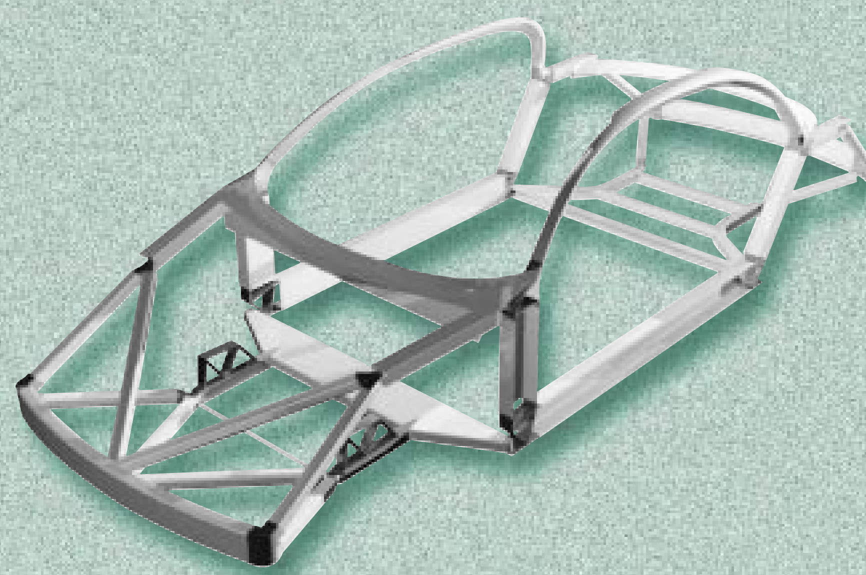
AUTOMOTIVE LIGHTWEIGHTING MATERIALS

2001
ANNUAL
PROGRESS
REPORT



U.S. Department of Energy

Energy Efficiency and Renewable Energy

Office of Transportation Technologies

ACKNOWLEDGEMENT

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Office of Advanced Automotive Technologies
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FY 2001

Progress Report for Automotive Lightweighting Materials

Energy Efficiency and Renewable Energy
Office of Transportation Technologies
Office of Advanced Automotive Technologies
Vehicle Systems Team

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1. INTRODUCTION

Automotive Lightweighting Materials R&D

As a major component of the Office of Advanced Automotive Technologies (OAAT) in the U.S. Department of Energy's (DOE's) Office of Transportation Technologies (OTT), the Automotive Lightweighting Materials (ALM) Program focuses on the development and validation of advanced lightweight materials technologies to significantly reduce automotive vehicle body and chassis weight without compromising other attributes such as safety, performance, recyclability, and cost. Through many of its technology research programs, OAAT has supported the government/industry Partnership for a New Generation of Vehicles (PNGV) since its inception. The PNGV leadership is now reevaluating the partnership goals to identify changes that will maximize the potential national petroleum-savings benefit of the emerging PNGV technologies. When these PNGV goal changes have been defined, the OAAT will adjust the focus of its technology research programs accordingly.

The specific goals of the ALM program are (1) by FY 2000, develop and validate materials technologies that, if implemented, will enable a reduction in automobile body and chassis weight by 50% relative to a 1994 baseline automobile, at 1.5 times the cost of using the baseline materials, while maintaining safety, reliability, and recyclability; and (2) by 2004, improve materials technology to enable a 50% reduction in the weight of automobile body and chassis components, and a 40% reduction in the overall vehicle weight (PNGV target), while achieving cost competitiveness with conventional materials.

The program is pursuing five areas of research: cost reduction, manufacturability, design data and test methodologies, joining, and recycling and repair. The single greatest barrier to use of lightweight materials is their high cost; therefore, priority is given to activities aimed at reducing costs through development of new materials, forming technologies, and manufacturing processes. Priority lightweight materials include aluminum, magnesium, titanium, and composites such as metal-matrix materials and glass- and carbon-fiber-reinforced thermosets and thermoplastics.

Collaboration and Cooperation

The ALM Program collaborates and cooperates extensively in order to identify and select its research and development (R&D) activities and to leverage those activities with others. The primary interfaces have been and still are with the Big Three domestic automotive manufacturers, namely the PNGV Materials Technical Team, the Automotive Composites Consortium (ACC) and the United States Automotive Materials Partnership (USAMP). This collaboration provides the means to determine critical needs, to identify technical barriers, and to select and prioritize projects. Other prominent partners include such organizations as the Aluminum Association, the American Iron and Steel Institute, the American Plastics Council, the Vehicle Recycling Partnership, the Society for the Advancement of Materials and Process Engineering, the International Magnesium Association, the International Titanium Association, and the Auto Parts Rebuilders Association. The program also coordinates its R&D activities with entities of other U.S. and Canadian federal agencies. Interaction with the DOE Office of Industrial Technologies (OIT) and Office of Heavy Vehicle Technologies (OHVT) and with the Department of Natural Resources of Canada is especially important by virtue of overlaps of interests in lightweight materials.

Once selected, R&D projects are pursued through a variety of mechanisms, including cooperative research and development agreements (CRADAs), cooperative agreements, university grants, R&D subcontracts, and directed research. This flexibility allows the program to select the most appropriate partners to perform critical tasks. The ALM efforts are conducted in partnership with automobile manufacturers, materials suppliers, national laboratories, universities, and other nonprofit technology organizations. These interactions provide a direct route for implementing newly developed materials and technologies. Laboratories include Albany (Oregon) Research Laboratory, Ames Laboratory (Ames), Argonne National Laboratory (ANL), Lawrence Berkley National Laboratory (LBNL), Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Oak Ridge National Laboratory (ORNL), Pacific Northwest National Laboratory (PNNL), and Sandia National Laboratories (SNL). PNNL manages the Northwest Alliance for Transportation Technologies, drawing on the expertise and developments in the Northwest. ANL oversees recycling efforts, and ORNL provides overall technical management, including management for the DOE cooperative agreement with USAMP.

Research areas and responsible organizations

Coordinated area	Organization
Production and fabrication of aluminum	The Aluminum Association, OHVT, OIT, Natural Resources of Canada (NRCAN)
Production and fabrication of magnesium	International Magnesium Association, NRCAN
Recycling, reuse, repair of automotive parts and materials	Auto Parts Rebuilders Association, OIT, Vehicle Recycling Partnership
Fabrication of steel and cast iron	American Iron and Steel Institute, the Auto/Steel Partnership
Fundamental materials research	DOE Office of Energy Research, National Science Foundation
High-volume composite processing	Department of Commerce—National Institute of Standards and Technology’s Advanced Technology Program
Electric and hybrid vehicle technology program	Department of Transportation
Materials research for defense applications	Department of Defense
Materials research for space applications	National Aeronautics and Space Administration
Crashworthiness	Department of Transportation
International vehicle material R&D	International Energy Association
Production and fabrication of titanium	The International Titanium Association
Production and fabrication of composites	American Plastics Council

FY 2001 Accomplishments

To meet the goals set forth for the program, the ALM Program is developing materials and material processing technologies; validating these technologies through fabrication and evaluation of representative, non-proprietary test components; and developing adequate design data to facilitate their beneficial application. The research is balanced between nearer-term objectives and longer-term, higher-risk research. As the technical barriers are removed, the technology is made available to industry. Because of the broad area of research and the limited resources, projects have been selected to overcome the most significant barriers within the technical areas that the materials community considers higher-risk but that, if successfully developed, would result in significant progress toward program goals.

Composite Materials

Low-Cost Carbon Fiber

One of the highest-priority technical needs is the development of low-cost carbon fibers. Carbon fiber composites are the lightest material available for making primary automotive structures. Their use in automotive structural composites could reduce the body and chassis weight of vehicle components by up to 67%. Currently, the use of these advanced lightweight composites in primary automotive structures is limited because carbon fibers are much more expensive than traditional automotive materials. To address this challenge, research is being conducted to reduce the cost of materials being converted into carbon fiber and the cost of the production processes used for making carbon fiber.

Originally, the program had four projects aimed at developing low-cost carbon fiber precursors. During the last year, that field was down-selected to the two most promising technologies. The two choices were a nearer-term, medium-risk project using commodity-grade polyacrylonitrile (PAN) precursor that could reduce finished fiber costs by $1.60 per pound; and a longer-term, higher-risk project using lignin-based precursors that could reduce the price of carbon fiber to near $3.00 per pound.

Textile-Grade PAN Precursors

During 2001 a method was successfully developed to produce lower-cost carbon fiber precursor using commodity-grade PAN. Commodity textile-grade PAN is commercially available in large quantities at about half the cost of large-tow PAN precursors. Pretreatment and processing methods were developed using textile-grade PAN precursors. Appropriate mechanical and chemical properties were determined, and a laboratory-scale proof of concept was demonstrated. The next step will be to scale this technology up for industrial applications. These new carbon fiber precursors should complete full-scale development within 18 months and be ready for implementation into production facilities shortly afterward. The resulting reduction in the price of carbon fiber is projected to be $1.60 per pound.

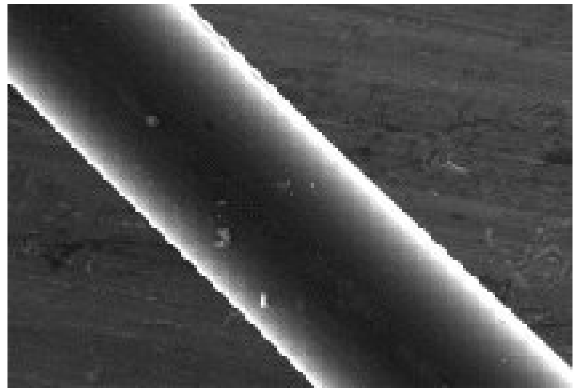
Textile PAN being processed into carbon fiber.

Before- and after-processing views of carbon fiber made from textile-grade PAN.

Lignin-Blend Carbon Fiber Precursors

It is expected that commodity-grade textile precursor will allow carbon-fiber–based composites to penetrate the automotive market at significant levels by allowing the $5.00 per pound goal for carbon fiber to be met. However, for carbon fiber composites to become more prevalent as structural materials, prices of $3.00 per pound must be approached. The second precursor project seeks to reach that goal by using lignin as the feedstock. Lignin is a by-product of papermaking and is often burned in paper mills to recover its energy value. It can be purchased on the open market for $40 per ton. During this year, very inexpensive feedstock materials were desalted and purified, and processing schedules were determined. Lignin blends were successfully spun, stabilized, carbonized, and graphitized in small batches. It was demonstrated that good-quality carbon fiber can be made from lignin feedstocks and that the process is economically viable.

Carbonized fibers from lignin blends.



As-carbonized surface of a polyethylene oxide/ lignin-blend fiber.

Microwave-Assisted Plasma Processing of Carbon Fiber Precursors

In addition to developing lower-cost precursors, the program has been working on developing lower-cost production methods for converting precursors into carbon fiber. In previous years, a process was demonstrated that used microwave energy to produce carbon fibers; it required processing times that were only a fraction of those necessary using conventional methods. The tow size of carbon fibers was small; and the process, after initial development, was small and the output line speed still much slower than desired. During the past year, the microwave-assisted plasma process for manufacturing carbon fiber using microwave energy was greatly improved. Scale-up of the process was conducted this year using large-tow (48,000-filament) precursors. Line speeds were increased from 4 inches to 48 inches per minute, and fiber quality was improved to match that of fiber commercially available from carbon fiber vendors. Development of this technology should be completed in the next 18 months.

Microwave-assisted plasma carbon fiber production unit.

Commercialization and Advancement of the P4 Process

The programmable powder preform process (P4) is a low-waste, high-speed method of making fiber performs that was developed under this program. (Preforms are the fiber mats that are infiltrated with a resin to make a composite.) In earlier work, the economic viability of P4 was demonstrated using glass fiber; this year two variations of this technology were commercialized on vehicle platforms. In addition, this year the process for making high-quality composite performs using carbon fiber was advanced past the proof-of-concept stage. The project identified the best fibers and made the first carbon fiber composite performs. The preforms were then molded into composite test panels.

Aston Martin Vanquish with front quarter panels made using P4.

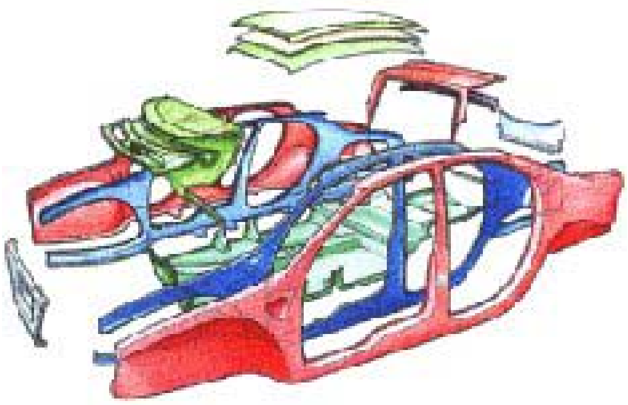
Chevy 1500 with pickup truck bed made using P4.

First Durability Guidelines Delivered for Designing with Carbon Fiber Composites
An ongoing program effort is to develop the durability-based design guidelines necessary to enable automotive engineers to design automotive structures made from composites and be assured that the structures will last for the life of the vehicle. To do so, it is necessary to account for the effects of all the various load histories, temperature extremes, and environmental stressors that a vehicle may see and then develop an understanding of their synergistic effects. In previous years, design guidelines were developed for glass-fiber composites. During this year, the program completed the first durability design guideline for carbon fiber composites. The first design guidance manual was distributed to all three domestic automakers; it includes the synergistic effects of multiple-load, environmental, and temperature histories on carbon-fiber–based composite materials used for primary automotive structures.

Completed Phase 1 of Focal Project 3
The program integrates all development projects into large demonstration projects that demonstrate that structures can be manufactured and assembled within required cycle times and at required costs while meeting performance targets acceptable to industry. The third focal project is the development of an all-carbon-fiber composite body-in-white. During this year, phase 1 of Focal Project 3 was completed. The resulting structure met or exceeded all design requirements and can be cost-effectively manufactured from carbon fiber composites. It is more than 60% lighter than the baseline steel structure. The figure on page 6 shows the structure and the amount of material used in it.

Metals and Alloys

Low-Cost Powder Metallurgy for Particle-Reinforced Aluminum Composites
Powder metallurgy particle-reinforced aluminum (PMPRA) composites exhibit a number of benefits for automotive applications. Unfortunately, several barriers, including cost, durability, and manufacturability, have limited their widespread implementation. A project led by USAMP has focused on developing the manufacturing methods, machining technology, and design methodologies necessary to establish the commercialization potential of these materials. The PMPRA project is nearing completion. Major process and material development milestones were achieved in FY 2001. The press and sinter sub-team has fabricated an aluminum-silicon-alloy PMPRA transmission oil pump gear set with new dimensionally accurate tooling to demonstrate the newly developed technology. Parts have passed two major durability hurdles, including wear tests. In addition, a set of sintering process conditions based on correlations to wear-resistant alloy microstructures and calorimetry measurements have been developed. The direct powder forging sub-team has established the feasibility of making connecting rods from PMPRA composites by demonstrating the fabrication of components in small lots using existing (non-optimized) tooling with an aluminum-SiC powder



Phase 1 results:
- 67% mass savings over baseline
- Bending stiffness exceeded 20%
- Torsional stiffness exceeded 140%
- Durability and abuse load cases satisfied
- Manufacturing strategy developed

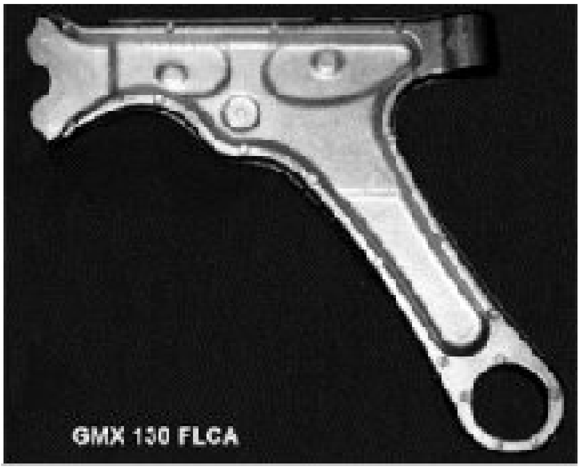
Materials/mass distribution:
- Chopped carbon—54.8 kg
- Carbon fabric—17.7 kg
- Core–3.2 kg
- Adhesive—1.6 kg
- Inserts—8.8 kg

blend and cold-isostatically-pressed preforms. The results of room-temperature tensile and fatigue tests and microstructural characterization indicate a high probability of achieving design and performance strengths. These activities have demonstrated a high potential for using powder metallurgy aluminum composite materials in both moderate-strength and high-strength/high-temperature applications in high-volume production.

Prototype 600-kg modular mixing system built by MC-21: melter (rear), mixer (middle), and holding furnace (foreground).

Low-Cost Cast Aluminum Metal Matrix Composites
In addition to the activity focused on powder-metallurgy–based metal matrix composites (MMCs), efforts have been supported in the development of low-cost cast aluminum MMCs. This project seeks to make aluminum MMC materials more attractive for widespread use in automotive applications by reducing the costs of the raw materials, the processing methods, and the machining and finishing operations. Initial activities have resulted in the selection and validation of lower-cost SiC reinforcement materials and the scale-up of the mixing and holding system for producing low-cost castings from 60 to 600 kg. Other activities focused on the development of innovative shape-casting and molding technologies—including centrifugal casting, pressure-infiltrated preforms, and ceramic-based composite samples—to produce aluminum MMC automotive braking systems. With the promise of a lower-cost aluminum MMC material, a less expensive compositing process, and supporting work on downstream processing, future efforts will focus on innovative brake system designs that take advantage of the unique properties of MMC materials to produce cost-effective, lightweight replacements for cast iron systems.

Improved A206 Alloy for Automotive Suspension Components
Aluminum alloy A206 is significantly stronger than the aluminum casting alloys normally used at present and has mechanical properties approaching those of some grades of ductile iron. It also has excellent high-temperature tensile and low-cycle fatigue strength. Consequently, this material has potential for use in a number of applications to reduce vehicle weight. Cost savings may also result because less material would be required to provide the strength needed for the application. In spite of its excellent properties, A206 alloy has not been used in significant quantities because of its propensity for hot cracking. The ALM Program is supporting a project team, under the direction of USAMP, to investigate the potential for improving the castability of A206 using a better method of grain refinement and a new ultrasonic inspection technique to

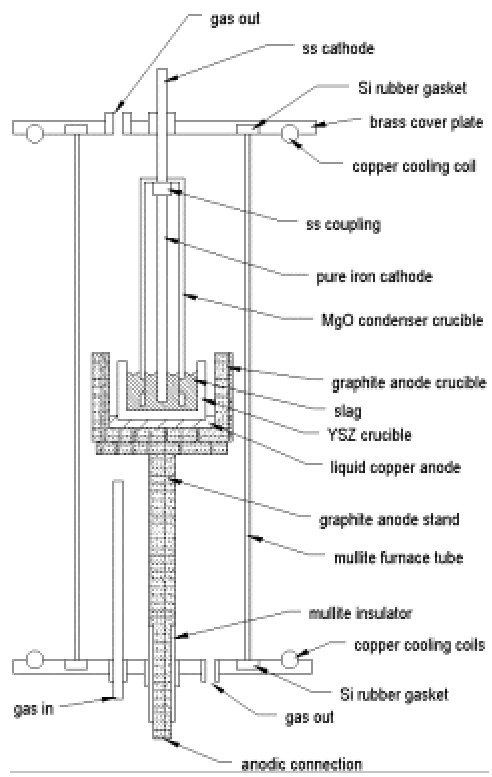


Lower control arm castings produced in Phase 1 trials. (Left) A front lower control arm for a GM Grand Am. (Right) A rear lower control arm for a GM Cadillac.

test for the presence of hot cracks. Suspension components have been successfully cast using a new experimental version of A206. Initial results indicate higher resistance to hot cracking and mechanical properties superior to those of conventional A356 alloy parts, even though the casting process had not been optimized. Further experiments were conducted to establish optimum process parameters for the production of more than 100 castings with little or no porosity under production conditions. Continued testing is expected to result in a database that the automotive industry can use to design and cast lighter-weight, higher-strength components.

Solid Oxygen-Ion-Conducting Membrane Technology

Because of their low densities, magnesium alloys have the potential for application in many automotive components. One of the barriers to the increased use of magnesium in automotive applications is the relatively high cost of the primary metal. The current methods for producing magnesium are either electrolysis from a halide electrolyte bath, which requires extensive and expensive feed material preparation, or metallothermic reduction at high temperatures, which involves expensive metal reductant. The ALM Program supports efforts aimed at developing a solid oxygen-ion-conducting membrane (SOM) process for producing magnesium from its oxide. The SOM process has the potential to be more economical, less energy-intensive, and more environmentally sound. In work to date, high magnesium production rates have been achieved in laboratory-scale demonstrations without damaging the yittria-stabilized zirconia membrane or other components of the cell. Steady state operation of the laboratory-scale cell has indicated superior performance compared with state-of-the-art processes for magnesium production and with Hall Cell aluminum production. Experiments to determine parameters critical to the potential scale-up of this technology for commercial magnesium production will continue.

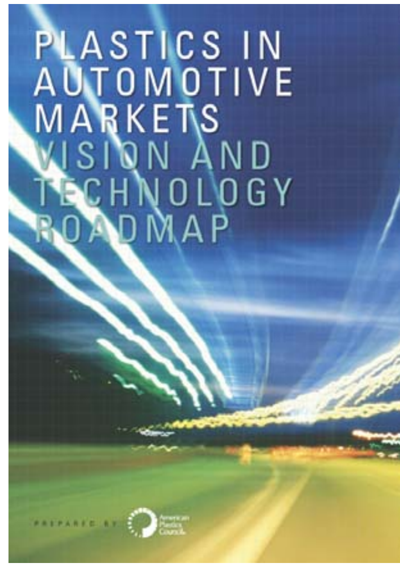


Apparatus for the laboratory-scale electrolysis and collection of magnesium metal using the SOM technology.

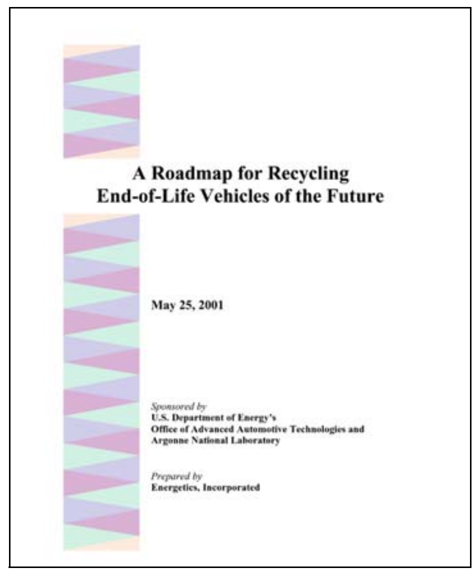
Roadmapping and R&D Planning

A report on initial planning was completed with the American Plastics Council (APC). The report was begun at a May 2000 workshop. A report on recycling, based on a September 2000 workshop, was completed as a joint effort of several interested organizations, such as the APC, the Aluminum Association, the American Iron and Steel Institute and the Auto Parts Rebuilders Association. The planning report is particularly notable in that it seems to have stimulated further planning on the part of the plastics-supplier industry in this area of plastics for the automotive market.

The first comprehensive program review by an independent panel of outside experts (outside the program) was completed. The panel consisted of four retired materials and manufacturing researcher/manager experts from the auto industry, one from the aluminum industry, one from the steel industry and one from the U.S. Department of Defense.



Planning report completed with American Plastics Council.



Roadmapping report for recycling of vehicle materials.

Future Direction

FY 2001 marked the completion of a ramp-down of R&D thrusts begun around FY 1994–1998, and completion of a ramp-up of new thrusts begun around FY 1999. Most projects—with the exception of one effort on casting of unreinforced aluminum and all projects on glass-fiber-reinforced polymer-matrix composites (PMCs)—were completed or began winding down toward final wrap-up in FY 2002. New projects on magnesium casting and on carbon-fiber-reinforced PMCs came up to full funding levels. These new efforts will demand most of the funds available in FY 2002 and most funds likely to be available in FY 2003. FY 2001 claims to have developed titanium production processes with potential costs compatible with some (higher-value) automotive applications were confirmed. Some of the titanium being produced will be assessed for suitability for fabrication in FY 2002 in anticipation of further escalation of the titanium activities in late FY 2002 or FY 2003. These events represent notable shifts away from aluminum—the prime candidate for meeting the original PNGV goal of a cost-competitive 40% weight reduction—to less-explored and higher-risk materials thought capable of enabling even greater weight reductions over the longer term.

Seven new projects on forming of components out of sheet steel or aluminum began in FY 2001, as did five on joining of steel, aluminum, and magnesium to one another and to PMCs. This effort signifies another programmatic trend toward downstream manufacturing, as opposed to the emphasis on upstream aluminum sheet production and alloy development during the earlier years of the ALM Program. The multi-material nature of the joining projects highlights still another trend that arises from recent thinking that various materials will compete for use in future vehicles, without domination by just one, steel, as in the past.
FY 2002 will be a year for planning future new efforts in the areas of polymers and recycling, based on the FY 2000 and FY 2001 roadmapping efforts (see Roadmapping and R&D Planning), that could be addressed as the new thrusts mentioned wind down. Further R&D efforts in the areas of MMCs, glass, and nondestructive evaluation will be considered as the present efforts in these areas wind down in FY 2002.

2. AUTOMOTIVE ALUMINUM R&D

A. Forming Advances for Aluminum Automotive Components and Applications

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Contract No.: XSU544C

Objectives

- Develop and demonstrate advanced forming processes to improve the formability of aluminum sheet for automotive panel applications.
- Improve the aluminum sheet metal stamping process through the development of a method for controlling the flow of metal into the tooling cavity during the stamping process through the application of variable binder loads.

OAAT R&D Plan: Task 2; Barriers A, B

Accomplishments

- Conducted single-cylinder testing and development at Erie Press to resolve a technical difficulty in controlling the binder cylinders that was uncovered during the full-scale press demonstration trials.
- Successfully reprogrammed the binder cylinder control system to obtain satisfactory results under actual press conditions.

Future Direction

- Ship unit to Troy Design and Manufacturing (TDM) for next phase press evaluations.
- Complete a final report on the technical and economic evaluations.

Introduction

More extensive use of aluminum on vehicles has been identified as an important means of reducing tailpipe emissions and fuel consumption. This contract comprised two distinct projects, process/press optimization and warm forming. Work on the warm forming project was completed in 2000. This report details progress on the process/press optimization project.

Research Results

The full-scale press demonstration trials at Troy Design and Manufacturing (TDM) in 2000 uncovered a technical difficulty in controlling the binder cylinders in the last 1–2 inches of the press stroke. The project team determined that the control system required re-programming using the control valves' actual pressure drop at various flow rates. The optimal procedure to accomplish this was to pull the full-size binder control system from the press at TDM and work on a single cylinder at Erie Press.

One complete binder cylinder assembly was removed from the fixture and placed into a test stand. The test stand consisted of a larger hydraulic cylinder directly opposed to the binder cylinder. The binder cylinder received its electrical commands and hydraulic fluid from the existing binder control system. The control of the opposed cylinder was derived from an external source. This control allowed the velocity of the opposed cylinder to be programmed.

A servo valve opening\flow\pressure drop spreadsheet was acquired from the valve manufacturer. These data allowed modification of the control algorithm to predict the valve opening required while maintaining binder cylinder force during the slower press velocities.

Initial tests were performed with the opposed cylinder traveling at constant velocities. The results were favorable, especially at the slower velocities that were not under control during the in-forming trial at TDM.

For the next test, we programmed the opposing cylinder to mimic the velocity curve of the press at TDM. It was observed that the binder cylinder pressure fell further below the set point as the velocity was reduced. The problem was traced to binder cylinder speed reference calculation. The speed reference was derived by examining the feedback from the binder fixture analog position sensor and calculating the rate of change during every controller scan. The position sensor's feedback contained a substantial amount of electrical noise that required us to incorporate digital filtering to produce a stable result. This filtering also dampened the speed reference signal's response to a change in velocity. Then a digital encoder and PC card were added to allow acquisition of digital (noise-free) position information at a 20-μs rate.

Testing resumed with the new encoder and PC card installed and the opposing cylinder pushing the binder cylinder down at the press velocity. It was observed that when the binder cylinder was first contacted, pressure control was satisfactory. However, as the stroke increased, the cylinder pressure became unstable. It was surmised that the sensitivity of the new speed sensor allowed the binder cylinder control system to respond much faster than did the previous method, and that it was compensating for the velocity changes (increasing and decreasing) as the opposed cylinder tried to maintain the press velocity curve. At that time, it was concluded that the simple hydraulic cylinder setup was not capable of simulating the inertial characteristics and velocity curve of a large mechanical press.

Next, the single binder cylinder assembly was installed in a 4000-T forging press. This test would eliminate the possibility that the binder cylinder pressure changes would affect the velocity of press and thus induce system instability. The press was operated at about 1/3 its normal speed in an attempt to match the TDM press velocity curve. The initial tests were encouraging, but the feed forward algorithm (derived from the valve manufacturer's data) was causing the pressure to rise significantly above the set point as the press reached bottom dead center. The data gathered from this test allowed the team to derive the proper equation needed to improve the feed forward algorithm.

With the new equation in place, testing resumed with acceptable results. Successful pressure profiles were obtained throughout the entire press stroke for the three pressure trajectories required from the finite element modeling of the process. Figure 1 shows the three prescribed pressure curve trajectories for groups of cylinders. Figures 2–4 show the actual single-cylinder pressure output under actual press conditions. (Note that 14.22 psi = 1kN.) This test completed the single-cylinder testing and development at Erie Press, and the unit was prepared to ship back to TDM for press trial reevaluations. At this time, the program management transferred to Mahmoud Demeri of Ford Motor Company.

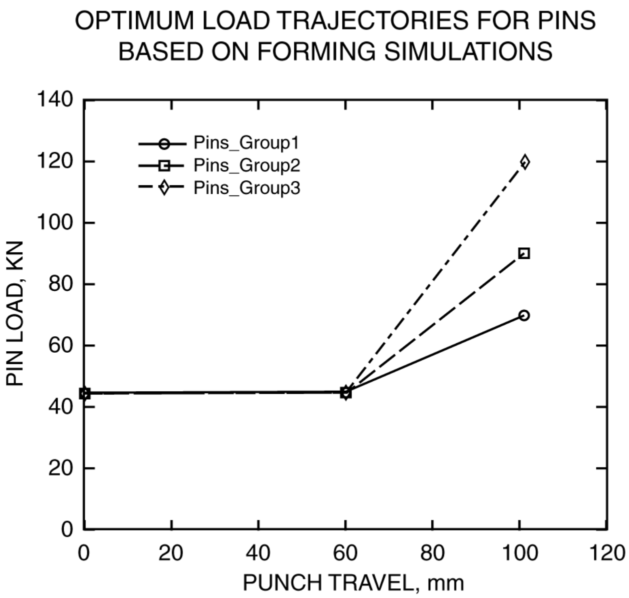

Figure 1. Recommended load trajectories from finite element modeling.

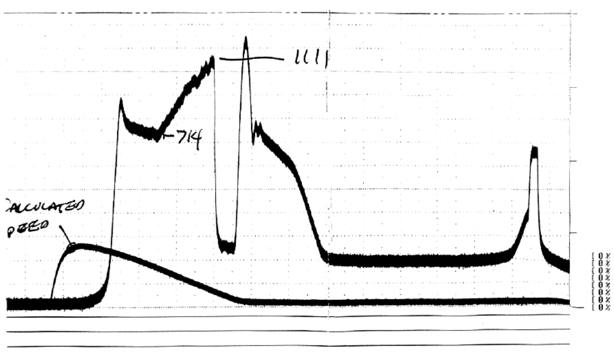

Figure 2. Actual cylinder pressure for pin group 1.

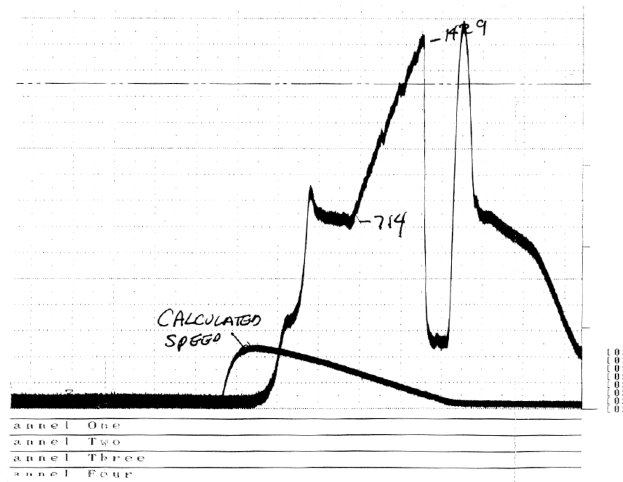

Figure 3. Actual cylinder pressure for pin group 2.

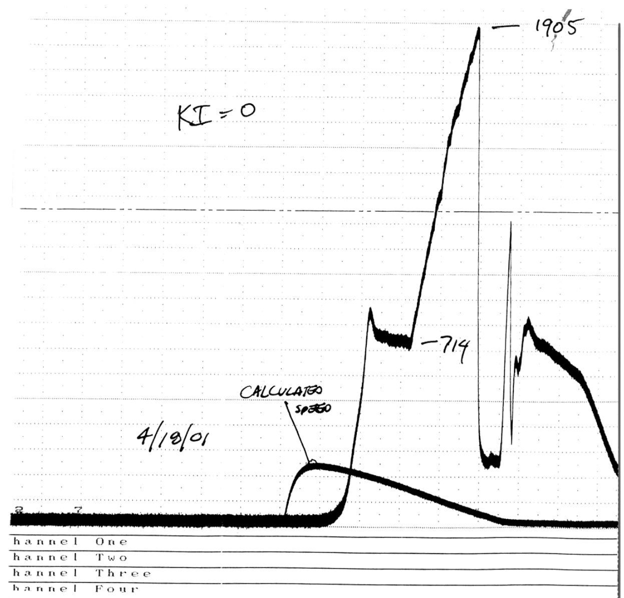

Figure 4. Actual cylinder pressure for pin group 3.

B. Active Flexible Binder Control System for Robust Stamping

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objective

- Develop and demonstrate, on an industrial scale, an optimized closed-loop flexible binder control system that can be installed in presses to improve the quality, reduce the variability and maintain the accuracy of stampings made from aluminum alloys and ultra-high-strength and stainless steels. The system will also reduce the cost for developing and setting production tools.

OAAT R&D Plan: Task 2, 13; Barrier B

Approach

- Conduct open-loop control demonstration of flexible binder technology.
- Develop methodology and guidelines for designing and building flexible binders.
- Develop computer simulation and process optimization capabilities for flexible binders.
- Develop a closed-loop flexible binder control system with appropriate sensors.
- Demonstrate closed-loop control of the flexible binder system on an industrial part.
- Evaluate technical and economic feasibility of flexible binder technology.

Accomplishments

- Completed project team formation and selection of academic and industrial partners.
- Initiated the project on March 20, 2001.
- Conducted monthly project, steering committee, and task meetings.
- Received from the Institute for Metal Forming Technology the design guidelines for segmentation of lower flexible binders and structural designs for rigid lightweight upper binders.
- Developed an original equipment manufacturer–approved 5-phase project plan for development of the computer simulation and process optimization capabilities. (Development work done by Ohio State University).
- Built tooling for testing single-point binder force control for axi-symmetric parts.

- Conducted computer simulation on blank holder force (BHF) optimization of the conical cup.
- Evaluated geometry- and energy-based wrinkling detection methods.
- Investigated ductile fracture and thinning as criteria for splits.

Introduction

Significant weight savings can be achieved by replacing parts made from mild steel with those made from lightweight materials (aluminum and magnesium alloys) and high-specific-strength materials (ultra-high-strength and stainless steels). Such materials are less formable than mild steel, and parts made from them lack dimensional control because of the significant amount of springback that they produce after forming.

Traditional stamping leaves no flexibility in the stamping process for using difficult-to-form materials and for responding to process variations (e.g., lubrication, material, die wear, blank placement) that can lead to stamping inconsistencies or even failure. It has been found that failure by wrinkling or tearing is highly dependent on the magnitude and trajectory of the binder force. Recently, dynamic variation of the binder force during the forming stroke has been shown to affect formability, strain distribution, and springback. Optimal forming trajectories can be obtained under constant and variable binder force conditions, but there is no guarantee that process variables will remain constant during the stamping process. Specifying a binder force trajectory is not easy because the part shape changes during forming. Also, stresses in the part cannot be determined because the coefficient of friction is not a controllable quantity and it varies from location to location. Therefore, the forming process must be controlled and a closed loop system with an appropriate local control parameter (friction, draw-in) must be used to track a predetermined optimum control parameter trajectory.

Scope

The project scope involves using flexible binder control technology in conjunction with innovative tool designs and closed-loop control to produce robust processes for stamping aluminum and high-strength-steel automotive panels. The focus of this project is to implement binder and feedback process control in the stamping industry to extend the forming window of difficult-to-form materials, thereby increasing the robustness of the forming process and improving the quality and consistency of stampings. This technology will use computer simulation and process optimization to predict the optimum binder force trajectory on hydraulic cushions with programmable multi-pins on mechanical transfer presses.

Details of Project and Team Formation

Significant effort was spent in detailing project tasks and in selecting team members. A steering committee for the project was formed, task leaders were identified, a project administrator was hired, and academic and industrial partners were selected. The project was initiated on March 20, 2001. Monthly project and steering committee meetings were conducted and a number of task meetings were convened. The meetings reflected a high level of participation and an increased interest in binder control technology. Tasks 1, 2 and 3 are being conducted simultaneously.

Task 1: Conduct Open-Loop Trials Using Flexible Binder and Liftgate Tooling

Task 1 was inherited from an earlier U.S. Automotive Materials Partnership/DOE project that ended in December 2000. Proceeding with Task1 was contingent upon receiving a functional binder control unit from that project. The control unit was designed and built by Erie Press Systems, and demonstration trials were planned at Troy Design and Manufacturing (TDM). Initial tests showed that the control unit did not function properly; therefore, it was sent back to Erie Press to find and fix the problem. Erie Press reprogrammed, reassembled, and retested the control unit and returned it to TDM for installation in a mechanical press to conduct tryouts on the liftgate inner tooling. Tests conducted in March 2001 showed a serious problem with controlling individual hydraulic cylinders in the binder control unit. The problem has been identified as a control problem: pressure within the hydraulic cylinders could not be achieved in a mechanical press with a simple proportional integral derivative (PID) control because the punch velocity is not constant as it is in hydraulic presses. A number of options are being considered to surmount the problem. It is important to realize that Task 1 cannot proceed until a functioning, reliable binder control unit becomes available.

Task 2: Develop Guidelines for Designing and Building Flexible Binders

The Institute for Metal Forming Technology of the University of Stuttgart in Germany pioneered the development of flexible binder technology. The Institute was selected to work on Task 2. Flexible binder technology uses innovative tooling designs (Figure 1). The lower binder has cone-shaped segments to provide local binder force control, and the upper binder employs structures to provide rigidity and light weight. Building flexible binders requires optimization of the tool designs to achieve flexibility in the lower binder and lightweight rigidity in the upper binder.

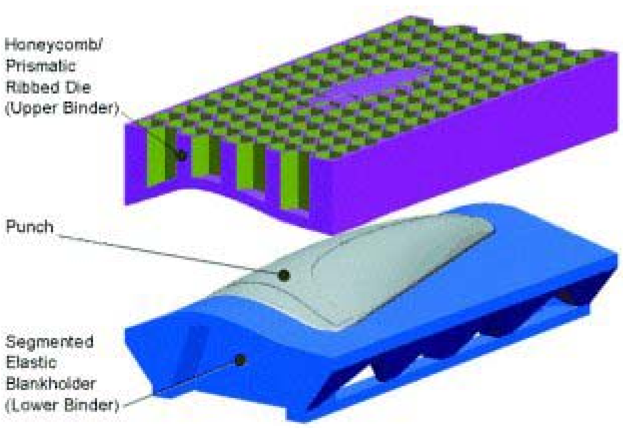

Figure 1. Flexible binder technology with flexible lower and rigid upper binders.

Task 2 objectives are to develop guidelines for designing and building flexible binders. Specific areas of development include these:

- Binder segmentation
- Cone shape, numbers, and size
- Lower and upper binder material
- Effect of stress concentration and fatigue on flexible binders
- Allowable stresses and strains in flexible binders
- Computer-assisted designs for flexible binder tools
- Hydraulic cylinder and pin sizes, capacity and characteristics

Task 2 is proceeding according to plan. Some of the accomplishments include design guidelines for convex, straight, and concave segments of lower flexible binders (Figures 2–4) and honeycomb, triangular, and circle designs for rigid lightweight upper binders (Figure 5).

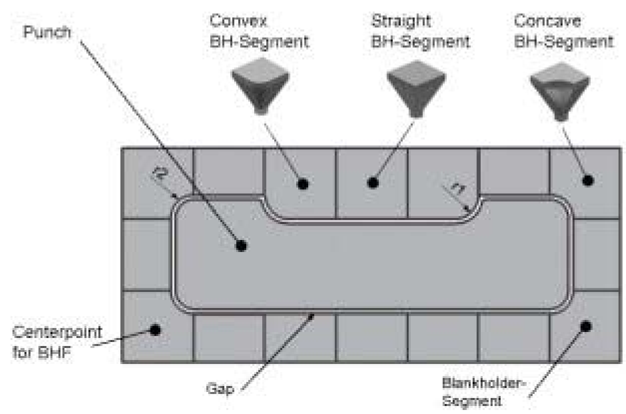

Figure 2. Design for lower binder.

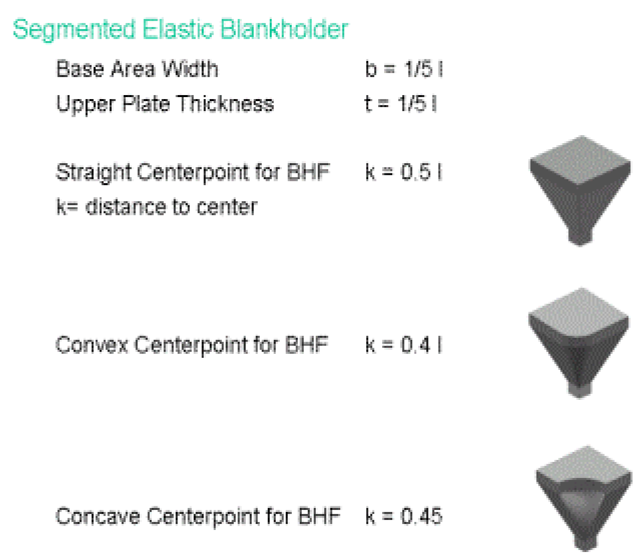

Figure 3. Designs for lower binder segments.

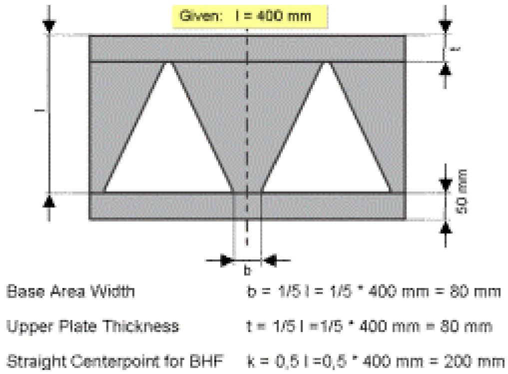

Figure 4. Designs for lower binder segments.

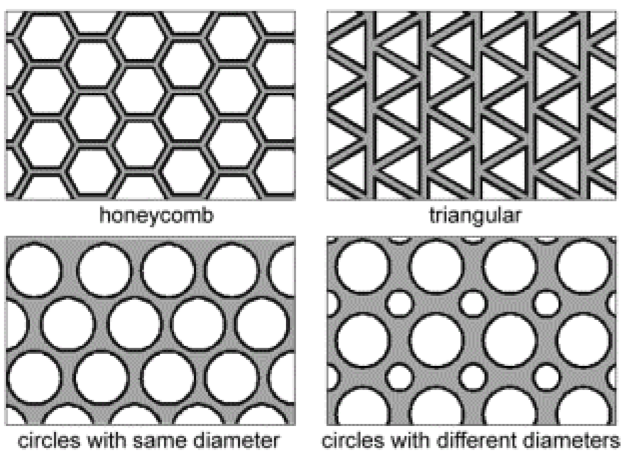

Figure 5. Designs for upper binder construction.

Task 3: Develop Computer Simulation and Process Optimization for Flexible Binders

The Engineering Research Center for Net Shape Manufacturing of the Ohio State University was selected to work on Task 3 because of its expertise and the availability of equipment and facilities needed to complete the work. Ohio State developed a 5-phase project plan for Task 3 that was approved by the original equipment manufacturers:

Phase 1: State-of-art review in stamping simulation for binder force control
Phase 2: Single-point binder force control on axi-symmetric parts
Phase 3: Single-point binder force control on non-symmetric parts
Phase 4: Multipoint flexible binder force control
Phase 5: Technology transfer

Work on the task is proceeding according to plan. A literature survey was conducted of issues related to temporal binder force control, such as fracture prediction, wrinkling detection, and temporal binder force control strategies. Several fracture prediction and wrinkle detection methods, based on part geometry and energy, were analyzed. A conical cup-drawing tool was designed and manufactured for use in developing temporal binder force optimization algorithms and in verifying the wrinkle-detection and fracture-prediction methods. The accomplishments include

1. Design and manufacture tooling for conical cup drawing
2. Preliminary simulations on BHF optimization of the conical cup drawing
3. Investigation of wrinkling detection methods (geometry-based and energy-based)
4. Investigation of fracture prediction criteria (ductile fracture and thinning)

Results from the conical cup drawing simulations conducted to determine the optimal linear variable BHF profiles are shown in Figure 6.

The types of wrinkle detection methods evaluated in this investigation fall into two categories: (1) geometry-based and (2) energy based. Wrinkle types obtained in a conical cup are flange wrinkles and sidewall wrinkles. One of the geometry-based methods used is measuring wrinkle amplitudes at different cross-sections (Figure 7).

The evolution of wrinkle severity of a drawn cup is shown in Figure 8. Results show that the average wrinkle amplitude varies with the draw depth of the conical cup.

Computer simulations were conducted on the conical cup to determine the optimal constant BHF profiles and to verify wrinkle and fracture predictions. These simulations were conducted with several different constant BHF values to determine how the applied constant BHF values affect the maximum obtainable drawing depths. Figure 9 shows plots of maximum obtainable cup depths at different constant BHF levels for two wrinkle amplitudes, recorded when failures were detected. Flange and sidewall wrinkles occur at lower BHF levels (<20 KN). Sidewall wrinkle is the only type of failure that occurs at medium BHF levels (20 KN~140 KN); fracture is the only failure type when higher BHF levels (>140 KN) are applied. From Figure 9, it is seen that at a constant BHF of

Better than construction BHF 140 KN Resultant drawing depth = 45.6 mm (7% improvement in depth)	Worse than construction BHF 140 KN Resultant drawing depth = 35 mm

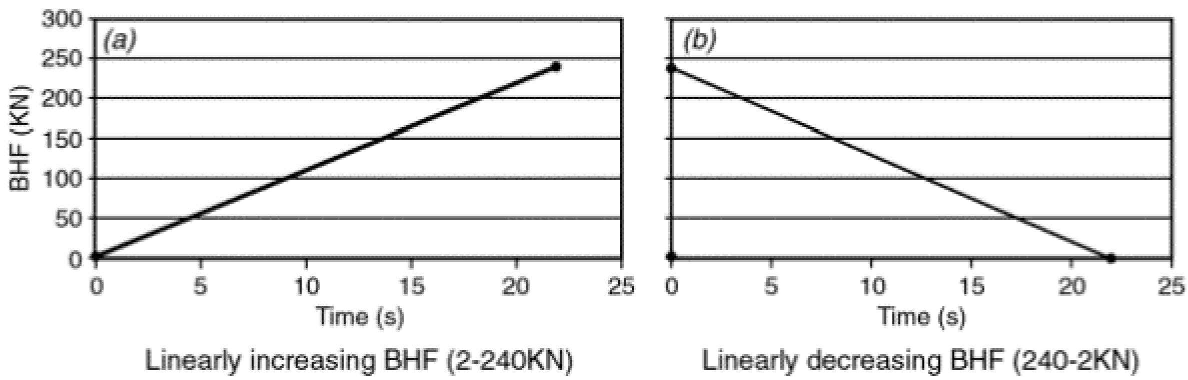

Figure 6. Linear variable blank holder force profiles used in finite element model simulations.

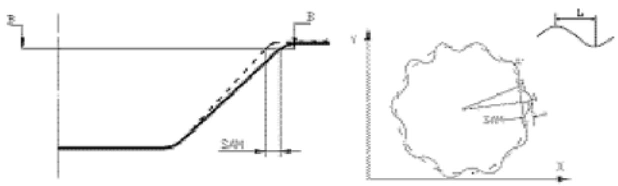

Figure 7. Wrinkle amplitude method.

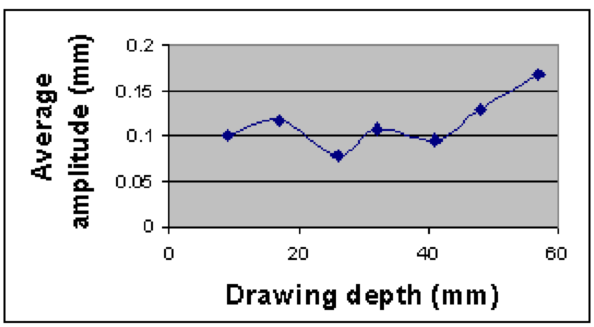

Figure 8. Wrinkle amplitude variation with draw depth in a conical cup.

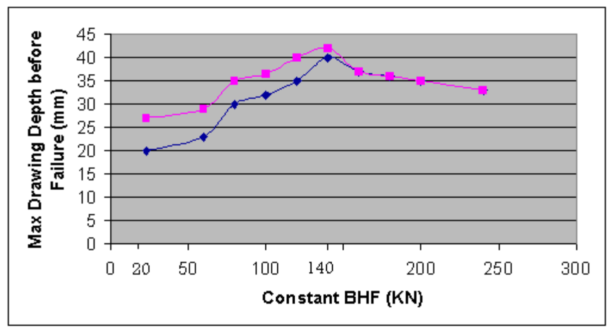

Figure 9. Effect of constant blank holder force on drawing depth of cup.

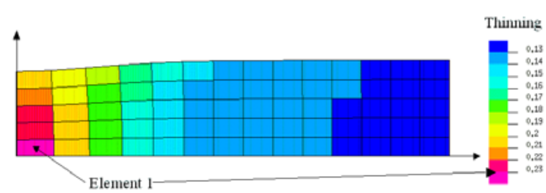

Figure 10. Thinning distribution on the tensile test sample (simulation).

140 KN, the maximum obtainable cup depth is 42 mm. In order to obtain a deeper cup (>42 mm), a non-constant BHF (i.e., varying BHF curve) must be used.

Using the stresses and strains obtained from the tensile test simulations and the ductile fracture constants, the ductile fracture criteria values were calculated for each element and each time step. Figure 10 shows the thinning distribution on a quarter mesh of the tensile test simulation when the ductile fracture criteria values become unity at element 1.

In order to investigate the effectiveness of the ductile fracture criteria, the fracture values (calculated at element 1 using the four fracture criteria) are plotted with respect to the engineering strain (along the longitudinal direction) in Figure 11. The thinning value (calculated at

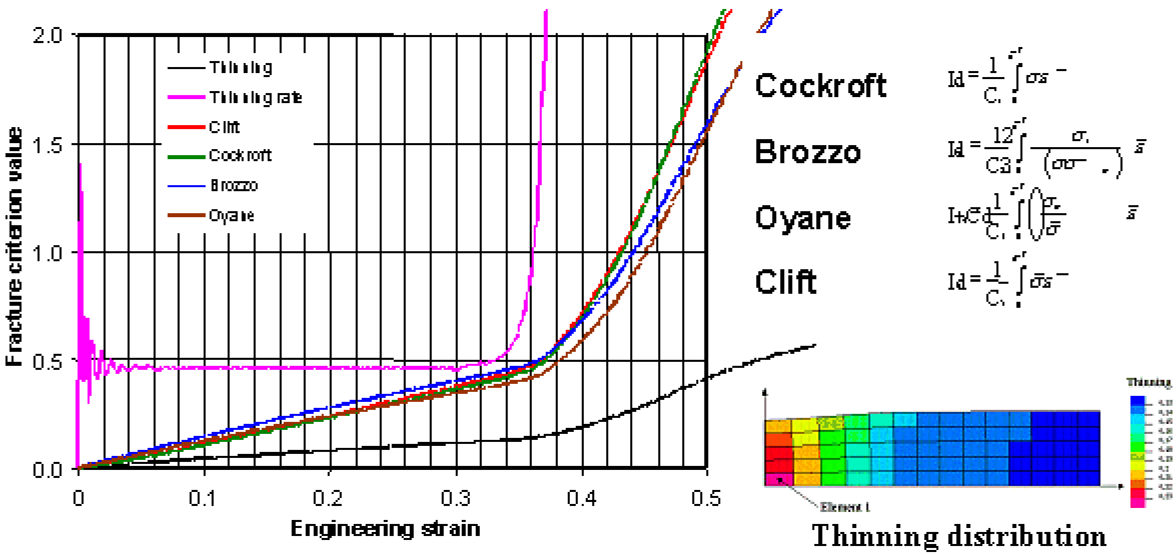

Figure 11. Fracture criterion values at element 1.

element 1) is also plotted. In Figure 11, ductile fracture criteria values become unity when the engineering strain is 0.43–0.45. This corresponds to thinning of 25–30%. It seems that these ductile fracture criteria can be used to predict fracture with reasonable accuracy.

Accomplishments for Task 3

- Literature survey on temporal BHF control and wrinkling and fracture detection methods were conducted and summarized.
- Conical cup tooling has been designed and built.
- Initial conical cup drawing simulations were conducted.
- Simulation results show that a linearly increasing BHF profile can increase the maximum drawing depth of the conical cup, compared with the cup drawing with a constant BHF.
- Four wrinkling detection methods have been introduced.
- Wrinkle amplitude and distance to ideal surface methods were found to be good in reflecting both (1) the detailed information of individual wall wrinkles and (2) the global severity of wall wrinkles of drawn parts. The surface area ratio method can only describe the global severity of a wrinkled part. The Nordlund's energy method can only reflect the location of individual wrinkles.
- Four ductile fracture criteria have been evaluated by conducting simulations of the tensile test and the conical-cup drawing operations.
- The use of the ductile fracture criteria in predicting splits in BHF optimization seems promising. However, because of the lack of experimental data to determine damage values for different materials (aluminum alloys and high-strength steels), a thinning limit will be used, for now, in the development of BHF optimization algorithms.

The following is the status of subtasks performed by Ohio State University:

- Development of wrinkle detection
  Geometry-based—100% complete
  Energy-based—100% complete
  Verify wrinkle detection with the conical cup drawing—50% complete
- Development of fracture detection
  Thinning/thinning rate—100% complete
  Forming limit diagram/forming limit stress diagram—50% complete
  Ductile fracture criteria—100% complete
  Verify fracture detection with the conical cup drawing—50% complete

- Determination of temporal binder force (conical cup)
  Optimization techniques—10% complete
  Closed-loop control techniques—10% complete
- Variable binder force experiments with the conical cup—10% complete

C. Warm Forming of Aluminum—Phase 2

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objective

- Prove the manufacturing and economic feasibility of the warm forming process for aluminum for industrial applications in a mass production environment:
  - — Improve the formability of the aluminum alloy.
  - — Develop a washable lubricant for use with heated blanks and dies.
  - — Optimize the temperature distribution of the die.
  - — Develop a rapid heating system for blanks.
  - — Optimize the process design and layout.

OAAT R&D Plan: Task 2, 13; Barriers A and B

Approach

- Develop alloys:
  - — Select candidate alloy compositions.
  - — Determine microstructures after heating.
  - — Perform tensile tests.
  - — Conduct preliminary forming tests.
  - — Analyze test results.
- Develop lubricant system:
  - — Select lubricant candidates.
  - — Perform screening tests.
  - — Design and lab test lubricant application system.
  - — Install and test system in forming facility.

- Conduct thermal analysis:
  - — Develop die and sheet models and equations for thermal analysis.
  - — Develop and verify predictions of die temperatures and distortions.
  - — Forecast sheet temperatures and distortions after stamping.
- Prepare for blank heating:
  - — Verify heater performance in University of Michigan Laboratory.
  - — Design, build, and validate production heater.
  - — Install and test infrared heating in stamping facility.
- Fabricate alloys:
  - — Prepare candidate material for laboratory testing at University of Michigan.
  - — Fabricate full-scale material for production stamping tests.
- Demonstrate full-scale process:
  - — Develop blank transfer mechanism.
  - — Install infrared heating system.
  - — Perform system tests.
  - — Perform production tests.
- Complete panel inspection and material analysis:
  - — Evaluate material strength and corrosion properties.
  - — Inspect panels.
- Evaluate the process:
  - — Develop the business case for warm forming.
  - — Evaluate the technical and economic feasibility for warm forming.

Accomplishments

- Held a project kick-off meeting at U.S. Automotive Materials Partnership on June 5, 2001.
- Met to gain consensus on process cost model on September 28, 2001.
- Formed a team to prepare a cost model straw man for team review in November.
- Initiated the lubricant selection process.
- Resolved intellectual property issues satisfactorily.
- Selected Camanoe Associates to perform technical cost modeling.
- Received a proposal from Oakland University to perform thermal analysis and requested a second proposal from the University of Michigan.

Future Direction

- Specify and produce four alloys for testing.
- Hot-test lubricant samples produced by Fuchs and continue evaluations to develop the optimum lubricant.
- Build a straw man technical cost model and present it to the team for evaluation.
- Build and deliver a lab heater for the University of Michigan.
- Select either Oakland University or the University of Michigan to conduct the thermal analysis.

Introduction

This project focuses on developing the materials, equipment, and processes to cost-effectively produce automotive panels from aluminum alloys using a technology called warm forming.

Aluminum alloys generally have limited formability, a characteristic that has limited their extensive use in the industry. To relax this limitation, DaimlerChrysler, General Motors, and Ford collaborated to investigate the feasibility of increasing the formability of aluminum using warm forming technology.

Phase 1 Accomplishments

The first phase of the project involved developing a modified 5000-series aluminum alloy, producing blanks, and performing warm forming trials on a Dodge Neon door inner, a door panel with deep-drawn profiles.

Stamping trials were performed at Sekely Industries, Inc., an industrial stamping supplier and member of the Phase 2 project. The principal result of Phase 1 was a stamping demonstration of Dodge Neon inner door panels that were formed in a single die stage with one hit. A typical panel from these tests is shown in Figure 1.

Figure 1. Dodge Neon door inner panel.

Producing these panels in a single stage was an encouraging feat because the equivalent steel panel required two stages. The newly developed alloys also showed significant promise for use as Class A automotive panels. Reaching this goal would have a significant payback because it would simplify procurement, production, and recycling of aluminum-intensive vehicles in the future.

Phase 2 Issues and Plans

Phase 1 focused on demonstrating the gains in formability due to the warm forming process. Since that milestone has been passed successfully, the next steps need to focus on the technical and economic feasibility of applying warm forming technology to the rigors of a production process.

Five critical issues must be addressed successfully to pass the Phase 2 milestone: alloy formability improvement, lubrication system development, blank heating, full-scale production rate demonstration, and cost analysis.

The first issue is improving the formability of the alloy to increase its applicability to parts requiring a material with more extreme drawing capability.

In Phase 1, graphite was used as the die lubricant. Although graphite performs wells as a lubricant at warm forming temperatures, it is not easily removed and does not satisfy painting requirements. The second Phase 2 issue is developing a lubricant that can be applied in a cost-effective manner and can be satisfactorily washed away to meet painting requirements.

The third issue is demonstrating that blanks can be uniformly heated and fed to a press at a speed commensurate with standard production rates.

The fourth issue is demonstrating that the heater, lubrication system, press, and feeder mechanism can function as a unit at production rates.

The fifth issue is building a technical process cost model of the warm forming process that would enable a cost comparison between the currently used process and warm forming and would flag areas where the product cost could be minimized.

Phase 2 Accomplishments

The project was initiated on June 5, 2001, with a kick-off meeting attended by the project team. All participants reviewed their roles in the project and summarized their deliverables. Team assignments were made, one of which was to seek contractors for performing the cost analysis and the thermal analysis.

A search was made to identify an appropriate contractor to perform the critical task of cost modeling. Camanoe Associates was selected based on its reputation for delivering quality products on other automotive industry processes.

Camanoe submitted a proposal, which was presented at a team meeting on September 28, 2001. The Camanoe proposal was accepted, and a purchase order is being processed.

The key issue raised at the September 28 meeting was the definition of the scope and parameters for the process cost model. A model team was formed that will be led by Camanoe and will include representation from DaimlerChrysler, Tower Automotive, and the University of Michigan.

Oakland University submitted a proposal to execute the thermal analysis task. A second proposal is expected from the University of Michigan. The proposals will be evaluated, and an award should be made before the end of November 2001.

Future Direction

Work is planned in five areas:

- Alloy selection: Four alloys will be specified by the University of Michigan and produced by Pechiney for testing at the University.
- Lubricant: GM will hot-test the lubricant samples produced by Fuchs, and Fuchs will continue evaluations to develop the optimum lubricant.
- Cost model: Camanoe will build a straw man technical cost model and present it to the team for evaluation.
- Infrared heater: IR Technologies will build and deliver the lab heater for the University of Michigan.
- Thermal analysis: The University of Michigan will be asked to submit a proposal for conducting the thermal analysis, and the project team will select either Oakland University or the University of Michigan as the contractor.

D. Hydroforming of Aluminum Tubes

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objective

- Develop material data, design rules, and computer modeling capability to enable the design and manufacture of hydroformed aluminum tubes for the reduction of mass in automotive bodies.

OAAT R&D Plan: Task 2, 13; Barrier B

Approach

- Undertake an experimental program to determine the basic material and frictional properties of the tube material.
- Design and carry out simple laboratory experiments to determine the response of the tubes to bending and hydroforming.
- Use the basic material and frictional properties measured in the laboratory to complete computer simulations in order to evaluate and improve our ability to model the bending and hydroforming of aluminum tubes.
- Use analysis of the laboratory data to determine simple design rules that product and process designers could use to ensure that hydroformed designs are manufacturable.
- Instrument a rotary draw bender to determine the exact forces imparted on the tubes and to explore the effects of process parameters on bending behavior and on subsequent formability.

Accomplishments

- Obtained tensile data for all materials and measured frictional data.
- Completed and documented free tube expansion tests.
- Completed and documented straight-tube corner fill tests.
- Began rotary draw bend tests.
- Began computer modeling of the tests that have been conducted.

Future Direction

- Install and fully instrument a bender on the University of Waterloo campus to bend tubes under a wide range of end feed and bend radii.
- Test these bent tubes at Industrial Research and Development Institute (IRDI) to determine their residual formability.
- Participate in model validation studies with the automobile companies and develop formability criteria suitable for finite element modeling.
- Characterize failure and damage metallographically.
- Complete work on IRDI's experimentally determined forming limit diagrams, correlate friction measurements and achievable corner radii in straight-tube corner fill tests, and determine an empirical prediction of burst pressure and corner radius.

Introduction

Automotive structural parts can be made from hydroformed tubes. These are tubes, not necessarily circular, which are first bent to shape in a rotary draw bender and then put into a die and expanded by fluid pressure to the die configuration. These parts have many advantages over the welded sheet metal parts they replace: They help reduce manufacturing costs (by decreasing part count, welding, and scrap) and improve performance (by reducing mass and increasing stiffness). Steel hydroformed tubes are finding increased application in the automotive industry, but the behavior of aluminum in the bending and hydroforming operations are currently poorly understood; therefore, aluminum hydroforming is not yet used in the U.S. automobile industry. Figure 1 shows a hydroformed engine cradle.

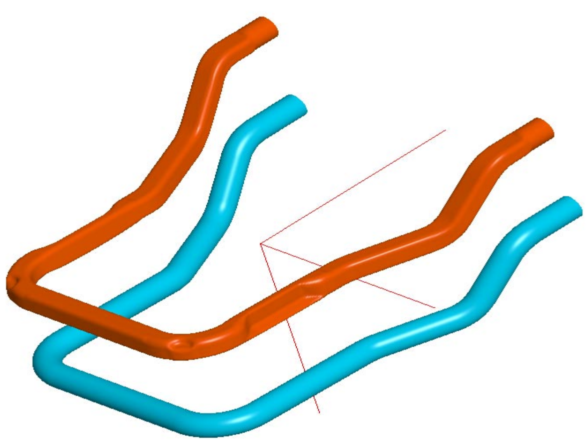

Figure 1. A hydroformed engine cradle.

This project brings together engineers from DaimlerChrysler, Ford, General Motors, Alcoa, Alcan, VAW, IRDI, the University of Waterloo, and several manufacturing concerns. The expertise of the group includes material science, production methods, computational mechanics and experimental methods. This group meets monthly to monitor progress and to plan future project work.

Accomplishments

Materials used in this project included welded Al3.5Mg alloy tubes from VAW and extruded tubes from Alcoa in alloys 6061-T4 and 6061-T6. Alcan's Kingston laboratories obtained tensile data for all materials, and IRDI measured frictional data.

Free-tube expansion tests— tests carried out without a die—were completed and fully documented. Data collected during these tests included the magnitude of expansion, the fluid pressure, the axial force, and the burst pressures. This information enables us to begin to estimate the basic formability of the tube material, and it provides experimental data that we can use to validate computer models. Figure 2 shows Al3.5Mg tubes, 3.5 mm in thickness, at maximum, medium, and minimum end feeds and as received (left to right).

Straight-tube corner fill tests were also completed and documented. These tests involve expanding a tube into a square die. In such a test, frictional forces between the tube and the die become important. Three different lubricants were used. Measurements included internal pressure, axial force, and corner displacement. It was found that the lubricants had a significant effect on the amount of

Figure 2. Al3.5Mg tubes, 3.5 mm in thickness, at maximum, medium, and minimum end feeds and as received (left to right).

corner expansion that could be achieved, and that this effect correlated quite well with the data taken in the basic friction tests carried out by IRDI. These results suggest that the friction tests are valid and that they might be enlarged in scope to provide guidelines for designing hydroforming systems. Figure 3 shows one of the tubes that did not burst. The variation in thickness is clearly evident.

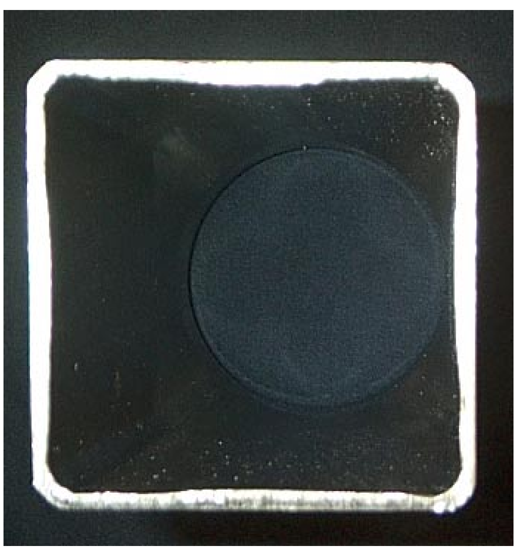

Figure 3. A test tube that did not burst.

Data from these tests also provide information on the basic formability of the tube materials.

Rotary draw bend tests were begun during FY 2001. These tests are not yet complete. When they have been completed, the bent tubes will be tested in hydroforming dies to evaluate their residual formability.

Computer modeling of the tests described has begun. Models of the rotary draw bending have been completed and compared with data from experiments, with quite good agreement. The free-tube expansion test has also been modeled, and comparisons with the experimental data are under way. A benchmark document has been prepared to assist in comparing the various computer models with each other and with the experimental data.

Much of the time in our monthly meetings for this time period was spent in laying out a course of work for the next phase of this project. In view of the high strains created in the bender, the lack of knowledge of subsequent formability, and the fact that the bending operation is responsible for most of the process variability in hydroforming, we decided to concentrate on the bender. The University of Waterloo will install and fully instrument a bender on campus. It will be used to bend tubes under a wide range of end feed and bend radii. These bent tubes will then be tested at IRDI to determine their residual formability.

Waterloo will also participate in model validation studies with the automobile companies and will develop formability criteria suitable for finite element modeling. Waterloo will also characterize failure and damage metallographically. IRDI will complete work on its experimentally determined forming limit diagrams. IRDI will also correlate friction measurements and achievable corner radii in straight-tube corner fill tests and will determine an empirical prediction of burst pressure and corner radius. The latter two projects are aimed at providing validated design rules for hydroforming systems.

E. Electromagnetic Forming of Aluminum Sheet

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL0 1830

Objective

- Develop electromagnetic forming (EMF) technology that will enable the economical manufacture of automotive parts from aluminum sheet. EMF is a desirable process because the dynamic nature of the deformation results in benefits including increased forming limits and reduced springback. These benefits will result in increased use of aluminum and, therefore, vehicles that are more fuel-efficient because their mass is reduced.

OAAT R&D Plan: Task 2, 13; Barriers A, B

Approach

- Address three main technical areas:
  - — Analysis methods for forming system designs.
  - — Development of durable actuators (coils).
  - — Industrial embodiment of the EMF process.

Accomplishments

- Completed a literature search for information on EMF, coil materials, and coil design/durability.
- Completed design and assembly of a 150-kJ pulsed power unit (Figure 1) at Los Alamos National Laboratory (LANL).
- Completed a C4 survey of the laboratories.
- Requested and received a proposal from LANL for a paper study on the design of durable coils.
- Installed the 150-kJ pulsed power supply at Pacific Northwest National Laboratory (PNNL).

Future Direction

- Establish an operating procedure for the 150-kJ pulsed power unit.
- Develop design concepts for durable coils for aluminum sheet.
- Design and build a test rig for coil durability testing.
- Develop concepts/designs for test systems that are suitable to generate hybrid forming (combined static and dynamic deformation) biaxial formability data.
- Establish project partners that can contribute to the development of technology for the industrial embodiment and formability areas.
- Explore and develop modeling capabilities that can assist in the design of EMF systems.

Introduction

In the EMF process, a transient current pulse of high magnitude is generated in a coil by a low-inductance electric circuit. During the current pulse, the coil is surrounded by a strong transient magnetic field. The transient nature of the magnetic field induces current in a nearby conductive workpiece that flows in the opposite direction to the current in the coil. The coil and the workpiece are set up as adjacent magnets with poles oriented in opposite directions to repel each other. The force of repulsion can be very high, equivalent to surface pressures on the order of tens of thousands of pounds per square inch. Thin sheets of material can be accelerated to a high velocity in a fraction of a millisecond.

