Metals and Ceramics Division

Heavy Vehicle Propulsion Materials Program
Quarterly Progress Report for
April through June 2001

D. R. Johnson
Technical Project Manager

Prepared for
U.S. Department of Energy
Assistant Secretary for Energy Efficiency and Renewable Energy
Office of Transportation Technologies
EE 07 01 00 0

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6066
Managed by
UT-Battelle, LLC
for the Department of Energy
Under contract DE-AC05-00OR22725

CONTENTS

Cost Effective High Performance Materials and Processing

Development of Low Cost Austenitic Stainless Diesel Engine Components with Enhanced High-Temperature Reliability (ORNL and Caterpillar, Inc.)

Cost-Effective Smart Materials for Diesel Engine Applications (ORNL)

Low-Cost Manufacturing Processes for Ceramic and Cermet Diesel Engine Components (SIU-C)

Low Cost High Toughness Ceramics (ORNL)

Utilization of Thermally Conductive Graphite Foam for Cooling Diesel Engine Blocks (ORNL)

Advanced Manufacturing Technology

Intermetallic-Bonded Cermets (ORNL)

Low-Cost Manufacturing of Precision Diesel Engine Components (ORNL)

Cylindrical Wire Electrical Discharge Machining (EDM) and Temperature Measurement (North Carolina State University)

Testing and Characterization

NDE Technology for Heavy Duty Diesel Engines: Fuel Delivery and Insulating Materials (Argonne National Laboratory)

NDE Development for Ceramic Valves for Diesel Engines (Argonne National Laboratory)

Processing and Characterization of Structural and Functional Materials for Heavy Vehicle Applications (North Carolina A&T State University)

Life Prediction of Diesel Engine Components (ORNL)

Field Emission Analytical Electron Microscopy for Characterization of Catalyst Microstructures (ORNL)

Durability of Diesel Engine Component Materials (ORNL)

Materials and Testing Standards

Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications (ORNL)

Standards for Reliability Testing of Heavy Vehicle Propulsion Materials (NIST)

Mechanical Property Testing and Standards Development (NIST)

Development of Low-Cost Austenitic Stainless Diesel Engine Components with Enhanced High-Temperature Reliability

P.J. Maziasz and R.W. Swindeman
Oak Ridge National Laboratory
and
M.E. Frary and M.T. Kiser
Caterpillar, Inc.

Objective/Scope

The objective of this work is to evaluate cast austenitic stainless steels as a high-performance alternative to SiMo ductile cast iron, which is currently the standard material used in most diesel engines for exhaust manifold and turbocharger housing components. The new material must be able to withstand prolonged exposure at temperatures of 750°C or above, as well as to survive the severe thermal cycling from near room temperature to such high temperatures without developing cracks. This project has tested commercially available cast alloys, as well as developed new, modified cast alloys with significantly enhanced performance. The ultimate project goal is to provide high-performance, reliable materials that are also cost effective for such applications. This advanced diesel engine work is part of a broader CRADA (Cooperative Research and Development Agreement) project (ORNL99-0533) that began July 21,1999, and was scheduled to end July 22, 2001, but has been extended for 6 more months. Any more detailed information on this project must be requested directly from Caterpillar, Inc.

Technical Highlights, 3rd Quarter, FY2001

Background

Advanced large diesel engines must have higher fuel efficiency as well as reduced exhaust emissions, with similar or greater durability and reliability. Therefore, exhaust manifold and turbocharger housing materials must withstand temperatures ranging from 70 to 750°C or higher in a normal duty cycle that includes prolonged, steady high-temperature exposure as well as more rapid and severe thermal cycling. New technology to reduce emissions like exhaust gas recirculation (EGR) will likely push temperatures in these critical components even higher. Current exhaust components are made from SiMo ductile cast iron, and increased engine temperatures would push such materials beyond their current strength and corrosion limits. Therefore, new data is needed on alternate higher performance materials like cast austenitic stainless steels to guide materials selection and component lifing. There is limited industrial experience on cast austenitic stainless steels like CN12, especially for diesel engine applications, and little high-temperature mechanical properties data available, particularly creep and fatigue. The main purpose of this program is to produce data comparing cast CN12 and SiMo cast iron for such diesel exhaust component applications, with a parallel alloy development effort to optimize the cost-effective performance of such cast austenitic stainless steels.

Approach

Commercial cast CN12 austenitic stainless steel (Fe-25Cr-13Ni-1.8Nb, C, N, S) was chosen as the baseline alloy for evaluation relative to SiMo ductile cast iron. Materials were evaluated in the as-cast condition as well as after appropriate thermal aging at temperatures up to 850°C. Tensile and isothermal cyclic fatigue properties were evaluated selectively from room temperature up to 900°C, together with some high-temperature creep rupture and oxidation/corrosion testing. Microstructure analysis was performed on some of the specimens to better understand mechanisms associated with the properties changes. In a parallel effort, new alloying element modifications were made to CN12 and studied to further enhance the high-temperature performance.

Technical Progress

To date for alloy selection, the commercial cast iron and baseline CN12 materials have been obtained, all mechanical properties specimens machined, and all tensile and creep testing of unaged and aged specimens have been done. High-cycle isothermal fatigue up to 700°C has been completed, and similar fatigue testing at higher temperatures, and thermal fatigue over this temperature range is in progress. Microstructural analyses of selected specimens of as-cast or as-cast and aged materials have been completed or are in progress to understand the properties behavior and to establish the effects of aging on the baseline CN12 material. CN12 has a clear tensile strength advantage (Figure 1) and an overwhelming creep-strength advantage (Figure 2) over SiMo cast iron above 550-600°C.

To date, eight smaller heats (15 lbs each) of modified CN12 austenitic stainless steels have been produced at ORNL and have been evaluated. Preliminary screening of as-cast or cast and aged material with tensile testing, and of as-cast material with creep-rupture testing at 850°C indicates that significant improvements have been achieved in high-temperature strength and aging resistance. The improvement of the best modified CN-12 alloys is five to ten times better relative to the standard commercial CN-12 alloy for creep-rupture testing at 850°C and 110 MPa.

