2. MATERIALS FOR FUEL SYSTEMS

A. Smart Materials for Diesel Fuel Injector

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No: DE-AC05-00OR22725
Subcontractor: Detroit Diesel Corporation, Detroit, Michigan

Objectives

- Develop technical requirements for a valve actuator system, including advanced piezoelectric materials that would enable faster and more precise control of the injection rate characteristics of a heavy-duty diesel injector.
- Enable the heavy-duty diesel engine to meet future emission standards without suffering gross fuel economy penalties, while mitigating the costs and complexities of prerequisite exhaust aftertreatment systems. Hardware fabrications in this project include a pre-prototype piezo-based actuator system suitable for bench test characterization and mechanical design integration with a production injector body.

Approach

- Characterize and validate, by static and dynamic testing, those candidate piezo stacks that promise to meet the gross requirements of the high-pressure diesel fuel injection application. Down-select the most promising candidate(s) for further evaluation and integration.
- Design and develop an amplification system to achieve the total required control valve stroke anticipated for the future diesel fuel injector.
- Complete a characterization study of advanced reference two-actuator fuel injector systems by
  - — experimental fixture
  - — engine testing
  - — simulation
- Synergize a combination of computational and experimental results to establish detailed material and system requirements for a superior piezoelectric material application in the heavy-duty diesel fuel injector.

Accomplishments

- Completed first-generation refinement of pre-load and electrical drive current in candidate piezo stacks to avoid high tensile forces, achieve minimum response time, and minimize displacement over-shoot under dynamic operation.
- Measured piezo stack power consumption to support the expectation for lower overall power consumption from a mature, well-applied piezo stack, as compared with a solenoid actuator performing a similar function.
- Designed and fabricated a hydraulic amplification module including the mechanical elements required to attach to a production injector body for bench demonstration purposes. Definitive finite element analysis confirmed the need for a design revision to the original hydraulic amplification module concept. Iterations on the design led to considerable simplification and implications of improved robustness, crucial to ultimately pursuing a production-feasible assembly.

  Integrated the revised amplification system module with a production injector body yielding a pre-prototype piezo injector, which was subsequently tested on an experimental fixture.

Introduction

The importance of research in fuel injection equipment to reduce environmental pollutants from the automotive industry, including light-duty and heavy-duty vehicles, has been greatly emphasized. One technical focus is the advancement of basic fuel injector technology to provide more precise delivery and atomization of fuel to the combustion chamber across a broader range of operational control.

The general objective of this project is to accelerate the assessment of the maturity of emerging piezoelectric materials to fulfill the requirements of future diesel engine fuel injectors. Specific requirements of this material application and challenges of the operating environment are being quantified.

An extensive candidate search was carried out for so-called smart materials, such as piezo stacks, that could actuate the control valve in a heavy-duty diesel injector.

This program is a joint research effort carried out by Wayne State University and Detroit Diesel Corporation and funded by DOE.

The more specific technical objective of this project is to use computational and experimental methods to substantiate a design and application strategy to maximize the advantage of piezoelectric materials, compared with conventional solenoid materials, in a diesel fuel injector operating environment. The premise of the base piezoelectric material advantage is that it can provide

- An order-of-magnitude reduction in response time
- Much higher force
- More precise control of the start, rate shape, and duration of the injection event
- More precise timing and delivery quantity of a multiple injection event
- Less energy consumption

The research was initiated with a screening study of available piezoelectric devices. Various piezoelectric devices were selected for this purpose. Broadly, these can be classified as either high-voltage or low-voltage/multilayer co-fired stacks. Key performance parameters of piezo stacks related to fuel injection applications were evaluated as the following table indicates:

Applied preload	Input voltage	Input current	Operating frequency	Duty cycle
Displacement	Rise time	Delay time	Fall time	System compliance

From the completed experiments, it was evident that the displacement produced by these candidate stacks was insufficient compared with the total displacement requirements of most historical control valve applications in the heavy-duty diesel fuel injection environment. Therefore, three options were possible:

1. Select much larger stacks that can produce higher displacements.
2. Design an amplification system to increase the stroke using existing stacks.
3. Search for the best fit of prevailing piezo stack capability with emerging fuel injector technology.

The first option was deemed impractical because of the physical limitations of the stack sizes, as well as the cost concerns for expensive piezo materials. The intermediate solution was deemed to be a reasonable approach in spite of considerations for implied energy losses and system complexities. Hence, conceptual design was initiated of a pre-prototype amplification system that could couple piezo stacks with a diesel fuel injector. Again, in the study of potential amplification design concepts, various approaches were considered, including mechanical, hydraulic, and hybrid amplification systems.

From these three general approaches, the hydraulic amplification system was selected because of its simplicity and inherently improved control of design issues. A prototype control valve actuator system was built and tested. This actuator system testing revealed that the system performance (e.g., valve stroke versus response time) would have to be improved substantially. Finally, a revised control valve actuator system concept—consisting of the actuator itself, the driving source, and the amplification system—was shown to achieve higher displacements.

Results

This report focuses on the activities completed for FY 2003:

1. Failure analysis of the piezo stack
2. Design and fabrication of the hydraulic amplification system
3. Integration of the hydraulic amplification system with the injector
4. Reference characterization of emerging two-actuator fuel injection systems

Failure Analysis of the Piezo Stack

As is well known, piezo stacks incorporate relatively brittle ceramic materials. Hence proper care must be taken to assemble the stack into the actuator mechanism. Various design issues must be resolved to ensure that the piezo stack operates efficiently and without failure, especially with the reliability and durability emphasis of the heavy-duty transportation industry.

Scanning electron microscopy (SEM) pictures from this project (Figures 1 and 2) show samples tested earlier this year. Stack layers are fractured, and electrodes are short-circuited and burned. Post-mortem analysis indicated that the stack would fracture only when the tensile forces in the stack were present or if there were intermittent impact loads on the stack.

Piezo stacks require some minimum preload force to preclude tensile forces from arising within the stack during normal cyclical operation. As the electrical current increases and in turn increases the force generated in the stack, higher-input electrical current calls for a higher preload. Otherwise, when the threshold is passed whereby inertial force becomes greater than the preload applied on the stack, even the modest tensile forces created can precipitate a physical failure of the stack.

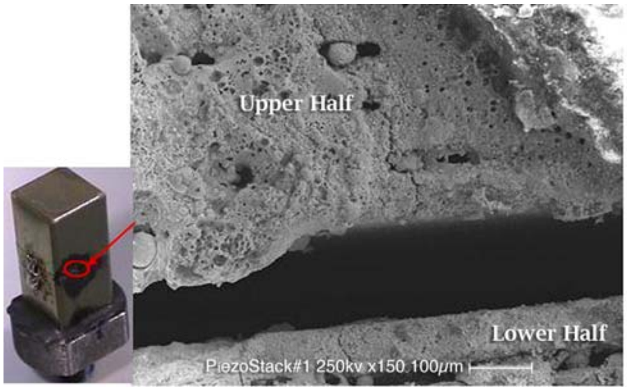

Figure 1. Failure analysis: Separation of layers of multilayer piezo stack.

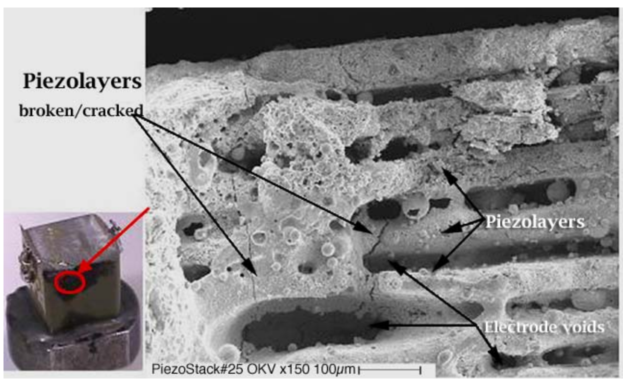

Figure 2. Failure analysis: Fractured piezo stack layers and burnt electrodes leaving voids.

Conversely, if the preloads are very high, then it is especially crucial to apply the preload uniformly over the entire stack cross-section to prevent the cracking of the ceramic layers. Otherwise, the stack is subjected to uneven forces and stress/strain gradients, with some portion of the stack under compression and some portion of the stack under tension, which will also lead the stack to fracture. If the stack is to operate in dynamic conditions, as expected for a diesel injector application, it becomes increasingly important to apply an appropriate level of uniform preload over the entire stack plane.

Therefore, the design of the marriage between the piezoelectric stack and its encapsulating structure must ensure that the loads and adequate preloads subjected to the stack are substantially co-axial with the stack axis. Similarly, the mating surfaces between the stack and the encapsulating structure must be within a predetermined tolerance of parallelism, influenced by local perpendicularity and planarity.

Design, Fabrication, and Integration of the Hydraulic Amplification System

The working principle of the amplification is illustrated by Figures 3 and 4. When the stack is actuated, the stack pushes the piston downward, which in turn pressurizes the working fluid in the fluid chamber. The fluid pushes the plunger upward along with the attached control valve. Thus the required control valve motion is achieved.