A recent interest in understanding the EMF of metals has been stimulated by the desire to use more aluminum in automobiles. The high workpiece velocities achievable using this forming method enhance the formability of materials such as aluminum. Also, the dynamics of contact with the forming die can eliminate springback, an undesired effect that cannot be avoided in other forming techniques such as stamping. The commercial application of this process has existed since the 1960s. The large majority of applications have involved either the expansion or compression of cylinders (tubes). The forming of sheet materials is considerably more complex and has received relatively little attention.

Project Deliverables

At the end of this program, methods and data to assist in the economical design of EMF sheet forming systems will be documented. These will include materials information and design methods for durable coils, coil durability test data for selected materials and design concepts, dynamic and hybrid formability data, methods for modeling the forming process, and concepts for the industrial implementation of the technology in an automotive manufacturing environment.

Planned Approach

This project will address three main technical areas. The first technical area involves establishing analysis methods for designing forming systems. These methods will be based on developed knowledge of forming limits and of relations between electrical system characteristics and deformation responses for specific aluminum alloys of interest. The second area of technical challenge is in coil durability. Existing knowledge of EMF and relevant knowledge from pulsed power physics studies will be combined with thermo-mechanical analyses to develop durable coil designs that will be tested experimentally. Until a more thorough understanding is achieved of economic factors determining the required durability, a nominal level of a 100,000-cycle coil life will be a project goal. The third technical area involves the industrial embodiment of the EMF process. In this project, EMF is expected to be hybridized with conventional sheet metal stamping. Different approaches to hybridization will be analyzed for issues affecting economical implementation in a modern stamping plant. Different system concepts will be developed and studied. Existing knowledge of the EMF process and technical achievements in this project will be combined to establish a methodology for designing hybrid forming systems that can be readily integrated into modern manufacturing facilities for the economical production of automotive sheet aluminum components.

EMF System Commissioning

The initial testing and trials of the new EMF system at PNNL were completed in September 2001. The trials consisted of assembling the new EMF power supply system, load cables, and inductive load coil. The apparatus used to conduct the experiments is illustrated in Figure 1. This figure shows the four parallel coaxial conductors connected between the power supply and the EMF coil. Figure 2 is an enlarged view of the single-turn coil. The coil used was a single-turn, low-inductance aluminum alloy coil made from AA6061-T6. Also shown in Figure 2 are multiple sheets of Mylar sheeting and G10 (glass-fiber composite) insulating materials. Not shown in the figure are the coil containment shroud and associated supports.

The experiments involved multiple cycles of charging and discharging of the capacitor bank through the load coil at various known energy levels. The capacitor bank was controlled via the custom system developed for the unit. The sequence of testing consisted of charging the capacitor bank, isolating the charging power supply, triggering (releasing) the capacitor charge, and monitoring the response of the system. The system response was recorded using a high-speed digital oscilloscope.

Figure 1. The capacitor bank and load cables connected to the forming coil.

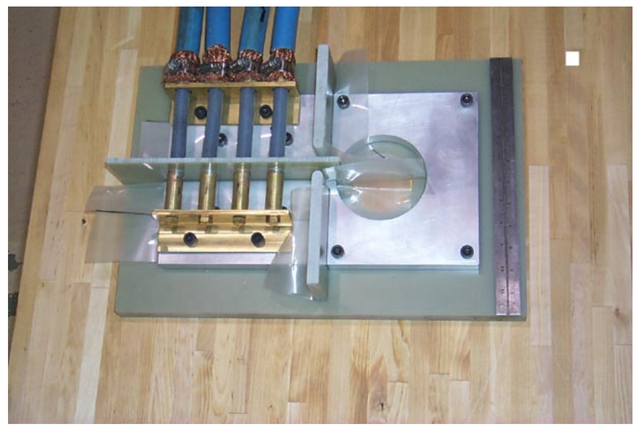

Figure 2. The single-turn forming coil and the load cable connections.

Figure 3 illustrates the typical response of the system during a 15-kJ discharge of the capacitor bank. This figure shows that the half-current of the system is approximately 86 kA, so that a total current of 172 kA passed through the load coil. The system rise time was shown to be approximately 26 microseconds. This power supply system performed as predicted during its design phase, and follow-on work is currently under way.

Coil Design Concepts and Durability

In EMF, the large forces that deform the workpiece are mirrored in the coil. The coil, insulators, and support structure must resist these forces, as well as related thermal cycles, without significant permanent deformation or material failure. In contrast to typical cylindrical coils, sheet forming will require coils with general three-dimensional shapes that are inherently less resistant

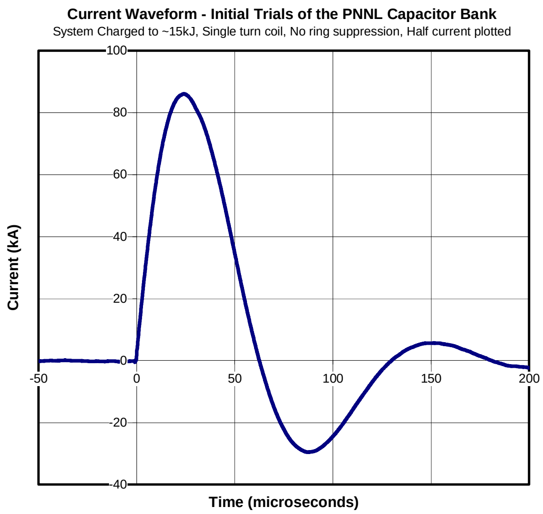

Figure 3. The current waveform that resulted during the initial system trials at PNNL.

to forces induced during forming. The key issues involve materials selection and design. Materials must be selected for both electromagnetic and mechanical properties. Materials must be compatible for bonding, presence of coolants, manufacture, and system integration for hybrid forming systems that combine conventional stamping and EMF. The design must integrate these elements while delivering the primary function of a spatial and temporal load distribution that achieves the desired deformations. Coil systems will have to be low-cost, modular, and highly durable (nominally 100,000 cycles) if they are to be relevant to automotive manufacture.

Conclusions

The technical feasibility of EMF for aluminum sheet in an automotive application has been demonstrated in a prior project conducted by the U.S. Council for Automotive Research. However, the durability of relevant coils systems and methods for the economical design, construction, and implementation of forming systems is yet to be demonstrated. There is also a need for dynamic formability data for relevant aluminum alloys. This project targets these issues. Progress has been made in assessing the current state of knowledge for materials, coil design, formability, and system design. Also, a pulsed power system has been designed and fabricated to serve in experimental testing of coil systems. As this project progresses, a balanced combination of analysis and experiment will be applied to develop durable coil systems that meet the performance requirements of automotive manufacturing. Formability data will be produced and combined with system design analyses to develop methods for designing EMF systems. Project partner(s) will be recruited to study the issues of industrial implementation of EMF sheet forming systems.

F. Optimization of Extrusion Shaping: Aluminum Tubular Hydroforming

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objective

- Develop and experimentally verify materials and forming models that can be used to optimize the hydroforming of complex aluminum tubular components. The project also focuses on developing predictive methods to determine the forming limits of extruded aluminum alloys during the hydroforming process.

OAAT R&D Plan: Task 2; Barrier B

Approach

- Evaluate existing finite element (FE) modeling codes and baseline capabilities.
- Select a representative complex three-dimensional hydroformed component to demonstrate integrated stretch bending and hydroforming process steps.
- Develop laboratory experimental hydroforming test equipment and evaluate candidate extruded aluminum alloys.

- Develop and verify mathematical-based methods for predicting material forming limits under hydroforming process conditions.
- Evaluate candidate aluminum alloys and tubular products under simulated hydroforming conditions.

Accomplishments

- Developed forming-limit diagrams (FLDs) for several aluminum alloys and compared the material formability with theoretical FLD calculation methods.
- Established the effects of non-proportional loading on the forming limits of aluminum alloy extrusions.
- Established the effects of tubing dimensions and strain hardening on forming limits of aluminum alloy extrusions.
- Determined the mechanical property gradients within structural aluminum alloy tubing that affect the overall formability of these tubes. Structural extrusions are normally made using the cost-saving manufacturing method known as "portal die extrusion."

Future Direction

- Carry out additional work to improve understanding of the differences between structural and seamless extruded tubing.
- Characterize seam-welded aluminum alloy tubing for a quality and cost comparison with extruded aluminum alloy tubing.
- Develop numerical algorithms coupled to FE codes that optimize or refine hydroforming process control prior to part prototyping phases, which would advance use of aluminum hydroforming.

Introduction

Aluminum is currently a principal lightweight material candidate for the Partnership for a New Generation of Vehicles (PNGV) initiative. However, some technical challenges remain that inhibit the economical and efficient use of aluminum in high-volume automotive manufacturing. A major challenge for many aluminum-intensive vehicle designs is the ability to predict the dimensional variations that develop during the forming of individual parts and structural elements, and to use these predictions to control and optimize the forming process that is used. The objective of this project is to develop analytical and experimental tools that can be used to model and predict the shaping and forming processes for individual aluminum structural elements. This project is mainly focused on numerical simulation of extrusion shaping processes and experimental evaluation of aluminum alloy formability.

Project Deliverables

Designing a vehicle by selecting among the multitude of different aluminum alloys, heat treatments, and manufacturing techniques for optimum performance in an automotive structure is a formidable challenge. Several manufacturing technologies have been identified that appear to have significant influence on the formability of aluminum extrusions. There are also major issues that will predict the manufacturability and dimensions of automotive components made from aluminum extrusions. The scope of these projects includes evaluating the limits of formability of different types of aluminum alloy tubing under hydroforming conditions investigating the capability to perform multiple sequential forming operations using FE analysis to accurately simulate the bending, die closure, and hydroforming of aluminum alloy tubing

Planned Approach

The approach and work plan of the project includes (1) evaluating material formability under laboratory hydroforming conditions, (2) performing prototype-scale commercial hydroforming trials, and (3) implementing FE codes and other analytical methods.

Experimental Characterization of Forming Limits

Tube hydroforming is a manufacturing process receiving considerable attention as a method to produce complex and precise structural automotive components. The hydroforming process typically applies internal pressure and axial compression to a tube material to manipulate its expansion to fill a precise die cavity. Figure 1 schematically presents the concept of tube hydroforming. Currently, the automotive industry is applying this process predominately with steel. However, in an effort to reduce vehicle weight and vehicle emissions, considerable attention is focused on applying aluminum alloys in applications formerly dominated by steel. One significant focus of this project was to evaluate the formability of various aluminum extrusions. This section presents the results of an experimental investigation of the formability of aluminum alloy extrusions, which is representative of much of the project's experimental focus. The experimental characterization of tubular materials under simulative hydroforming conditions requires a special apparatus. The system required the simultaneous application of internal pressure and axial load. The system also required unique specimen grips and process control capabilities. The mechanical apparatus used to perform the experiments is shown in Figure 2. The base system for the free hydroforming tool was a 300-kN uniaxial hydraulic load frame. The system was fitted with a 222-kN load cell and unique grips to support both axial load and internal pressurization. The load frame actuator controlled the axial position and force imparted to the tube, and it had a maximum travel of 152 mm. The specimen grips consisted of a locking collet, an internal mandrel, and a seal assembly that are typically used for hydrostatic testing of tubular products. The internal pressurization system consisted of an air-driven water pump that was capable of achieving pressures up to 69 MPa. A programmable valve regulated the internal pressure during testing. The hydroforming control system consisted of a multi-axis digital servohydraulic controller, which controlled forming process parameters and data acquisition.

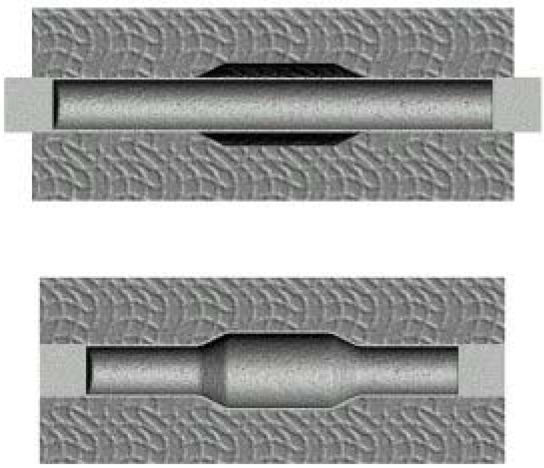
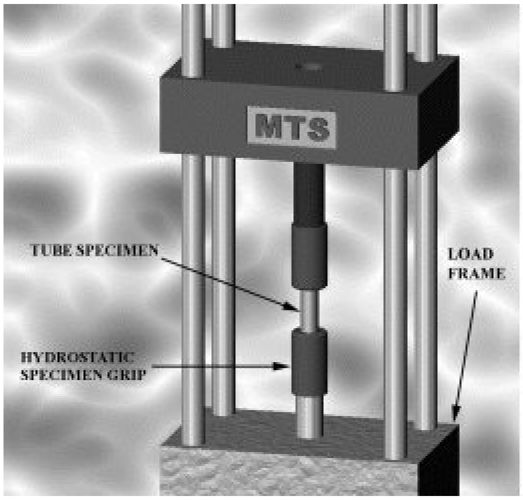


Figure 1. Schematic of the tube hydroforming process before and after forming.

Many different populations of aluminum alloy extrusions were evaluated. This report presents two of the subject materials, which are AA6063-T4 and AA6061-T4. The AA6063-T4 population dimensions were an outside diameter (OD) of 76 mm with a wall thickness of 3.5 mm. The AA6061-T4 population dimensions were OD 50 mm with a wall thickness of 2.0 mm. The overall lengths of the tubes tested during experimentation were 560 mm for AA6063 and 460 mm for AA6061. The unsupported lengths of tube between grips were 420 mm and 320 mm, respectively, with 70 mm of tube inserted into each grip. Each subject tube specimen had a 2.54 mm^2 grid of fine lines applied to its surface prior to deformation, and the development of plastic strain was quantified using strain grid analysis. The strain grid analysis results were compiled to create an FLD for each tube population.

The experiments included uniaxial tensile tests that determined the strain hardening coefficients of $n = 0.26$ and $n = 0.27$ for AA6061-T4 and AA6063-T4, respectively. After the uniaxial tensile properties were determined, each sample population was subjected to a series of experiments to determine the level of plastic strain that developed under various proportional loading ratios of internal pressure and axial load. Figure 2 illustrates the typical results obtained for a single population of samples tested under free hydroforming conditions. The specimens are shown so that the bottom specimen received the highest amount of axial compression during testing, and the top specimen was the uniaxial specimen. The strain-gridded regions surrounding the fractures were characterized using a strain-grid analysis system. This system determined the magnitude of strains that developed in the specimen in the region



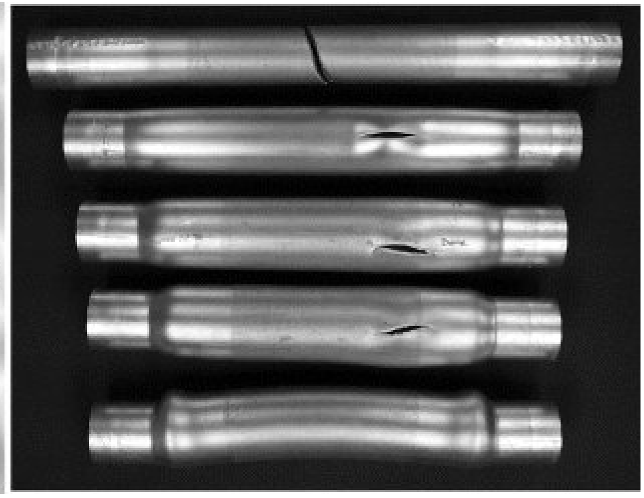


Figure 2. The apparatus used to perform free hydroforming experiments (left). Photograph showing the results of applying increasing magnitudes of axial compression during internal pressurization on the AA6063-T4 population (right).

around the failure. Each of the originally 2.54-mm^2 grids characterized in the area surrounding the fracture was identified as necked if the nodes near the failure showed elevated levels of local strain (necking) in the grid, or the fracture had penetrated under (undercut) the grid surface. Figure 3 shows the results of strain-grid analysis for both populations plotted on a circumferential-versus-axial-strain FLD.

These laboratory hydroforming experiments have shown that the forming limits of aluminum alloy tubing may be determined using a laboratory apparatus that incorporates both axial end feed and internal pressurization. For the AA6061-T4 and AA6063-T4 materials presented, the limits of the material formability have proved to be significantly different even though they have a similar n-value (strain hardening coefficient).

In addition to the seamless 6000-series extrusions presented, this project evaluated the formability of 5000-series materials, 6000-series alloys subjected to retrogression heat-treatment, and various structural extrusions produced using portal die extrusion techniques. Additional work was conducted to improve understanding of the differences in formability of various AA6061-T4 extrusions with different grain sizes and microstructures.

Laboratory Hydroforming with Dies—Hydrocone Experiments

Commercial hydroforming is significantly different from the free hydroforming experiments described. Free hydroforming is a valuable technique to quantitatively determine and distinguish the performance of different materials, but this technique does not offer all the insight needed for commercial hydroforming predictions. Therefore, a laboratory apparatus was devised that was more representative of commercial hydroforming. Figure 4 is a schematic representation of the hydrocone assembly developed and fabricated at Pacific Northwest National Laboratory. This system was designed to simulate the friction, end feed and sealing, and buckling mode constraints that often exist in commercial hydroforming. This arrangement allows tests to be conducted under more severe axial compression than free hydroforming. Figure 5 is a photograph of an AA6061-T4 tube with an OD of 50 mm and a wall thickness of 2 mm tested in the hydrocone apparatus. This tube achieved increased circumferential expansion compared with same materials under free hydroforming, which is consistent was commercial experience. Figure 6 illustrates an FLD showing the relative change in plastic strain achieved using the hydrocone compared with free hydroforming. This apparatus

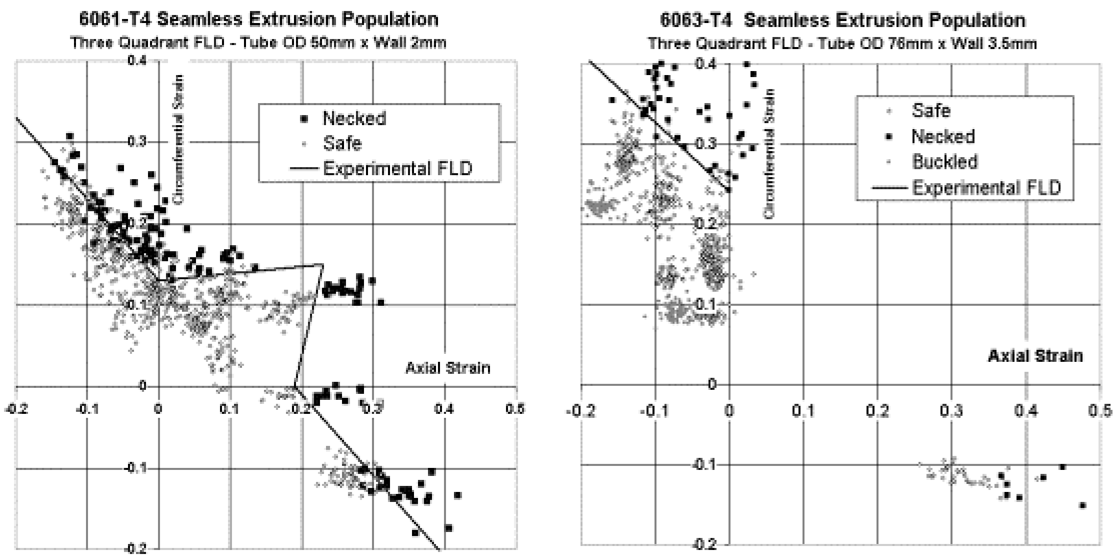


Figure 3. Form-limiting diagrams for the two sample populations determined via free hydroforming.

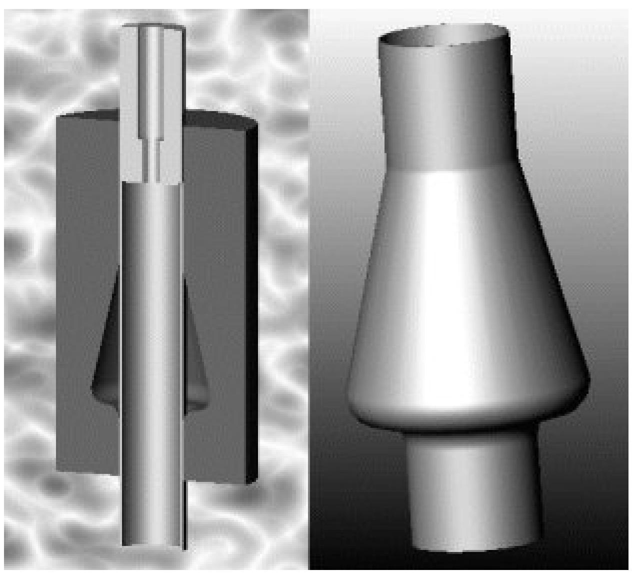


Figure 4. Schematic illustration of the Pacific Northwest National Laboratory hydrocone assembly.

Figure 5. Photograph of an AA6061-T4 tube tested in the hydrocone apparatus.

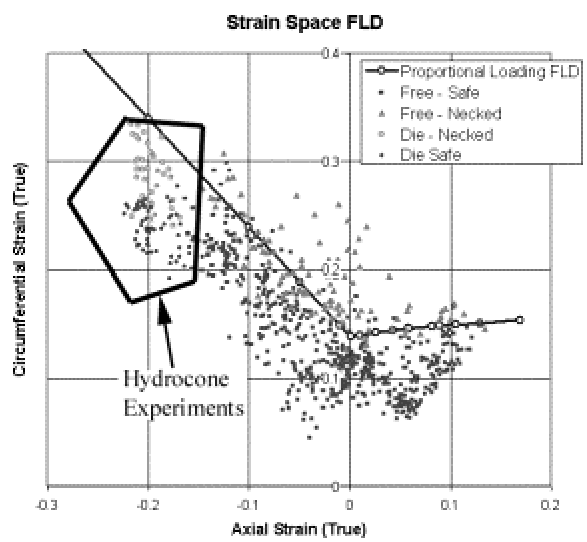


Figure 6. The friction and reduced buckling in the hydrocone dies increases the axial (compressive) end feed of the tube prior to buckling and increases the circumferential expansion.

enables the investigation of more severe non-proportional loading with respect to axial end feed, which is more representative of commercial hydroforming.

<u>Sequential Forming Numerical Simulation</u>

Modeling of extrusion shaping was performed to develop reliable methods for simulating 3-dimensional extrusion forming operations so that the forming process path and final dimensional characteristics of the part can be predicted. This type of work requires accurate material models, realistic failure criteria, and control methodologies that allow modeling and prediction of a successful forming process. This report describes a FE modeling and simulation study performed for sequential stretch-bending and hydroforming of an aluminum extrusion. The purpose of the study was to investigate and develop procedures for three-dimensional FE modeling of sequential forming operations and to assess both the viability of implicit-versus-explicit solution formulations and the utility of 3-dimensional versus two-dimensional models. The complete sequence of forming operations was simulated, from initial clamping of the ends of the extrusion for stretch-bending, to depressurization and release of the part from the hydroforming dies.

Figure 7 illustrates a representative 3-dimensional FE model of a complex hydroforming problem. This figure illustrates the final step in a sequence of stretch forming and hydroforming of an initially non-circular 6000-series extrusion. Results of the simulations, including springback, residual stresses, and deformed shape, showed the value of 3-dimensional models compared with 2-dimensional models. Figure 8 shows the predicted final shape and contours of plastic strain from a sequence simulation encompassing both stretch bending and forming.

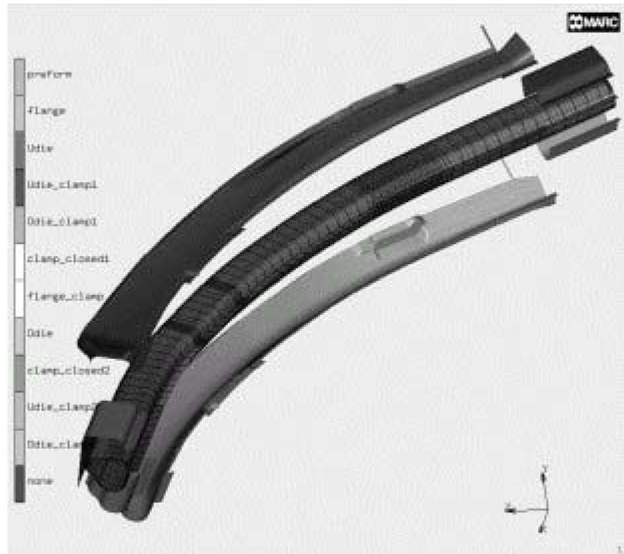


Figure 7. A finite element simulation of a complex hydroform component. This was the final step in a sequence of numerical models used to predict the formability, failure, and final dimensions of the manufacturing process.

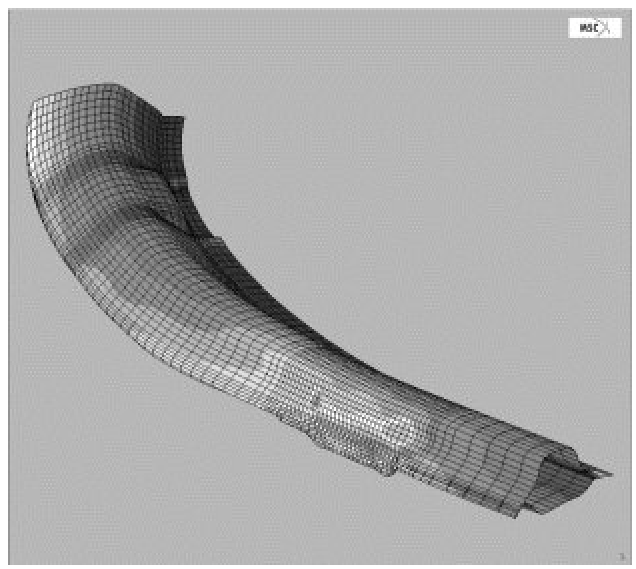


Figure 8. An automotive hydroformed component as predicted by the sequential numerical simulations.

G. Improved A206 Alloy for Automotive Suspension Components

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Demonstrate the capability to produce suspension parts in 206 alloy without hot cracks.
- Establish the quality of 206 control arms.
- Provide design data and guidelines needed to produce other parts in high-strength 206 alloy.
- Determine if a quick and inexpensive ultrasonic test can replace dye penetrant inspection.

OAAT R&D Plan: Task 12; Barriers A, B

Approach

- Establish that it is feasible to produce 206 alloy control arm castings by employing a combination of improved grain refinement and correct casting process parameters.
- Optimize the alloy chemistry and casting parameters for a GMX-130 control arm and make a production quantity of these castings.
- Provide detailed mechanical property data for the resulting control arms, at room temperature and at 250°F, and provide engineering and manufacturing design guidelines for production of other castings in 206 alloy.
- Establish whether the quick, inexpensive ultrasonic test method, developed at Stevens Institute of Technology, is a reliable indicator for the presence of cracks.

Accomplishments

- Completed Task 1 and manufactured two control arm castings in 206 alloy.
- Completed Task 2 and made more than 100 production-quality castings.
- Partially completed Task 3 (some preliminary mechanical property data are available.

Future Direction

- Complete testing of the phase 2 castings.
- Prepare design guidelines.
- Complete a final report on the project by February 2002.

Introduction

A206 alloy is significantly stronger than aluminum casting alloys normally used currently and has mechanical properties approaching those of some grades of ductile iron. It also has excellent high-temperature tensile and low-cycle fatigue strength. Consequently, this material could be used in a number of applications to reduce vehicle weight. Cost savings may also result, because less material would be required to provide the strength needed for the application.

In spite of its excellent properties, 206 alloy is seldom used because of its propensity for hot cracking. A better method to grain-refine this alloy has been discovered, which has the potential to eliminate the hot-cracking problem. In addition, a new ultrasonic inspection technique to test for the presence of hot cracks has been developed at Stevens Institute of Technology. Hence, it is time to reconsider the commercial feasibility of 206 alloy.

The high-strength 206 alloy has a number of potential applications, but its high strength and excellent ductility make it an ideal candidate for suspension components. Consequently, control arms will be poured to establish the viability of this material for suspension components.

An extensive material properties database has been developed for aluminum alloys under the current U.S. Council for Automotive Research (USCAR) project titled “Design and Product Optimization for Cast Light Metals”. However, no information on 206 alloy is presently available in this database.

Justification

Automakers will be under increasing pressure to reduce CO_2 emissions through increased Corporate Average Fuel Economy (CAFE) standards. Because of its higher strength, 206 alloy structures have the potential to reduce vehicle mass, which is directly linked to improved CAFE and vehicle performance. There is also a potential for cost savings.

Program and Deliverables

This project will take approximately 12 months to complete and will proceed in four stages. The total project cost is $232,000, 56% of which is provided by in-kind contributions from the participants and the three automobile companies. DOE funds provide the balance.

Phase 1

The viability of casting the 206 alloy as suspension components is to be established. Two General Motors (GM) control arms will be poured at Stahl Specialty Company. The tooling for these castings is available at Stahl and will be used to make A206 (conventional) and A206E (experimental) versions of 206 alloy. These castings will be examined for cracks by die penetrant tests, and mechanical properties will be determined for test bars cut from the castings.

Phase 2

Assuming the results in phase 1 confirm the viability of casting the A206E alloy, the second stage of the program will begin. Further casting trials will be conducted at Stahl Specialty with the object of “fine tuning” or optimizing the casting process for the GMX-130 casting. When the “sweet spot” for the casting has been found, a significant number of production castings will be made. Dr. Donskoi from Stevens Institute of Technology will participate in the evaluation of crack inspection techniques at Stahl Specialty during these trials.

Phase 3

The third stage of the program includes a full spectrum of mechanical properties and other inspection tests. Most of these will be conducted at

Westmoreland Mechanical Testing Laboratories. The results will give USCAR a good comparison of properties between A356 and A206 alloys poured in the same mold. In addition to the mechanical property tests, nondestructive tests will be conducted at Stevens Institute of Technology.

Phase 4

In the fourth and final stage of the project, GKS Engineering will prepare a detailed analysis and report of important experimental results obtained during the program. The report will

- Determine if the hot cracking problem can be eliminated by a combination of improved grain refinement, proper mold design, correct casting parameters, and better on-line inspection techniques.
- Provide detailed mechanical properties data for the A206 alloy control arm, at room temperature and at 250°F.
- Provide engineering and manufacturing design guidelines for production of castings in 206 alloy
- Establish whether the quick and inexpensive ultrasonic test method, developed at Stevens Institute of Technology, is a reliable indicator for presence of cracks

Results Obtained

Phase 1

Two control arms were selected for these tests. Both parts have been produced commercially in the past by using tilt-pour molds in A356 alloy, heated, treated, and aged to maximum strength (T6 condition). Two versions of A206 alloy were poured, using virtually the same melt and process parameters used for A356 alloy. No changes were made in the mold. Minor changes in tilt rate and ejection times were made for the A206 alloy trials, but no attempt was made to fully optimize the process. The intention was to establish the viability of pouring automotive suspension components in A206 alloy. The two castings are both GM parts. They are shown in Figure 1. The casting on the right hand side of Figure 1 is a rear lower control arm for GM's Cadillac. This part is a substitute for a steel stamping and was not designed primarily to be an aluminum casting. The geometry is complicated, the draft angle on the deep ribs is minimal, and it is a large casting (weighing 6.7 lb or 3 kg). This casting was selected as a worst-case scenario. Contrary to our expectations, we were successful in producing this part. The most serious problem was cracking during ejection. The tool used in our trials was old and showed a significant amount of erosion at the in-gates. Thus the part tended to hang up in the mold. Only with careful maintenance of the mold

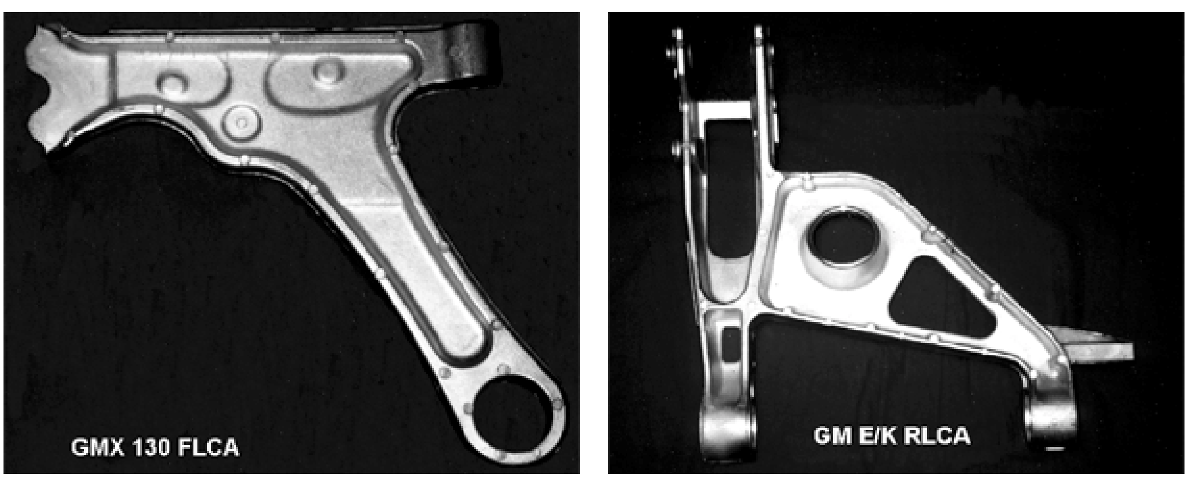

Figure 1. Lower control arm castings produced in phase 1 trials. (Left) A front lower control arm for a GM Grand Am. (Right) A rear lower control arm for a GM Cadillac.

coating in these areas could the part be ejected without cracking.

The casting on the left hand side of Figure 1 is a front lower control arm for GM's Grand Am. This part was originally designed to be an aluminum casting.

The GMX-130 castings were subjected to dye penetrant tests. For the parts produced with the regular version of A206 alloy, 4 of 24 castings or 16.6% exhibited cracks. For castings poured with the improved A206E alloy, 5 of 52 castings or 9.6% exhibited cracks. The other control arm was also examined for cracks, but these data are less meaningful, because the cracks were caused by a die ejection problem. The results consequently reflect the state of the die coating.

The castings were heat treated, and tensile bars were cut from primary load-bearing parts of the casting, where mechanical properties are critical. The average values of the tensile properties measured are summarized in Table 1. Data for the same castings made with A356 alloy are also included for comparison.

Table 1. Average values of the properties measured for the tensile bars cut from heat-treated castings

Mechanical properties of RLCA castings			
Alloy	Yield (ksi)	Ultimate (ksi)	Elongation (%)
206-T4	36.6	54.3	12.6
206E-T4	33.5	51.7	15.8
356-T6	31.7	40.1	9.1

Mechanical properties of GMX-130 castings			
Alloy	Yield (ksi)	Ultimate (ksi)	Elongation (%)
206-T4	33	51.2	17.0
206E-T4	34.3	53.4	17.8
356-T6	28.6	41.6	9.6

The phase 1 results were encouraging. We were successful in making both control arm castings. The new experimental (E) version of 206 alloy appears to offer better resistance to hot cracking, and the mechanical properties are comparable to those of the conventional version of the alloy. Also, the 206 control arm castings are stronger than conventional 356 alloy parts, even though the casting process has not been optimized for 206 alloy.

Phase 2

A week-long casting trial was held in August at Stahl Specialty Company in Kingsville, Missouri. A set of statistically designed experiments was used to establish optimum process parameters for the GMX-130 casting. When the optimized process was used, X-ray inspection of parts showed the casting to have excellent soundness (no porosity). Then one and a half shifts of production castings were made using the optimized process.

During these trials, the chemistries of the alloy and of the heat treatment parameters were adjusted slightly to increase the strength of the castings.

In addition to the GMX-130 control arm castings, separately cast test bars and step-mold castings were poured for comparison. The step mold has five sections of different thickness. Tests of tensile bars cut from each section will give us an idea of how the section thicknesses, and the resulting solidification rates, affect the strength of castings made in 206E alloy.

Phase 3

The extensive series of destructive and nondestructive tests of the castings made in phase 2 was just beginning as this report was being prepared. Only preliminary test data on separately cast test bars are available.

One of the problems with the use of 206 alloy is that little technical information about the material is available in current handbooks. As a consequence, an extensive literature search was made regarding the Al-Cu–based and Al-Cu-Mg–based alloys.

Studies published on other Al-Cu-Mg alloys nearly 50 years ago suggest that 206 alloy should be subject to a natural aging process—holding castings at room temperature after quenching from the high-temperature solution heat treatment. We therefore decided to study this strengthening process by measuring the Brinell hardness of step-mold castings and the tensile strength of separately cast (ASTM B-108) test bars. Tests were made on the as-cast samples and on castings at times of 1 hour, 8 hours, and 1,2 3, 4, and 7 days after the water quench at the end of the solution treatment. The averages of test results for five castings are presented in Figure 2. The natural aging process is seen to be complete after 3 days of holding. At this time, the average values for the tensile properties (in separately cast permanent mold test bars) are

Yield strength	37,800 psi (261 MPa)
Ultimate	62,200 psi (429 MPa)
Elongation	20%

The Brinell hardness is 106.

Several points should be considered regarding the results shown in Figure 2.

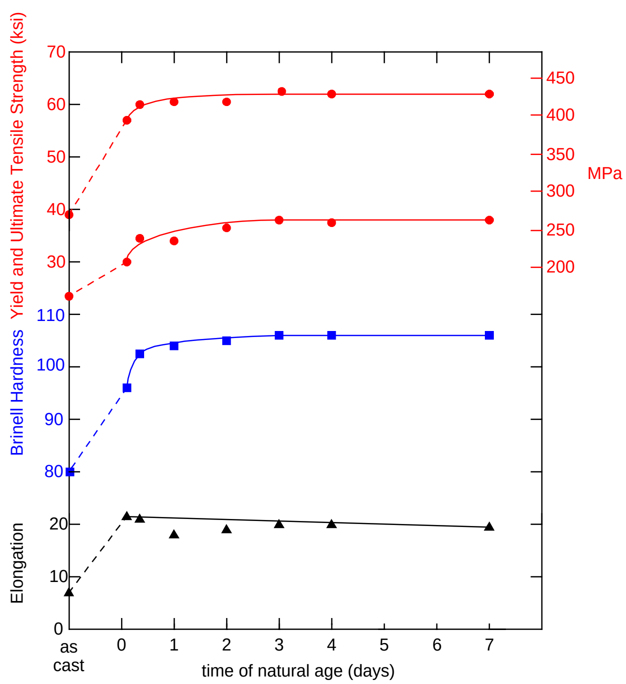

Figure 2. Mechanical properties of 206 alloy during natural aging.

First, the average elongations shown appear to be controlled largely by the presence of small oxides in the test bar castings. These are sometimes observed on the fracture surface of the bar, and they lower the elongation to fracture. When the metal is completely free of oxides, the elongation varies from 25% at 1 hour after the water quench, to 22% at 3 or more days after the solution treatment.

To a lesser extent, the ultimate tensile strength (UTS) is also influenced by the presence of oxides. When oxides are present, the UTS is usually lowered by about 1000 to 2000 psi (7–14 MPa).

The first test results, obtained on control arms in phase 1 of this study and presented earlier in Table 1, were made within 24 hours after the high-temperature solution treatment. Thus full strength was not achieved in these castings at the time tests were made.

The complete test results from the phase 2 castings should be available by the end of January 2002. The design guidelines should also be prepared by then.

The final report for this project should be complete by the end of February 2002.

H. Die-Casting Die Life Extension

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Rod Whitbeck, Project Coordinator, Ford Motor Company

Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objective

- Extend the useful life of the dies that are used in die casting aluminum for automobile components. Extending the life of dies will make it more economically attractive to replace steel components with aluminum components.

OAAT R&D PLAN: Task 12; Barriers A, B

Approach

- Identify failure mechanisms.
- Develop an analytical modeling capability to improve understanding of the relationships between the thermal cycle, stress, and the amount of accumulated damage in a die.
- Identify potential solutions, with emphasis on surface modifications.
- Develop a predictive modeling capability to help identify solutions.
- Perform in-plant demonstrations of enabling technologies.

Accomplishments

- Completed dry-lube and gas-cooling demonstration project. Results showed that this process has commercial potential.
- Completed commercial demonstrations of laser-heat-treated die inserts. The heat-treatment process reduced cracking in sensitive areas.
- Completed commercial demonstrations of laser-surface-coated die inserts. Two coatings were tested, both of which showed good initial soldering resistance but were eventually consumed.
- Completed residual stress study on a four-cavity diode plate die. An unexpected reversal in stress was identified after an extended service life, and analytical modeling was used to help identify the cause.

Future Direction

- Complete a few tasks remaining after completion of four major project milestones during the last 3 months of FY 2001.
- Document the project results in a final report.

Introduction

In the first phase of the die life extension program, the national laboratory participants identified and categorized the fundamental failure mechanisms associated with reduced die life. The study included metallurgical analysis of die components furnished by industrial partners, literature reviews, interviews with field experts, and analytical modeling. Environmentally-assisted cracking, which had not been considered before, was identified as a potential contributor to limited die life. Crack growth studies were performed with several parting agents, i.e., lubricants; and it was determined that crack growth rates were accelerated in the presence of some of them. Localized overheating was also identified as a major factor contributing to heat-checking. Tests showed that infrared heating and laser surface processing had the potential to reharden temper-softened die surfaces by selectively heat-treating only the affected areas. Analytical modeling was used to confirm the benefits of these approaches and to help determine the role of residual stress in die performance.

Project Deliverables

Demonstration projects were organized to evaluate new technologies and approaches that showed promise for extending die life.

To reduce the debilitating effects of splashing cold water on hot dies, a process involving gas cooling and the application of a lubricant in the form of a dry powder was evaluated for commercial potential. A pilot-scale demonstration project, funded by the automobile company partners, was successfully performed at a commercial die-casting facility. The castings produced by this method met all the quality requirements of a production part.

Surface heat-treating was identified as a method to increase the resistance of dies to heat-checking and extend the life of dies overheated in service. Several die inserts were heat-treated on the surface by a laser and run successfully in commercial production at a General Motors (GM) plant.

Laser surface-coating processes were developed at the University of Tennessee and Argonne National Laboratory. Coatings were applied to parts of the same dies that had been heat-treated. These coatings also performed satisfactorily in commercial operation.

A four-cavity diode plate die and matching pairs of die sets made from H-13 and KDA-1 alloy steel were removed from service at intervals over an extended period of time and tested at Oak Ridge National Laboratory (ORNL) for residual stress levels. It was discovered that the residual stress at the die surfaces cycled between tension and compression over time.

Planned Approach

Summaries of the completed demonstration projects follow.

Pilot Testing of Gas Cooling and Dry Powder Lubricant

Prior modeling work and experimental studies performed on the thermal fatigue apparatus at Case Western Reserve University suggested that resistance to heat-checking could be improved by substituting gas for water during the cooldown portion of the die-casting cycle. Eliminating water, which is also used to apply the lubricant (i.e., parting agent), required a dry lubricant application process. A commercially available delivery system for powder lubricant was custom-fit to an existing production die-casting press, and over 6000 castings were produced at the MagTec casting facility in Jackson, Michigan. The results of the quality comparison showed that castings produced with liquid lubricant and those produced using the dry lubricant were comparable, and both were free of porosity defects. Large-volume production experience in Japan has reportedly shown a reduction in porosity defects when dry powder lube systems are used.

The degree of heat-checking was undetectable in both processes. This result was not unexpected, because the runs were not of sufficient duration to generate surface cracks. Japanese studies have also reported a 20-X increase in die life when heat-checking is the primary failure mode.

Soldering was observed on the barrel insert for the throttle body casting (see Figure 1), but the amount of solder build-up was actually less for the powdered lubricant process than for the conventional water-cooled cycle. However, soldering increased when the dry-lube process was shortened to reduce cycle time. These results correlated with an increase in the die insert surface temperature as recorded by thermographs and an imbedded thermocouple.

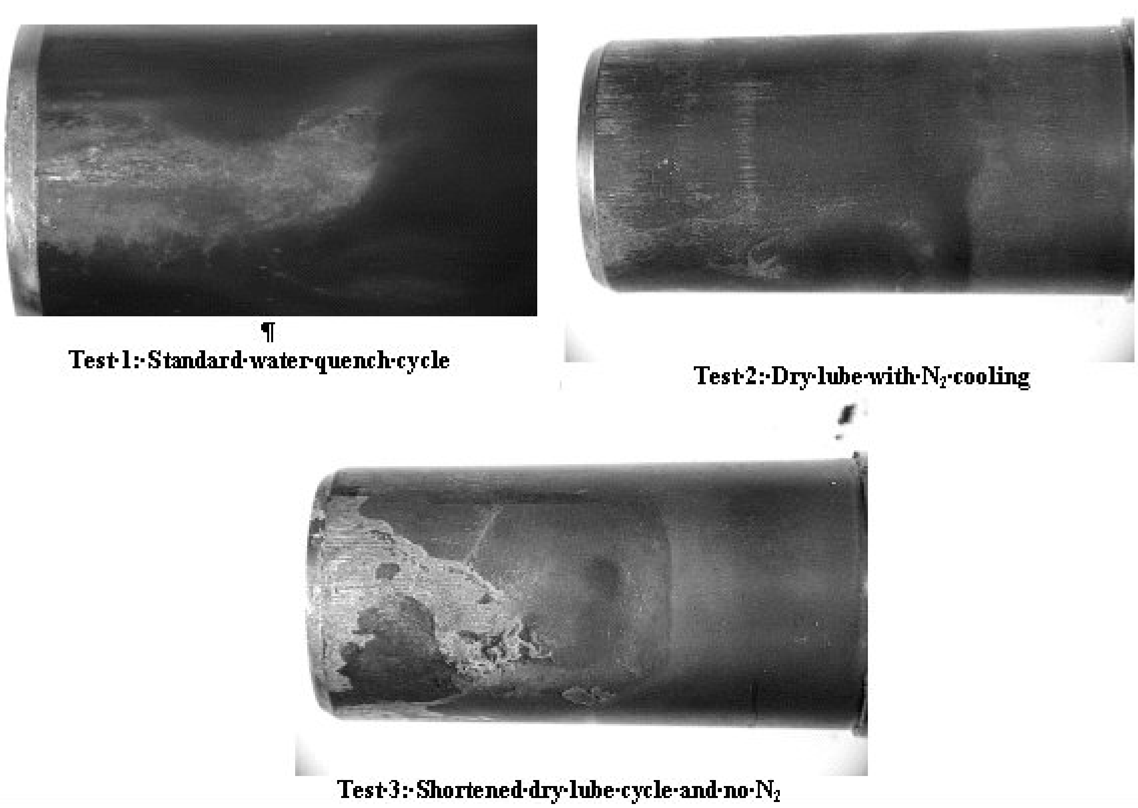

Figure 1. Soldering patterns on large barrel inserts for experiments comparing wet and dry lube cycles.

Laser Surface Heat Treating

Experimental studies performed earlier on the Case Western Reserve University thermal fatigue testing system had shown that surface heat-treating significantly reduced heat-checking. Figure 2 shows a die insert designated as GM detail 504 and heat-treated on the surface with a 1.6-kW pulsed Nd:YAG laser equipped with a fiber-optic beam delivery system by Argonne National Laboratory.

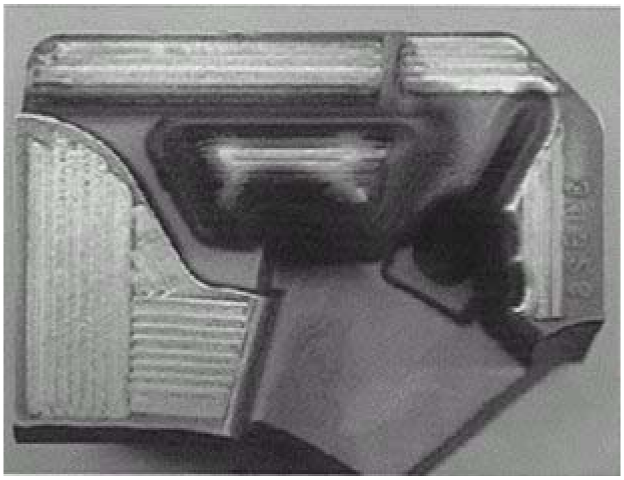

Figure 2. Photograph showing laser surface heat-treated areas on a GM die insert. The heat-treating was performed by the Laser Applications Laboratory at Argonne.

The laser treatment leaves overlapping bands on the surface of the die that can readily be discerned in the photograph. This insert and another one heat-treated by the University of Tennessee were placed into commercial production at the GM Bedford facility. The two dies collectively produced more than 50,000 castings.

Another view of a standard 504 insert that was not surface heat-treated is shown in Figure 3. Extensive heat-checking can be observed in the region designated as Area 1 in the photograph. For comparison, Figure 4 shows the identical area on the insert that was surface heat-treated with a continuous-wave multi-kilowatt industrial Nd:YAG laser equipped with a 600-micron-diameter fiber optic for beam delivery at the University of Tennessee. Virtually no evidence of cracking can be seen on the laser surface-treated insert in the affected area. However, photomicrographs have revealed that a few small cracks do exist in the

Figure 3. Photograph of a used GM insert, detail 504, showing heat-checking in area 1.

Figure 4. Photograph of laser heat-treated and laser-coated GM die insert after 33,285 shots. Vertical laser coating bands are still visible on one surface.

curved area adjacent to the heat-treated region. Further improvements in controlling the laser heat-treating process along concave and convex surfaces will be required to enhance the efficacy of this process.

Laser Surface Coating

Originally developed as a cladding process, laser surface processing has been modified to apply cermet coatings consisting of a solder-resistant ceramic that is cemented to the die surface with a metallic binder. A coating developed at the University of Tennessee, consisting of titanium diboride particles in a ferrous alloy binder, was applied to the post or pillar portion of the die insert in Figure 4. No soldering can be observed on this surface after 33,000 shots; however, very little coating remains. The opposite surface—which cannot be seen in the photograph—was heavily soldered, and no remnants of the coating could be detected on it. Castings that were examined at two prior service intervals showed little evidence of soldering. These results suggest that the coating, while resistant to soldering, was slowly consumed. Heavy soldering apparently occurred very rapidly once the coating was gone. Similar observations were made on another insert coated with tungsten carbide by Argonne National Laboratory.

Residual Stress in Production Dies

Ford Motor Company provided a set of female die halves from four cavity diode plate die sets. Two of these were made with premium-grade H-13 die steel and two with KDA-1 alloy. Residual stresses were measured on these dies at ORNL using X-ray diffraction methods over a 2-year period. The initial measurements were made when the dies were new—that is, at zero service life—and then after 25,000, 113,000, and 235,000 shots. The residual stress results are plotted as a function of service life in Figure 5, together with other data reported by Iwanaga[1] and Case Western Reserve.[2]

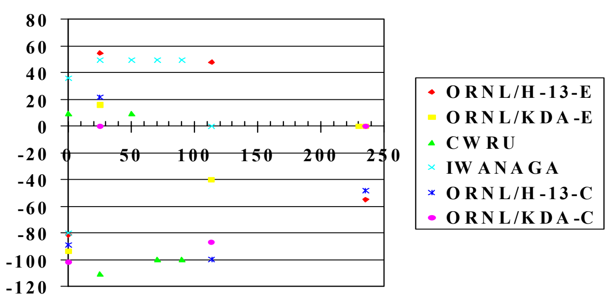

Figure 5. Comparison of the residual stresses measured in various commercial dies as a function of cycle time multiplied by 1000.

It was previously shown that dies begin life in a state of residual compression as a result of the surface polishing that is performed as the last step prior to the die's being placed into service. The dies then go immediately into a state of residual tension as a result of the compressive plastic strains imposed during the injection of hot molten aluminum into the die cavity. The active stress in the die is reversed when cold water is applied to cool the hot surface; but the temperatures are low enough, and the yield strength of the steel high enough, to preclude plastic tensile strains from developing. Thus the residual stress remains tensile. Prior observations reported by Case Western Reserve University on the redevelopment of compressive residual stresses were not widely published because they were believed to be erroneous. However, these new measurements, some of which were made at extended service life, show clearly that compressive residual stresses can be reestablished.

The potential consequence of this residual stress reversal is reduced die life, because the stresses are a result of plastic tensile strains being imparted into the die. These strains will promote crack nucleation and accelerate the growth of existing cracks. Analytical modeling has been used to create a scenario that helps explain why this reversal is occurring. Initially, an insulating layer of baked-on die lube builds on the surface. This layer retards solidification of the casting and extends the casting cycle, maybe by as much as 7 seconds in some cases. To compensate, additional cold water is sprayed onto the die while it is hotter than normal. Tensile stresses develop rapidly, and the temperature-dependent yield strength of the die is exceeded. Plastic tensile strains form in the surface layers of the die, and these force the residual stress back into compression.

References

1. S. Iwanaga, "Initiation and Propagation of Heat Checking and Variation of Residual Stress in Aluminum Die Casting Dies," *Transactions, 19th International Die Casting Congress and Exposition*, paper T97-081, 1997.

2. Dave Schwam, Case Western Reserve University, private communication with Edward Courtright, Pacific Northwest National Laboratory, March 2001.

3. ADVANCED MATERIALS DEVELOPMENT

A. Low-Cost Powder Metallurgy for Particle-Reinforced Aluminum Composites

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363
DOE CRADA No.: 96-Mult-AMP-0444(EE-07-02)

Objectives

- Develop low-cost powder metallurgy (PM) manufacturing methods for particle-reinforced aluminum (PRA) composite components.
- Advance PRA machining technology and PRA composite design methodologies.
- Provide sufficient information that U.S. Automotive Materials Partnership (USAMP) members and suppliers can make sound commercialization decisions regarding PM PRA technologies.
- Through participation by leading suppliers, establish (enhance) technical and manufacturing capabilities in North America to provide low-cost PM PRA components to the automotive industry through the USAMP partners.

OAAT R&D Plan: Task 12; Barriers A, B

Approach

During this 6.5-year project, the following tasks will be executed in two sub-projects, press and sintering (P&S) and direct powder forging (DPF):

- Establish property requirements and cost targets.
- Develop and assess low-cost PM processes for PRA components.
- Develop and apply process models for PM PRA technologies.

- Develop machining technology for PM PRA components.
- Develop mechanical behavior and wear database for PM PRA materials.
- Develop PRA composite design optimization methodologies.
- Combine new process, machining technology, and composite design methodologies to optimize and fabricate PRA materials for at least two components.
- Bench and engine test at least three PM PRA components.
- Develop and continuously update cost models for PM PRA materials, fabrication processes, and PRA machining processes.

Accomplishments

P&S sub-project to develop moderate-strength aluminum composites:

- Fabricated Al-Si alloy PM PRA transmission oil pump gear sets with new dimensionally accurate tooling, demonstrating team-developed technology. Parts have passed two major durability hurdles, including wear tests. Supplier has nearly completed evaluation of all candidate new coatings and the testing of compacted gears to meet more stringent wear and durability test regime defined with the original equipment manufacturer (OEM) pump test engineers. P&S material/process design database is being finalized.
- Tested new wear coatings for further evaluation based on cost performance.

DPF sub-project to develop high-strength, high-temperature aluminum composite applications:

- Established the feasibility of PM PRA connecting rod by demonstrating component fabricated in small lots using existing (non-optimized) tooling with Al-SiC powder blend and preforms.
- Selected the coarse/coarse powder blend for process optimization. Additional commercial powder blending methods are being evaluated with suppliers to optimize processing and handling.
- Conducted tensile and fatigue property tests at room temperature and conducted microstructure studies. The results indicate a high probability of achieving design and performance strengths. Elevated temperature testing is needed on the optimum blend.

Future Direction

- Complete all wear/durability/dimensional conformance studies to recommend optimized P&S technology for high-volume gear set production (prior to OEM evaluations of supplied gear sets).
- Complete evaluation trials of commercial preforming, blending, and agglomeration technologies for DPF technology; fabricate/test 150 connecting rods from optimum parameters for OEM evaluation.
- Complete composite material/process design guidelines and database for both P&S and DPF projects.
- Identify and explore additional opportunities for cost reduction and new powertrain applications of Al powder blends.

Major Project Deliverables

In FY 2001, the P&S and DPF teams delivered the following items to USAMP:

- A set of sintering process conditions (correlated to a wear-resistant alloy microstructure) for the P&S process, based on microstructural analysis and calorimetry measurements.
- For the DPF program, an alternative powder shape consolidation process (that does not require a binder or lubricant additive) for fabrication of forging preforms was developed and recommended for the model test shape.

- Investigation of the commercial potential for incorporating the process in a connecting-rod manufacturing line.
- Specimen fatigue testing, mechanical property, and modeling data for the quantitative assessment of material alloy and blend characteristics in DPF PM PRA.
- Identification and test results from investigation of commercial powder blending technologies required for the agglomeration of new aluminum alloys and composite materials using PM processes and best practices.
- The PM PRA project management team directed an effort led by Aluminum Consultants Group to develop a project database for the P&S and DPF projects. Its purpose is to capture key technical information from the project for use by powertrain product designers and process engineers. The project website, maintained by Technologies Research Corporation–National Center for Manufacturing Sciences, is being used for real-time development of the P&S database, with the final version to be produced on CD for archival and distribution purposes. The team has also finalized an alloy designation/nomenclature system for the DPF project.

Conclusions

This DOE-funded USAMP program is nearing completion with major process and material development milestones achieved during FY 2001. The P&S project has demonstrated high potential for using PM aluminum composite materials in both moderate strength and high-strength/high-temperature applications in high-volume production. The insights gained from the DPF project materials development, processing, and handling experience gathered thus far suggest that design rules and tooling (presently applicable to ferrous-based materials) may need to be revised in order to completely exploit the benefits of using aluminum PM composites.

The PM PRA program expects to achieve several key objectives in FY 2002. Note that the P&S effort with the supplier team will formally end after the first quarter of FY 2002. The original equipment manufacturers (OEMs) will evaluate the results and components for bench testing and dynamometer testing during the reminder of FY 2002.

The emphasis of the team will shift in FY 2002 to supporting the DPF team, led by Metaldyne, in successfully demonstrating the DPF process for manufacturing optimized high-strength Al-SiC composite bearing caps for preforms and demonstrating Metaldyne’s sinter-forge processing to permit evaluation of base material property and dimensional and thermal fatigue. Another objective in FY 2002 is the optimization of powder flow technology. Next, prototype aluminum connecting rods and tooling will be designed and optimized to meet the OEM engine design specification. The original objective to produce up to 150 connecting rods for evaluation in bench test devices still stands. Analyses from fabricated connecting rod samples will be used to optimize processes and tooling and perform machining evaluations. The DPF program is scheduled to conclude at the end of the first quarter of FY 2003.

The USAMP team stands committed to achieving its major P&S and DPF technology milestones in FY 2002 for component fabrication, testing, and validation.

B. Low-Cost Cast Aluminum Metal Matrix Composites

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objectives

- Develop low-cost aluminum (Al) metal matrix composite (MMC) materials that are cost-competitive with typical Al alloys used in the automotive industry.
- Develop a modular mixing and holding system for low-cost Al MMC materials.
- Develop innovative technologies to produce Al MMC automotive braking systems and powertrain components.

OAAT R&D Plan: Task 12; Barriers A, B

Approach

- Develop rapid MMC mixing process and lower-cost, castable Al MMC material.
- Conduct a foundry evaluation of castability.
- Evaluate mechanical and physical properties.
- Conduct friction and wear testing of sample products.
- Explore innovative casting and finishing options.
- Develop innovative brake system designs to utilize Al MMC materials.

Accomplishments

- Completed scale-up of rapid MMC mixing process from 60-kg batch sizes to 600 kg. The cost of developing the larger equipment was shared by MC-21, Inc.
- Completed the processing of innovative cast and pressure-infiltrated samples for wear testing.
- Completed wear and friction testing of seven candidate Al-based MMC materials at Rockwell Science Center using an instrumented test system.
- Provided sample materials for brake wear simulation testing by Ford Research Laboratory.
- Initiated collaboration with Visteon Chassis System for the development and testing of Al MMC brake system components.

Future Direction

- Continue development work in the area of evaluating innovative casting and machining concepts, using the low-cost MMC material approach. Development of a brake system design task involving automotive industry Tier I suppliers will occur in FY 2002. This project will be focused around the use of Al MMC materials in automotive braking systems.

Introduction

The initial goal of the project is to develop the next-generation mixing process for producing low-cost Al MMC materials for casting applications. The concept is to use a modular melting-mixing-holding unit, designed to be placed on the foundry floor, in which the MMC material can be mixed as needed for casting various products. The rapid mixing process, combined with the use of lower-cost SiC reinforcement feedstock, was selected to reduce the overall cost of particulate-reinforced Al MMC materials. The target material cost, which was established by the domestic automotive industry, is approximately $1/lb for production of a range of Al MMC products.

As part of the development of the modular mixing concept and low-cost Al MMC material, it is necessary to evaluate the materials that are generated from the new process and equipment and compare them with a currently available commercial product. The Al MMC material produced by Duralcan, Ltd., is considered to be the industry standard for stir-cast Al MMC material. Therefore, mechanical and physical property evaluation trials were conducted to compare the properties of the new low-cost materials against those of the equivalent Duralcan material.

The ultimate goal of the project is to demonstrate the ability to reduce the cost of cast MMC powertrain and brake system components to that of a comparable system consisting of cast iron components in an automobile application. In addition to reducing the cost of the feedstock Al MMC material, manufacturing costs must be reduced to produce a component at a cost-effective price. Therefore, to further reduce the cost of Al MMC components, innovative casting technology projects have been initiated to address the issues and the low-cost requirements of casting/manufacturing of MMC brake system components.

Modular Rapid Melting/Mixing/Holding MMC System

To make Al MMC materials more attractive for widespread use in automotive applications, raw-material costs will need to be reduced. The current cost for castable Al MMC material is $2–3 per pound, depending on the quantities ordered from the supplier. The domestic automotive industry has estimated that it would require a feedstock cost of approximately $1 per pound to make MMCs more attractive for use in powertrain and brake system components. Therefore, the primary goal of this project is to develop the processing methods to produce Al matrix composites at a lower cost, with no detrimental effect on the mechanical and physical property performance. In addition to reducing the compositing time through the application of a rapid mixing process, a further cost savings can be realized by reducing the raw material cost of the SiC particulate used.

MC-21 in Carson City, Nevada, developed an evolutionary compositing process to mix MMC materials. The design of the mixer incorporates a rapid mixing process that significantly shortens compositing time and, therefore, reduces labor costs associated with production of the MMC material. The concept of the rapid mixing process calls for in-plant placement of the system so that the MMC material can be produced as needed. This arrangement can eliminate the need for remelting prior to casting, which can result in added expense and detrimental effects to the material quality. The system is modular in design, so it can be scaled according to the casting foundries' business needs, thus providing for a cost-effective flexible compositing method. In addition, the ability to produce smaller batch sizes potentially allows rapid changeover between matrix alloy/reinforcement product combinations, at a reduced cost. A U.S. patent (6,106,588) issued in 2000 covers the rapid mixing concept and the details of the process.

Along with a less labor-intensive compositing process, a lower-cost SiC reinforcement can be utilized for further cost savings. For most stir-cast Al MMC materials, a specific grade of SiC particulate is used as reinforcement. The designation for this grade of SiC is F500, which indicates that a narrow size distribution of particle sizes is used—approximately 15–25 µm. The added separation required to achieve the narrow size distribution increases the cost of the raw material and adds to the overall cost of the MMC material. For the production of an Al MMC material with the MC-21 process, a lower-cost SiC material was selected that had a wider size distribution of approximately 3–30 µm. MC-21 estimates the price for a 359/SiC/20p composite material, using the lower-cost SiC and the rapid mixing process, at $1 per pound.

During FY 2001, MC-21 completed the scale-up of the modular mixing process from the previous 60-kg capacity to 600 kg (Figure 1). The larger equipment consists of a separate melting furnace, a modular mixing unit, and a separate holding unit capable of keeping the reinforcement phase properly mixed while controlling pour temperature. All three units were demonstrated during two 600-kg material runs. The resulting cast ingot material was delivered to Pacific Northwest National Laboratory (PNNL) for microstructural evaluation. Additional materials will be provided to selected foundries and casting houses for evaluation. The estimated production rate for the 600-kg system is two batches per 8-hour shift with an electric melter unit, and four to six batches per 8-hour shift with an auxiliary reverb melter. The system is designed in a modular fashion in order to promote the installation on a foundry floor, and the capacity can be sized to meet specific needs.

Figure 1. Prototype 600-kg modular mixing system built by MC-21: melter (rear), mixer (middle), and holding furnace (foreground).

Low-Cost Al-MMC Material Testing and Evaluation

During FY 2001, MMC testing and evaluation has focused on microstructural, mechanical property and thermal property evaluations of the material systems that are included in the innovative casting task. Included in the materials evaluations are the baseline Duralcan F3S.20S and the new MC-21 low-cost SiC materials, both statically cast at 20-volume fraction reinforcement. Innovative shape casting and molding concepts include the centrifugally cast Duralcan and MC-21 materials and the pressure-infiltrated preform materials produced by Metal Matrix Cast Composites (MMCC) in Waltham, Massachusetts. In addition, ceramic-based composite samples were evaluated that were made by BFD, Inc., in Columbus, Ohio, using BFD's ONNEX process. Microstructural evaluations have focused on characterization of reinforcement volume fraction, reinforcement distribution, and porosity levels in feedstock and cast materials. Oak Ridge National Laboratory (ORNL) is performing thermal physical property measurements, including thermal conductivity, thermal diffusivity, and coefficient of thermal expansion. Sample materials for the thermal property measurements were delivered to ORNL near the end of the second quarter. Updated property data are provided in Table 1 on tensile properties for the MC-21 material, with comparable data on the Duralcan F3S.20S material. As indicated by the data, the mechanical properties are in line with the benchmark Duralcan data.

Table 1. Tensile data for MC-21 and Duralcan materials

Composite material	Modulus[a] (GPa)	Elongation[b] (%)	Yield strength		UTS[c]	
			T6 (MPa)	T71 (MPa)	T6 (MPa)	T71 (MPa)
MC-21 359/SiC/20p w/F500SiC	100.5	0.38	290	NA	310	NA
MC-21 359/SiC/20p w/low cost SiC	102.3	0.62	274	214	288	240
Duralcan F3S.20S ingot tested	100.4	–	*d*	NA	290[e]	NA
Duralcan F3S.20S product data	98.6	0.40	310	214	217	262

[a]Results obtained by ultrasonic testing.
[b]T6 condition values.
[c]UTS = ultimate tensile strength.
[d]0.2% off set not achieved.
[e]Fracture strength.

Because of the unique structures that were generated during centrifugal casting and preform infiltration, the evaluation of mechanical properties from representative reinforced areas of the samples focuses primarily on compression testing using cylindrical samples. Compressive properties are felt to be representative of the primary loading conditions that occur in brake rotor applications, and they provide a basis for comparison among the various candidate materials. Room-temperature compression tests were performed on the materials made by the innovative shape-casting methods; the results are tabulated in Table 2. The data represent the compressive strength of the reinforced areas, with the centrifugal cast materials showing the greatest strength. The volume fraction in the reinforced region of the centrifugal cast components is approximately 36–38%, while the pressure-infiltrated MMCC materials have a reinforcement volume fraction of 40%. The compressive strengths are consistent with the expected values for Al MMC materials with this level of reinforcement.

Table 2. Compression data

Material	0.2% offset yield (MPa)
Duralcan F_3S_{20} reinforced region	352
MC21 new pig ingot reinforced region	349
MMCC Al-Mg/Al_2O_3/40p reinforced region	204
MMCC Al-Si/SiC/40p reinforced region	269
BFD ONNEX cermet rotor	248

Innovative Casting and Finishing Technology

The goals of the Partnership for a New Generation of Vehicles (PNGV) project depend significantly on the ability to achieve weight reduction cost-effectively. One area that has been targeted for weight reduction is the brake system, in which replacing cast iron with an AL MMC is desirable. Although the current prototype PNGV vehicles have Al MMC brake rotors, they are considered too expensive for high-volume use. The target is to achieve the weight reduction at cost parity with cast iron.

It is recognized that meeting the goal of a low-cost brake rotor will require more than just a lower-cost Al MMC material. There are opportunities to be realized in reducing costs for brake system components through the application of innovative manufacturing methods and design. Therefore, work was initiated on innovative casting and machining technologies to address downstream processing methods and costs related to the production of an automotive brake rotor.

During FY 2001, contracts were placed with BFD and MMCC to produce Al MMC test disks for brake wear testing and for use in characterizing materials properties and microstructures. In addition to the BFD and MMCC test materials, PNNL prepared a series of test disks using conventional static gravity casting and centrifugal casting to produce a segregated, selectively reinforced disk. Samples of each material set were then machined into 200-mm-diameter, 12.5-mm-thick test specimens and delivered to Rockwell Science Center for wear testing. Wear and friction testing were conducted at two initial operating temperatures, 150ºC and 400ºC. The initial brake pad materials were the Plymouth Prowler rear pads (which operate on an aluminum MMC brake rotor) and a standard cast iron pad for the baseline iron rotor tests. Results of the testing are shown in Figure 1. Note that using the Prowler MMC formulated brake pad material with the MMC rotor specimens resulted in very high wear rates for the friction pad. Additional work performed by Rockwell Science Center investigated the use of more aggressive friction pad materials, resulting in more favorable pad wear rates.

Future Work

With the promise of a lower-cost Al MMC material, along with supporting work on downstream processing, it is important to consider the opportunities for innovative brake system design. Designs must take advantage of the different physical properties of Al MMC materials, as well as their unique manufacturing requirements compared with cast iron, rather than simply substituting them in existing designs. Therefore, as part of the FY 2002 activities, the project will secure the participation of a Tier 1 brake system supplier in a cost-shared project to develop innovative brake rotor designs that take full advantage of the light weight of Al MMCs as a cost-effective replacement for cast iron. It is envisioned that this project would proceed through the steps of design, prototype manufacturing, and testing.

During the last half of FY 2001, a collaboration was initiated with Visteon Chassis Systems (Dearborn, Michigan). Visteon is now in the process of signing a cooperative research and development agreement with PNNL and will participate in the design and testing of brake systems that incorporate Al MMC brake rotors. During the first half of FY 2002, Visteon will develop the design for the test rotors and provide it to PNNL and the selected MMC producers. It is expected that test rotors will be produced by PNNL, BFD, MMCC, and THT Presses in Dayton, Ohio (which uses the squeeze casting process). PNNL, in conjunction with ORNL, will characterize the mechanical and thermal properties of the scaled-up test rotors and supply material properties data to Visteon for evaluation and incorporation into its brake system models. PNNL and the Aluminum Consultants Group will update cost models for the new rotor and brake system designs and compare them with the baseline cast iron system. In addition to developing the MMC brake rotors, the team will examine additional Al MMC applications in conjunction with the Original Equipment Manufacturers Steering Committee and Visteon. As required, additional friction and wear evaluations will be performed at the Rockwell Science Center.