In addition to the modified CN12 austenitic stainless steels, similar efforts were made to modify a less costly and more castable CF8C (Fe-19Cr-12Ni-Nb, C) austenitic stainless steel that has previously been considered as a candidate for diesel exhaust applications, and which also finds use in some gas-turbine exhaust components at 650°C or below. Three smaller heats of modified CF8C were made at ORNL (one baseline unmodified alloy and two modified alloys) and screened using tensile tests and creep-rupture testing at 850°C. The dramatic improvement in creep-resistance of the modified CF8C alloys at 850°C relative to commercial material is shown in Figure 3. The two modified CF8C alloys are several orders of magnitude better than the standard commercial steel, and are still in test, with times exceeding 12,000 h. Additional creep testing done this last quarter shows that the best-modified CF8C steels have creep strength that is the same or slightly better than standard, commercial CN12 steel at the same test conditions, and those tests will also continue into next quarter.

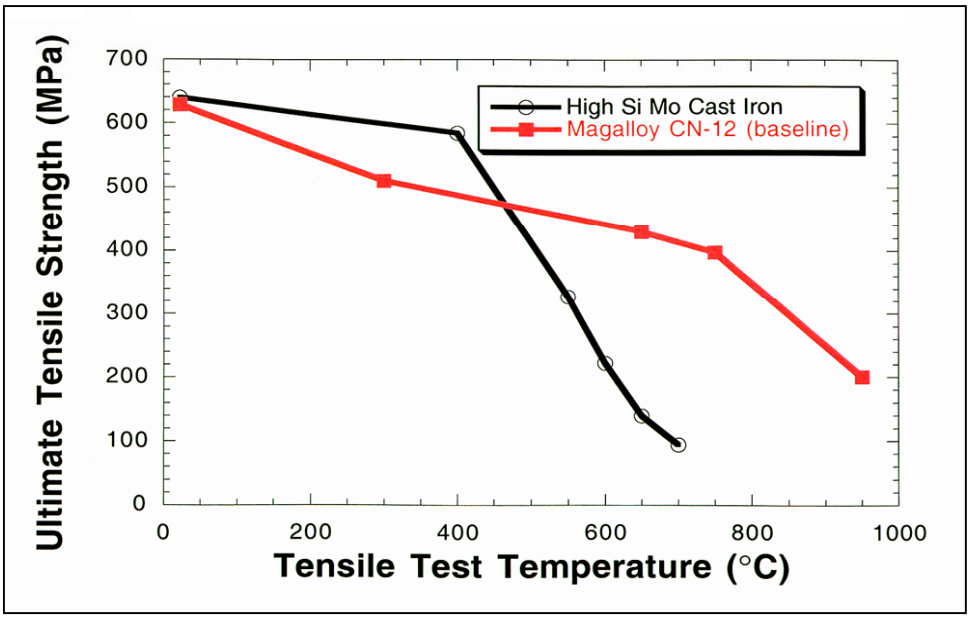


Figure 1. Plots of ultimate tensile strength versus test temperature for commercial SiMo cast iron and a commercial standard (baseline) CN-12 cast austenitic stainless steel.

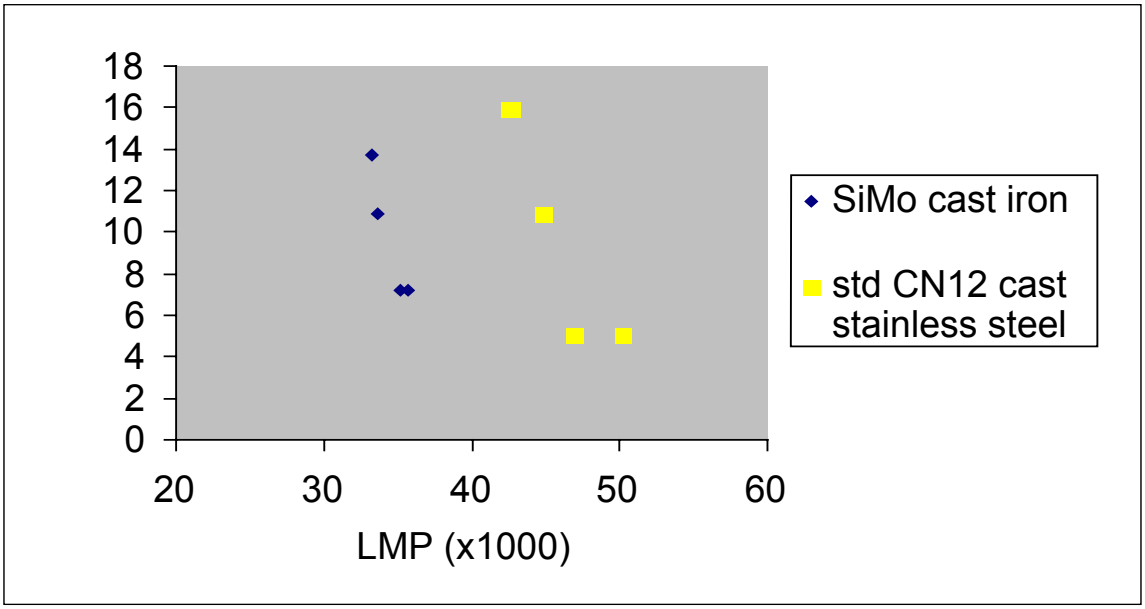


Figure 2. A plot of Larson-Miller Parameter (LMP = (T+ 460)(20+log t_r), where T is creep test temperature in degrees Farenheit and t_r is the time to rupture in hours) versus creep-rupture stress (vertical axis in ksi) for various creep tests run on SiMo cast iron (550-650°C) and commercial CN-12 cast austenitic stainless steel (750-900°C). Cast CN-12 austenitic stainless steel has an overwhelming advantage in creep strength.