The development of the motion amplification system progressed through

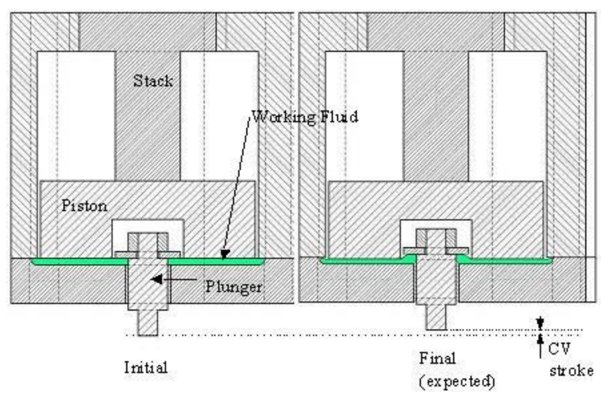

Figure 3. Working of hydraulic amplification system for piezo injector.

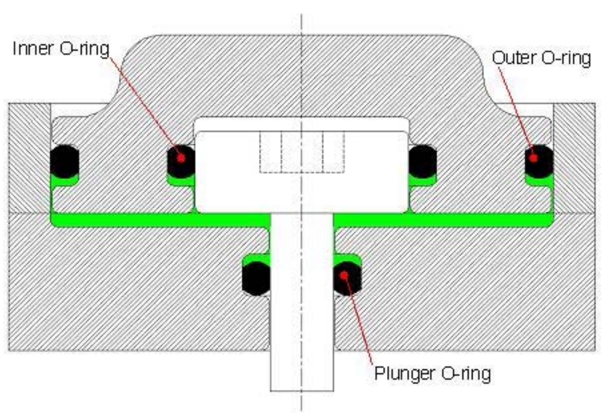

Figure 4. Hydraulic amplification system details with three O-rings.

several design iterations. Design was largely guided by finite element analysis, spring and O-ring calculations, and numerous interactions with vendors. The final design of the amplification module is represented by Figure 5, while the exploded view of the production "mule" injector body outfitted with the piezo electric actuation system is shown in Figure 6. The amplification system was assembled (Figure 7) and tested on an experimental fixture (Figure 8).

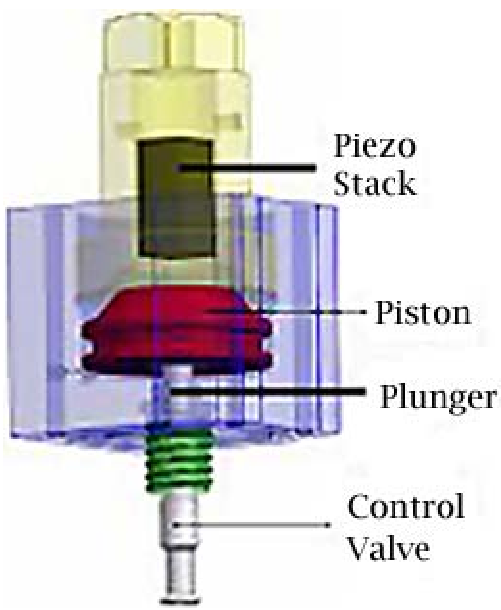

Figure 5. Final design: Piezo-electric control valve actuator of electronic unit injector.

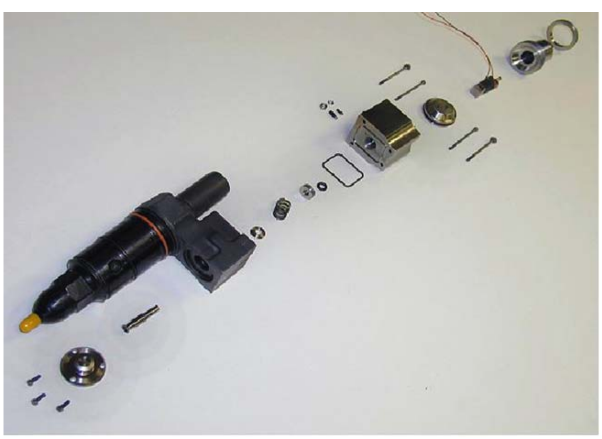

Figure 6. Piezo injector assembly: Exploded view.

Figure 7. Piezo injector with integrated amplification system module.

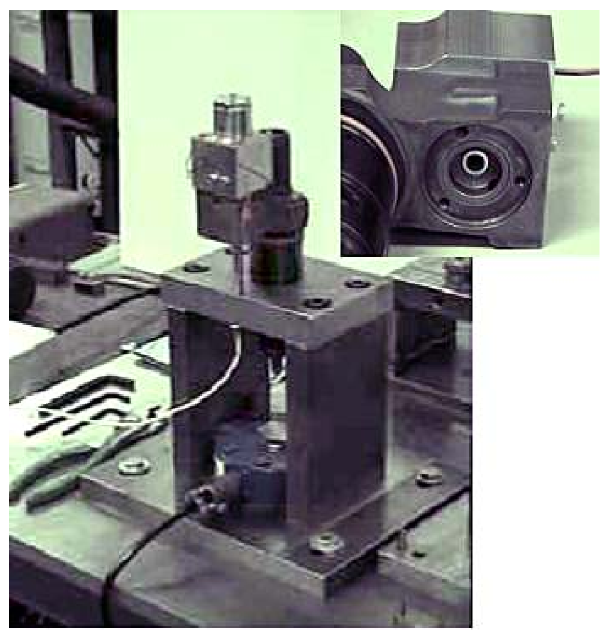

Figure 8. Test fixture for piezo injector.

Feasibility Study of Two-Actuator Fuel Injection System for Heavy-Duty Application

Two-actuator injectors exhibit promising potential compared with the single-actuator injector. Fixture test results quantified that a range of pressure traces and instantaneous rate shape control, as depicted in Figure 9, could be achieved by a two-actuator injector system, even though operational instability is apparent for some operating conditions. Nonetheless, NO_x reduction with marginal fuel economy penalty using the two-actuator injector (Figure 10) was readily demonstrated with engine testing.

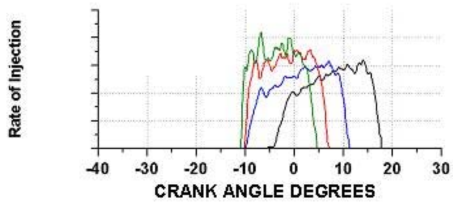

Figure 9. Two actuator fuel system—fixture test results: Injection rate characteristics.

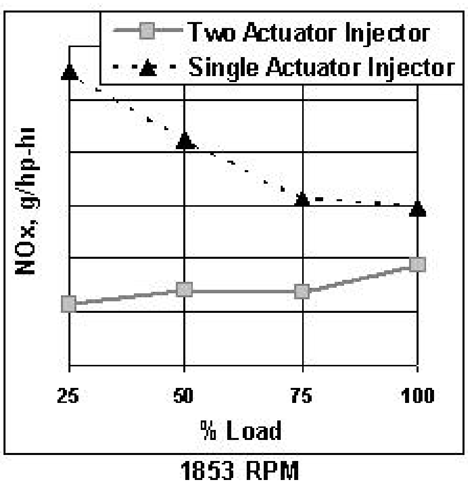

Figure 10. Two actuator fuel system—engine test results: NOx benefits.

Simulation of the two-actuator injector was performed while incorporating representative performance characteristics of available piezo stacks. Such characterization confirmed that the linear displacement versus voltage characteristics of the piezo stack could be exploited under dynamic operation. Relative to a solenoid candidate, response times were halved while overshoot travel was contained to within 30% of total displacement. Force measurement also confirmed the need to adjust to a higher preload during dynamic operation of the stack in order to avoid failure (Figure 11). Research shows that displacement losses are high in the amplification system of the piezo injector module. Hence a more feasible solution is application of the piezo stack to the needle control valve of the two-actuator injector. The potential of this application was gauged by simulating the needle control valve of a two-actuator injector with shorter response, as promised by piezo stack characterizations. Ensuing simulation results (Figure 12) suggest that more precise minimum pilot injection quantities and pilot gaps are possible when a faster needle control response is implemented.

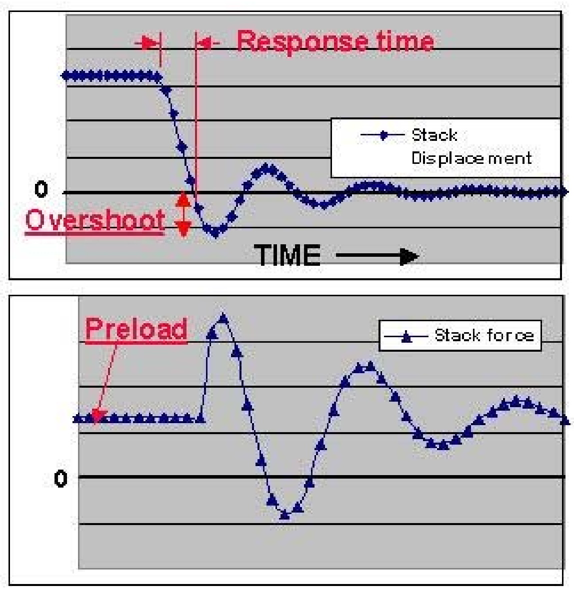

Figure 11. Hydraulic simulation results for piezo stack characterization: Displacement and force characteristics.

