

Evaluation and Characterization of Lightweight Materials: Success Stories from the High Temperature Materials Laboratory (HTML) User Program

DOE 2010 Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

Dr. Camden Hubbard
HTML User Program

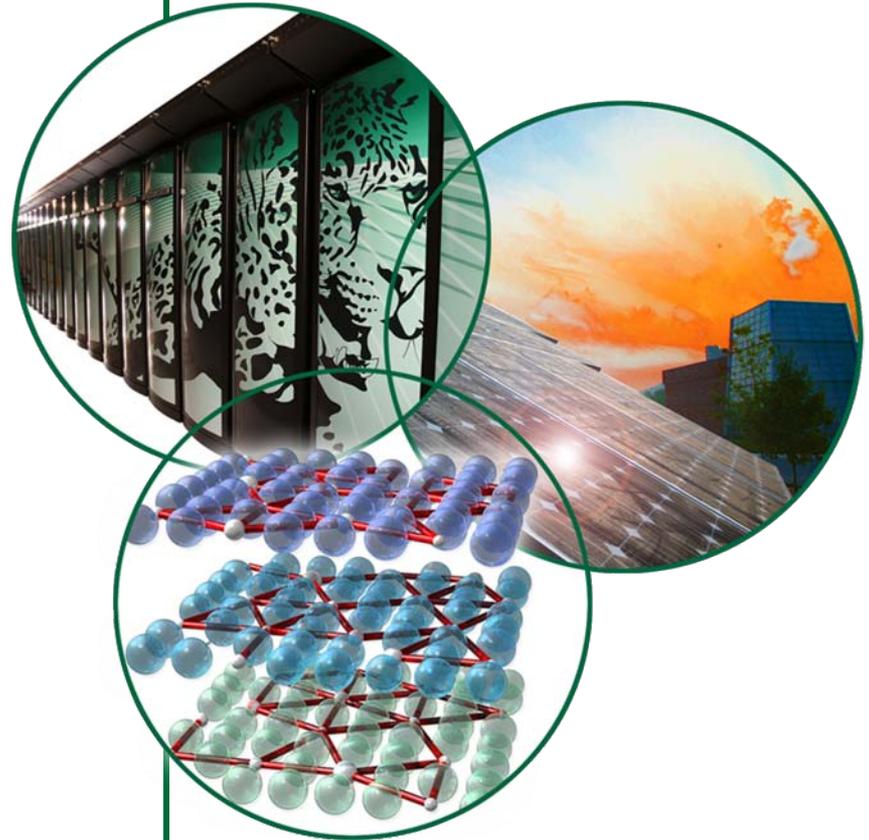
Materials Science and Technology Division
Oak Ridge National Laboratory

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and Renewable Energy, Office of Vehicle Technologies



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The HTML User Program: Background

- The HTML is a National User Facility that supports the missions of DOE, EERE and the Vehicle Technologies Program in particular, by working with industry, universities, and other national laboratories to develop energy-efficient technologies that will enable the U.S. to use less petroleum. The HTML is organized into six user centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.
- Access to the HTML User Program is provided through the HTML User Program proposal process. Research proposals are reviewed by a committee and approved based on scientific merit, relevance of the proposed research to the mission of DOE's Vehicle Technologies Program, and feasibility. Projects have a well-defined scope, and research is completed within 24 months and normally involves one or more user visits to the HTML.
- Both nonproprietary and proprietary research is conducted within the HTML User Program. There are generally no charges for nonproprietary research projects, and users conducting nonproprietary research must agree to submit research results for publication in the open, refereed literature. A nonproprietary project is complete when the results are published in the open literature and/or presented at a professional conference. For proprietary research, the user owns the research data, and all costs at the HTML are paid by the user based on DOE guidelines for ORNL costs.

The HTML User Program – FY2009 Activity

During FY2009, the HTML User Program collaborated with 11 companies, 14 universities, and 3 national laboratories on 41 user projects addressing critical technical barriers to achieving the goals of DOE's Vehicle Technologies Program. There were 107 researchers who visited the HTML for a total of 562 days to conduct experiments.

The HTML User Program FY2009 budget was \$5,066,946 and was allocated as follows:

- Capital equipment: \$514,025
- Operations: \$4,552,921

Users cost-share their HTML user projects through:

- 1) direct involvement with HTML staff members during the development of the user project;
- 2) funding their travel to the HTML to perform research;
- 3) cost of materials provided by the user or the research performed prior to the user project;
- 4) collaboration with HTML staff members to analyze the data and publish the results.

The HTML also supports the education and preparation of a new generation of scientists and engineers. During FY2009, students and professors from 14 universities participated in the HTML User Program. Four of those students earned their Ph.D. degree and three earned their M.S. degree based in part on research they conducted through the HTML User Program.