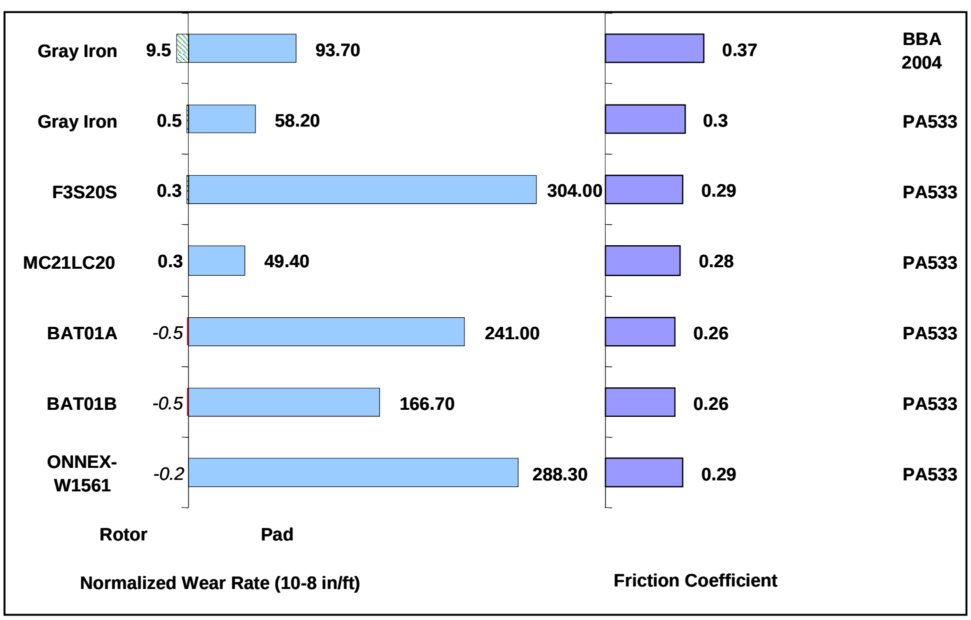

Figure 1. Aluminum metal matrix composite rotors tested against PA533 at 400°C .

C. Structural Cast Magnesium Development

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363
CRADA No.: 00-Mult-AMP-0596

Objectives

- Develop the science and technology necessary to implement a front structural cradle (and two other magnesium castings) that will interface with other concurrent magnesium programs proposed for the U.S. automotive industry.

OAAT R&D Plan: Task 11, 12; Barriers B, C

Approach

- Investigate various methods of casting and techniques used.
- Develop mold fill parameters.
- Develop/investigate corrosion factors and methods of protection.
- Develop nondestructive evaluation (NDE) and sensor applications.
- Enhance the current design guideline.
- Develop and continue the existing database.
- Develop joining and best practices.
- Develop a business case, cost model, and actual casting of a Corvette front magnesium cradle.
- Transfer technology for all project items to industry.

Accomplishments

- Initiated the development of a magnesium material design property database.
- Started collecting magnesium production castings for the development of the material property database.
- Selected a Corvette front cradle (currently a production aluminum production casting) for conversion to magnesium.
- Selected two additional magnesium production castings for project development.
- Established nine steering committees to work with the core team on solving the project tasks.
- Collected commercial software from industry suppliers for evaluation by Oak Ridge National Laboratory (ORNL). This software will be used for future project modeling work.
- Started corrosion evaluation and casting protection work.
- Started quantitative characterization of cast microstructures on the magnesium production castings.
- Developed a magnesium metal material design property database.

Future Direction

- Award a tooling order (for a magnesium cradle) to one of the project participants.
- Follow the tasks listed in the project statement of work to complete the project on time.
- Continuously solicit several additional industrial (equipment) participants.

Introduction

The structural cast magnesium development (SCMD) project objectives focus on resolving critical issues that now limit the large-scale application of magnesium castings in automotive components. This project will develop the science and technology necessary to implement a front structural cradle (and two other magnesium castings) that will interface with other concurrent magnesium programs proposed for the U.S. automotive industry. Such components offer all of the difficult manufacturing issues—including casting processes (e.g., high-pressure die casting and semisolid, low-pressure, and squeeze casting) and joining—along with harsh service environment challenges, such as corrosion, fatigue, and the stress relaxation associated with fasteners.

The project team includes personnel from

- The Big Three automotive companies
- More than 34 companies from the casting supply base
- Academic personnel
- Independent testing and research laboratories
- American Foundrymen's Society
- Technical associations
- ORNL
- Sandia National Laboratories (SNL)
- Lawrence Livermore National Laboratory (LLNL)

Figure 1 illustrates the technology we are trying to develop to improve the properties of cast components. Figure 2 illustrates a generated

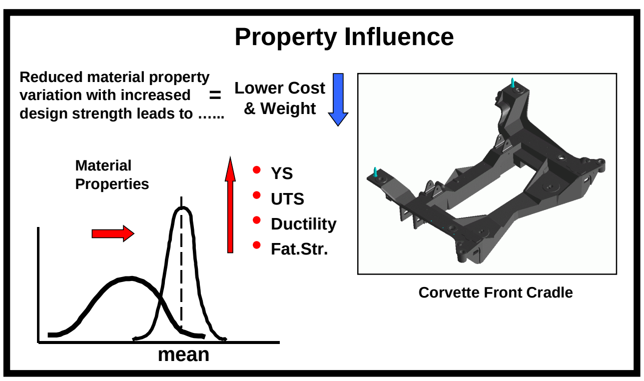

Figure 1. Technology will be developed that will help engineers and product designers reduce cast component variation, thereby yielding lower component cost and weight.

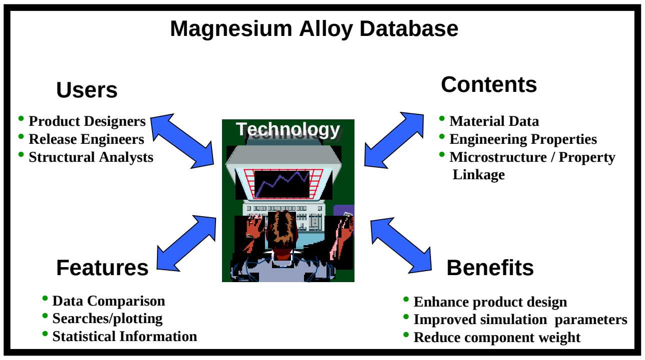

Figure 2. Comprehensive materials database.

database that uses an entirely different architecture for comparing magnesium. It includes historical literature data and comprehensive mechanical property data derived from samples excised from actual production magnesium castings. Casting processes examined include high-pressure die-casting and gravity, permanent-mold, semisolid, low-pressure and squeeze casting.

Monitoring Technologies

- Development of on-line process monitoring, feedback control, and non-destructive evaluation technique(s) to ensure cast component consistency and quality.

  LLNL previously adapted and applied rapid-response temperature optical sensors for process monitoring of two production casting facilities in a previous project. Similar applications will be used for the SCMD project (Figure 3). LLNL

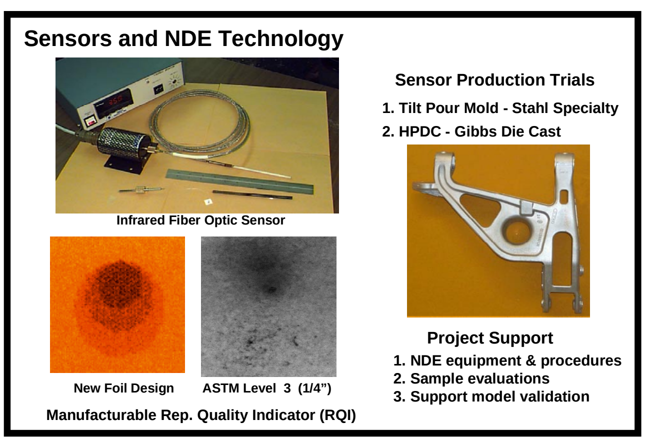

Figure 3. Process sensors, radioscopic quality indicators and nondestructive evaluation project support.

has begun evaluating quality assurance methodologies/equipment and will further develop a new radioscopic standard for magnesium castings based on improving American Society for Testing and Materials (ASTM) standards.

- Evaluation and development of numerical modeling techniques to predict cast microstructure and subsequent mechanical properties throughout cast component sections.

  A major goal of the project is to develop math-based models that predict the microstructure and subsequently the mechanical (tensile and cyclic) properties of cast components from the mold, casting, and component function criteria (Figures 4 and 5). The results of this work will be a joint effort of industry participants, national laboratories, and academia.

Microstructure

The study of variability in ductility and ultimate tensile strength of production casting of magnesium alloys has started. These samples have been successfully correlated to the presence of defects through careful qualitative and quantitative fractographic observations and quantitative microstructural data obtained through digital image analysis and stereological techniques. Quantitative correlation between tensile elongation and total area

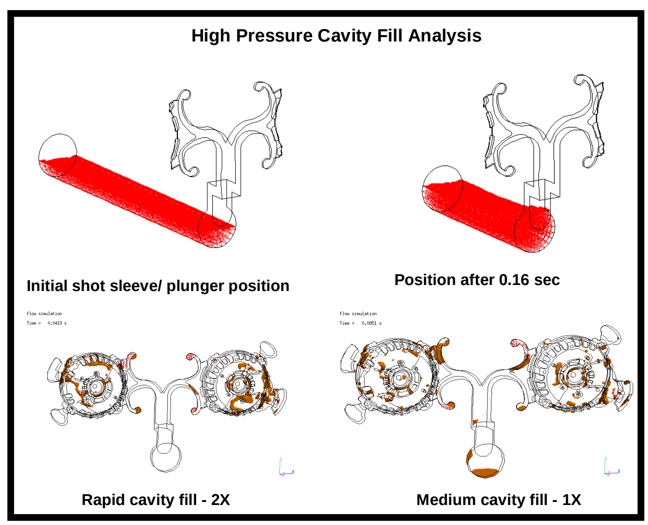

Figure 4. Math-based simulation model that predicts cast microstructure.

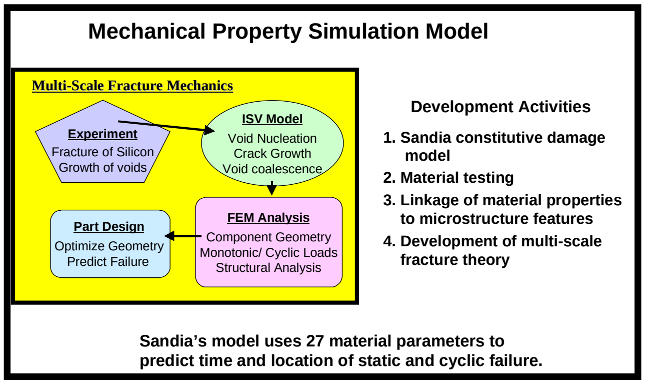

Figure 5. Material property simulation model.

fraction of defects (e.g., oxides, pores, flux residues) of the fracture surface has already been demonstrated for various groups of production castings on the previous project. Fractographic analysis has already been completed of the tensile test specimens of AM60 Mg-alloy specimens from an instrument panel. Georgia Tech completed this analysis, which will be useful for the modeling work performed by ORNL and SNL.

Milestones

Cooperative Agreement

- A front cradle has been chosen for conversion from aluminum to magnesium.
- Two additional magnesium castings have been chosen for evaluation by the project team.
- Detailed quantitative microstructural characterization has been completed for production components cast with AM 60 and AZ 91 magnesium alloys.
- Nine project steering committees have been established and are actively working with the project team and all industrial participants on major task issues.

Cooperative Research and Development Agreement

- **ORNL—**ORNL has received modeling software from three of the supply teams and started the evaluation review. The resulting ORNL models will be used in current commercial software.
- **SNL—**Finite element simulations of AM60B notch tensile tests were performed to corroborate experimental load-displacements curves with porosity data from the interrupted X-ray tomography. Comparisons are encouraging, considering that no coalescence is included yet in the magnesium model, which only considers void growth. We expect to include more about pore-pore coalescence as the project continues. We have also quantified some microstructure-property relations for fatigue based upon some microstructural evaluations of AM60B and AZ91D magnesium alloys. This information will be used for our multi-scale fatigue model.
- **LLNL—**Radiographic analysis of validation mule castings has started to determine discontinuity types and grades at predicted high stress locations. Implementation testing of a fiber optics in-mold thermal monitoring system for high-pressure die-casting will be initiated in several high-volume production facilities. Laboratory personnel will start computed tomography of notched tensile bar magnesium samples to measure porosity void nucleation and growth for constitutive equation modeling parameters.

Project Benefits

The expected benefits to DOE and U.S. industry from successful completion of this research would include the following:

- Reductions in mass for ground and air transportation vehicles will lead to reduced fuel consumption, emissions, and dependence on foreign oil.
- Automakers are under increasing pressure to reduce CO^2 emissions and increase Corporate Average Fuel Economy (CAFE) standards. The North American auto industry currently uses approximately 70,000 MT of magnesium/year, which is equivalent to some 3.5 Kg per vehicle.
- The ability to significantly increase magnesium usage will help the auto industry meet future Federal CAFE targets and reduce exposure to CAFE penalties. Cast magnesium structures have the potential to reduce vehicle mass by an over 100 Kg, which could reduce emissions by 5% and reduce fuel consumption by approximately 1.0 mpg (ignoring secondary mass savings).
- Light metal alloys have greater recycling value and reduce the energy required for recycling compared with plastics (including melting,

machining, handling, and transportation energy requirements).

- The competitive global posture of the Big Three U.S. automakers will increase if they can design and manufacture vehicles that offer greater consumer value. This could improve the nation's trade balance with countries that market vehicles that are more fuel-efficient than those produced in North America.
- Health and environmental issues for workers are reduced during light metal casting operations, compared with such problems in ferrous foundries and polymer molding operations.
- The national laboratories will gain valuable manufacturing development and product application experience.
- The national laboratories will gain an opportunity to develop math-based simulation models and NDE technologies that benefit both the auto industry and federal technology programs.

The project scope of work will be followed to complete the project in time. However, the project has been somewhat curtailed by

- the delay by the Big Three automakers in signing the cooperative research and development agreement
- the delay and requirement by the U.S. Council for Automotive Research Business and Policy Committee that all existing 34 project participants re-sign and comply with the recently designed generic work agreement.

Two important elements are associated with the SCMD project:

- The research institutions participating in the project—universities, the national laboratories, industry, and CAMNET—must work together to carry the scientific research that is required (e.g., microstructure; effects of modeling; corrosion and fastening properties, NDE methods).
- Industry needs a fast-track effort to convert an existing aluminum cradle to magnesium and have a part ready for testing on an actual vehicle in 3 years. Therefore, the industrial participants cannot wait for all of the intricate scientific aspect to be understood before a process is chosen, the tooling built, parts cast and the validation tests are run.

Each "side" of the project, researchers and industry, must remain constantly aware of the needs and progress of the other.

D. Magnesium Powertrain Cast Components

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC-05-95OR22363

Objectives

- Demonstrate and enhance the feasibility and benefits of using magnesium alloys in engine components.
- Design an ultra-low-weight, cost-effective-performance engine block, bedplate, and structural oil pan using the best low-cost, recyclable, creep- and corrosion-resistant magnesium alloys.
- Compare and select the alloys on the basis of common-protocol casting and testing. (The resulting cast specimen database will provide the necessary design data for the components.)
- Design and build the component dies.
- Cast and test the components in operating engines (dynamometer or vehicle).
- Validate the performance benefits, component durability, and system costs.
- Create a material specification for magnesium powertrain alloys common to original equipment manufacturers.
- Identify critical challenges for future magnesium alloy and component developments and use this information to promote scientific research in North America.

OAAT R&D Plan: Task 11, 12; Barriers A, B

Approach

- Conduct the Magnesium Powertrain Cast Components (MPCC) project in two phases, each containing three tasks. A decision gate separates the two phases.
    - — Phase I: Produce the finite element analysis (FEA) engine design, the alloy property requirements for the engine, the comparison of the alloys in a cast-specimen material property design database, a cost model of the engine, and a set of scientific challenges to serve as the basis for Requests for Proposals for fundamental research programs to be conducted in North America.

— Phase II: Produce the magnesium-intensive-engine test results using cast components, assembled and tested engines to validate Phase I predictions, an extensive design database using alloy specimens excised from cast components, the common magnesium materials specification, and the results of the fundamental research programs, the need for which were identified in Phase I.

Accomplishments

- Launched the MPCC project and achieved participation of the key magnesium alloy producers and die casters, as well as a full complement of industrial partners.
- Selected the engine to serve as the baseline for the design of the magnesium-intensive version.
- Defined the mechanical property database content and format to support the FEA design needs for the engine components and to compare and select the best magnesium alloys for the components.
- Completed alloy literature review and compilation of producers' alloys properties to enable the initial screening and downselection of the alloys for inclusion in the cast-specimen mechanical property database.
- Defined the alloy casting protocol and evaluation criteria.

Introduction

The MPCC project will provide comprehensive answers to the questions of the feasibility and cost-benefit of using magnesium in powertrain components. Although magnesium has been demonstrated to significantly reduce weight at acceptable automotive costs in many areas of the vehicle, structural powertrain components have not benefited from this material. The reasons for this are as follows:

- high cost of alloys that are able to withstand the operating temperatures of the engine without deforming under load (creeping)
- limited powertrain design experience with magnesium alloys
- no long-term field validation or controlled-fleet testing data of magnesium powertrain components
- limited scientific infrastructure in the United States that is directed toward acquiring a fundamental understanding of magnesium alloys and casting processes.

With the recent development of several potentially low-cost, creep-resistant alloys, the growth of non-powertrain magnesium alloy design experience—including the launch of the Structural Cast Magnesium Database (SCMD) project by DOE (see article 3C in this progress report), and the renewed recognition of U.S. dependence on foreign oil supplies, DOE and the U.S. automotive industry undertook the MPCC project to extend the weight-reduction potential of magnesium to the powertrain.

The project will increase interest in magnesium powertrains and promote greater competition for and development of magnesium sources, manufacturing capability, and engineering expertise. It will also encourage scientific research into the properties, processing, and behavior of magnesium by universities and national laboratories in North America. The project was started in 2001. It is being conducted in two phases separated by a decision gate, at which point the predicted feasibility and cost-effective performance benefits will be evaluated prior to entering Phase II.

The engine chosen for the MPCC project is the Ford Duratec, as shown in Figure 1. The components of this V-6 engine to be designed for magnesium are the block, the bedplate, and the structural oil pan. The bedplate and oil pan will be high-pressure die cast, and the block will be sand cast.

Details of Phase I

The first-phase goals of the project comprise three tasks: (1) evaluation of the alloys, (2) FEA design of the magnesium engine components, and (3) identification of the critical scientific knowledge necessary for future magnesium powertrain materials.

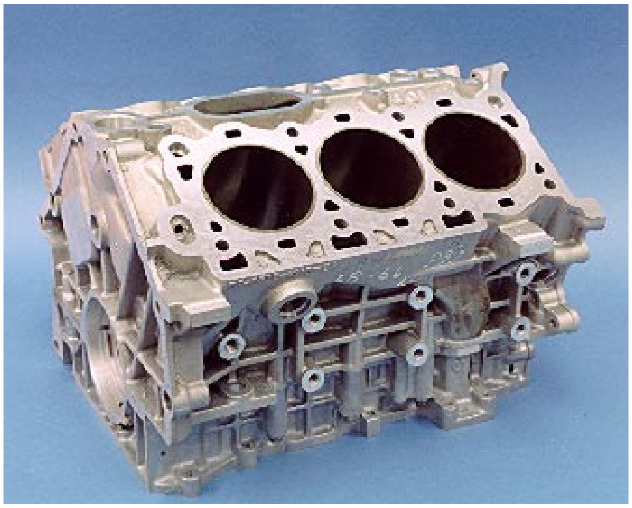

Figure 1. The Ford 2.5L Duratec engine.

Task 1

Task 1 is the evaluation of the new creep-resistant alloys based on cast specimens using a common die and standardized mechanical property and corrosion tests to yield a short list of suitable alloys. Evaluation will be based on tensile and fatigue properties, creep and corrosion resistance, castability, recyclability, and estimated alloy costs.

On the basis of already published data and properties data provided by alloy suppliers, the initial set of candidate alloys has been identified, their producers have agreed to participate in the program, and the test matrix has been defined. The alloys will be cast into the test specimens for evaluation. The documentation of the test results will be based on the same database structure and format as that developed in the project "Design and Production Optimization for Cast Light Metals" that was completed for aluminum and launched for magnesium (SCMD) in 2001. MPCC and SCMD will have a common database of non-powertrain magnesium alloys, benefiting both groups of potential magnesium alloy users.

Task 2

Task 2 is the FEA design and cost model of the engine and its magnesium components. The initial designs will be based on the property data for the alloys that came from the literature evaluation in Task 1. As casting and test results are acquired, the model will be refined to achieve ultra-lightweight, cost-effective performance. The design and cost model results using the best alloys from Task 1 will form the basis for the decision gate for entry into Phase II of the project.

Task 3

Task 3 identifies the fundamental scientific challenges of using magnesium alloys and casting processes in powertrain components. While alloy development is not within the scope of this project directly, the purpose of Task 3 is to promote new and strengthen existing scientific research in North America, specifically for future magnesium alloy and component developments (e.g., determining detailed mechanisms of creep and fatigue of magnesium alloys at elevated temperatures and establishing thermodynamic and phase equilibrium databases). Several such needs have already been identified in the project, and these will be presented in appropriate forums in the near future.

Details of Phase II

The second-phase goals of the project also comprise three tasks: (4) casting the engine components, (5) completing the magnesium alloy property database, and (6) conducting validation tests of the assembled engines. Upon entry into this phase of the project, its specific goals and objectives will be reconsidered and altered as necessary to best serve the overall goals of the MPCC project. In addition, the identification and promotion of scientific research projects will continue.

Task 4

Task 4 will accomplish the casting of the engine components, which will ultimately be validation-tested in Task 6. The dies for the bedplate and structural oil pan and patterns for the engine block will be designed from fill and solidification models and fabricated. Components will be cast, inspected, and approved for assembly. Allowances for up to three iterations of casting trials and subsequent die and pattern modification have been included in the project schedule.

Task 5

Task 5 will complete the magnesium alloy database and will use alloy specimens excised from cast components obtained in Task 4. This database will not comprise all the initially considered alloys; rather, only those selected at the end of Tasks 1 and 2 will be included. Validation of “actual” properties may result in identifying additional areas of technical challenge and the need for further scientific research, thereby furthering the objective of Task 3.

Task 6

Task 6 will achieve the completion of the project: the cast components assembled into complete engines and tested on dynamometers or vehicles to validate the design, performance, and durability of the components. All tested engines will be torn down for inspection; and the final report will contain the test and teardown results, the FEA designs, the fill and solidification models, the alloy property databases, the final cost model, a common magnesium materials specification for powertrain components, and recommendations for future work.

Summary

The MPCC project is an aggressive attempt to address key concerns regarding the future prospects for a magnesium-intensive powertrain. Upon completion of the project, in addition to addressing these concerns, additional scientific and economic implementation barriers, if any, will have been identified and programs to overcome the scientific barriers will have been undertaken by qualified North American universities and laboratories.

E. Advanced Magnetherm Process for Production of Primary Magnesium

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Contractor: ALCOA, Inc., Alcoa Center, Pennsylvania
Contract No.: 306420-AN7

Objective

- Design, fabricate, construct, and demonstrate the process, equipment, and operating parameters required to produce magnesium economically using plasma torch technology and atmospheric condensation of magnesium.

OAAT R&D Plan: Task 11; Barriers A, B

Accomplishments

- Completed an economic model and demonstrated the successful operation of the plasma torch furnace at Mintek (Randburg, South Africa) in the submerged arc mode.

- Made a decision not to pursue the development of a liquid condenser, based on difficulties encountered in the operation of the prototype condenser.

Summary

The prototype liquid condenser experienced two difficulties: (1) durability of the high-temperature seal, and (2) stresses that exceeded the design limits for the condenser material because of the temperature needed to operate a liquid condenser. A decision was made to hold off on further development of the liquid condenser since a satisfactory solution has not been found for these issues. This decision was also based on the economic model identifying potential major cost savings from changes in slag chemistry and associated feed materials that are feasible with the increased heat from the plasma torch (i.e., from Mintek portion of the work) and not the productivity increases associated with operation of the liquid condenser that would allow continuous tapping of liquid magnesium.

An engineering study is in progress to retrofit an existing furnace with a graphite electrode for the plasma torch operation. Testing at Mintek revealed that it may be feasible to operate with a submerged arc environment powered by the Northwest Alloys' AC transformers. However, a decision by ALCOA to shut down its Addy, Washington, magnesium reduction facility resulted in a decision to terminate this project.

F. Solid Oxygen-Ion-Conducting Membrane Technology for Direct Reduction of Magnesium from Its Oxide at High Temperatures

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Contractor: Boston University
Contract No.: C#407200-AN8

Objective

- Demonstrate the technical and commercial viability of the solid oxygen-ion-conducting membrane (SOM) process for producing magnesium directly from its oxide. The SOM process employs yttria-stabilized zirconia (YSZ) as a solid-oxide, oxygen-ion-conducting membrane to separate the anode and cathode of a high-temperature electrolytic cell. This process exhibits several advantages over existing magnesium production routes, including improved economics and reduced environmental impact.

OAAT R&D Plan: Task 11; Barriers A, B

Approach

- Identify molten flux systems to operate the SOM process at 1300–1400°C.
- Examine the long-term stability of the YSZ SOM membrane in the candidate flux systems.
- Determine key physical properties of the candidate flux systems: volatilization rate, ionic conductivity, and mass transfer characteristics.
- Conduct laboratory-scale experiments with various anode-electrolyte-cathode configurations in order to determine the dissociation potential and the I-V characteristics for producing magnesium from its oxide in the selected flux systems.
- Evaluate various methods of magnesium collection during the SOM process.
- Operate the SOM cell between 1300 and 1400°C at current density on the order of 1 A/cm^2 and maintain low overall ohmic and polarization losses in the system so that the applied electric potential does not exceed 5V. This arrangement will keep the power consumption at less than 13 KWh/kg of magnesium produced.

Accomplishments

- Conducted experiments to examine potential interactions between the YSZ membrane material and two candidate flux compositions: 29 wt% SiO_2–51 wt% MgF_2–20 wt% MgO and MgF_2–5.3 wt% MgO. Based upon these results, the binary eutectic MgF_2–5.3 wt% MgO flux was selected as the candidate flux for the SOM process.
- Conducted thermogravimetic experiments to determine the volatility rate of the MgF_2–5.3 wt% MgO flux at temperatures of interest for the SOM process.
- Assembled the experimental apparatus for the in situ investigation of possible molten flux/YSZ interactions using impedance spectroscopy.
- Assembled an SOM cell incorporating the MgF_2–5.3 wt% MgO flux, the YSZ membrane, and a magnesium vapor collection apparatus for the laboratory-scale electrolysis of magnesium between 1300 to 1400^0C.
- Achieved high magnesium production rates (Faradaic current densities in excess of 1 amp/cm^2) without damaging the YSZ membrane or other components of the electrolytic cell. The applied electric potential was below 5 V.
- Operated the SOM cell successfully at 1300°C for more than an hour with a steady-state current density of 800 mA/cm^2. The applied electric potential was 4.0 V, and the power consumption was estimated to be 10 KWh/kg of magnesium produced; these figures are clearly much better than the state-of-the-art processes for magnesium production and also the Hall Cell for aluminum production. Magnesium metal vapor that was produced was successfully condensed as crystals in the condenser.

Future Direction

- Complete the impedance measurement experiments and model the long-term stability of the YSZ membrane in contact with the MgF_2–5.3 wt% MgO flux.
- Determine the ionic conductivity of the MgF_2–5.3 wt% MgO flux.
- Continue the laboratory-scale magnesium electrolysis experiments to identify the rate-limiting process steps, and optimize the SOM cell configuration and the magnesium vapor condenser.
- Use the results obtained to verify the energy requirements and the environmental and economic benefits of magnesium production by the SOM process. The data will be used to model the process for scale-up in order to evaluate commercial viability.

Introduction

The SOM process is a generic environmentally sound and energy-efficient alternative for oxide electrolysis using an inert-oxygen-ion-conducting membrane-based anode. The process can be an attractive alternative for the synthesis of high-energy-content metals such as aluminum, magnesium, titanium, high-purity silicon, chromium, and value-added ferroalloys. This project focuses on the synthesis of magnesium metal using this technology.

The current production methods for magnesium are either electrolysis from a halide electrolyte bath, which requires extensive and expensive feed-material preparation, or metallothermic reduction (the magnetherm process) at high temperatures (1600°C) involving expensive metal reductant (FeSi). The electrolytic magnesium process is based on an anhydrous chloride feed material that is recovered from brines via an elaborate and expensive front-end dehydration process. This feed preparation process step may represent 80% of the plant footprint and 30% of its capital cost. This process also generates a chlorine by-product at the graphite anode that may represent an environmental concern and requires additional capital and operating resources. The alternative metallothermic process for producing magnesium is based on the thermal reduction of calcined dolomite or magnesite with ferrosilicon to form a magnesium vapor that is collected in an attached condenser. The ferrosilicon reductant represents 12 KWh/Kg, or roughly 40% of the total energy required to produce magnesium; it also represents roughly a third of its total production cost. Furthermore, this batch process generates about 4–5 tons of slag per ton of magnesium that must be properly disposed of.

In the SOM process, the oxide reduction is electrochemical, but it dramatically alters the existing electrolytic magnesium process flowsheet. It replaces the magnesium chloride dehydration process with a simple magnesium oxide calcining operation, thereby significantly reducing front-end capital and operating costs. It also reduces the overall energy consumption of magnesium production by roughly 50%. In addition, the elimination of the magnesium chloride cell feed eliminates the production of chlorine and chlorinated hydrocarbons. Compared with the current metallothermic and the electrolytic processes, the SOM process has the potential to be more economical, less energy-intensive, and more environmentally sound.

The fundamental configuration of the SOM process, shown in Figure 1, consists of a solid-oxygen-ion-conducting stabilized-zirconia electrolyte (membrane) that separates the anode from the melt containing the oxide of the metal to be reduced.

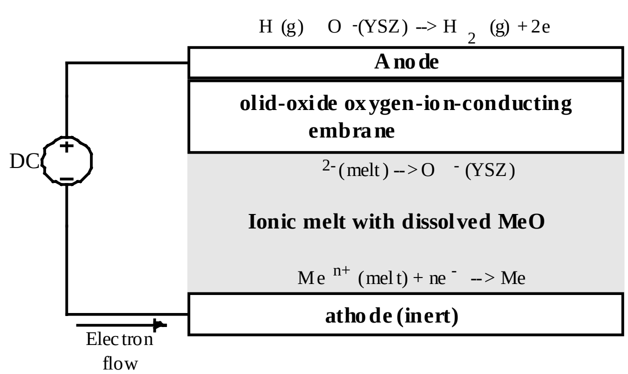

Figure 1. Solid oxygen-ion-conducting membrane process for metal (Me) production by direct reduction of its oxide.

An inert cathode is placed in the melt. When the applied electric potential between the anode and the cathode exceeds the dissociation potential of the oxide to be reduced, the desired metal cations are reduced at the cathode; oxygen ions migrate through the membrane and are oxidized at the anode. The applied electric potential between the electrodes can be increased as long as the potential at the melt-zirconia interface does not exceed the dissociation potential of the solid zirconia and undesired oxides are not reduced at the cathode. Therefore, larger potentials can be applied between the electrodes in order to increase the rate of production of the desired metal. The full benefit of the SOM process can be realized if the process is conducted at temperatures between 1200 and 1400°C.[1,2] At these temperatures, the overall resistance drop across the stabilized zirconia membrane and the melt are expected to be sufficiently low to allow high current densities on the order of 1 A/cm^2 or greater to be obtained. In addition, at these temperatures, the process efficiency can be further increased by directly reforming hydrocarbon fuel over the anode. It should be noted that several attempts have been made to employ SOMs at temperatures below 1000°C. However, these efforts have not been successful in developing a commercially viable process mainly because sufficient high-current densities could not be obtained through the membrane.[3–8]

Work in Progress

Keeping commercial viability in mind, the present research is aimed at developing a SOM cell capable of producing and collecting magnesium with nearly 100% current efficiency at steady-state current density on the order of 1 A/cm^2 or greater with applied electric potentials not exceeding 5V. This development would result in a magnesium production rate that is at least 1.33 times the current aluminum mass production rate in Hall cells at comparable power consumption. The following sections summarize the various components of current research efforts toward this goal.

Molten Flux Selection

Two melt systems were selected as candidate fluxes for the SOM process for magnesium production. Selection criteria include low volatility at the temperatures of interest, viscosity of less than 10 poise, electrical conductivity of greater than 1 (ohm-cm)$^{-1}$, and MgO solubility of at least 5 wt%, at temperatures from 1300 to 1400°C. These requirements are expected to provide kinetically favorable conditions suitable for scale-up.[7, 8, 9–11] The first flux considered was the eutectic composition of the binary MgF_2–MgO system (MgF_2–8mol%

MgO.) The second flux investigated was based on the eutectic composition of the ternary MgO–MgF_2–SiO_2 system; the exact composition was 29 wt% SiO_2–5 1wt% CaF_2–20 wt% MgO.

For the proposed SOM technology to be technically feasible on a commercial scale, it is critical that the YSZ membrane be reasonably stable in the molten flux. Therefore, to identify a single best candidate flux composition for the SOM electrolytic cell, preliminary experiments were conducted to evaluate the stability of the SOM material, YSZ, in the two melts described earlier. In these experiments, YSZ crucibles containing the respective candidate fluxes were heated to the proposed operating temperature of the cell (1300°C). The flux-containing crucibles were held at temperature for periods of 10 and 30 hours and then cooled to room temperature. After cooling, the interfaces between the YSZ crucible and candidate fluxes were examined using optical and scanning electron microscopy for signs of dissolution, erosion, or general reaction. Chemical analysis of the region near the slag-crucible interface was performed using an electron microprobe equipped with a wavelength-dispersive X-ray spectrometer.

Examination of the solidified samples indicated that YSZ is relatively stable in contact with both slags. For both slag compositions, the reacted zone between the slag and crucible wall was less than 500 µm after 30 hours. For all experiments, microprobe analysis of the crucible wall near the solid-liquid interface indicated a deficiency in yttria. This suggests that a selective dissolution of yttria occurs at the liquid-solid interface. It may be possible to alleviate the problem by adding yttria to the initial flux composition to reduce the driving force for yttria dissolution from the YSZ membrane. It was found that the degree of reaction appeared to be slightly higher for the ternary slag containing silica. In addition, it was found that some pitting of the YSZ crucible occurred in the vicinity of the free surface of the silica containing ternary slag. This phenomenon was not observed for the binary MgO–MgF_2 slag. Based on these observations, it was concluded that YSZ is more stable in contact with the binary MgO–MgF_2 slag, and this flux composition was selected for further experimentation.

Characterization of MgF_2–8mol%MgO Flux

An investigation is in progress to accurately determine certain key properties of the binary magnesium oxy-fluoride slag, which has been selected for use in the SOM electrolytic cell at temperatures between 1300 and 1400°C. These properties include

- precise erosion/corrosion rate of YSZ by this flux
- its volatility
- its ionic conductivity
- its mass transfer properties

The first parameter in the list is critical. The feasibility and design of a large-scale SOM-type reactor for the electrolysis of magnesium are highly dependent upon the long-term stability of YSZ in the proposed flux. Therefore, laboratory experiments are planned to precisely determine the rate of attack of YSZ by the magnesium oxy-fluoride slag. For these experiments, the plan is to use an impedance spectroscopy technique that has been successfully employed in the past to investigate refractory-melt interactions in other systems.[11,12] The scientific principles of this technique will be only briefly explained here. The impedance of an electrochemical cell is a function of several parameters, including the wetted area and geometric configuration of the electrodes and the conductivity of the electrolyte. For carefully designed experiments, very small changes in the wetted area of the electrodes can be detected by monitoring changes in the impedance of the cell. In the present study, two cylindrical electrodes with a known and constant separation distance will be inserted into the molten MgO–MgF_2 flux contained in a YSZ crucible and the impedance measured as a function of time. Interaction between the MgO–MgF_2 flux and the YSZ will result in a change in the measured impedance.

Thus far, the experimental apparatus for these experiments has been assembled. The cell constant and the dependence of the cell impedance upon the wetted area of the electrodes has been determined for the proposed apparatus using a standard solution of KCl in water with a precisely known conductivity. Impedance data regarding the stability of YSZ in the MgO–MgF_2 flux as a function of temperature is being gathered and will be presented in subsequent reports. These experiments will also allow for the precise determination of the ionic conductivity of the flux.

The specific rate of volatilization of the MgO–MgF_2 flux is also an important process parameter. Significant flux losses due to volatilization will not only complicate the laboratory experiments in progress, but also decrease the attractiveness of a commercial-scale process based on this flux. Therefore, thermogravimetric (TGA) experiments are in progress to accurately determine the rate of volatilization of this slag at the proposed operating temperatures for the SOM process. Experiments have been conducted using a high-temperature D101-02 AT Cahn Balance.

For these experiments, approximately 20 grams of slag were contained in a graphite crucible having an internal diameter of 2.2 cm and suspended from the microbalance using a mullite rod. All experiments were conducted in an inert atmosphere with a purging flow of argon gas. Volatilization experiments have been conducted at various temperatures from 1300 to 1550°C. At these temperatures, the rate of volatilization has been found to be in the range of 10^{-7} to 10^{-6} g/cm^{2}sec. This rate is relatively low and will not be a problem for either the laboratory experiments or the eventual commercial process. Based upon these experiments, the temperature dependence of the volatilization rate, R, was calculated in g/cm^2-sec as

$$R = -3.96 \times 10^{-4} \exp\left(\frac{-14281}{T}\right)$$

Laboratory-Scale Electrolysis and Collection of Magnesium

An apparatus for the electrolysis and collection of magnesium on a laboratory scale using the SOM technology is schematically shown in Figure 2. The major features of this cell include an iron cathode; a magnesia collection and condenser tube to capture the magnesium metal vapor that is produced at the cathode; a YSZ crucible containing the magnesium oxy-fluoride flux, serving as the SOM; and a liquid copper bath maintained at a very low oxygen chemical potential, serving as the anode. As experiments continue, the apparatus shown in Figure 2 is likely to evolve.

Potentiodynamic and potentiostatic experiments have been conducted to evaluate the performance of the apparatus and the behavior of the magnesium oxy-fluoride flux at 1300°C. Results of these experiments are extremely promising. Figure 3 shows the experimentally determined current-potential behavior of the cell up to a current density of 1 amp/cm^2. The current density is calculated using the active YSZ membrane area because it was found that the membrane area was rate-limiting. Power consumption at 1 amp/cm^2 is estimated to be 12 KWh/kg of magnesium produced. The results of this and other potentiodynamic experiments indicate that the MgO dissociation potential under the imposed experimental conditions is around 1 V, and the cell resistance is primarily ohmic in nature; no diffusion-limited current develops as long as the flux contains an adequate source of MgO.

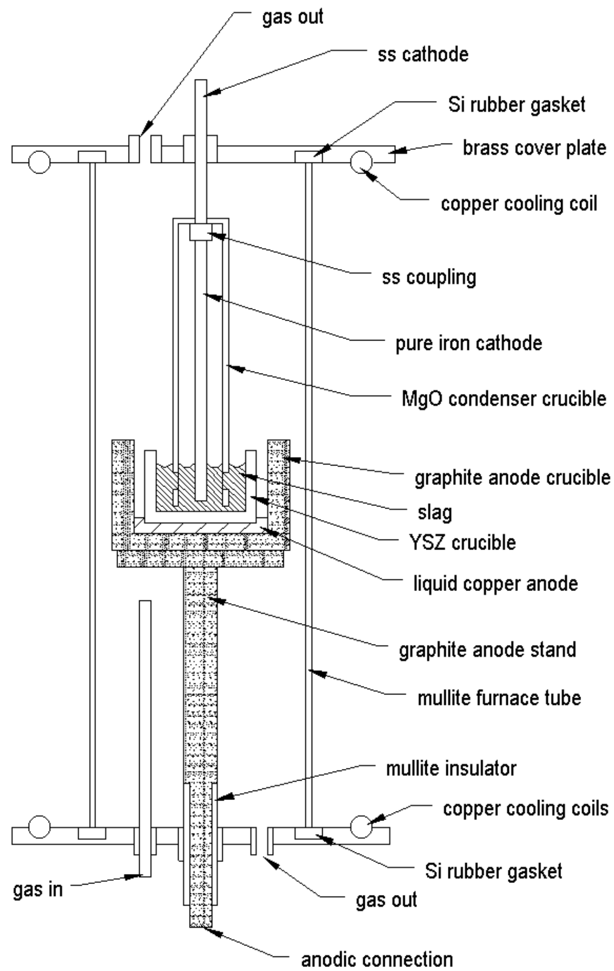

Figure 2. Apparatus for the laboratory-scale electrolysis and collection of magnesium metal using the SOM technology.

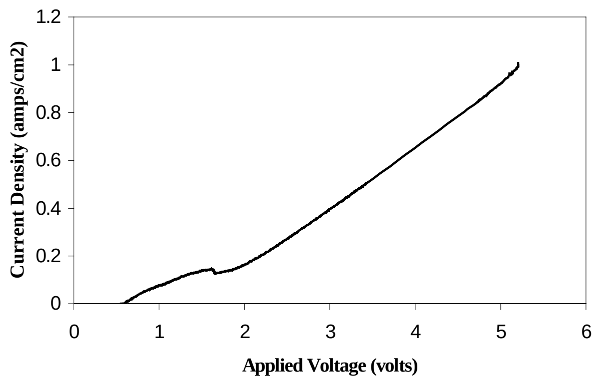

Figure 3. Potentiodynamic sweep conducted on the magnesium oxy-fluoride flux using the SOM cell shown in Figure 2 at 1300°C.

In addition, the cell has been operated under potentiostatic conditions at a current density of around 0.8 A/cm^2 for more than an hour (Figure 4). The applied electric potential was 4 V, and the corresponding power consumption was 37 kW/g or 10.3 kWh/kg of magnesium produced. It is notable that the electrical efficiency achieved is better than that in the highly efficient modern aluminum Hall cell. A significant quantity of magnesium metal was deposited in the crystalline form in the cooler regions of the condenser tube (Figure 5). The magnesium metal analysis was confirmed using a scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (Figure 6).

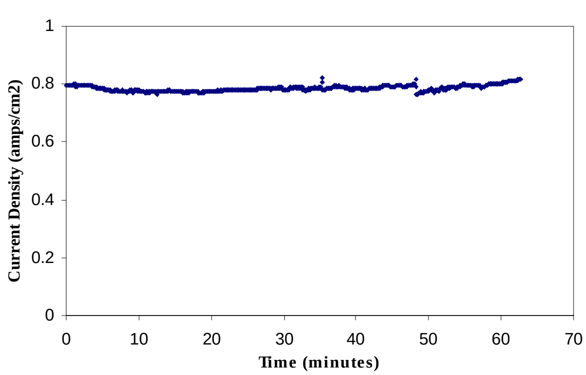

Figure 4. Steady-state current in the SOM cell under potentiostatic conditions (4.0 V) at 1300°C.

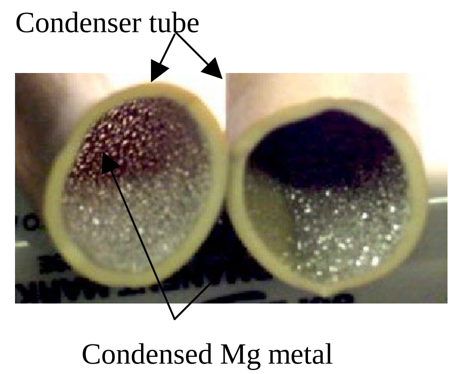

Figure 5. Condensed magnesium metal vapors produced in the SOM cell at 1300°C.

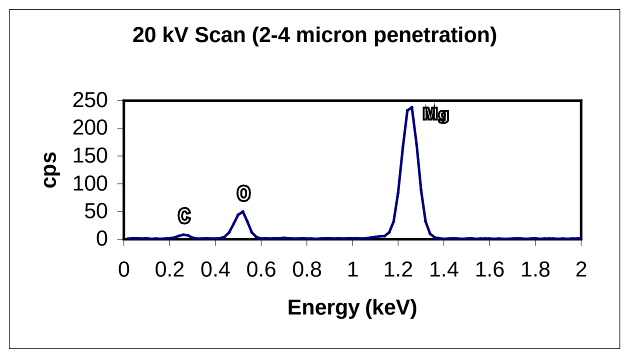

Figure 6. Energy-dispersive X-ray spectra scan of the magnesium metal deposit in the condenser tube.

Summary

Significant progress has been made in this multi-faceted research program to evaluate the use of the SOM technology for the direct electrolysis of magnesium from its oxide. Laboratory experiments to determine parameters critical to the potential scale-up of this technology for commercial magnesium production will continue.

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G. Understanding the Economics of Emerging Titanium Production Processes

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RLO 1830

Objective

- Explore the potential for the widespread adoption of titanium in automobile structures by understanding the manufacturing economics of emerging processes for its production and for forming it into parts.

OAAT R&D Plan: Task 11; Barrier A

Approach

- Identify promising titanium production processes. These include
  - Plasma quench process being developed by Plasma Quench Titanium Incorporated (PQTI)
  - Armstrong process being developed by International Titanium Powders (ITP)
  - FFC Cambridge process being developed by British Titanium
- Catalog technical fundamentals of processes of interest and their potential for technological development.
- Develop process-based cost models for each of the titanium production processes.
- Use the cost models to explore the behavior of each process's manufacturing economics.
- Identify a probable potential lowest production cost for each process.
- Develop a process-based cost model of automotive parts production using titanium.
- Use the cost model to reveal the cost drivers associated with titanium parts production.

Accomplishments

- Constructed cost models of PQTI and ITP processes.
- Analyzed the economic behavior of each process, including quantifying parametric sensitivity and identifying key cost drivers.
- Assessed titanium production cost under likely current, pessimistic, and optimistic development of each technology.
- Constructed cost model of hot isostatic pressing.
- Analyzed the sensitivity of part production cost to key cost drivers.
- Generated and delivered a report summarizing these results.

Future Direction

- Develop an analogous cost model of the FFC process.
- Use this model to generate analyses of FFC process economics.

Introduction

An automobile design trend that has received much attention is the reduction of vehicle mass. Reducing mass can improve both performance and fuel economy. Although design changes can play a large role in reducing mass, ultimately, large reductions will require the substitution of higher-specific-strength/stiffness materials for the now universal carbon steel. Primary contenders in this race are high-strength steels, aluminum, magnesium, and fiber-reinforced polymer composites. One material that is not on this short list but that could provide reductions in selected applications is titanium. Although titanium is light and strong, its role in the automobile has been almost nonexistent because of its exorbitant price. This high price is a direct result of the current production route, the Kroll process, which is time-consuming; energy-, capital- and labor-intensive; and batch-based.

However, new technologies are emerging that may change the characteristics of the titanium market. In particular, these technologies may reduce the price of titanium sufficiently to allow it to compete in high-volume markets, possibly even automotive markets. This project examines the production costs for three of these technologies in detail.

Project Deliverables

Three processes were identified by the Northwest Alliance for Transportation Technology that have the potential for significantly reducing the cost of producing titanium. They are

- plasma quench (PQTI)
- Armstrong
- FFC

To understand the economics of these processes, process-based cost models were created for the PQTI and the Armstrong processes. (A model for the FFC process is under development.) Furthermore, to understand how changes in raw material price ultimately impact part cost, a model of a part forming process, hot isostatic pressing, was developed.

Planned Approach

Because all three processes represent technologies that are not yet implemented at full manufacturing scale, traditional accounting-based approaches to cost are ineffective. However, a set of methods, collectively referred to as “technical cost modeling,” has been developed to address such questions. Technical cost models (TCMs) derive manufacturing costs by building up from the engineering realities of a process. Specifically, TCMs combine engineering process models, operational models, and an economic framework to map from the details of product and process to operating costs. Because TCMs are built around technical details, they allow an exploration of how cost evolves as a technology changes. Because of the obvious applicability, the TCM approach was adopted for this project.

Analysis Summary

The cost analyses of both of these processes proved to be very promising. Figures 1 and 2 give a summary look at these costs for the baseline scenarios considered. These charts indicate the costs for producing briquettes by lightly sintering powders. As the diagrams show, both processes can deliver titanium powder at a price well below the prevailing market price. In fact, under a range of operating conditions, these processes could manufacture powder for less than the selling price of sponge today.

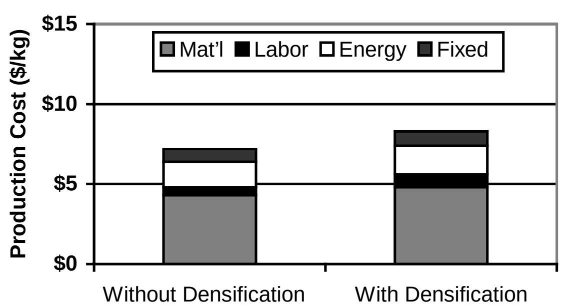

Figure 1. Baseline cost breakdown by major elements for PQTI process.

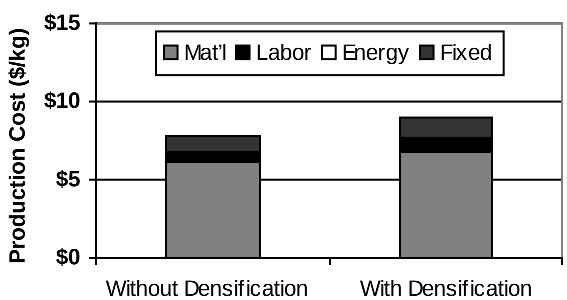

Figure 2. Baseline cost breakdown by major elements for Armstrong process.

Plasma Quench Process

The plasma quench process involves the thermal dissociation and reduction of titanium tetrachloride ($TiCl_4$). This is accomplished by passing these reactants through an electric arc. The resultant plasma is drawn through a Delaval nozzle. The nozzle accelerates and expands the gas, quenching it rapidly enough to prevent significant back reaction. The actual output from the PQTI process is, in fact, titanium hydride (TiH_{1-2}), which contains ~97% titanium by weight, and hydrochloric acid (HCl). The principal costs for the PQTI process are

- $TiCl_4$
- electricity
- quench gas
- capital expenditures

In addition to the sale of Ti powder, HCl can provide a significant source of revenue.

In addition to a number of specific analyses, the economic performance of the PQTI process was investigated under three development scenarios: (1) optimistic development, (2) likely current implementation, and (3) pessimistic development.

Figure 3 shows the production costs associated with each of these scenarios. Notably, using optimistic assumptions, the titanium powder production cost falls nearly to half of the current sponge price. Furthermore, although the range from “optimistic” to “pessimistic” cost covers nearly a factor of three, the “pessimistic” result is still well below prevailing prices for titanium powder.

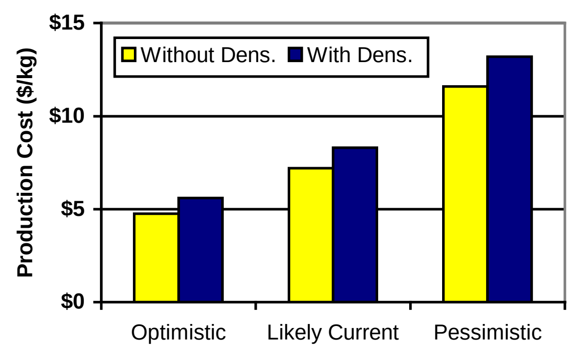

Figure 3. PQTI costs across development scenarios.

Armstrong Process

The process used at ITP, referred to as the Armstrong process, involves the reaction of gaseous $TiCl_4$ with sodium to produce titanium. In realizing this process, ITP’s clear breakthroughs are permitting continuous operation of the process as well as the ability to produce powder directly. Because their product is in powder form, they can much more readily and effectively extract the titanium from the byproduct NaCl and any remaining sodium. Because of the strong exothermic nature of the reaction, the primary energy demands derive not from initiating the process, but rather from cooling its products.

The principal costs for the ITP process are

- $TiCl_4$
- sodium
- capital expenditures

In addition to the sale of Ti powder, NaCl can provide a source of revenue, albeit a small one.

Figure 4 shows production costs for the Armstrong process across the scenarios investigated. Again, all cases represent notable decreases in titanium production cost, with the optimistic scenario coming in below current production costs of Kroll sponge.

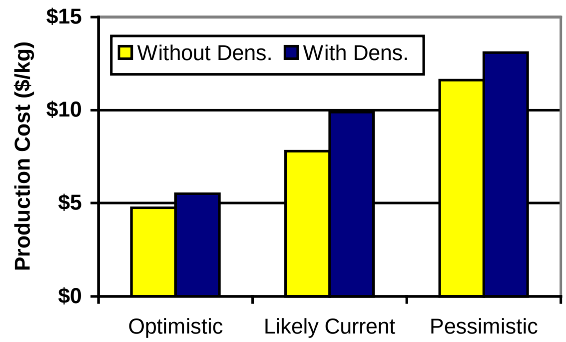

Figure 4. Armstrong costs across development scenarios.

H. Structural Reliability of Lightweight Glazing Alternatives

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objective

- Create lightweight glazing products for automotive applications. The technical focus of this project includes the following tasks:
  - Develop lightweight laminated windshield and body glass by using thin glass layers and asymmetric construction.
  - Characterize the contribution of thin glazing to the structural rigidity of automobiles through instrumentation and modeling.
  - Characterize the strength of new and used typical automotive glass and thin lightweight glass.
  - Characterize stresses imparted on automotive glazing systems that are due to external factors such as stone impacts, handling and mounting, and aerodynamic loads.
  - Develop numerical models to study the behavior of automotive glass (typical and new innovative windshields) under stone impacts.
  - Develop numerical modeling and simulation tools to design and assess the structural behavior and reliability of new, thinner glazing systems.

OAAT R&D Plan: Task 8; Barriers B, C

Accomplishments

- Fabricated new lightweight windshield and side-body glasses that are 30% lighter than conventional glazing systems.
- Fabricated new glass with polyvinyl butyral (PVB) thicknesses ranging from 0.5 to 0.76mm.
- Determined the contribution of the glazing system to overall car rigidity.
- Developed new tests for measuring bending and edge strength of finished windshield specimens and for measuring stone impacts.
- Determined the tin/air side strength and the effect of temperature on glazing strength.
- Developed models to simulate the through-thickness damage evolution in monolithic and layered glass subjected to blunt and sharp indentors.
- Designed a new computational tool for design of lightweight glass.

Future Direction

- Develop lightweight side-door glass.
- Study asymmetric glass construction and/or the use of coatings to harden the outer surfaces of glass for weight reduction.
- Investigate the effect of strength and thickness of PVB on overall windshield/side-door strength and performance.
- Investigate the overall thermal behavior based on the new lightweight glass, including the effect of the laminated side-door glass.
- Characterize the strength of the new lightweight glass.
- Verify and extend the predictive stone impact models to investigate the effect of the impact of sharp indenters on glass.
- Enhance and extend the numerical tool for design of lightweight glass to include stresses encountered during fabrication of glass.
- Develop a methodology for modeling acoustical response of glass.

Introduction

This project is a cooperative research and development agreement (CRADA) between DOE, Pacific Northwest National Laboratory (PNNL), Visteon Automotive Systems (Glass Division) and PPG, Inc. PPG joined this project at the beginning of CY 2000. The project started in June 1998.

Reduction of weight is a major concern for all future cars because weight can contribute significantly to fuel economy. The 100–150 lb of glass in current cars can potentially be cut by a third or more by creating tempered thin glass, asymmetric glass construction, and glass/plastic bi-layers. The development of experimental procedures and of accurate and efficient numerical methods to predict the impact of stresses on the structural reliability of new, lightweight glazing products—such as glass/plastic bi-layers and tempered thin glass—is the key for ensuring widespread use of these products in PNGV. With tools developed in this project, glass will also potentially begin playing more of a structural role so that metal weight can be reduced. Although 50 lb is the minimum savings, there may be a cascading impact on weight savings in other areas. For example, it may be possible to decrease weight in other vehicle parts as a result of increased body stiffness imparted by stronger, thinner glass. The contribution of improved thinner glass properties to enhancing thermal management may also make it possible to reduce weight in thermal management components.

Lightweight Glass Manufacturing

New lightweight windshield glass (Figure 1) was fabricated using 1.6-mm, 1.8-mm, 2.0-mm, and 2.1-mm glass plies (a conventional automotive glass ply has a thickness of between 2.4 and 2.6 mm; total windshield thickness is usually over 5.2 mm). The new glass was formed and laminated at the Visteon Tulsa Plant. The new glass constructions were

- 2.1 mm glass, 0.76 mm PVB, 1.6 mm glass (29% weight reduction)
- 1.8 mm glass, 0.76 mm PVB, 1.8 mm glass (31% weight reduction)
- 2.0 mm glass, 0.76 mm PVB, 1.8 mm glass (27% weight reduction)

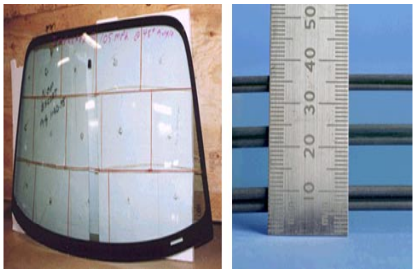

Figure 1. Lightweight glass materials windshield.

New lightweight laminated side glass (Figure 2) was fabricated using 1.6-mm, 2.1-mm, and 2.6-mm plies. The new side-glass constructions were

- 1.6 mm glass, 0.76 mm PVB, 1.6 mm glass
- 2.1 mm glass, 0.76 mm PVB, 2.1 mm glass
- 2.6 mm glass, 0.76 mm PVB, 2.6 mm glass

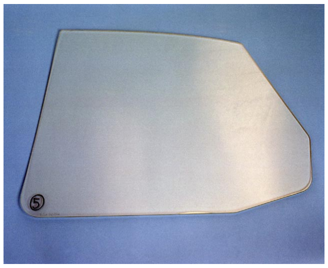

Figure 2. Lightweight glass materials sidelight.

Studies on other asymmetric glass constructions, on coatings to harden the outer surface, and on tempering will continue during FY 2001. We will continue to seek more weight reduction while maintaining or enhancing glass performance.

Contribution of Glass to Structural Rigidity

Experiments and modeling were conducted to determine the contribution of glass to overall car rigidity, and the stresses transferred to the glass. The work focused on the 200 CW 170 Focus and the P2000 vehicles. Visteon Glass Division investigated the strain induced in a windshield for a 200 CW 170 Focus subject to road load vibration, body flex, and mechanical loading. The vehicle was instrumented for acquisition of strain and acceleration data. Three tests were performed—road load data acquisition, direct mechanical loading of windshield, and torsional body flexure. Based on the observed strain, it does not appear that the loading methods produced significant strains in the windshield.

A finite element model of the P2000 body in white was obtained from the Vehicle Safety Research Department at Ford. It was used to study the contribution of the front windshield and back-glass to the overall rigidity of the P2000, as well as the effect of the glass thickness and molding stiffness on both the contribution of the glass to overall rigidity and the loads transferred to the glass.

Analyses have been conducted using the P2000 model to determine the contribution of the front windshield and back-glass to the overall rigidity of the P2000 [ref. 1]. The analyses showed that the torsional rigidity of the P2000 is 24.29 kNm/deg with windshield and backlight glass, and 16.44 kNm/deg without glass (i.e., the glass contributes about 30% of the overall car rigidity). Reducing the glass thickness by 40% resulted in a minor reduction in the rigidity to 23.26kNm/deg.[1]

Additional analyses were conducted to investigate the effect of glazing molding stiffness on the contribution of the glazing system to the P2000’s torsional rigidity. The results show that increasing the glazing molding stiffness results in a marginal gain in torsional rigidity, but decreasing the glazing molding stiffness results in a significant loss in torsional rigidity.

Strength Characterization

PNNL built unique experimental tools for measuring (1) the bending strength of finished windshield specimens, (2) the edge strength of windshields, and (3) the effects of precision impacts with blunt and sharp indentors. The bending strength apparatus is similar to a standard ring-on-ring apparatus that produces a uniform bending stress in flat glass; however, the new device accommodates the radii of finished manufactured windshields. Extensive strain gage data and experimental windshield strength results have been obtained to validate the apparatus. We measured the strength and flaw measurements for conventional automotive windshields in new and used conditions. We also examined the effect of high-speed impact damage on windshield strength. The strength of single glass plies, notably in the edge region, was also examined. The data were used to design lightweight glazing systems for future vehicles. These are some of the conclusions from the experiments on conventional used and new windshields:

- The maximum strength of carefully handled new windshields approaches 30,000 psi.
- The minimum strength of production-quality new windshields is 7500 psi, or 25% of the maximum strength. This is comparable to the strength of used windshields with 53,000 road miles, indicating the severe handling damage during manufacturing.
- The adhesion of a PVB layer is critical, not only for containment of glass fragments during an accident, but also for maximizing the load-bearing capacity of laminated windshields.
- The well-bonded PVB layer transmits a membrane stress of as much as 21% of the

maximum tensile stress in each of the glass plies during ring-on-ring flexure testing.

Figure 3 shows the flexible ring-on-ring apparatus and Figure 4 shows the edge strength test. (See ref. 2 for more details.) Figure 5 shows the edge strength of single plies. We obtained minimum strength estimates of 29, 23, and 19 MPa at failure probabilities of 1×10^{-4}, 1×10^{-5} and 1×10^{-6}, respectively. These are only 20 to 30% of the maximum edge strength of 86 MPa, indicating the need for better control of edge quality during finishing and handling. The combination of edge flaws, inner band residual tension, and bending stress induced by lamination can initiate premature cracking from the edge and undermine the long-term durability of the windshield. Such premature cracking is generally minimized by improving edge quality through diamond grinding and/or by introducing moderate levels of compression in the edge region by convective cooling following sag-bending of individual plies.

Figure 6 shows the bending strength of square plate specimens measuring 70 × 70 mm and 2.0 mm thick. These were prepared from float glass sheets and tested in the concentric ring fixture (b = 9.5 mm, c = 28.6 mm) in ambient conditions at a crosshead speed of 1.27 mm/min, the bending strength of single plies. The key message conveyed by Figure 6 is that the air side is consistently stronger than the tin side. This may be helpful for manufacturing side-door glass because the optical requirements for it are less restrictive.

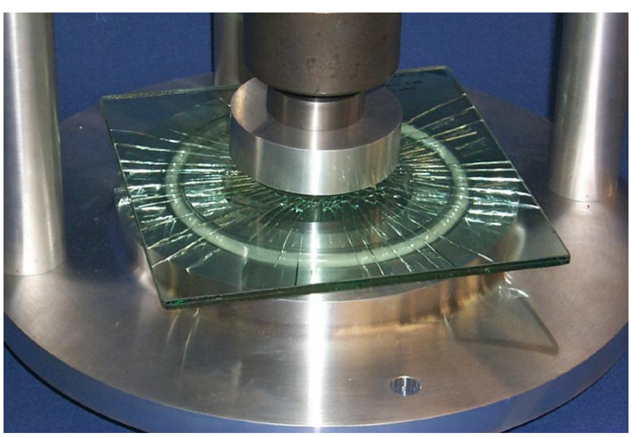

Figure 3. Photograph of the ring-on-ring apparatus during a test.

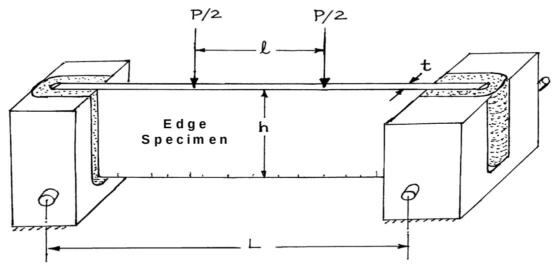

Figure 4. Setup for edge strength test.

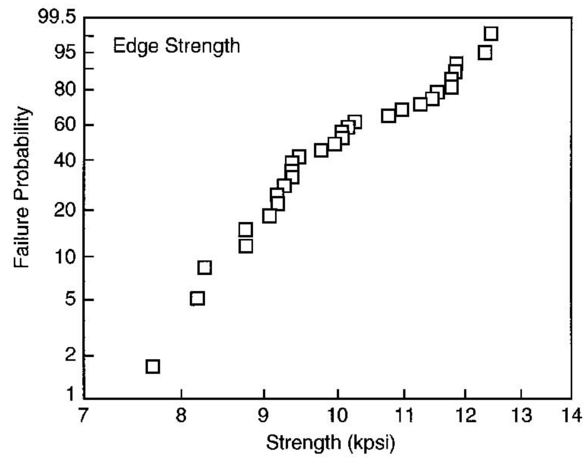

Figure 5. Weibull distribution of edge strength of single glass plies.

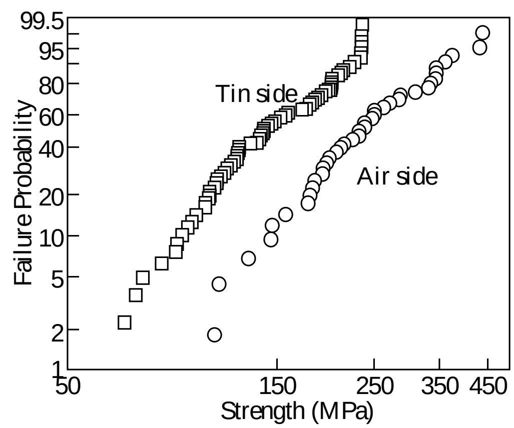

Figure 6. Weibull plot of strength distribution of tin versus air side of single plies.

Future windshield designs are required to be safer and more economical without compromising performance. In this regard, it is critical to understand the strength behavior of conventional laminated windshields under various operating conditions—notably temperature and relative humidity. All of the strength measurements were carried out in controlled-temperature environments and ambient humidity. The data in Figure 7, as might be expected, show that the biaxial strength is 28% lower at 50ºC and 21% higher at −40ºC than that at room temperature. In addition, the scatter in strength data at 50ºC is significantly higher than that at 25ºC and −40ºC (see ref. 3 for more details). Figure 8 shows the experimental set-up for a precision impact of glass (up to 70 miles/hour), and Figure 9 is a representative result. PNNL built the impact apparatus to experimentally investigate the response of automotive glazing to impact events. The apparatus uses a spring-loaded projectile launch system, capable of firing symmetric or asymmetric projectiles. The system measures the speed of the projectile immediately prior to impact via two pairs of infrared beam sensors. The system precisely positions a strain-gage-instrumented single-ply or laminated glass sheet into the impact system and measures the dynamic response of the glass to the impact. The preliminary results show that a single-ply sheet of 3.6-mm glass develops 4000 psi of tensile stress on the glass sheet immediately opposite to the impact event.

Residual Stress Characterization

Measurements of the surface stress distribution were obtained for three automotive windshields. Measurements were performed using two techniques. For the first technique, we used a laser

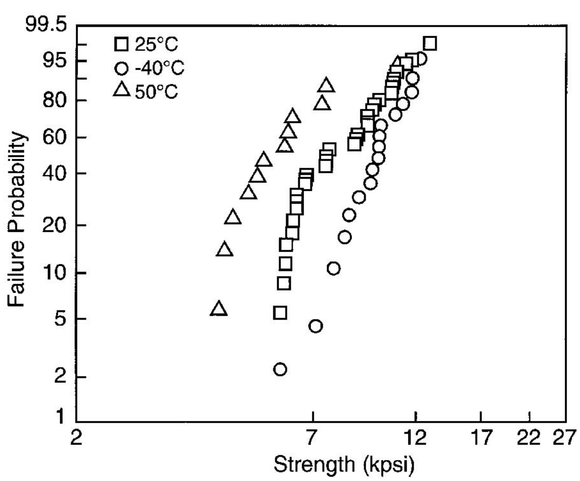

Figure 7. Weibull plot of strength distribution of new windshields.

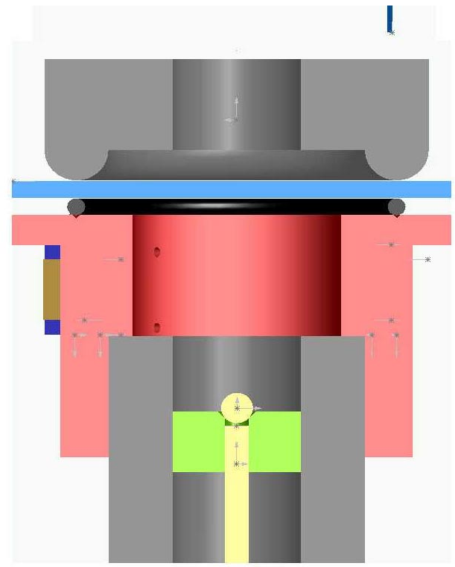

Figure 8. Diagram of an impact apparatus to measure the response of automotive glazing to impacts.

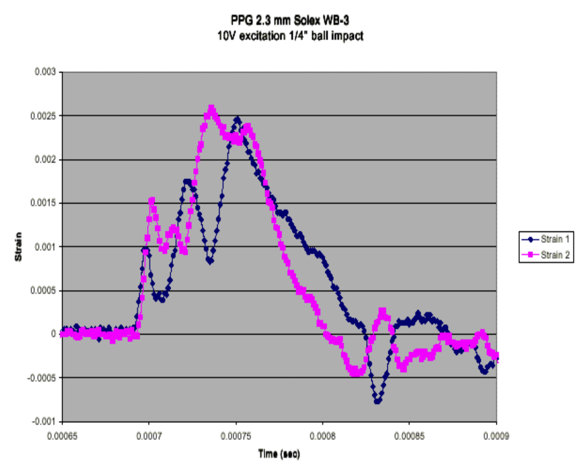

Figure 9. Strain data from a test on the impact apparatus.

grazing angle surface polarimeter (GASP) instrument manufactured by StrainOptics, Inc.[4] This instrument is capable of measuring surface stresses only. For the second technique, an instrument was used to measure the polarization change after double-passing light through the glass. The innovation here was to make this measurement simultaneously over a whole windshield with 2-in.-diameter optics.[5] Figures 10 and 11 show the maximum principal stresses on the windshield's

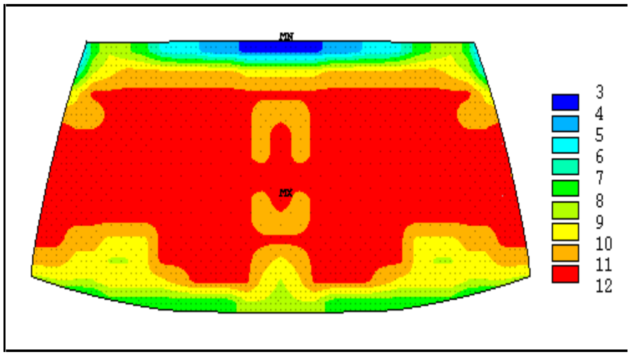

Figure 10. Maximum principal stress (in MPa) on outer surface of windshield.