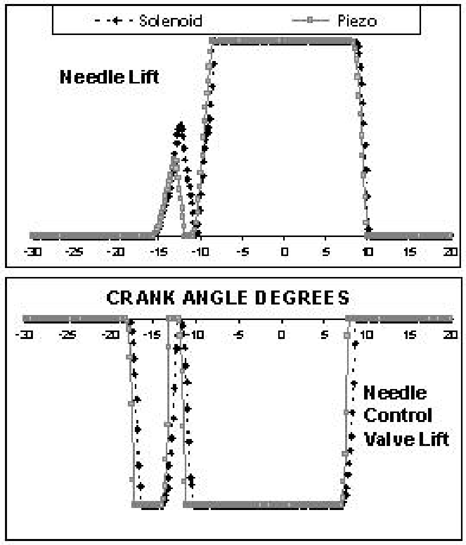

Figure 12. Hydraulic simulation results for two actuator fuel injector: Multiple injection.

Key Technical Observations

- In combination with the pre-prototype amplification system module, the demonstrated piezo injector control valve motion was limited. The main causes can be enumerated as
  - — High displacement losses in the amplification system module during return motion due to local O-ring expansion
  - — Higher-than-estimated friction forces in various moving parts
  - — Aeration in the hydraulic chamber
  - — Insufficient spring return force
- Reference characterization of the two-actuator injector highlighted that the most feasible and promising application of the piezo stack is to apply it synergistically to emerging two-actuator fuel injection technology. Similarly, the application of piezo stacks is most suitable for those actuators requiring smaller displacements, such as the needle control valve application (Figure 13). In this way, the need for motion amplification may be reduced or eliminated while the benefits of short piezo actuator response times are maximized with the flexibility of dual actuation.

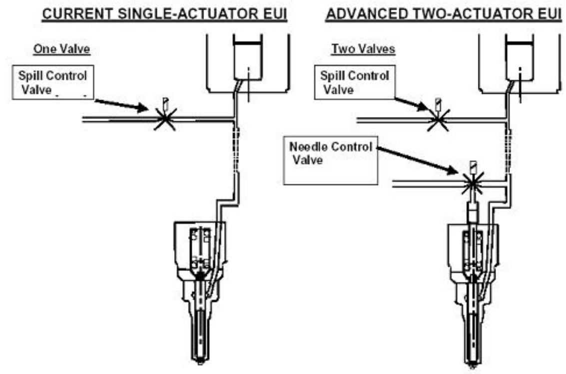

Figure 13. Schematic of single-actuator EUI and advanced two-actuator EUI.

Conclusions

- The piezo stack is a viable actuation device for future diesel fuel injection equipment involving multiple actuators.
- Piezo actuation provides substantially faster mechanical and hydraulic response times compared with prevailing commercial solenoid actuator technology.
- Hydraulic amplification of piezo stack displacement can generate required diesel injector control valve motion.
- Hysteresis of a piezoelectric stack is not expected to play a significant role in a pulsed application such as a fuel injection system.
- Open-loop control of input voltage may be of adequate complexity for application of a mature piezo actuator to a diesel fuel injection system.
- Commercially available piezo stacks have adequate material properties and technical characteristics for fuel injection application.
- Both stack failures encountered in this program are attributed to mechanical system design and not to piezo material or stack manufacturing issues.

- Piezo stacks cannot operate in tension. Adequate compressive preload must exceed tensile forces of stack acceleration in dynamic operation.
- Uneven loading of the stack can readily destroy piezo stack layers and must be avoided.
- The actuator design has to overcome various challenges, namely local aeration, O-ring forces, displacement losses, and force balance. Increased control of the return motion of the control valve could be achieved with such improvements.
- Better fuel injection characteristics and emission benefits are achievable with a two-actuator injection system.
- Application of smart materials to the needle control valve of the two-actuator injector would improve the multiple injection capabilities of the two-actuator fuel injection system.

B. Cost-Effective Smart Materials for Diesel Engine Applications

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No: DE-AC05-00OR22725

Objectives

- Develop a lower-cost lead zirconate titanate (PZT) powder for PZT stack actuators used for fuel injectors.
- Improve the mechanical properties, in particular the strength, of the low-cost PZT materials.
- Manufacture multilayer PZT stacks with low-cost PZT powder.
- Develop a test apparatus for measuring the reliability of PZT stack actuators.

Approach

- Reformulate commercial PZT-4 powder by including a low-temperature sintering aid and incorporating high-energy attrition milling.
- Investigate chemical additives that can strengthen the low-cost PZT materials.
- Manufacture multilayer stacks from low-cost PZT powders using tape casting and lamination techniques. Use screen printing to apply silver or Ag-Pd (90 wt%/10 wt%) for the interlayer electrodes.
- Fabricate a computer-controlled test apparatus for testing the reliability of commercial and in-house PZT stacks.

Accomplishments

- Developed ceramic additives that allow a commercial PZT powder to sinter at a temperature below the melting point of silver. These additives also enhance the piezoelectric properties of the low-temperature sintered material.
- Increased the fracture strength of the low-temperature sintered material by about 50% by including a non-soluble oxide into the matrix.
- Fabricated multilayer stacks with silver and Ag-Pd (90 wt%/10 wt%) interlayer electrodes.

- Fabricated a computer-controlled test apparatus for measuring the reliability of multiplayer PZT stacks.
- Stroked a commercial PZT actuator stack for >50 million cycles with continuous data collection using the Oak Ridge National Laboratory (ORNL) reliability apparatus. The data indicate that the displacement of a commercial stack steadily degrades with increasing cycles.

Future Direction

- The initiative on low-cost PZT materials for PZT stack actuators has been completed and will not continue for FY 2004. However, development plans for continuation of this effort are in place should additional effort be required by heavy vehicle stakeholders. In this regard, discussions are under way with a diesel engine manufacturer regarding reliability testing of PZT stacks for fuel injector applications.

Introduction

Major drivers in diesel engine research are the desire to increase fuel efficiency and to reduce the pollution of the environment. Recent studies have shown that multiple fuel injections per combustion cycle (rather than the conditional one injection per cycle) can increase the fuel efficiency and lower the NO_x and particulate emissions from diesel engines. Varying the rate and shape of the injection cycle and using multiple injections can improve power, emissions, and fuel economy. Because of the present speed limitations of magnetic solenoid valves, multiple injections of fuel are possible only at engine idle speeds. The need for multiple injection capability per combustion cycle has stimulated research into the development of high-speed actuators. The leading candidate devices for these actuators are piezoelectric, based on PZT, and magnetostrictive, based on Terfenol-D. Because of its lower material costs and performance capability, PZT is the favored candidate.

PZT materials are widely used for actuators in applications requiring precision control and high force with small sizes. Multilayered PZT materials have been studied and are favored for their low power consumption and high precision control.[1,2,3] In comparison with conventional actuators, PZT transducers demonstrate higher energy densities, larger actuation forces, greater positioning accuracy, and shorter response times. However, widening the application of PZT transducers requires improvement in the properties of the PZT materials (greater strains and temperature stability desired) and reduction of the materials costs, where the Pt-Ag or Pd-Ag electrode materials represent the largest material factors.

Approach

The following approach was taken to study the effect of low-temperature sintering agents and strengthening agents on PZT-4 powder. Numerous PZT powder batches were prepared using attrition milling with sintering aids and several types of strengthening agents. For the initial powder batch, the particle size was measured after defined milling periods to determine optimum milling time. After milling, the slurries were dried, the powder cakes were crushed, and the powders were sieved. PZT disks were pressed in a steel die and sintered at temperatures suitable for silver electrode applications. Samples were poled, after which the electrical and mechanical properties were measured. Optical microscope and scanning electron microscope (SEM) examination were performed to analyze the microstructures of the materials.

The following approach was taken for the fabrication of multilayer stack actuators from the low-temperature sintering PZT-4 materials. Thin sheets (0.02 cm) of the powder were tape-cast, and then silver or Ag-Pd (90/10 wt/wt) electrodes were screen-printed onto the dried tapes. Then 25 to 35 discs were punched from a tape, aligned on top of one another in a steel die, and laminated to make a stack. Laminate samples were sintered and densities measured. Silver ink electrodes were painted on the outer flat surfaces of each stack, and the ink was used to create layer-to-layer interconnections down the side of the stacks. The stacks were fired at low temperature to bond these silver electrodes. Several samples were poled and electrical properties measured. Select samples were sliced into sections with a diamond saw for optical and SEM analysis.