Relevance to the VT Program

- The Vehicle Technologies Program funds the operation of the HTML User Program to maintain world-class expertise and instrumentation capabilities for materials characterization to work with industry, universities and national laboratories toward the goals of the Vehicle Technologies Program.
- The HTML User Program capabilities also support the activities of the Vehicle Technologies Program's subprograms in Lightweight Materials, Propulsion Materials, Energy Storage, and Thermoelectric Conversion at the Oak Ridge National Laboratory.
- This poster presentation highlights **six** of the 41 user projects managed by the HTML User Program during FY2009. The user projects in this poster presentation address critical barriers for the market viability of advanced lightweight materials for automotive and commercial vehicle applications. These include cost, design data, joining, manufacturability, and performance.

ORNL Polymer Matrix Composites Group

“Development of next generation low-cost carbon fibers: Surface and microstructure analysis”



Timeline

- Start date: 5/5/09
- End date: 5/1/11
- % complete: 75%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Cost
- Design Data
- Manufacturability

Collaborators

- **ORNL Users:** Soydan Ozcan and Felix Paulauskas
- **HTML Staff:** Rosa Trejo and Andrew Payzant

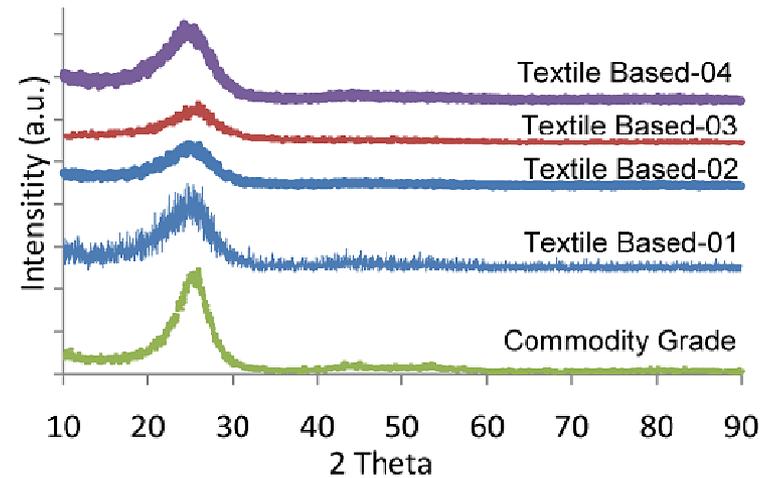
ORNL Polymer Matrix Composites Group: User project background of study



Research problem: To evaluate the microstructure of carbonized fibers obtained from various precursors and processing conditions.

Technical approach: X-ray diffraction and nanoindentation were performed to determine the elastic modulus and crystallographic characteristics of carbon fibers.

Implications: High cost of carbon fibers is the greatest single barrier to their use in automotive and commercial vehicle applications. Also, adequate design data are necessary for the widespread application of these materials.

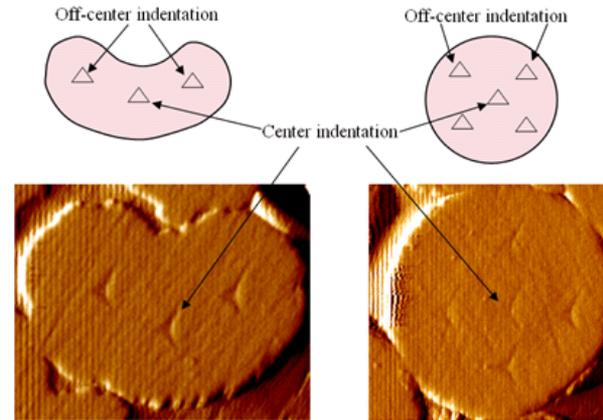


Wide-angle x-ray diffraction patterns of various textile-based carbon fibers compared with commodity grade commercial carbon fiber.

ORNL Polymer Matrix Composites Group Accomplishments

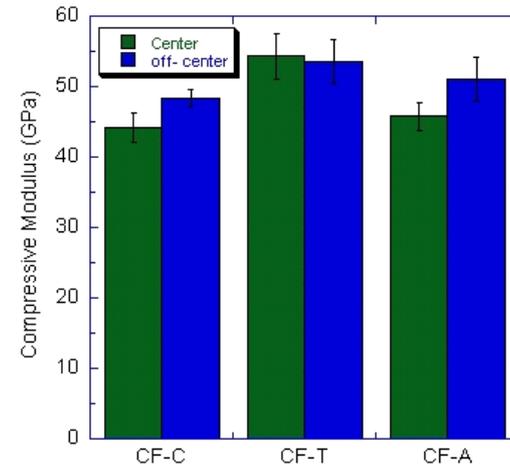


- The compressive elastic modulus of three carbon fibers from different precursors was determined from nanoindentation tests. Indents were made at on-center and off-center (close to fiber edges) locations of the fiber's cross-section.
- Elastic modulus values were found to be lower at the center of the fiber, which is believed to result from differences in the microstructure that arise from the kinetics of oxidation.
- Analysis of the X-ray diffraction data suggest that the elastic modulus of carbon fibers is a function of the orientation of crystallites but independent of the length and thickness of crystallites.
- The information obtained from this project will be used to optimize accelerated processing of carbon fiber precursor conversion to significantly reduce the cost of fiber production.