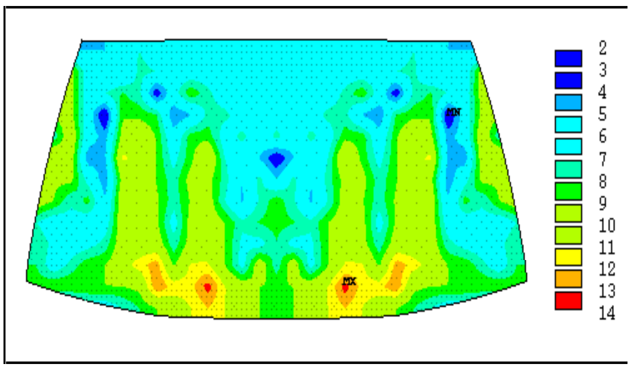

Figure 11. Maximum principal stress (in MPa) on inner surface of windshield.

outer and inner surfaces, respectively. We are using the residual stresses as input to the stone impact models and the design tools. The measured residual stresses are also guiding the manufacturing of the lightweight glass to determine suitable forming times and tempering.

Windshield Resistance to Stone Impact

We developed axisymmetric finite element models to simulate the glass damage evolution when a windshield glass is subjected to impact loading of stones. The windshield glass consists of two glass layers laminated with a thin PVB layer. A model is used to predict and examine through-thickness damage evolution patterns on different glass surfaces. It also examines cracking patterns for windshield design factors such as symmetric or asymmetric glass construction, thickness of glass plies, and curvature. These models are used to determine appropriate glass construction schemes that meet requirements such as strong resistance of glazing to abrasion and particle impact, while at the same time minimizing weight.[6]

We are performing stone impact experiments for conventional and lightweight glass. The windshields are impacted with ¼-in.-diameter steel spheres to simulate windshield damage due to stone impacts. These experiments are used to validate the models. We are also doing staircase (threshold-seeking) stone impact experiments to better determine the values of parameters used in the models. Our models are being validated using controlled experiments done at PNNL. Figure 12 shows a half-symmetry model, and Figure 13 shows the contour plots for web-shape damage for the tri-layered glass constructions under a ball bearing impact of 60 mph. It was predicted that visible damage would be produced on surface no. 2 for all of the four tri-laminated cases. It is important to note that the least damage results when the thin piece of glass is used as the inside layer.

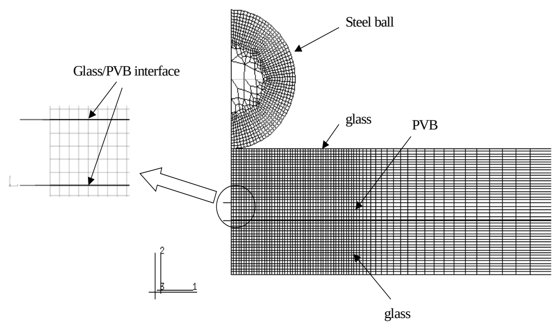

Figure 12. Half-symmetry representation of the finite element model.

A Numerical Tool for Design of Lightweight Glass

We have developed a method for predicting structural failure probabilities for automotive windshields (see ref. 7 for more details). The predictive model is supported by the data from strength tests performed on specimens of automotive glass. Inputs for stresses are based on finite element calculations, or from measurements of the residual stresses that arise from fabrication. The failure probability for each surface or edge subregion of a windshield is calculated from the local state of stress, the surface area or length of the subregion,

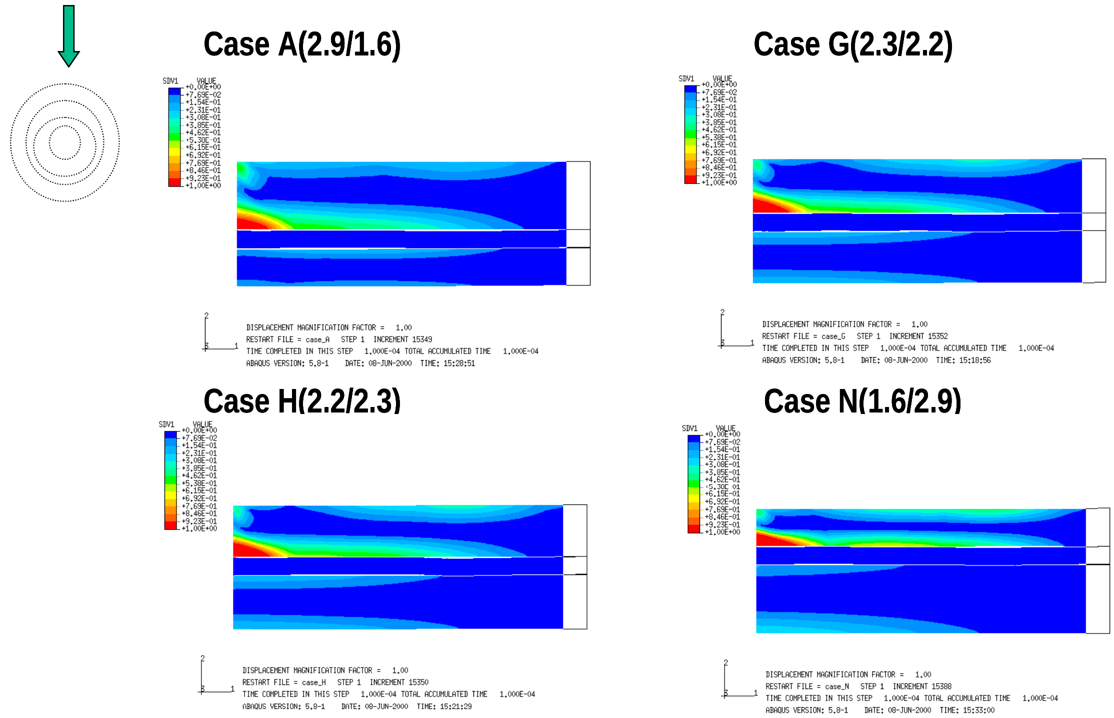

Figure 13. Contour plots of predicted damage for total thickness of 5.2mm (web shape).

and the statistical distribution of glass strengths. Figure 14 shows failure probabilities for an entire windshield due to residual stresses. This new tool will be used to design lightweight glass with reasonable reliability levels as compared with conventional automotive glass. Recent work has resolved concerns with the original probabilistic formulation and has validated the numerical results for cases of high failure probabilities. The work will now focus on applications of the failure prediction model. We have obtained data from PPG for

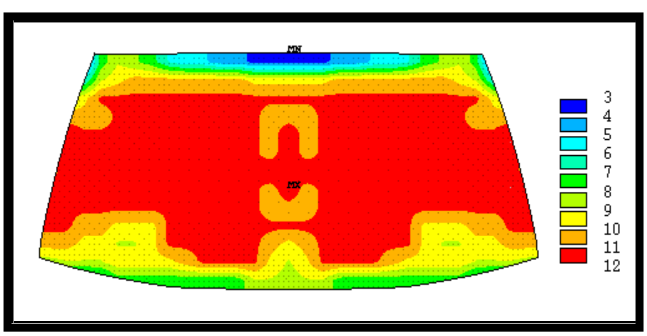

Figure 14. Predicted failure probabilities due to residual stresses (failure probability due to edge flaws is 4.2E-05, and due to surface flaws is 2.0E-07).

measured edge strengths of laminated side-light glass and for measured stresses induced by door slam tests. This information will be used as input to predict failure probabilities for the loading condition of concern. Results will be compared with results of similar calculations to be performed by PPG research staff.

<u>Side Door</u>

New lightweight laminated side glass using 1.6-mm, 2.1-mm, and 2.6-mm plies was fabricated. The new side glass constructions were

- 1.6 mm glass and 0.76 mm PVB or 0.5 mm PVB and 1.6 mm glass
- 2.1 mm glass and 0.76 mm PVB or 0.5 mm PVB and 2.1 mm glass
- 2.6 mm glass and 0.76 mm PVB or 0.5 mm PVB and 2.6 mm glass
-

Side door impact has been conducted at PPG. Figure 15 shows typical strain measurements.

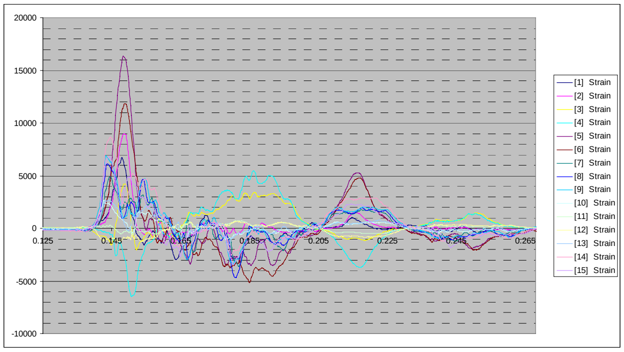

Figure 15. Measured strains from slam test

Future Work

Future work will focus on fabrication of lightweight glass using cost-effective manufacturing technologies. We will continue to seek more weight reduction while maintaining or enhancing glass performance. The static and dynamic strength of the glass will be characterized, and the computational models will be verified. Modeling efforts will continue to focus on stone impact studies and acoustics. Work will also be conducted to enhance and extend the numerical tool for design of lightweight glass.

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4. POLYMER COMPOSITES R&D

A. Development of Manufacturing Methods for Fiber Preforms

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Contractor: DOE Cooperative Agreement
Contract No.: DE-FC05-95OR22545

Objectives

- Develop and demonstrate new fiber preforming processes to decrease cost, increase manufacturing rates, and improve reproducibility of large preforms for composite molding.
- Provide process development support, mainly development of a suitable carbon-fiber roving for use in the programmable, powdered preforming process (P4), in support of Automotive Composite Consortium (ACC) Focal Project 3.

OAAT R&D Plan: Task 3, 10; Barriers A, B

Accomplishments

- Tested a variety of carbon-fiber rovings supplied by various manufacturers in P4. All rovings supplied were essentially “off-the-shelf” and unfortunately did not exhibit characteristics suitable for chopped-fiber preform processing.
- Fabricated flat-panel preforms from carbon-fiber rovings supplied by several carbon-fiber manufacturers. Subsequently used these performs to support structural reaction injection molding trials and material characterization efforts within the ACC.
- Designed a modified fiber-handling system and fabricated a prototype system.
- Performed several upgrades on the ACC’s P4 preforming machine during the first half of FY 2001.
- Tested prototype thermoplastic string binder materials (supplied by PPG) in P4.
- Successfully tested a choppable version of the Vetrotex Twintex roving (commingled polypropylene/glass fiber) in P4.

Future Direction

- Continue to develop and evaluate new carbon rovings.

Carbon-Fiber Roving Development

Currently available off-the-shelf carbon-fiber materials are not suitable for use in chopped-fiber preforming processes. These materials were originally designed for use in filament winding, pultrusion, woven/engineered fabrics, and preimpregnated material manufacture. Due to this fundamental difference in processing methodology, current materials cannot successfully be processed in high-speed chopped-fiber applications. In order to achieve low-cost, random carbon-fiber composites that are suitable for use in the automotive industry, a carbon-fiber roving suitable for use in a chopped-fiber preforming application must be developed. Based on the lack of availability of suitable carbon-fiber materials, a carbon-fiber roving specification was developed and distributed to several carbon-fiber manufacturers. Carbon-fiber suppliers then developed several experimental carbon-fiber rovings for use in chopped-fiber preforming processes.

The ACC's P4 preforming machine located at the National Composite Center in Dayton, Ohio, was used to perform all material evaluations. All materials were processed through an Aplicator SMART chopper gun system. During the carbon-fiber testing program, two separate preform tools were utilized to examine the processing characteristics of carbon rovings and to manufacture panels for destructive evaluations. The two tools are the shape preforming tool (Figure 1) and the flat-panel preforming tool (Figure 2).

Figure 1. Shape preforming tool.

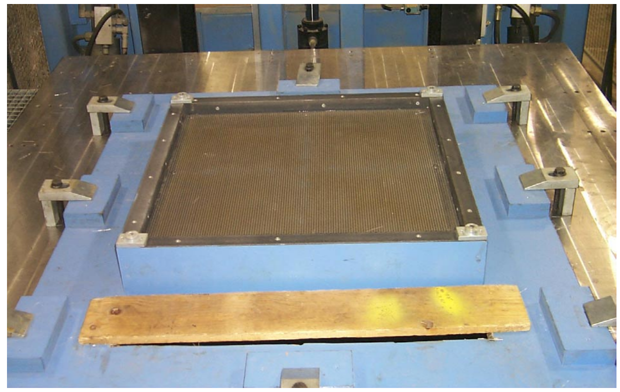

Figure 2. Flat-panel preforming tool.

The shape tool is approximately 195 × 600 × 325 mm ($l \times w \times h$) and contains five separate two-dimensional surfaces. These five surfaces are (from the uppermost surface downward) as follows (angle/$l \times w$): 0°/95 × 600 mm, 90°/115 × 600 mm, 0°/60 × 600 mm, 45°/110 × 600 mm, and 0°/90 × 600 mm. Each surface is blended to another with either a concave or convex radius (6 mm). This allows for a variety of surfaces to be examined for material deposition and conformability characteristics. Shape preforms are also destructively evaluated using a cut-and-weigh procedure to determine areal density distribution. The flat-panel preforming tool is 700 × 700 mm, and the consolidation thickness can be varied with the use of shims along the perimeter of the tool. Flat-panel preforms fabricated on this tool are used for destructive evaluation to determine areal density distribution. In addition, flat-panel preforms were supplied to support ACC molding development and material characterization efforts.

All of the carbon-fiber rovings tested failed to meet the ACC carbon-fiber roving specification and were also determined to be unsuitable for high-volume applications because of preform processing issues. Unfortunately, the material form has not improved significantly, and processing of all the available materials remains somewhat of an issue. Successful development of carbon-fiber rovings will depend upon extensive research and development at the carbon-fiber suppliers to produce rovings to specifications. Currently, carbon-fiber roving development at suppliers is minimal, and off-the-shelf materials not suitable for chopped-fiber processing are being supplied. Unless funding of development programs at carbon-fiber manufacturers can be realized, advanced carbon fibers for chopped-fiber applications will not be developed in a timely manner.

Because of lack of carbon-fiber supplier support in manufacturing carbon-fiber rovings according to the ACC's specifications, new programs are being developed to fund programs for roving development at carbon-fiber manufacturers. It is still unclear whether these programs will be funded directly or via the cooperative agreement. This change in programmatic approach is still under investigation and is dependent upon supplier willingness to perform the proposed research. P4 carbon-fiber preforming on the ACC's preforming machine is currently an ongoing effort, with new materials being developed and tested. The ACC will continue to develop and evaluate new carbon rovings in an effort to create a low-cost carbon-fiber gun roving suitable for use in the P4 process.

P4 Machine Upgrades

P4 machine upgrades included relocation of the binder hoppers, new surface veil choppers with electric servo control, quick electrical connects and disconnects to facilitate moving of the P4 equipment, improvements to the chopper gun SLC program, resolution of hydraulic system oil-leaking issues, replacement of damaged electrical cables, and an improved robot hose and chopper gun offset beam design. Many of the modifications were due to wear and tear of the equipment, and the remaining modifications are the current state of the art in P4 technology.

Prototype Fiber-Delivery System

The prototype fiber-handling equipment reduced friction when processing carbon-fiber rovings, thus allowing improved processing characteristics, including a reduction of fiber fuzzing. The main focus of the prototype system was a reduction in roving contact surface area within the fiber-delivery system. Based on the experimental test results, a second-generation fiber-handling system is currently being designed to further reduce friction by utilizing eyelets with less contact surface, which will improve carbon-fiber roving processibility. In addition, the new handling system will improve processing of both thermoset and thermoplastic string binder materials due to friction reduction.

PPG String Binder

PPG string binder material is a thermoplastic binder strand comingled with the glass-fiber rovings. The thermoplastic strands are composed of either polystyrene and polypropylene or a co-polyester and polyester. Preliminary materials could not be successfully processed due to issues related to fiber strength; however, development of a more suitable material is ongoing. In addition, equipment modifications, including redesigned ejector tubes and a delivery system to reduce friction, are being investigated to process this material more efficiently.

Vetrotex Twintex

Vetrotex Twintex material was processed favorably by P4. Several issues emerged; however, these are not deemed to be severe. A major issue identified was the high preform loft immediately following chopping and also following preform consolidation. Preform loft following chopping was approximately 10 times that of the desired molded part thickness. In the same way, preform loft following consolidation was approximately 5 times that of the desired molded part thickness. The high loft may cause problems on component surfaces with relatively small draft angles (i.e., less than 7°), leading to "wiping" when both the preforming and molding tools are closed. Additionally, the relative lack of stiffness within the thermoplastic strands can lead to fiber jamming within the output tubes. Based on the preliminary test results, a more detailed investigation is currently being planned.

B. Composite-Intensive Body Structure Development for Focal Project 3

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC-59OR22545

Objectives

- Support the goal of Focal Project 3 (FP3) of the Automotive Composites Consortium (ACC) to design, analyze, and build a composite-intensive body-in-white (BIW), while meeting structural and production objectives.
- Achieve BIW structure—compared with steel—(1) achieves cost parity, (2) yields 60% mass reduction, (3) provides equivalent or better structural performance, and (4) affords equivalent better dimensional tolerance.
- Develop high-volume production techniques (>100,000 units per year).
- Provide a focus for bringing together technology developed by each of the ACC working groups through emphasis on carbon-fiber-reinforced composites and the use of hybrid materials, faster manufacturing processes, design optimization, and rapid joining methods.

OAAT R&D Plan: Task 10; Barriers A, B, C

Approach

- Optimize the design and complete the finite element analysis (Phase 1).
- Construct one part of the BIW to demonstrate high-volume processing methods for both component and assembly fixtures (Phase 2). The body component will be tested before continuing with the complete BIW build.
- Build the complete body-in-white (BIW), ensuring that the properties of each part are consistent with those that will be obtained from production tools (Phase 3).

Accomplishments

- Completed Phase 1 of the program, including design optimization and finite element analysis of the selected BIW structure. (The structure meets or exceeds all design requirements.)
- Identified critical manufacturing issues for Phase 2 of the project.

- Began work to build a section of the body side, which will be used as a learning tool to conduct further preforming and molding research.

Future Direction

- Develop the programmable powdered performing process (P4).
- Produce a learning tool to address manufacturing issues.

Introduction

Much of FY 2000 was devoted to completing detailed design and analysis of the chosen BIW concept. The analysis predicts the mass of the BIW to be 86.2 kg, a 67% reduction over that for the steel. The actual mass will be slightly higher because of manufacturing constraints on the variations in thickness; however, we still expect to exceed the target of 60% reduction.

Details of Phase 1

The package was determined to be roughly equivalent to the JA; therefore, extensive concept and styling changes were not possible. The first stage was to optimize the geometric structure to be efficient for composite materials. The philosophy was to produce a BIW with a low panel count, good connectivity, efficient load paths, stability, and—above all—maximized section properties.

Performance, cost, processibility, and minimum thickness were key issues. The structure is based on three sets of carbon materials: random chopped fibers for the majority of panels; stitched or woven material (with core) for the flat, regularly shaped floor and roof structures; and either braided or helically wound lower rails.

From the sound platform of optimized geometry with efficient part integration, the process of optimizing the material content against the prescribed load cases was undertaken. Topological and topographical optimization techniques were developed and utilized on the random material. The considerations for the sheet materials utilized ply-management techniques for local patch and insert reinforcement.

The load cases assessed were bending and torsion stiffness (which were exceeded by 80 and 200%, respectively) and durability and abuse loads to ensure that material fatigue allowables were not exceeded. The finite element model of the final concept for the BIW is shown in Figure 1.

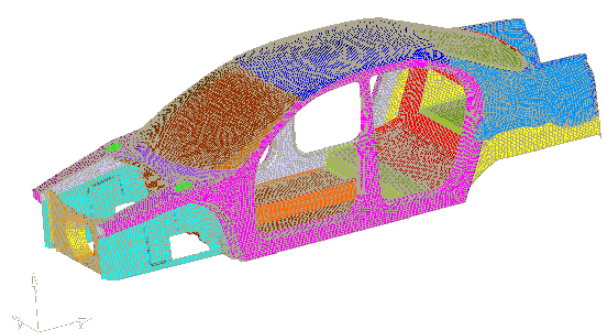

Figure 1. Finite element model of body-in-white.

Crash performance of the structure was developed with respect to two key criteria: controllable crush of the front rails and stability and integrity of the backup safety structure. Nonlinear crash analysis capability is not yet fully developed and therefore was not used for this study. High safety margins are placed on the backup safety structure throughout the stable crush of the rails. The loads from the predicted crash pulse show that a factor of safety of 3 exists for material allowables.

The predicted mass of the complete structure is 86 kg, a 67% reduction over that for the steel BIW mass and comfortably below the 60% target reduction.

The feasibility study undertaken demonstrates that the broad project objectives are feasible, and the group is continuing with Phase 2.

Details of Phase 2

Processing technology is being developed in conjunction with the materials and processing groups. These include

- high-volume processes and assembly
  - expansion of the programmable, powdered preforming process (P4) for carbon
  - evaluation of other processes (as needed)
  - identification of methods to reduce molding time
  - adhesive bonding
- material property characterization of carbon-reinforced composites
- sandwich construction.

The materials and processing groups are developing P4 for carbon fiber. P4 development plans needed for FP3 have been discussed, and preliminary work has begun. Plans are in place to produce a learning tool to address the necessary manufacturing issues to be able to successfully build the demonstration part. The tool incorporates challenging issues requiring development, such as preforming and molding sections of variable thickness. The demonstration part will also be used to address issues related to bonding.

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C. Study of Thermoplastic Powder-Impregnated Composite Manufacturing Technology for Automotive Applications

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Contract No.: DE-AC06-76RL01830

Objectives

- Demonstrate and develop the manufacture of glass- and carbon-fiber-reinforced thermoplastic matrix composites at production rates consistent with those of high-volume automotive structural applications.
- Investigate and develop methods for shortening the process cycle times and improving materials performance.
- Develop methods for achieving a Class A surface finish requirement, consistent with outer body panels for automotive use.
- Investigate the potential for cored (sandwich) structure composites with thin-walled thermoplastic matrix composite skins.
- Determine the cost-effectiveness of thermoplastic matrix composites in production volumes consistent with those required for automotive applications, and the potential to achieve cost targets in the future.

OAAT R&D Plan: Task 10; Barriers A, B

Approach

- Use data from previously completed parametric studies of time, temperature, and pressure profiles required to achieve properties for structural automotive composites and determine combinations of processing factors that will accelerate the process cycle to under one minute.
- Develop methods of heating and cooling and process operations to achieve consistent cycle times.

- Analyze alternative methods of melt processing for the various operations required for consolidation, including experimental evaluation of cored structures.
- Apply a multi-scale materials modeling approach to develop predictive numerical models for thermal and structural responses for constituent materials and architectures; apply those models to processing conditions for cycle optimization, material properties predictions, and end-use performance predictions.
- Use data to influence materials suppliers in modification of base material feeds in ways that support increased cycle times.
- Use complex-shaped components to determine process limitations and factors for economic evaluation.
- Evaluate methods of achieving a Class A surface finish with reinforced thermoplastic matrices and analyze them in production cycles on complex tooling.

Accomplishments

- Developed a two-press system and mold clamping/shuttle arrangement to integrate novel process parameters for accelerating heating and cooling cycles.
- Used the heated/cooled cone mold for process and formability trials to fabricate test components.
- Developed a method for tabbing and analyzing thin, curved compressive test samples from the conical parts fabricated in the formability mold.
- Investigated all compatible core materials for processing and properties and determined two acceptable core materials, based on structural and processing factors. (Core processing appears to be mainly useful with higher-temperature matrices if a two-step process is used. Lower-temperature thermoplastics will limit automotive applications.)
- Developed a new processing method for continuous processing with multiple tooling sets and with optimized heat/cooling methods. Delphi has filed a record of invention for the process.
- Analyzed surface films with in situ and post-processing methods for achieving a Class A surface on highly loaded structural components. Methods with films and veils were generally unsuccessful, but this work has led to a novel method developed by Pacific Northwest National Laboratory (PNNL) of process and material modifications that have promise for achieving Class A structural thermoplastic composites. PNNL has filed invention disclosures on the system.
- Developed a method of materials processing at PNNL to integrate with the traditional heating/cooling cycle, which has achieved greatly reduced cycle times in small panel bench tests. The method is being further developed to determine whether scaling is a factor and whether it will be applicable to production cycles.
- Analyzed materials production and materials forms with the suppliers to determine cost and volume potential and material supply limitations.
- Determined cost/volume scenarios for thermoplastic TP components for different processing methods.
- Developed initial macro-model results indicating the potential to predict important process parameters.
- Refined meso-scale models of the fabric structure to predict formability relationships for stamp forming of 2×2 twill-weave fabrics.
- Developed a family of micro-scale models for unidirectional composite (fiber bundle) property predictions.

Future Direction

- Refine process models to predict formability limits under various process conditions.
- Continue meso-scale (fabric) model developments to investigate effects of weave architecture on woven lamina properties.
- Fully develop, integrate, and document the models in the multi-scale approach.

- Develop process and test equipment for a production cycle demonstration of thin cored structures.
- Develop a methodology for control of "print through" to achieve a Class A surface and adapt to higher-rate processing.
- Develop tooling and production cycle for demonstration of continuous process methods for achieving cycle times of less than 1 minute.
- Develop novel process methods to determine their capability for implementation into higher-volume production.
- Work with materials supplier to adapt to other materials forms that are more cost-effective and that can achieve increases in production cycle times.

Introduction

Polymer matrix composites are a class of materials identified as having a combination of properties that are required to achieve significant reductions in vehicle mass compared with conventional materials. Although they are widely regarded as being the material with the most potential for future weight savings, their use is severely restricted by fundamental issues with achieving the necessary production volumes and with the cost of basic materials. These issues have been studied and analyzed based primarily on liquid thermosetting methods for matrix composites, such as structural reaction injection molding (SRIM) as used in the Automotive Composites Consortium (ACC) focal projects. The cost and availability of fibers and materials, especially carbon fibers, are being addressed by DOE under the low-cost carbon fiber projects. This project has identified carbon fiber thermoplastic matrix composites as having significant potential to meet structural property requirements and to achieve rapid processing cycles similar to the cycles for stamping of metal components. To recognize the potential for widespread use of polymer matrix composites in automotive applications, two other fundamental issues must be addressed—cored, thin-walled structures (for optimum structural performance and minimum weight) and Class A surfaces in semi-structural components. Developing thermoplastic composites to meet these requirements will require a combination of experimental approaches, materials and process modeling, and modification of materials forms and processing methods to achieve an optimum manufacturing process.

Project Deliverables

This multi-year program will develop and demonstrate knowledge concerning four technology areas associated with the thermoplastic composite forming process: (1) results of process cycle times and materials properties achievements at high forming rates; (2) results of Class A surface finish trials; (3) methods for the manufacture of thin face-sheet cored structures; and (4) modeling tools for predicting and optimizing the thermal and structural materials behavior for fiber-reinforced thermoplastics.

Planned Approach

Experimental and analytical methods are being used and developed for high-rate forming of thermoplastic composite sheet and cored structures. The project team is focusing on achieving automotive stamping production rates and on materials modifications to achieve rapid flow and consolidation. By integrating these two approaches, the project is able to determine where difficulties in the process and materials form occur and suggest methods to overcome these barriers. Several significant invention disclosures have resulted, and a further focus on the process and materials kinetics will allow the bench scale demonstration of high-rate stamping.

Experimental Methods

The initial experimentation used flat plate molds and developed a process diagram based on achieving a pre-defined "acceptable" quality of consolidation, as measured by compressive strength of the coupons. These process conditions were used as a basis for the processing of a conical mold that had varying angles of fabric shear (deformation) and varying forming radii (both convex and concave) to provide an understanding of the forming limits under different process conditions (Figures 1 and 2). Coupons have been taken from the conical mold and tested in compression to match the consolidation results to the results from the basis flat plate mold. Areas showing varying degrees of resin content and fiber deformation were selected for study. Compressive strength data collected so far on cone mold formed parts are summarized in Table 1.

Methods for achieving rapid process cycles were developed as an ongoing part of the conical mold trials, and a two-press system was developed over the past year to simulate a newly developed continuous cycle process. The approach will be developed and refined in the coming year with novel methods of integrating the heat and cooling cycles to reduce overall cycle time and to address thermal load issues.

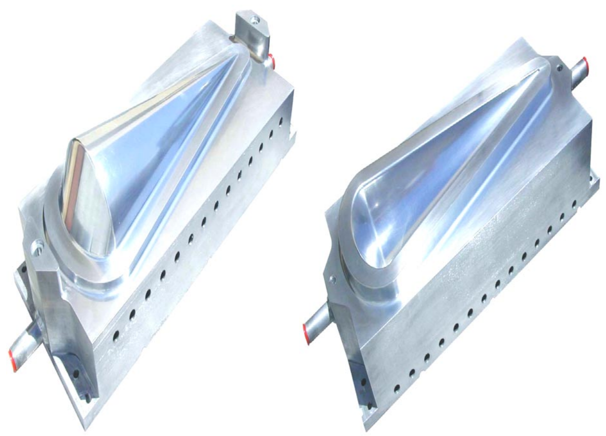

Figure 1. Heated/cooled cone mold for formability trials.

Figure 2. Forming trial parts.

Class A surface trials were run with films and veil materials that have been employed in the liquid molding industry for improving surface finish. These trials were conducted with the flat and cone mold tooling. Under the higher pressures and thermal cycle conditions for this process, they proved ineffective for approaching Class A surface conditions, except at unacceptably large thicknesses. The development and study of conditions have led to a novel method of controlling surface finish that is under experimental and theoretical investigation now, which could allow a significant reduction in the thickness requirement of the surface film/veil.

An experimental study was made of potential core materials for one-stage and two-stage forming of cored structures. The analysis proved valuable in separating manufacturer claims for capabilities of the core materials from the actual behavior demonstrated in rapid process cycle conditions (Figure 3). The materials that seem to have the most potential for a one-step process are the phenolic and end-grain balsa cores, since both can withstand the temperatures and pressures of processing. Cored panels are very sensitive to requirements for face-sheet processing; therefore, the selection of materials and processing parameters has to be based on part requirements. A process cost study was done to determine the effectiveness of the different approaches (Figure 4).

Analytical Methods

A multi-scale modeling approach is being applied to develop modeling tools with long-term value to the technology of fabric-reinforced thermoplastic composites. As illustrated in Figure 5, three important scales have been addressed. At the micro-scale, the fiber and matrix properties are captured, along with their interactions that depend on microstructure. At the meso-scale, the fiber bundle properties from the micro-scale models are used to simulate the composite material response for particular weave architectures. Finally, properties predicted at the meso-scale are applied in macro-

Table 1. Maximum failure stress of formed sections (MPa)

P (MPa)	Specimen location	1 minute				3 minutes				6 minutes			
		Center	12.5 mm from center	25 mm from center	37.5 mm from center	Center	12.5 mm from center	25 mm from center	37.5 mm from center	Center	12.5 mm from center	25 mm from center	37.5 mm from center
1	Left side	259	130	318	214	279	165	187	225	257	382	298	304
	Right side	325	298	92	251	345	172	252	221	319	333	349	251
1.7	Left side	289	262	224	190	341	354	314	255	181	302	310	251
	Right side	214	189	126	93	402	376	342	255	241	303	244	303
2.4	Left side	147	310	162	206	262	276	195	207	286	329	214	258
	Right side	163	122	230	127	263	259	179	213	280	200	216	248
	Average	233	218	192	180	316	267	245	229	261	308	272	269

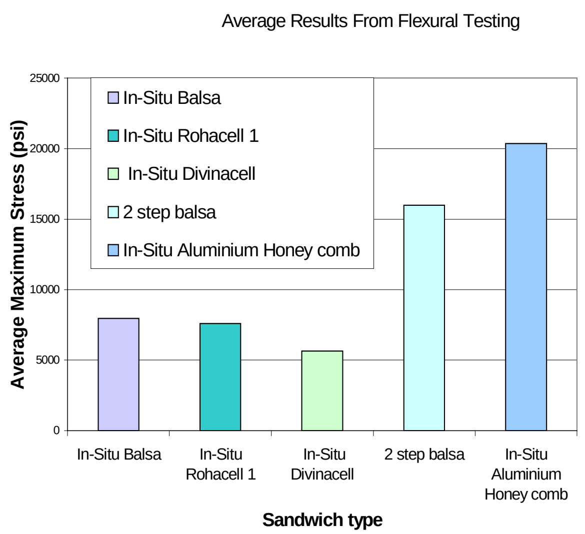

Figure 3. Sandwich panel mechanical testing evaluation.

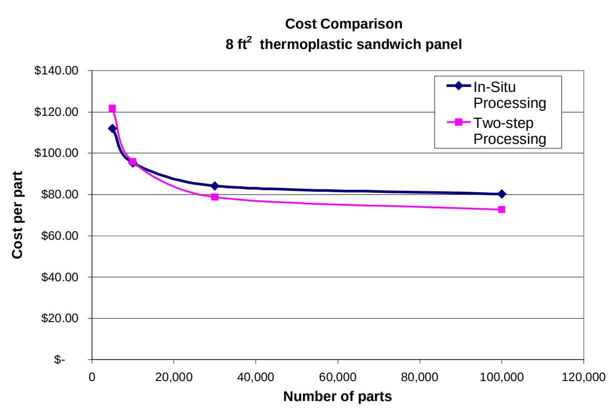

Figure 4. Cost evaluation of core panel manufacturing.

scale models that predict performance in the manufacturing and application environments. The implicit goal of any multi-scale modeling approach is to create ties between properties applied at the macro-scale and the features of micro- and meso-scale models. This is particularly desirable for composite materials because the macro-scale properties are difficult to characterize completely by experimental testing alone.

The finite element method is the fundamental general-purpose modeling tool applied at all three scales. The unit of construction for a fabric architecture is a bundle of fibers that, at any point along the bundle, consists of unidirectional fibers adhesively bound together by a thermoplastic polymer. Therefore, the properties of the bundle can be represented by the unidirectional fibers with interstitial polymer. Here, it is assumed that the fibers are arranged in a periodic array so that a small periodic unit is representative of the entire bundle. Several variations of this micro-scale model have been developed and used to predict thermal and structural properties of consolidated fiber bundles.

At the meso-scale, the fiber bundles are arranged in woven patterns so that the fiber bundles are intertwined in coherent form to facilitate the formation of thin planar structures. By nature, the weaves have a periodic structure that facilitates a small representative volume of the fabric as the basis for a meso-scale model that can predict the detailed behavior of the fabric, which in turn can be homogenized to estimate macro properties of the composite. Thermal and structural models of a plain weave have been constructed, and thermal models

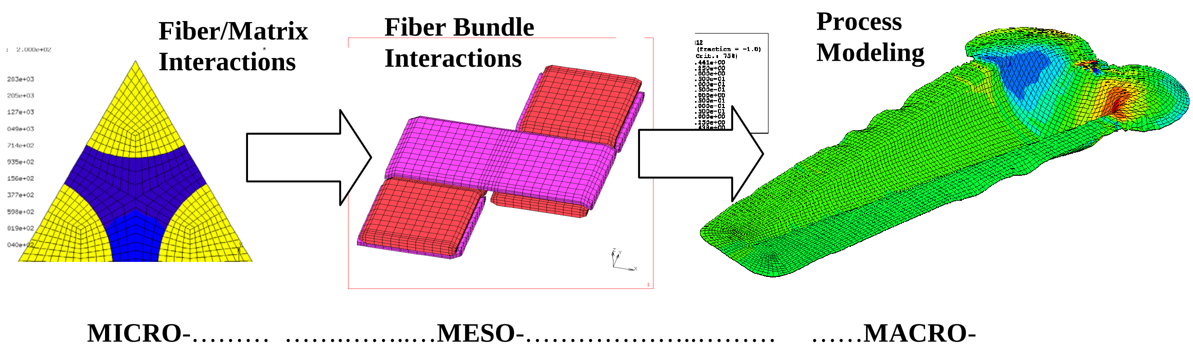

Figure 5. Multi-scale modeling of fabric-reinforced composites. Shown are a hexagonal fiber pack unit cell for micro-scale modeling, a 2×2 twill fabric unit cell for meso-scale modeling, and cone forming process model results at the macro-scale.

have been developed for a 2 × 2 twill weave material. For further understanding of the effects of fiber architecture, work has been initiated on a 4-harness satin weave meso-scale model. At the macro-scale, it is desirable to predict performance in the manufacturing and application environments. In this project, the immediate interest is in modeling of the forming process as it relates to the formability of particular weaves and heat transfer properties under forming cycle conditions. Process models have been developed to initially study the effect of in-plane shear stiffness on formability and drape characteristics for the cone-shaped mold. In addition, macro-models are being applied to predict in-mold pressure distributions during forming. These pressure distributions and their evolution relate to the time required at pressure for complete consolidation of the composite to occur.

Infrared tomography experiments have been conducted to collect data for verification of thermal model predictions. However, the matrix materials under consideration are strongly transparent to infrared radiation. This infrared transparency has the effect of "seeing" the fiber surfaces internally, rather than an overall thermal response of the composite. This effect has complicated the interpretation of the experimental data for comparison with the models.

Future work will be directed at better numerical representations of the die surfaces and material properties. Improved properties will result from predictions of the meso- and micro-scale models. In particular, meso-scale models will predict in-plane shear stress-strain histories that ultimately relate to formability at the macro level. In addition, alternate element formulations will be employed to permit through-thickness stresses currently unavailable from the shell element used in the forming process models. This will be useful for predicting final thickness distributions and transient pressure distributions. As the models are refined, more comparisons will be made with experimental data associated with cone mold forming trials.

Conclusion

The technical feasibility of stamp forming of thermoplastic carbon/nylon composites for automotive applications has been demonstrated for simple flat panel shapes and is being further developed for complex-shape forming. The investigation and modeling results have led to the development of some novel processes and technologies that have the potential to further advance the forming science, surface appearance, and applicability of these material forms. Significant challenges still lie in optimization of the process and its development into a commercially viable robust process, especially given the requirements for cored thin-wall structures and Class A surfaces for structural components. Materials costs and production volumes are within acceptable limits for large-scale automotive applications.

5. LOW-COST CARBON FIBER

A. Low-Cost Carbon Fibers from Renewable Resources

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objective

- Investigate and establish proof-of-principle processing conditions for several types of low-cost carbon fiber precursor materials that are suitable—in properties, volume, and cost—for use by the automotive industry.

OAAT R&D Plan: Task 5; Barriers A, B

Approach

- Evaluate the spinning of fiber from high-volume renewable and recycled fiber streams.
- Evaluate carbonization and graphitization of renewable and recyclable fibers using a variety of bench techniques (e.g., thermogravimetric analysis, micrography, resistance/conductance).
- Evaluate the effects of modern carbon fiber production technologies (e.g., hot stretching, controlled thermal and atmospheric environment) on selected fibers to improve properties.
- Focus on factors (feedstock availability and cost, yield) critical to economic feasibility.
- Evaluate mechanical and composite properties of graphitized melt-spun fibers.

Accomplishments

- Found effective stabilization, carbonization, and hot-stretching techniques for single-strand, melt-spun, lignin-based fiber.

- Developed compositions that decrease water uptake during storage and shipment of lignin-blend fibers.
- Prepared and evaluated scanning electron micrographs of a wide variety of raw, carbonized, and graphitized fibers.
- Obtained preliminary data that indicate that yields of 50%, consistent with those obtained commercially for lignin-based activated carbon, are feasible.
- Working with Westvaco, prepared and provided large amounts of hardwood kraft lignin for project use.
- Successfully prepared lignin-blend fibers with a dominant graphitic content at a firing temperature consistent with intended use.

Future Direction

- Spin, characterize, and oxidize/carbonize/graphitize a wide variety of single fibers.
- Evaluate data on the physical properties of several recyclable petrochemical polymer and lignin-blend fibers using bench-scale oxidation and carbonization facilities.
- Scale up fiber spinning and treatment to permit evaluation of mechanical properties of several experimental fibers.
- Systematically evaluate fiber properties and economics to downselect the number of fiber formulation systems under active investigation.
- Attempt melt spinning of multiple fibers to create a small tow (20–40 fibers) to (1) provide a better basis for understanding downstream spinning issues and (2) provide material for small composite tests of successful fiber systems. Contracts for this activity are being placed with North Carolina State University and the University of Tennessee.

Introduction

This project focuses on development of carbon fibers from high-volume, low-cost, renewable or recycled fiber sources to reduce precursor and processing costs. Use of these materials also decreases the sensitivity of cost to changes in petroleum production and in energy costs. Earlier literature reports indicate that a wide variety of fibers—kevlar, nylon, polyacetylene, polyethylene, polystyrene, and pitch—as well as natural and renewable products such as wool, rayon, and lignosulfonate were also used to make low-performance carbon fibers in the 1960s and 1970s. In quality, cost, and volume, kraft lignins and recycled petrochemical polymers are attractive in the United States. This project will evaluate the feasibility of preparing carbon fiber precursors from a variety of high-volume renewable and recycled polymers.

Project Deliverables

By the end of this multi-year program, the production of one or more environmentally friendly, economically feasible carbon fiber precursors will be demonstrated.

Planned Approach

Producing low-cost, high-volume carbon fiber precursors requires the simultaneous development of processes for making the feedstock fibers and of downstream processing techniques that improve the properties of each type of fiber. Because of high levels of emissions and costs typically associated with spinning of fiber from liquids, first priority was placed on development of melt-spinning techniques for fiber. The use of lignin and other non-nitrogenous feedstocks would eliminate cyanide emissions during furnacing. The use of modern furnacing techniques, such as hot-stretching and controlled-atmosphere processing, is being evaluated to improve the properties and yield of carbon fiber precursors from feedstock. Promising fiber blends will be prepared as precision fibers and will, additionally, be graphitized to permit the evaluation of finished fiber structure and mechanical properties of individual fibers and of composites.

Carbon fiber for use in aerospace or recreational products is typically produced in three steps: (1) spinning, (2) stabilization/ oxidation [with hot stretching, in the case of polyacrylonitrile (PAN], and (3) carbonization/ graphitization under reduced oxygen or inert atmosphere to increase strength and stiffness. Systematic evaluation will be required to find appropriate conditions for spinning and furnacing melt-spun lignin-based fibers.

Fiber Spinning

Melt-spun polymer blends of kraft lignin, which is commercially available as a byproduct, received significant attention. Lignin is a ubiquitous polymer that typically constitutes 20–30% of wood and woody biomass. In pulping, lignin and lignin compounds are separated from cellulose and either burned or recovered and sold as a byproduct. Detailed estimates place the volume of lignin produced and burned by the U.S. paper industry at around 1000 times the present volume of worldwide carbon fiber production. Thus kraft lignin could support production of the amounts of fiber needed by the automotive industry. Gasification, which is being evaluated as an alternative to current lignin combustion processes, could provide significant amounts of lignin as well as both high-temperature process heat and low-cost electricity. Development of this process could greatly increase availability of carbon fiber and decrease the price.

Studies of melt-spun single lignin-blend fibers have continued during this year. Because of its narrow melting point range, Westvaco hardwood kraft lignin has been used. As reported earlier, it was desalted prior to use to prevent void formation during graphitization.

Single fibers were spun to our specifications from prepared lignin mixtures by North Carolina State University staff and returned on cardboard spools for our evaluation. Although the current fibers are strong enough to withstand high-speed spooling and to permit hand lay-up on quartz for furnacing, fibers produced by the Atlas Laboratory mixing extruder have varying diameters that are typically larger than those of fibers typically used in composites. These problems can be solved with more uniform stretching techniques and larger draw ratios.

Since results with single fibers are favorable, a decision was made to spin amounts of multiple precision fibers for graphitization and mechanical property evaluation. Subcontracts for multiple fiber spinning are being placed.

Stabilization, Carbonization, and Graphitization

Conditions that provide good stabilization of lignin fiber blends containing polyesters, polyolefins, and polyethers have been profiled. Computer-controlled furnacing and a novel technique for stretching single fibers are producing dense, solid fibers with little or no inter-fiber adhesion or melting.

After stabilization, the lignin-blend fibers are carbonized in a reducing atmosphere, again with hot stretching. Carbonized lignin-blend single fibers are dense and compact with relatively few visible defects.

Lignin-blend fibers have been graphitized to temperatures ranging between 1600 and 2400°C (Figure 1). Yields remain near 50%.

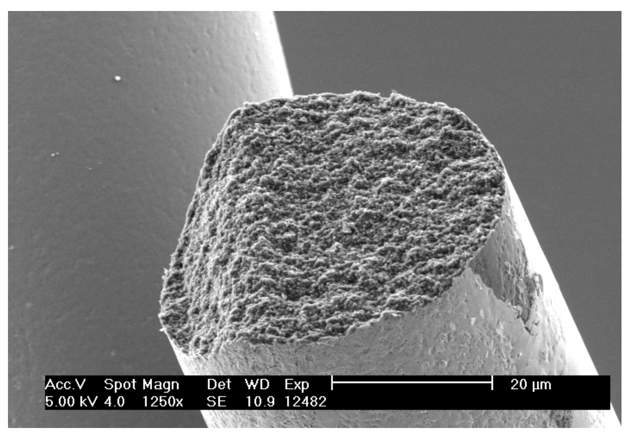

Figure 1. A scanning electron micrograph showing the end of a graphitized lignin-blend fiber.

Development of Graphitic Structure

Raw, stabilized, carbonized, and graphitized lignin-polyester blend fibers have been evaluated. Crystalline structure was not apparent in raw, stabilized, or carbonized materials. As shown in Figure 2, graphitic structure is increasingly pronounced as firing temperature increases, as indicated by powder X-ray diffraction analysis.

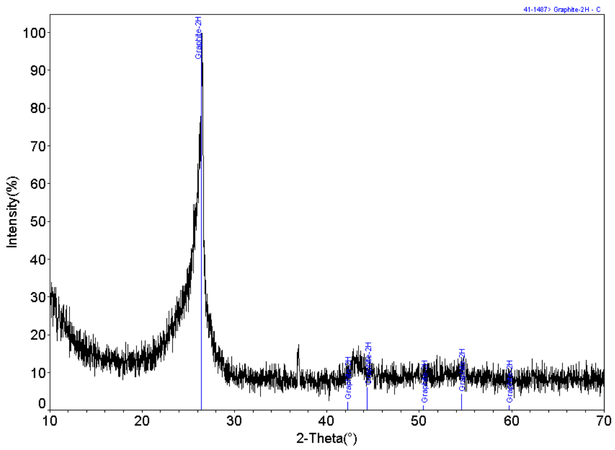

Figure 2. Powder diffraction data on graphitized lignin-blend fiber showing the predominance of graphitic structure.

Future Directions

Proof-of-concept production of single lignin-blend fibers indicates the potential for use of this material as a feedstock for industrial-grade carbon fibers. To determine whether the technology is feasible, melt-spinning and processing of multiple fibers will be evaluated. Hot-stretching and furnacing conditions for multiple fiber tows will be evaluated. Surface treatments and sizing to permit the production of small composites for properties tests will be evaluated.

Conclusions

Several lignin-based blend fibers that can be oxidized, carbonized, and graphitized have been demonstrated using equipment and techniques developed during this project. Preliminary evaluations indicate that production of carbon fiber precursor from renewable and recycled materials is likely to be feasible. The yield of fiber is ~50%.

Further studies are under way to (1) extend the range of evaluated fibers, (2) continue evaluation and improve processing conditions, (3) spin and process multiple small-diameter fibers, and (4) begin evaluation of multiple fibers and composite properties.

B. Low-Cost Carbon Fiber Development Program

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Contractor: Hexcel Corporation
Contract No.: 450001675

Objective

- To define technologies needed to produce a low-cost carbon fiber (LCCF) for automotive applications at a cost of $3.00 to $5.00 per pound in quantities greater than one million pounds per year. The required carbon fiber properties are tensile strength of greater than 400 ksi, modulus of greater than 25 Msi, and strain at failure greater than 1%.

OAAT R&D Plan: Task 5; Barriers A, B

Approach

- Develop new precursors that can be converted into carbon fiber at costs below the costs of current processes.
- Explore processing by other methods than thermal pyrolysis.
- Develop technologies for significant improvement in current production methods and equipment.
- Develop alternative methods for producing carbon fiber from pitch, polyacrylnitrile (PAN), or other precursors.
- Reduce precursor cost by the use of commercially available energy-efficient precursors and high conversion yields.
- Improve precursor production economics of scale and throughput.
- Introduce novel low-cost carbon fiber production methods.

Accomplishments

- Evaluated proposed research areas through laboratory trials and refinement of manufacturing cost analyses. These focused on
  - PAN-based precursors: large-tow benchmark, commodity textile acrylic tow, chemical modifications, acrylic fibers spun without solvents, and radiation and nitrogen pretreatment of PAN-based materials.
  - Precursors other than PAN: polyolefins—polypropylene (PP), linear low-density polyethylene (LLDPE) and high-density polyethylene (HDPE); polystyrene; and polyvinyl chloride (PVC) pitch.
- Scaled up proposed research areas to pilot line trials.
- Assessed the technical and economic feasibility of the proposed research areas.
- Down-selected the most promising technologies to meet the program objectives:
  - Commodity textile acrylic tow with chemical modification and/or radiation pretreatment.
  - Polyolefins (LLDPE and PP).
- Developed detailed manufacturing cost models for the down-selected technologies.

Future Direction

- Continue pilot-scale and scale-up experimental trials and assessment of the manufacturing cost of the down-selected technologies.
- Down-select the LCCF technology(ies) to be developed for large-scale engineering and commercial manufacturing feasibility.
- Develop manufacturing project plans that include the configuration of facilities, sites, supply of raw materials, and economic feasibility studies for the selected technology(ies) for the large-scale production of LCCF for automotive applications.

Introduction

The goal of this program is to define and demonstrate technologies needed for the commercialization of LCCFs to be used in automotive applications. Lighter-weight automotive composites made with carbon fibers can improve the fuel efficiency of vehicles and reduce pollution. In order for carbon fibers to compete more effectively with other materials in future vehicles, their cost must be reduced.[1–3] Specifically, this program targets the production of carbon fibers with adequate mechanical properties, in sufficiently large quantities, at a sustainable and competitive cost of $3 to $5 per pound.

Project Deliverables

At the end of this multi-year program, technologies for LCCF production will be defined. This definition will include the required materials and facilities and will be supported by detailed manufacturing cost analyses and processing cost models. Laboratory trials and pilot-scale demonstrations will be performed to support the defined technologies.

Planned Approach

This program was divided into two phases (Figure 1). **Phase I** (15 months): Critical review of existing and emerging technologies, divided into two tasks:

Task 1.1. Literature review and market analysis.
Task 1.2. Laboratory-scale trials and preliminary LCCF manufacturing cost assessments of the proposed technologies. Phase 1 led to further refinement and down-selection of the most promising technologies for Phase 2 (Table 1).

Phase 2 (27 months): Evaluation of selected technologies using pilot-scale equipment and cost models. Phase 2 was divided into three tasks:

Table 1. Phase 1 low-cost carbon fiber (LCCF) development program technology downselection

Precursor	Technology	Primary LCCF development issues		Continued in Phase 2?
		Technical	Cost	
PAN-based	Large-tow benchmark		X	–
	Textile acrylic	X		X
	Chemically modified	X		X
	Acrylic fibers spun without solvents	X	X	–
	Radiation-treated	X		X
	N_2-prestabilized	X		X
Not PAN-based	Polyethylene	X		X
	Polypropylene	X		X
	Polystyrene	X	X	–
	Polyvinyl chloride	X	X	–

Task 2.1. Pilot-scale design for the evaluation of selected LCCF technologies. This included modifications of a PAN spinning pilot line and two different carbon fiber conversion lines (a single-tow research line and a multi-tow pilot line) and the construction of continuous sulfonation processing equipment for polyolefin fibers.

Task 2.2. Experimental evaluation of down-selected LCCF technologies, including commodity textile tow PAN (with chemical modification and radiation and/or nitrogen pretreatment) and polyolefins (LLDPE and PP).

Task 2.3. Large-scale feasibility study of selected LCCF technologies.

The planned approach included decision points within each of the tasks in order to further down-select and define the most promising technology(ies) that will meet the program objectives, as indicated in the overall LCCF program timeline (Figure 1).

<u>Polyacrylonitrile</u>

Large-Tow Benchmark

Carbon fibers from PAN precursors can be made in a variety of product formats (e.g., tows, mats). High-rate automotive composite manufacturing processes such as the programmable powdered performing process (P4) (ref. 1) use continuous tows as their feedstock. Large tows (>24,000 filaments) can be produced more economically than typical aerospace-grade tows (≤24,000 filaments). However, the manufacture of carbon fibers from large tows presents a variety of technical challenges. These challenges were addressed in three ways. First, small tows were combined to investigate the effects of various parameters (tow count, filament diameter, tow width, air flow velocity and orientation) on large-tow processability. We then carried out the conversion of a large-tow PAN precursor regarded as an industrial benchmark (Acordis large-tow precursor). The equipment modifications and parameters needed to safely process the precursor in a carbon fiber pilot line were established. As a result, large-tow precursors were processed and converted to carbon fibers with mechanical properties in excess of LCCF program targets (tensile strength of up to 516 ksi, modulus of up to 30 Msi). However, the high cost of this precursor precludes its use for LCCF purposes. We then contacted alternative large-tow PAN manufacturers and obtained less expensive developmental precursors for testing. Work on large-tow developmental precursors again allowed us to exceed LCCF targets (tensile strength of up to 463 ksi, modulus of up to 30 Msi).

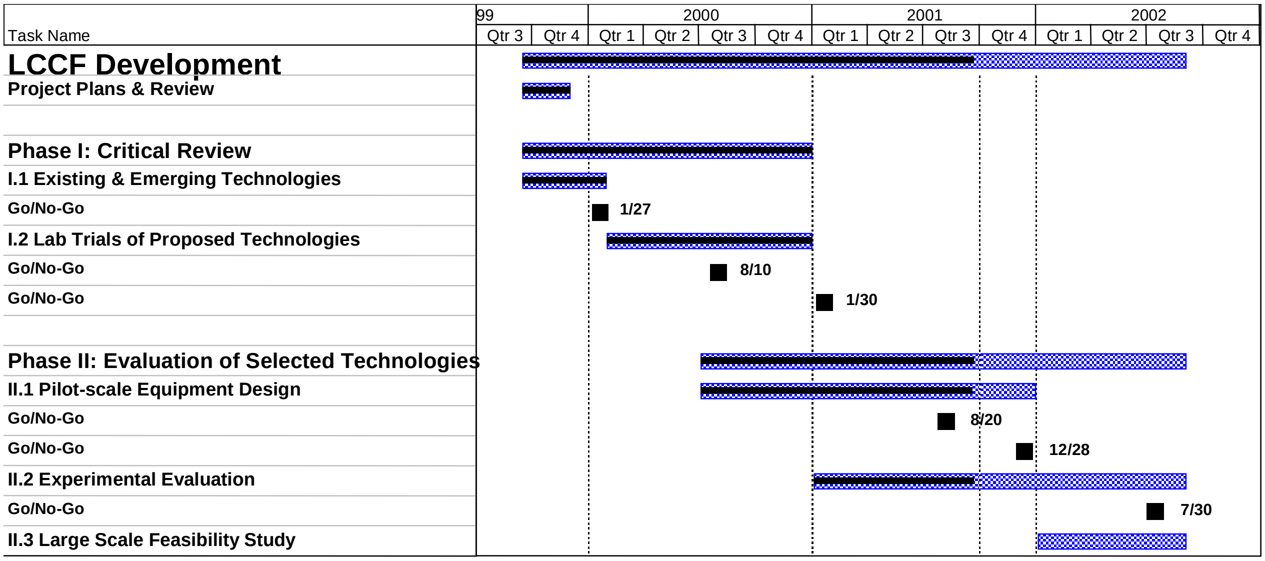

Figure 1. Low-Cost Carbon Fiber Development Program timeline.

Extensive discussions with large-tow PAN suppliers also allowed us to address factors that may lead to lower LCCF costs. These include PAN chemistry, spinning parameters, PAN filament characteristics, tow band uniformity and splittability, and tow packaging issues. For instance, supplying tows in compact bales instead of boxes could reduce packaging and shipping costs, and hence PAN fiber costs, by as much as 15%. However, additional trials with developmental precursors indicate that cost-saving measures can compromise their LCCF performance. The knowledge gained in processing large-tow PAN precursors is being applied to support our work with more economical large-tow PAN derived from commodity-textile-grade PAN polymers.

Commodity Textile Acrylics

Commodity-textile-tow PAN is commercially available in large quantities at about half the cost of large-tow PAN precursors. Its use as a LCCF precursor could thus lower the carbon fiber manufacturing costs to LCCF program target levels. However, the properties of commodity textile PAN are less than optimum from a carbon fiber conversion perspective. Textile-grade PAN polymers are usually made with relatively large amounts of vinyl acetate or methyl acrylate comonomers, and without acidic comonomers or other stabilization enhancers. These polymers are generally wet-spun or dry-spun in large production lines. The large tows are combined, sized, crimped, and compacted in bales. The resulting tows are unsplittable and are difficult to process in conventional carbon fiber lines.

To address those difficulties, raw materials (fibers and polymers) were obtained from various textile PAN suppliers. Initially, wet-spun or dry-spun textile PAN tows made with methyl acetate or vinyl acetate comonomers, and commercially available in small filament deniers, were tested. Lab trials followed by pilot line conversion trials of selected materials yielded conditions at which thermal runaway reactions, filament fusion, and other processing difficulties could be overcome. However, the mechanical properties of the resulting carbon fibers fell below the LCCF program targets (see Table 2). In addition, the relatively long oxidation time and low carbon yield of textile PAN (Table 2) can be expected to increase LCCF manufacturing costs. Laboratory-scale development of novel PAN modifications and conversion technologies led to several ways to improve the potential of commodity textile PAN as an LCCF precursor. These include chemical modification, radiation treatment, and N_2 prestabilization.

Table 2. Comparison of properties of carbon fibers made from large-tow polyacrylonitrile (PAN)

Parameter	Large-tow PAN benchmark	Developmental PAN precursor	Commodity textile tow PAN
Filaments per tow	322,000	332,000	666,000
Tensile strength, ksi	516	463	121
Modulus, Msi	30	30	11
Strain to failure, %	1.6	1.5	1.1
Carbon fiber density, g/cm^3	1.79	1.77	1.67
Carbon fiber yield, % wt.	54	43	45
Relative oxidation time	1	1.36	1.61

Chemically Modified Acrylics

Chemical modification of acrylics is one of several methods reported in the literature to accelerate the rate-limiting stabilization stage in the carbonization process. Lab-scale investigation identified an inorganic oxidizing agent (hydrogen peroxide) in an alkaline medium as the most effective modifier of PAN polymer/fiber. Evaluation of modified polymers and fibers by various analytical methods indicated that partial oxidation of PAN had been accomplished. Pilot-line trials demonstrated that adding a chemical modification step in the PAN spinning line prior to drying would induce a similar degree of partial oxidation in the spooled precursor fiber. Several factors—such as modifier composition, fiber morphology, and application process—were optimized. As expected, optimally modified PAN stabilized in approximately half the time required for its virgin counterparts. Properties of carbon fiber made from chemically modified precursor are shown in Table 3.

Table 3. Properties of carbon fibers produced from chemically modified specialty polyacrylonitrile (PAN)

Tensile strength (Ksi)	Tensile modulus (Msi)	Strain at failure (%)	Density (g/cm^3)	Mass per unit length (g/m)
420	24.5	1.71	1.746	0.215

Following these encouraging initial results, the focus turned toward adapting the modification process to more economical textile PAN fibers. Exploratory lab work on chemical modification of textile PAN polymers indicated partial oxidation similar to that in the precursor polymer. Adaptation to an in-line modification process for textile fiber, similar to that used in the PAN pilot-line trials, began in the fourth quarter of the second year of this project. Textile polymer with vinyl acetate (vinyl acetate is more cost-effective than methyl acetate) was purchased from a textile fiber manufacturer. It was successfully spun on the PAN pilot line and enough textile polymer–based fiber was made for further work. In parallel, a cost analysis of carbon fiber made from chemically modified textile PAN fiber showed that reduced stabilization time translates to lower capital investment and ultimately to lower carbon fiber production cost.

Work for year 3 of the program is planned as follows:

1. Spin textile polymer (control and chemically modified) on the PAN pilot line.
2. Prepare several tow sizes (3, 12, 48, 84 K, etc.) by combining smaller tows.
3. Carbonize various tow sizes to model the effect of tow mass on various stages in the conversion process.
4. Optimize the oxidation/carbonization process to maximize mass throughput and mechanical properties and minimize carbon fiber cost.
5. Identify process parameters for scaling up to standard textile tow counts (>300K).

6. Collaborate with textile fiber manufacturers to implement the process on a production line.

Acrylic Fibers Spun Without Solvents

Work on this topic was substantially reduced in the fourth quarter of 2000 and was halted by March 2001 for the following reasons:

1. The costs of a melt-spun acrylic fiber were estimated using other melt-spinning systems as models. The results showed that the expected lowest cost, for state-of-the-art large-scale manufacture, could not fall below $0.50–0.55/lb, with the actual selling price probably needing to be much higher (current polyester filament prices give some guidance in this regard, at $ 0.65 to 0.70/lb). Set against significant uncertainties related to technology implementation, and capital risk, we see only a small economic justification for pursuing melt-acrylic technology, possibly $0.20/lb as carbon fiber. The only reason for prioritizing this work would be a compelling technical advantage over textile acrylic fibers. These are available at ~$0.75/lb, and the range of acrylic polymer variants that might be made available at this price is limited only by the scale of the market and by customer/supplier interests.
2. The problem of extruding a melt filament is not the primary technical challenge. In the first and second quarters of 2001, it became apparent that an intractable problem with melt-acrylics was stabilization without fusion in a cost-effective time frame. Radiation treatment was found to be helpful in inhibiting the degree of fusion experienced and to permit thermal oxidation to be applied, but the required time frames were simply too long for carbon fiber operations to be practical.

Based on these findings, work on this subtask was halted. In subsequent work during 2001, it has become even more apparent that textile acrylic fibers have more LCCF potential than had been previously realized.

Radiation Pretreatment

Radiation pretreatment of PAN precursors to reduce carbon fiber manufacturing costs by decreasing stabilization times was technically demonstrated and has been an option since the late 1970s. Recent advances in radiation treatment technology and reduced processing costs have rekindled interest in this approach. For LCCF development, this route is especially attractive if it could be applied to low-cost textile PAN fiber with the same effect as those observed for PAN precursors. As noted under “Commodity Textile Acrylics,” commodity textile acrylic fibers can be purchased for approximately half the cost of large-tow precursors currently available in the market.

A significant technical problem associated with the conversion of textile fibers to carbon fiber is the long stabilization time necessary before carbonization. The low reactivity of textile PAN fibers relative to PAN precursors demands high temperatures to initiate oxidation (compared with those required for precursors). Since the stabilization process is accompanied by a high residual heat of reaction, the use of high temperatures leads to an uncontrollable exothermic process, resulting in fiber fusion and/or burning. Preliminary lab-scale evaluation of textile fiber samples irradiated with either gamma or E-beam sources indicated a reduction in heat evolution and the initiation of oxidation at lower temperatures. Analysis of the two radiation treatments, from both cost and operability points of view, favored the E-beam radiation treatment as the process of choice. The treatment dose level was optimized to obtain maximum benefit in terms of reducing both heat evolution and treatment cost. Figure 2 shows cost model calculations derived from optimization trials. The optimum dose level for textile acrylic fibers was defined as 30 Mrad.

Commercial textile PAN fiber is crimped to maintain the tow integrity of the >300K filaments and is packaged in bales. Radiation facilities in general are not equipped to handle large tows or the packaging formats of standard textile PAN shipments. For these reasons, textile fiber was spun on Hexcel’s PAN pilot line using commercially available textile polymer (~90–92% acrylonitrile + 8–10% vinyl acetate) to demonstrate the technical feasibility of radiation treatment on textile PAN tows. A 12-K tow spool was radiated in a continuous line operation with E-beam equipment at an off-site

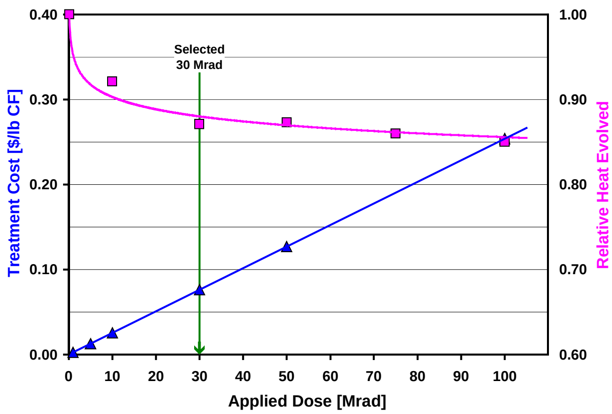

Figure 2. Effect of applied E-beam radiation dose on treatment cost and relative heat evolution of commodity textile tow PAN.

facility. The line speed was set to treat the fibers at 30 Mrad. Radiated fibers and as-spun virgin fibers were then subjected to batch oxidation/stabilization studies. The results from these studies (Figure 3) showed that a total oxidation time of at least 75 minutes was necessary to stabilize the pre-irradiated fibers (to an oxidized fiber density of 1.324 g/cm^3) to enable the fibers to withstand typical carbonization temperatures. By comparison, the oxidized fiber density of control fibers reached only 1.329 g/cm^3 after 135 minutes.

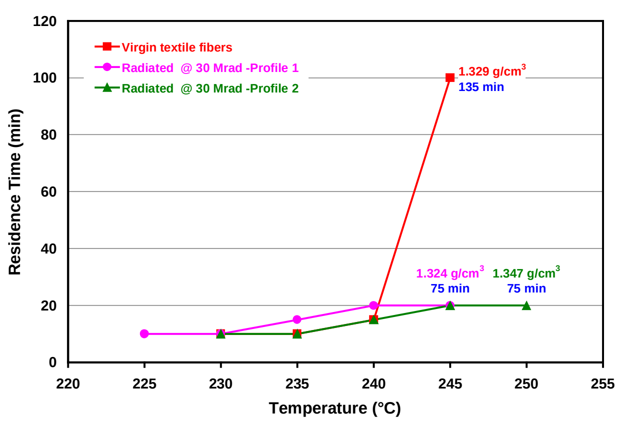

Figure 3. Effect of air stabilization profile on ox-density of textile PAN fibers with and without E-beam radiation pretreatments.

These results clearly showed that, in these trials, E-beam irradiation led to a 45 % reduction in the overall stabilization time of textile PAN fibers. The temperature/ time profile in oxidation was also optimized, without increasing the total heating time, in order to obtain a higher carbon yield (profile 2 in Figure 3). The radiated fiber was oxidized and carbonized on the research line in a continuous process. The control fiber could not be carbonized continuously because of its poor strength. The properties of the carbon fiber obtained are shown in Table 4.

Table 4. Carbon fibers properties after E-beam pretreatment of textile polyacrylonitrile fiber @ 30 Mrad

Tensile strength (Ksi)	Tensile modulus (Msi)	Strain at failure (%)	Density (g/cm^3)	MPUL (g/m)
256	22.7	1.10	1.71	0.776

These trials demonstrated the concept of using radiation as a pretreatment for reducing carbon fiber conversion costs through a reduction in overall stabilization time. In this first attempt to carbonize the sample, the carbon fiber modulus obtained almost met the LCCF program's target of 25 Msi. While the tensile strength is still below the LCCF target of 400 ksi, the reported strength is very encouraging for fibers derived from a textile PAN polymer. In fact, many options are available to improve the mechanical properties of these textile PAN fibers. Higher strengths can be expected when an optimum degree of stretch is applied in the oxidation and tar removal stages of the process. In addition, the property envelope could be pushed further if a commercial source of PAN polymer with compositions of less than 8% vinyl acetate and more than 92% acrylonitrile could be identified. This source should be able to provide the material at a cost similar cost to that of the textile PAN polymer used for the trials.

Based on the experimental results obtained, economic analyses performed to date, and practical options available for improvement in carbon fiber properties, textile PAN radiation pretreatment is the most promising route to achieve the LCCF development goals. As a result, work has been planned for Year 3 of the program as follows:

1. Carbonize radiated Hexcel-spun 48K textile acrylic fiber.
2. Explore the effects of small changes in vinyl acetate content on carbon fiber properties.
3. Design hardware (with vendors) to be able to radiate 83K and >300K tows uniformly.
4. Carbonize various tow sizes to model the effect of tow mass on various stages of the conversion process.
5. Optimize the oxidation/carbonization process to maximize mass throughput and mechanical properties and minimize carbon fiber cost.
6. Identify process parameters needed for scaling up the technology to handle standard textile tows (>300K).

Nitrogen Prestabilization Treatment

Work within the LCCF program confirmed earlier observations that N_2 prestabilization can help to reduce the overall processing time of precursor and textile-grade PAN. Heating PAN in N_2 promotes cyclization reactions without parallel oxidation reactions. This greatly reduces the reaction onset temperature and the amount of exothermic heat released upon oxidation. Hence, N_2-prestabilized PAN can be processed more quickly and safely through conventional oxidation ovens. Bench-scale LCCF work showed that N_2 prestabilization treatments were most effective when they were carried out (a) at relatively high temperatures and (b) for short times. For instance, large tows of textile PAN can ignite if oxidized directly at temperatures as low as 240°C. In contrast, subjecting textile PAN to a single N_2 prestabilization step at 280°C for 5–30 minutes showed the potential to cut down its overall stabilization time by as much as 30%. The elimination of any possible filament fusion by employing a two-stage N_2 prestabilization treatment (including a low-temperature N_2 pretreatment step) was also investigated. However, preliminary LCCF manufacturing cost models indicated that other LCCF technologies are more promising than N_2 prestabilization in terms of overall cost reduction. For that reason, the decision was made to halt work on this subtask and to focus on the most promising LCCF technologies.

Non-Polyacrylonitrile

Polyolefins

Polyolefin fibers are appealing as LCCF precursors because of their commercial availability, high carbon content, and relatively low cost. Recycled polyolefin resins are also available at low cost, and their use would reduce the impact of industrial polyolefins on the environment. However, commercial polyolefin fibers melt before they can be thermally stabilized. Their melting can be suppressed through stabilization treatments that promote chain cross-linking while minimizing fusion or destructive chain scission. Such treatments, when effective, tend to involve the use of hazardous and/or costly materials. A few open literature reports show that some of those treatments have been employed in the past to successfully convert polyethylene (PE), but not PP to carbon fibers. In those few instances, short lengths of non-commercial, expensive PE fibers were subjected to a variety of batch stabilization treatments. In one experiment, batch-stabilized HDPE fibers were subsequently converted to carbon fibers with mechanical properties that approach LCCF program targets (tensile strength of up to 369 ksi, modulus of up to 22 Msi).[4]

In contrast to the processes discussed in literature reports, our approach involved taking commercially available polyolefin tows and developing an industrially scalable method for their conversion to low-cost carbon fibers. Accordingly, polyolefin tows with 1000 to 3000 filaments and 2 to 3 denier per filament were obtained from various commercial suppliers. Portions of the tows were subjected to sulfonation, radiation, or combined treatments with or without cross-linking additives. Preliminary results and manufacturing cost model calculations indicated that polyolefin fiber treatments in hot, concentrated sulfuric acid could provide adequate stabilization within LCCF program targets. A multi-stage sulfonation reactor was built to allow the continuous feeding, prewetting, sulfonation, washing, and winding of polyolefin tows. Line speeds were set to allow the direct carbonization of sulfonated and washed polyolefin tows if desired. LLDPE was selected over HDPE based on its lower cost, easier spinnability, and more open structure. Initial trials allowed the determination of the experimental conditions at which either LLDPE or PP tows could withstand continuous immersion in hot sulfuric acid without breaking. These trials confirmed that both PE and PP tows can be successfully stabilized in a continuous process.[5] However, tests with the more economical PP indicated that the conditions needed to fully stabilize it led to poor tow processability and quality. The trials carried out to date also confirmed the importance of careful control of temperature stability, line tension, acid concentration, and drying conditions.