The third major work this year involved the fabrication of a computer-controlled test apparatus for testing the reliability of multiplayer PZT stacks. ORNL personnel fabricated a custom apparatus.

Results

Work was performed with an attrition-milled PZT-4 powder containing the ORNL sintering aids. This material was densified to full density after 3 hours of heating at temperatures greater than 850°C. This low sintering temperature can allow the use of cheaper silver inter-electrodes in laminated stacks. Furthermore, Figure 1 shows that these materials, when sintered at temperatures from 800 to 950ºC, have piezoelectric properties equivalent to those reported for PZT-4 that was sintered at temperatures exceeding 1200ºC.

However, the strength of the low-temperature sintered material is lower than that of material that is sintered at the higher temperatures. Consequently, part two of the study involved the use of ceramic additives to increase the strength of the low-temperature sintered material. Figure 2 shows that the addition of type 3 ceramic additive enhances the strength of the low-temperature PZT by approximately 50%. Figure 3 shows the fracture and polished surfaces of a part containing the type 3 additive. Each grain of the PZT has several of the additive particles both at the grain boundaries and within the grains. It is believed the ceramic inclusions prevent excessive grain growth and may help bond PZT particles together more strongly.

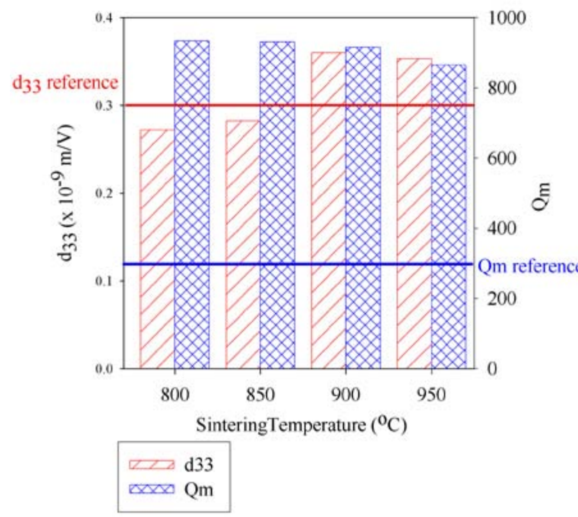

Figure 1. Piezoelectric properties for PZT materials sintered at from 800 to 950°C.

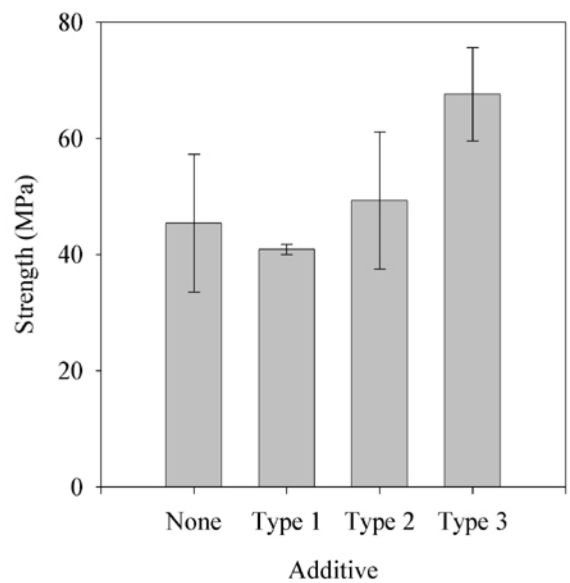

Figure 2. Strength values for low-temperature PZT materials containing strength additives.

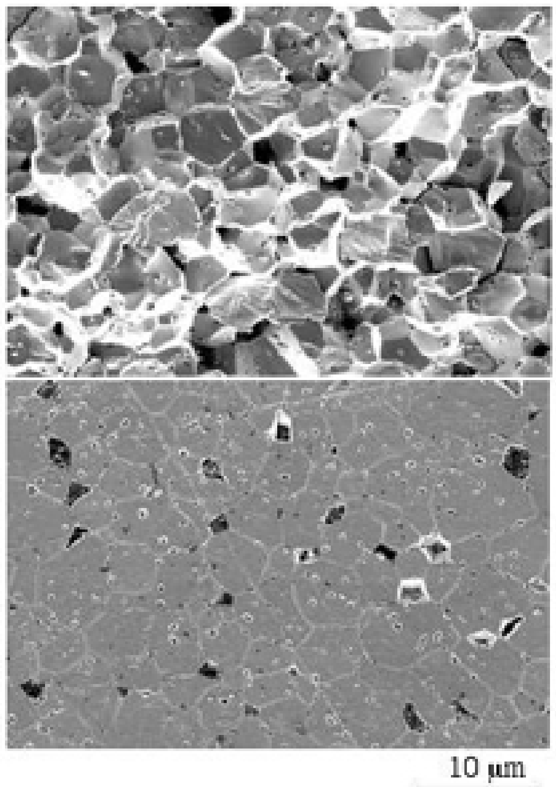

Figure 3. SEM photos of the fracture surface (top) and the polished surface (bottom) of ORNL PZT with type 3 strength additive.

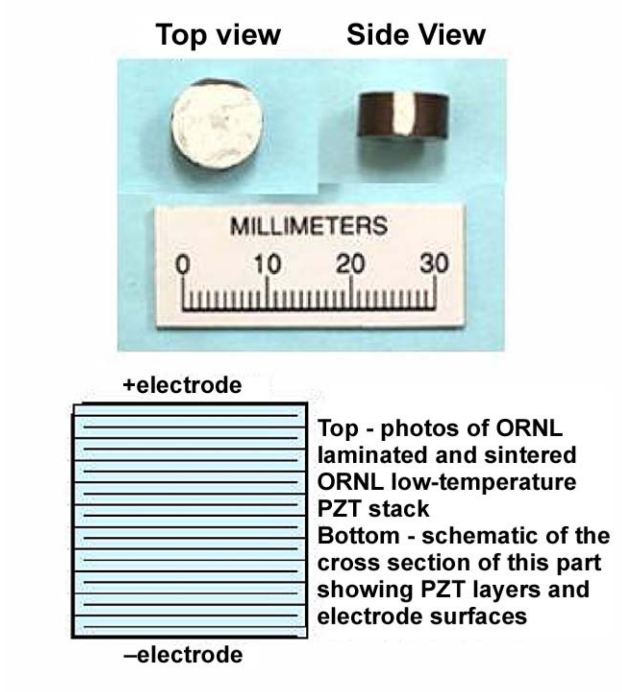

Figure 4. Photos of an ORNL PZT stack (top) and a schematic that shows the architecture of the PZT layers inside the part (bottom).

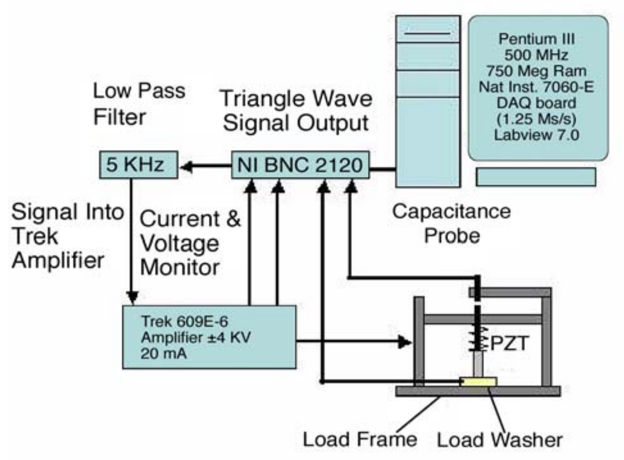

Figure 5. Schematic of the ORNL custom-built reliability apparatus for testing PZT stacks.

Several multilayer laminate PZT stacks were fabricated as part of the milestones for this year. The stacks are made from low-temperature PZT materials made at ORNL. The inner electrodes consist of a lower-cost silver and silver/palladium electrode material. Figure 4 shows a 33 layer-multilayer laminate.

Fabrication and initial testing of a PZT actuator reliability test system were completed to reach a program milestone. Figure 5 shows a schematic of the test apparatus. Figure 6 shows photos of all of the major components. The test system includes a fixture to hold a PZT stack in compression and to measure the displacement, a power supply and other electronic equipment needed to cycle a piezoelectric stack actuator, and a computer with Labview software that provides signals to the power supply needed for continuous cycling of a PZT actuator. The Labview system also collects the voltage, current, and displacement during testing. Two commercial actuators, 26 mm in length and 8.25 mm in diameter, have been tested

(Top) reliability apparatus electronic equipment and stack holder (left) holding fixture for PZT stack

Figure 6. Photos of the ORNL reliability apparatus.

using this apparatus. One of the actuators was tested for >50 million cycles.

Displacement data in Figure 7 show that this actuator slowly degraded in performance during the test. An additional power supply, computer, and associated electronics have been purchased to build a second test system that can stroke at 10 times the rate of the present system and allow faster reliability testing.

Conclusions

ORNL has developed a piezoelectric powder that has the potential to lower the cost of diesel fuel injection actuator stacks by allowing the use of internal electrodes made from metals that are less expensive than platinum or palladium.