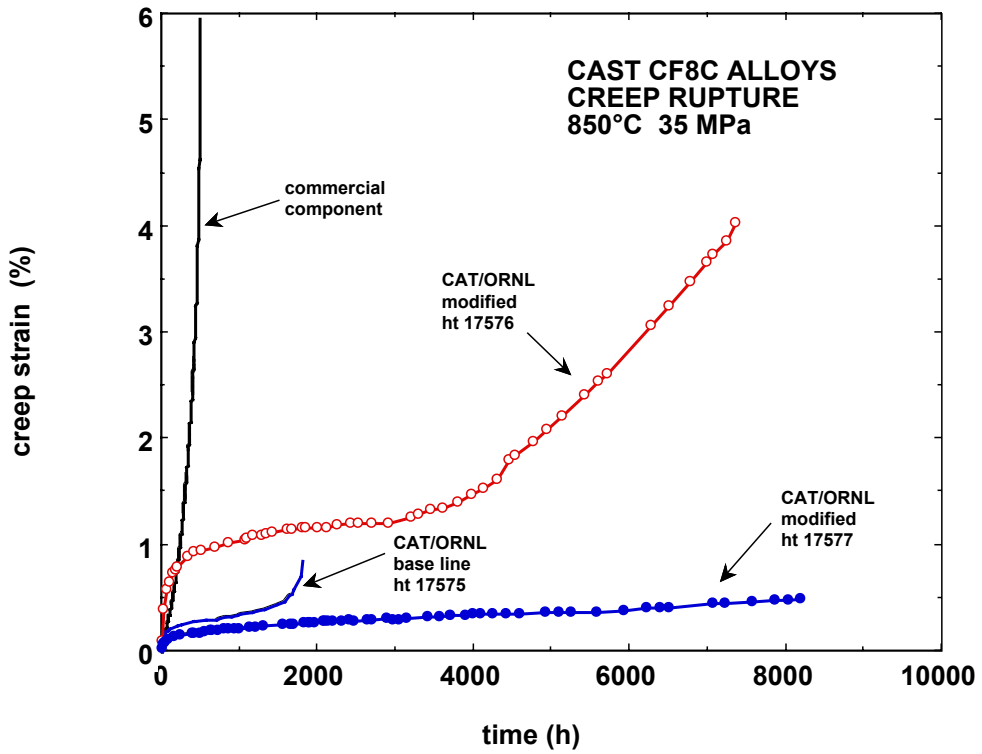


Figure 3. Creep-rupture curves of creep strain versus rupture time for a commercial standard CF8C cast austenitic stainless steel and several laboratory heats of baseline or modified CF8C cast austenitic stainless steel developed by ORNL and Caterpillar. Creep-rupture tests of the two best-modified CF8C steels are still on going, and exceed 12,000 h.

Communications/Visits/Travel

Team communications between ORNL and Caterpillar occur on a regular basis. As part of the user-agreements imbedded in the CRADA, Mike Pollard of Caterpillar worked for 1 week at ORNL in January 2000, and Tim McGreevy (formerly at Caterpillar and now at Bradley University) worked for 2 weeks at ORNL in June 2000. Phil Maziasz of ORNL visited the Caterpillar Technical Center in Mossville, IL, for technical discussions and to summarize the progress of this project. Chad Siebenaler took over the project from Tim McGreevy, and is summarizing the technical results of the project, but has moved to another portion of the Caterpillar technical organization. Megan Frary is a recent Caterpillar employee who has taken over this project.

An invention disclosure and patent application on cast austenitic stainless alloys with improved performance were completed by Caterpillar and filed with the U.S. Patent Office in December 2000. It is entitled "Heat and Corrosion Resistant Cast Stainless Steels With Improved High

Temperature Strength and Ductility," by P. J. Maziasz (ORNL), T. McGreevy (U. of Bradley/CAT), M. J. Pollard (CAT), C. W. Siebenaler (CAT), and R. W. Swindeman (ORNL).

Status of Milestones

Formal milestones are imbedded in the CRADA and are not part of the HVPM Program FWP. However, all milestones have been achieved on or significantly ahead of schedule.

Publications

None.

Cost-Effective Smart Materials for Diesel Engine Applications

J. O. Kiggans, Jr., T. N. Tiegs, F. C. Montgomery
and L. C. Maxey
Oak Ridge National Laboratory

Objective / Scope

There are two objectives for this project. The first is to evaluate the cost-effectiveness and maturity of various "Smart Materials Technologies," which are under consideration for diesel engine applications, such as fuel injection systems. The second is to develop "Smart Materials" to be incorporated into working actuators and sensors.

Task 1 - Multilayer Electroded Laminates
The purpose of this study is to find satisfactory methods for the preparation of PZT laminates with internal electrodes from tape cast materials. This task is in progress with no new results to share in this report.

Task 2 – Compositional Alteration of PZT-4
Experiments are under design to improve the Qm or loss factor of low-sintering compositions, which have been discussed in previous reports.

Task 3 – Fabrication of Piezo Amplifier Device

Space constraints in present fuel injector designs limit the size of piezoelectric actuators that may be utilized to open and close the fuel valve. Due the small stroke (25 μm) delivered by a standard, commercial 25-mm-long piezoelectric stack, stroke amplifier devices are needed to increase the overall valve stroke to >100 μm which is needed by the injector device.

Because of unique ORNL experience in motion-amplifying devices and the need of the diesel companies for this type of hardware in fuel injectors, a subproject was begun to fabricate cost-effective, compact amplifiers. To define the overall size of the device, a piezoelectric stack was purchased which could provide at least 25 μm of stroke and 250 lb force. Table 1 shows the specifications for the EDO Inc. Model E100P-4 stack actuator. Using this information, an ORNL amplifier design was scaled to provide >100 μm stroke with >50 lb of output needed for fuel injection operation. A drawing of the first prototype amplifier produced for this purpose is shown in Figure 1. Figure 2 shows areas of high stress in the amplifier calculated by the finite element software "Cosmos." This model shows areas of high stress. The first prototype was fabricated using 7075 aluminum alloy, a grade of aluminum that is employed by the aircraft industry for areas of the plane that undergo repeated cyclical motion, for example the wings. Figure 3 shows photos of the final amplifier, including the EDO piezoelectric stack.