Polystyrene

Like polyolefins, polystyrene resins are also appealing as LCCF precursor materials because of their commercial availability, high carbon content, and relatively low cost. But unlike polyolefins, polystyrene is not available commercially in the form of fiber tows. After contacting a series of polystyrene resin suppliers and commercial pilot-line melt spinners, it was possible to produce multi-filament tows of polystyrene fibers and to acquire hands-on experience with their manufacture. Owing to their molecular characteristics, polystyrene fibers can be melt-spun; but the resulting fibers are so brittle that they cannot be drawn at reasonable line speeds. Higher spinning temperatures improved their spinnability but enhanced their degradation rate. Literature reports provide potential pathways to improve the manufacture and properties of melt-spun polystyrene fibers (e.g., through multi-stage draw processes). Some evidence also indicated that polystyrene fibers can be cross-linked much more quickly and effectively than polyolefin fibers. In fact, patent literature reports indicate that large-diameter carbon fibers with low mechanical properties (tensile strengths of up to 71 ksi and modulus of up to 9 Msi) have been obtained by carbonization of polystyrene fibers treated with warm sulfuric acid for a few minutes.

Based on these observations, and on kinetic modeling parameters derived from successful bench-scale stabilization and carbonization trials, a detailed LCCF manufacturing cost model was developed. The conclusion from the cost model calculations was that polystyrene fibers, if made available, could not offer any significant cost advantages as LCCF precursors compared with PAN fibers. In view of the technical difficulties and unappealing cost model estimates, work on polystyrene was halted to focus on the most promising LCCF technologies.

Polyvinyl Chloride Pitch

The strategy of producing carbon fiber from PVC pitch was abandoned as a sub-task in early 2001. Several factors contributed to this decision. The negative factors that were known prior to experimental work included a very low theoretical carbon yield (resulting from the presence of chlorine in the polymer backbone); the necessity to discard or recycle hydrogen chloride), a relatively low-value pollutant and byproduct of PVC pitch pyrolysis; and the rather poor carbon fiber made via this route as reported in the literature. Based on optimistic assumptions about the ability to develop a process that achieves the LCCF program target properties at a reasonable stabilization time, this sub-task remained a marginal candidate on cost potential through the year 2000. This was primarily because PVC is the cheapest commercial polymer available, and the conversion of pitch fiber to carbon fiber is reasonably well understood.

Developing a process for extruding pitch fiber with good conversion potential brought additional technical problems, some of which were eventually solved. However, little evidence was developed that suggested a pitch could be made via this route that would be superior to commercially available pitches as a carbon fiber precursor. This conclusion was due to the rather narrow window of PVC-to-pitch processing conditions available to make a pitch with a reasonable softening point.

It was shown, however, that extrudable pitches could be made by heat-treating a standard grade of PVC powder in the range of 25 to 45 minutes of reaction time at 400°C. Extrusion experiments with these samples and a control, a commercial coal tar-based pitch, were carried out with the aid of a capillary rheometer. Although the rheometer is not an ideal tool for melt-spinning and fiber collection, the experiments allowed some qualitative judgments to be made. It was not possible to wind fiber from the rheometer extrude from PVC-pitch samples that possessed a reasonable softening point, and the relatively low softening point that resulted from the longest stabilization time would indicate great difficulty in stabilization. The only recourse to carry this work further would have been to attempt a scale-up of the PVC-pitch pyrolysis to generate sufficient quantities for a true melt-spinning study. Given the lack of encouragement from the bench-scale work, this did not seem to be worth the effort and expense, relative to other, more promising sub-tasks.

Cost Modeling of LCCF Technologies

Manufacturing cost modeling has been an essential component of all the LCCF technologies investigated. Based on an earlier model by Cohn and Das,[6] derived from conventional PAN-based carbon fiber manufacturing practices, costs of potential LCCF technologies were initially divided into four main categories: precursor, stabilization, carbonization, and other.[2, 3, 6] The most significant cost contributor to the conventional carbon fiber manufacturing process is the precursor.[3] This is followed by the precursor stabilization step and to a lesser extent by the carbonization step. Hence, initial LCCF efforts were geared toward uncovering less expensive precursors and stabilization methods. The cost model incorporates fixed capital, maintenance, taxes, insurance, labor, and utility costs, as well as variables such as sales and administrative/research and development (SA/RD) costs and return on investment (ROI). Technical factors such as carbon fiber yield, line speed, and productivity were also incorporated into the model. In each case, capital investment costs were estimated for a production volume of 4 million lb/year of carbon fiber in two parallel manufacturing lines. This resulted in estimated capital expenses on the order of $36 million for the baseline case using a commercial large-tow PAN precursor, while required capital expenses for the candidate LCCF technologies ranged from $30 to $40 million. The projected carbon fiber manufacturing costs for the LCCF technologies deemed most promising at the end of Year 2 of the LCCF program are shown in Figure 4.

Figure 4 shows that the $5.00/lb upper LCCF program cost target cannot be met in a sustainable manner (that is, without allowing for overhead or profit margins) based on the use of commercially available large-tow PAN precursor. In fact, the present cost model suggests that to meet the ~$6.00 to $6.50/lb sales price currently claimed by some large-tow carbon fiber manufacturers, the ROI margin could only be within ~5.5 to 11.1%. The predicted costs of the other four technologies, including 10% SA/RD but excluding ROI, are seen

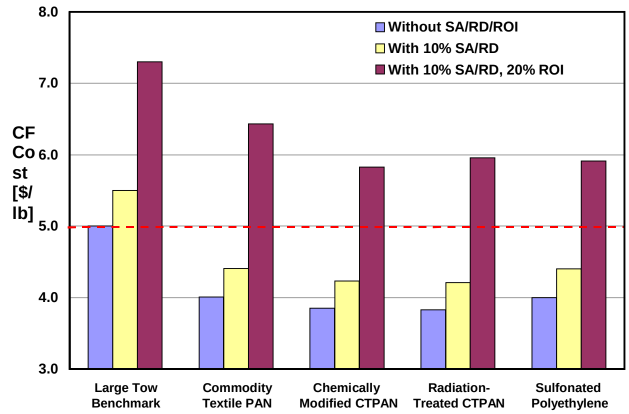

Figure 4. Predicted carbon fiber manufacturing costs for selected technologies.

to fall well below the $5.00/lb upper LCCF program boundary. If 20% ROI is included for these LCCF technologies, the minimum attainable carbon fiber costs would be on the order of ~$5.60 to 5.90/lb. A distribution of costs into their various components (Figure 5) shows that the savings relative to the large-tow benchmark (baseline) technology would mostly be attained by using less expensive materials as precursors, and to a lesser extent by reducing capital costs.

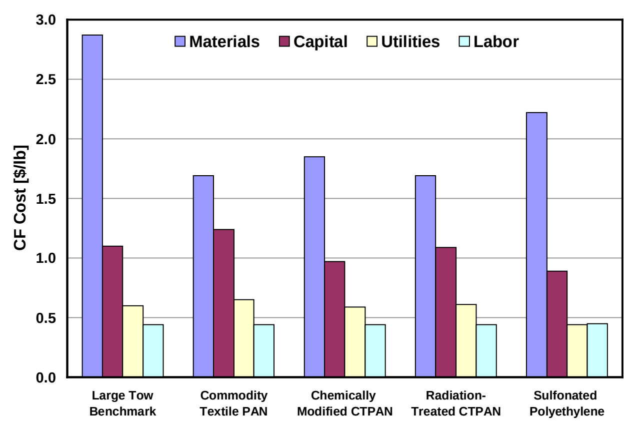

Figure 5. Distribution of carbon fiber manufacturing costs for selected technologies.

Conclusions

The results of the laboratory trials of proposed technologies) were as follows.

1. We halted further work on acrylic fibers spun without solvents, plasticized PAN,

PVC, and polystyrene because of technical, environmental and cost issues (Table 5).

2. We evaluated and selected the following most promising LCCF technologies for Phase 2: commodity textile PAN-based precursors (as-received and with pretreatment using chemical modification, radiation, and nitrogen prestabilization technologies) and polyolefin precursors (LLDPE and PP).
3. We used a large-tow PAN precursor technology benchmark as a metric to evaluate the proposed technologies in terms of their potential to meet the LCCF program's cost targets. The difference between commodity textile PAN and large-tow precursor, based on carbon fiber cost, is approximately \$1.80 vs \$3.10/lb.

Summary

These are highlights of the progress during FY 2001.

1. Processed and evaluated various state-of-the-art PAN and developmental PAN precursors that meet the targeted properties for the LCCF program using a single-tow research line and a multiple-tow pilot line.
2. Developed the continuous in-line chemical modification of PAN precursor on research-line and pilot-line scales and demonstrated a reduction in stabilization times of ~50% while producing carbon fiber properties that meet the LCCF program requirements using standard Hexcel's precursor PAN.
3. Evaluated and selected E-beam radiation technology for the prestabilization of commodity textile acrylic tow to achieve the cost and mechanical properties targeted by the LCCF program. This was demonstrated by working with acrylic textile producers and E-beam and gamma radiation equipment manufacturers and by acquiring and processing acrylic polymer materials.
4. Developed a continuous two-stage sulfonation process for the stabilization of LLDPE and PP fibers. This was demonstrated by acquiring polyolefin fibers and producing sulfonated samples at various processing conditions and carbon fiber conversion levels.
5. Developed cost models for the selected technologies: large-tow benchmark, commodity textile acrylic fibers with chemical modification and radiation pretreatments, and LLDPE sulfonation. The cost models incorporate fixed capital, maintenance, taxes, insurance, labor, and utility costs, as well as variables such as S/RD and ROI costs. Technical factors such as carbon fiber yield, line speed, and productivity were also incorporated into the model. For comparison purposes, capital

Table 5. Technologies eliminated and the reasons for their elimination

Eliminated technologies	Reasons for elimination		**Comments**
	Technical	Cost	
Acrylic fibers without solvents	X	X	Fusion Insufficient cost incentive to solve technical problems
Plasticized PAN	X	X	Capital-intensive Needs non-aqueous solvent
Polyvinyl chloride	X	X	Low yield Cost of environmental compliance
Polystyrene	X	X	Spinning issues Environmental issues No cost advantages

6. investment costs were estimated for a production volume of 4 million lb/year of carbon fiber in two parallel manufacturing lines. This resulted in estimated capital expenses on the order of $36 million when using a commercial large-tow PAN precursor (baseline) and $30 to $40 million for other LCCF technologies. The predicted costs of the other four technologies, including 10% SA/RD but excluding ROI, were found to fall well below the $5.00/lb upper LCCF program boundary. If 20% ROI is included for these LCCF technologies, the minimum attainable carbon fiber costs would be on the order of ~$5.60 to 5.90/lb. It was also concluded that the lowest cost for polymeric precursor fibers cannot be less than $0.50/lb. Submitted two records of invention: "Polyolefin Sulfonation and Carbonization" and "Chemical Modification of PAN" and a public disclosure on "Chemical Modification"
8. Produced three papers [two published during Society for the Advancement of Materials and Process Engineering (SAMPE) 2000, and one accepted for SAMPE 2001].

Commodity textile acrylic fiber costs are ~$0.70 to $0.90/lb. At current carbon fiber consumption levels, one acrylic facility could supply the world demand for carbon fiber precursor. Also, very large carbon fiber lines are not decisive in achieving low carbon fiber costs. Individual units at 2 million lb/year can show favorable economicsA significant cost benefit would be realized by moving from large-tow precursor to commodity acrylic, with savings of about $1.30/lb of carbon fiber. However, manufacturing a carbon fiber with a tensile strength of 400 ksi from commodity acrylic is challenging.

Enhanced stabilization rates using radiation and/or chemical modification should lower carbon fiber costs to ~$4/lb (without ROI, SA/RD). The best results will be achieved by working closely with acrylic fiber manufacturers to improve the carbon fiber conversion potential of currently available material.

Developmental "precursors" have met the LCCF program's targets, but only within a very narrow cost window. Also, high-volume melt-processed polyolefins present a competitive alternative to PAN. Polyolefins have been shown to approach the LCCF program's targets, but a continuous large-scale process must be developed.

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C. Low-Cost Carbon Fiber for Automotive Composite Materials

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Contract No.: 4500011036

Objective

- Develop a cost-effective route to producing low-cost carbon fiber (<$5/lb) with a tensile strength of greater than 2.7 GPa, a modulus of >170 GPa, and a strain-to-failure rate of >1%. This range of carbon fiber properties has been deemed necessary for generating composite materials suitable for automotive applications.

OAAT R&D Plan: Task 5; Barriers A, B

Approach

- Develop melt-spinnable polyacrylonitrile (PAN) copolymers by controlling the copolymer ratio, molecular weight, and end-groups.
- Identify a benign solvent or plasticizer for PAN copolymers, allowing precursor fibers to be generated by melt-spinning techniques.
- Identify an alternate melt-spinnable precursor system containing a higher carbon content.
- Develop a thermal treatment for oxidative stabilization and carbonization of fibers made from different precursors to produce low-cost carbon fibers with desired mechanical properties.
- Develop a new stabilization process based on ultraviolet (UV) irradiation to cross-link the fibers.

Accomplishments

- Successfully prepared melt-processable acrylonitrile (AN) copolymers by copolymerizing methylacrylate (MA) with AN to produce a series of copolymers of controlled molecular weight but of variable MA content.
- Found that compositions in the range of 88/12 to 90/10 AN/MA with small amounts of chemical stabilizers (e.g., 1 wt% boric acid) exhibited sufficient stability for spinning on a laboratory system.
- Treated fibers with a UV-sensitive coating, which allowed the surface to be stabilized by UV radiation. (The fibers could not be stabilized by standard thermal oxidation treatment.) The fibers could then be subsequently stabilized (i.e., cross-linked and cyclized) in an oxidative step.
- Successfully carbonized the stabilized fibers.
- Successfully saturated copolymers of AN and MA, with levels of AN as high as 90 mole%, with CO_2 and extruded them at temperatures low enough to prevent significant cross-linking. Carbon dioxide is an environmentally benign processing aid that will diffuse out and leave the fiber in a state that will allow it to be stabilized by thermal oxidation.
- Discovered and developed an environmentally attractive, economical, and solvent-free polymer system that has the potential to serve as a carbon fiber precursor.

Future Direction

- Fine-tune the AN/MA melt-spinnable copolymer composition so that it is sufficiently stable for spinning in larger-size equipment, but yet has the potential to be stabilized using a reasonable oxidation schedule. This will be accomplished by selecting an appropriate AN/MA ratio (between 85 and 90 mole% AN), the amount of stabilizer (i.e., boric acid), and the polymerization mechanism.
- Develop fundamental information on the copolymers concerning rheological properties as a function of temperature, time, and shear rate needed to design a large-scale spinning system.
- Scale up the reactor for producing the AN/MA copolymer precursor for pilot-scale melt spinning.
- Fine-tune the UV stabilization process and scale it up for handling multiple-filament bundles.
- Design a spinning pack to handle marginally stable precursor copolymers.
- Reduce the cost of the carbon fibers further by synthesizing AN/MA/epoxy terepolymers that can be photochemically stabilized in minutes rather than hours.

- Continue to develop alternate methods for reducing oxidative stabilization time by introducing sites in the AN/MA copolymers that provide sites for cross-linking at lower temperatures.
- Continue to search for a lower-cost, higher-carbon-content precursor that can be photochemically rather than oxidatively stabilized.

Introduction

To use plastics in the manufacture of automobiles to reduce weight and hence improve energy consumption, it is necessary to reinforce them with glass or carbon fibers. Glass is inexpensive (less $1.00 per pound), but its reinforcing capabilities and density make it less desirable than carbon fiber. At present, carbon fiber suitable for use in automotive composites costs more than $7.00 per pound, a cost prohibitive for general use. It has been determined that in order to economically use carbon fiber in the manufacture of automobiles, the price must be less than $5.00 per pound. Approximately half of the present cost for carbon fiber is associated with the production of the carbon fiber precursor. For automotive composites, the precursors at present are generated from PAN copolymers that are solution-spun using highly toxic solvents. Considerable savings could be recognized if these copolymers could be melt-spun without the use of the highly toxic solvents. Furthermore, as the PAN copolymers contain only about 50 wt% carbon, new precursors with higher carbon levels would reduce the cost even more.

Project Deliverables

At the end of this multi-year program, a melt-spinnable polymer system will be developed and scaled up that is suitable for producing carbon fiber precursors that can be stabilized more rapidly by means of radiation. It will lead to a significant reduction in the cost of producing carbon fiber.

Planned Approach

To meet the target of generating carbon fiber costing less than $5 per pound, the present approach using PAN copolymers must be significantly modified. These modifications include the development of melt-spinnable PAN copolymers that can be stabilized and carbonized efficiently and economically, and the identification of a precursor system that contains higher carbon levels.

Melt-Spinnable PAN Copolymers

Melt-processable AN copolymers were successfully prepared by copolymerizing MA with AN to produce a series of copolymers of controlled molecular weight but of variable MA content. In a significant departure from past procedures, copolymers were prepared with an azo-initiator, which produced non-associative end groups and thereby led to a significant reduction in melt viscosity. These materials were successfully extruded on a laboratory scale (see Figure 1) to

Figure 1. Micro-spinline for producing carbon fiber precursors from small quantities of polymer (<50 g).

produce carbon fiber precursors. Efforts must be continued to obtain the highest AN-to-MA ratio possible while keeping the molecular weight at the highest level possible in order to provide adequate melt strength for melt-spinning. The approach we are taking requires that a delicate balance be maintained between a copolymer that is sufficiently stable to be melt-spun but eventually will become unstable, so that cross-linking and cyclization can take place in a subsequent step. Therefore, we are investigating other avenues for stabilizing the fiber precursor besides direct thermal oxidative methods. One approach employs a UV-sensitive coating applied to the fibers which, when subjected to UV radiation, will initiate the cross-linking of the copolymer at the surface. The fiber can then be subjected to heating to promote thermal oxidative stabilization of the remainder of the fiber (see Figure 2). It is desirable to make the whole fiber

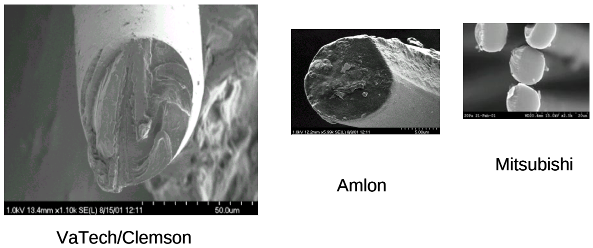

Figure 2. Scanning electron microscope micro-graphs of UV–oxidatively stabilized fibers (UV 2.5 h–150°C, 180–220°C).

UV-sensitive by synthesizing an AN/MA/epoxy terepolymer. Significant cost reductions can result from eliminating or reducing the time required for oxidative stabilization. At present, we have shown that fibers stabilized in this fashion can be converted to carbon fiber (Figure 3). This is the slowest step in

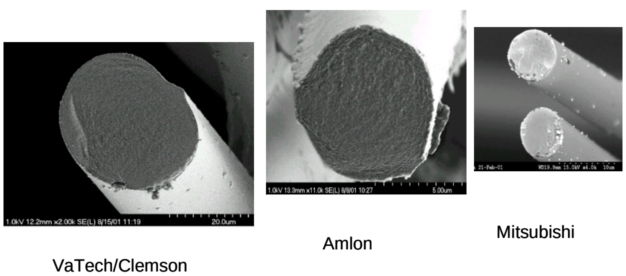

Figure 3. Scanning electron microscope micro-graphs of carbon fibers produced by UV–oxidative stabilization (2.5 h–150°C, 180–220°C) followed by carbonization (1500°C–30 min).

the carbon fiber manufacturing process. Another approach, which is available as a backup in case we cannot produce a system that is sufficiently thermally stable, is the use of CO_2 as a plasticizer for PAN copolymers. Carbon dioxide is a benign solvent that is obtained from air and can be released back to air. PAN copolymers that are currently solution-spun may be melt-spun in the presence of small amounts of CO_2 (~5 wt%). Although we will not actively pursue this approach in the coming year, it could be an important technique for handling the UV-sensitive terepolymers.

<u>Higher-Carbon-Level Precursors</u>

Another factor that must be considered in reducing the cost of carbon fiber is the use of a precursor material that contains higher carbon content. We have tested an entirely different polymer, syndiotactic polybutadiene, as an alternate precursor because it has a theoretically higher yield of carbon. However, thus far it has been found to be inferior to PAN-based precursors in high-temperature processing steps involved in the preparation. Future efforts will be concerned with adding a photosensitive group that would allow the polymer to be cross-linked by means of UV radiation.

D. Economical Carbon Fiber and Tape Development from Anthracite Coal Powder and Development of Polymer Composites Filled with Exfoliated Graphitic Nanostructures

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Contractor: Cornerstone Technologies, LLC
Contract No.: 4500011695

Objectives

- Develop low-cost fiber spinning and tape casting/extrusion processes from low-cost, carbon-based raw materials.
- Produce high-stiffness, high-strength carbon-fiber-like particles for automotive composite structural applications.

OAAT R&D Plan: Task 5; Barriers A, B

Accomplishments

- Produced a unique new structural/conductive graphite platelet material, exfoliated graphite nanostructures (EGN), with the promise of breakthrough performance as an injection-molded graphite-polymer composite component.
- Conducted trials of EGN-filled thermoplastic resin with successful injection molding at virgin-resin conditions.
- Evaluated compounding and resin processing technologies that generate unique EGN-filled polymeric composites.
- Identified additives for EGN-polymer formulations to enhance its compatibility and to improve the performance of EGN-polymeric composite components.

Future Direction

- The project concludes at the end of FY 2001.

Introduction

The use of polymeric composites in materials-based industries has almost quadrupled during the last two to three decades, especially in automotive applications. The benefits of polymerics include lower cost; weight reduction; styling potential; functional design; and new effects, including superior acoustic characteristics, reduced maintenance, and corrosion resistance. In most applications, however, the resin system must be reinforced by fibers and/or particulates to yield the required stiffness and strength. The resins are mixed with various additives for processing and durability requirements and sometimes with fillers for further cost reduction. These modifications cause major reductions in toughness and processability and difficulties in controlling surface properties of the final product.

Expanded graphite is used along with carbon black, various types of carbon fiber, metal fibers, and ceramic nitrides as fillers to improve the electrical and thermal conductivity of normally non-conducting or poorly conducting polymeric materials. Generally, these are used in combination with other fillers such as glass, talcs, aramid fibers, mica, and the like to obtain additional property enhancements for overall heat transfer solutions in polymer applications.

Exfoliated graphite nanostructure (EGN) is a graphitic layer in platelet form, consisting of multi-aromatic carbon ring nanostructures (see Figure 1). EGNs are identical to the graphite ribbon component of carbon fiber and are capable of performing in thermoplastics as a low-cost carbon reinforcement. Current EGN platelets have high aspect ratios of 100:1 to 1000:1. In the fourth quarter of 2001, EGN particles haves been reduced in to 5 µm in width by 20 nm in thickness. EGN micro-particles should act as agents to inhibit micro-crack propagation, the dominant damage mechanism producing composite and polymer failures.

EGN-filled polymer resins can be conventionally compounded using twin-screw extrusion techniques. Compounding trials demonstrate excellent compounding characteristics at substantially reduced molding pressures and shearing forces. The resulting extrudate can be compression-molded, injection-molded, extruded, cast, blow-molded, vacuum-formed, poltruded, and formed with other processes common to the plastics, pre-impregnated textile, and polymeric composites industries. The primary advantage of a low-cost, high-performance powder component for polymer composites such as EGN is to drive polymer performance in automotive applications closer and closer to that of metals such as aluminum and high-cost engineered fiber reinforcements currently used for body, under-the-hood, and interior components.

Anticipated Property Benefits of Exfoliated Graphite Nanostructures

EGN has the advantages of

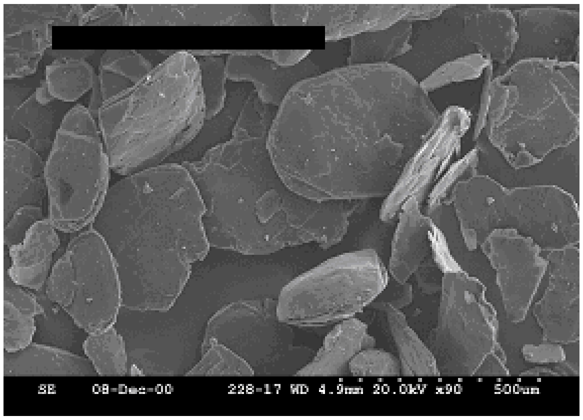
- electrical conductivity
- thermal stability
- heat transfer
- flexural modulus
- tensile modulus
- ductility
- heat distortion temperature
- abrasion resistance
- surface quality

Table 1 lists some common uses of polymeric composites in automobile manufacturing. Table 2 lists some of the disadvantages of those materials in automotive applications.

Core Process Technology

Cornerstone’s process technology works by focusing the kinetic energy of a high-pressure liquid so that concentrated destructive forces are applied to raw material feedstocks. It attacks particles along natural cleave planes and defects and high-energy grain boundaries, and applies hydro-wedging attacks on particle grain boundaries. The process generates extremely intensive turbulence, shearing, high-velocity

Crystalline graphite precursor (non-expanded)
Magnification 90X



Exfoliated graphitic nanostructures
Magnification 3000X
Note: Aspect ratio exceeds 100:1.

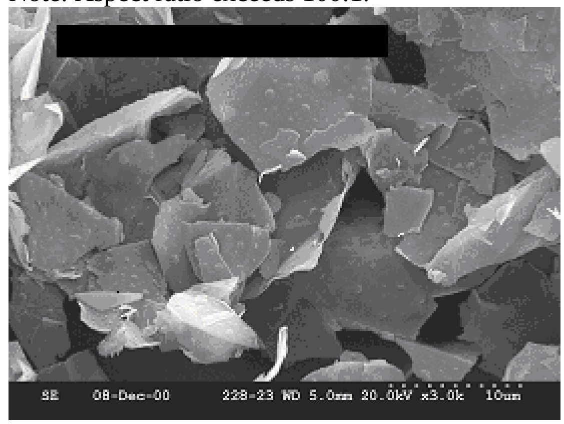


Figure 1. Graphite precursor and EGN platelets.

Table 1. Typical polymers used in automotive applications

Resin system	**Automotive application**
Glass-reinforced polypropylene	Battery trays, brackets
Mineral-filled polypropylene	Heating, ventilating, air-conditioning, instrument panel, structural, under-hood
Thermoplastic olefins	Fascias, sound abatement, moldings, step pads
Unfilled polypropylene	Interior trim, splash shields, wiring conduit
Mineral-filled nylons (6 and 6,6)	Covers, gears, connectors
Acetal	Interior (knobs, levers), structural
Acrylonitrile butadiene styrene (ABS)	Fascias
Urethanes	Instrument panel, interior trim
Polyvinyl chlorides	Interior trim
Polyphenylene sulfide	Gears, connectors, fuel pump components
Polyethlene terephthalate (high-impact)	Structural, exterior

Table 2. Issues with current automotive resins

Resin system	**Issues**
Thermoplastic olefins/polypropylene/olefin	Stiffness, heat stability
Nylons	Impact strength, cost
Polyvinyl chloride, urethanes, acrylonitrile butadiene styrene (ABS), acetal	Cost (ABS, urethanes), recycling (all)
Thermosets	Impact, recycling
Fiber-filled resins	Surface/aesthetics/paintability cost of chopping fibers and blending
All polymer-based systems	Electrostatic paintability

collision/abrasion, and destructive cavitation. Its combined hydraulic forces result in the penetration, pressurization, and ultimate exfoliation of layered structures. It successfully comminutes a diverse range of materials including graphite, coal, silica, wollastonite, zirconia, alumina, ferrochrome, chromium metal, cordierite, and other difficult-to-process materials. High-aspect ratio particles are produced at the sub-micron and nano-scales at relatively low labor and energy costs.

EGN Product and Process Performance

To date, uncoated and unmodified EGN (18-mm D_{50} particle size, 50–100 nm in thickness) has been successfully compounded with nylon 6 and polypropylene copolymer from 5% through 70% by weight. EGN-filled polypropylene and nylon 6 pellets were injection-molded into formed articles using virgin resin conditions.

EGN has been produced in a coated concentrate (dust-free powder) format of 80%–90% EGN with a coating of functionalized polymer or polymeric-compatibilizer. A batch of 1000 lb has been produced, and production tooling is in place for volume output. EGN production capacity is 1500 lb per hour for a single production system.

Experimental Results

Table 3 and Figures 2–4 show the mechanical, electrical, and thermal properties of polypropylene copolymer composites with various levels of EGN filler.

Exfoliated graphite nanostructure advantages in polyolefins

- Stiffness increase of up to 700% in initial study of EGN advantages in polyolefins
- Tensile strength increase of up to 30%
- Impact decrease of only 50% at maximum mechanical property improvements
- Thermal performance surpassing that of nylons
- Electrical properties modified from insulating to dissipative/conductive
- Excellent surface quality at loadings of up to 50–55% in polypropylene copolymer
- Potential unique combinations of mechanical, electrical, and thermal properties
- Processability comparable to that of unfilled (injection molding) at loadings of up to 55%
- Substantial improvement of ductility and tensile with property compatibilizers

Exfoliated graphite nanostructure advantages in nylon 6

- Tensile strength increase of up to 55% Tensile modulus increased by 450%
- Flexural modulus increased by 500%
- Heat deflection temperature raised by 300%
- Impact decrease of only 55% at maximum strength increase
- Thermal performance approaching that of high-performance liquid crystal polymers
- Electrical properties modified from insulating to dissipative/conductive
- Excellent surface quality at up to 50% EGN loading
- Potential unique combinations of mechanical, electrical, and thermal properties
- Processability comparable to that of unfilled (injection molding) at up to 50% loading

<u>Nanotechnology Breakthrough</u>

Cornerstone's core technology focuses high-pressure liquid in a manner that applies concentrated destructive forces on raw material feedstocks, creating hydro-wedging attacks on particle grain boundaries, extremely intensive turbulence, shearing, high-velocity collision/abrasion, and destructive cavitation. These combined hydraulic forces result in the penetration, pressurization, and ultimate exfoliation of layered structures. In the case of

Table 3. Mechanical properties of EGN-filled polypropylene copolymer

Polymer/filler	Yield stress (PSI)	Modulus		Elongation		Izod impact	Heat deflection temperature
		Tensile (PSI)	Flexural (PSI)	Yield (%)	Break (%)	(ft*lb/in.)	(°C)
Fina 7825	3563	47400	115000	5	715	0.76	46
5% EGN	3920	56500	173700	4	592	0.68	48
10% EGN	3896	59100	211000	4	490	0.67	49
20% EGN	3916	63700	281000	6	71	0.69	55
36% EGN	4100	85400	514000	3	13	0.40	65
53% EGN	4516	94600	912000	5	6	0.40	82

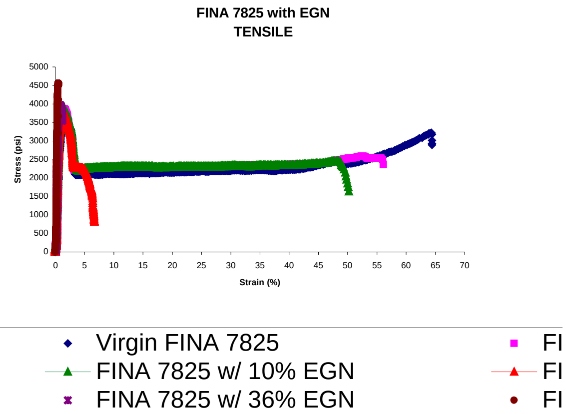


Figure 2. Polypropylene/exfoliated graphite nanostructure stress-strain curve.

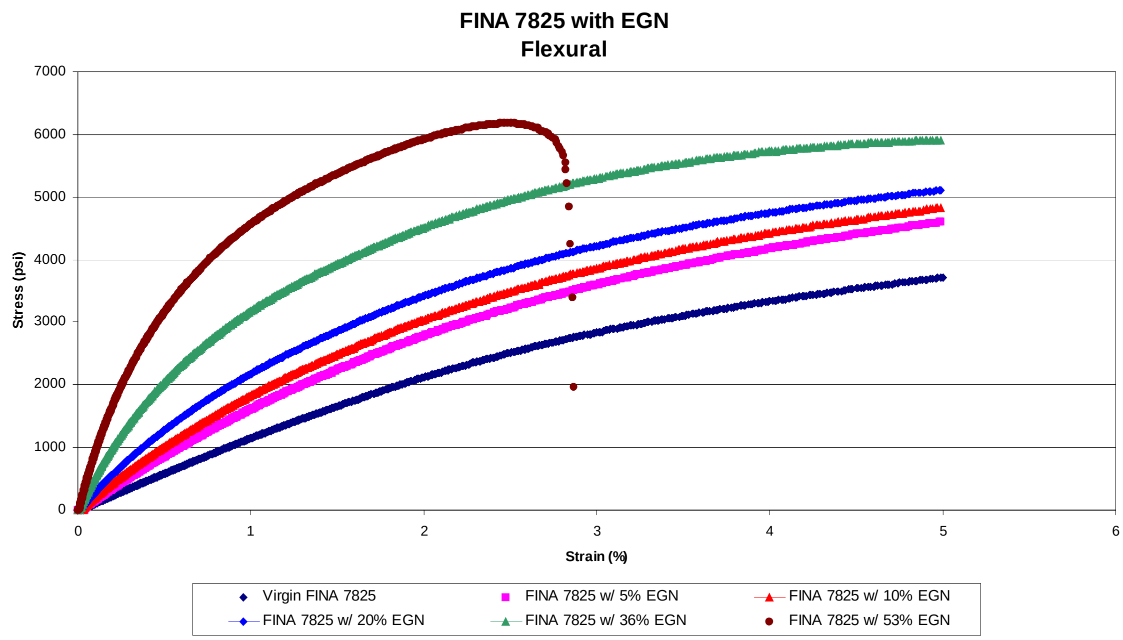


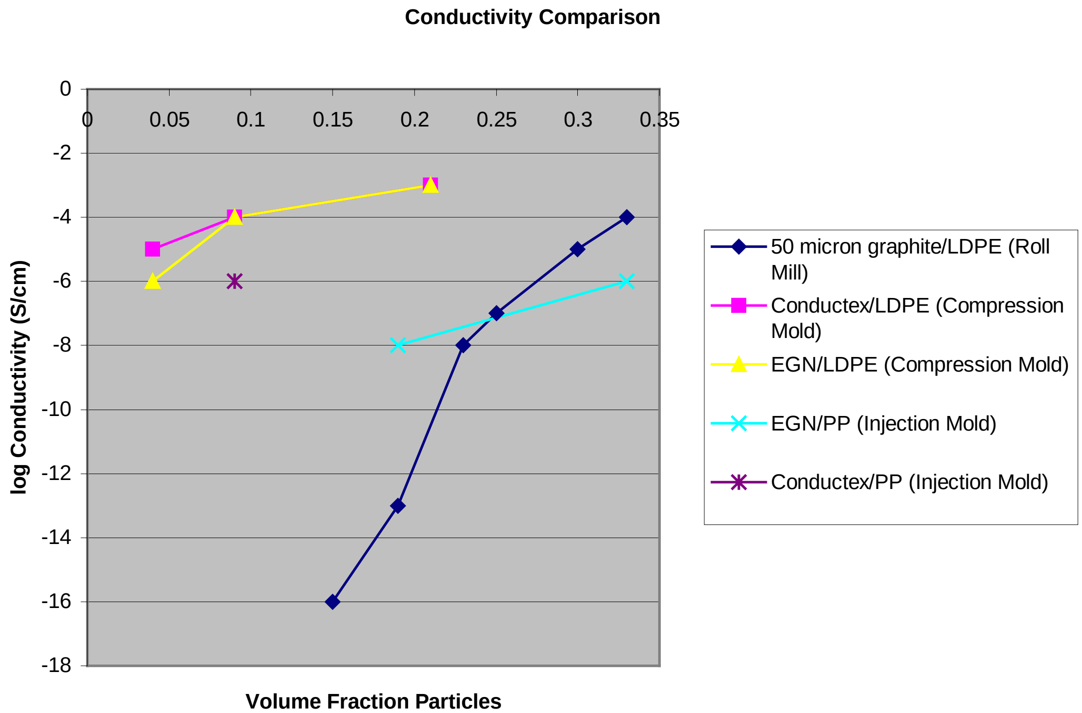
Figure 3. Conductivity in low-density polyethylene, injection-molded, and roll-milled commercial.

graphite, Cornerstone has created a unique, high-aspect ratio morphology, with platelets exhibiting nano-scale thickness.

Conclusions

From extensive preliminary studies and literature searches, it was concluded that dissolving anthracite coal in chemical agents was neither feasible nor cost-competitive. Cornerstone's generation of a new exfoliated graphite particle has permitted a modified approach. The approach is to demonstrate the feasibility of commercializing carbon-reinforced polymer products that are not polyacrylonitrile (PAN) based, use low-energy processing, and reduce costs compared with PAN-based alternatives.

It is anticipated that Cornerstone's technology will help the U.S. carbon and automotive industries develop advanced processes for manufacturing new products at reduced costs that can be passed on to the consumer. In addition, a new process-reactor technology will be introduced



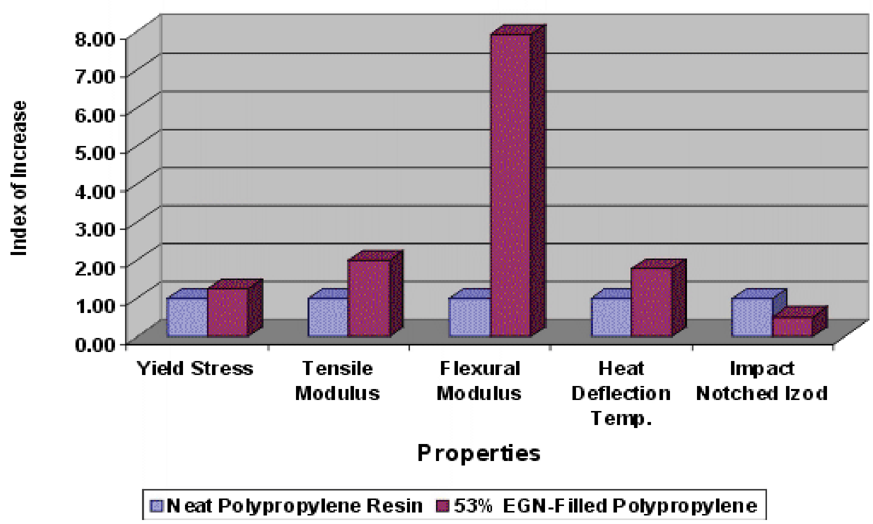


Figure 4. Comparison of mechanical properties of polypropylene resin and polypropylene with EGN filler.

for the efficient production of carbon/graphite products.

The development of enhanced EGN-filled polymeric composites would fulfill a number of automotive and truck project goals and objectives. Low-cost, conductive, engineered plastic composites could enable the commercialization of paintable, abrasion-resistant, high-performance body components; functional under-the-hood parts; and lightweight injection-moldable energy management materials.

E. Microwave-Assisted Manufacturing of Carbon Fibers

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objectives

- Investigate and develop a microwave-assisted technical alternative to carbonize and graphitize polyacrylonitrile- (PAN-) based precursor.
- Prove that carbon fibers with properties that make them suitable for use by the automotive industry can be produced inexpensively using microwave-assisted processing.
- Demonstrate that microwave-assisted processing can produce acceptably uniform properties over the length of the fiber tow.
- Show that, for specified microwave input parameters, fibers with specific properties may be controllably and predictably manufactured using microwave furnaces.
- Demonstrate the economic feasibility of producing fibers of approximately 30-Msi modulus at a significantly lower cost than fibers produced conventionally.

OAAT R&D Plan: Task 5; Barriers A, B

Approach

- Conduct parametric studies with the existing single-tow, pilot continuous carbon fiber processing unit to provide data about unit performance, energy balance, limitations, and thresholds.
- Characterize the properties of carbon fibers produced by microwave-assisted plasma (MAP) processing.

- Use results of the parametric and fiber characterization studies to develop and incorporate modifications necessary to transform the single-tow processing unit from a low-production-speed line to a high-production-speed line.
- Continuously evaluate the underlying technology, hardware, and ideas required to transform this laboratory uni-tow, high-production-speed line pilot unit to a multi-tow, moderate- to high-production-speed line.

Accomplishments

- Completed the construction and basic evaluations of the MAP continuous carbon fiber processing prototype laboratory unit. (This is a single carbon fiber tow processing facility.)
- Demonstrated the processing of PAN-based stabilized precursor to carbon fiber in a continuous mode.
- Acquired, modified, and installed two modular microwave units for use as an independently controlled preheating section. Each unit has a nominal output power of 3 kW. The units are part of a hardware upgrade leading toward a pilot processing unit, with a high-production-speed line, for single-tow carbon fiber.
- Completed development of the required methodologies for morphological (crystallographic) evaluation of the MAP-produced and conventionally produced carbon fibers. For these crystallographic evaluations, wide-angle X-ray diffractometry was used. This permitted a fundamental comparison at the level of atomic crystal unit/configuration of the MAP fiber and conventionally produced carbon fibers.
- Developed methodologies for characterizing elemental chemical composition and functional groups present on the surface of the carbon fiber. This characterization was undertaken with conventionally produced PAN-based carbon fibers. It is performed with X-ray photoelectron spectroscopy and secondary ion mass spectroscopy.
- Continued the effort to put in place the required underlying methodologies and resources for other types of characterizations of the MAP-manufactured continuous carbon fibers on a routine basis.
- Designed, constructed, installed, and evaluated a unique vacuum eyelet system in the processing unit. These new devices will guarantee proper vacuum during processing and reduce frictional forces in the fiber tow, thereby reducing the amount of filament breakage.
- Filed a new patent application for a spin-off technology from this carbon fiber manufacturing program: No. IR-0841: "Microwave and Plasma-Assisted Modification of Composite Fiber Surface Topography."

Future Direction

- Study process parameters, limitations, and threshold in this continuous pilot facility and incorporate modifications.
- Incorporate required new modifications and advancements into this continuous unit with the intent to achieve higher production-line speed and multiple-tow capability.
- Complete the required characterization for the MAP-processed carbon fiber and compare it with conventionally processed, low-cost, PAN-based carbon fibers.
- Initiate preliminary work in demonstrating related technologies in the area of carbon fiber manufacturing (e.g., surface treatment) as resources are available.

Introduction and Progress

In the earlier stages of this program, microwave-assisted processing of PAN precursor for the manufacturing of carbon fibers was proved to be a feasible technical alternative to conventional oven processing. Proof of this concept had been limited to batch or quasi-batch processing. Test results on processed carbon fibers indicated that this technology produces fibers with high-density values, acceptable electrical resistivities, and standard fiber diameters. The weight loss in the fibers ranged from 45% to 55%, which is equivalent to the values obtained from conventional processes. The mechanical properties were slightly lower than but reasonably close to those of conventionally processed carbon fibers. Required processing times showed good potential for this process to produce a significant cost savings.

With this knowledge, a more advanced continuous processing unit was conceived, developed, and built. Additional technical issues were resolved, such as the need for a suitable vacuum device/system to maintain an adequate processing vacuum during operation in this unit. Fiber-tow-handling devices (e.g., feeding, tensioning, adjustment, take-up) were also developed or bought to suit this project. The core of the unit is the 18-foot-long reactor, which contains the carbon fiber processing discharge tube. Nitrogen gas is leaked into the discharge tube under controlled conditions to maintain the required (vacuum level) operating pressure. A section of this reactor features a rectangular waveguide with periodic openings in two opposite sides to enable monitoring of the temperature of the tube via a two-color infrared system during operation.

MAP carbon fiber was produced at speeds of up to 3 in/min. All these MAP fibers were analyzed for mechanical and electrical properties and for their densities, tow cross-sectional area (calculated), and single filament diameter (calculated). Mechanical evaluations of these runs in the manufacturing of carbon fiber in continuous mode indicated that the product made steady progress toward meeting the PNGV requirements during the first half of the reporting period. More recent evaluations of these manufactured carbon fibers indicate that they now surpass all technical requirements of the PNGV program. (The minimal requirements of the PNGV program are tensile strength of 300 ksi, modulus of 25 Msi, elongation at break of 1%.)

Table 1 shows a comparison between conventionally manufactured carbon fiber and carbon fiber manufactured by the Oak Ridge National Laboratory MAP process. The MAP process was characterized at different line high speeds (7.5, 20.5 and 34.6 in./min). Both conventional and MAP processes were evaluated for 50-K tows. In general, these results indicate that the MAP carbon fibers are comparable to and, in the case of some specific properties, even better than the corresponding conventionally processed, low-cost carbon fibers. Furthermore, the results of morphological analysis (X-ray diffractograms) indicate that these MAP fibers possess a degree of carbon fiber graphitization that is comparable to or higher than that of conventionally processed carbon fibers (see Figure 1). It was further demonstrated that the morphological properties of the fiber produced by the MAP process do not vary significantly as a function of position along the tow length. Figure 1 also indicates the production flexibility in the MAP process to manufacture carbon fiber to the required (average) level of graphitization.

Table 1. Testing data for PAN-precursor-based fibers, 50-K tows, MAP carbon fibers, immediately after processing

Physical testing data						
		MAP			Conventional	
					Panex 33	Fortafil F3 (C)
Production line speed	(in./min)	7.5	20.5	34.6	–	–
Density/pycnometer	(gr/cm^3)	1.83	1.82	1.77	1.81	1.77
Calculated tow area	($\times 10^{-2}$ cm^2)	1.90	2.03	2.04	1.96	2.17
Calculated filament diameter	(µ m)	6.97	7.19	7.20	7.37	7.43
Tow linear electric resistance	($\times 10^{-2}$ Ω/cm)	8.37	9.80	8.90	8.23	7.80
Electrical resistivity	($\times 10^{-3}$ Ω/cm)	1.57	1.89	1.82	1.62	1.67

Mechanical testing data							
		MAP			Conventional		**PNGV Program Goals**
					Panex 33	Fortafil F3 (C)	
Production line speed	(in./min)	7.5	20.5	34.6	–	–	**–**
Modulus	($\times 10^6$ lb/in.^{2})	31.7	30.5	29.0*	26.1	31.1	**25**
Ultimate strength	($\times 10^3$ lb/in.$^{2)}$	424.1	384	341.9*	407.8	485.3	**300**
Elongation at break	(%)	1.3	1.22	1.25*	1.5	1.5	**1.0**

*These fibers were neither surface treated nor sized and tested 4 months after the manufacturing date.

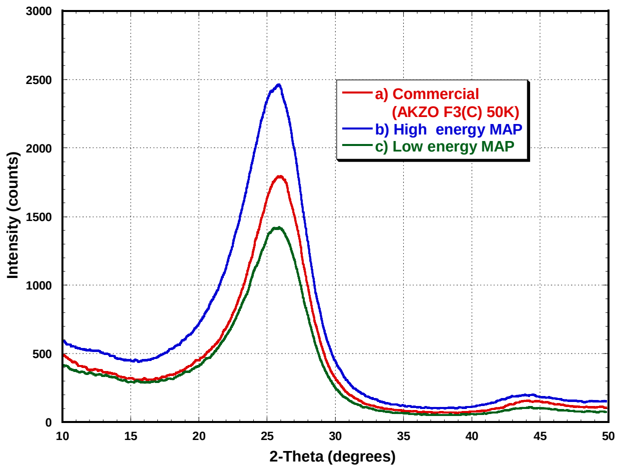

Figure 1. Comparison of the X-ray diffractograms for commercial and MAP carbon fiber produced under different microwave energy processing levels (curves not normalized).

Project Deliverables

At the end of this multi-year program, a protoype high-production-speed line for continuous MAP processing of carbon fiber will have been developed, constructed, and tested. A final report will be issued with the results of tests of the carbon fibers processed with this unit. Appropriate industry briefings will be conducted to facilitate commercialization of this economically enabling technology.

6. RECYCLING

A. Sorting Mixed Alloys from Shredded Automobiles

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Contract No.: DE-FC05-95OR22363

Objective

- Identify, evaluate, and validate methods for separating and sorting aluminum in shredded automotive scrap.

OAAT R&D Plan: Task 9; Barrier E

Approach

- Demonstrate the capability to separate wrought from cast materials.
- Demonstrate existing and developed scrap sorting capability using color and laser- induced breakdown spectroscopy (LIBS).
- Integrate the various elements (scrap preparation, decoating, sorting, recycling) required for commercial recycling of automotive wrought alloy scrap.

Accomplishments

- Optimized Alcoa's proprietary caustic etching alloy-coloring process to provide color tints to the alloy mix in order to facilitate color sorting.
- Conducted melt tests to establish a composition for each color grouping.
- Used the LIBS technology to sort 5xxx alloys from 6xxx alloys.

- Conducted trials evaluate bulk cleaning procedures for the alloy samples.
- Demonstrated a clear, consistent separation of 5182 and 6111 alloy samples into separate bins.

Future Direction

- Conduct sorting tests of additional alloys to establish that the specific separations are possible and establish procedures to group materials by major alloying elements.

Project Rationale

The fraction of the wrought aluminum used in the automobile grows, the automotive aluminum alloys in which use of aluminum in automobiles is becoming increasingly widespread. Previously, this aluminum was predominantly cast material for applications in the power train. Now much more wrought aluminum, both sheet and extrusions, is being used in the bodies of vehicles.

In the past, all aluminum that was recycled from auto shredders was remelted into casting alloys and then reused in cast components. As the amount of wrought material increases, this procedure will eventually become unacceptable because the wrought materials are compositionally distinct from and metallurgically incompatible with the cast materials.

As the fraction of wrought aluminum used in automobiles grows, the scrap-consuming alloys currently used—mainly foundry 380 and 319—will no longer be able to use the entire volume of automotive aluminum scrap. The recycled content of some other alloys will have to be increased to keep the total aluminum recycling system functioning efficiently. In the long term, this change will require the capability to batch desired “hardener”" compositions by sorting mixed alloy scrap shred.

While dismantling of autos will clearly be used to some extent to recover the aluminum, the overwhelming portion of aluminum will be recovered from shredded automobiles. Accordingly, it is envisaged that there will be a modification to the current recycle process. Figure 1 illustrates schematically the additional processing of alloy sorting that will be inserted into the overall recycle loop (see the hatched area). Alloy sorting will require decoating and pre-cleaning of the shredded particles plus separation of aluminum from other metals. Technologies to accomplish these tasks are already all commercially practiced.

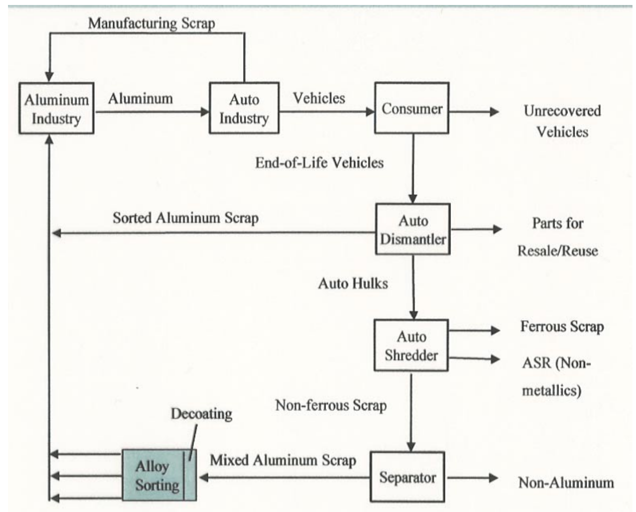

Figure 1. Future automotive aluminum recycling process. *Source*: “The Aluminum Industry Roadmap for the Automotive Market,” The Aluminum Association, May 1999.

There are intermediate aluminum sorting steps that may open some other markets, both automotive and non-automotive, for the shredded aluminum scrap and delay the date when the recycling stream will need composition-based alloy sorting technology. These intermediate steps include separation of cast from wrought particles and further property-based grouping within the wrought and cast alloy families. Accordingly, a more comprehensive picture of the future recycling loop incorporating composition-based alloy sorting is shown in Figure 2. The additional elements are shown in the bottom left side of the figure.

Using alloy-sorting technology to batch tailored hardener compositions will enable keeping the various loops of the total aluminum recycling system closed without requiring that each alloy in a car have substantial recycled content. Thus designers will be able to keep optimizing component

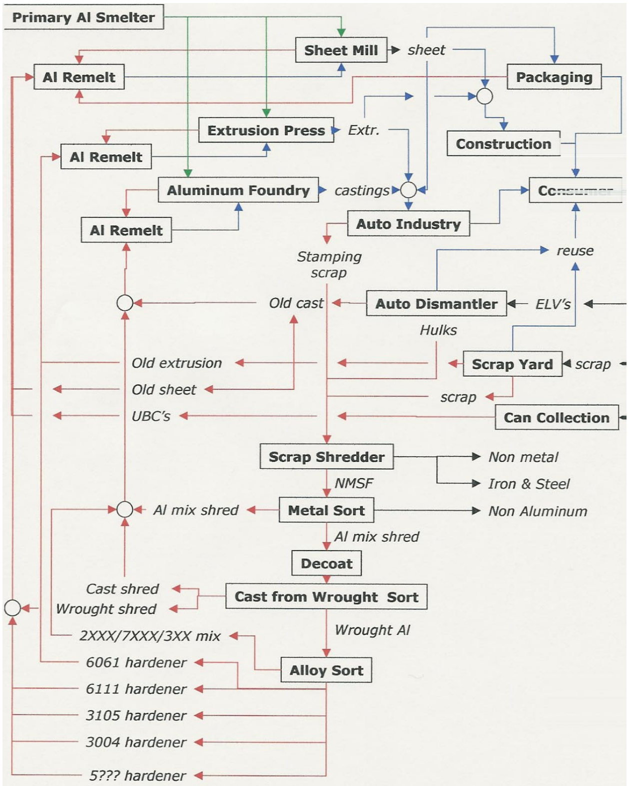

Figure 2. Future aluminum recycling system using alloy sorting technology.

performance and minimizing vehicle weight to make the maximum reductions in greenhouse emissions.

Collection, Identification, and Preparation of Scrap for Sorting Demonstration

The Huron Valley Steel Corporation (HVSC) location in Belleville, Michigan, was the site for the sorting tests. HVSC has developed significant capabilities for the separation of scrap materials and is in an advanced phase of the development of technology for the sorting of aluminum alloys. HVSC has provided mixed-alloy aluminum shred from its current production in quantities of 100–200 tons for plant testing and 1–2 tons for pilot plant testing.

All participants were asked to contribute materials of known composition for the sorting tests. Materials were either as-received from auto shredders or cut to < 4 in. in the largest dimension, and were then hammer-milled to simulate the shredding operation.

The materials collected were designed to anticipate those wrought alloys that will be added to the recycling stream in 10–15 years. The following wrought alloys were already collected in ~100 kg quantity: 2036, 3003, 4147, 5182, 6061, 6111, 7003, 7016, and 7129. Additional samples of 5052, 5754, 6016, 6022 and 6063 have been sought. These known alloys will be seeded into the current unknown alloy mix of both wrought and cast shred pieces. Additional alloy material is continuously being sought. Recently, Ford Motor Company donated three AIV hulks to be shredded and added to the material mix for separation.

The approximate composition of samples of unknown shred pieces was determined using a hand-held optical emission spark spectrometer. HVSC has used this technique to characterize the current generation of the aluminum alloy shred mix, analyzing and identifying ~6,000 individual shred pieces. These pieces are ready for LIBS composition-based sorting.

The HVSC pilot plant was used for the sorting tests. This pilot line simulates full-scale plant operation and operates at a production rate of 7000 lb per hour. Plant tests on separation of cast from wrought alloys were done at the HVSC Belleville and Overpelt, Belgium facilities on a scale of 100–200 tons of aluminum mix input.

Sorting Test Results

Cast from Wrought Alloys

Based on its prior experience, HVSC developed and implemented a proprietary wrought–cast separation method. This method has been tested in plants at both Belleville and Overpelt. The test at Belleville involved 90 tons of material; the resulting products of these tests have been sold commercially, indicating the beginnings of a market for sorted aluminum material.

Table 1 gives the results of the initial Belleville test, expressed in terms of the percentage of the component recovered. The starting material contained some 60% wrought material. The final wrought component contained 85% of the original wrought material, and the final cast component contained 88% of the original cast component.

The demonstration of the viable wrought–cast separation is a major achievement in the

Table 1. Recovery of component in product in the 90-ton test at Belleville

Components	Products	
	Wrought	Cast
Wrought	84.6%	14.3%
Cast	6.3%	88.0%

development of an overall sorting strategy for shredded automobile material, and the development of commercial sales is a further positive sign for the use of aluminum in automotive applications.

Alloy Family Grouping by Selective Etching and Color Sorting

A proprietary alloy coloring process developed by Alcoa was used to provide a color tint to the alloy mix to facilitate color sorting. In this process, exposure to a caustic etching process colors various alloy families differently. With the support of Paul Schultz of Alcoa, the caustic etching process was optimized. The specific conditions chosen following extensive testing were exposure to 10% NaOH solution at 50°C for 60 seconds. These conditions enabled good control and tint reproducibility and minimized metal loss, environmental fumes, and cost.

In Figure 3, material to be sorted passes through the etching process before being placed on the belt. The conveyor belt takes the material under a camera that then enables the sorting process.

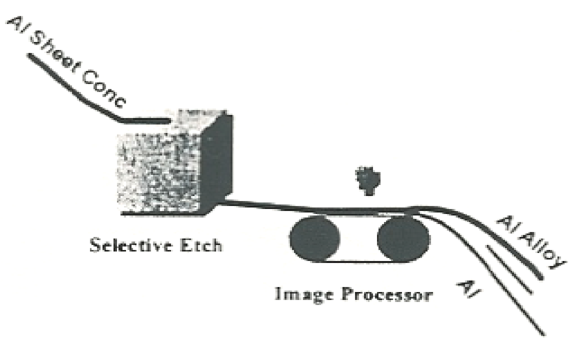

Figure 3. Color sorting process.

Batch samples of alloyed shred were subjected to coloring by selective etching under previously described conditions. Samples of both known and unknown alloys were etched and sorted. The final sort of the HVSC unknown alloy mix was based on a sample of ~400 kg of shred. Color sorting separated the unknown mix into three distinct categories: "bright"—pure aluminum and binary AlMg alloys, "gray"—AlMn, AlSi, and AlMgSi alloys, and "dark"—alloys containing zinc and copper (see Figure 4).

Figure 4. Scrap pieces can be sorted into three major groupings following etching.

Subsequent melt tests were conducted to establish a composition for each color grouping. Compositional and mass balance analysis indicated the following results:

- There is a small metal loss of ~0.5% from the etching process.
- The contrast developed during etching can be increased by higher temperatures, concentrations and times; however, this also increases the metal loss.
- The color sorting method is well suited to separating mixtures of known alloys with good color contrast. Unfortunately, however, there is no direct correlation of color to alloy family group. The sorting method collects unknown alloy mixes into color groups that roughly correlate to the composition of individual pieces.
- The family grouping will be sufficient to open up some new market for auto shred material.

Scrap Sorting Using LIBS

To batch a tailored hardener for a particular alloy from shredded scrap, a sorting method based on chemical composition, rather than physical properties, will be required. The LIBS technology permits quick, precise, non-contact, elemental chemical analysis of the surfaces of randomly shaped scrap particles. This technology is based on optical emission spectroscopy, is more complex and costly than other technologies, and likely will be used only as a final sorting step in combination with other techniques.

Recent proprietary work at HVSC has been successful in integrating LIBS technology to full production scale on a pilot line. Specifically, the integration of laser operation, particle tracking, analysis, and separation has been achieved at production rates of up to 50 shred particles per second.

While some laser ablation of the surface is possible during the LIBS analysis, for optimum analysis results, the surfaces need to be cleaned of all paint and protective coatings. The LIBS technique is schematically illustrated in Figure 5.

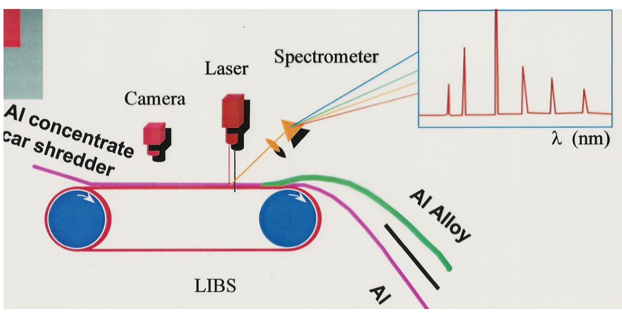

Figure 5. Schematic of the LIBS sorting process.

Sorting Alloy by Alloy

Additional work on the LIBS technology has been conducted during the past period of the contract. The sorting of 5xxx alloys from 6xxx alloys has been conducted. This is of especial interest to the automakers because it is anticipated that these alloy families will compose the bulk of wrought material used in future vehicles.

For this sorting demonstration, attempts were made to separate the alloys 5182 from 6111. Both these alloys are being used in current vehicle production. For the purposes of this demonstration, the pilot plant sorter at HVSC was used. Sheet samples of known 6111 alloy (containing ~1%Mg) and 5182 alloy (containing ~5%Mg) were placed randomly on the sorter belt moving at 300 ft per minute. Following calibration experiments, it was determined that it was best to attempt the sorting based on a distinction of the magnesium concentration in the two alloys, as opposed to silicon or other alloying additions.

Additional trials were conducted to evaluate bulk cleaning procedures for the alloy samples. Specifically, in these experiments, the LIBS Mg/Al and Si/Al spectral intensity ratios, measured for freshly ground surfaces, were compared for other methods of cleaning. On freshly ground surfaces, it was found that that a stable intensity ratio was achieved from the first laser shot (note: there are 4 energy pulses within each laser shot). For other cleaning procedures, ranging from water washing to caustic etching, more than one multi-pulse laser is necessary to achieve the stable intensity ratio, which is assumed to be representative of the bulk alloy composition.

When the experimental conditions had been optimized, the 5182 / 6111 alloy sorting experiment was conducted. It was found that 5182 samples were diverted consistently to bin 1 and 6111 samples were collected in bin 2. A clear separation of 5xxx and 6xxx alloys had been demonstrated, which is highly significant from the viewpoint of enhancing the economics and efficiency of automotive recycling.

Given this successful demonstration, additional alloy sorting tests have been scheduled. For example, the separation of the following alloy groups is under way or is planned:

- 3003/3004
- 3003/6111/5182/2036/7129
- 5182/5754/5052
- 6111/6061/6063/6082

In addition to establishing that these specific separations are possible, these tests will also establish procedures to group materials by major alloying elements. Alternatively, these tests could enable sorting of particles compatible with a particular alloy or batching of materials to achieve a particular alloy composition. All this should make it possible to batch a product with the particular selected proportions of alloying elements to provide a “tailored hardener,” as was discussed in the context of the future aluminum recycling scheme shown in Figure 2.

Process Improvements

Since the start of the project, several process improvements have been incorporated into the

HVSC pilot line. For example, a faster and more powerful laser system has been acquired, and a more efficient spectrometer has been received and incorporated into the line. Also, new laser beam delivery optics and light collection optics are due to be installed during the October–November timeframe. The latter equipment is designed to minimize variations in the LIBS spectral line intensity resulting from variations in particle height or position on the moving belt. These improvements will further improve the effectiveness of the LIBS sorting system.

Finally, the pilot line has been modified to enable "multi-stream diversion." Initial test results showed 95% diversion of the targeted particles into the first two product streams and ~80% into the third stream at industrial-level belt speeds and loadings.

However, it is already clear from testing on the HVSC pilot plant that the process of LIBS is sufficiently robust to sort particulate material from shredded autos on a particle-by-particle basis. In fact, HVSC is moving forward with plans to develop a commercial facility in anticipation of the developing market for the recycling of aluminum-containing material from shredded automobiles.

Summary and Outlook

Test materials from shredded automobiles have been assembled and prepared for sorting tests. Etching procedures have been developed to provide a color tint to a mix of shredded aluminum auto material, and techniques have been developed to enable this material to be sorted by color. Additional work will be needed to refine and verify the process economics.

The separation of cast from wrought shredded auto material has been demonstrated in two large-scale tests in the United States and Europe. This process has already been industrially implemented in the HVSC plants, opening new markets for the wrought aluminum shred from auto shredders.

The sorting of alloys using the LIBS process has also been shown to be technically feasible and can clearly separate material on an alloy-by-alloy basis. Specifically, it has been shown that it is possible to readily separate 5xxx from 6xxx alloys. This is especially significant to automakers because wrought material from these alloy families will be used extensively in future vehicle production. The project has demonstrated that technology will be available to sort auto shred material when the market need arises. Accordingly, there should be no concern that the growing quantities of aluminum going into the automotive cannot be recycled in the future.

The large market for recycling wrought aluminum from automotive is probably still at least 10 years away, since the current vehicles with high aluminum content will be used by the consumer for the next decade. Exactly how the technology will be deployed in the future will depend on many factors, such as the specific alloys used, the cost of process sensors, the amounts of aluminum used in future vehicles, and the overall economics of the sorting process. However, there is little doubt, based on the demonstrations conducted to date on this project, that scrap sorting technology will be available when it is needed.

Additional Reading

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B. Recycling of Polymer Matrix Composites

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Contractor: Argonne National Laboratory
Contract No.: W-31-109-Eng-38

Objective

- Develop efficient and cost-effective processes for recovering carbon fibers from polymer matrix composites (PMCs).

OAAT R&D Plan: Task 9; Barrier E

Approach

- Conduct process improvement studies to determine the optimum operating conditions.
- Determine disposal and treatment requirements of the effluent stream.
- Develop a conceptual design for a full-scale plant.

Accomplishments

- Evaluated three processes to separate carbon fibers from PMC scrap: thermal treatment, chemical treatment, and thermal shock. The thermal treatment method, a single-step process, recovered carbon fibers with properties equivalent to those of their virgin counterparts. Economic analysis of this method also showed it to be potentially economical. Therefore, its development was continued in FY 2001.

Future Direction

- Work with a carbon fiber manufacturer to produce and test PMC panels made with recovered fibers and compare the properties of these panels with those of similar panels made with the same type of virgin fibers.
- Produce about 200 lb of recovered carbon fibers for use in an automotive application.
- Investigate the technical feasibility and advantages of using a hybrid treatment process (a thermal process followed by a chemical process, and vice versa).

Process Improvement Study of the Thermal Treatment Method

An experimental study was conducted to determine efficient and economical process operating conditions for recovering fibers from both urethane and epoxy substrates. Samples as large as 4 × 12 in were treated at temperatures of up to 1750°F in air or in an inert environment. The pilot-scale and the bench-scale experiments produced consistent results.

Test Results Obtained From Treating PMC Samples Made With Urethane-Based Substrates

At temperatures greater than 500°F, the carbon fibers were completely freed from the polymer, given enough residence time. At 1250°F, the required residence time was about 5 minutes. About 45% of the weight of the samples was the polymer substrate that was pyrolized when the PMC samples were treated in an inert environment at different temperatures for prolonged periods of time. Appreciable oxidation of the fibers did not start until after 8 minutes. To determine the degree of oxidation, we repeated the experiments at 1250°F in a nitrogen environment. The results are shown in Figure 1. The recovered carbon fibers were also examined under a microscope. Figure 2 is a picture of recovered fibers at 1250°F in air for 5 minutes. This picture shows that the fibers are free of contamination. The individual fibers are also free from each other, and their surfaces are smooth and have the same diameter. These results indicate that no oxidation has occurred at the surfaces of these fibers.

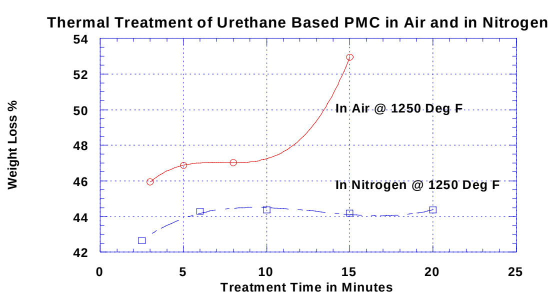

Figure 1. Weight loss of polymer matrix composite samples with urethane-based substrate treated in air or in nitrogen at 1250°F.

Figure 2. Carbon fibers recovered at 1250° F in air for 5 minutes from a polymer matrix composite sample with a urethane substrate.

Test Results Obtained From Treating PMC Samples Made With Epoxy-Based Substrates

Two types of PMC materials made with epoxy substrates were tested. The first was supplied by Oak Ridge National Laboratory (ORNL) and was about the same thickness (~ 1/16 in.) as the samples made with urethane substrate; however, it was cylindrical in shape. The other type was in the form of plates of about 1/16 in. in thickness, and was supplied by Hexcel Corporation. The Hexcel samples had well-specified properties and materials of construction. The ORNL samples were more difficult to treat than the samples made with the urethane substrate, even though the substrate constituted only about 25% of these pieces by weight. Further, unlike the urethane-based PMC samples that produced loose fibers when treated, the ORNL samples remained somewhat intact and retained some level of rigidity and stiffness after treatment. However, when a mechanical force was applied to the treated samples, such as repeated manual bending, the fibers became soft and loose; and when they were aerated using compressed air or placed in water with agitation, most of the residual solid flakes fell off. These samples also suffered some oxidation when treated in air, as shown in Figure 3.

These data show that the treatment process was completed in about 10 minutes. When the treatment was carried out in air, about 8% of the fibers were lost as a result of oxidation. The reason for the higher level of oxidation is not clear at this time.

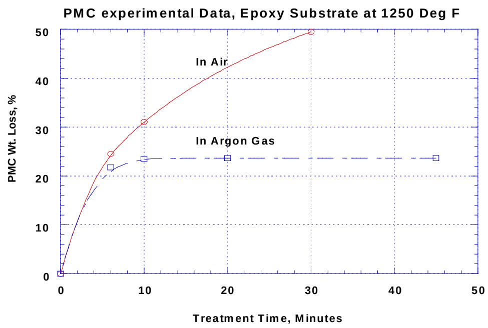

Figure 3. Weight loss of the ORNL polymer matrix composite samples with epoxy substrate treated in air or in argon gas at 1250°F.