ORNL has found a ceramic additive that can strengthen the low-temperature sintered piezoelectric powder without degrading the piezoelectric properties.

The ORNL automated test stand for measuring the effect that voltage cycling has on the displacement of a piezoelectric stack will be an important tool to aid in the reliability testing of fuel injection system actuators.

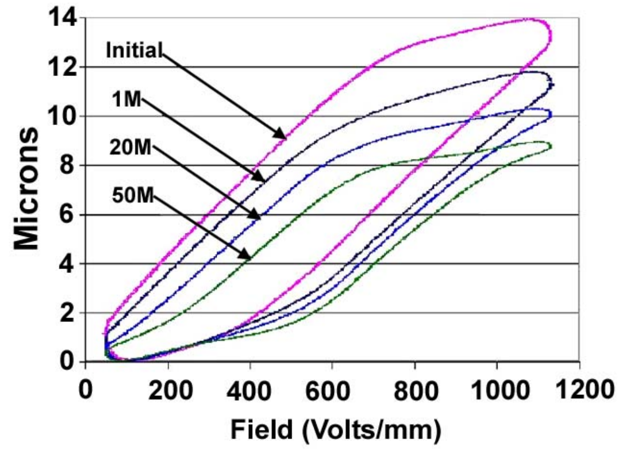

Figure 7. Displacement curves for a commercial stack actuator tested on the ORNL reliability apparatus.

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C. Low-Cost Manufacturing Processes for Ceramic and Cermet Diesel Engine Components

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No: DE-AC05-00OR22725
Subcontractor: Southern Illinois University, Carbondale, Illinois

Objectives

- Investigate low-pressure injection molding and continuous sintering as low-cost manufacturing processes for ceramic and cermet diesel engine components.

Approach

- Process cermet compositions containing titanium carbide (TiC) and an intermetallic matrix phase into simple cylindrical shapes by low-pressure injection molding.
- Following debinding, sinter the cermets by continuous sintering and by a vacuum-pressure sintering cycle developed by Oak Ridge National Laboratory (ORNL).
- Measure physical properties and compare them with those of conventional methods and materials.
- Investigate the economics of the process by a model developed at Southern Illinois University–Carbondale (SIUC) to determine if the process is competitive with current manufacturing methods.

Accomplishments

- Processed simple cermet shapes by low-pressure injection molding and continuous sintering. This method potentially offers a competitive, economical process for the manufacture of small parts for diesel engine components.
- Developed an economic model to determine the cost-effectiveness of processes.
- Designed a precision mold for a fuel injector plunger that will allow the use of low-pressure injection molding to produce very-near-net-shape parts for further testing.
- Continued interaction with ORNL, CoorsTEC, and Cummins to promote commercialization of cermet diesel engine components.

Future Direction

- Conduct injection molding trials of the precision mold for the fuel injector plunger that is being machined.
- Modify the continuous sintering furnace to allow for extremely high transport rates through the furnace. If successful, this will allow for improvement of the economics of the process and further reduce the cost of the manufacturing process.

Introduction

All manufactured parts potentially can benefit from improved manufacturing processes and the incorporation of advanced materials. Injection molding is a highly cost-effective method for producing precision parts with a minimum of labor cost, and continuous sintering has been proven industrially to provide an economic advantage for sintering many advanced ceramics. More recently, continuous sintering has been used as a means of rapidly sintering cermets composed of TiC in an intermetallic matrix.

To reduce costs, it is essential to minimize material waste while maximizing the yield of in-specification finished parts. Continuous sintering is a potential means of maximizing the furnace yield of parts that are within specifications while minimizing material losses due to furnace-related problems, thereby reducing part cost. The continuous furnace at SIUC has been used to sinter a wide range of pre-alloyed intermetallic-TiC formulations and similar formulations where the intermetallic is formed by reaction sintering of the individual elements. Based on this work, the most promising intermetallics contained 30–50 vol% of nickel aluminide (NiAl), nickel-chromium (NiCr), or nickel-chrome-iron (NiCrFe) added to a fine-grained commercial TiC. These formulations were found to have high strength, hardness, toughness, and corrosion resistance. In addition, their thermal expansion can be engineered to be very close to that of cast iron and steel, which will reduce thermal expansion mismatch in several key diesel engine applications.

The present work has focused on developing an economic model to compare the costs associated with processing and sintering of these cermets by conventional and continuous sintering. To further refine this economic model and determine conditions that may make cermets more economically attractive to the diesel engine industry, more experimental data are required.

In addition, a precision mold for the low-pressure injection molder needed to be designed and machined to allow for the processing of very-near-net-shape parts for this study. Interaction was continued with ORNL, CoorsTEC, and Cummins to promote commercialization of these cermets for diesel engine components.

Approach

Economic Model

One of the primary tasks involved the development of an economic model for comparing both materials and processing methods for cermets targeted for use as diesel engine components. The cermet is a combination of an intermetallic and TiC; the intermetallic can be NiAl, CrAl, NiCr, or another alloy. Modification of these intermetallics may be necessary to obtain the properties desired.

The costs associated with the various processes involved in producing these intermetallic bonded cermets were compiled and input into an economic model that was originally developed at SIUC for silicon nitride. The model was refined to include raw powder cost, binder costs, and process costs related to injection molding, debinding, isopressing and sintering. Both the continuous sintering process and a commercial process based on a vacuum/pressure cycle were explored. In previous work, continuous sintering was found to afford a reduction of up to 50% in the cost of sintering over conventional methods. Figure 1 shows the continuous sintering furnace at SIUC. In the sintering of cermets, higher sintering rates and more rapid throughput are possible with continuous sintering. However, the properties required or acceptable for cermets for diesel engine applications have not been identified.

Figure 1. Continuous sintering furnace at SIUC.

Low-Pressure Injection Molding

In the current work, simple right cylinders have been produced by low-pressure injection molding. This requires additional manufacturing cost to machine this shape into a precision fuel injector plunger. In order to make prototypes of plungers for fuel injectors that are very near net shape, it was necessary to design and machine a die set for the low-pressure injection molder. Figure 2 shows the low-pressure injection molder at SIUC.

Figure 2. Low-pressure injection molder at SIUC.

Industrial Collaboration

Collaboration among SIUC, ORNL, CoorsTek, and Cummins has continued since the start of this project. Cermets processed by low-pressure injection molding and continuous sintering have been submitted to ORNL for wear testing, and materials have been submitted to CoorsTek for evaluation. Recently CoorTek has submitted materials to SIUC for sintering and evaluation.

Results

Economic Model

One of the major tasks involved the development of an economic model for comparing both materials and processing methods for cermets targeted for use as diesel engine components.

In this model, the most cost reduction can be obtained by developing a system that can take advantage of continuous sintering. For this model, the following parameters were used:

- Production was assumed to be 95% efficient for continuous sintering and

85% efficient for the production batch furnaces.

- Production was held constant at ~750,000 parts per year.
- Each part was assumed to weigh 20 g in the green state.
- Powder costs included binders, and the composition was 40 vol% Ni3Al and 60 vol% TiC with processing by reaction sintering.
- Gas, electric, labor, equipment depreciation, and replacement parts were set according to the furnace type.

For this model to be reliable, the production of two commercial batch furnaces was used, which fixed the production at ~750,000 parts per year. This production level uses only 26% of the capacity of the continuous furnace. At 100% capacity, the continuous furnace would be capable of sintering about 3 million parts, which is the production capacity of nine commercial batch furnaces. This means that for higher production levels, the proportional cost would be lower for continuous sintering than predicted by the model.

Figure 3 shows a comparison of the total cost of a 20-g cermet part processed by low-pressure injection molding and then sintered either by continuous or batch sintering. The continuous furnace uses flowing argon gas, while the batch furnace requires a purge/vacuum cycle followed by low-pressure argon sintering. The costs are based on real production cost, with 250% overhead attached to the manufacturing cost and assuming an added cost of 25% for machining costs. The continuous optimized cost is based on optimizing the production volume for the continuous furnace.

It is believed that with higher sintering rates, cermets can be manufactured to be competitive with or lower in cost than ceramics, such as zirconia, for these applications. The advantages of the cermets over the ceramics are

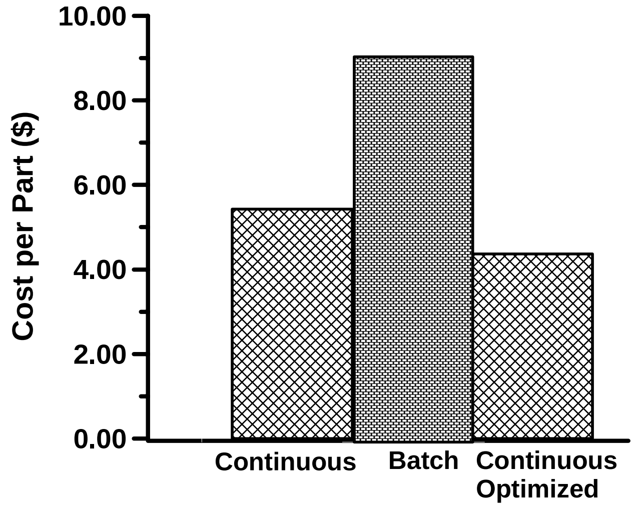

Figure 3. Relative cost comparison for continuous sintering vs. batch sintering for diesel engine components.