Schematic and images taken after indentation test, kidney shape textile-based carbon fiber (CF-T; left) and aerospace grade carbon fiber (CF-A; right).

Compressive elastic modulus results for commodity (CF-C), textile and aerospace grade fibers.



Plasan Carbon Composites

“Testing of Composite Braided Beams for Automotive Door System”



Timeline

- Start date: 7/20/09
- End date: 7/15/11
- % complete: 85%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Cost
- Design Data
- Manufacturability

Collaborators

- **Users:** Andy Rich (Plasan Carbon Composites)
Phil Lariviere (A&P Technologies)
- **HTML Staff:** Don Erdman and Mike Starbuck

Plasan Carbon Composites

User project background of study



Research problem: To quantify the crashworthiness of carbon fiber-reinforced composites for automotive door systems.

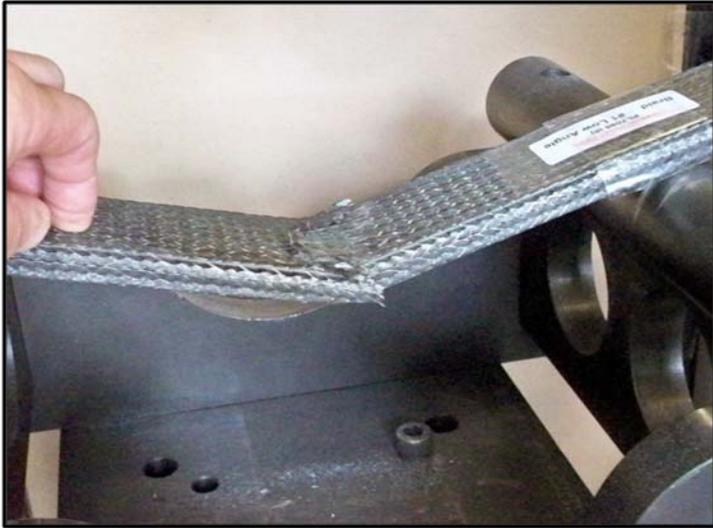
Technical approach: Evaluated the effect of fiber architecture and strain rate on the flexural strength of braided composite structures.

Implications: Adequate design data and methods for the cost-competitive, high-volume production of advanced lightweight materials are necessary for their widespread use in automotive components.



Andy Rich (Plasan Carbon Composites) and Phil Lariviere (A&P Technologies) examine braided composite components prior to testing.

Plasan Carbon Composites User Project Accomplishments



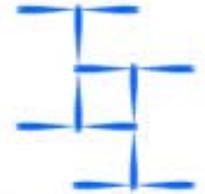
Braided composite after a high-rate impact test.

- Tests were performed on composite beams in 4-pt bending configuration using ORNL's Test Machine for Automotive Crashworthiness (TMAC) at crosshead speeds of 1 mm/s and 8 m/s.
- Sandwich structures consisting of braided panels over foam cores were found to be significantly stronger than plain pultruded composite beams without core.
- Metallic structures were also tested to provide benchmark energy levels associated with currently used metallic components.

Plasan Carbon Composites expects to redesign and optimize their composite beams to take advantage of the lessons learned at the HTML. The results from this user project will further enable the introduction of composite structures in automotive applications.

Innegrity, LLC

“Toughness of high performance Innegra™ S fibers at high strain rates”



Timeline

- Start date: 2/26/09
- End date: 12/31/10
- % complete: 95%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

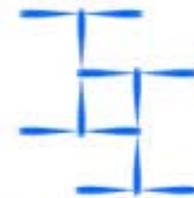
- Cost
- Design Data
- Performance

Collaborators

- **Innegrity Users:** Michael Grah, Loren Chambers, and Brian Follo
- **HTML Staff:** Don Erdman and Barbara Frame

Innegrity, LLC

User project background of study



Research problem: To characterize and understand the mechanical properties of Innegra™ fibers and its composites. Innegra™ S fibers, which are highly oriented polypropylene fibers, are the lightest fibers currently available (0.67 g/cm^3).

Technical approach: Determined the effect of strain rate on the tensile strength of Innegra™ yarns using ORNL's high-rate servohydraulic testing machine. Special grips were developed for these tests.

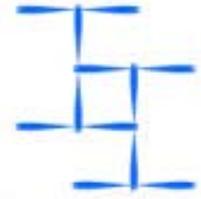
Implications: Adequate design data (material property databases), test methods, and durability data are necessary for widespread applications of advanced lightweight materials.



Michael Grah from Innegrity cuts a test specimen yarn prior to high-rate tensile testing.

Innegrity, LLC

User Project Accomplishments



Brian Follo inserts yarn into the high-rate tensile testing machine.