Testing will begin next quarter to determine the maximum speed and displacement for the piezoelectric amplifier assembly.

Status of Milestones

- Evaluate and characterize commercially available PZT materials. Seek methods to improve the properties of these materials through alternative processing and forming methods. Fabricate new PZT compositions that allow sintering of the PZT materials at lower temperatures.
  Status: The evaluation of new commercial materials and low temperature PZT materials is in progress.
- Initiate studies on important variables in the fabrication of multilayer co-fired PZT stacks. Fabricate multilayer PZT stacks for testing.
  Status: Co-fire studies are presently in progress.
- Use ORNL expertise in motion amplifier devices to design and fabricate motion-amplifying fixtures for fuel injector assemblies.
  Status: An amplifier fixture has been designed at ORNL. The first amplifier has been fabricated.

Communications/Travel/Visits

None

Publications

None

Table 1. Performance specifications of EDO Inc. model E100P-4 stack actuator

Displacement Range (μm)	0 - 24
Diameter (mm)	9
Length (mm)	25.2
Maximum Preload (lb)	200
Stiffness (N/μm)	70
Free Capacitance (nFarads)	110
Frequency Range Hz	DC to 1000
Actuator Weight (grams)	9
Peak Voltage (V)	800

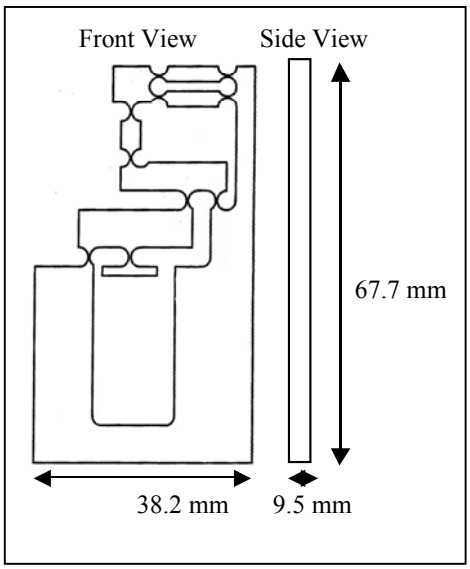


Figure 1. Drawing of ORNL amplifier

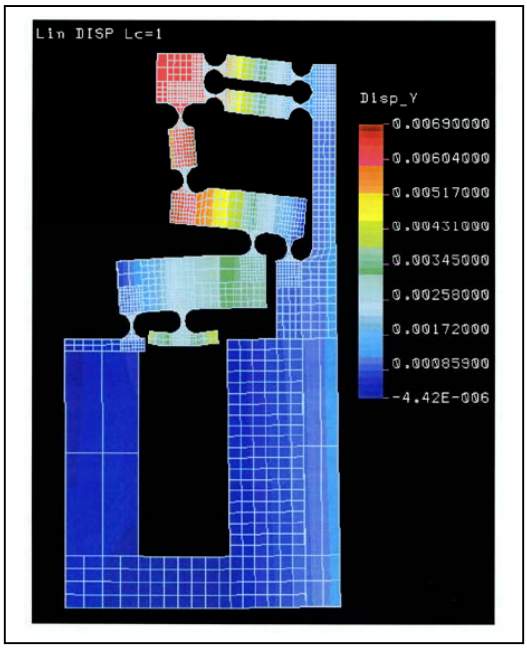


Figure 2. Cosmos stress analysis of ORNL amplifier

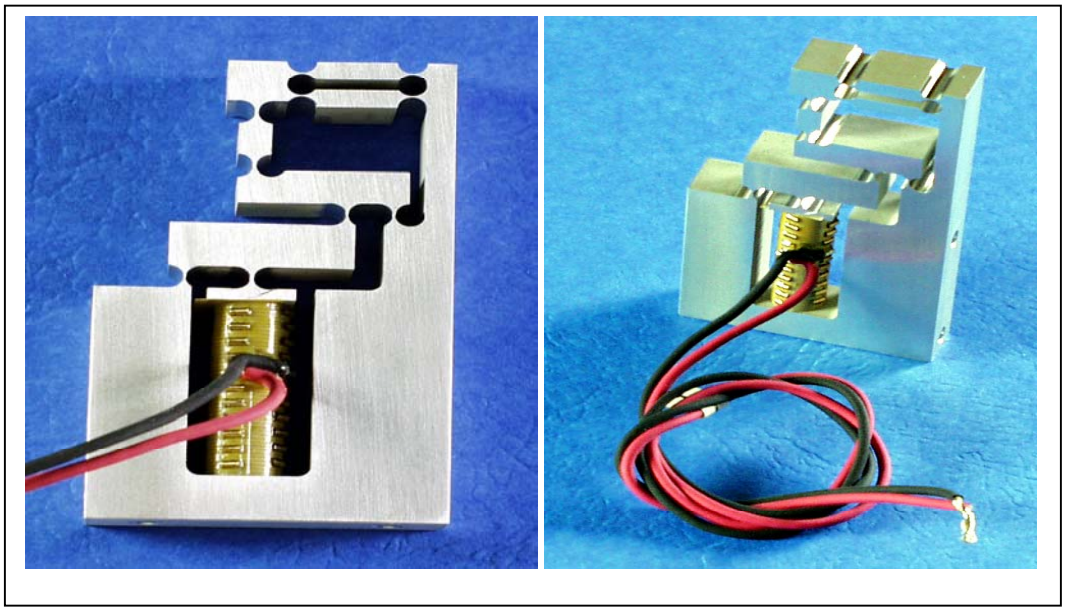


Figure 3. Photo of assembled EDO piezoelectric stack and ORNL amplifier

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

D. E. Wittmer
Southern Illinois University

Objective/Scope

The purpose of this work is to investigate the potential of low-cost manufacturing processes for ceramic and cermet diesel engine components. The primary task is to develop cost-effective processing, forming and sintering methodologies for cermet and ceramic formulations used by industrial diesel engine manufacturers.