The Hexcel PMC samples containing the epoxy substrate were more readily treatable than the ORNL samples. Samples of various dimensions were treated at temperatures in the range of 850 to 1500°F in air and in a nitrogen environment. These samples were made with about 30% epoxy substrate by weight and about 70% fiber. The results of the treatment are presented in Figure 4. Significant oxidation of the fibers did take place after prolonged treatment in air. The treatment was essentially completed in 5 minutes when conducted in air at 1250°F, and in about 10 minutes when conducted in nitrogen at the same temperature. We plan to process about 40 test panels from Hexcel in FY 2002. The fibers recovered from these panels will be characterized to determine the physical properties of recovered fibers relative to those of "virgin" fibers. The recovered fibers will also be used to produce 20 "recycled" test panels to determine the physical properties of the recycled panels vis-à-vis the properties of panels made of virgin fibers. The results of the process improvement study suggest treating PMC scrap at 1250°F in air

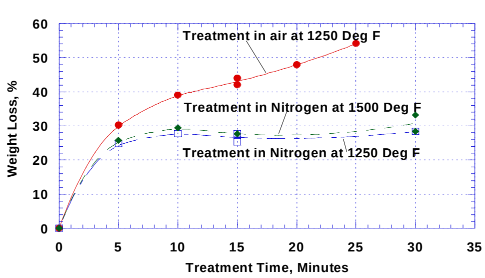

Figure 4. Weight loss of the Hexcel epoxy sample.

for about 10–15 minutes will enable the operator to recover carbon fibers from the PMC scrap with minimal loss of fibers and without having to use nitrogen. Changes in these operating conditions may be necessary to accommodate different types of scrap.

Determination of the Disposal and Treatment Requirements of the Effluent Stream

The PMC waste stream will contain scrap made with various types of thermoset and thermoplastic substrates. The thermal treatment method will be capable of treating all of these types. However, different substrates will generate different pyrolysis and oxidation products, including volatile and semi-volatile organic compounds. Based on the heating values of polymers, we estimate that at least 60% of the energy requirements of the process could be satisfied by the energy value of this stream. In most cases, the PMC panels are made with about 50% polymeric substrate that has a heating value of over 18,000 Btu/lb. The energy released upon combustion of the polymeric substrate is enough to maintain the treatment reactor at the desired temperature; therefore, the system can be self-sufficient in energy. An afterburner is required to destroy residual carbon monoxide and organic compounds. The process does not generate liquid or solid waste streams.

Development of a Conceptual Design for a Full-scale Plant

A conceptual design for the process was developed, including subsystems for loading the PMC scrap and for collecting the recovered fibers. We used this conceptual design and the data from the process improvement study to update the economic analysis for a full-scale plant processing 1,000,000 lb/year of PMC scrap. The following assumptions were also made:

- The recoverable carbon fiber in the PMC scrap is 40% of the scrap weight
- The scrap contains 50% by weight polymeric substrate that has a heating value of 18,000 Btu/lb.
- The recovered fibers are valued at $1.50 per pound, based on a value of $3/pound for the virgin fibers

We also estimated the cost of the system to be \$1,000,000. The results, summarized in Table 1, suggest a payback of less than 2 years.

Table 1. Process economics (annual basis) for carbon fiber recovery from obsolete PMC materials

Revenues, \$1000/year	
Recovered carbon fibers @ \$1.5/lb	600
Other	0
Total revenues	**600**
Operating costs, \$1000/year	
Feedstock (credit for avoided disposal)	–10.0
Waste disposal	0.0
Utilities	25
Labor	50
Maintenance	20
Total operating costs	**85**
Net income, \$1000/year	**515**
Capital cost, \$1000	**1,000**

Milestones

The following milestones of the proposed effort are to be conducted in FY 2002.

- Complete testing of the PMC panels made with recovered carbon fibers.
- Complete production of 200 lb of recovered fibers for testing in an actual application.
- Assess the technical feasibility and advantages of using a hybrid treatment process (thermal followed by chemical and chemical followed by thermal).

Conclusions

Work conducted so far has demonstrated that the recovery of carbon fibers from PMC scrap is technically feasible and potentially economical. Establishment of the properties of the recovered fibers and of products made with recovered fibers is essential in order to identify applications for the recovered fibers.

C. Investigation of the Cost and Properties of Recycled Magnesium Die Casting for Employment in Automotive Applications

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Contract No.: DE-AC06-76RL01830

Objective

- Investigate the recycling of magnesium alloys—AZ91D, AM60, and AM50—from previously cast Class 1 and Class 2 die-cast material.

OAAT R&D Plan: Task 9; Barriers A, E

Approach

- Determine the quality of the cast metal from individual melts cast by permanent molding and squeeze die casting in two recycling shops. The effects of various flux additions and of the refining process used were determined by the tensile strength and ductility.
- When the quality of the metal produced in the permanent molds is not adequate, repeat the process using other fluxes and improved processes.
- Using this procedure, obtain desirable properties for all three alloys. (Some difficulty was experienced with the AZ91D composition.)

Accomplishments

- During the refining process, planned and conducted tests on AZ91D, AM60, and AM50 alloys in the Case Western Reserve University (CWRU) foundry and at the cooperating business recycling shops at Garfield Alloys and MagReTech. The overall purpose of the tests was to develop a technical procedure that will produce good die-casting melt quality in the three alloys from Class 1 and Class 2 scrap.

Introduction

Magnesium alloys have many advantages for specific structural applications. Until recently, most magnesium castings were used in specialized applications, such as aircraft or hand tools, where lightness is important to minimize weight, improve handling, or reduce inertia. Cast products offer advantages over some wrought products because work-hardening of the HCP lattice structure in the latter can cause difficulties. However, the generally higher premium cost and variable price of magnesium vis-à-vis aluminum has interfered with the commercial growth of magnesium alloys. Many of these economic factors either have been corrected or are being muted by current developments. The die-casting process has been an excellent method of producing magnesium castings because of the rapid production of parts with close-to-final dimensions.

These improvements have resulted in the growing use of magnesium casting for more general usage, particularly in automotive applications because of their light weight. The present technical literature[1–7] continues to state the advantages of magnesium alloys, including its increased availability, improved machineability, and light weight. These articles do highlight some problems, such as the need for careful handling or particularly, with the requested properties, for some purposes. One of the problems in improving the development of magnesium alloys has been the reclamation of the scrap produced.[1–7] When the scrap is in the form of Class I material, such as gates and rejected castings, it can frequently be processed through normal procedures recommended for magnesium. However, lighter Class 2 scrap, such as turnings and thinner materials, requires considerably improved handling. The development of improved sludging, evaporation, and filtering technologies will be of assistance in processing this Class 2 material so that it can be used to produce magnesium alloy of the proper quality.

In addition to these issues, the presence of some elements (Table 1) has to be controlled. They exert an effect on the behavior of magnesium alloys with which they are in close contact.

Table 1. Effect of alloying elements on magnesium alloys

Manganese
- Does not affect mechanical properties
- Promotes corrosion resistance

Silicon
- Present in small amounts in most alloys
- Improves creep strength by producing Mg_2Si phase

Beryllium
- Sometimes added at low levels <10 ppm to minimize oxidation of the molten alloy
- Does not affect mechanical properties

Impurities—Iron, Nickel, and Copper
- Controlled at ppm levels
- Negligible effect on mechanical properties
- When in excess of spec cause large loss in corrosion resistance

Background of the Project

The casting group at CWRU has been proceeding in several directions to satisfy the requirements in the statement of work. First, an arrangement was made with Garfield Alloys and MagReTech to work with CWRU in preparing and testing magnesium alloys that are preferable to those produced with currently used procedures. The activities have included casting and testing of the alloys produced with various processing techniques at these two shops and at CWRU. This work has included tests on the tensile properties of bars cast from these alloys. Under the arrangement, CWRU personnel visit both Garfield Alloys and MagReTech, and the supervisory personnel of these plants visit CWRU. Both organizations have benefited from the arrangement. CWRU has run several small-scale tests on reclaiming material and has been aided by the comments of the two companies. The supervisory personnel from the plants have made several recommendations for controlling the processing equipment employed at CWRU. CWRU has analyzed many of the fluxes used by these companies and has made specific recommendations.

In addition, the CWRU Foundry Group has maintained a close relationship with a local die-casting shop that is using reclaimed scrap from Garfield Alloys and MagReTech to produce magnesium castings on a production basis. This die-casting shop, Magnesium Aluminum Corporation, has been providing magnesium alloys castings for 40 years on a commercial basis. Personnel from CWRU have visited the die-casting shop and shop personnel have visited the operation at CWRU.

Comments from supervisors at the shop have been used to improve CWRU's process.

Investigation Processes

The first project task was to develop sludging, evaporation, and filtering processes to produce magnesium alloys, using Class 1 scrap from a commercial magnesium recyclers, with the same properties as virgin magnesium die-cast AZ91D, AM50, and AM60. As part of this work, Garfield Alloys and MagReTech cooperated in a series of experiments involving Class 1 scrap. The scrap material was melted in 2300-lb steel crucibles at Garfield Alloys and 6500-lb steel crucibles at MagReTech. The Class 1 scrap was melted in the furnace with a control cover of flux, and a standard fluxing and sludging treatment were used throughout the working cycle. Tests were conducted and the properties of the different alloys measured when the fluxing started after the Class 1 alloy was charged in the furnace. A cover and drying flux were used as the test proceeded. The compositions of these fluxes were changed somewhat to improve the quality of the alloys as the treatments proceeded. The fluxes that were employed followed the general pattern shown in Table 2.[8]

The processing of AM60 and AM50 proceeded without problems. However, adjustments in the analysis of the treatment slags were found to be necessary to obtain good properties with AZ91D. necessary to obtain good properties with AZ91D. This alloy is very susceptible to difficulties and receives special treatments when fluxing and sludging are employed.

The results for AM50, AM60, and AZ91D show that AZ91D is a more difficult alloy to process than the others because of the presence of oxide inclusions that form readily in it. Treating for long periods of time with the fluxes produces alloys that are generally clean, without inclusions. Another problem that occurs is black spots on the fracture surface of the tensile bars. The composition of these fluxes and the analysis of these black spots are shown in Figure 1. A brightmeter has been obtained from Daimler Chrysler for use in testing and in analyzing the results of these tensile tests.

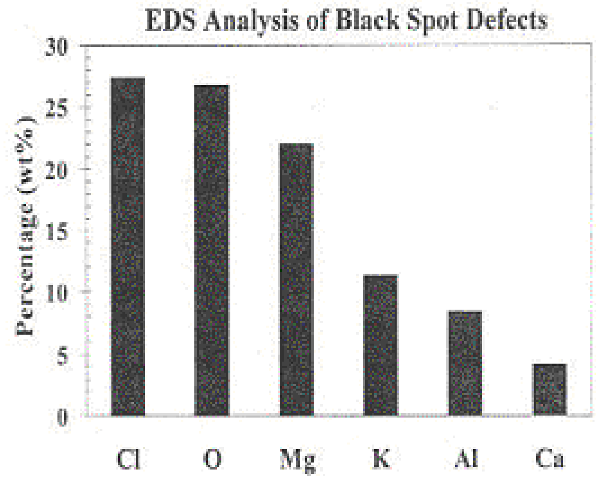

Figure 1. Black spots on the fracture surface of PM AZ91D.

This initial work was conducted with Class 1 scrap at each plant. Subsequent work at Garfield Alloys was conducted on AM60 alloy using Class 2 scrap to attain the required results. The work with the Class 2 scrap is considerably more difficult and time-consuming than the work with the Class 1 material. After some of the difficulties with handling Class 2 scrap were determined, it was decided to limit the amount of work being conducted at the two cooperating shops and conduct the work at CWRU. The loss of time and production interferes with production schedules at the commercial shops.

Table 3 shows the properties of the permanent mold test pieces based on a permanent mold test bar shown in Figure 2. This mold was used to cast test bars from the alloy product during the refining operation that employed the standard fluxing and sludging at Garfield Alloys and MagReTech. To obtain good properties, it was necessary to change the fluxes to those with higher fluoride content and to slow the operation down. The specialized work to obtain the AZ91D alloy properties using this special fluxing and sludging procedures eliminated the inclusions.

Table 2. Fluxes for magnesium-alloy melting

Flux No.	KCl	$MgCl_2$	MgO	CaF_2	$BaCl_2$	$MnCl_2$	Use
230	55.0	34.0	….	2.0	9.0	….	Remelting pots, fluid slag
232	37.5	42.0	7.5	8.5	4.5	….	Remelting pots, fluid slag
310	20.0	50.0	15.0	15.0	….	….	Crucible melting, dries during use

Table 3. Mechanical properties of permanent-mold and squeeze-cast magnesium alloys

Mechanical properties magnesium alloys	UTS (ksi)	Elongation (%)
AM50—Permanent mold (as cast)	27.7	10.8
AM60—Permanent mold (as cast)	29.7	11.2
AM60—Squeeze case (as cast)	31	7.25
AZ91D—Permanent mold (as cast)	23.4	3

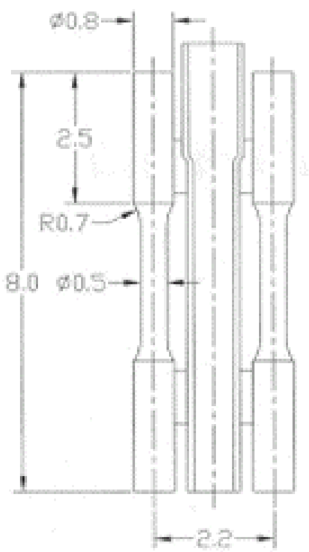

Figure 2. Permanent mold for tensile test bar.

The results for the refined alloys are reported for the test casting in permanent mold test bars. These molds produced a constant quality of casting when the material being cast was properly treated. Throughout the work being processed, the reclaimers employed for processing were excellent at both locations. However, when the squeeze-cast mold shown in Figure 3 was employed at CWRU, the strength and ductility of the test bars were somewhat lower. It became apparent that the bars did not have a consistent cross-section without the presence of inclusions or segregations. To improve the properties of the squeeze-cast bar test, a faster pour in a hotter mold was needed. A comparison of

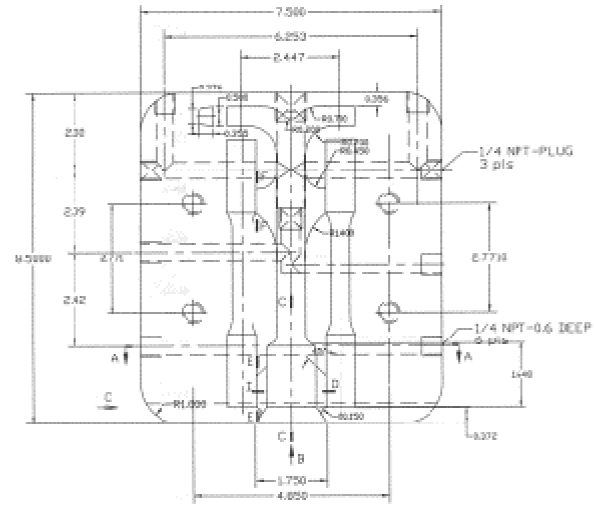

Figure 3. Squeeze-cast mold for test bar.

the structures for these bars show a dendrite arm spacing (DAS) of about 25 microns in the permanent mold bar and only 15 to 16 microns in the squeeze-cast bar. Previous work[9] has indicated that the closer spacing of the DAS squeeze-cast bar should normally provide better properties in the absence of defects. However, attaining the appropriate conditions will require improvements in casting procedures.

The squeeze-cast bars were produced in the mold shown in Figure 3, where the general pattern of feeding from both ends is present. However, the colder die and rapid solidification did not allow the test bar to attain the appropriate structure along its length. It is apparent from these results that it is necessary to pour the casting into a mold faster at a higher temperature. The results we have obtained to date show tensile strengths that approach and even exceed those of the bars made in permanent molds in some cases, as shown previously. Because of the finer DAS of the squeeze-cast bars compared with the permanent mold bars, a higher tensile strength was expected. However, the facilities we have available cannot attain the shot speed or the higher die temperature on a regular basis. Although these results will eventually be obtained, it will require more equipment than is available within the limits of this agreement. For this reason, the metallographic problems with the squeeze-cast test parts require that the permanent mold be widely used. The results from the permanent mold tests are generally used in the work on reclaiming magnesium alloys made from Class 1 and Class 2 types of scrap.

Our experience has indicated that the difficulties with the Class 2 alloy involve the presence of inclusions and small particles of scrap material still remaining in the melt. These types of inclusions have reduced the properties significantly. Methods of eliminating these inclusions, so that they do not lower the tensile properties or appear as defects in the brightness examination, are to be developed using our large melting furnace and tapping the alloy into the ingot molds after further melting and processing. This equipment is shown in Figure 4.

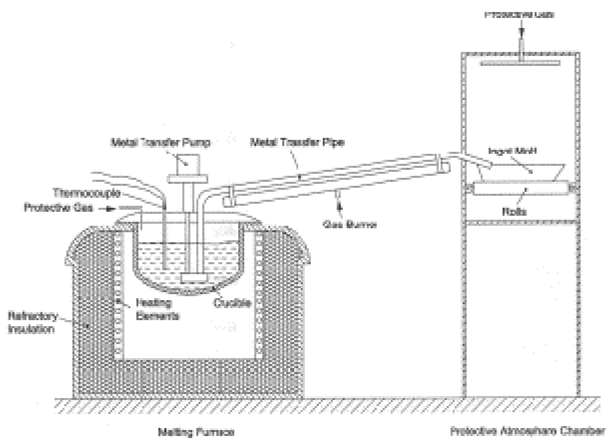

Figure 4. Experimental set-up for recycling magnesium alloys.

The equipment uses a gaseous, non-salt cleaning procedure in which inclusions in the melt are removed by skimming with argon gas through the melt. The skimming causes the inclusions to rise to the surface so that impurities can be removed via a non-fluxing skimming method before casting. This equipment has been used experimentally for non-fluxing procedures to produce the good-quality test runs shown in Figure 5. This method has shown its efficiency in producing the better properties of the clean alloy.

Conclusions

At present, the investigation that we have undertaken has shown that excellent tensile properties and quality of metal can be obtained for AM60, AM50, and AZ91D with proper reclamation. These investigations have included both Class 1 and Class 2 scrap from other die casting operations. Attaining these properties required the use of fluxing, sludging, and skimming removal

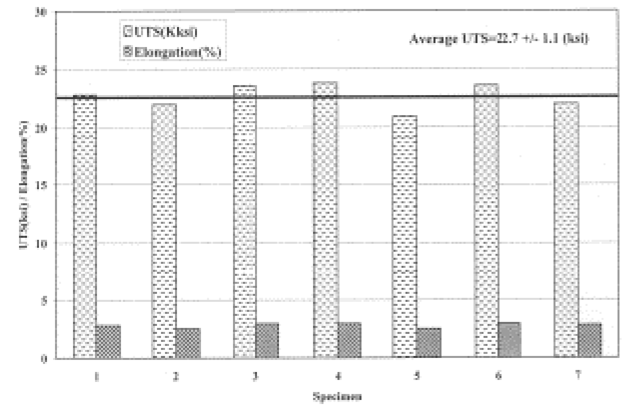

Figure 5. Ultimate tensile strength and elongation of fluxless recycled AZ91 at CWRU 032001.

techniques. The tensile test results obtained have required considerable periods of time for these operations. The results have included the large 3600-lb and 5400-lb steel melting furnaces at Garfield Alloys and MagReTech.

Excellent results have also been obtained with smaller heats processed at CWRU using the same three alloys. These results are reported based on the tensile properties and fracture surfaces of the test bars produced as permanent test bar castings. Both Class 1 and Class 2 scrap have been tested at CWRU.

The results obtained with squeeze-cast test bars, however, have not been consistently up to the standards attained in the permanent mold test bars. The difficulty has been that the squeeze-cast bars have contained areas of eutectic alloy. These areas have formed during the solidification of the test bars and have not exhibited the strength expected in this process.

The overall results of the work indicate that the reclamation at Garfield Alloys, MagReTech, and CWRU produced quality products. The primary work with the magnesium processor was for Class 1 scrap with alloys of AM50, AM60, and AZ91D. The work conducted with CWRU included both Class 1 and Class 2 alloys. The processing at Garfield Alloys and MagReTech used the conventional fluxing procedure. The CWRU process included treatment of the AZ91D alloy with a non-fluxing method that bubbled argon through the melt and removed the impurities by skimming.

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D. Recycling Assessments and Planning

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Contract No.: W-31-109-Eng-38

Objectives

- Establish priorities for cost-effective recycling of advanced automotive technology (AAT) materials and components associated with PNGV and beyond.
- Focus research on near-term recycling on the technologies critical to achieving 85% recyclability when the first wave of the PNGV fleet reaches end-of-life status.
- Focus long-term efforts on enhanced recovery/sorting procedures and advanced recycling technologies that are enabling factors in a vision for 100% recyclability.

OAAT R&D Plan: Task 9; Barrier E

Approach

- Consult with automotive manufacturers and recycling industries, the U.S. Council on Automotive Research (USCAR) and its affiliates, national laboratories, universities, and other relevant organizations to assess critical recycling needs/barriers.
- Develop a recycling research and development (R&D) program plan that will serve as a working document to guide DOE/OAAT in establishing priority goals, with an initial emphasis on lightweight body and chassis materials.
- Assist DOE in establishing advanced recycling R&D initiatives and provide technical oversight to ensure that priority objectives/goals are accomplished.

Accomplishments

- Organized the AAT Recycle Roadmapping Workshop during the fourth quarter of FY 2000. The workshop was facilitated through a subcontract with Energetics. The roadmap document, *A Roadmap for Recycling End-of-Life Vehicles of the Future*, was completed in May 2001 and will now provide the basis for structuring specific projects consistent with the needs of the key stakeholders.

- Identified related federally funded research projects, one funded by the U.S. Department of Commerce and three funded by the National Science Foundation during 1997 through 2000. The principal investigators of those projects have been contacted and reports from their projects have been requested.
- Continued to work with the Automotive Aluminum Alliance on its project to develop technology to sort aluminum alloys by class using color sorting and/or laser-induced breakdown spectroscopy. Both technologies are being tested on a large scale at Huron Valley Steel Corp.

Future Direction

- Structure a suite of prioritized projects over the next year in collaboration with the stakeholders.
  - Continue work on development of the AAT Recycling Program R&D Plan through collaboration with the USCAR Vehicle Recycling Partnership and associated USCAR affiliates, the Advanced Battery Consortia, the Automotive Parts Rebuilders Association, remanufacturing industries, materials trade organizations, and other appropriate industries involved in automotive recycling.
  - Continue evaluations of recycling technology progress in Europe and Japan, along with assessments of critical recycling technologies as new needs are identified.
  - Continue to work with the Automotive Aluminum Alliance, which is working with Huron Valley Steel Corporation to demonstrate technology developed by Huron Valley Steel for separation of aluminum alloys.
- Continue project efforts to assist DOE in preparation of AAT planning documents, priority recycling R&D needs, proposal reviews, and related tasks throughout FY 2002.

Summary

The recycling program objective is to establish priorities and develop cost-effective recycling technologies and strategies in support of long-term DOE OAAT objectives and goals. Automobile recycling is the final productive use of end-of-life vehicles (ELVs). The obsolete car has been a valuable source of recycled raw materials and useable parts for repair as long as cars have been mass-produced. Today, cars that reach the end of their useful service life in the United States are profitably processed for materials and parts recovery by an existing recycling infrastructure. That infrastructure includes automotive dismantlers who recover useable parts for repair and reuse, automotive remanufacturers who remanufacture a full range of components—including starters, alternators, and engines—to replace defective parts, and ultimately the scrap processor who recovers raw materials such as iron, steel, aluminum, and copper from the remaining auto “hulk” after components have been recovered for recycling. Each of these activities contributes to the recycling of obsolete vehicles.

Today, less than 25% by weight of obsolete cars is not profitably recoverable for recycling and is therefore landfilled. Over the past 10 years, the original equipment manufacturers—Ford, General Motors, and DaimlerChrysler—through the Vehicle Recycling Partnership and other organizations (including the Aluminum Association, American Plastics Council, Institute of Scrap Recycling Industries, Automotive Recyclers Association, Automotive Parts Rebuilders Association, and the federal government) have been working both collaboratively and independently to address technical, institutional, and economic issues that limit the recycling of ELVs. Progress has been made toward understanding some of these issues, and technology has been developed that can impact the level of ELV recycling.

The recyclability of ELVs is currently limited by a lack of commercially proven technical capabilities to cost-effectively separate, identify, and sort materials and components, and by a lack of profitable post-use markets. While nearly 75% by weight of ELVs currently are recycled in some form, the remaining 25% is sent to landfills each year. Over the next 20 years, both the number and complexity of ELVs are expected to increase, posing significant challenges to the existing recycling infrastructure. The automobile of the future will use significantly greater amounts of lightweight materials (e.g., ultralight steels, aluminum, plastics, composites) and more sophisticated/complex components.

New technology is and will continue to be needed to improve vehicle recyclability. Using the Roadmap as a framework, we will work with the key stakeholders over the next year to structure a suite of projects to be undertaken in this program area.

The following strategy was developed as part of the Roadmap to maximize the value recovered from ELVs.

- Come together as a unified recycling community to share the costs of the development of required new technologies.
- Incorporate reuse, remanufacturing, and recycling into the design phase for cars whenever possible.
- Recycle as early in the recycling stream as possible while relying on the market to optimize the value and amount recycled at each step.
- Maintain a flexible recycling process that can adapt to diverse model lines fabricated with different techniques and materials from various suppliers.
- Develop automated ways to recover bulk materials.
- Emphasize R&D on post-shredding material identification, sorting, and product recovery
- Focus R&D efforts on materials not currently recycled by sorters (e.g., post-shredding glass, rubber, fluids, textiles, plastics).
- Develop uses for recovered materials (whether in the same or different applications) and testing specifications.
- Encourage investment in the infrastructure needed to achieve the recyclability goal. Build on the existing infrastructure.
- Develop a means to prevent the entry of PCBs and other hazardous materials into the recycling stream and promote acceptable limits in shredder residues.
- Consider the recycling requirements of new technologies entering fleets as early as possible.

7. ENABLING TECHNOLOGIES

A. Durability of Carbon-Fiber Composites

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Contract No.: DE-AC05-00OR22725

Objective

- Develop experimentally based, durability-driven design guidelines to ensure the long-term (15-year) integrity of representative carbon-fiber-based composite systems that can be used to produce large structural automotive components. Durability issues being considered include the potentially degrading effects of cyclic and sustained loadings; exposure to automotive fluids; temperature extremes; and the low-energy impacts of such events as tool drops and kickups of roadway debris on structural strength, stiffness, and dimensional stability.

OAAT R&D Plan: Task 4; Barriers A, C

Approach

- Characterize and model the durability behavior of a progression of three representative carbon-fiber composites; each has the same thermoset urethane matrix but with a different reinforcement type and configuration: (1) reference crossply, (2) quasi-isotropic, and, (3) random chopped fiber.
- Replicate on-road conditions in laboratory tests of each composite to generate durability data and models.
- Develop and publish durability-based design criteria for each composite.

Accomplishments

- Published a report on durability-based design criteria for the reference crossply carbon-fiber composite (ORNL/TM-2000/322).
- Completed the required experimental characterization of the durability of quasi-isotropic carbon-fiber composite.
- Developed a suitable chopped-carbon-fiber composite system that met Focal Project 3 design requirements and that will be the basis for durability characterization and modeling [Automotive Composites Consortium (ACC)].

Future Direction

- Develop durability-based design criteria for quasi-isotropic carbon-fiber composite and publish a report.
- Publish a report on the development of a mechanical model for predicting the long-term, time-dependent, nonlinear response of directed-carbon-fiber composite from short-time tests.
- Complete durability characterization and modeling of Focal Project 3 chopped-carbon-fiber composite and publish a design criteria report.
- Address the durability of thermoplastic carbon-fiber composites.

Material Development

The three molded thermoset composites being addressed are differentiated by their carbon-fiber preforms, which are depicted in Figure 1. All three have the same Bayer 420 IMR urethane resin matrix. The required experimental characterization of the quasi-isotropic composite is complete. The chopped-fiber composite will next be addressed.

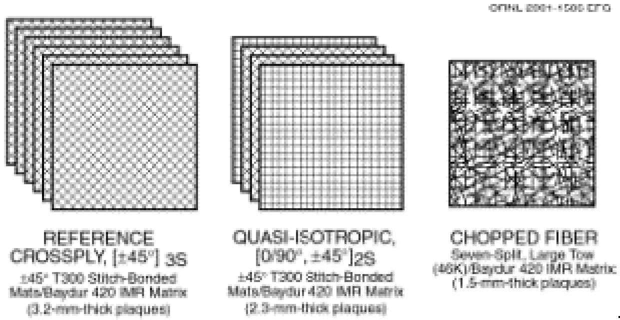

Figure 1. Preforms used in progression of carbon-fiber thermoset composites being characterized and modeled.

In developing a viable chopped-carbon-fiber composite, the ACC Materials group has focused its work on the requirements being developed for Focal Project 3. A 60% mass reduction and cost parity with steel have directed work toward the low-cost, high-tow carbon products from suppliers such as Zoltek. Further, the high productivity requirements (100,000 units per year) have similarly directed work toward liquid molding and the programmable powdered performing process (P4) chopped-fiber technology demonstrated in the ACC Focal Project 2 truck box program. The material requirements resulting from these developments are for a chopped-carbon-fiber–reinforced composite containing 40 vol % fiber and capable of being molded at a thickness of 1.5 mm. In chopped-fiber composites, both the 40 vol % fiber content and the 1.5-mm thickness represent substantial technology challenges.

Several chopping and molding runs were carried out using high-tow fibers from Hexcel, Toray, and Zoltek. Also, several sizings were used. Sizings are materials introduced by the fiber manufacturers to hold and stabilize fiber tows (bundles) during handling and shipping. “Hard” sizes keep the bundles tightly together during molding; “soft” sizes allow the bundles to break down into individual fibers or smaller fiber bundles. Soft sizes lead to better properties, but they result in more difficult preforming and higher molding pressures.

Ultimately, Zoltek Panex 33, seven-split 46K carbon fiber with an X8 size package, was found to perform the best, both in terms of ease of performance using the ACC P4 machine and in terms of the resulting mechanical properties. Using a

1500-ton press, the 1.5-mm-thick plaques have been produced with fiber volumes ranging from 42 to 46%. An ultimate tensile strength (UTS) of 250 MPa, tensile modulus of 45 GPa, and failure strain of 0.6% easily satisfy Focal Project 3 design requirements.

This composite has consequently been identified as the main stream material for the ACC Focal Project 3 low-mass body-in-white structure. The next step is to produce a quantity of plaques of the chosen material for complete ACC characterization, for durability characterization and modeling, and for university programs.

Basic Properties and Environmental Effects

Basic short-time properties used to develop design criteria include tensile, compressive, shear, and flexural strength. These have been developed for the quasi-isotropic composite (40 vol % fiber) over the automotive design temperature range of –40°C to 120°C. Also, the effects of fluids on each property have been determined. Average tensile, compressive, and shear properties are tabulated in Table 1 at three key temperatures. Multiplication factor curves have been derived from these, and from data at other temperatures, to allow the determination of a property at any temperature from the corresponding room-temperature value.

Table 1. Baseline tensile, compressive, and shear properties

	Temperature		
	–40°C	–23°C	120°C
Tension			
Modulus, GPa	33.7	32.4	29.8
Strength, MPa	292	336	272
Compression			
Modulus, GPa	32.7	32.1	27.3
Strength, MPa	230	225	131
Shear			
Modulus, GPa	12.1	12.2	10.9
Strength, MPa	240	226	133

The basic short-time allowable stress, S_0, for design is defined as two-thirds of the minimum UTS. The B-basis minimum UTS used is based on statistical treatment of 86 strength values so that the survival probability at the minimum stress is 90% at a confidence level of 95%. The resulting room-temperature S_0 value is 201 MPa. Values at other temperatures are obtained by using the aforementioned multiplication factors.

The effects of two standard bounding fluid exposures—1000 h in distilled water and 100 h in windshield washer fluid (70% methanol)—on baseline properties were investigated. The distilled water had the greatest effect; a factor of 0.94 bounds the effects on strength and stiffness in tension, compression, and shear.

The effects of temperature cycling, such as that experienced by automotive structures, were investigated by cycling groups of tensile, compressive, and shear specimens between –40°C and 120°C twenty-five times prior to testing. The only significantly deleterious effect of the cycling before testing was on shear stiffness, which was reduced by 25%.

Some bending is unavoidable in structures. While the quasi-isotropic composite is isotropic in the plane, it is anisotropic in bending because of the directionality of the outer surface fibers. Thus uniaxial bending tests were performed on specimens with surface fibers oriented both perpendicular and parallel to the specimen axis. As expected, the lowest strength values were for specimens with transverse surface fibers. The modulus of rupture, which is the elastically calculated maximum bending stress (assuming an isotropic, homogeneous material), was 1.8 times the UTS at room temperature and 1.2 times the UTS at 120°C. These values will be utilized in the design criteria. The flexural strength at 120°C was 0.44 times that at room temperature. The same factor was obtained in biaxial flexure tests.

Cyclic Fatigue

As was done for the basic property determinations, cyclic fatigue tests were performed at temperatures over the –40°C to 120°C range of interest and in distilled water with a 1000-h presoak and in windshield washer fluid with a 100-h presoak. The setup for performing tests in a fluid is shown in Figure 2. Tensile fatigue curves at room temperature and at 120°C are shown in Figure 3. Similar curves exist for –40°C and 70°C and for the two fluid exposures. Factors relating the failure

Figure 2. Tensile fatigue specimen immersed in fluid during cyclic loading. Extensometer probes pass through the container to measure stiffness loss during cycling.

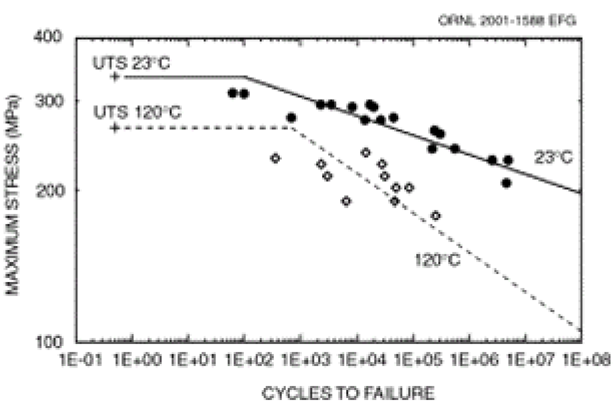

Figure 3. Cyclic-fatigue curves at room temperature and at 120°C in air.

stresses from these curves to those from the room-temperature curve are tabulated in Table 2.

A room-temperature design curve was developed by placing two reduction factors on the room-temperature curve shown in Figure 3. First, a factor of 20 was placed on cycles to failure. Maximum stresses were then multiplied by 0.90, the ratio of minimum to average UTS. The resulting curve and the factors in Table 2 can then be used to determine design-allowable cyclic stresses for other temperatures and environments.

Table 2. Fatigue strength multiplication factors

Environment	Cycles to failure			
	10^2	10^4	10^6	10^8
–40°C air	0.89	1.00	0.96	0.88
70°C air	1.00	0.97	0.89	0.82
120°C air	0.97	0.78	0.62	0.50
Distilled water	0.92	0.91	0.97	1.03
Windshield washer fluid	0.97	0.92	0.95	0.98

Long-Term Creep-Rupture Strength

Long-term creep-deformation and creep-rupture tests are performed in deadweight lever-arm test machines. Most specimens are instrumented with strain gages to measure long-term time-dependent creep. Tensile creep test results have been generated at room temperature and at 120°C and in distilled water and windshield washer fluid. Creep-rupture curves at room temperature and at 120°C are shown in Figure 4. The curve at room temperature is very flat, but there is a significant increase in slope at 120°C.

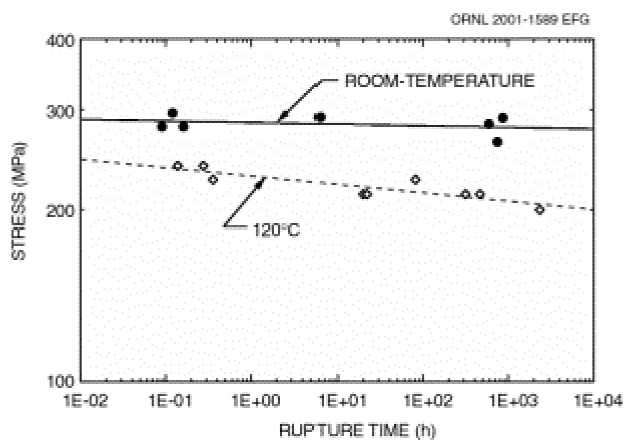

Figure 4. Creep-rupture curves at room temperature and at 120°C in air.

Table 3 is a tabulation of multiplication factors for relating tensile creep-rupture strength at 5000 h and 15 years, for 120°C and for the two fluid environments, to the corresponding strength at room temperature in air. The 5000-h time corresponds to the maximum assumed operating time of a vehicle; 15 years is the assumed design life.

Table 3. Creep-rupture strength multiplication factors

Environment	Rupture time	
	5000 h	15 year
120°C	0.73	0.70[a]
Distilled water	0.98	0.99
Windshield washer fluid	0.95	0.94

[a]This is an unlikely condition because 5000 h is the maximum time a component would be hot.

A room-temperature creep-rupture design curve is derived by taking 80% of the stresses from a minimum curve determined from the room-temperature data in Figure 4. The factors in Table 3 can then be applied to the design curve. This, together with reduction factors for compressive stresses, provides the time-dependent allowable stresses in the durability-based design criteria.

Damage Tolerance

Damage tolerance is defined as a measure of a structure's ability to sustain a level of damage and still safely perform its function. Types of damage of concern in automotive composite structures include low-level impact damage or the presence of a flaw. Both have been investigated for the quasi-isotropic composite.

In the case of impact damage, pendulum drop tests were used to represent such things as tool drops, while an air gun with a small projectile was used to represent the other extreme, such as kickups of roadway debris. Figure 5 is a photograph of the backside of a typical impacted plate specimen. Lower-energy impacts can also produce damage that is not visibly detectable. Ultrasonic C-scans were used to quantify the damage areas, which in turn were correlated with impactor mass and velocity. A single design curve was developed that conservatively predicts damage, including damage in specimens impacted at a temperature of –40°C and in specimens impacted by dropped bricks.

ORNL 2001-1590C EFG

Figure 5. Typical impact damage area on backside of impact plate specimen.

To determine the degradation of strength due to impact damage, special 76.2-mm-wide compression-after-impact specimens were cut from the impacted plate specimens and tested. The results are shown in Figure 6 as the ratio of the degraded strength to that of undamaged specimens. A "design curve," shown in the figure, was developed to conservatively estimate the degradation.

ORNL 2001-1591 EFG

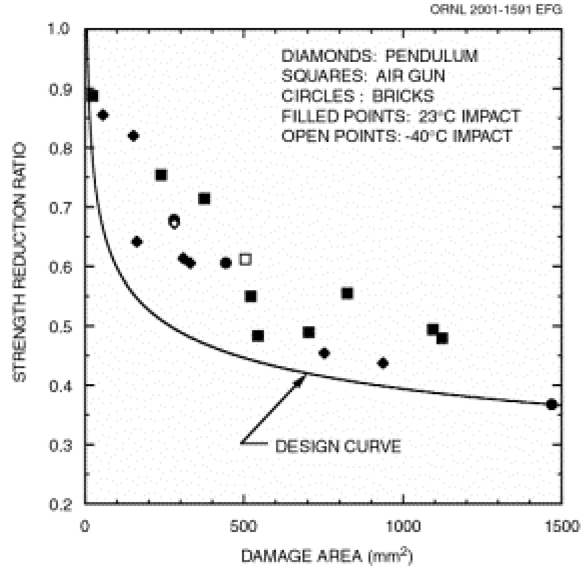

Figure 6. Residual compression-after-impact strength vs impact damage area.

B. Creep, Creep Rupture, and Environment-Induced Degradation of Carbon and Glass-Reinforced Automotive Composites

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Contractor: U.S. Automotive Materials Partnership (cooperative agreement)
Contract No.: DE-FC05-97OR22545

Objectives

- Design and develop low-cost, reliable fixtures and methods for the characterization of creep-rupture behavior of automotive composites with and without environmental exposure. Confirm results generated by the new fixture with those from conventional testing systems.
- Incorporate these fixtures and methods into industry-standard test methods for automotive composites.
- Use results of short-term tests to develop predictive models for lifetime property degradation.
- Investigate the fundamental damage mechanisms in polymer-matrix, E-glass, and carbon-fiber composites as a function of specific varied mechanical loading with concurrent environmental exposure.

OAAT R&D Plan: Task 4; Barriers A, C

Approach

- Design and develop a compact fixture system for creep-rupture testing and confirm its performance.
- Use the new fixture system to develop a creep-rupture database.
- Develop a standard procedure for creep-rupture testing using the new system.
- Develop and verify damage and creep-rupture models for structural automotive composites.

Accomplishments

- Tested the first pre-prototype. This process revealed the necessary concept modifications.
- Developed a new feasible concept using pulleys and a lever arm.
- Fabricated the final prototype.
- Tested the final concept using Bayer programmable powdered performing process (P4) materials.
- Implemented various improvements to the fixture in the areas of loading procedure and load measurement.

Future Direction

- Continue prove-out of fixture system.
- Evaluate varying environments on the fixture system and the test materials.
- Use the new fixture to perform modeling.

Introduction

Because of insufficient information concerning the long-term durability of lightweight composite materials, reliable methods and models requiring relatively short-term tests are essential if composites are to achieve their full potential in the automotive industry. The purpose of this project is to develop simple low-cost fixtures and methods for the creep and creep-rupture characterization of automotive composites and to confirm the in situ creep-test fixture results with those obtained using conventional testing methods.

Initial Design Concept for the Creep-Rupture Fixture

The first pre-prototype included a speed multiplier coupled to a lever arm. Testing of this design concept for the creep-rupture fixture showed difficulty maintaining constant load as strain increased. This prototype emphasized the critical need to minimize friction in a creep-rupture fixture. From these observations, a second design concept, combining two pulleys and one lever arm was developed.

This second concept showed much improvement in the ability to maintain a constant load while a specimen was undergoing creep. This modified concept consisted of a load multiplier composed of two pulleys and a vertical lever arm that fed into the creep fixture. Results from a series of creep tests compared well with accepted values for the tested material. These experimental results were encouraging. Specimens were able to creep, and the results compared well with those of Tulsa University and Oak Ridge National Laboratory (ORNL) models.

This pre-prototype was assembled in order to test the concept of using two pulleys and a lever arm for the load multiplier. However, the use of a lever arm within the load multiplier showed some disadvantages such as travel limitation. Therefore, the lever arm was replaced by a pulley.

Final Concept of the Creep-Rupture Fixture

Based on the results obtained from the first prototypes, some improvements were made. A final concept was produced. This later prototype consists of two pulleys, two sprockets, and a lever arm (Figure 1) and is designed to achieve a ratio of up to 216:1. The actual ratio is currently being measured to account for friction within the system.

A series of tests were performed on Bayer P4 composite specimens. These tests successfully verified the ability to creep and to rupture, therefore proving the feasibility of the concept selected. Furthermore, test data showed encouraging results and compared well with ORNL results generated on standard deadweight machines.

A series of improvements was implemented on the prototype. A clutch was added to adjust the weight height during a test without perturbing the specimen. The loading procedure was also modified to obtain the desired loading rate. Initial tests after these modifications showed the need for further improvement. This is currently being addressed.

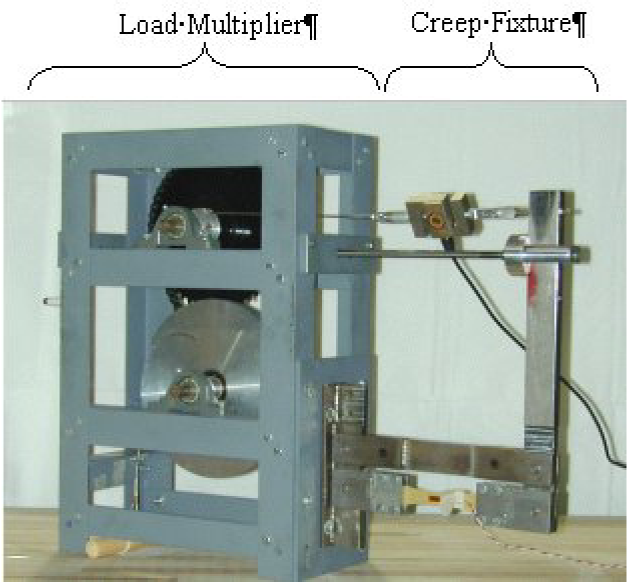

Figure 1. Final prototype.

Finally, in addition to strain, temperature and humidity, load is now integrated in the data acquisition system and can be measured simultaneously (Figure 2).

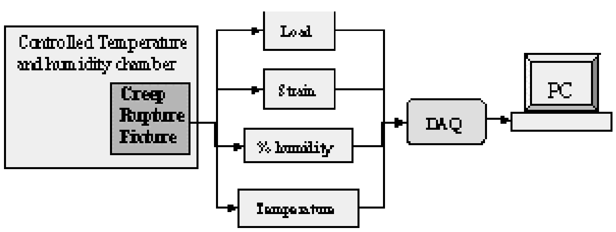

Figure 2. Schematic of the fixture and data acquisition system.

Creep-Rupture Test Fixture Details

As stated in the previous report, to achieve the goal of developing a compact, inexpensive creep-rupture fixture, several design specifications were targeted. These specifications are as follows:

- capability of testing materials such as aerospace-grade carbon-fiber composites
- maximum load application of 5650 lb on a test specimen (assuming an ultimate tensile strength of 90 ksi for a specimen of 0.5×0.125 in. cross section)
- load multiplication of at least 120:1
- geometric envelope: $500 \times 250 \times 500$ mm
- lightweight: less than 45 lb
- ability to test specimens in various environments (water, etc.)
- maintenance of $\geq 95\%$ of the initial load
- inexpensive in comparison with a conventional testing machine: fixture cost less than approximately $4000 without the data acquisition system
- easy conversion to the spring-loaded in situ creep fixture previously developed (patent pending, U.S. Patent Office no. 09/821,280)

A load multiplication of at least 120:1 reduces the required input load to a maximum of no more than 50 lb, for a maximum specimen load of 5650 lb.

The final prototype is composed of two main parts: the fixture and the load multiplier, as shown in Figure 1. One lever arm, two pulleys, and two sprockets enable the fixture to achieve the desired ratio of 180:1. The lever arm is connected to a small pulley located on the upper axle of the load multiplier. This axle is coupled to the lower axle using two sprockets and a chain. A small amount of tension was applied on the chain to reduce friction within the system. A weight hangs from a large pulley next to the small sprocket on the lower axle. Because of the load multiplication of the fixture, the weight moves much farther than any of the other components in the system. For this reason, it is useful to be able to raise the weight without disturbing the other components. To accomplish this, a one-way clutch was installed on the large pulley, as shown in Figure 3.

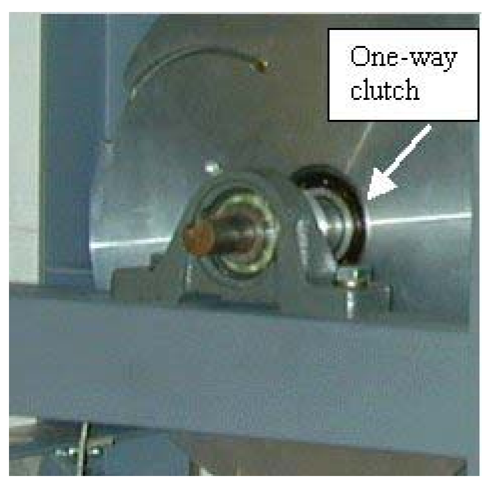

Figure 3. Pulley modification.

A load cell was placed between the lever arm and the cable to record any load variation during a test, as shown in Figure 1. A Labview program was developed to record the load simultaneously with strain, temperature, and humidity during a test in order to assess any environmental influences on the overall fixture. Finally, a first attempt was made to load the specimen at a constant chosen loading rate. A threaded rod was placed horizontally in the load multiplier and touched the lever arm (Figure 4) to stop it from deflecting. To load the specimen, the threaded rod would turn at a constant rate to slowly release the lever arm, thereby placing the specimen in tension. However, high-compression forces on the threaded rod made loading the specimen difficult. In fact, the rod could barely turn. Following this experiment, a second version of that concept was developed and is currently installed on the fixture and ready for testing.

Figure 4. Loading procedure modifications (first attempt).

Test Results Using the Creep-Rupture Test Fixture

Figure 5 shows results of five creep tests on Bayer P4 composite made using the new fixture. Also shown in Figure 5 are the upper and lower bounds of a nonlinear model developed by ORNL for the same P4 material.

Figure 6 shows the curve fit of test data obtained with the new fixture at a stress level of 104 MPa. For this stress level, the ORNL nonlinear model was applied, showing the upper and lower bound for the tested material. With one exception, the strain curve fell within or on the boundary of the model for each

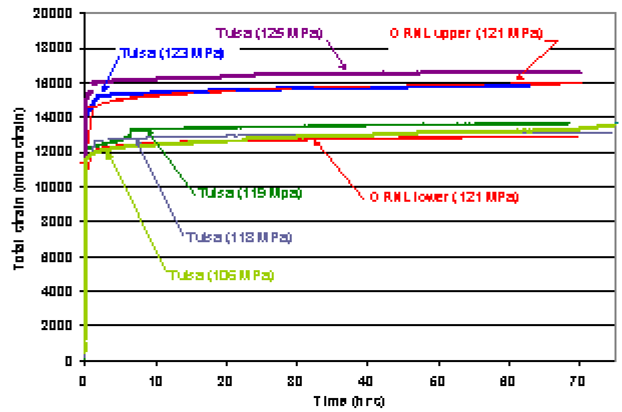

Figure 5. Results of five creep tests conducted at various levels of stress using the new creep-rupture test fixture. These results are compared with upper and lower bounds from an ORNL model.

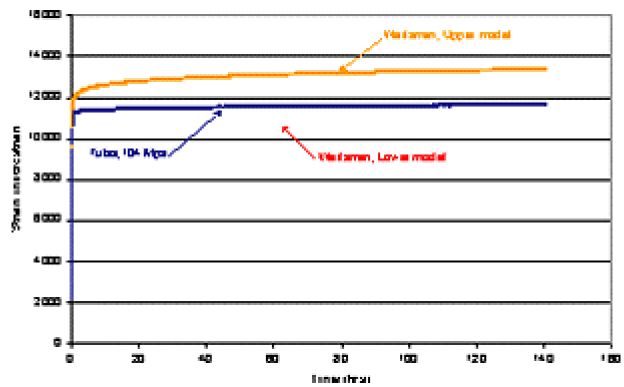

Figure 6. Test data compared with those of a nonlinear model.

test on the fixture. These results were encouraging in comparison with ORNL results.

For each test, the load was recorded to verify the ability of the fixture to maintain load. Figure 7 shows some results obtained from the load measurement. The load loss during a test remains under 5% on average. However, some fluctuations in the load do occur. These are currently being studied.

When examining the data recorded during a typical experiment, it was noted that temperature and humidity varied significantly throughout the test. Figure 8 is an example of these variations. The load fluctuation is being further analyzed to see if temperature and humidity affect the load variation. If the load is affected by the environmental conditions, it will be necessary to better understand the fixture and account for it.

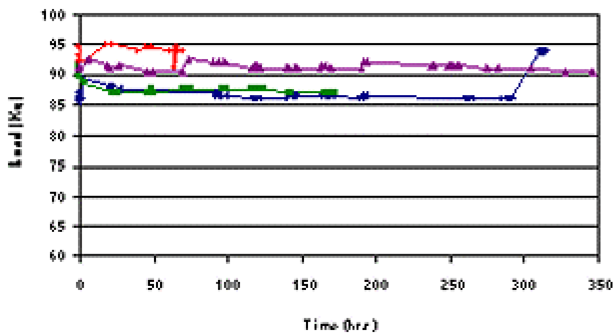

Figure 7. Load fluctuation for four different tests.

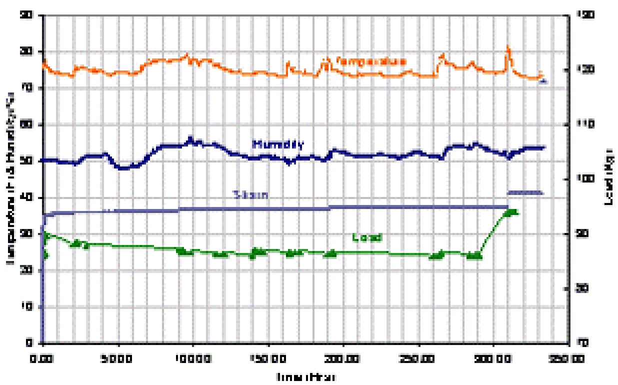

Figure 8. Possible effect of environmental condition on load and strain. (No scale is shown for the strain.)

Ongoing Work

A modification to the loading procedure was recently implemented in the fixture and is ready to be tested. The load multiplication ratio of the creep-rupture test fixture will be experimentally verified. Finally, a series of tests accounting for environmental effects on the creep-rupture fixture system will be performed to better understand how the fixture behaves in different environmental conditions. Because the load varies (i.e., does not only decrease) during a test, it is possible that an increase or decrease in temperature and/or humidity might have an influence on the fixture. It is important to determine what this influence might be in order to account for it when a test is performed. This series of tests will consist of a designed experiment having two parameters: temperature and humidity. For each parameter, two values will be chosen. For example, room and elevated temperatures could be selected, and the humidity levels could be 25% (or 50%) and 90%. Creep tests will be run at each set of conditions, and the results will be used to determine environmental influences on the fixture.

C. NDE Tools for Evaluation of Laser-Welded Metals (Steel and Aluminum)

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Contract No.: DE-FC05-95OR22363

Objectives

- Develop fast, accurate, robust, noncontact nondestructive evaluation (NDE) tools and methodologies to replace current manual destructive testing of laser-welded sheet and structures in zinc-coated steel and aluminum.
- Demonstrate accuracy and repeatability of the technologies developed or applied.
- Eliminate the need for a highly trained/experienced NDE evaluator.

OAAT R&D Plan: Task 6, Barriers A, C, D

Approach

- Phase 1: assess state-of-the-art technologies, down-select, conduct validation testing using fabricated welded coupons, correlate NDE test results with destructive test results, and select technologies.
- Phase 2: develop and build of bench prototypes of the selected NDE methodologies, conduct further validation testing using laser-welded production parts, and correlate NDE test results with destructive test results.

Accomplishments

- Completed preliminary weld flaw characterization of metals from original equipment manufacturers.
- Completed selection of target laser weld application.
- Completed preliminary NDE system functional specifications.
- Completed initial assessment of NDE state-of-the-art technologies.

Introduction

Laser welding has been widely accepted by the automotive industry as an industrial process. Uses range from welding of tailor-welded blanks to transmission components to air-bag inflation modules. The use of lasers continues to increase, with some manufacturers considering the use of lasers for welding of the "body in white."

While laser welding has been accepted by the industry, cost factors have always been an issue, particularly in the area of weld defects. Given the high speed and high volume of laser welding coupled with the relatively small critical flaw size, finding defects can be time-consuming and difficult and, hence, expensive. If not detected before subsequent processing and/or use, these defects could cause failures to occur later during processing or during use. For example, pinhole porosity in a laser-welded tailor blank can cause failure during the stamping operation, which can in turn damage the dies and thus cause the press to be out of operation for a period of time. To prevent this, laser welding needs to be accomplished with no detrimental flaws and be monitored with a high degree of reliability.

There have been a number of systems developed to monitor laser welding systems in real time. Generally, these systems examine the by-products of the laser-to-metal interaction to determine the quality of the weld. These detection methods may include examining the frequency and intensity of the light that is given off and comparing it with a known "acceptable" weld. Most of these monitoring systems use this "training" method as a basis for determining acceptable welds versus welds to be rejected. Generally, these systems interpolate between known parameter variations. Some systems use software that is neural network based rather than function based. As of this writing, these systems have had limited success in the production environment.

The U.S. Automotive Materials Partnership (USAMP) is exploring NDE tools for use upon completion of the weld. Of specific interest is the identification of progressive and emerging technologies in NDE. As part of this effort, the AMD 303 project performed a technology assessment of NDE. From this assessment, specific technology recommendations are being reviewed, with the goal being to have an NDE prototype system ready for installation in a factory setting for in-plant evaluation by the end of FY 2002.

Approved for incorporation into the USAMP/AMD project portfolio in January 2001 and launched in May 2001, this project has two primary investigative missions: (1) evaluate and develop new NDE tools for laser-welded steel (zinc or organically coated) and (2) evaluate and develop new NDE tools for laser-welded aluminum. The investigative strategy is to

- Conduct a comprehensive assessment of existing and emerging NDE tools for steel and aluminum.
- Down-select technologies to the most promising methodologies.
- Conduct validation testing of the selected methodologies against the destructive test method currently in use first for fabricated welded coupons and then production laser-welded parts.
- Demonstrate the selected methodologies through bench prototype systems.

Details of Phase 1

Phase 1 is the assessment of state-of-the-art technologies, down-selection, validation testing using fabricated welded coupons, correlation of NDE test results with destructive test results, and technology selection. As of this writing, the following Phase 1 activities had been accomplished.

The NDE system sought was conceptually defined in terms of its functionality, and the laser weld flaws or defects that it must be able to detect were characterized. Some examples of weld defects can be seen in Figures 1–6.

An initial assessment of NDE technologies was completed. Available commercial off-the-shelf systems were identified in addition to emerging NDE technologies. The goal of this project is to identify techniques that can be applied to steel and aluminum structures. The techniques identified by our initial assessment are applicable to both materials. However, one should bear in mind that procedures or equipment developed for one material would likely require extensive redesign before they could be applied to the other material. For example, ultrasonic transducers developed for steel would need to be completely redesigned for aluminum due

Figure 1. Pinholes in a laser weld.

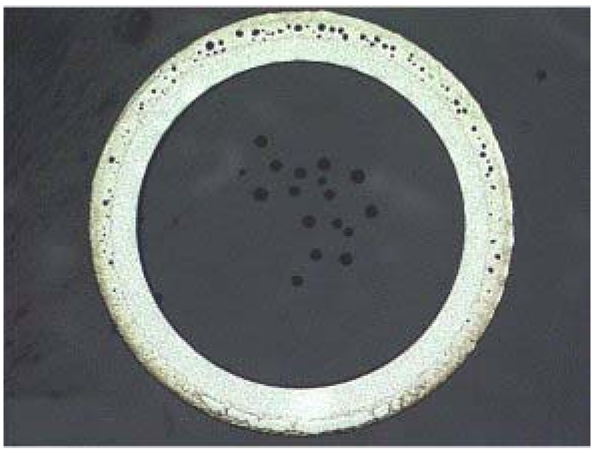

Figure 2. Porosity in cross-sectional sample of a laser weld.

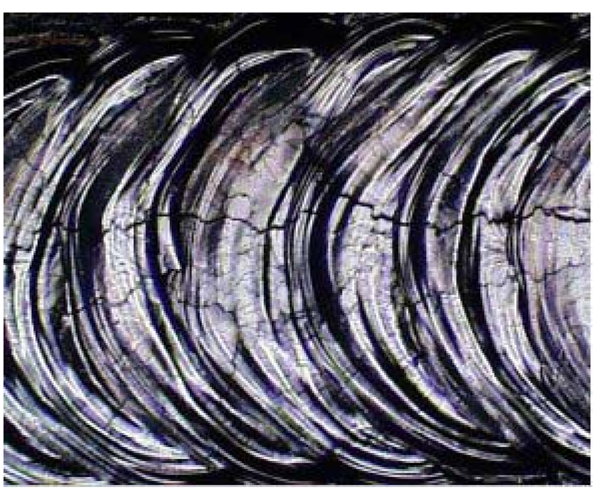

Figure 3. Centerline cracking of laser weld.

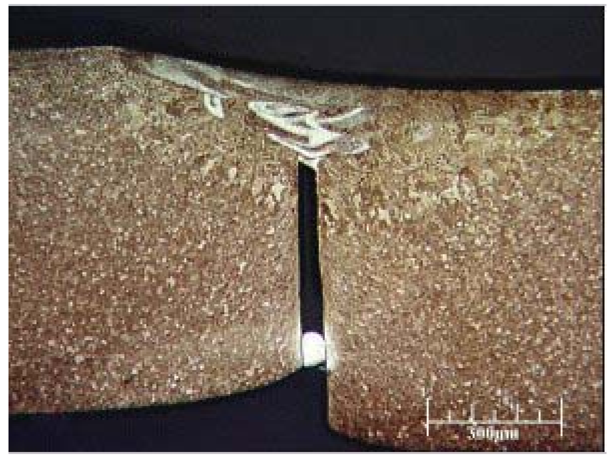

Figure 4. Incomplete penetration in laser weld.

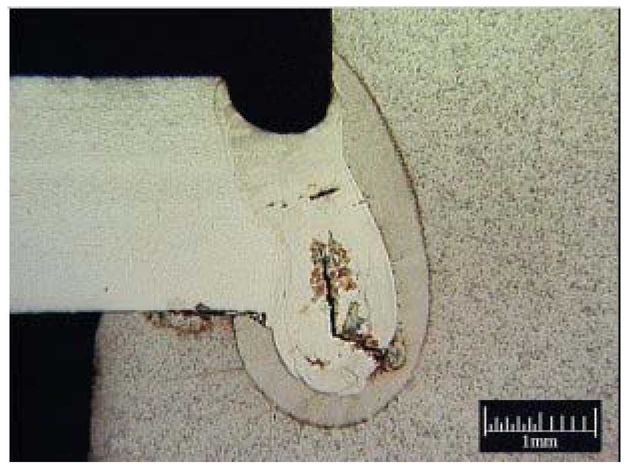

Figure 5. Laser weld with solidification defect at weld centerline.

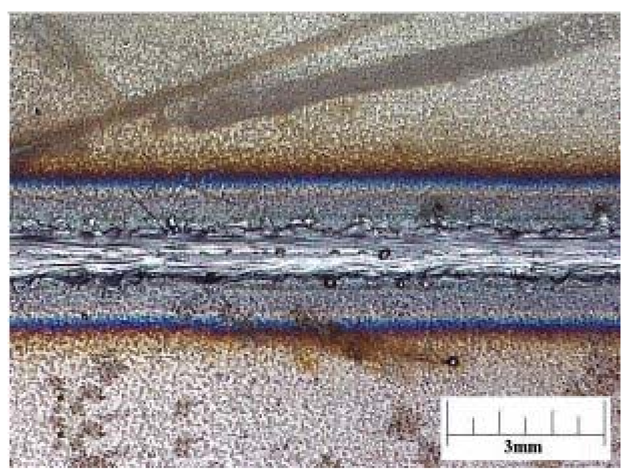

Figure 6. Contaminated laser weld.

to the difference in sound characteristics between the two materials. Another example would be for radiographic testing. Aluminum requires much less radioactive energy than steel to achieve an image of sufficient density for interpretation. Consequently, an X-ray tube suitable for aluminum inspection may not be adequate to penetrate steel to achieve sufficient density in a timely manner. Factors such as these will be given serious consideration as the project progresses toward down-selection of the technologies.

Preliminary findings arising out of our initial assessment have provided further insight into the functionalities required from the eventual NDE prototype system built by this project. The prototype system should be

- Capable of autonomously evaluating and assessing welds in the production system with minimal operator intervention and a high degree of reliability.
- Adaptable to a variety of weld joint geometries and materials.
- Capable of being mounted on a robot arm.
- Capable of detecting and evaluating discontinuities in accordance with a uniform standard.

The AMD 303 project team continues to evaluate the findings arising from the initial technology assessment study, and down-selection of the methodologies to those that merit validation testing is imminent.

D. Modeling of Composite Materials for Energy Absorption

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Contractor: Oak Ridge National Laboratory, Lawrence Livermore National Laboratory
Contract No.: DE-AC05-OR22725, W-7405-ENG

Objective

- Develop analytical and numerical tools that efficiently predict the behavior of carbon-fiber–based composites in vehicular crashworthiness simulations. These predictive tools are intended to decrease the automotive design process time and cost by reducing component testing and to increase the simulation accuracy of carbon-fiber-reinforced structures. The developed tools are used in conjunction with existing crash simulation software.

OAAT R&D Plan: Task 7; Barrier C

Accomplishments

- Developed a nonlinear undulation model, incorporated it into the previously developed triaxially braided constitutive model, and validated it against detailed finite element simulations of the braid's unit cell.
- Developed a constitutive-level enhanced-strain element formulation for the braid model to simulate tensile failure.
- Formulated and implemented a compressive damage surfaces and evolution equations for the braid model.

Future Direction

- Validate the braid material model by simulating previously conducted strip and tube crush experiments.

Introduction

Automotive structures manufactured from carbon-fiber composites (CFCs) offer the potential for significant advantages in weight, durability, design flexibility, and investment cost. Although there is substantial experience with graphite-fiber laminated composites in the aerospace community, there is little knowledge of how CFCs would respond in automotive applications during impact-induced "crash" loading conditions (i.e., "crush"). Furthermore, predictive analytical and numerical tools required to accurately evaluate and design carbon-fiber automotive structures for crush do not exist. This project aims to understand and quantify the basic deformation and failure mechanisms active in carbon-fiber materials during vehicular crush conditions. The project entails modeling, numerical, and experimental components and deals exclusively with automotive-type materials: braided, textile, and chopped random-fiber architectures.

Experimental Investigation of Braided Carbon-Fiber Composites

Basic material tests were performed on the braided CFC at quasi-static and elevated rates to quantify and characterize the material behavior. While the resin demonstrated a marked dependence upon strain rate, little rate dependence was observed in the failure behavior of the $0^o/\pm30^o$ braided CFC tested. In addition to the physical experiments, detailed microscopic examinations were performed on the failed specimens tested at Lawrence Livermore National Laboratory (LLNL), strip specimens crushed by Oak Ridge National Laboratory (ORNL), and automotive tubes dynamically crushed by the Automotive Composites Consortium (ACC). The controlling failure mechanisms appear to be inter-tow rather than intra-tow ones. Results from all of LLNL's traditional and non-traditional material testing and post-mortem examinations of the present CFCs are summarized in ref. 1.

Damage Model for Braided Carbon-Fiber Composites

A framework to numerically simulate the behavior of large-tow triaxially braided CFC was developed in FY 2000.[2] It represents the layered composite material on a layer-by-layer basis and depicts the complex fiber geometry using a simplified unit-cell approach. The material within the braid's unit cell is partitioned into three distant layers, and then each layer is individually homogenized. The behavior within each layer is simulated using a tow-level constitutive model, which represents the basic elastic, plastic, and damage behavior of the straight fibers; and an undulation model, which replicates how the curved fiber regions respond.

In FY 2001, the basic tow-level constitutive model was enhanced in several ways. First, a nonlinear undulation model was formulated and integrated into the tow model. Second, the matrix behavior was expanded to include rate-dependent plastic deformation. Third, compressive and tensile damage mechanisms were derived and incorporated into the constitutive relationship.

A micro-mechanics–based nonlinear undulation model was formulated, implemented, and numerically validated. Detailed elasto-plastic finite element (FE) simulations of the curved tow region (shown in Figure 1) were used to distill the macro undulation response under uni-axial tensile and compressive loadings. The newly developed nonlinear undulation model idealizes this complex behavior. It accounts for limited plastic deformation and stiffening in tension (due to tow straightening). Figure 2 shows the excellent agreement achieved between the macro stress-strain response of the undulation region obtained by the FE simulations

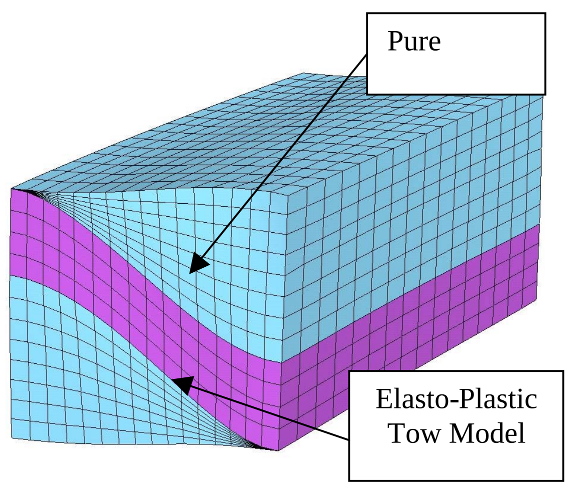

Figure 1. Model of an undulation region in a $0^o/\pm45^o$ triaxially braided carbon fiber composite.

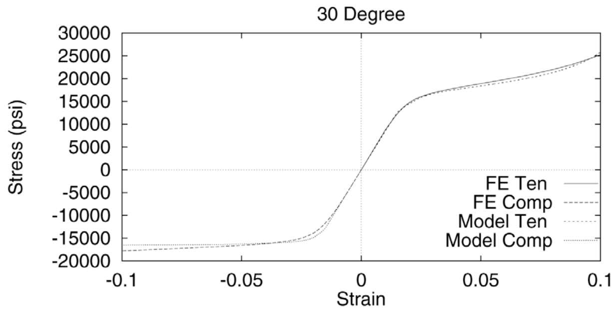

Figure 2. Simulated (finite element) and predicted (model) macro response of an undulation region in a $0^o/\pm30^o$ tri-axially braided carbon fiber composite.

and from the new undulation model for a $0^o/\pm30^o$ braid. The undulation model produced similar behavior and agreement for the other architectures examined.

The nonlinear undulation model is necessary to accurately represent braided CFC under shear and transverse loadings. Figure 3 shows the transverse extensional response obtained from detailed FE simulations of the braid's representative volume element (RVE)[3] and the newly enhanced three-layer model. The agreement between the two results is greatly improved by the nonlinear undulation model, as is the shear response (not shown in figure).

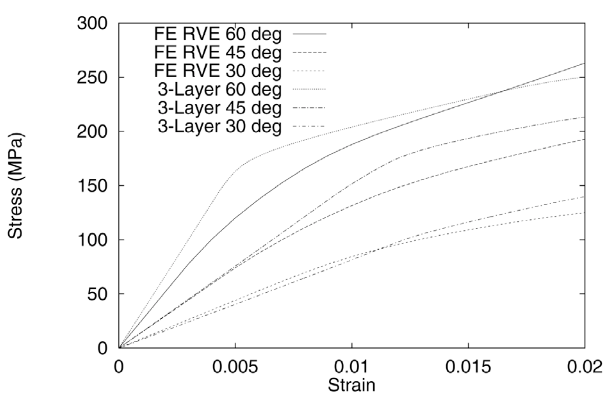

Figure 3. Three-layer model and detailed finite element representative volume element predictions of the transverse extensional response of various triaxially braided carbon fiber composites.

Guided by experimental observations, two compressive and two tensile damage mechanisms were derived and incorporated into the elasto-plastic tow-level constitutive model. They allowed separate formulations to be developed for damage in the fiber and transverse directions, as well as for different mechanisms in tension and compression.