- Yarns with a gauge length of 25.4 cm were evaluated at strain rates of $0.1s^{-1}$, $1.0s^{-1}$ and $10.0s^{-1}$.
- The tensile strength of Innegra™S yarns was found to increase with strain rate from 8.69 g/denier at $0.1s^{-1}$ to 9.38 g/denier at $10s^{-1}$.
- The toughness of Innegra™S yarns decreased with strain rate.

Innegrity is developing hybrid composites using Innegra™S fibers in combination with carbon, glass and aramid fibers. The mechanical properties of these hybrid composites will be evaluated as a function of strain rate in a future HTML User Program project.

Deere and Company, Moline Technology Innovation Center



“Effect of cooling rate on residual stresses in gray cast iron”

Timeline

- Start date: 4/4/08
- End date: 4/4/10
- % complete: 100%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Adequate design data (material property databases), test methods, analytical tools (i.e., models), and durability data are inadequate for widespread applications of advanced lightweight materials.
- Methods for the cost-competitive production of components for heavy vehicles in volumes of interest to the heavy vehicle industry are not sufficiently well developed.

Collaborators

- **Deere Users:** Eric Johnson, El-Zein Mohamad
- **HTML Staff:** Tom Watkins, Amit Shyam, Josh Schmidlin, Cam Hubbard

Deere and Company, Moline Technology Innovation Center:



User project background of study

Research problem: Build and validate casting modeling computational tools in support of Deere's goal of implementing virtual design, testing and manufacturing of its vehicles by 2015.

Technical approach: Measure the residual stresses in cast ductile iron components using through-thickness mapping at the HTML User Program's neutron residual stress facility (NRSF2) and compare results with model predictions. Two castings were chosen for this study: a transmission housing and a stress-lattice test specimen.

Implications: Data from this research will be used to validate models to enable Deere to increase casting rates in foundries, lower vehicle weight, and reduce production scrap rate.

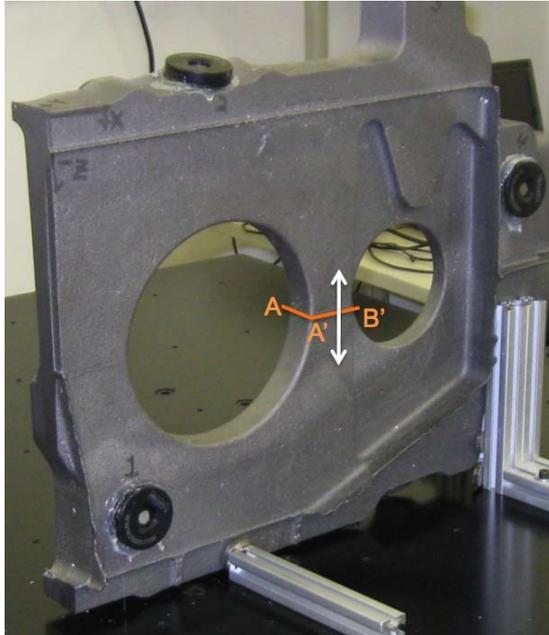


Eric Johnson of John Deere uses HTML's laser scan arm to prepare for neutron diffraction residual stress mapping measurements.

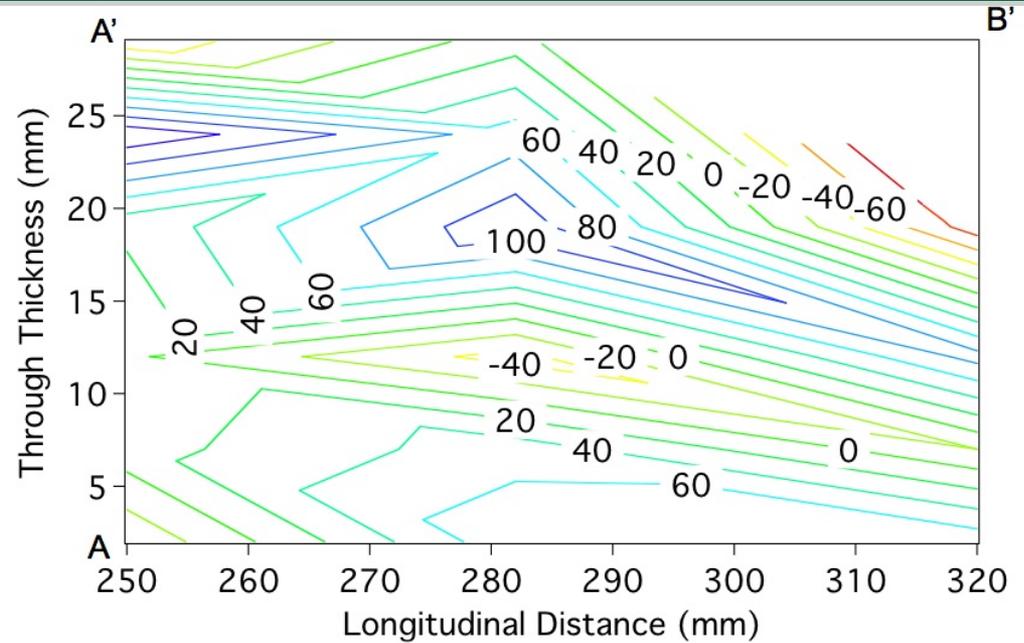


Stress-lattice test specimens

Deere and Company, Moline Technology Innovation Center: User Project Accomplishments