Technical Highlights

Task 1. Collaboration with industrial partner(s).

This task involves the collaboration with industrial partners to assist them in processing and sintering of their diesel engine components. Our goal is to provide assistance in processing and sintering which may result in a reduction in surface reactions and part warping. Moreover, this may also provide an alternative sintering process that will allow improved throughput efficiency and manufacturing economy. Due to the proprietary nature of this task, any research data generated from this task is normally controlled by the terms of each specific confidentiality agreement. The reporting of this data and any results are the responsibility of the industrial partner(s).

Task 2. Cost Effective Processing and Sintering

During this reporting period, concentration continued on binder removal of the low-pressure injection molded cermets. Five batches of NiCrFe-TiC and 4 batches of Ni3Al-20%Fe-TiC cermets were processed. All of the batches contained 40 vol % of the intermetallic and 60 vol % TiC (Starck high-purity grade). The objectives were to obtain near fully dense cylinders and have them ground into test bars for hardness and strength measurements and for observation of the microstructures.

For the NiCrFe batches, the highest density obtained was 95.4% of theoretical for a 60 vol % solids loading. The binder ratio was 55:44:1 (petroleum jelly:parafin wax:stearic acid), and the injection temperature was 160°F at a pressure of 60 psi. These specimens were sent out for machining/finishing.

For the Ni3Al-20Fe batches, the highest density obtained was 92.9% of theoretical for the 60 vol % solids loading. The binder ratio was 54.5:43:5.2 (petroleum jelly:parafin wax: stearic acid), and the injection temperature was 160°F at a pressure of 60 psi. These

specimens were sent out for machining into test bars and the hardness and 4-point flexural strength were measured. The hardness varied from a low of 292 kg-mm/s^2 to a high of 951 kg-mm/s^2, and the 4-point flexural strength varied from a low of 403 MPa to a high of 972 MPa. The variability in this data was a direct result of large random pores that resulted from the processing and sintering with the present lot of TiC powder. In previous work full density was achieved for continuous sintering of the same formulations. Typical microstructures are shown in Figure 1 for the fully dense cermets. It is believed that higher oxygen contents of the powders and potentially some residual binder, caused by incomplete removal, have affected the wetting of the TiC by the intermetallic. In addition, surface oxygen, that may not be removed during continuous sintering, may be partly responsible for these results.

These problems are currently being investigated and the binder removal process is being adjusted accordingly. In addition, a fine-screening step has been added to the injection molding process to reduce TiC agglomerates in the injection-molding batches.

The batch sizes have also been increased to reduce the effects of low volume in the injection molder and it appears that the trapped porosity has been reduced.

Status of Milestones

1. Collaboration with Industrial Partners	On Schedule
2. Cost Effective Processing and Sintering	On Schedule

Communications/Visits/Travel

D. E. Wittmer to ORNL to discuss program objectives and new initiatives with D. Ray Johnson and Terry Tiegs.

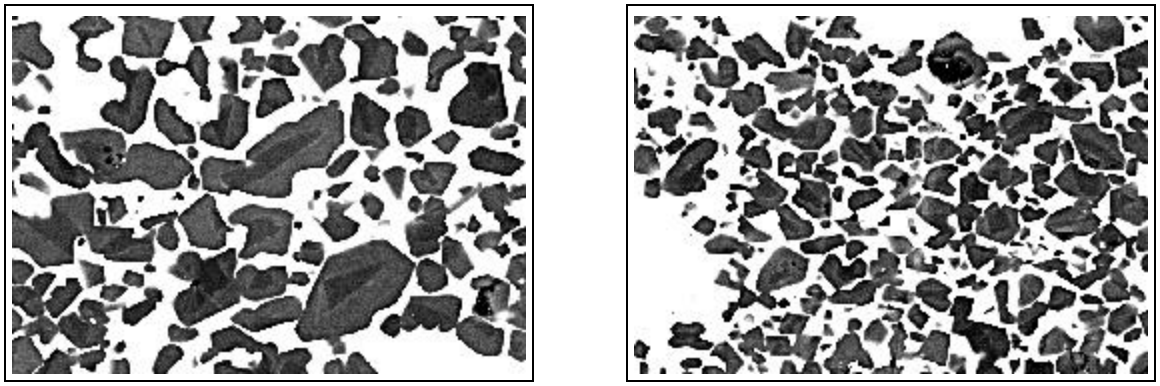
Graduate student, Jeffrey Hazelwood started his internship at ORNL as part of the ORISE program and will be transferring the injection molding technology to ORNL.

Problems Encountered

None

Publications and Presentations

D. E. Wittmer, A. Woods, F. Goransson, T. Tiegs, and P. Menchhofer, AContinuous Sintering of NiCr and NiCrFe-TiC Intermetallics,@ Metal Powder Industries Federation, Princeton, NJ, presented at the APMI-MPIF conference in New Orleans, LA, May 13-17, 2001.



(a) 10 μm (b) 10 μm

Figure 1. Microstructures of continuously sintered 40 vol % NiCr-TiC, sintered at 1450 EC using a belt speed of (a) 18.3 mm/minute and (b) 36.6 mm/minute, showing the finer TiC grains achieved for the higher belt speed.

Low Cost-High Toughness Ceramics

T. N. Tiegs, F. C. Montgomery, P. A. Menchhofer, and D. L. Barker
Oak Ridge National Laboratory

Objective/Scope

Significant improvement in the reliability of structural ceramics for advanced diesel engine applications could be attained if the critical fracture toughness (K_{Ic}) were increased without strength degradation. Currently, the project is examining toughening of ceramics by two methods: microstructure development in oxide-based ceramics, and incorporation of ductile intermetallic phases.