- Lower sintering temperature
- Potentially faster sintering cycle and lower processing costs
- Machinability by electro-discharge machining (EDM)

The intermetallic phase of the cermet is available as an alloy or as independent metals or metallic compounds that can be reaction-sintered to form similar alloys. The advantage of the reaction sintering is a reduced cost for the raw materials and the capability to produce a finer grain structure in the finished cermet. The intermetallic used in the cost model was Ni_3Al, formed by reaction sintering of NiAl and Ni, and the source of TiC was Kennametal.

Other combinations are possible where the intermetallic can be NiAl, CrAl, or NiCr with or without iron and boron additions. Further modification of these intermetallics may be necessary to obtain the properties desired. Currently the powders used for reaction sintering are comparable in cost with high-purity ceramics. In article 2E of this volume, ORNL reports that a catalytic grade containing aluminum and nickel could possibly reduce the material cost even further.[1] The ceramics can be processed in air, while the cermets require a non-oxidizing atmosphere for sintering. The ceramic cannot be easily machined into the geometry required for specific diesel applications, especially where porting is required. Since the cermets investigated can be machined by EDM, these complex geometries can be readily produced. This advantage in manufacturability could make the cermets more economically feasible than ceramics.

Low-Pressure Injection Molding

Previous work in low-pressure injection molding focused on developing and optimizing a binder system to produce a simple cylinder shape approximately 9 mm in diameter by 7.5 cm in length. Figure 4 shows these cermets in the various stages of processing, and Figure 5 shows the typical microstructure present in the cermets investigated. Figure 6 shows the hardness results obtained for the cermets, compared with values for cast iron.

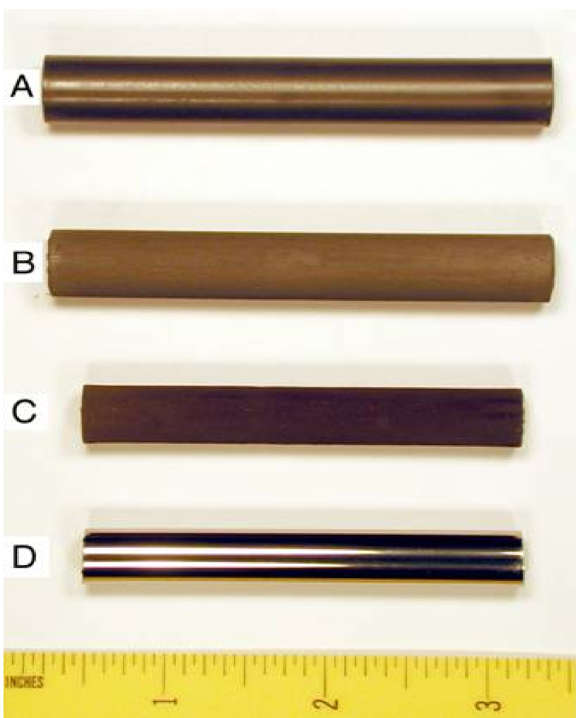

Figure 4. Cermets produced at SIUC (a) as injection molded, (b) following binder removal, (c) following continuous sintering, and (d) following machining.

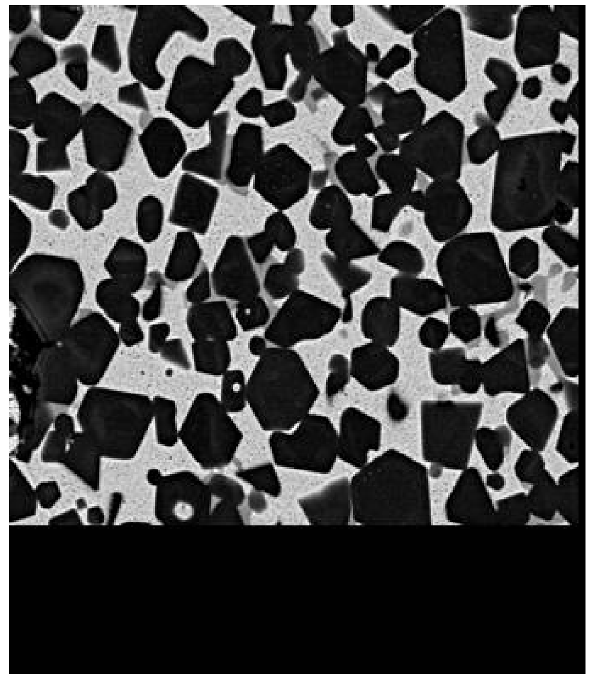

Figure 5. Typical microstructure for NiAl-TiC cermet.

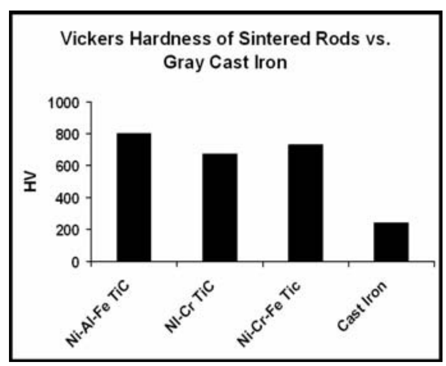

Figure 6. Hardness comparison of cermets investigated with cast iron.

To better demonstrate the capabilities of this process, a commercial fuel injector was obtained and dismantled. Figure 7 is a schematic of the commercial fuel injector showing the exposed fuel injector plunger.

Figure 7. Fuel injector layout.

Based on this fuel injector, a near-net-shape mold for making the injector plunger was designed and machined at SIUC for use on the Peltzman low-pressure injection molding machine shown in Figure 2.

Industrial Collaboration

SIUC, ORNL, CoorsTek, and Cummins have been involved in a confidential collaboration; and all data related to this project have been the responsibility of the individual contributors. What can be reported are relative results and results obtained independently. CoorsTek and SIUC have independently sintered cermets that were processed at SIUC and sintered by both CoorsTek and SIUC. The results for relative density and hardness for one cermet formulation are given in Figure 8. As can be seen from these results, marginally higher density and hardness were obtained for the cermets sintered at SIUC by continuous sintering. More recently, a cermet received from CoorTek has been received for continuous sintering and evaluation.

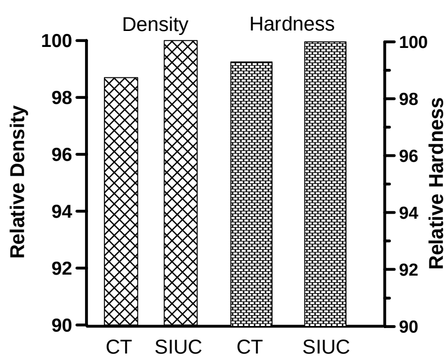

Figure 8. Density and hardness results for a cermet sintered at CoorsTek (CT) and SIUC.

The results of wear testing have been very favorable for many of the cermets investigated. The hardness, toughness, and strength of the cermets investigated are responsible for the very high wear resistance. The effects of the intermetallic and of particle size of the TiC in the cermet on the wear resistance are not very well understood. Several of the simple cylindrical shapes produced at SIUC have been submitted to Peter Blau at ORNL for sliding wear evaluation. Preliminary results are encouraging, and the final results will be reported when they are received.

Conclusions

An economic model was developed that indicates that cermet components for diesel engine applications can be competitive with the advanced ceramics currently employed. Simple cermet shapes were produced by low-pressure injection molding and continuous sintering. These methods potentially offer very competitive, economical processes for the manufacture of small parts for diesel engine components. A precision mold was designed for a fuel injector plunger that will allow the use of low-pressure injection molding to produce very-near-net-shape parts for further testing. Industrial collaboration and wear testing of cermets will be continued.

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D. Intermetallic-Bonded Cermets

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Objectives

- Develop materials for diesel engine applications, specifically for fuel delivery systems and wear components (e.g., valve seats and turbocharger components). Requirements are excellent corrosion resistance in a diesel engine environment, high flexure strength and fracture toughness, and compatibility with steels. In addition, they should not cause excessive wear of the steel counter face

Approach

- Develop processing to combine carbide phases with tri-nickel aluminide (Ni_3Al) with a range of nickel aluminide contents by liquid-phase sintering.
- Analyze nickel aluminide to (1) optimize the cermet thermal expansion coefficient to that of selected steel compositions, (2) maximize fracture strength and toughness of the cermet, and (3) minimize wear of both the cermet and steel components.
- Support efforts at Cummins Engines and CoorsTek to develop advanced fuel injector systems through materials and failure analysis of prototype components.