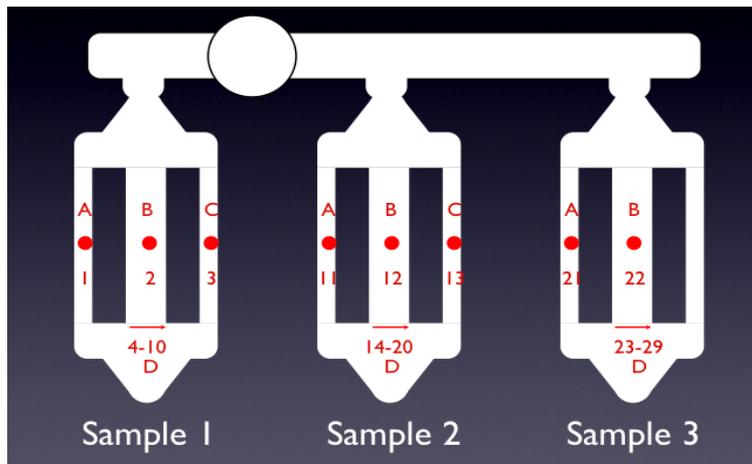
Close-up of transmission housing specimen showing the “knit line” or sample mid plane (plane AA'BB') where two fronts of molten metal meet during casting.



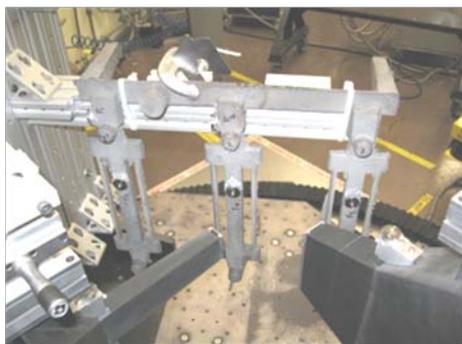
Map of the stresses (MPa) perpendicular to the “knit line”. The upper right corner is a cut-in portion of the casting.

Most of the stresses perpendicular to the “knit line” are tensile, which might result in reduced strength and fatigue life.

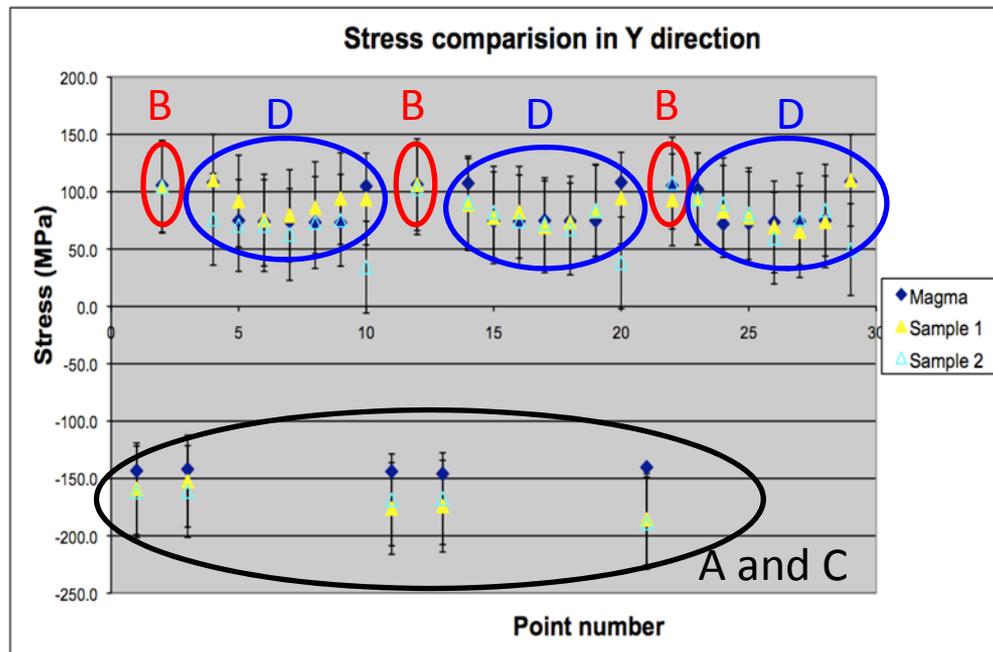
Deere and Company, Moline Technology Innovation Center: User Project Accomplishments



Stress-lattice test specimen measurement locations for neutron diffraction stress mapping.