Technical Highlights

Previous studies have shown that the properties of the aluminide-bonded ceramics (ABC) are attractive for diesel engine applications and consequently, development of these materials was started. At the present time, TiC-40 vol % Ni_3Al composites are being developed because they have expansion characteristics very close to those for steel. Preliminary wear testing indicated that improved wear resistance could be achieved by decreasing the grain size of the TiC. Achieving fine grain size with the high binder contents is difficult because of the large inter-grain distances. In addition, it was thought that changing the TiC grain shape from a highly faceted one to a more-rounded, equiaxed grain would reduce localized stress at sharp corners. This, in turn, would improve abrasion resistance from any wear debris. Consequently, grain-size refinement is presently being studied. Several approaches can be used to control the final TiC grain size. The methods studied in the present report include: (1) use of additives to change the interface behavior of the growing TiC grains, and (2) reduction of the initial TiC particle size.

Reduction of Oxygen Pickup During TiC Milling by Use of Additives

Previous work has shown significant oxygen pickup when the TiC was milled to particle sizes <0.5 µm. One of the methods to minimize oxygen pickup by non-oxide materials is to add surfactants that will absorb onto the 'fresh' surfaces and physically prevent oxygen from interacting with the surface. Several samples were fabricated (as indicated in Table 1), where oxalic acid or polyvinylpyrrolidone (PVP) were used to inhibit oxygen pickup. Another method, that is used in the hardmetals industry, is to mill the carbide powders with the metal particles and 'smear' the metal over the carbide surfaces. This method was also used as shown in Table 1.

The results on the oxygen pickup by the powders during processing have been reported previously. Flexural-strength testing is summarized in Fig. 1. As shown, significant variations in the observed strengths were obtained which were not readily explained. Examination of the fracture surfaces by SEM showed an unusual surface morphology on the tensile side of the test specimens. The bars were machined by EDM and the effects of this method are being examined further as an explanation of the variable strengths for this series of materials.

Additional samples have been fabricated to study the effect of Zr additions on the properties of the TiC-Ni_3Al cermets. Earlier work had suggested that Zr additions to the alloy improved

wetting of the TiC and could be beneficial to counteracting the effects of oxygen pickup during processing. Additions of NiZr, $ZrSi_2$, and ZrC were used as shown in Table 2. Results on the sintering behavior (also shown in Table 2) indicate the Zr-based additions have a serious detrimental effect on the densification. As a result, additional examination of these samples is not being planned.

Status of Milestones
On schedule.

Communications/Visits/Travel
Travel by T. N. Tiegs to Indianapolis, IN, April 15-18, 2001, to attend the American Ceramic Society Annual Meeting.

Travel by T. N. Tiegs to New Orleans, LA, May 13-17, 2001, to attend the PM^2TEC Conference and present a paper entitled "Microwave Sintering of TiC-Based Cermets."

Problems Encountered
None.

Publications
T. N. Tiegs, J. O. Kiggans, and P. A. Menchhofer, "Microwave Sintering of TiC-Based Cermets," to be published in Proceedings of PM^2TEC Conference (2001).

Table 1. Summary of processing variables on samples to minimize oxygen pickup for different TiC-40 vol % Ni_3Al composites.

Sample No.	Oxalic Acid Addition (wt %)*	PVP Addition (wt %)*	Ni & NiAl Milled With TiC	Sintered Density (% T. D.)
TMPO-1	2	0	No	100
TMPO-2	2	0	Yes	99.8
TMPO-3	1	1	Yes	99.1

Table 2. Summary of sample compositions using Zr-based additions to minimize oxygen pickup for different TiC-40 vol. % Ni_3Al composites .

Sample No.	Alloy Additive (wt %)*	PVP Addition (wt %)*	Ni & NiAl Milled With TiC	Sintered Density (% T. D.)
TMPO-5	7 NiZr (70:30)	2	Yes	85.4
TMPO-6	7 $ZrSi_2$	2	Yes	89.2
TMPO-7	5 ZrC	2	Yes	86.7

* Based on the TiC content.

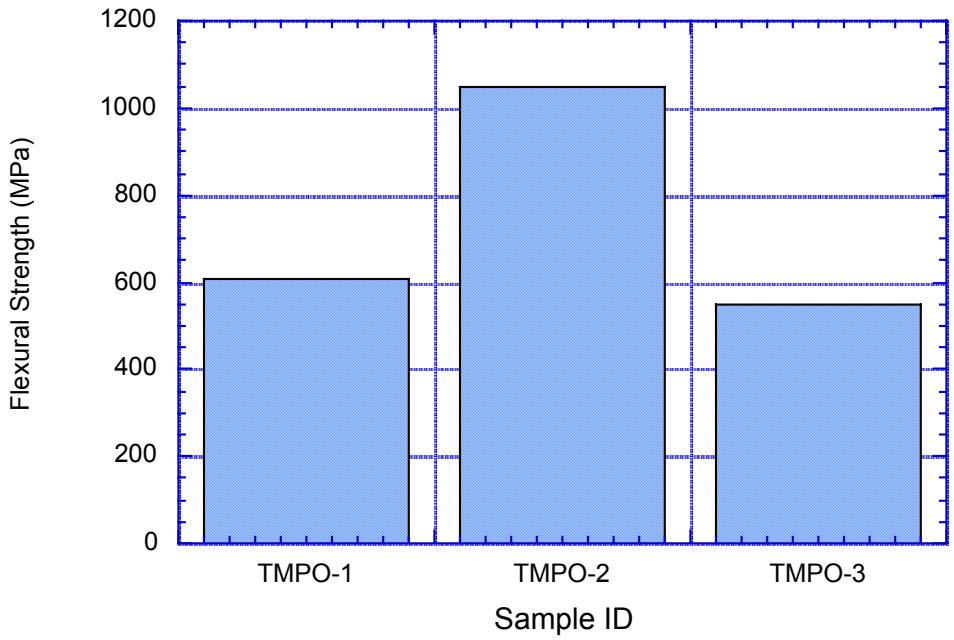


Figure 1 - Summary of flexural strengths for TMPO-samples.