While both tensile and compressive loadings generate inter- and intra-tow micro and macro cracking, commingling of the debris and the undamaged material causes the compressive stress-strain response to reach a non-zero saturation level after the peak load. In the fiber direction, this is simulated by abruptly lowering the maximum stress the fiber can carry to a prescribed level after the (in-plane) axial fiber strain reaches are at a critical level. Similarly, in the transverse direction, the matrix flow strength is reduced to a saturation value after the onset of damage. Motivated by recent triaxial experimental work on uni-directional CFC material, the matrix stresses are used in a J-2 plasticity-like criterion to determine when transverse damage commences.

The post-peak tensile response in each direction is replicated using a pseudo enhanced-strain element formulation.[4] In this approach, the total displacement field is additively decomposed into a material displacement field and a crack displacement field. The material strains, calculated from the material displacements, are used by the tow-level constitutive model to predict the bulk stresses, while the crack displacements and a rigid-softening traction-displacement law replicate how the "crack" evolves with deformation. Auxiliary constraints, which ensure that the bulk stresses and crack tractions are consistent, complete the formulation.

The present tensile damage formulation ensures that a fully damaged material supports no load across the postulated crack and is fully consistent with fracture mechanics concepts. Figure 4 shows

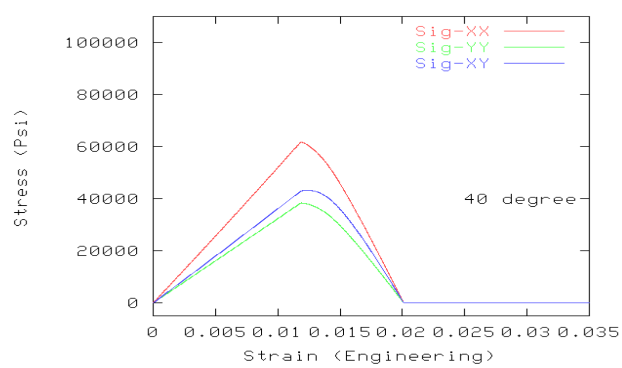

Figure 4. In-plane stress vs applied (x-direction) strain for a uni-directional carbon fiber composite lamina. The fibers are oriented 40° from the x-axis.

the predicted in-plane stresses, as a function of the applied strain, for a uni-directional CFC lamina subjected to in-plane uni-axial straining. A square shell element, aligned with the x-y coordinate system, is used, and the fibers are oriented at 40° from the x-axis. Notice that the resultant stress-strain curves reach a peak and then decay to a zero stress level as desired. Similar behavior was demonstrated for fiber orientations between –180 and 180° (i.e., any angle).

Conclusions

A three-dimensional micro-mechanical–based finite-deformation constitutive model has been development and implemented into the nonlinear, FE code DYNA3D. The model attempts to replicate progressive damage in CFCs and is intended for use in automotive crashworthiness applications. The new undulation component added in FY 2001 allows the model to accurately predict the elasto-plastic pre-damage response of braided CFC, while the tensile and compressive damage mechanisms that were added facilitate modeling of the post-peak material response.

Future Work

With the basic formulations of the braid model complete, the future emphasis will be upon numerically "tuning" the model as necessary and validating it against experimental results, as well as preparing documentation on its theoretical formulation. Using the existing experimental data for a $0^o/\pm30^o$ braid CFC and uni-directional CFC tows, the necessary material properties will be determined and used to validate the model against previously conducted strip tests and drop-tower tube crush tests of $0^o/\pm30^o$ braid architectures. The model will then be used to predict drop-tower tube crush tests of $0^o/\pm45^o$ and $0^o/\pm60^o$ braided CFC architectures.

Budget

FY 2001	Carry-over	New money	Current spent	YTD spent
	−1.5k	325K	169.5k	306k

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3. Zywicz, O'Brien, and Ngyuen, "On the Elasto-Plastic Response of a Large-Tow Triaxial Braided Composite," in *Proceedings of the Fifteenth Annual Meeting of the American Society of Composites*, 2000.

4. Zywicz, "A Constitutive-Level Enhanced-Strain Element Formulation for Simulating Brittle Damage," presented at the workshop Multiscale Modeling of Materials: Strength and Failure, Bodega Bay, Calif., UCRL-145514, 2001.

E. Composite Crash Energy Management

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Contractor: U.S. Automotive Materials Partnership (cooperative agreement) Automotive Composites Consortium Energy Management Working Group
Contract No.: DE- FC05-95OR22363

Objectives

- Experimentally determine the effects of material, design, environment, and loading on macroscopic crash performance to guide design and development of predictive tools.
- Determine the key mechanisms responsible for crash energy absorption and examine microstructural behavior during a crash to direct the development of material models.
- Develop analytical methods for predicting energy absorption and the crash behavior of components and structures.
- Conduct experiments to validate analytical tools and design practices.
- Develop and demonstrate crash design guidelines and practices.
- Develop and support design concepts for application in demonstration projects.
- Experimentally determine the effects of material, design, environment, and loading on carbon fiber–reinforced tubes to guide automotive design and analysis development.
- Conduct microscopic characterization of crush mechanisms for a range of material types and loading to identify the key mechanisms responsible for crash energy absorption and to direct the development of material models.
- Experimentally determine the sensitivity of tube crush performance to impact velocity.
- Determine the mechanisms that control the observed impact velocity effect to support the development of analytical models.
- Experimentally determine the relative energy absorption due to each failure mode of a progressively crushed composite tube and isolate that portion of energy absorption due to friction. Then evaluate differences in friction energy absorption between quasi-static and dynamic crush.

- Experimentally determine strain fields around an advancing damage zone emanating from a notch in a braided carbon fiber composite under biaxial load. Determine the effects of load biaxiality and microstructure on damage accumulation and assess the potential for developing continuum damage mechanics formulations.
- Evaluate the performance of bonded structures under crash loads.
- Examine the influence of bond design concepts, impact velocity, environmental conditions, adhesive type, and other material issues.
- Fabricate new molding tools to produce simulated automotive structures.
- Evaluate the performance of carbon composites and section geometries under angular impact.
- Study the mode of collapse of carbon composite structures subjected to combined loading.
- Establish the operating envelope for structures that will maintain progressive axial crush mode of failure for maximum energy absorption.
- Investigate the viability and crashworthiness of novel sandwich composite concepts for automotive applications.

OAAT R&D Plan: Task 7; Barrier C

Approach

- Conduct experimental projects to enhance understanding of the global and macro influences of major variables on crash performance.
- Create crash intuition, guidelines and rules of thumb, and data for validation of analysis developments.
- Conduct microscopic experimental characterization to define the mechanisms that occur during and as a result of the crash process.

Accomplishments

- Completed material property characterization tests of low-cost carbon fiber composites.
- Completed drop tower tests of 2-in.-square tubes.
- Fabricated additional tubes (2- × 4-in. and 4-in.-square cross section) for axial and off-axis dynamic impact testing.
- Obtained approval of a final design of the intermediate-rate machine. The build phase of the project has been initiated.
- Completed off-angle compression tests on thick unidirectional carbon/vinyl ester specimens to examine failure mechanisms at the fiber/resin interface.
- Performed preliminary testing of 2-in.-square tubes for relative energy absorption and friction energy absorption.
- Conducted off-axis tests to characterize homogenized inelastic stress-strain response of the composites.
- Completed fabrication of test samples used to evaluate the performance of carbon composite and section geometries under angular impact.

Future Direction

- Focus efforts on Partners' needs in the areas of materials, processes, and applications.
- Determine bending and friction energy absorption modes.

Project Accomplishments

Carbon-Reinforced Tube Performance

Experimental determination of the effects of material, design, environment, and loading on carbon fiber reinforced tubes to guide automotive design and analysis development. Conduct microscopic characterization of crush mechanisms for a range of material types and loading to identify the key mechanisms responsible for crash energy absorption and to direct the development of material models.

The material property characterization tests of the low-cost carbon fiber composites have been completed. Materials tested included composites with unidirectional, stitched, and woven fabric as well as biaxial and triaxial braids of Fortafil #556 80k fibers and some Grafil 34-700 12k fibers. Plaques with 40k fiber are being fabricated for additional material property characterization tests. To begin investigating the crush mechanisms associated with dynamic axial crush, drop tower tests of 2-in.-square tubes were completed. Additional tubes of 2×4 in. and 4-in.-square cross section were fabricated during the year for axial and off-axis dynamic impact testing. Fiber architectures were selected based on the 2-in.-square tube results. Dynamic impact tests of these new geometries have been initiated and will be completed in FY 2002. Energy dissipation mechanisms include resin fracture and plastic deformation, fiber fracture and pullout, and inter-ply delamination.

Static vs Dynamic Performance

Experimentally determine the sensitivity of tube crush performance to impact velocity. Determine the mechanisms that control the observed impact velocity effect to support the development of analytical models. This work is supported by an additional university research project and a planned intermediate-rate testing facility at ORNL.

A final design of the intermediate-rate machine has been approved, and the build phase of the project has been initiated. The intermediate rate machine will be delivered to ORNL by mid-2002. Off-angle compression tests on thick unidirectional carbon/vinyl ester specimens have been completed to examine the failure mechanisms at the fiber/resin interface. These tests showed that there is a transition in the failure mode between low- and high-angle specimens. This result is shown to be due to the nonlinear stress-strain behavior of the matrix.

Friction Effects on Crash Performance

Experimentally determine the relative energy absorption due to each failure mode of a progressively crushed composite tube and isolate that portion of energy absorption due to friction. Evaluate differences in friction energy absorption between quasi-static and dynamic crush.

This project was begun in July 2001. Preliminary testing using 2-in.-square tubes of (0/900/90/0) fiberglass reinforced vinyl ester resin composite has been performed. Baseline energy absorption of the tube in axial crush has been measured at 4950 J. Tensile test results have been used to calculate the energy absorption due to tube corner splitting at 98 J or 2% of the total energy absorbed. End-notch shear test results were used to calculate energy absorption due to ply delamination at 377 J or 7.6% of the total energy absorbed. Finally, a shallow-angle (5º) trigger was used, with the total energy absorbed measured at 764 J. The shallow-angle trigger test effectively eliminated the energy absorption due to bending and much of the friction energy absorption. The remaining energy absorption modes, bending and friction, have yet to be measured. A standard tube crush trigger and a shallow-angle tapered trigger coated with a low-friction diamond-like carbon coating will be used in future tube crush tests to further discriminate the energy absorption due to friction. A test method to measure bending energy absorption will be developed.

Biaxial Material Studies

Experimentally determine strain fields around an advancing damage zone emanating from a notch in a braided carbon fiber composite under biaxial load. Determine the effects of load biaxiality and microstructure on damage accumulation. Assess the potential for developing continuum damage mechanics formulations based on biaxial test data for braided carbon composites.

A detailed study of the braided carbon fiber strips from the tubes revealed an understanding of the size effects. It also provided a simple benchmark for verifying detailed nonlinear analyses. Analysis of the detailed 3D model was carried out to comprehend the role played by material nonlinearity and natural geometric imperfections in the braided structure. Off-axis tests to characterize homogenized inelastic stress-strain response of the composites were carried out. Biaxial tests with plaques having the same thickness as the tubes revealed a bending problem. As a result, thicker plates are being used in biaxial testing. Continuum damage mechanics models are being studied to relate to biaxial test data for predictive capability. The project has been extended by 6 months at no cost. Detailed study of the biaxial test data and relationships to Continuum Damage Mechanics will be reported at the end of the project.

Impact Performance of Bonded Structures

Evaluate the performance of bonded structures under crash loads. Examine the influence of bond design concepts, impact velocity, environmental conditions, adhesive type and other material issues. Fabricate new molding tools to produce simulated automotive structures. This is a joint program with the Automotive Composites Consortium (ACC) Joining Work Group and part of Focal Project 3 design studies.

Supplier involvement has been obtained to conduct this effort jointly with Focal Project 3 and ACC Joining. The program has been planned and will begin in FY 2002.

Off-Axis Impact

Evaluate the performance of carbon composites and section geometries under angular impact. Study the mode of collapse of carbon composite structures subjected to combined loading. Establish the operating envelop for structures that will maintain progressive axial crush mode of failure for maximum energy absorption.

The fabrication of the test samples was completed. The tubes are constructed of carbon fibers and Hetron resin. Square and rectangular section tubes (100 mm × 100 mm, 100 mm × 50 mm) will be tested statically and dynamically at impact angles between 0° and 20°. The study will be completed in the first quarter of FY 2002.

Sandwich Structures

To investigate the viability and crashworthiness of novel sandwich composite concepts for automotive applications.

A review of the literature on the subject was conducted. Various topics were highlighted as being areas requiring further research. A specialized sub-team was formed, and additional independent external researchers were identified and contacted for possible collaboration. Various primary and secondary vehicle components were identified for possible sandwich composites employment, as well as potential material candidates for the face sheets and core. An experimental testing program is currently being set up in which different specimen sizes and loading configurations are being considered.

F. Intermediate-Rate Crush Response of Crash Energy Management Structures

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-SL05-01OR22866

Objectives

- Develop a unique characterization facility for controlled, progressive crush testing, at intermediate rates, of automotive materials (polymer composites, high-strength steels, aluminum) and structures.
- Study the deformation and failure mechanisms of automotive materials subjected to crush forces as a function of impact velocity for different velocity profiles, including constant velocity.
- Obtain specific energy absorption and strain data and correlate them with deformation and failure mechanisms to describe the unknown transitional effects from quasi-static to high-rate for polymer composites.
- Characterize the strain rate effects for metallic materials and components.

OAAT R&D Plan: Task 7, 10; Barriers A, B, C

Approach

- Develop a unique high-force (50,000 lb), high-velocity (160 ips) servo-hydraulic to conduct progressive crush tests on tubes and cones at intermediate rates.
- Use high-speed imaging to observe and document deformation and damage mechanism during the crush event.
- Obtain specific-energy absorption as a function of impact velocity.
- Take strain measurements at discrete locations and investigate full-field measurements of strains and curvatures.
- Coordinate polymer composites investigations with the Automotive Composites Consortium (ACC) Energy Management Group via Ari Caliskan of Ford.
- Coordinate steel investigations with the Automotive Steel Partnership via Srdan Simunovic of Oak Ridge National Laboratory.

Accomplishments

- Developed specifications for a high-force (60,000 lb), high-rate (160 ips @ 60,000 lb) hydraulic test machine.
- Developed specifications for the Interactive Dynamic Inverse Model software and its interaction with the Iterative Drive File Generation software for pre-optimization of the servo drive file prior to testing and final optimization via learning algorithms to achieve the specified velocity profile.
- Let a contract to MTS for procurement of the machine in February 2001.
- Completed the mechanical design and released it to manufacturing in July 2001.
- Procured a hydraulic pump for the machine (in-kind contribution from the ACC) and installed it at the National Transportation Research Center.

Future Direction

- Procure a high-speed imaging system in the first quarter of FY 2002.
- Procure a high-speed synchronous data acquisition system during the first quarter of FY 2002.
- Take delivery of the machine early in the second quarter of FY 2002.
- Test programs for steel and composites to be initiated in the third quarter of FY 2002.
- Survey full-field measurement techniques for strain (in-plane) and curvatures (out-of-plane) for applicability and explore them to the extent funds are available.

Background

Polymer matrix composites show great promise for crash energy management in transportation applications. Researchers worldwide have reported higher specific energy absorption values for composites materials with respect to traditional metal counterparts. However, the energy absorption mechanisms are much different than in metals; and relatively little is known regarding the influence of material and structural parameters on the damage mechanisms that develop during a crash event. This knowledge must be gained before composites can be most efficiently utilized for crash management structures.

The ACC's Energy Management Group, with the assistance of national laboratories and selected universities, is undertaking the development of the knowledge base, both analytical and experimental, that will lead to a predictive capability for composites subjected to crash events. Previous ACC research has demonstrated a dramatic difference in the energy absorption of composite tubes when they are tested quasi-statically (2 in./min) and dynamically (3–10 m/s). The difference between quasi-static and dynamic crush is as much as 2 to 1, respectively. However, it is not clear how the transition from one failure mode to another occurs at intermediate rates (0–3 m/s) (Figure 1). Currently, there is no practical opportunity to study the crush response of tubes tested at intermediate rates. Under this project, the lack of test capability at intermediate rates will be addressed to facilitate the acquisition and documentation of data describing the transition from quasi-static to high-rate. Specifications for a high-force (60,000 lb), high-rate (160 ips @ 60,000 lb) hydraulic test machine have been developed (Figure 2).

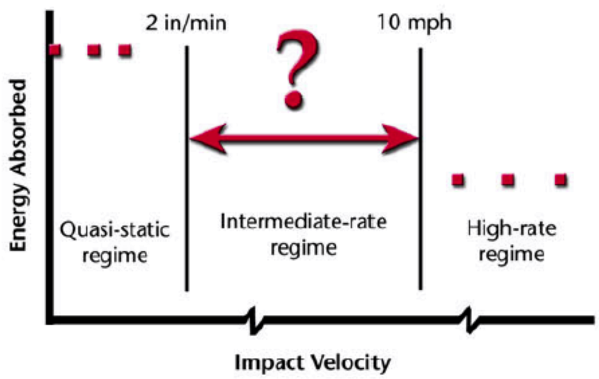

Figure 1. It is unclear how transition from one failure mode to another occurs at intermediate crash rates.

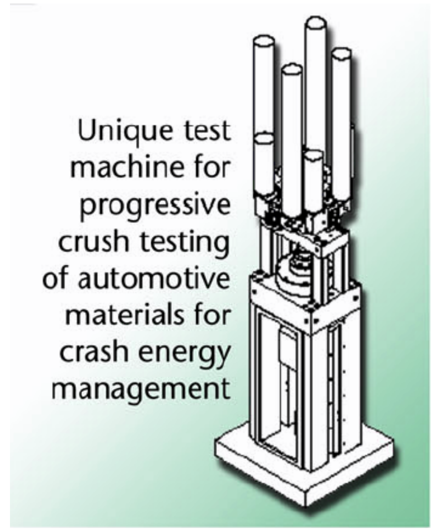

Figure 2. This high-force, high-velocity servo-hydraulic will be used to conduct progressive crush tests on tubes and cones at intermediate rates.

G. Long-Life Electrodes for Resistance Spot Welding of Aluminum Sheet Alloys and Coated High-Strength Steel Sheets

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Survey the currently available technology for achieving long electrode life.
- Comparatively test a broad selection of existing and developmental electrode technologies that have technical merit.
- Confirm the test results through electrode metallography and computer modeling.
- Evaluate the best practices through beta-site testing of automobile applications.

OAAT R&D Plan: Task 6; Barriers A, C

Approach

- Conduct benchmarking (Phase 1) in order to produce a state-of-the-art report on electrode wear. This will be accomplished through a review of open literature and available corporate literature, along with interviews of industry experts.
- Screen candidate electrode technologies, conduct in-depth testing of electrodes, and perform beta testing of selected electrode technologies in an automotive production environment (Phase 2).
- Conduct computer modeling of the electrode metallurgical and mechanical changes that occur as a result of electrode wear (Phase 3). The models will be used to define the mechanism(s) of electrode wear when resistance spot welding aluminum and high-strength coated steels.

Accomplishments

- Began the process of forming the project team.
- Obtained approval of the work plan.

- Began to conduct interviews with industry experts and to perform a review of the literature relating to electrode wear.
- Obtained approval for Oak Ridge National Laboratory (ORNL) to conduct computer modeling.

Introduction

Resistance spot welding has been widely adopted by the automotive industry because of its relatively low capital and operating costs and its potential for high production rates. However, electrode wear of coated steels and aluminum has been a continuing and significant problem. Electrode wear adversely affects the cost and productivity of automotive assembly welding because it reduces the reliability and robustness of the weld product. This requires additional inspection and mandates more strict control of the welding parameters to ensure quality. Ultimately, worn electrodes can result in deteriorated weld performance. Consequently, the production manufacturing cost savings of doubling the electrode life are substantial.

As technology has developed over years of use, few engineering solutions have been successfully introduced into the manufacturing process to manage the issues resulting from electrode wear. Weld current steppers and electrode cap dressers have been used for many years to minimize the effects of electrode wear, but these techniques do not address the underlying causes of electrode degradation. More-recent efforts to remedy electrode wear have resulted in the development of new electrodes, equipment, and sheet metal surfaces. Innovations in electrode technology include new compositions, inserts on the electrode face, coated electrodes, new manufacturing processes, and new electrode geometries. The objective evaluation of these types of existing and developmental technologies is the subject of the present initiative.

Work Team Development

A work team is being assembled, with a representative from DaimlerChrysler leading the team. In January 2001, representatives from Geneal Motors (GM), Ford, and DaimlerChrysler met to discuss the program. They selected Edison Welding Institute (EWI) to manage the program and perform a benchmark study (Phase 1). Brush-Wellman, a copper producer, and electrode producers Nippert Company and Huys Industries are currently part of the program as well. Additional electrode and copper producers are being sought. ORNL will be performing the computer modeling of the electrodes. Representatives from both steel and aluminum producers may also be invited to participate.

Work Plan Approval

The team was presented with a work plan, which has been approved by GM, Ford, and DaimlerChrysler. This plan calls for three phases, which are illustrated in Figure 1. Phase 1 is a comprehensive benchmarking study that will include a literature review, interviews with industry experts, a review of the available internal corporate documents and data, and identification of commercial and developmental technology for inclusion in Phase 2. This activity will be completed by December 2001 and will be the foundation for the next two phases.

Phases 2 and 3 will run concurrently and will test those electrode geometries and compositions that could technically provide significant improvements in electrode life. These technologies will be selected via analysis of the information made available during the benchmarking study. Phases 2 and 3 include activities such as electrode metallography, computer modeling, and beta-site testing in an effort to provide both practical and technical confidence for successful implementation in automotive production. The anticipated completion date of the initiative is the 4th quarter of FY 2003.

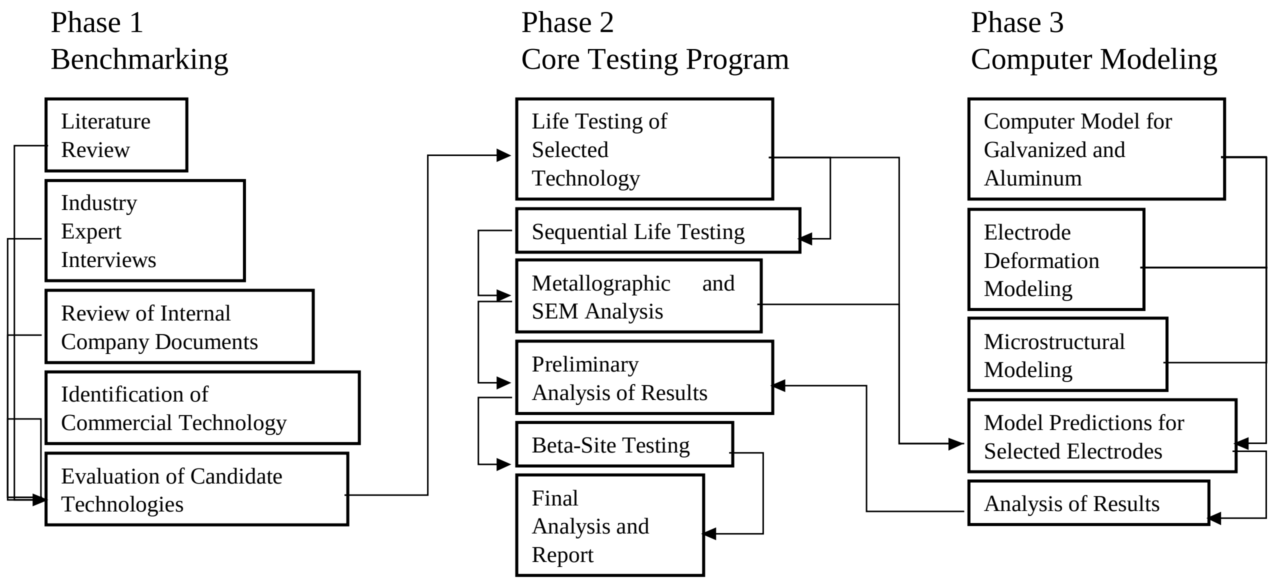

Figure 1. Long electrode life initiative.

Phase 1—State-of-the-Art Benchmarking Study

The objectives of Phase 1 are to collect all pertinent data and technologies for consideration in an evaluation of technologies that improve electrode wear. The open literature will be reviewed to identify the current theories of electrode wear mechanisms and phenomena. Experts in the automobile industry will be interviewed to determine the issues of electrode wear in practice, the solutions, and the production constraints within which potential solutions must work.

Data on electrode wear phenomena and potential solutions to electrode wear will be collected during these interviews. If made available, internal corporate documents will be reviewed. Both corporate test results and nonproprietary production welding practices will also be summarized.

The existing commercial technologies suggesting improvement in electrode wear will be surveyed in an attempt to obtain test results, determine the mechanism of electrode wear improvement, and assess implementation requirements.

The information from the above tasks will be reviewed, and specific candidate technologies will be recommended for inclusion in Phase 2. The minimum requirements for improved electrode wear will be established.

Phase 2—Core Testing Program

Electrode life testing using standard techniques will be performed to assess the electrode life of competing technologies. The results will be evaluated against the minimum standards established in Phase 1. Those technologies that appear to substantially improve electrode life will be evaluated further. The data from this task will also be used as input to the computer modeling work in Phase 3.

For selected technologies that perform at or above the selected minimum expectations, interrupted electrode life tests will be performed. Standard life testing will be performed using identical procedures but will be interrupted at 5, 10, 25, 50, and 100 welds, etc. At each break point, the electrodes will be permanently removed from the holders and electrode wear measurements will be assessed. These electrodes will be metallographically sectioned and compared using optical and scanning electron microscopy (SEM) techniques to assess the stages of electrode wear. The data will be analyzed to determine the nature of electrode life improvement. These results will be compared with those from the computer modeling in Phase 3.

Phase 3—Computer Modeling of Electrode Wear

An existing computer model of electrode performance will be adapted for use with galvanized steel and aluminum. This model will be enhanced to provide for both the global and electrode-sheet deformation events central to electrode wear phenomena. The model will be further enhanced to include the microstructural changes that occur between zinc and copper during the electrode wear phenomena and will be used to predict the deformations and alloy layer development for the selected electrodes. These predictions will then be compared with the metallographic results. After assessing the validity of the model, it will be used to better understand the causes of electrode wear and interpret the mechanism of electrode life improvement as evidenced by the results. These results will be used to help select one or more beta-testing candidate technologies.

Project Summary and Beta-Site Testing

The data from the electrode life and metallographic testing, as well as the computer modeling results from Phase 3, will be analyzed. The technologies offering the best opportunity for improved electrode life when welding aluminum and galvanized steel will be selected for beta-site testing. The tests of selected technologies will be performed in an automotive production application(s) on aluminum and coated high-strength steels. The existing operating conditions and electrode life will be assessed before implementation of the new technology.

Summary of Current Ongoing Activities

Interviews with industry experts are presently under way to better define electrode wear and how it occurs in practice, to obtain input on the factors contributing to electrode wear, and to identify potential existing and developmental technologies that may help to avoid the effects of electrode wear. At this time, 25 interviews have taken place and more than 15 are scheduled.

The level of activity with regard to the literature review is accelerating. Papers are being received, and a few have been reviewed.

H. Plasma Arc Welding of Lightweight Materials

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Develop and verify the joining technology required for the joining of lightweight materials (aluminum and magnesium) utilizing plasma arc spotwelding technology.
- Develop the necessary weld parameters and techniques required for a robust joining process.
- Develop guidelines for testing mechanical properties for this new technology that are appropriate for the various anticipated applications.

OAAT R&D Plan: Task 6, 13; Barriers C, D

Approach

This project has been divided into three key phases: testing, analysis and summary.

- Phase I: produce approximately 5000 material coupons that will be used for tensile, shear, and metallurgical analysis and testing.
- Phase II: modify the material alloys based on Phase I results.
- Phase III: produce guidelines for welding parameters and quality control.

Accomplishments

- Formed project team and conducted official meetings.
- Finalized test matrix (material selections and combinations).
- Obtained material from ALCAN to produce coupons for testing.
- Secured agreement from ALCAN and ALCOA to supply required material for the program.
- Secured ABB robots for the program [accomplished by General Motors (GM)].
- Designed and fabricated fixture to ensure uniform coupon production.
- Redesigned plasma arc torch (reduced tip size) to allow for reduction in flange size.

Future Direction

- Proceed with Phase I testing.
- Conduct Phase II testing based on Phase I results.
- Document and report results of the research.

Introduction

The current joining technology for aluminum relies heavily on resistance spotwelding, which at best is a marginal process. High maintenance and tip wear continues to be a major concern. Rivets and or mechanical clinching are costly alternatives that require high capital investment. The viability of plasma arc spotwelds has entered the picture through the efforts of Arc Kinetics, Ltd. Arc Kinetics is the leading developer of the plasma arc welding process in both steel and lightweight materials. The company developed its original process for single-sided plasma arc spotwelding for Jaguar Motor Cars for Jaguar's sheet metal floor pan assembly, which was not accessible with conventional resistance spotwelding equipment. In a separate effort, the company developed a process called aluminum plasma arc welding (APAW). By combining single-sided spotwelds and APAW, Arc Kinetics developed a process that demonstrates excellent potential for the joining of lightweight materials, aluminum and magnesium.

Team Development

The project team was formed with a representative from DaimlerChrysler as the leader. Representatives from GM, Ford, DaimlerChrysler, and Arc Kinetics met to discuss the program in February 2001. They selected a previous team member, F. G. LaManna, (LaManna Enterprises, L.L.C.) to manage the program.

Additional team members were solicited from the automotive industry. Rite-on Industries, ABB Robots, ALCAN, and ALCOA joined the team and are currently a part of the program. Additional suppliers may be also be invited to participate in the program.

Work Matrix Approval

A work matrix (material) presented to the team has been approved by GM, Ford, and DaimlerChrysler. This plan calls for initially two phases, which require producing material coupons for various tests: tensile, shear, and metallurgical analysis/testing.

Evaluation of these test results will determine the next phases of the program. Phase II will be a repetition of Phase I with modification to the material alloys based on Phase I results. Phase III will be the reporting phase of the program. Completion of the initiative is expected in the fourth quarter of 2002.

Phases I and II—Base Testing Programs

Coupon testing will consist of tensile, shear, and metallurgical analysis. Coupons will be evaluated to ensure weld integrity and establish equipment parameters to be used in future applications.

Project Summary

The data from the testing in Phases I and II will be compiled and used to produce guidelines for welding parameters and quality.

I. Performance Evaluation and Durability Prediction of Dissimilar Material Hybrid Joints

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objective

- Develop new experimental methods and analysis techniques to enable the use of hybrid joining as a viable attachment technology in automotive structures. The work includes evaluating the mechanical behavior of composite/metal joints assembled using a variety of hybrid joining methods and quantifying the resultant damage mechanisms under environmental exposures, including temperature extremes and automotive fluids, for the ultimate development of practical modeling techniques that offer global predictions for joint durability.

OAAT R&D Plan: Task 6; Barriers C, D

Approach

- Characterize the structural hybrid joint under quasi-static load conditions.
- Characterize the response to fatigue, creep, and environmental exposures.
- Conduct predictive analysis.

Accomplishments

- Identified a candidate hybrid joint with the Partnership for a New Generation of Vehicles (PNGV) Joining Task Force.
- Procured mechanical fasteners (rivets), steel and composite substrates, and adhesive system for manufacture of the candidate hybrid joint.
- Completed the fabrication of test specimens and mechanical characterization (tensile and shear properties) of the joint adhesive in conjunction with the University of Michigan and the Automotive Composites Consortium (ACC).

- Developed basic mechanics and finite element models to estimate stress fields in the hat-section geometry.
- Completed the mechanical test set-up and associated instrumentation for the first series of structural hat-section tests.

Future Direction

- Complete mechanical testing to characterize structural behavior under various in-service load conditions and environmental conditions.
- Identify failure modes and damage accumulation and progression and correlate with measurable physical response.
- Develop realistic, practical models to predict joint durability and/or failure under a variety of load conditions and environmental exposures.

Introduction

Automobile weight can be reduced and fuel efficiency increased without compromising structural integrity or utility by incorporating innovative designs that strategically utilize modern lightweight materials—such as polymeric composites—in conjunction with traditional structural materials such as aluminum, magnesium, and steel. Despite the advantages associated with dissimilar or hybrid material systems, there is a reluctance to adopt them for primary structural applications. In part, this reluctance can be attributed to the limited knowledge of joining techniques for such disparate materials. Traditional fastening methods such as welding, riveting, screw type fasteners, and bolted joints may not be appropriate.

One solution to this problem is the use of hybrid joining techniques in which a combination of two or more fastening methods is employed to attach similar or dissimilar materials. One example is a mechanically fastened joint (i.e., bolted or riveted) that is also bonded with adhesive. These types of joints could provide a compromise between using a familiar mechanical attachment that has proven reliability, and reducing problematic issues such as stress concentrations and crack nucleation sites introduced by using mechanical fasteners with polymeric composites.

The use of hybrid joining could also lead to other benefits such as increased joint rigidity, which contributes to overall stiffness gains and reduced vehicle mass. Additionally, adhesives used in conjunction with mechanical fasteners could significantly reduce stress concentrations, which serve as locations for crack starters. Hybrid joining methods can also provide additional joint continuity to allow increased spacing between fasteners or welds.

Although numerous benefits are derived from using hybrid joining techniques, and the joining of dissimilar materials is becoming a reality, little or no practical information is available concerning the performance and durability of hybrid joints. Therefore, this project has taken on the task of developing new technologies to quantify joint toughness and predict long-term durability. This will necessitate identifying and developing an understanding of key issues associated with hybrid joint performance, such as creep, fatigue, and the effects of environmental exposure.

Candidate Hybrid Joint Selection

To initiate this study, it was necessary to choose a candidate hybrid joint representative of those typically encountered in automobiles. Because of their wide applicability in automotive structures, several combinations of hat-section geometries were considered. Hat sections can be incorporated into a variety of generic automotive structural components, such as crush-tubes or frame rails, when they are bonded and mechanically fastened to other geometries. For the current study, the Joining Task Force selected a composite hat-section bonded and riveted to a steel base as shown in Figure 1. This selection was made on the basis of general applicability to a

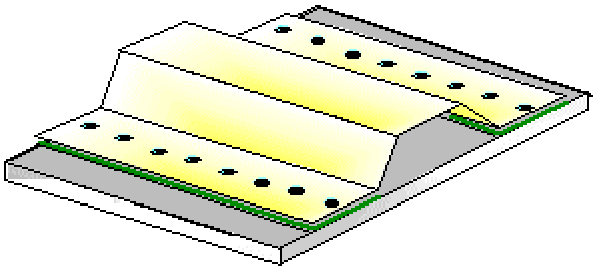

Figure 1. Candidate hybrid joint consisting of a composite hat-section bonded and riveted to a steel plate.

variety of automobile structural components. Members of the Joining Task Force identified industry partners for sources for the steel, rivets, composite hat section, and adhesive.

Adhesive Characterization

Characterization of the adhesive for the hybrid joint was carried out at Oak Ridge National Laboratory (ORNL) and at the University of Michigan in a joint project sponsored by the ACC. The motivation for this collaboration was a mutual need of the two programs for material properties and the desire of the Hybrid Joining Project to obtain independent data from a second source.

Testing consisted of flat coupon tensile tests for axial stiffness, strength, and stress-strain behavior and torsional testing of cylindrical specimens for shear properties. Failed tensile specimens are shown in Figure 2, where typical

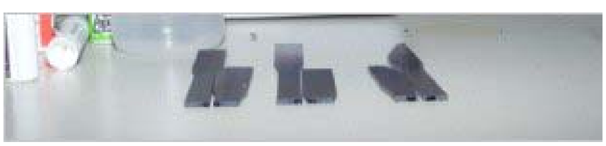

Figure 2. Cast-panel tensile dog-bone specimens revealed the presence of trapped gas bubbles.

bubbles are observed on the fracture surface as a result of gases trapped during casting of the flat specimens. A torsional specimen prior to testing is shown in Figure 3. Note that all cylindrical specimens were manufactured using curing procedures newly developed at ORNL to prevent exothermic reactions (see Figures 4 and 5), which result in large material porosity from combustion of the adhesive. Additionally, it was necessary to

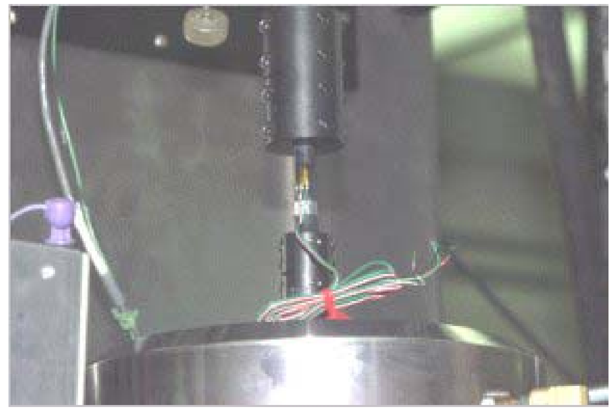

Figure 3. Torsion test setup in the AT machine (note: the setup used a 600 in.-lb torque cell).

Figure 4. Exotherm resulting in obvious porosity in the first exploratory specimen yielded much lower torsional strength.

Figure 5. Improved cure with “pre-gel” of next six specimens resulted in no noticeable porosity and significant improvement in specimen torsional strength.

establish new test procedures and apparati to accommodate the cylindrical specimens.

Typical results for the tensile and shear tests are plotted in Figures 6 and 7. Reduction of the data indicated that consistent stiffness and strength results were obtained between the tests carried out at ORNL and the University of Michigan; the consistent results indicated that specimen preparation and testing were comparable. In addition, calculation of Poisson's ratio from the tensile and shear data produced results typical of the adhesive system.

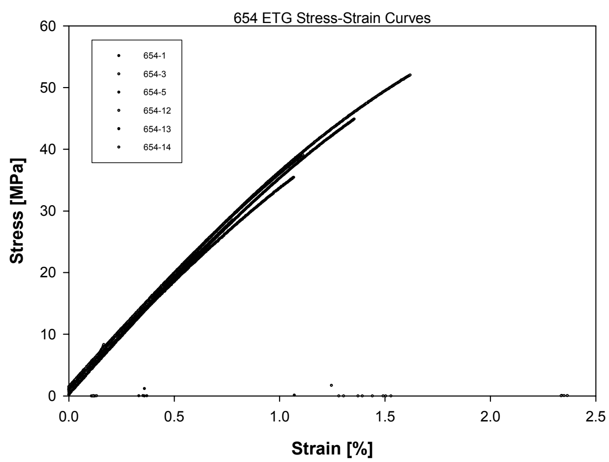

Figure 6. Stress–strain curve for the flat adhesive tensile specimens.

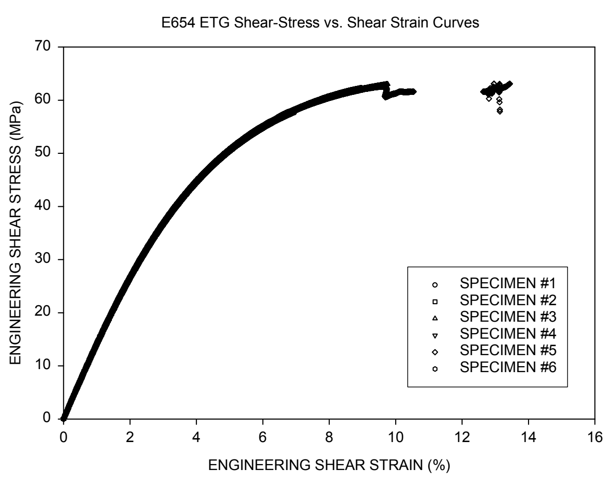

Figure 7. Shear stress vs shear strain test results for the torsional adhesive specimens.

Modeling Efforts

A primary long-term goal of this project is to predict the durability of the hybrid joint structure under load, using detailed models that account for interactions between the dissimilar materials, adhesive bonds, and mechanical fasteners. However, we decided to initiate the analysis for this project by constructing simpler models to estimate stresses prior to running the first tests. The results from this work can be used to determine the locations of stress concentrations and likely damage initiation sites. Additionally, this analysis will provide some indications of the types of failure modes (e.g., crack growth, material yielding) that can be anticipated during the initial testing.

The first of these models is a simple composite beam analysis based on a two-dimensional mechanics-of-materials approach, which accounts for the dissimilar material properties in the response of the structure but ignores the adhesive and the rivets. The interface between the disparate materials is considered a rigid joint. The second model is a three-dimensional finite element analysis that includes the adhesive layer but excludes the rivets. This model provides details of the stress field throughout the structure, especially variations of the shear stresses, which are not addressed in the first model.

These models will serve as an important starting point for the long-term modeling goals projected to be initiated in the final years of the research program. A typical plot of shearing stresses obtained from the finite element model is shown in Figure 8.

Mechanical Test Set-Up

To carry out the hybrid joint structural tests, it was necessary to modify the load train fixtures to accommodate the newly developed rail geometry of the stiffened hat-joint specimen. Additionally, the data acquisition system (computer and associated instrumentation) was updated to the current version of LabView (a data acquisition and control graphical programming language). All data acquisition and control software is being re-compiled to be consistent with the latest versions of the programming software.

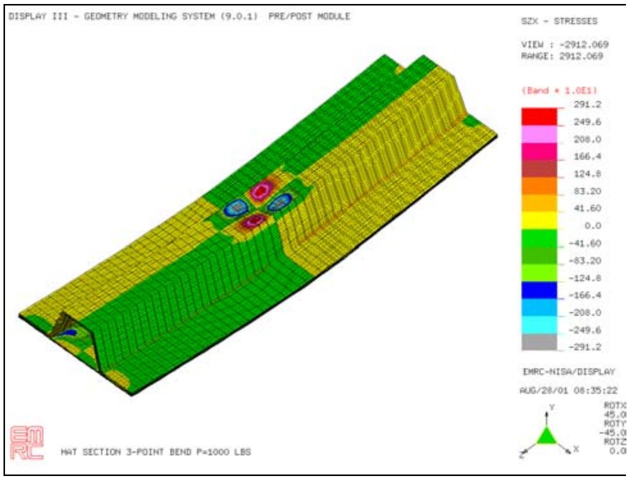

Figure 8. Preliminary finite element model results: shear stress for the hybrid joint hat-section specimen.

Preliminary testing within the linear region of a hat section without the steel base was conducted to estimate the required load capacity. Based on the mechanical response of the specimen, this exploratory test warranted installation of a larger load cell (10 kip, 2.2 kN) to achieve the desired levels of displacement for the upcoming tests.

Upon the arrival of the steel being supplied, expected in the near future, all aspects of the mechanical test system will be ready and will be ebugged to begin the first exploratory tests with the new specimen.

Conclusions

Although this project was started relatively late in the fiscal year, a proficient start has yielded excellent progress and all planned tasks were accomplished on schedule. Specifically, the identification of a candidate joint meaningful to automobile industry partners has been selected, preliminary testing of the adhesive system has been completed, and preliminary modeling of the hybrid joint has provided the necessary information to proceed with the static tests slated to start during the first quarter of FY 2002. Additionally, all necessary modifications to the test apparatus identified at this time have been completed. With the groundwork now completed, it is estimated that there will be little or no delay in accomplishing the next year's objectives.

J. Joining of Dissimilar Metals for Automotive Applications: From Process to Performance

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Contractor: Pacific Northwest National Laboratory
Contract No.: DE-AC06-76RL01830

Objectives

- Develop and evaluate different joining technologies for dissimilar aluminum alloys and for joining aluminum to steel.
- Characterize the performance of these joints.
- Develop a unified modeling procedure to represent these joints in vehicle structural simulation. The steel materials include mild, high-strength low-alloy (HSLA), and dual-phase steels.

OAAT R&D Plan: Task 6; Barriers C, D

Approach

- Further develop and/or enhance self-piercing rivets and resistance spot welding, with and without adhesives, for joining dissimilar metals.
- Develop a database for the static, dynamic, fatigue, and corrosion behavior for dissimilar material joints consisting of different material selections and different joining techniques.
- Incorporate and represent the joint performance data into current computer-aided engineering (CAE) codes for evaluation of the impact and fatigue performance of joint components.
- Develop design guidelines in the form of tables and charts for use in joint structural and crash designs.

Accomplishments

- Selected two promising joining techniques for joining dissimilar metals.
- Experimentally and numerically investigated the effects of specimen design on reported joint performance.

Future Direction

- Join dissimilar aluminum alloys (AA5182 and AA6111) and steel (SAE 1010, HSLA, dual-phase) using self-piercing rivets and resistance spot welding with and without adhesives.
- Develop interlayer material for the use in spot welding of dissimilar metals.

- Conduct mechanical and microstructural evaluation of dissimilar metal joints subjected to static, dynamic, and fatigue loads. In addition, investigate the effect of structural adhesives, temperature, and loading rates.
- Develop joint failure criteria for CAE safety and fatigue simulations based on the performance database achieved in the experiments.
- Develop design guidelines in the form of a design space that contains strength, energy absorption, normalized dimensional parameters, material combinations, and so on.

Introduction

This project, which started in April 2001, is a collaborative effort among DOE, Pacific Northwest National Laboratory (PNNL), and the metals joining team of the U.S. Council on Automotive Research.

The automotive industry envisions that the optimized vehicle, in terms of performance and cost, can be achieved only by using different materials at different vehicle locations to utilize the materials' functionalities to the fullest extent. Currently, aluminum and steel are the most important construction materials for the mass production of automotive structures. High-volume non-steel joining is a significant new problem to the industry. For joining dissimilar aluminum alloys, the leading candidate joining methods are spot welding and self-piercing rivets, with or without adhesives. The major concerns regarding aluminum spot welding are its high energy consumption, low electrode life, and structural performance concerns related to weld porosity. The technology for joining aluminum to steel with which the industry is currently comfortable is self-piercing rivets (with and without adhesives). However, there are a number of barriers to the widespread exploitation and high-volume production of the riveting technology, one of which is limited performance data relative to automotive applications.

On the other hand, in order to shorten the vehicle development cycle, more and more CAE analyses are performed before the actual prototype is built. The question CAE engineers ask most often is how to represent the structural joints in crash simulation and fatigue simulation. Currently, there is no unified approach to representing structural joints that works for different material combinations under multi-axial loading. This is particularly true for dissimilar material joints, where even the basic performance information on the joint coupon level does not exist.

Joint Selection and Development

The U.S. Automotive Materials Partnership (USAMP) joining team and PNNL staff selected the most relevant material set and material gage based upon the most relevant vehicle applications. The material set used to fabricate dissimilar metal joints includes

- steel (mild steel: SAE 1010; HSLA; ultra-high strength steel: dual-phase steel)
- aluminum (AA 5182 and 6111)
- combinations of aluminum and steel

The type of adhesives to be used in the joint fabrication has also been identified by the project team. During the first phase of the program, Dow's Betamate 4601 and Eftec's WC2309 will be examined. These adhesives will be applied in addition to the structural joint (weld or rivet); therefore, the role of the adhesives on the impact, fatigue, and corrosion performance of the joint can be evaluated.

Joint Performance Characterization

It is well known in the automotive welding community that joint performance can be greatly influenced by different attributes of the joint sample design. However, the lack of a unified joint design standard—including the American Society for Testing and Materials (ASTM), American Welding Society, and International Institute of Welding (IIW)—has hampered the integration of joint performance data obtained from different sources. To overcome the joint design issues and ensure the validity of the performance data obtained during the later phase of the program, a coupon design sensitivity study is being performed for tensile shear specimens of both steel spot welds and aluminum self-piercing rivets.

Coupon design factors such as coupon width, sheet overlap, and the effect of forming a C-channel on the coupon flange have been investigated experimentally and numerically. Since some of the joining team members are also members of the Auto/Steel Partnership (A/SP) task force on high-strength steel joint performance, and since A/SP has used the C-channeled tensile shear design previously, coupons with C-channels are also included in our initial coupon design table. For example, Figure 1 shows the measured quasi-static load versus displacement curves for 1.0-mm SAE1008 tensile shear spot-welded coupons with different coupon widths and C-channels. The weld diameter used is 5.0 mm.

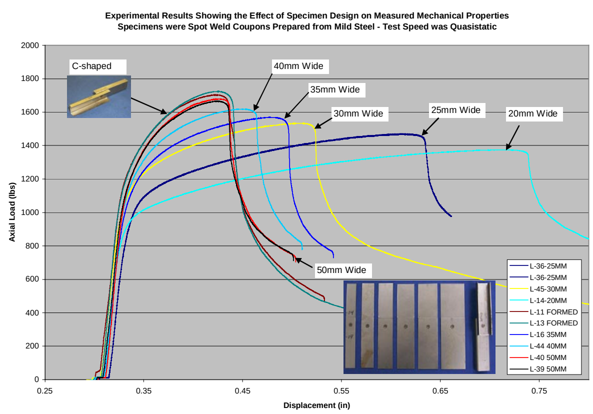


Figure 1. Experimental quasi-static measurement of tensile-shear spot-welded samples of different coupon designs.

Static weld tensile shear measurements shown in Figure 1 validate the conclusion that weld performance varies with joint coupon design. The joint strength—that is, the peak load value of the load-displacement curve—increases with increasing coupon width and converges to a single curve after a coupon width of 50 mm is reached. The displacement at peak load increases with decreasing coupon width because the overall cross-section moment of inertia is reduced when coupon width is reduced. Therefore, coupon rotation is more prominent during the deformation process. This large displacement at failure does not indicate the weld ductility; rather, it is caused by the rotation of the two sheets during the deformation process. On the other hand, when the coupon width is increased or a C-channel flange is introduced, the overall cross-section moment of inertia is increased, and there is little or no coupon rotation during weld failure. Therefore, the true weld strength and energy absorption should be evaluated using a coupon width of larger than 50 mm or a coupon with a C-channel for this joint.

Similar experiments have been carried out for tensile shear riveted aluminum coupons. For example, Figure 2 shows the quasi-static testing results for 2-mm AA6111 riveted tensile shear coupons with different widths and C-channels. A similar conclusion can be derived that a coupon width of 50 mm should be sufficient to reduce the sheet bending effect and, therefore, capture the true rivet strength and displacement at failure for the energy absorption.

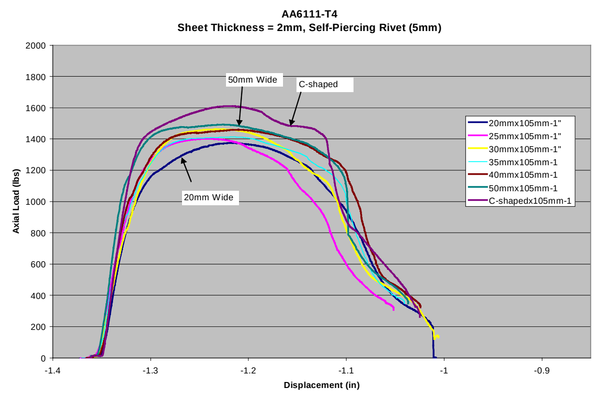


Figure 2. Experimental quasi-static measurement of tensile-shear riveted aluminum samples of different coupon designs.

Based on these experimental results, the joining team and PNNL staff decided to use the flat tensile-shear samples with a 50-mm coupon width to achieve the convergent joint strength.

Similar work on joint design issues of cross-tension samples is being conducted at PNNL and will be reported during the next quarterly review meeting.

Joint CAE

To allow flexibility in investigating the influence of different weld attributes to determine the critical coupon width for tensile shear tests, a generic finite element procedure was developed. It allows the coupon width, weld diameter, sheet thickness, and sheet metal overlap to be changed parametrically so that the effect of different design attributes can be studied. This work parallels the experimental work, and it enables the study of more parameter combinations than the experiment could easily achieve. The model is validated by comparing the predicted load versus displacement curves. The experimental results are shown in Figure 1.

Figure 3 shows the model geometry and the detailed failure mode of the weld region. This generic analysis tool is used to study the coupon width and overlap effect. Figure 4 shows the predicted coupon width effect on the overall coupon rotation at failure, and Figure 5 shows the predicted overlap effect on static load-displacement behavior. The results show that the overlap width is not a critical factor influencing the static strength of the joint. Above 20 mm, the predicted static behavior of the samples with different overlap widths is almost the same. However, it should be mentioned that the minimal coupon width needs to be observed to avoid the bearing deformation of the parent near the rivet (see Figure 6).

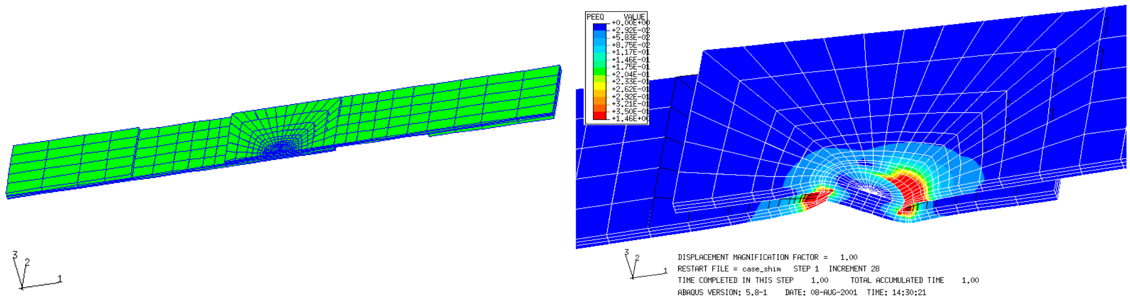
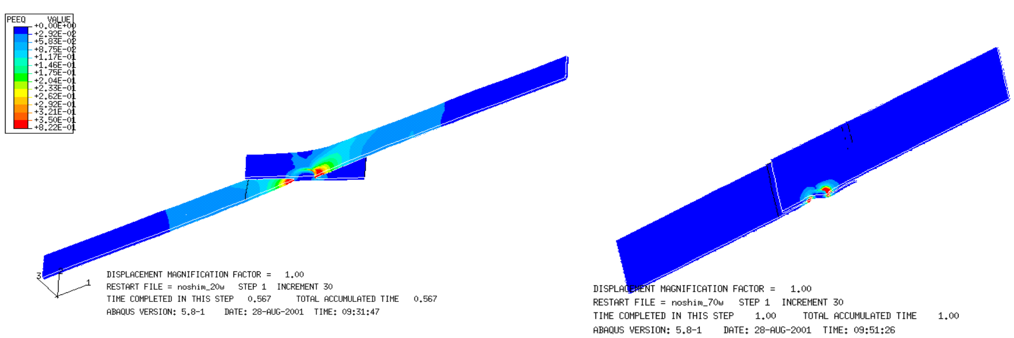


Figure 3. Model geometry and detailed failure mode of weld region.



Width = 20mm Width = 70mm

Figure 4. Predicted coupon-width effect on tensile shear samples.

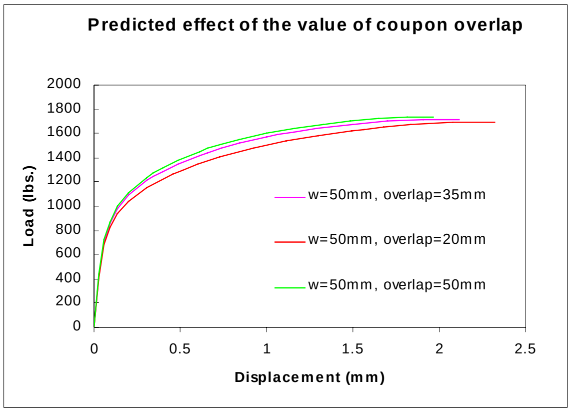


Figure 5. Predicted effect of coupon overlap.

Figure 6. Bearing deformation occurs in the base metal since the overlap width is too small.

K. Technical Cost Modeling

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objectives

- Address the economic viability of new and existing lightweight materials technologies.
- Develop technical cost models to estimate the cost of lightweight materials technologies.

OATT R&D Plan: Task 8, Barrier A

Approach

- Address the economic viability of lightweight materials technologies supported by the Automotive Lightweight Materials (ALM) program.
- Use cost modeling to estimate specific technology improvements and major cost drivers that are detrimental to the economic viability of these new technologies.
- Derive cost estimates based on a fair representation of the technical and economic parameters of each process step.
- Provide technical cost models and/or evaluations of the "realism" of cost projections of lightweight materials projects under consideration for ALM Program funding.
- Examine technical cost models of lightweight materials technologies including (but are not limited to) aluminum sheet; carbon fiber precursor and precursor processing methods; fiber-reinforced polymer composites; and methods of producing primary aluminum, magnesium, and titanium, and magnesium alloys with adequate high-temperature properties for powertrain applications.

Accomplishments

- Conducted a limited cost analysis of alternative precursor materials for the low-cost carbon fiber development program.
- Examined the economic viability of nondestructive testing (NDT) methods for testing of joints.
- Examined the economic viability of polymer composites and how the R&D supported by ALM is responding to the needs of the industry from an economic viability perspective.
- Identified and tested methods appropriate for estimating the benefits attributable to research and development projects funded by ALM.

Future Direction

- Focus on the fabrication technologies for composites.
- Explore the economic viability of a carbon fiber composite-intensive body-in-white.
  - Continue individual project-level cost modeling to identify specific technology improvements and major cost drivers that are detrimental to the economic viability of these technologies.
  - Continue the benefit evaluations of the R&D projects funded by the ALM Program.

Major Accomplishments

The major accomplishments of this effort include cost assessments of alternative precursor materials for low-cost carbon fiber and NDT methods for testing of joints. Another highlight was benefit evaluations of the R&D projects supported by the ALM Program. In addition, the composites R&D portfolio of the ALM program was examined in the context of satisfying the current needs of the industry from an economic perspective.

Analysis of Alternative Precursor Materials

To produce a low-cost carbon fiber for automotive applications in the range of $3 to $5 per lb, several alternative precursor materials are under consideration. To achieve the target cost, a significant reduction is needed in precursor and capital costs, which, combined, currently contribute to more than 60% of the total carbon fiber production cost. Melt-processable acrylonitrile copolymers have the potential to reduce costs by about $0.60/lb, compared with the conventional solution-spinning process, by eliminating safety and recovery costs associated with the use of solvents. Melt-spun lignin, relatively less expensive than polyacrylonitrile and a major by-product of the paper and pulp industry, can further reduce the carbon fiber cost by about $0.25/lb. The use of commodity textile acrylic tow as the alternative precursor material lowers the precursor cost significantly, but the overall cost reduction potential is only about $0.90/lb because of reduced overall process yield and higher capital costs resulting from a slower stabilization process and higher processing temperature.

Other approaches considered for low-cost carbon fiber include the preprocessing of precursors by chemical modification or radiation in order to enhance the stabilization step of the carbon-fiber production process. The combination of using commodity textile acrylic tow as the precursor material and preprocessing either by chemical modification or radiation can reduce the carbon fiber cost by $1.50/lb and $1.30/lb, respectively. The cost reduction potential is lower in the case of radiation preprocessing, mainly because of the higher capital cost of electron beam processing. Sulfonation of linear low-density polyethylene has the most cost-reduction potential—about $1.60/lb, mainly due to higher overall process yield (70% vs. 48% for the conventional process). The preliminary cost analysis further confirmed that a combination of low-cost precursor material and enhancements in the processing speed (particularly at the stabilization step) would be necessary to achieve the target carbon-fiber cost for automotive applications.

Viability of NDT Joint-Testing Methods

The cost-effectiveness of current (i.e., conventional pry) and potential (i.e., ultrasonic and global resonance) NDT methods was based on a potential demonstration of these methods on a particular application (i.e., a compact automobile door), assuming spot welds are being tested. The part-test cost is estimated to be $0.60 for the global resonance method, the least out of three NDT processes considered in the study. The part-test costs of the other two offline methods are considerably higher, mostly because of the labor-intensive nature, in the case of the conventional pry method. Labor dominated the total part-test cost (accounting for more than 85%); therefore, the automated online global resonance method allowing computer-based determination of joint integrity showed the most favorable economics. There is no doubt that NDT methods are more cost-effective, as more ultrasonic testing is being used for the evaluation of spot welds in the automobile industry today. In the future, the automobile industry's use of advanced lightweight materials that require adhesive bonding will necessitate the development of on-line nondestructive evaluation methods to provide a means of controlling the production process and ensuring the integrity of the vehicles. The cost-effectiveness of global resonance for spot-welds estimated in this study can be extrapolated to weld-bonded and adhesively bonded joints because the testing methodology would be the same for these types of joints.

Economic Viability of Polymer Composites

A literature review of the cost studies of composites shows some general qualitative trends in the economic viability of composites. To date, most of the cost analyses of polymer composites are for body-in-white (BIW) applications because of the significant weight-reduction potential these offer. The viability of composites is still seen predominantly in non-structural elements, such as the bolt-on exterior panels of today's vehicles; and most composites are glass-fiber–reinforced thermoset polymers, such as sheet-molded composites, used in niche-market vehicles with annual production volumes of less than 80,000. At a higher annual production volume of 250,000, for example, an evaluation of the most efficient composite monocoque design indicates that the cost of glass-fiber–reinforced thermosets and carbon-fiber–reinforced thermoplastics are 62% and 76% higher, respectively, than the cost of a conventional steel unibody.

Even on a life-cycle basis, the cost of polymer composites is estimated to be higher than that of steel unibodies. For composites to be cost-competitive in a part-by-part substitution, improvements are necessary in cycle times and material utilization, which in some cases have been estimated to contribute 60% and 21%, respectively, of the total cost of carbon-fiber–reinforced thermoplastics. The material cost plays a key role in the economic viability of polymer composites, particularly at higher production volumes and for carbon-fiber–reinforced thermoplastic composites. The carbon-fiber cost needs to drop by 50% (i.e., to the $3–$5/lb range), and smaller cost reductions in other thermoplastic materials are needed for these composites to be economically viable.

Responding to the Needs of the Composites Industry

DOE, in partnership with the Automotive Composites Consortium of the U.S. Council for Automotive Research, is sponsoring research under the ALM Program that seeks to overcome the barriers to more widespread use of composites in automotive applications. DOE is attempting to take a comprehensive look at the research needs of the composites industry and has prioritized certain areas, such as low-cost carbon fiber production, thermoplastic structural composites, and the development of new reinforcement technologies such as nanocomposite technology. Its research portfolio is appropriately focused both on its ongoing research and its 5-year research plan that covers five major barrier areas: cost, manufacturability, design data and test methodologies, joining and inspection, and recycling and repair. Although cost reduction is a pervasive factor in all the composites R&D activities, it is appropriate to focus the cost area on materials, primarily carbon fiber. To improve the manufacturability of polymer composites, development of high-volume production manufacturing processes should remain one of DOE's research priorities. More research is needed in the areas of carbon-fiber–reinforced polymer composites for these to be economically viable automotive materials. It is clear that for polymer composites to become the material of choice for automakers, an aggressive R&D portfolio should be followed to achieve major breakthroughs that are necessary for several orders of magnitude of cost reduction.

Estimation of Benefits

Through a thorough literature review, three methods were identified to estimate benefits attributable to ALM R&D projects: qualitative assessment, National Research Council indicators, and benefit-cost analysis. This combination addresses all-important aspects of the benefits of R&D projects. The qualitative assessment addresses short-term project outcomes at the project level. The indicators address standard measures associated with the quality of research projects. The benefit-cost analysis addresses both short-term and long-term benefits associated with commercializing new technologies. Since ALM projects encompass both the creation of new knowledge and the commercialization of new technologies, both the indicator and benefit-cost approaches were found to be appropriate for a comprehensive benefits assessment.

Three ALM R&D projects evaluated for the application of these benefit assessment methods were low-cost, continuous-cast aluminum sheet; advanced forming technologies for aluminum; and manufacturing of composite automotive structures. Figure 1 shows the market forecasts for the manufacturing of composite automotive structures, which were used to estimate its benefits and costs.

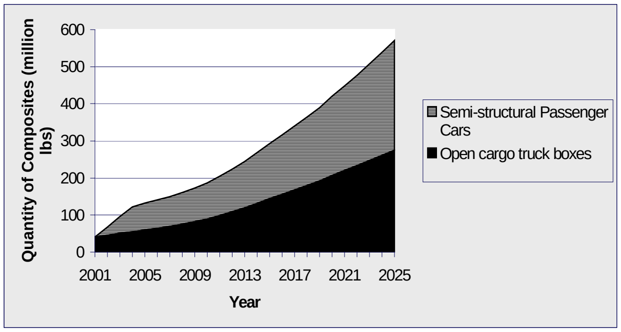

Figure 1. Forecasts of manufacturing of automotive composite structures.

The three projects assessed all appear to have yielded high levels of benefits. Based on the qualitative assessment, all met their technical goals, increased knowledge, and led to increased collaboration. It is not likely that these projects would have been undertaken without federal support, or at least undertaken as soon as they were. The only open question pertains to commercialization. With respect to the National Research Council indicators, there is a mixed rate of publication, but each project is shown to improve U.S. competitiveness in its respective area. The benefit-cost analysis yields impressive benefit-to-cost ratios, ranging from 168:1 to 699:1, when environmental benefits are taken into consideration. In order to improve the assessment of long-term benefits, publications and market penetration rates should be periodically revisited, and case studies need to be undertaken to identify any valuable spin-off technologies.

L. Nondestructive Evaluation Techniques for On-Line Inspection of Automotive Structures

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Contract No.: DE-AC03-765F0095

Objective

- Evaluate and develop nondestructive evaluation (NDE) and testing techniques that are sufficiently fast, robust in manufacturing environments, accurate, and cost-effective to be suitable for on-line inspection of automotive structures.

OAAT R&D Plan: Task 6, Barriers A, C, D

Approach

- Evaluate the state of the art of existing NDE technologies in conjunction with efforts to develop improved systems and new techniques that are necessary either to meet industry requirements or to meet the challenges posed by the introduction of lightweight materials.
- To develop the necessary technology, perform laboratory experiments in parallel with the development of sensors, models, data-acquisition systems, and data post-processing software toward the goal of producing prototype inspection systems for specific automotive-manufacturing applications.

Accomplishments

- Carried out site visits to commercial vendors of spot-weld-inspection systems and phased-array technologies.

- Conducted laboratory experiments using acoustic and thermographic techniques to evaluate conventional and phased-array spot-weld inspection systems.
- Developed a finite-difference code that allows modeling of wave propagation in anisotropic and viscoelastic media to study propagation of acoustic waves in spot-welded samples.
- Evaluated the state of the art of phased-array technologies and their suitability for spot-weld inspection.

Future Direction

- Evaluate commercially available spot-weld inspection systems; summarize in a report the results of round-robin evaluations completed by vendors and researchers.
- Identify and describe technologies viable in a manufacturing environment with the short- and long-term potential to improve the accuracy, robustness, speed, cost-effectiveness, and ease of use and implementation of spot-weld inspection systems.
- In conjunction with automobile manufacturers and suppliers, and government and university researchers, develop a roadmap to identify barriers to increased used of NDE in automobile manufacturing and determine strategies for implementing NDE inspection and process-monitoring techniques for specific applications.

Introduction

The NDE Industry Steering Committee is composed of representatives from DaimlerChrysler, Ford, and General Motors with expertise in manufacturing, materials, and NDE. The committee asked Lawrence Berkeley National Laboratory (LBNL) to evaluate the state of the art in spot-weld inspection and to determine the potential of acoustic phased arrays for inspecting welds. To accomplish these tasks, LBNL was asked to attend the annual Review of Progress in Quantitative Nondestructive Evaluation (QNDE) to learn more about recent advances in the development of phased-array techniques; evaluate commercially available spot-weld inspection systems; visit those vendors/institutions with the most promising conventional and phased-array systems; and evaluate the results of a round-robin experiment in which test specimens prepared by Ford are being inspected by vendors and institutions with applicable technologies.

To date, LBNL has visited IRT Scanmaster Systems; Krautkramer, Inc.; Sonoscan, Inc.; Imasonic SA; and Imperium, Inc. Meetings have been held with representatives from R/D Tech; Panametrics, Inc.; Commission de l'Energie Atomique (CEA, France); FORCE Institute (Denmark); Laboratoire de Mécanique Physique (University of Bordeaux, France); and Ohio State University. LBNL is in contact with Innerspec Technologies, Inc.; Edison Welding Institute; and Kumamoto University (Japan).

Project Deliverables

- Report summarizing the results of round-robin sample evaluations performed by commercial vendors.
- Paper summarizing the laboratory experiments and modeling studies, suitable for publication.
- Roadmap of strategies to increase the use of NDE in automotive applications.

Planned Approach

Assessment of the state of the art in conventional and phased-array spot-weld inspection is being accomplished by attending professional technical meetings; consulting with industry, government, and academic experts; visiting vendors and institutions with promising technologies; and analyzing the results of measurements performed by commercial vendors on test specimens provided by industry partners. In addition, LBNL is conducting laboratory experiments and modeling studies as necessary to evaluate phased-array technology and commercially available spot-weld inspection systems.

The round-robin testing being performed by commercial vendors is focused on determination of the size of the weld nugget, which is an industry criterion for distinguishing between good and bad welds.[1] Work being performed by LBNL is more broadly focused; NDE techniques are being evaluated based on

- their ability to identify the many different kinds of defects that occur in spot welds, including inclusions and voids
- their sensitivity to surface conditions, including zones of zinc expulsion and surface roughness
- their ability to inspect welds used in a variety of configurations, including triple-thickness joints and joints formed between sheets with different material properties and thicknesses
- their potential for miniaturization as necessary to inspect welds in locations where access is severely restricted
- the level of operator training required.

Viable systems must also be fast, robust, and able to issue a warning if they are not properly deployed.

Emerging phased-array technologies and commercial spot-weld-inspection systems are ultrasonic techniques. Evaluating these systems requires a thorough understanding of acoustic-wave propagation in spot-welded joints, including how acoustic waves are affected by defects in the welds, changes in material properties in the heat-affected zone, surface roughness, coupling between the sensor and test specimen, and orientation of the sensor. Laboratory and modeling studies were performed to help us understand these issues and interpret the results obtained with commercial systems.

Acoustic microscopy is a high-resolution technique used to create images of surfaces and bulk microstructures.[2,3] A focused transducer generating acoustic waves is used to scan the test specimen; images are generated by measuring the amplitude of reflected waves, which is directly proportional to changes in impedance in the sample. Acoustic microscopy measurements were made at the Laboratoire de Mécanique Physique in France. A stainless-steel test specimen with five spot welds is pictured in Figure 1. The quality of the welds was varied by varying the welding current between 5,800 and 10,500 A. The surface of the test specimen was polished because of the sensitivity of the method to surface conditions. Experiments are being conducted in several types of steel; stainless was used here to obtain baseline data. Welds in galvanized steel are more complicated to evaluate because the zinc coating has a lower melting temperature than the base material; the zinc tends to be expelled from the fused zone, forming a weak solder joint around the weld nugget.

Figure 1. Five spot welds joining stainless-steel plates. The quality of the welds was varied by varying the welding current between 5,800 (first weld) and 10,500 A (fifth weld).