Sample mounted on the neutron diffractometer.



Measured residual stresses for all three directions validate the stress predictions for the stress-lattice test specimen.

Deere found the correlation of the measured and predicted results for the two castings exceptionally good. Deere is using the validated models to optimize casting operations.

MIT's Impact and Crashworthiness Lab

“In situ neutron diffraction of residual strains and monitoring of martensitic phase transformation in austenitic stainless steel sheet”



Timeline

- Start date: 9/2/09
- End date: 9/1/11
- % complete: 50%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Adequate design data (material property databases), test methods, analytical tools (i.e., models), and durability data are inadequate for widespread application of advanced lightweight materials.

Collaborators

- **MIT Users:** Allison Beese, Scott Speakman
- **HTML Staff:** Camden Hubbard, Josh Schmidlin

MIT's Impact and Crashworthiness Lab: User project background of study



Research problem: To develop models for the transformation kinetics and plasticity laws of 301LN Advanced High Strength Steel (AHSS).

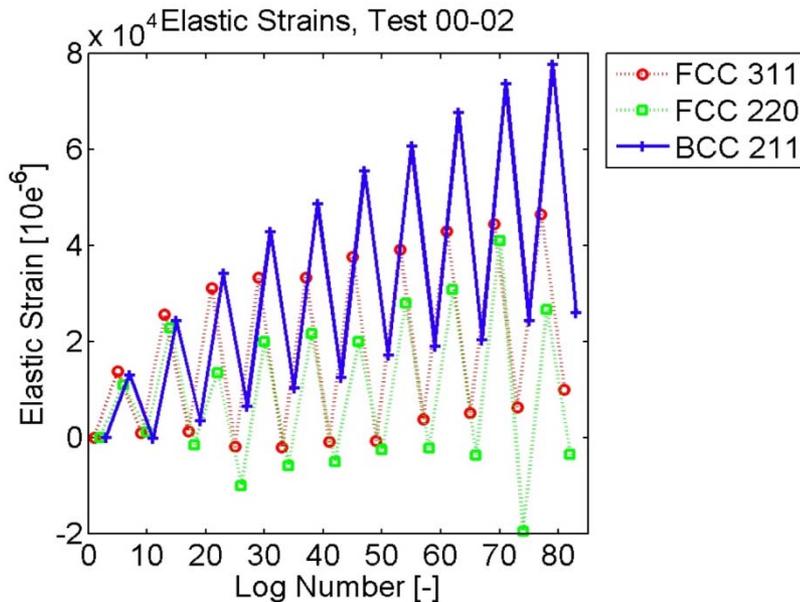
Technical approach: Collect *in situ* neutron diffraction data at the HTML User Program's neutron residual stress facility (NRSF2) to develop models for phase transformation, texture, and stress as functions of applied uniaxial load.

Implications: Constitutive and fracture models for this AHSS could lead to vehicle weight reduction without sacrificing safety.

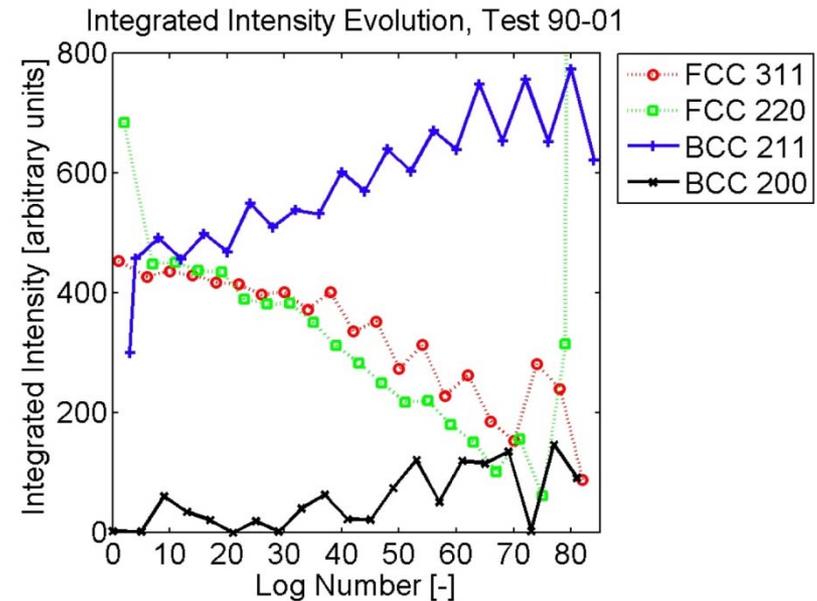


MIT's Allison Beese prepares for an *in situ* loading neutron diffraction measurement at NRSF2 of 301LN sheet samples.

MIT's Impact and Crashworthiness Lab User Project Accomplishments



Measured diffraction elastic strain versus log number for two FCC and the BCC 211 diffraction lines for grains parallel to the loading direction. The load was step-wise increased and then released after each increase, leading to the saw-tooth patterns.