Accomplishments

- Revealed that Ni_3Al-bonded titanium carbide (TiC) cermets with flexure strengths in excess of 1 GPa, fracture toughness values in excess of 18 MPa√m, and thermal expansion coefficients approaching those of steels could be fabricated by liquid phase sintering.
- Transferred technology to industry and provided support in developing prototype components for high-pressure fuel injector systems.

Future Direction

- This task met its objectives and was completed at the end of FY 2003.

Introduction

The goal of this task is to develop materials for diesel engine applications, specifically for fuel delivery systems and wear components (e.g., valve seats and turbocharger components). These applications will require materials that have a minimum hardness of 11 GPa and a thermal expansion coefficient of between 10 and 15×10^{-6}/°C. The material should also have excellent corrosion resistance in a diesel engine environment, flexure strength in excess of 700 MPa, and fracture toughness greater than 10 MPa√m to ensure long-term reliability. The material should also be compatible with steels and should not cause excessive wear of the steel counter face. The upper temperature limit is 200°C for fuel delivery system applications and 815°C for other applications. For reciprocating components, a low density or specific weight is most desirable. Finally, the total material processing costs for these advanced materials should be competitive with those of competing technologies such as TiN or other ceramic coatings on high-speed tool steels.

Approach

The objectives listed in the introduction provided the guidelines for materials selection and the choice of the use of the cermet concept to impart toughness to a ceramic-based system. The constituent materials that were most attractive were based on advanced Ni_3Al, which can exhibit exceptional ductility over a broad range of temperature and have excellent corrosion resistance. To improve the wear resistance and hardness, TiC was selected for the matrix material. The combination of these two materials offers the opportunity to tailor the composition so as to match the thermal expansion of steels and develop cermets with low specific weight together with high fracture toughness and strength. In addition, the TiC-Ni_3Al combination was readily amenable to fabricating components using the standard powder metallurgical processing used extensively in industry. These factors combined to offer a promising approach to developing cermet components for advanced fuel injectors, which was the first application. The task undertook a study of optimizing the cermet properties for this application. It also provided technical support to CoorsTek, which was responsible for scaleup of the process, and to Cummins Engines Company, which was responsible for testing of the fuel injector components.

Results

The composition of the TiC-Ni_3Al cermets influences the wear characteristics as observed in pin-on-disk rotating wear studies. Evidence of this is seen in cermets containing 40 vol % in which molybdenum or Cr_3C_2 powder was added to the TiC powder prior to attrition milling to reduce the TiC particle size. Molybdenum is employed as has been indicated to improve the wetting of TiC by nickel during liquid-phase sintering and improves the strength of the interface between the TiC and the Ni.[1] Chromium additions are seen to increase the yield stress and hardness of the Ni_3Al phase to a greater extent than does molybdenum,[2,3] but carbon is seen to be much more effective.[2,4] The thought was that increasing the hardness of the Ni_3Al might improve the wear resistance. However, evaluation of cermets fabricated in this project using these additives reveals that the addition resulted in better wear performance with regard to the wear of both the cermet pin and the steel disk materials (Figures 1 and 2). Based on the wear surface observations, it appears that pull-out and fracture of the TiC grains is diminished with the molybdenum addition, compared with the response with the Cr_3C_2 addition.

Fracture analysis was conducted on a number of metering plungers that had been machined by Cummins Engines from rod stock provided by CoorsTek using the TiC-50 vol % Ni_3Al composition. These plungers had been machined to specifications for insertion

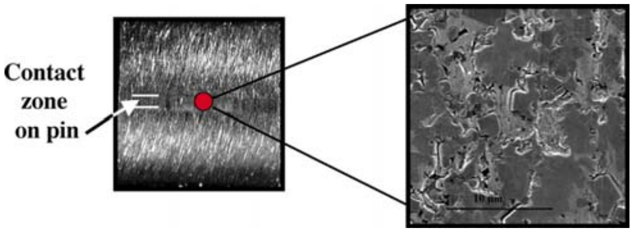

Figure 1. Contact surface (left) of (TiC+2 wt % Mo) + 40 vol % Ni_3Al cermet pin after pin-on-disk (tool steel) wear test. Low wear coefficients were obtained for both pin and disk materials. This is reflected by the fact that within the contact zone (right) the angular TiC grains (darker gray grains) did not cleave and form abrasive debris.

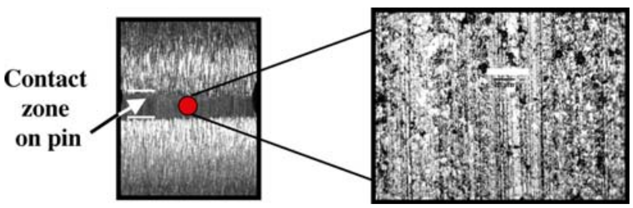

Figure 2. Wear coefficients of both pin and disk increase when a Cr_3C_2 addition to TiC is substituted for molybdenum, as reflected by the wide wear track on the pin (left) and the pullout of TiC grains from the surface, evidenced by the dark pits within the contact zone (right).

into a high-pressure (>315 MPa) fuel injection test. During preparation, the starting rods are machined to profile; a steel end cap is added by press fitting; and, finally, the end cap is machined to specifications. Four of these plungers were received for failure analysis and are shown in Figure 3. C10-3 and C10-7 both failed during machining of press-fit steel end cap, C10 failed in a 20-hour overpressure test, and C8-5 successfully completed the 20-hour overpressure test.

Fractographic analysis revealed that the two plungers that fractured during machining of the steel "top hat" cap exhibited large pores and defects on the fracture surfaces located in the reduced section just below the steel-capped end of the plunger. The greatly reduced diameter in this area would experience high stresses if

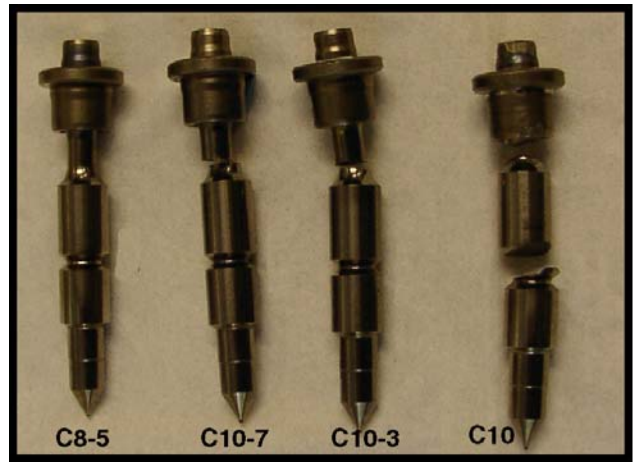

Figure 3. TiC-Ni_3Al cermet fuel injector plungers fabricated through collaborative efforts at ORNL, CoorsTek, and Cummins Engine Company. C8-5 successfully completed a 20-hour overpressure injector test at Cummins. C10 failed at some time during that test. Both C10-3 and C10-7 failed during the machining of the steel "top hat" at Cummins.

flexure occurred during machining. The presence of large pores and defects implies a need for improvement in the fabrication procedures. Subsequent fractographic analysis of flexure bars made from rod stock fabricated from one of the powder batches that was used (Batch 10) confirms that processing defects were an issue. The one plunger that failed during the overpressure injector tests at Cummins Engines also exhibited defects on a secondary fracture surface.

Conclusions

Cermets composed of TiC grains bonded by a continuous Ni_3Al phase, and fabricated by industrial-based powder metallurgical processing routes, were developed with strengths in excess of 1000 MPa, toughness values of ~0 MPa•$m^{1/2}$, and thermal expansion coefficients of up to 11 ppm/°C. Characterization of cermet pins tested against a rotating steel disk in a wear facility at Cummins Engine revealed that minor additions of molybdenum improve the wear resistance compared with chromium-bearing additions. Molybdenum has also been found to enhance the liquid-phase sintering and mechanical properties of the cermets. Evaluation of cermet plungers, both those prepared for injector rig tests and those exposed in the overpressure injector tests, reveals that processing defects were a factor in limiting the performance of the cermet plungers. Further improvements in the cermet processing should improve the reliability of the cermet plungers. At the same time, it is strongly recommended that the actual conditions under which plungers fail (e.g., during machining) be carefully documented to aid in determining steps to take to improve the reliability.

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E. Low-Cost, High-Toughness Ceramics

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No: DE-AC05-00OR22725

Objectives

- Increase the critical fracture toughness (K_{Ic}) of structural ceramics for advanced diesel engine applications without strength degradation.
- Develop high-toughness materials that are also low in cost.

Approach

- Develop TiC-based composites with 40–60 vol. % nickel aluminide (Ni_3Al) because they have expansion characteristics very close to those for steel.
- Conduct the development effort in collaboration with CoorsTek, Inc., and Cummins Engine on processing scale-up and engine testing tasks.

Accomplishments

- Supplied large numbers of sintered blanks for engine testing.
- Examined several different raw material powder combinations.
- Found that when elemental aluminum was used, the samples exhibited macroscopic cracking due to differential heating and shrinkage resulting from the exothermic reaction between the nickel and aluminum.
- Found that only composites made from the NiAl + nickel combination had mechanical properties that were acceptable for engine applications.