The acoustic microscopy results for the spot-welded sample shown in Figure 1 are displayed in Figure 2. Blue corresponds to areas where reflections have relatively low amplitude compared with areas with high-amplitude reflections, shown in red. The scans were performed with the transducer focused at the interface between the two steel sheets. In the area of the weld nugget, where the steel sheets are fused together, only small-amplitude reflections are observed. Outside of the fused zone, the interface between the sheets generates high-amplitude reflections. Defects in the weld nugget are also visible; they can be observed in the first three images, where defects are evident as small bumps, indicating reflections with relatively high amplitudes. Work under way includes determining the relationship between the low-amplitude fused zone visible in the images, and the size of the weld nugget measured when the joint is peeled open.

A finite-difference model was developed to study acoustic-wave propagation across complicated boundaries such as those around the weld nugget and inclusions inside the weld. The code allows modeling of wave propagation in anisotropic and viscoelastic media. A staggered grid was implemented to increase the accuracy of the model without increasing the computation time. The method achieves second-order accuracy in time and fourth-order accuracy in space. Viscoelastic behavior is modeled using a tensor that allows different attenuation coefficients to be specified for different directions of propagation. Figure 3 shows a micrograph of a spot weld containing zinc inclusions. The finite-difference code described earlier was used to model the weld pictured in Figure 3. Figure 4 shows two snapshots of acoustic

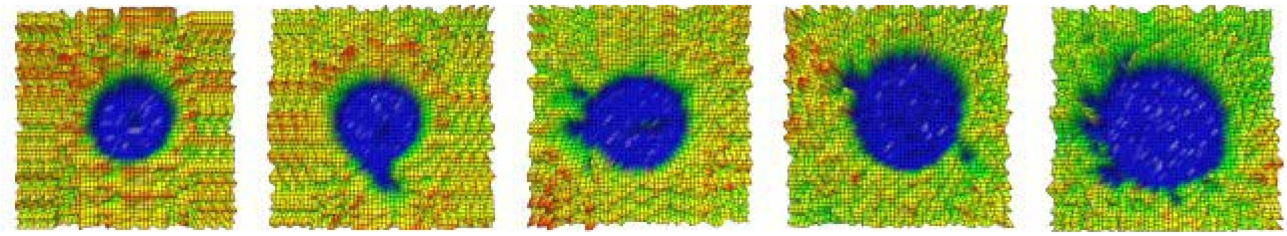

Figure 2. Acoustic microscopy images of the five spot welds pictured in Figure 1. Blue and red correspond to low- and high-amplitude reflected waves, respectively. The elements in the grid are 0.25-mm square.

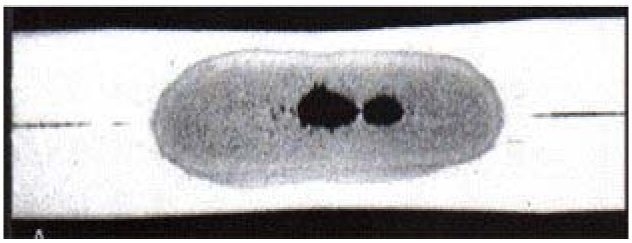

Figure 3. Cross section of a spot weld joining steel plates. Two inclusions in the weld nugget are clearly visible.

waves propagating in the sample; the dark lines represent the propagating waves, before (Figure 4a) and after (Figure 4b) the waves interact with the interfaces and defects in the sample that cause reflection and diffraction of the waves.

Work performed previously demonstrated that thermographic images of spot welds can be analyzed to provide information on weld quality[4–7]. For work in progress, thermographic images are being used to provide independent data to help interpret acoustic measurements. Infrared (IR) imaging radiometers are used to measure thermal-radiation energy; any change in thermal resistance in the direction of the heat flux results in a change in the temperature gradient. The spot-welded sample that produced the acoustic images displayed in Figure 2 was also evaluated using steady-state IR thermography. The resulting thermogram is shown in Figure 5; red corresponds to relatively warm areas compared with surrounding cooler areas, indicated in blue. The results show close agreement with acoustic microscopy measurements. Three spot welds appear to be satisfactory, while the two others appear to be undersized. These results will be confirmed when the welds are peeled open to reveal the size of the weld nugget.

Conclusions

Commercially available ultrasonic spot-weld inspection systems are based on time of flight and wave attenuation. These systems are relatively inexpensive and are widely used in Europe. Measurements are very sensitive to positioning of the probe, and operator training is critical. Work by LBNL and others suggests that the measurements are also affected by surface roughness and indentation of the surface caused by the welding electrodes. Existing commercial systems may not be viable for inspecting welds in aluminum or high-strength steel. However, modifications to these systems as required to improve performance may be a more cost-effective approach for certain applications than development of phased-array systems.

Industry's NDE Steering Committee asked LBNL to evaluate ultrasonic phased arrays against several criteria, including sensor technology, maturity of technology, industrial applications, robustness, and cost. Although phased arrays provide the ability to scan, focus, and steer with a fixed probe, they do not change what can be measured; that is, things that cannot be measured with conventional ultrasonic techniques cannot be measured with phased arrays. Based on site visits, discussions with experts, and a review of the literature:

- The number of elements in an array is not an issue except for cost, which is primarily in electronics.
- Although different materials are available and used for transducers, piezocomposites appear to be the most appropriate material for arrays because it is easier to minimize acoustic interaction between elements in them than with other materials.

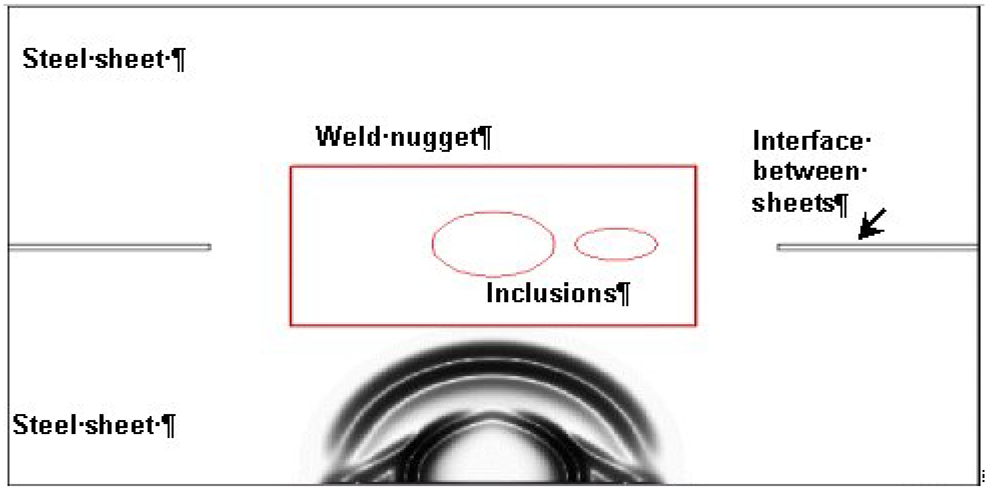

Figure 4a. Snapshot in time of acoustic waves propagating in a spot-welded sample (Figure 3) calculated using a finite-difference model developed to help interpret results obtained from spot-weld inspection systems. The image shows longitudinal, shear, conical, and surface waves generated from a source located at the center of the bottom surface. The image shows the waves propagating from the source before they interact with the weld nugget (red box) and interfaces between the two steel sheets.

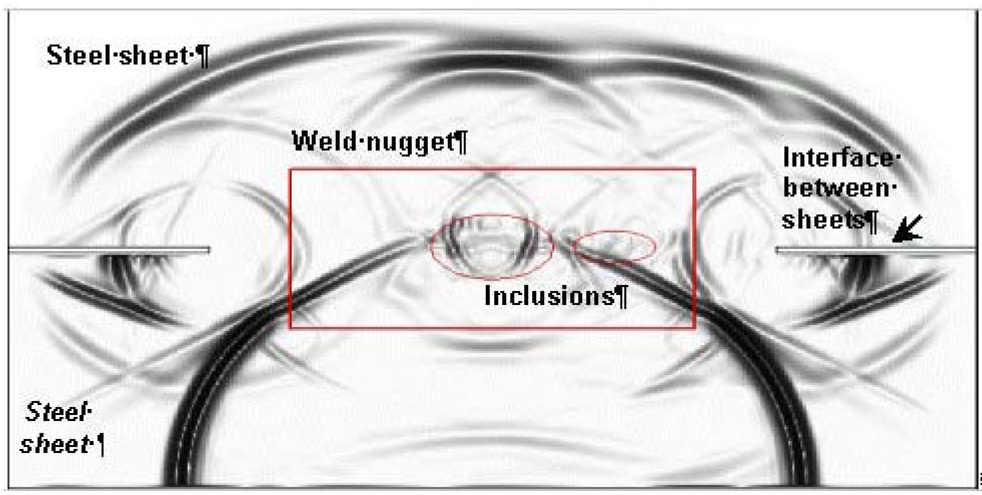

Figure 4b. Snapshot of propagating acoustic waves at a later time than that captured in Figure 4a, showing interaction of the waves with the weld nugget, inclusions inside the nugget, and the interface between the steel sheets. Reflections, mode conversion, and diffraction can be observed.

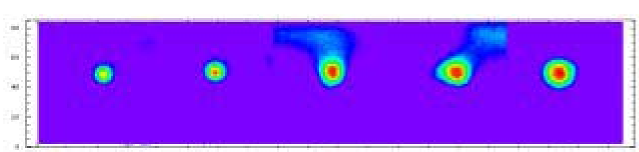

Figure 5. Thermograms of the five spot welds pictured in Figure 1. Red in the welds corresponds to relatively warm areas compared with surrounding cooler areas, shown in blue.

- Commercially available arrays all allow scanning, as well as steering and focusing of the acoustic beam with a single probe.
- At present, there are limitations on element size and frequency. The smallest element available is about 0.5 mm; smaller elements can be manufactured, but as the size decreases, the stress acting on the element increases, making it more difficult to control the frequency. The highest frequency that can be obtained at present is on the order of 18 MHz.
- Electronics are the most expensive components in a phased-array system, typically costing more than ten times as much as the probe.
- Costs are likely to drop in the future with increased competition.
- Surface roughness and coupling are still issues. Problems in interpretation are similar to those with conventional systems; for example, for galvanized steel, it may be difficult to differentiate between the heat-affected zone, zinc solder joint, and weld nugget.
- At present, no 2-dimensional matrix array is commercially available; systems have to be custom-ordered and built for a particular application.

Because the measurements made with phased arrays are comparable to those made with conventional ultrasonics, existing inspection systems and laboratory measurements can help determine the viability of phased arrays. Issues including cost and manufacturability may limit the viability of phased arrays for spot-weld inspection in the near future. If so, modifications to existing systems to improve their performance, and ease of use may be a cost-effective solution for the near term. In addition, recent advances in computer science, statistics, and signal processing provide new opportunities for in-process monitoring and for reducing the reliance on trained operators.

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8. HIGH-STRENGTH STEELS

A. Enhanced Forming Limit Diagrams

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-SL05-01OR22866

Objective

- Document previously unavailable relationships between stamping die variables and increased forming limits that will be necessary to facilitate the manufacture of lightweight steel vehicles. This objective will be accomplished by using unique capabilities developed for this project, which continue to be refined.

OAAT R&D Plan: Task 12, 13; Barriers B, C

Approach

- Phase 1: Extend the predictive relation for aluminum-killed draw quality (AKDQ) steel to the high-strength steels critical to the lightweighting of steel passenger car bodies.
- Phase 2: Develop an application methodology for use on the original equipment manufacturer’s vehicle programs.
- Phase 3: Develop training modules for effective transfer of the technology to the manufacturing floor.

Accomplishments

- Designed experimental equipment and developed procedures to quantify improvements in the breakage limits.
- Used a channel draw die to produce open-ended channel sections using varying draw bead geometries.
- Completed a predictive model for AKDQ steel.
- Measured and analyzed data from the channel draw tests.
- Performed longitudinal and transverse tensile tests from the wall of the channel draw specimens. These included 19 conditions and 2 directions, in triplicate, for a total of 114 tensile tests.
- Analyzed the data and extended the predictive model to the high-strength steels needed for the A/SP lightweighting initiatives.

Background

The Enhanced Forming Limits project team was formed to quantify increases in breakage limits to allow greater use of the intrinsic formability of sheet steel. This goal is particularly important for mass reduction initiatives because the required high-strength steels exhibit reduced formability. It thus is critical to use all of the formability that is available. This technology reduces die tryout costs by avoiding the delays associated with solving forming problems, and it can reduce manufacturing costs through improved blank utilization. In addition, this effort provides a framework for future studies of sheet metal stamping that would broaden the range of parts that can be successfully produced with advanced high-strength steels.

B. High-Strength Steel Stamping Project

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Determine how to accurately predict the degree of springback in a variety of flanging conditions on parts made from high-strength steel (HSS) sheets prior to the construction of the production tooling.
- Identify part designs and manufacturing processes that will reduce springback and other part distortions and recommend them to design and manufacturing engineers.

OAAT R&D Plan: Task 12, 13; Barrier B

Approach

- Enhance HSS stamping springback predictability through finite element analysis (FEA).
- Improve HSS stamping springback control by developing the required knowledge of part design geometries that affect flange springback and die processes that help to control springback.
- Constructed, or obtained on loan, dies offering different panel conditions for the Auto/Steel Partnership for process development.

Accomplishments

- Studied FEAs of part designs and computer simulations of manufacturing processes to determine factors causing springback of HSS sheet metal stampings.
- Established optimum stamping die processes and die metal clearance values through comparison of simulation output to actual stamping results. Although the most accurate springback prediction was noted using a die

clearance of 1.4 times the metal thickness (1.4t), the least amount of actual part springback occurred when the die clearance was reduced to 1.1 times metal thickness (1.1t). The simulation group is working to improve the FEA predictive accuracy with varying metal clearances to produce the least amount of springback and variation.

- Conducted springback control experiments using tooling designed to replicate actual production stamping die processes. These experiments confirmed the importance of optimizing part geometry and the stamping process in order to reduce residual stress, flange springback, and part-to-part variation.
- Measured parts and recorded and analyzed the data. Results will be published in hard copy and on the Auto/Steel Partnership web site.

Introduction

The use of HSS in automotive applications has been increasing over the last 10 years. Owing to the mechanical properties of HSS, the springback after forming and the geometric dimensional control of the stamped parts have been critical issues in stamping tool construction and in the stamping process. Because the actual dimensions of the HSS stamping off the tool (e.g., for a structural rail-type stamping) are unpredictable with current tools and technology, the average die face re-machining process may be four to six times that required for mild steel applications and result in 2 to 3 months of added tryout time.

Computer simulation technology has been widely applied in the stamping industry and has been recognized as a common virtual stamping tool to identify the formability issues and evaluate the solutions before the actual stamping dies are made. Actual experience has demonstrated that computer simulation data have not been reliable in predicting the degree and modes of the springback for rail-type stampings, even with small form radii of two to three times the metal thickness.

Details

To assess the accuracy of the current forming/springback simulation technology, a full-size structural rail stamping die and a smaller die representing a segment of the full-size die were built. Three different HSS materials—40, 50 and 60 KSI—were used to stamp rails, and the finished panels were measured. A correlation between actual and simulation data quantified the differences. The simulation task team proposed that predictions be developed for the die segment, stampings be manufactured with varying material clearances in the die, and simulation predictions be compared with the measured stamping data. The segment findings will then be extrapolated over the full-size panel (Figure 1). The purpose of this proposal is to allow the continuation of stamping trials and computer simulation comparisons, but with the additional element of working with known die gaps, rather than assumed clearances between the punch and the die.

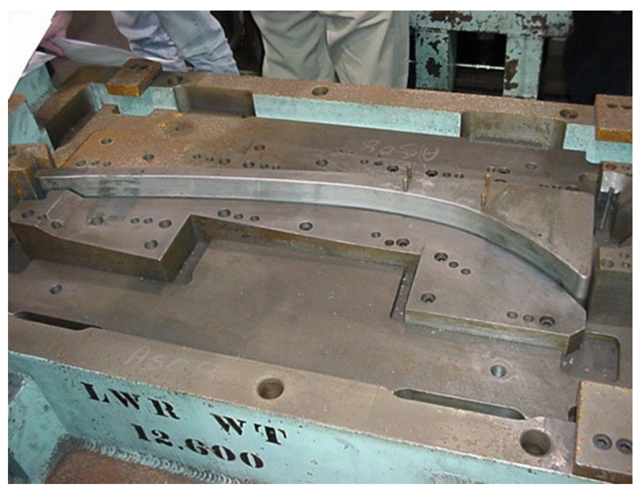

Figure 1. Full-scale rail stamping die (lower half).

C. Hydroform Materials and Lubricants Project

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Develop mechanical test procedures for tubes.
- Improve the accuracy of and confidence in finite element modeling of tubular hydroforming.
- Develop an understanding of steel and lubricant requirements for hydroforming, using a combination of experiments and finite element modeling.

OAAT R&D Plan: Task 8, 13; Barriers B, C

Approach

- Conduct a series of experiments to determine the free expansion and corner fill capabilities of various mild and high strength steel tubes using internal pressurization.
- Conduct other experiments for tube expansion to determine the effects of axial compression in combination with internal pressurization and the effects of pre-bending and pre-forming on subsequent formability.
- Use the collected data to develop forming limit diagrams for tubular hydroforming. These are required for the computer simulation of hydroforming.

Accomplishments

- Worked with the Industrial Research and Development Institute (IRDI) of Midland, Ontario, Canada, to perform free expansion and corner fill experiments with mild and high-strength steel. Existing mechanical property testing methods for tubes have been evaluated, and new testing methods are being developed as needed.
- Started evaluating the validity of the traditional forming limit curve for sheet metal stamping and developing correction factors for tubular hydroforming.
- Initiated a designed experiment using both mild and high-strength steels to gain an empirical understanding of material and lubricant requirements in order to develop test cases for validation with finite element modeling.

The experiment covers free expansion and corner filling of tubes, as well as internal pressurization and axial compression with straight and bent tubes.

- Completed the free expansion and corner fill portions of the experiment. As of October 30, 2001, tubes are in the process of being made and bent for the ongoing internal pressurization and axial compression experimentation that will continue into 2002.

Background

The formability limits for steel in tubular hydroforming applications are poorly understood. Practitioners are using the sheet steel forming limits developed for conventional stamping processes to assess the formability of hydroformed components. As an example, the effect of axial compression on the forming limits of steel is unknown. Likewise, the effect of prior strains induced in the tube-conversion and tube-bending processes that precede the hydroforming operation have been ignored. The accuracy of material characteristics needs to be assessed and improved to allow the optimum application of tubular hydroforming in vehicle lightweighting activities.

In addition, the tests that tube producers are using to evaluate tube quality do not properly address metal forming issues relevant to tubular hydroforming. The current tests focus on the weld quality and dimensional accuracy of the tubes, but they do not adequately describe the amount of ductility left in the tube for post-forming operations such as hydroforming. There is a need to develop and evaluate tube-testing methods that are suitable for evaluating and comparing material formability and are also suitable for input into finite element models.

Furthermore, additional information is needed on the fundamental material attributes that control the hydroformability limits of steel. This knowledge will allow steelmakers to develop new steel grades or apply existing steel grades to hydroforming applications (Figure 1). Experiments will be conducted using the facilities of the IRDI of Midland, Ontario, Canada.

Figure 1. Section of die and part for hydroforming corner-fill experiments.

D. Sheet Steel Joining Technologies

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC-59OR22363

Objective

- Facilitate the increased application of advanced high-strength steels (AHSSs) in Auto/Steel Partnership (A/SP) lightweighting projects through the development of weld parameters and schedules that will produce quality welds in 12 different grades of these advanced steels. The project will be pursued from several perspectives, including weld lobe evaluation under peel test conditions to determine the optimum welding for weld time and current. Weld-bonded joints will be evaluated, as well as straight-welded joints. Impact fracture and fatigue characteristics of these welds will be documented and compared with the weld strengths (cross-tension and tensile shear) in the development of a standard for partial thickness fractures.

OAAT R&D Plan: Task 6, 8; Barrier D

Approach

- Establish a weld window for the 12 different AHSSs using two resistance spot-welding systems, scissor gun, and “C” gun, and evaluate the welds through peel test, tensile shear, cross-tension, microhardness, and metallographic examination.
- Develop a fatigue test coupon and evaluate weld fatigue properties for the various AHSS materials.
- Develop an impact test model and evaluate impact characteristics for the various AHSS materials.

- Characterize the scissor gun and C gun machines as to rigidity and weldability.
- Characterize the effects of electrode design, metallurgy, and coatings on the weldability of the particular steels.

Accomplishments

- Established the procedure for weld lobe study, testing, and machine characterization.
- Acquired 200 ft^2 of material for 10 of the 12 materials and delivered the material to the test facility, where it has been sheared into coupons.
- Completed the weld lobe study to the 75% level, including weld peel, shear coupons, and micro-examination.
- Prepared metallographic samples and took photographs as peel tests were completed.

Background

The use of AHSSs is integral to the steel industry effort to make automotive components more lightweight while not sacrificing strength or escalating cost, an issue with the application of other lightweight materials. The development of welding parameters and physical testing parameters for the new steels qualifies these for various automotive components. Extensive work has been attempted on spot welding of mild steels during the Intelligent Resistant Welding Project of the Auto Body Consortium, and various A/SP resistance welding and adhesive bonding projects.

E. Sheet Steel Fatigue Characteristics

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objectives

- Investigate the work-hardening effects of stamping and the strengthening effects of paint baking on the grades exhibiting such effects on constant-amplitude strain-controlled fatigue properties, and compare them with similarly strained and baked conventional high-strength steels.
- Investigate the variable-amplitude performance of low-carbon steel compared with that of interstitial-free steel.
- Investigate other new advanced high-strength steels, possibly with various joining methods.

OAAT R&D Plan: Task 8, 13; Barriers B, C

Approach

- Characterize the fatigue properties of selected grades of steel by identifying three fatigue coefficients and three fatigue exponents.
- Compile the results into a database that can be used by designers and design engineers to retrieve and compare the fatigue lives of sheet steel structural components in the design phase.

Accomplishments

- Completed phase 2 and phase 3 testing.
- Developed a sheet steel fatigue database and populated it with fatigue data on 54 coils of steel. Team members are evaluating the database as a resource for fatigue information.

Introduction

In the era when vehicle mass was not a drawback, and in fact was sometimes considered an advantage, the primary concern in body design was rigidity. More recently, the need to reduce body mass in order to comply with mandated corporate average fuel economy (CAFE) standards has caused design engineers to reexamine design procedures and materials. High-strength steels, judiciously selected and applied, emerged as potential low-cost, reliable materials for reducing vehicle mass. As structural components are optimized and thinner-gauge, higher-strength materials are assessed, component fatigue life becomes a consideration. To assess the performance of a material in the design phase, its fatigue characteristics must be known.

This project is addressing steel grades identified by the lightweighting initiatives (closures and front end structures) of the Auto/Steel Partnership. The objective of this 3-year program is to continue the investigation of particular formable and high-strength steels used in automotive components. Advanced high-strength steels are being investigated by the Lightweight Front End Structure Project Team for their potential to reduce weight and manage crash energy. The work will also investigate the performance of steels currently being used under variable-amplitude conditions. The results are expected to improve durability modeling and simulation.

Details

The long-term strategy for the project to research sheet steel fatigue characteristics consists of four phases of work. Phase 1 objectives were to develop and establish confidence in the testing procedure. This phase included selecting machine shops and laboratories to prepare the test samples and perform the tests. Test samples must be prepared in a manner that does not alter the as-rolled properties of the sheet steel. For example, stamping the samples will induce work hardening in the immediate area of the cut edge. The work hardening is essentially eliminated by machining the samples according to carefully determined procedures. The design of the machined test sample is shown in Figure 1.

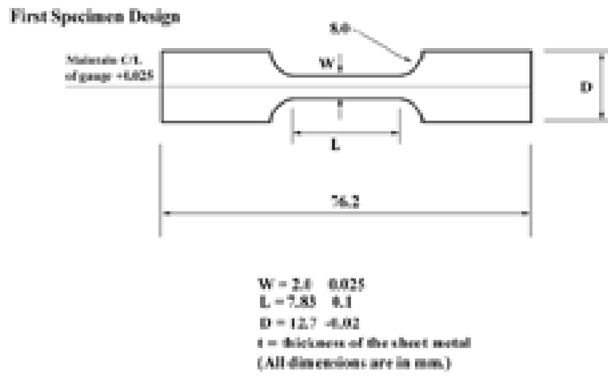

Figure 1. Design of the machined test sample.

Selection of qualified test laboratories capable of performing the work is even more critical. Since participating project team members had extensive experience with a number of qualified machine shops and laboratories, phase 1 was completed on March 1, 1997. To maximize the consistency among testing services, a standard test procedure was written for use by all test laboratories.

Phase 2 was the testing, based on procedures established in phase 1, to determine the fatigue properties of the following steel grades:

- Interstitial-free hot-dipped galvanized
- Drawing-quality, special-killed, cold-rolled
- High-strength low-alloy (HSLA) 50 ksi (345 MPa) hot-dipped galvanized
- High-strength low-alloy 50 ksi (345 MPa) galvannealed
- Interstitial-free hot-dipped galvannealed
- Structural-grade 40 ksi (275 MPa) galvannealed

The tests for each grade of steel were normally based on samples taken from material supplied by three different steel suppliers. Samples from each supplier were taken from three different locations on each of three coils of steel from the supplier. Therefore, each data set for each grade of steel was based on samples taken from $3 \times 3 \times 3 = 27$ locations. A sufficient number of test coupons were prepared from each location that each fatigue test was run at multiple strain amplitudes. The results were plotted on a log-log scale of cycles versus strain amplitude, and a theoretical curve was constructed.

This work was reported as completed at the January 10, 2001, team meeting. An Access database was developed, and all the material properties, including strain based fatigue, were entered.

Phase 3 is the verification testing to determine and demonstrate the benefits of the work accomplished in phases 1 and 2. Verification was performed on DDQ+ steel. Two SAE spectra were tested: transmission time history and suspension time history. The test matrix consisted of three replicates at five strain/load levels for each spectrum for a total of 30 tests. The range of strain/load for each spectrum was approximately 3 to 1.

Phase 4 was initiated upon agreement of the team members. This phase of work comprises two projects:

Project 1 is an analysis of the strain + bake properties of dual-phase (DP) 600 vs. transformation-induced plasticity 600 vs. HSLA 340 (50 ksi). Two prestrain conditions were specified.

- Balanced biaxial stretch with an equivalent strain of approximately 2% for the DP 600+ and HSLA 340 steels
- Balanced biaxial stretch with an equivalent strain of approximately 8% (or ~6% if 8% cannot be achieved) for all three materials

This work is scheduled for completion in March 2002.

Project 2 is a comparison of low-carbon drawing steel to the interstitial-free steel tested in Phase 3 under variable amplitude loading conditions. This work is scheduled for completion on October 31, 2001.

The sheet steel fatigue database, noted above under Phase 2, will be an essential product of this program. That database is intended to provide a user-friendly compilation of the fatigue data generated in Phase 2, which was reported on a series of spreadsheets. Compilation into the database will give the user access to data in one location. It also provides features such as data sorting, plotting and data averaging.

F. Strain Rate Characterization

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objective

- Embark on a 4-year program that will result in the development of test methods, constitutive materials models, and finite element analysis (FEA) guidelines for industry-wide use.

OAAT R&D Plan: Task 8; Barrier C

Approach

This project addresses two aspects of the simulation accuracy problem:

- Accurate representation of material properties:
  - Develop a standard test for sheet metal at high strain rates to characterize materials with minimum testing efforts.
  - Evaluate Hopkinson bar and servo-hydraulic load frames to capture the behavior of sheet metal at typical crash strain rates, to understand the relationship between tension and compression, and to understand and effectively model the hardening behavior of sheet steel under typical crash conditions.
  - Develop a database of properties for existing and new materials being developed for use in automotive construction, including advanced high-strength steels.
- Optimization of material constitutive models and finite element models:
  - Develop the necessary guidelines for structuring finite element problems to ensure that all inputs, including material properties, are accurately reflected in the results.
  - Develop validation experiment(s) for evaluating the different materials and material models that they represent.
  - Optimize the use of new element technology, time-step control and integration, modeling of contact conditions, and modeling of joints.
  - Evaluate stochastic approaches to crash modeling.
- Evaluate the influence of part variability on the stability of collapse modes in components and in assemblies.

- Recommend robust, accurate computationally efficient materials models for FEAs.
- Evaluate the influence of work hardening, bake hardening, and strain-rate sensitivity effects on constitutive models.
- Develop models for non-ferrite materials such as dual-phase and transformation-induced plasticity steels, as well as models for multi-axial loading.

Accomplishments

- Completed a project through the University of Dayton Research Institute for compression and tensile "split Hopkinson bar" tests at strain rates of 300/s and 1000/s on drawing-quality, special-killed and DF140T steel grades. The final report was delivered.
- Completed review of the Callidine and Avalle papers to arrive at recommendations on the test procedures for the cylindrical tube crush test.
- Continued to support the Oak Ridge National Laboratory project "Strain Rate Sensitivity Study for Automotive Structures."
- Completed specification development work for quoting the axial hinge plate and the cylindrical tube crush experiments.
- Submitted purchase order requests for axial hinge plate crush and cylindrical tube crush experiments.
- Issued a purchase order to a supplier for the manufacture of steel tubes that will be used in the tube crush experiments. The high-strength steel materials to be used in experiments—drawing-quality mild steel, high-strength, low-alloy 350 Mpa steel, and dual-phase 600 Mpa steel—will be supplied by partnership member companies.

Background

Over the past several years, evaluating crashworthiness in new vehicle designs has become more and more dependent upon math-based engineering tools, including FEA. The use of these tools enables rapid optimization of product designs, thereby reducing development time and cost while minimizing product mass. The growing dependence on these techniques and the desire to accelerate the replacement of physical testing with analytical validation of design solutions is driving the need to improve the predictive capability of the math-based tools.

The accurate representation of the mechanical properties of the sheet steels used in current and future vehicles is a key element in the development of accurate math-based tools. One key aspect of these properties is the strain rate dependence of the stress/strain behavior. Previous Auto/Steel Partnership research has experimentally demonstrated the positive influence of strain rate sensitivity on dynamic performance of sheet steel structures. Efforts to accurately simulate this behavior using math-based tools have not, however, been completely successful.

G. High-Strength Steel Tailor-Welded Blanks

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-SL05-01OR22866

Objectives

- Investigate the formability and weld properties of high-strength steel (HSS) and advanced HSS (AHSS) tailor welded blanks (TWBs).
- Determine the fatigue properties of laser and mash seam welds in AHSS.
- Investigate the weight and cost savings potential for patch-type vs laser-welded TWBs.

OAAT R&D Plan: Task 6, 8, 13; Barriers B, C

Approach

- Prove the concept of using patch-type TWBs on door inner panels.
- Make stampings from automotive door inner dies to produce doors with patch-type TWBs.
- Conduct fatigue tests on AHSS blanks with laser and mash seam welds.

Accomplishments

- Worked with Oxford Automotive to make stampings with patch-type blanks from automotive door inner panel dies.
- Determined testing procedures and began identifying prospective testing laboratories for fatigue tests.

Introduction

The tailor welding of sheet steel blanks—that is, the use of varying gauges and grades of steel within a specific panel "tailored" to provide added strength in select areas—continues to gain favor in the product design community. Industry data show that production of TWBs has grown from just over 1 million in 1993 to nearly 24 million in 1999, with the market potential estimated at 50 million blanks by the year 2003. TWBs have gained acceptance in the automotive industry as a means of simultaneously achieving cost and weight reduction. The use of conventional high-strength low-alloy steels, HSSs, and AHSSs—such as dual-phase and transformation-induced-plasticity steels—offers the potential for further weight reduction. However, uncertainty exists regarding the weldability of these products and the resultant ductility of the weld and heat-affected zone. Manufacturing techniques must be developed and validated to produce reliable, high-volume HSS and AHSS TWBs.

The purpose of this focused 3-year project is to investigate the manufacturability and formability of HSS and AHSS TWBs and demonstrate that these products can produce a front-end structure of significantly lower mass.

In addition to supporting the work of the Auto/Steel Partnership Lightweight Front End Structure Project, the Tailor Welded Blank Project Team is investigating the wider application of patch-type blanks to reduce the weight and cost of automotive door assemblies for the Auto/Steel Partnership Light Weight Closures Project.

Approach

The TWB project team has worked with the Lightweight Closures Project Team to prove the concept of using patch-type TWBs on door inner panels (Figure 1). This type of tailored blank reduces weight because the spot-welded patch can

Figure 1. Door inner hinge reinforcement made from a patch-type blank.

be made to the smallest required irregular shape, whereas the laser weld must be a straight line, making the heavy metal member of the blank much larger and heavier than necessary. The cost of spot welding the patch-type blank is also much less than the cost of laser welding.

The TWB Project Team worked with Oxford Automotive to make stampings from automotive door inner dies to produce this type of door. Initial reviews by representatives from General Motors and DaimlerChrysler were generally positive, with requests to repeat the stamping trials on dies for doors with more complex configurations.

Fatigue test planning has been done in conjunction with the needs of the Lightweight Front End Structures Project. As they defined the materials they would use for tailor welded blanks in the motor compartment rails (Figure 2), the Tailor Welded Blank Project Team has determined the required test procedures and is determining prospective laboratories to perform this work.

Figure 2. Example of a patch blank on an underbody rail part.

Results

Most of the work undertaken in the first 6 months of 2001 involved the patch-type tailored blank for the Lightweight Closures Project. Patch-type blanks have been used in some less complex production applications to integrate parts and reduce tooling cost. The stamping die tryout work with Oxford Automotive indicates the potential for this process to reduce the cost and weight of select door assemblies. Doors with a straight hinge pillar surface, such as rear doors, appear to have the most potential at this time. Minor distortions at the sealing surface are a problem, but they can be corrected in subsequent die operations.

H. Tribology

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Contractor: U.S. Automotive Materials Partnership
Contract No.: DE-FC05-95OR22363

Objective

- Conduct stamping simulation tests to study the effects of tribology—the interrelationship of lubrication, friction and wear—on the stamping performance of advanced high-strength steels. (Stamping performance in this project is defined as minimizing die wear and maximizing dimensional stability. Understanding of the contribution of lubricants to errors in springback prediction when using finite element analysis will also improve dimensional performance.

OAAT R&D Plan: Task 8, 13; Barrier B

Approach

Phase one:

- Select the sheet high-strength steel material grades for the tests.
- Secure the material and ship it to TribSys Inc. to do the testing. Using its twist compression tester and draw bead simulator, TribSys will perform friction testing.

Phase two:

- Compare the wear rate of draw beads using different lubricants and high-strength steel materials. Restraining force will be compared with temperature, contact area, and wear on the draw beads. These data will be correlated with the results of the twist comparison test and draw bead simulator test conducted in phase one.

Accomplishments

- Changed the name of the project team from sheet steel lubricants to tribology.
- Invited lubricant suppliers to address the project team with their product recommendations along with supporting performance data. Participating companies included Quaker Chemical, Diversified Chemical Technology, Inc., Fuchs Chemical, and Midstate Chemical.
- Member company Dofasco Steel did a presentation titled " Lubricant/Steel Characterization Study Using the Draw Bead Simulator"
- Installed Alan Pearson of General Motors as the new project team chairman on July 1, 2001

Introduction

Lubricants have historically been used in the forming of sheet metal stampings to reduce friction, improve formability, and minimize die wear. They also provide corrosion protection. The anticipated increase in the use of advanced high-strength steels places a greater emphasis on understanding the process parameters associated with die wear, such as heat build-up, die scoring, and dimensional variation caused by springback variation. The contribution of interfacial friction to springback variation is not known. Advanced high-strength steels may require different lubricant and/or die materials to minimize the friction and die wear.

I. Modeling of High-Strain-Rate Deformation of Steel Structures

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Contract No.: DE-AC05-00OR22725

Objective

- Develop numerical modeling guidelines to realistically assess the influence that the properties of strain-rate-dependent materials exert in crashworthiness computations. The dynamic loading problems are modeled using diverse combinations of modeling approaches (sub-models) that are essential in describing strain-rate sensitivity in computational simulations.

OAAT R&D Plan: Task 7, 8, 13; Barrier C

Accomplishments

- Performed a literature review for progressive crushing and characterization and modeling of strain-rate effects in automotive impact.
- Developed computational models for steels under various strain rates based on experimental data.
- Developed computer programs for calculating instantaneous strain rates that are used for evaluation of constitutive models in computer programs.
- Evaluated finite element model (FEM) element performance for modeling of axisymmetric crushing of circular tubes.
- Determined the effects of various material parameters on the crush behavior of axisymmetric tubes.
- Developed experiments for characterizing structural and material aspects in component crashworthiness.

Future Direction

- Analyze transitions from axisymmetric to antisymmetric mode in progressive crushing and evaluate the performance of FEM formulations for modeling of transition problems.
- Determine the effects of FEM shell element discretization and formulation on model accuracy.
- Determine optimal FEM formulations for modeling of crushing of rectangular tubes.
- Develop an experimental program for crushing of rectangular tubes.
- Develop modeling guidelines for rectangular tubes.

Introduction

In the past several years, the development of the crashworthiness of new vehicles has become more and more dependent upon math-based engineering tools, including finite element analysis. The use of these tools enables the rapid optimization of product designs, reducing development time and cost, as well as minimizing product mass. The growing dependence on these techniques, and the desire to accelerate the replacement of physical testing with analytical validation of design solutions, is driving the need to improve the predictive capability of the math-based tools.

The accurate representation of the mechanical properties of the sheet metals used in contemporary vehicles is a key element in the development of accurate math-based tools. An important aspect of these properties is the strain-rate dependence of the stress-strain behavior. Previous research has experimentally demonstrated the positive influence of strain-rate sensitivity on the dynamic performance of sheet steel structures.[1, 2] However, efforts to accurately simulate this behavior using math-based tools have not been completely successful. Three aspects to this problem must be addressed:

Accurate material properties. Several test methods are available to measure material properties under stress-strain conditions representative of crash events, such as the Hopkinson pressure bar and high-capacity servo hydraulic load frames coupled with advanced extensometry for strain measurement. The relatively low thickness of sheet metals typically used in vehicle structures presents a special problem in accurate mechanical testing of these materials. The characteristics of load transfer from grips to sample in high-strain-rate tensile tests are not well known. Compression tests must be done through the thickness. Nonetheless, accurate properties are the cornerstone of accurate simulations. Recommendations are needed for robust, accurate testing methods for sheet materials.

Constitutive material models. The mechanical properties of the materials must be accurately represented in the computational simulation programs. This is done either by direct representation of the stress-strain curves; by development of a mathematical model of the stress-strain behavior, using empirical or fundamental equations; or by a combination of the two. The models must allow the computer code to efficiently and accurately select the appropriate material properties for the stress-strain situation at any given time step in the crash simulation. The models should also comprehend the changes in properties due to processing during vehicle manufacture, including work hardening (under various strain paths) and bake hardening. These models are evaluated by way of a validation test, where a structure is deformed in a known manner, and FEM modeling is used to predict the deformation behavior. Accurate validation tests have been developed and documented for bulk materials, but they need to be developed for sheet materials.

Finite element codes. The FEMs must accurately predict the stress, strain and strain rate for each element in the model under sometimes violently changing loading conditions and under conditions of rapid buckling. This requires robust finite element codes, meshing practices, and elements. The introduction of strain-rate-sensitive properties adds new complexity to these calculations, which must be understood. In the end, guidelines are needed for setting up and running FEM crash analyses, which maximize the accuracy of all aspects of the model, including the material properties.

The objective of the project is to develop numerical modeling guidelines for strain-rate-dependent materials in crashworthiness computations. The scope of the project is to study specific, experimentally well-defined structural problems in automotive impact. The dynamic loading problems will be modeled using diverse combinations of modeling approaches (sub-models) that are essential in describing strain-rate sensitivity in computational simulations. Sub-models to be examined include finite element formulations, constitutive materials models, contact conditions, and so on. The trends, influences, and direct effects of the modeling techniques employed will be identified and documented. The relative significance of the sub-models employed will be established, particularly in relation to the strain-rate effect resulting from the material constitutive models.

In particular, the project will

- Evaluate the behavior of the available strain-rate material models on the automotive component level
- Examine the constraints of FEM technology used in explicit time integration codes with respect to strain-rate modeling
- Use data from existing experimental programs to verify modeling methodology
- Implement, modify and utilize strain-rate-dependent material models into a crash simulation code if the existing model implementation is not available
- Help define new experimental programs to support the modeling of material strain-rate effect
- Utilize supercomputing resources to provide sufficient model resolution and fidelity
- Develop an Internet-based, password-protected project site for project progress reporting, verification and dissemination of results
- Define the requirements for transfer of project developments to different commercial FEM codes

Modeling of Strain Rates in Steels

The response of metals under a multiaxial state of stress and varying strain rates is still far from being described by a unified theory. During multi-axial large plastic deformations, material undergoes significant changes of microstructure and texture that lead to changes in material properties on the macro level. From a practical standpoint, the best that we should hope for is that the selected material model is applicable to the ranges of loading and deformation for the problem at hand. In tubular crush devices that are used as energy absorbers in vehicles, metallic sheets are subjected to large, localized deformations that organize into global collapse mechanisms. In the past, analytical and semi-analytical methods were the only feasible routes for analyzing progressive structural collapse. These methods start from kinematics assumptions and idealization of material response, and derive the expressions for the structural response based on energy minimization principles. The advances in FEM [3] and in computational power have provided a framework within which the constraints of analytical approaches can be largely eliminated, and radically more complex deformation and material response can be modeled.

Body-centered cubic (BCC) metals, such as steel, exhibit high strain-rate sensitivity that can increase flow stress several times over the quasi-static values for deformation rates of interest in automotive impact.[4] Therefore, incorporation of strain-rate sensitivity into material models for crashworthiness analysis is the first logical step in improving the accuracy of analysis.

Currently, the most widely used approach to modeling strain-rate-sensitive material in tube crushing is to use isotropic plasticity models with rate-sensitivity components that have moderate requirements on the experimental program. The isotropic plasticity models that incorporate strain-rate sensitivity and that were recently reported for tube crush simulations are the Johnson-Cook model,[5] the Zerilli-Armstrong model,[6] and the piecewise linear strain-rate-sensitive material model. The models are appealing because they have been implemented in commercial codes used for crash simulations and have a limited (less than 7) number of material parameters that must be determined by experiments. In the case of the piecewise linear plasticity model, effective strain-stress curves are directly fed into the material models and require the least amount of effort for model development.

The standard practice for extracting material parameters is to perform experiments with uniaxial loading configurations. The ductility of uniaxially tensile-loaded specimens is limited by the geometric instability; and for automotive steel sheets, it limits the magnitude of uniform plastic strains to about 25%. However, the strains that are measured and modeled during tube crushing far exceed these values and can reach 100%. The physical reality of such large deformations is not in question. The biaxial loading and bending provides additional stability that allows the utilization of a material's strain hardening, ductility, and corresponding large energy dissipation.

Modeling of tube folds is the area where material models and finite element formulations are intrinsically linked. In the case when we want the kinematics of the deformation to be accurate, the resulting maximum plastic strains are approximately four times the experimental strain range. On the other end of the spectrum, when the finite element resolution is coarse, the material model extrapolation is not a concern because the plastic strains get smeared over larger volumes and are within the range of uniaxial experiments. The problem is then the ability to correctly describe the kinematics of the problem. In models based on shell finite elements, which are currently the standard way of modeling tubular crush, the issue of element resolution boils down to the number of bi-linear shell elements that are used for modeling of plastic folds. The curvature of plastic folds is of the order of the material thickness; and for the kinematic representation of the fold to be accurate, element length should approach shell thickness. It is at this point that standard shell theory tends to break down, and material models extrapolate far outside the experimentally verified range.

In both of these scenarios, in order to match the experimental results with models, material parameters are commonly modified. While the practice may be appalling to material scientists, the reason for modification in one case is to compensate for the inability to represent local deformations; in the other case, the reason is to account for the significance of large strain regions for which experimental data are not available. The flexibility of computer programs now even provides methods for definition of optimization problems where the material parameters are determined so that they result in the optimal match between the structural simulations and experiments. These approaches are inadvertently linked to specific structural problems, FEM mesh configurations, and loading situations. The best that we can hope for is that the deformation modes and characteristic deformation mechanisms remain essentially the same, in which case we are able to cover a certain family of problems.

In addition to better representation of the material response, strain-rate sensitivity has an added benefit of promoting the computational stability of simulations. The pathological mesh sensitivity that is characteristic of modeling of localized deformations is alleviated when strain-rate-sensitive materials are used. Material rate dependence introduces a length scale that is proportional to the length of propagation of elastic waves. This length scale has a physical character as opposed to the length scale imposed by the element mesh spacing that governs the localization response when a rate-independent material model is used.

Impact Simulations using Johnson-Cook and Zerilli-Armstrong Models

Strain rates in automotive impact problems are reported to be less than 1000/s. Most of the published studies on automotive-related impacts with strain-rate-sensitive material models employ Johnson-Cook and Zerilli-Armstrong material models. These two models have been developed for modeling metals subjected to large strains, high strain rates, and high temperatures occurring during projectile impact. The popularity of the Johnson-Cook model is promoted by its simplicity and ease of implementation. The Zerilli-Armstrong model, on the other hand, is appealing because of its roots in the dislocation mechanics and the possibility for physical interpretation of the model parameters.

The strain-rate sensitivity of BCC materials is attributed to the rate-controlling mechanism of the thermal component of the flow stress. Because the activation volume for BCC metals is considered to be constant, the increase in strain rates should theoretically be described by an upward translation of the quasi-static strain-stress curve. Experimental results show there is also a corresponding reduction of the strain-hardening rate so that the parallel strain-stress curves can be viewed as a limit case.

The Johnson-Cook model is expressed in a multiplicative form of strain, strain-rate, and temperature terms:

$$\sigma = (A + B\varepsilon^n)(1 + C\ln\dot{\varepsilon})(1 - T^m) \qquad (1)$$

The consequence of the multiplicative form is that the strain hardening rate at a certain strain will increase when the strain rate increases. The strain-stress curves for increasing strain rates will tend to “fan out,” which is in contrast to the experimental results for steel sheets. The material parameters can be selected so that this differential hardening is insignificant and curves become parallel in the limit case. However, the overall feature still cannot accommodate the global trend of reduction of the strain hardening rate with increasing strain rate that is observed in experiments.

The Zerilli-Armstrong model for BCC metals is written in an additive form akin to the standard materials science expression for flow stress as an aggregate of various strengthening mechanisms:

$$\sigma = c_0 + B_0 e^{-(\beta_o - \beta_1 \ln \dot{\varepsilon})T} + K\varepsilon^n \tag{2}$$

The strain hardening for various strain rates is constant; therefore, the corresponding strain-stress curves are parallel. The correlation of yield with the Zerilli-Armstrong model has been shown to be very good. However, the inherent inability to model the dependence of strain hardening on strain rate will unavoidably lead to adjustment of the element discretization and selection of specific strain intervals for material parameter derivation, as it does for the Johnson-Cook model. Because of the inability of the models to match the experimental results across all ranges of strain and strain rate, these models were not selected for the detailed tube crush simulations that follow.

It was shown that the strain hardening has a crucial role in development of a collapse pattern and bifurcation between the modes in tube crushing. Material models such as Bodner-Partom and Khan-Liang can offer added flexibility for modeling of strain hardening, but they have not yet been implemented in commercial crash codes. In the future work, these models will be implemented, and their performance for the modeling of progressive crush problems will be evaluated.

Impact Simulations using Piecewise Linear Plasticity Model

Another popular approach for modeling of strain-rate sensitivity is to use a tabulated form of strain-stress curves for different strain rates. The resulting stress in simulations is interpolated between the known values of strains and strain rates. The blessing and the curse of this approach is that the experimental data fit the model exactly, and that any testing artifacts or errors contained in the experimental data will be carried over to the simulations. The highest strain rate in experimental data acts as a saturation plateau for strain-rate effects.

Single Finite Element Under Constant Strain-Rate Loading

Figure 1 shows a numerical test configuration for evaluation of a material constitutive model.

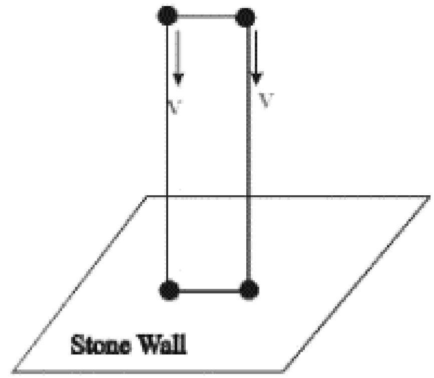

Figure 1. One-element strain-rate test.

The imposed velocity that will result in a constant strain rate is

$$v = \dot{\varepsilon} L_0 \exp(\dot{\varepsilon} t) \tag{3}$$

where L_o denotes element length and t denotes time. The experimental true plastic strain-stress data under various strain rates for drawing-quality, special-killed steel were provided by the Auto/Steel Partnership and are shown in Figure 2.

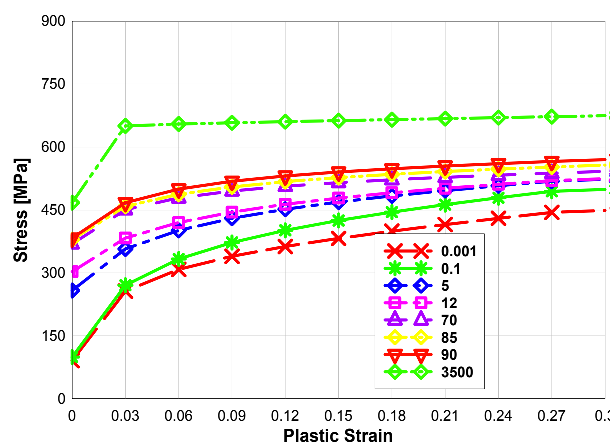

Figure 2. Drawing-quality, special-killed steel material data.

Simulation of the constant strain-rate test shows the exact overlap of experimental data and simulation in Figure 3.

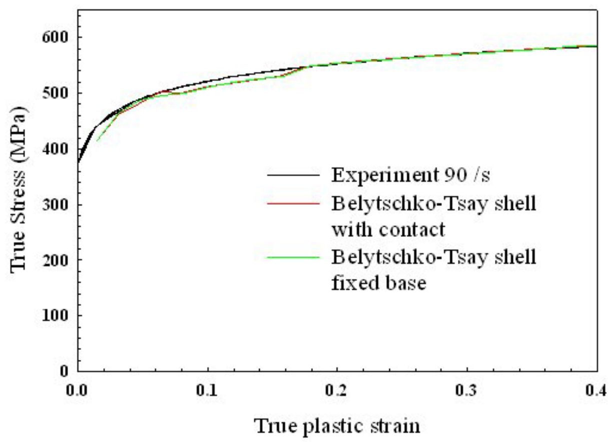

Figure 3. Plastic strain evolution in the element.

The only variation comes from the effects of contact conditions on the base; but, as can be seen, they can be neglected so long as the time increment and contact penalty parameters meet the stability criteria.

The important question in implementation of the piecewise linear plasticity model is the maximum strain rate for which the stress-strain data should be tested. Another is the number and arrangement of curves across the strain-rate range. In Figure 2, it is clear that one curve per order of magnitude would suffice. The material model uses linear interpolation between the curves, whereas it has been shown that the stress increment relation is logarithmic. However, when sufficient experimental data are available, the effects of linear interpolation should not be significant. One possible improvement over the existing model is to develop a logarithmic fit for the available curves with respect to strain rate and then extrapolate it outside the plateau value.

Strain-Rate Calculation in FEM Simulations

Crashworthiness simulations are almost exclusively done using FEM programs with explicit time integration.[7] The explicit time integration algorithms are only conditionally stable, meaning that the integration time step must be smaller than the time for a disturbance to travel across the smallest finite element in the model. For example, for steel and a finite element characteristic length of 5mm, the condition requires that the time step be smaller than 1 microsecond. The material constitutive relations are evaluated at each computational time increment. The strain rates are also calculated at this smallest time scale where wave propagation effects are important. The experimental data are determined on the higher time scale where the wave effects are not measured except for the highest-rate experiments. Therefore, it is important to determine the values of the strain rates in the explicit time integration calculations in order to define the extent of the material characterization experimental program. It is also important to investigate the effects of FEM formulations and element discretization on the magnitudes and distribution of plastic strains and strain rates in the areas of large plastic deformations that determine the crush mode. So far, no studies have been reported on the analysis of strain-rate calculations in explicit FEM programs.

We have developed a set of programs that take the data from the FEM computations at each time increment, process them on the fly, and store results for further analysis. The development of new programs was necessary because storing information for each time increment is not feasible.

The results show a clear dependence of the strain-rate calculations on the element discretization. Strain-rate calculations are shown to be much more sensitive to FEM formulations than are plastic strains. The magnitudes of strain rates are of the order 10^3/s, so that the experimental maximum strain rate of that order is required for the material model development. Currently, we are analyzing distributions of strains and strain rates in the area of tube folds for different discretizations. The simulations will be compared with tube crush experiments on the ORNL Intermediate Strain Rate Machine.

Axisymmetric Crushing of a Steel Tube

The axisymmetric crush mode energy has larger energy dissipation than antisymmetric modes and would be preferred for energy-absorbing devices. The mode is difficult to achieve in practical automotive impact problems, but it is useful for studying interplay between materials and structures in the crush problems. The axisymmetric folding is assisted by the localized deformations driven by low strain hardening. The new high-strength steels for automotive applications, such as dual-phase steel, exhibit very high strain hardening, which tends to precipitate bifurcation into antisymmetric collapse modes. This makes the modeling of high-strength steel more sensitive to details of material models and element formulations.

The objective of our study on axisymmetric crushing was to investigate the trends and magnitudes in strain rates as calculated by the finite element program. As stated earlier, this information is important in deciding the extent of the experimental program for derivation of material models. Contrary to the plastic strains that tend to monotonically increase, strain rate exhibits intermittent bursts of activity in the area of evolving folds. The effects of different cutoff strain rates on the evolving deformation are currently under investigation and will be reported in a journal publication.

A typical setup for the tube crush experiment is shown in Figure 4. The tube is clamped on the bottom and is impacted on the top with a moving mass. The experimental configuration from ref. 8 was used. The tube is made of mild steel that is modeled using the piecewise linear plasticity model with strain rate tabulated curves as shown in Figure 2. The tube thickness is 1.2 mm, the radius is 28 mm, and the length is 180 mm.

The axisymmetrically crushed tube is shown in Figure 5.

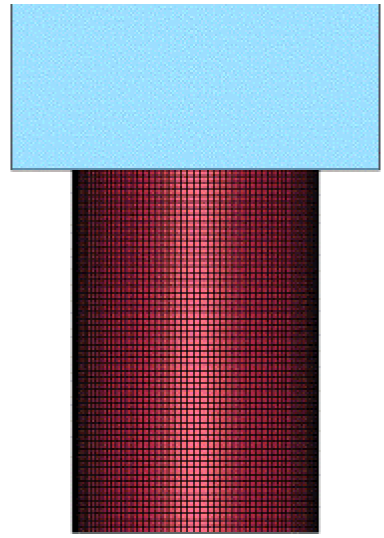

Figure 4. Tube crush setup.

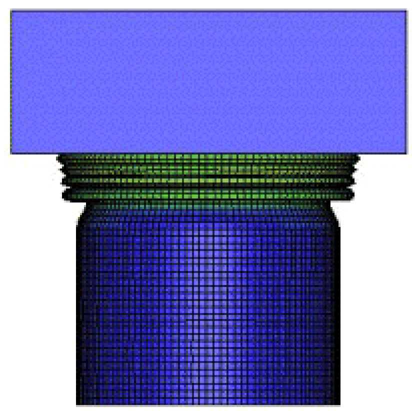

Figure 5. Axisymmetric tube crush.

Effects of Strain Rate on Tube Crush

Because of the axisymmetry of the problem, we can simplify the problem by analyzing a segment of the tube. The tube is modeled by a 2°-wide cylinder segment with symmetry boundary conditions imposed on the radial sides. Figure 6 shows the deformed tube segment for simulations using material with and without strain-rate sensitivity. It is clear that the material strain-rate sensitivity is an important effect and must be used for crush simulations.

Effects of Element Discretization

In order to investigate the effect of element discretization on the accuracy of the FEM simulations, we used 23, 46, 92, and 160 elements along the segment length. Figure 7 shows final deformation of the tube for different element discretizations. The case when 23 elements are used across the tube length is apparently too coarse to describe the resulting crush mode. As a consequence, when the entire circumference of the tube is modeled, the crush mode will not be axisymmetric or regular. For the case when 46 elements are used along the tube length, the general features of the crush are acceptable but are not in agreement with the experimental results. For 92 and 160 elements, the results are in

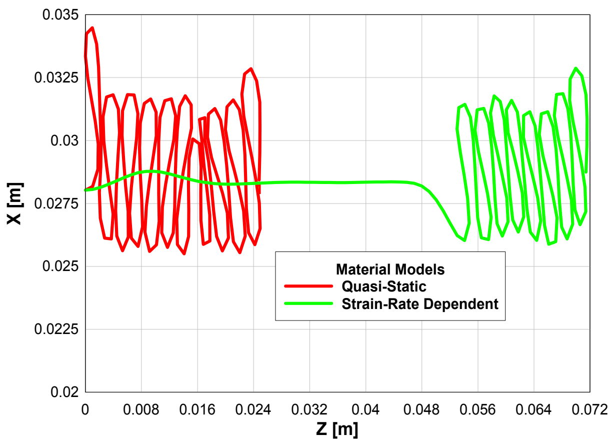

Figure 6. Deformed configurations for different material models.

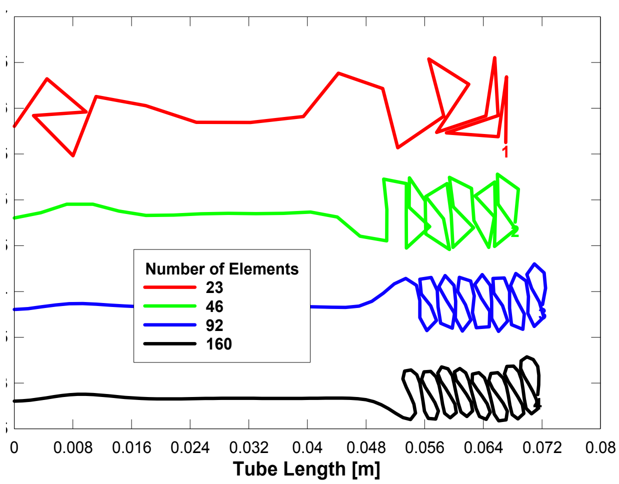

Figure 7. Deformed configurations for different FEM element discretizations.

agreement with experiments where the finest mesh shows the smooth deformation characteristic of the tube crush.

However, when 160 elements along the length are employed, the element length is of the order of element thickness; and the shell element theory shows certain deficiencies for modeling of large bending, as shown in the following figures. The curvature of the tube fold is of the order of its thickness, and the standard shell element theory with small strain assumptions is not able to model the strain fields correctly. The curvature along the undeformed length of the tube for different element discretizations is shown in Figure 8.

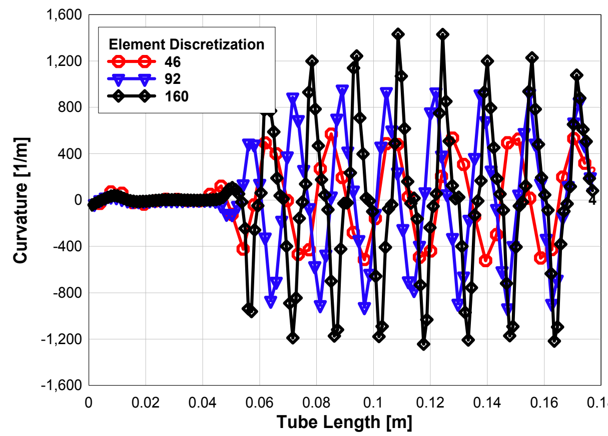

Figure 8. Curvature of the tube for different element discretizations.

The calculated curvature for the finest element mesh is larger than the expected value. However, experimental measurements were not available for the curvature measures. The experiments in FY 2002 will provide information on deformation curvatures, and the data will be used for FEM validation.

Effects of FEM Element Formulations on Tube-Simulated Tube Deformation

Simulations have shown that the FEM shell formulation has significant influence on the predicted mode of deformation. Shell formulations in the FEM program LS-DYNA3D [7] were used for different tube configurations, and it was shown that the fully integrated shell element based on Bathe-Dvorkin formulation has superior performance compared with other element formulations. This element formulation gives a good approximation of warping and transverse shear fields and predicts the correct mode of deformation with increasing mesh resolution. The performance was analyzed on existing and new benchmark problems and on the full-tube crush simulations.

Figure 9 shows the antisymmetric mode of deformation that was predicted by an FEM shell element formulation commonly used in FEM crush simulations. This formulation predicts the axisymmetric mode for coarse element discretizations; but for fine discretizations, the mode bifurcates.

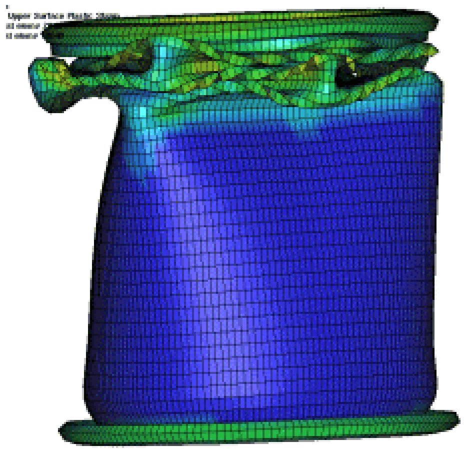

Figure 9. Crush simulation with Hughes-Liu shell element formulation.

When the Bathe-Dvorkin shell element formulation is used, the mode remains the same with increasing mesh density, as shown in Figure 10. The element is computationally more expensive than the standard reduced integration shell finite elements; for the detailed models of the crush, the increase in computational cost is offset by the increase in accuracy of the crush mode prediction.

Effects of Strain Hardening and Strain-Rate Sensitivity on Tube Crush

One of the objectives of the project is to evaluate the influence of various material parameters on tube crush. The effect of strain rate and strain hardening on tube crush has been analyzed. It has been shown that material hardening has a dominant effect on fold length, while strain-rate sensitivity influences the number of folds that is created. Strain hardening and strain-rate sensitivity both have relatively equal importance for onset of bifurcation. These findings are illustrated in Figures 11–16. Figures 11 and 12 show the tube crush for strain-rate sensitive material with strain hardening.

When material does not strain-harden, the fold lengths are reduced, as shown in Figures 13 and 14. The antisymmetric mode after mode bifurcation has more periods than the previous case. The onset of mode bifurcation is similar in both cases.

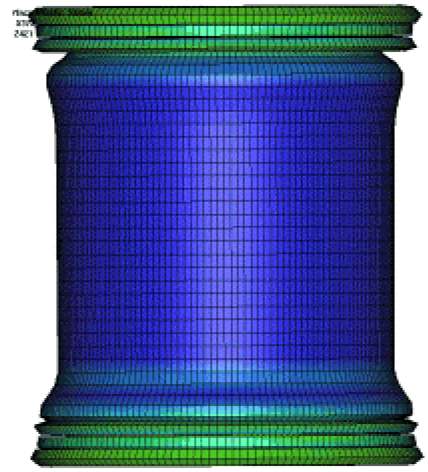

Figure 10. Crush simulation with Hughes-Liu shell element formulation.

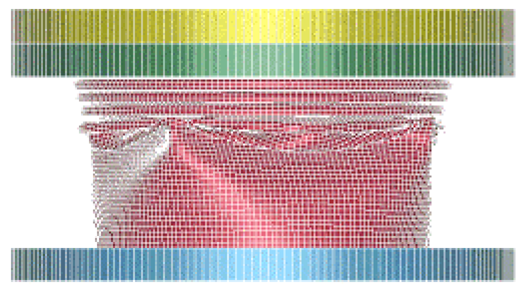

Figure 11. Crushed tube for strain-rate sensitive material with strain hardening.

Figure 12. Fold profile for strain-rate sensitive material with strain hardening.

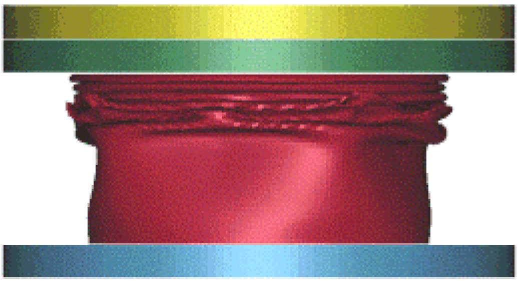

Figure 13. Crushed tube for strain-rate sensitive material without strain hardening.

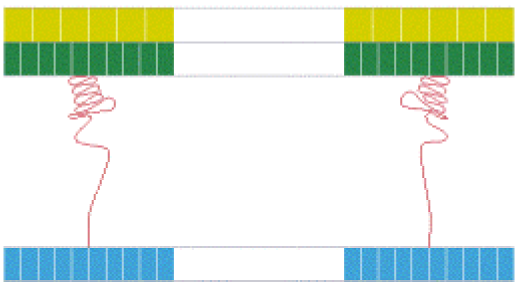

Figure 14. Fold profile for strain-rate sensitive material without strain hardening.

Figures 15 and 16 show tube deformation for rate-insensitive material without strain hardening. The model shows no bifurcation from axisymmetric into antisymmetric crush mode.

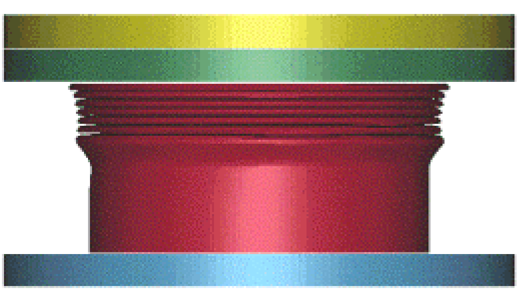

Figure 15. Crushed tube for strain-rate insensitive material without strain hardening.

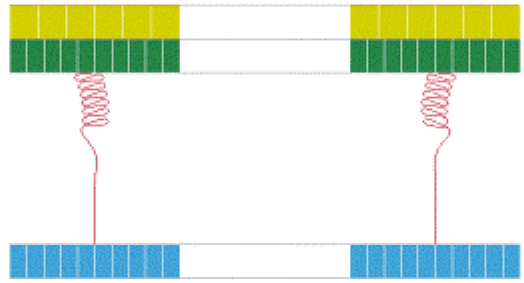

Figure 16. Fold profile for strain-rate insensitive material without strain hardening.

Future Work

The work on the project in FY 2002 will focus on two aspects: development of modeling guidelines for modeling of steel tube crushing and the support of the new intermediate-strain-rate experiments. The experiments will provide the crush data for high-strength, low-alloy steels and dual-phase steels. These two high-strength steels have comparable yield stresses but drastically different strain hardening characters.

On the modeling front, we will analyze transitions from axisymmetric to antisymmetric mode in progressive crushing and evaluate the performance of the FEM shell formulations for modeling of transition problems. We will analyze the effects of the FEM shell element discretizations and formulations on model accuracy through computational simulations and validations using experimental data.

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Appendix A. ACRONYMS AND ABBREVIATIONS

A/SP	Auto/Steel Partnership
AAT	Advanced Automotive Technology
ACC	Automotive Composites Consortium
AHSS	advanced high-strength steel
ALCOA	Aluminum Company of America
ALM	Automotive Lightweighting Materials
ALTC	Automotive and Light Truck Committee
AMD	Automotive Metals Division
ANL	Argonne National Laboratory
APAW	aluminum plasma arc welding
ASTM	American Society for Testing and Materials
BHF	blank holder force
BIW	body-in-white
CAE	computer-aided engineering
CAFE	Corporate Average Fuel Economy
CFC	carbon-fiber composite
CRADA	cooperative research and development agreement
CWRU	Case Western Reserve University
DOE	U.S. Department of Energy
DPF	direct powder forging
E	experimental
EGN	exfoliated graphite nanostructure
ELV	end-of-life vehicle
EMF	electromagnetic forming
FE	finite element
FEA	finite element analysis
FLD	forming-limit diagram
FY	fiscal year
GASP	grazing angle surface polarimeter
GM	General Motors
HDPE	high-density polyethylene
HSLA	high-strength low alloy
HSS	high-strength steel
HTML	High Temperature Materials Laboratory
HVSC	Huron Valley Steel Corporation
ICS	Industrial Ceramic Solutions
IIW	International Institute of Welding
IRDI	Industrial Research and Development Institute
ITP	International Titanium Powders
LANL	Los Alamos National Laboratory

LBNL	Lawrence Berkeley National Laboratory
LCCF	low-cost carbon fiber
LIBS	laser-induced breakdown spectroscopy
LLDPE	linear low-density polyethylene
LLNL	Lawrence Livermore National Laboratory
MAP	microwave-assisted plasma
MMC	metal matrix composite
MMCC	Metal Matrix Cast Composites
MPa	megaPascal (a unit of pressure)
NDE	nondestructive evaluation
NDT	nondestructive test
NRCAN	Natural Resources of Canada
OAAT	Office of Advanced Automotive Technologies
OEM	original equipment manufacturer
OHVT	Office of Heavy Vehicle Technologies
OIT	Office of Industrial Technologies
ORNL	Oak Ridge National Laboratory
OTT	Office of Transportation Technologies
P4	programmable powder preform process
P&S	press and sintering
PAN	polyacrylonitrile
PC	personal computer
PE	polyethylene
PID	proportional integral derivative
PM	powder metallurgy
PMC	polymer matrix composite
PMPRA	powder metallurgy particle-reinforced aluminum
PNGV	Partnership for a New Generation of Vehicles
PNNL	Pacific Northwest National Laboratory
pp	polypropylene
PQTI	Plasma Quench Titanium, Inc.
PRA	particle-reinforced aluminum
PVB	polyvinyl butyral
PVC	polyvinyl chloride
R&D	research and development
ROI	return on investment
RVE	representative volume element
SA/RD	sales and administrative/research and development
SAMPE	Society for the Advancement of Material and Process Engineering
SCMD	structural cast magnesium development
SEM	scanning electron microscope
SNL	Sandia National Laboratories

SOM	solid-oxygen-conducting membrane
SRIM	structural reaction injection molding
TCM	technical cost model
TDM	Troy Design and Manufacturing
TGA	thermogravimetric
USAMP	U.S. Automotive Materials Partnership
USCAR	U.S. Council for Automotive Research
UTS	ultimate tensile strength
YSZ	yttria-stabilized zirconia

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- Automotive Lightweighting Materials
- Automotive Propulsion Materials
- Fuels for Advanced CIDI Engines and Fuel Cells
- Spark Ignition, Direct Injection Engine R&D
- Combustion and Emission Control for Advanced CIDI Engines
- Fuel Cells for Transportation
- Advanced Technology Development (High-Power Battery)
- Batteries for Advanced Transportation Technologies (High-Energy Battery)
- Vehicle Power Electronics and Electric Machines
- Vehicle High-Power Energy Storage
- Electric Vehicle Batteries R&D

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