The drop of intensity of the two FCC lines and increase of intensity of the BCC lines provide quantitative data for monitoring transformation of grains parallel to the loading direction as plastic deformation increases. The BCC 200 lines have near zero intensity due to high texture in the as prepared sample (log 0).

The *in situ* neutron results from this investigation are contributing to the advancement of Advanced High Strength Steels for lightweighting vehicular structures.

Michigan State University and Magna Cosma International



“Residual stresses in automotive alloy castings as a function of metal and glass shot peening”

Timeline

- Start date: 5/5/09
- End date: 8/30/09
- % complete: 100%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Design data (material property databases), test methods, analytical tools (i.e., models), and durability data are inadequate for widespread application of advanced lightweight materials.
- Materials needed to achieve performance objectives in specific engine and ancillary components may not exist as durable, reliable, well-characterized and understood materials.

Collaborators

- **MSU Users:** Bryan Kuhr, Alex Ritter, Carl Boehlert
- **Magna User:** Xiaoping Niu
- **HTML Staff:** Camden Hubbard, Tom Watkins, Burl Cavin, Josh Schmidlin

Michigan State University and Magna Cosma International: User project background of study



Research problem: To understand the through-thickness effect of shot peening on the microstructure, residual stresses and fatigue strength of aluminum alloy A356.2

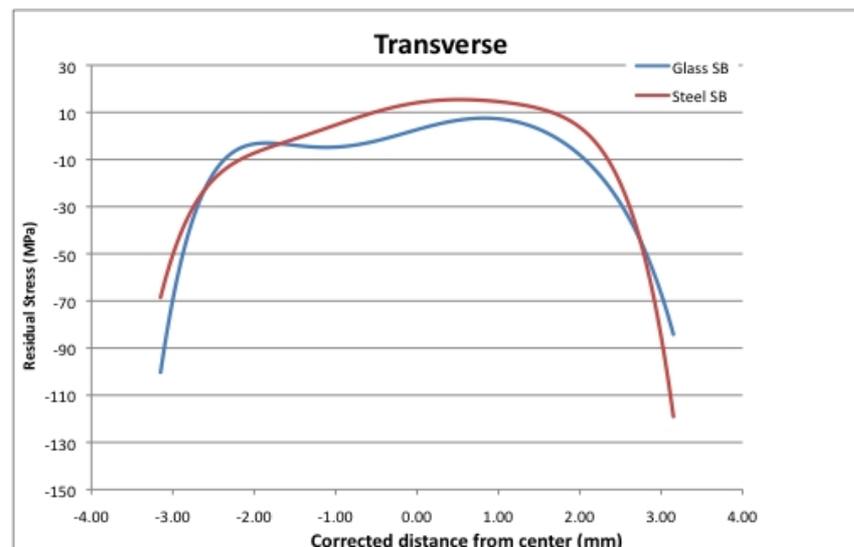
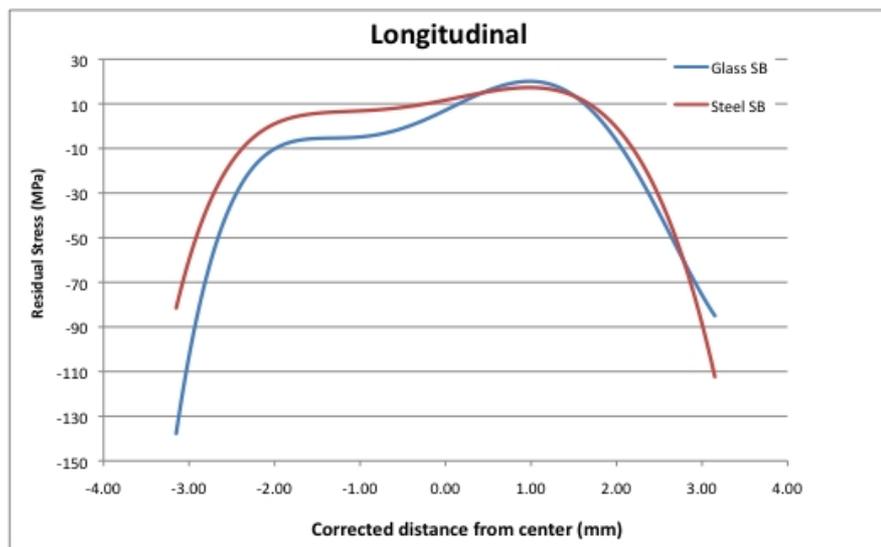
Technical approach: Conduct shot peening with glass and steel shot to constant Almen number, measure fatigue life, and determine the distribution of residual stresses through the thickness of test specimens at the HTML User Program's neutron residual stress mapping facility (NRSF2).

Implications: Understanding if different shot types have similar or different stress distributions with depth will lead to optimizing shot peening to extend the fatigue life of automotive components.



Michigan State's Bryan Kuhr and Alex Ritter align shot-peened samples for neutron residual stress measurements.