Future Direction

- Refine the processing parameters with the alternate precursors to improve the mechanical performance.
- Use alternate NiAl sources including an Al-Ni catalyst material, TiC-Ni_3Al mixtures developed by the University of Colorado, and Ni-Al-TiC blends for reaction synthesis.

- Continue to work with CoorsTek Inc. (a parts supplier) for scale-up of the processing and supply it with pilot-plant-scale quantities of powder mixtures for injection molding trials.
- Supply sintered parts produced in conjunction with CoorsTek to Cummins Engine Co. for rig testing of machined components.

Introduction

TiC-Ni_3Al composites are under development for application in diesel engines because of desirable physical and mechanical properties. For these applications, the Ni_3Al volume contents are on the order of 30 to 50 vol % in order to match the thermal expansion of steel. Typically, flexural strengths greater than 1000 MPa at temperatures up to 800°C and fracture toughnesses higher than 15 MPa√m are obtained for the composites. The composites are densified by liquid-phase sintering. Most of the early work used gas-atomized Ni_3Al particles with fine TiC powders. Later work was done using nickel and NiAl powders (along with the TiC) to form Ni_3Al by an in-situ reaction during sintering. Over the past few years, the in-situ reaction process was developed significantly because it produced high mechanical properties and developed a fine TiC grain size. The finer grain sizes were favored because of better wear resistance.

The fabrication techniques and equipment employed in production are very similar to those used for the fabrication of WC-Co hard metals; thus the processing costs are well established. However, when the economics of producing the TiC-Ni_3Al composites were examined, a significant cost was associated with the use of the NiAl precursor powder (about 55% of the total raw material cost). Because the costs of the starting raw materials can be a significant fraction of the total cost of a component, alternative materials for fabricating the cermets are of interest. Part of the reason for the high cost of the NiAl is that it is produced only as a specialty powder at present. Elemental nickel and aluminum powders are produced in sufficient quantities to be relatively cost-effective, so tests were done to maximize their usage in the composites. Currently, Al-30% Ni powder is produced in commercial-level quantities for use as a catalyst; consequently, it is a cost-effective alternative for an Ni-Al alloy for fabricating TiC-Ni_3Al composites.

Results

Large batch processing of TiC-Ni_3Al composites. As reported previously, a series of large batches (≥2 kg) was processed and sent to CoorsTek for green forming into rods. These were subsequently returned and sintered at Oak Ridge National Laboratory (ORNL) (Figure 1). The rod blanks were then sent to Cummins for final machining into test parts.

Figure 1. Portion of the as-sintered rods prior to machining into rig test parts.

Recently, several large batches of composite powder have been produced and sent to CoorsTek for injection molding testing. To date, five 3-kg mill runs (15 kg total) were processed of a composition of

50 % TiC-Ni_3Al (with 2 % molybdenum). The different runs were blended prior to shipping. Injection moldings tests are in progress.

Alternate precursors for Ni_3Al formation. All previous work used a combination of nickel and NiAl for an in-situ reaction to form the Ni_3Al. Nickel and aluminum powders are produced in sufficient quantities to be relatively cost-effective, so tests were done to maximize their usage in the composites.

Cermet compositions containing NiAl+Ni or the Al-30 Ni product produced by the catalyst manufacturer show good densification behavior, and the densities achieved were >99.5 % T. D. However, the samples using elemental aluminum (i.e., Ni+Al or Ni+Al+NiAl) show severe macroscopic cracking caused by differential shrinkage during densification (Figure 2). Typically, this behavior is a result of non-uniform heating of the compact. The most likely cause of the temperature difference was the exothermic reaction that occurs as a result of the reaction of the nickel and aluminum to form the Ni_3Al intermetallic. Using the heat of formation for the reactions of nickel and aluminum, the adiabatic temperature rise for the $3Ni+Al \rightarrow Ni_3Al$ reaction in the TiC-based composite is >1200°C (considering no heat loss to the surroundings) and as such is a maximum condition. Since the reaction commences at ~650°C, the temperature increase is higher than the melting point of the Ni_3Al. In contrast, the adiabatic temperature rise in the composite for the $NiAl+2Ni \rightarrow Ni_3Al$ reaction is <300°C. Such an increase would not be sufficient to lead to significant melting of the Ni_3Al that is formed.

The two compositions that did not crack were examined in greater detail for mechanical properties and microstructures. The toughness and hardness were very similar for the different cermets; however, the fracture strength for the sample made by the reaction of Al-30 Ni (catalyst precursor) and nickel had very poor strength compared with the other composite (Figure 3). Examination of the microstructures revealed a significant amount of a second phase within the composite made with the Al-30 Ni (catalyst precursor), as shown in Figures 4 and 5. X-ray diffraction could not identify any other phases besides the TiC and Ni_3Al typically observed in these materials.

Figure 2. Visual appearance of samples after sintering at a heating rate of 10ºC/minute. Sample compositions: NiAl+Ni (left), Ni+Al (center), and NiAl+Ni+Al (right).

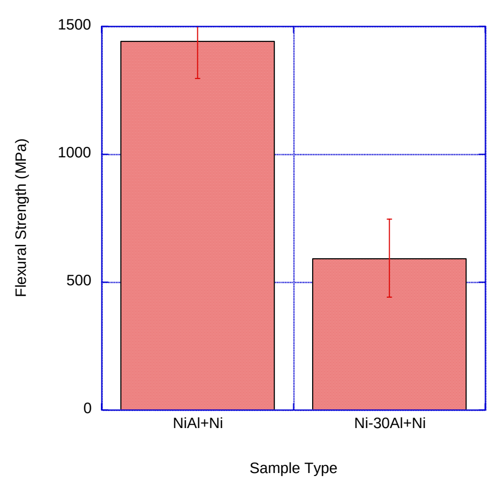

Figure 3. Results from tests of fracture strength of composites made with either NiAl+Ni or Al-30 Ni+Ni.

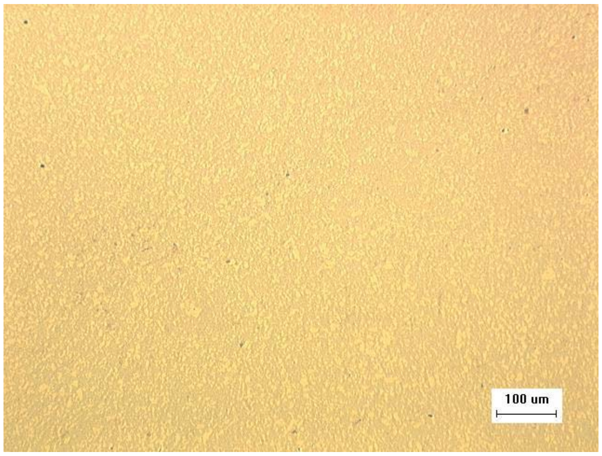

Figure 4. Microstructure of sample made with NiAl+Ni showing uniform distribution of TiC grains in Ni_3Al matrix and no secondary phases.

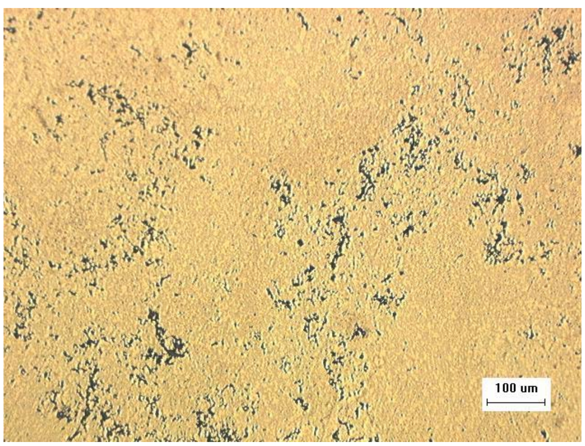

Figure 5. Microstructure of sample made with Al-30 Ni+Ni showing a second phase (black spots) distributed within the TiC and Ni_3Al.

Conclusions

It has been determined that the Ni-Al precursor type can have a significant effect on the densification behavior and final mechanical properties of TiC-Ni_3Al composites. When elemental aluminum was used, the samples exhibited macroscopic cracking as a result of differential heating, and shrinkage resulting from the exothermic reaction between the nickel and aluminum. Reducing the aluminum content and slowing the heating rate during the exothermic reaction did not eliminate the cracking problem. Samples fabricated using the Al-30 Ni precursor exhibited a second phase in the microstructure that is believed to be the cause of the low observed strength. Only the composites made from the NiAl + Ni combination had mechanical properties that were acceptable for engine applications.

Publications/Presentations

T. N. Tiegs, F. C. Montgomery, and P. A. Menchhofer, "Effect of Ni-Al Precursor Type on Sintering and Properties of TiC-Ni_3Al Composites," to be published in *Proceedings of International Conference on Powder Metallurgy and Particulate Materials*, Las Vegas, NV, June 2003.