Utilization of Thermally Conductive Graphite Foam for Cooling Diesel Engine Blocks

Ronald D. Ott and James W. Klett
Oak Ridge National Laboratory

Objective

The objective of this effort is to determine the feasibility of using the high thermally conductive graphite foam, developed at Oak Ridge National Laboratory, as a cooling medium in a diesel engine block. Cooling problems exist in certain areas of diesel engines that are not adequately cooled by water. There are attempts to cool these areas by the lubricating oil but hot spots around the cylinder block and head still exist. It may be possible to strategically place the graphite foam in these areas to aid in rapidly removing, or redistributing, the heat to the nearest cooling path, be it oil, coolant, or air.

In recent years there have been efforts to study the performance of, and methods to improve, the cooling system of diesel engines. For a diesel engine to operate at optimal performance, the thermal stability of the engine is critical. Cooling of the engine block is accomplished by the coolant, lubricating oil, exhaust, and losses due to radiation and convection to the surroundings. Approximately 36% of heat removed is achieved by the coolant and the lubricating oil. Almost half of that 36% of the heat removed is accomplished by lubricating oil passages that have been placed in areas where there are high levels of heat, due to inadequate cooling from the coolant. By effective controllable cooling, the life of the engine can be increased, as can the fuel economy and performance of the diesel engine.

Technical Highlights

A meeting was held in July with Ron Ott and Dave Stinton from ORNL and Tim Theiss, Robert Wagner, Norberto Domingo, and Jim Conklin from the Advanced Propulsion Technology Center to discuss ways in which the graphite foam could be utilized in cooling diesel engine blocks.

The initial discussion was focused on determining locations within the diesel engine where hot spots commonly occur. One area under consideration was around the edges of the exhaust valves, but consensus was that in order to incorporate a cooling device manufactured from graphite foam, considerable alterations to the manufacturing process would have to be undertaken. Another area that could incorporate a device such as a heat pipe would be between siamese cylinder bores where there is no cooling. Such a device could greatly reduce cylinder distortion, thus increasing engine life.

Heat pipe configurations were discussed and it was discovered that this is not a new concept for engine cooling, but was evaluated in the 1980s. There were cracking issues, leading to sodium leaks, and the heat pipes were very expensive. The group did not go into any depth discussing heat pipe configuration utilizing graphite foam. The discussion continued with evaluating other areas in which the graphite foam could be utilized in the engine block. Several ideas came up including oil pans, oil windage trays, transmission coolers and housings, radiator reservoir tanks, and torque converters.

Contacts have been made at Thermacore/Modine, Caterpillar, and ArvinMeritor. Each of these companies has the ability to contribute to the study considering their diverse areas of expertise. Thermacore is an industry leading company in thermal management with extensive background in heat pipe manufacturing. Caterpillar would be an ideal partner to help in identifying the feasibility of introducing the graphite foam into the diesel engine block for cooling purposes. ArvinMeritor is a world leader in aftermarket products for the automotive and heavy truck industry, and could contribute in evaluating add-on cooling components to diesel engines.

In July, a meeting was held with James Klett, April McMillan, Nidia Gallego, and Ron Ott of ORNL, Stephen Memory of Modine, and Jon Zuo of Thermacore. Although this meeting was not geared toward evaluating the graphite foam in cooling engine blocks, interest was shown and the topic was discussed. Jon Zuo is to contact Ron Ott with the appropriate person at Thermacore to investigate the feasibility in further detail.

From that meeting, a plan of action was laid out to effectively evaluate the performance of the graphite foam in a heat pipe configuration. Thermacore is able to advise ORNL on how to tailor the graphite foam's properties to better suit a heat pipe configuration and manufacturing. A simplified version of a heat pipe utilizing the graphite foam can be manufactured to evaluate its performance. In conjunction, discussions with Caterpillar and Advanced Propulsion Technology Center need to be ongoing in order to evaluate areas within the engine where heat pipes would be advantageous from the cooling aspect, as well as practicable from the manufacturing aspect.

Mike Ready of Caterpillar visited ORNL in July and showed interest in assessing the feasibility of using the graphite foam in cooling diesel engine blocks, other than radiator applications. The appropriate contact at Caterpillar will be identified to further investigate our interaction.

Sam Ciray of ArvinMeritor visited ORNL in June and showed interest in developing the graphite foam for heat pipe applications. ArvinMeritor was excited at the possibilities the graphite foam holds for heat management. An appropriate contact at ArvinMeritor is to be appointed for further communication.

Status of Milestones

The first milestone, evaluating the possibilities of utilizing the graphite foam for cooling diesel engine blocks, has begun. Significant industrial partners have been identified and the proper contacts within each organization are currently being determined.

Communications/Visits

No visits to report, but upcoming trips to the aforementioned organizations are to be scheduled.

Publications

No publications to report.

Intermetallic-Bonded Cermets

P. F. Becher and S. B. Waters
Oak Ridge National Laboratory

Objective/Scope

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). This will require materials which have a minimum hardness of 11 GPa and a thermal expansion coefficient of between 10 to 15 x 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 not cause excessive wear of the steel counter face. The upper temperature limit for fuel delivery systems applications is 540°C, and for the other wear applications, the limit is 815°C. Finally, the total material processing costs for these advanced materials should be competitive with competing technologies such as TiN or other ceramic coatings on high-speed tool steels.

Technical Highlights

The linear thermal expansion coefficients of several TiC-Ni_3Al cermets prepared in a collaborative effort between CoorsTek and ORNL were evaluated; these results were found to be comparable to those reported in the Second Quarterly Report.[1]

In the past, considerable effort was expended in successfully developing processing methodologies that are (1) consistent with industrial practice, (2) user friendly, and (3) economical. This included means to generate sinterable submicron TiC powders by attritor milling that is economically viable. The methodologies also maintain the combined high fracture strength (≥ 1GPa) and high fracture toughness (up to 18 MPa√m) that make these cermets so attractive. Currently, a study has been initiated of processing factors that might influence the optimization of the properties with scale-up in production levels.