Michigan State University and Magna Cosma International: User Project Accomplishments



Longitudinal (left) and transverse (right) residual stress measurement through the thickness of the aluminum plates for glass shot and steel shot blasting (10 Almen, 0.058 mm diameter, 45-90 incidence angle) was demonstrated. Glass and steel shot blasting at a constant Almen level yielded equivalent stress distribution in the 6.2 mm thick aluminum plates. Near surface compressive stresses extend to 1 mm depth.

The depth dependence of residual stresses was measured. This information can be used by design and manufacturing engineers to improve the fatigue resistance of lightweight vehicular components.

Summary

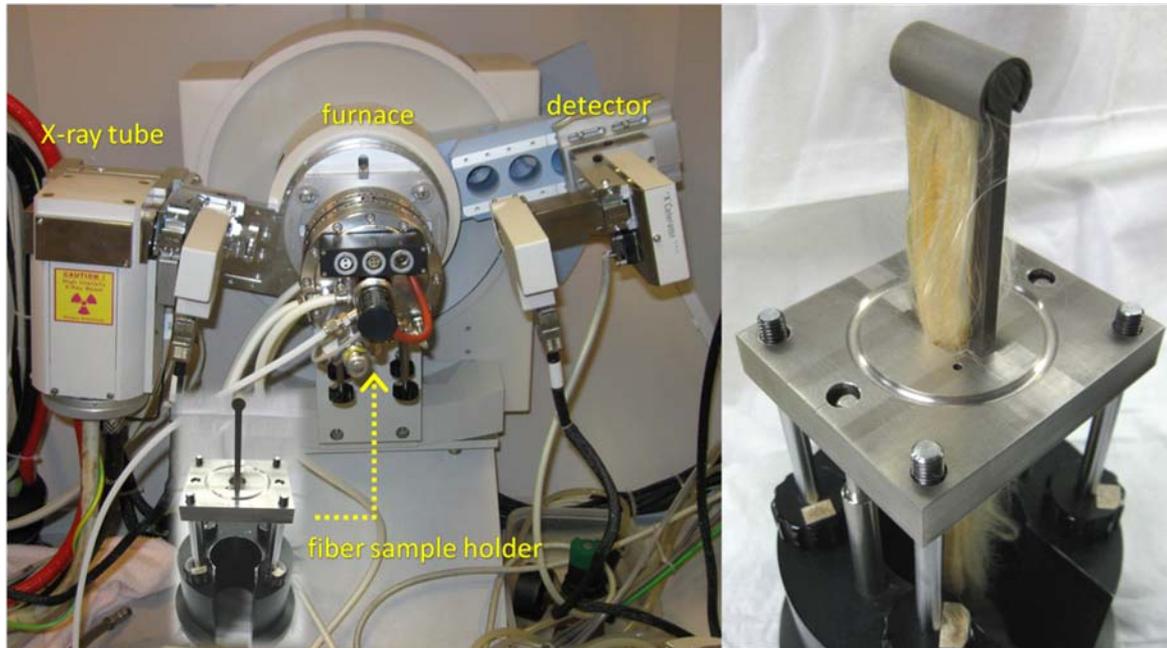
- The HTML User Program maintains world-class capabilities for materials characterization.
- During FY2009, the HTML User Program collaborated with 11 companies, 14 universities, and 3 national laboratories on 41 user projects addressing critical technical barriers to achieving the goals of DOE's Vehicle Technologies Program. 14 of those projects were focused on Materials Technologies, including Lightweight Materials. HTML user projects typically last from a few months to two years.
- This poster presentation reviews HTML User Program projects with industry, universities and national laboratories that address cost, design data, joining, manufacturability and performance barriers for the development and commercialization of carbon fibers, fiber-reinforced composites, magnesium and aluminum alloys, high strength steels and cast iron.

Future Work

- The HTML User Program will continue its collaborations with industry, universities, and national laboratories to address critical barriers to achieving the goals of DOE's Vehicle Technologies Program.
- Marketing efforts will continue to be focused on developing collaborations that contribute to the goals of increasing the fuel efficiency of passenger vehicles and reducing greenhouse gas emissions through increased utilization of lightweight materials.
- Special tools are under development to enable the *in situ* characterization of materials and processes, such as monitoring the microstructural evolution of carbon fibers under tensile loads during oxidation and carbonization and high-speed extensometry to measure deformation of materials and structures at high-strain rates.

Example of developing HTML User Program capabilities to support VT projects:

In Situ XRD Capabilities for Characterization of Low-Cost Carbon Fiber Processing



Unique *in situ* XRD attachment for carbon fiber processing characterization under development

- Fibers held in tension
- Furnace provides uniform temperature
- Extent of graphitization and texture determined by XRD as function of fiber precursor, process conditions such as tension and heating rates.

Barriers to be addressed:

- Cost – by developing processing methods to significantly reduce the cost of producing carbon fibers.