Status of Milestones

On Schedule

Communications/Visits/Travel

On June 12-13, discussions were held with Jim Stephan of AMT/CoorsTek on the properties of larger cermet components prepared by reaction sintering in collaboration with T. N. Tiegs of ORNL. A list of action items was developed aimed at optimizing the processing of the components.

On July 12, a presentation on the development of TiC-Ni_3Al cermets was presented to the staff of Caterpillar.

Publications

P. F. Becher, K. P. Plucknett, and C.-H. Hsueh, "Toughening and Strengthening Response in Ni_3Al–Bonded Titanium Carbide Cermets," *Zietschrift für Metallkunde*, in press.

References

1. Heavy Vehicle Propulsion Materials Program, Quarterly Progress Report for January through March 2001, Oak Ridge National Laboratory, Oak Ridge, TN (<http://www.ms.ornl.gov/programs/energyeff/hvpm/quarterly2.pdf>).

Low-Cost Manufacturing of Precision Diesel Engine Components

S.B. McSpadden, Jr.
Oak Ridge National Laboratory

Objective/Scope

To develop and demonstrate optimized, cost-effective fabrication processes for the production of precision components for use in diesel engines. To develop and demonstrate optimized, cost-effective, non-destructive testing methods for the detection and prevention of machining-induced damage in engine components.

Technical Highlights

Caterpillar CRADA – The final report for CRADA No. ORNL98-0501 has been completed and issued. This brings to a close a multi-year, multi-task effort to address the manufacturing issues associated with the use of ceramic materials for diesel engine components. Follow-on collaborations are currently being planned and will address manufacturing issues related to cylinder head inserts, valves, seals, and fuel injector components.

Collaborations with Cummins Engine Company and CoorsTek – Cummins Engine Fuel Systems Division, CoorsTek, and ORNL are working jointly to evaluate non-destructive testing methods for the detection of machining damage in ceramic fuel system components. In addition to OHVT support, the work is being partially funded by the State Partnership Program. The state energy offices in Indiana and Colorado will participate in the dissemination of results.

A group of 150 transformation-toughened zirconia rods has been received from CoorsTek. The rods are being ground into a rectangular shape suitable for MOR bars, after which they will be divided into groups of 30. The excess stock will be removed under conditions chosen to deliberately generate varying degrees of machining damage. The scanning acoustic microscope at ORNL is being repaired and will be used to evaluate the ground specimens.

Communications/Visits/Travel

Sam McSpadden attended the Quality Expo in Chicago to evaluate the current state of the art of non-destructive test equipment that might be useful for detecting machining damage.

Cylindrical Wire Electrical Discharge Machining (EDM) and Temperature Measurement

Albert Shih
North Carolina State University

Objective/Scope

To develop precise, efficient, and cost-effective cylindrical Wire Electrical Discharge Machining (WEDM) process for cermets and other electrically conductive advanced engineering materials, and temperature-measurement methods for grinding and diesel exhaust aftertreatment devices.

Technical Highlights

Three highlights in this quarter are the surface finish improvement, investigating the surface integrity by SEM, and testing of wire EDM fine grain size cermet developed by ORNL.

1. Surface finish

The goal was to adjust EDM process parameters to achieve the best possible surface finish. Two types of materials, brass and carbide, were used. The carbide material is tungsten carbide with 10% cobalt bond. After conducting preliminary cylindrical wire EDM experiments, two process parameters, the pulse on-time and wire feed rate, were identified to have significant effects on surface finish. Key process parameters for Experiments I and II are listed in Table 1.

Four pulse on-times and three wire feed rates were tested for brass and carbide. The cutting configuration shown in Fig. 1 with $\alpha = 0$, $R = 2.59$ mm, and $r = 2.54$ mm was used. A thin, 50 μm layer of work-material was removed. This so called "skim cut" helps improve the surface finish. A small pitch of p=0.01 mm was set for all tests to minimize surface roughness.

Table 1. Machine Setup for Experiments I and II.

Work-material	Carbide	Brass
Spark cycle (μs)	28	20
Pulse on-time (μs)[#]	2~5	2~5
(Percentage of on-time)	(7~18%)	(10~25%)
Wire tension (g)	1800	1800
Gap voltage (V)	57	57
Wire feed rate v_f (mm/min)[#]	0.25, 0.375, 0.5	0.5, 1.0, 1.5
Part rotation speed ω (rpm)[+]	25, 37.5, 50	50, 100, 150

[#] denotes the set up parameters

Figure 2 shows the surface finish results. The shorter pulse on-time and lower feed rate, in general, created better surface finish. At the shortest pulse on-time, 2 μs, significant decreases in the surface roughness can be observed. Shorter pulse on-time generates smaller sparks, which, in turn, creates smaller craters and better surface finish. This can be verified in the SEM micrographs of EDM surfaces. The best R_a generated on carbide is 0.68 μm. These values are

comparable to that of rough grinding, which makes the cylindrical wire EDM process suitable for precision machining of the difficult-to-machine materials.

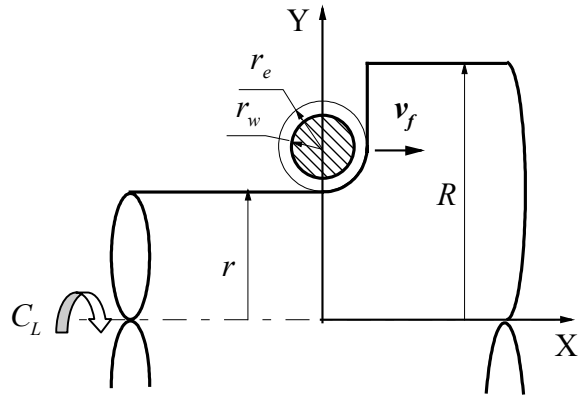


Figure 1. The configuration of roundness experiment

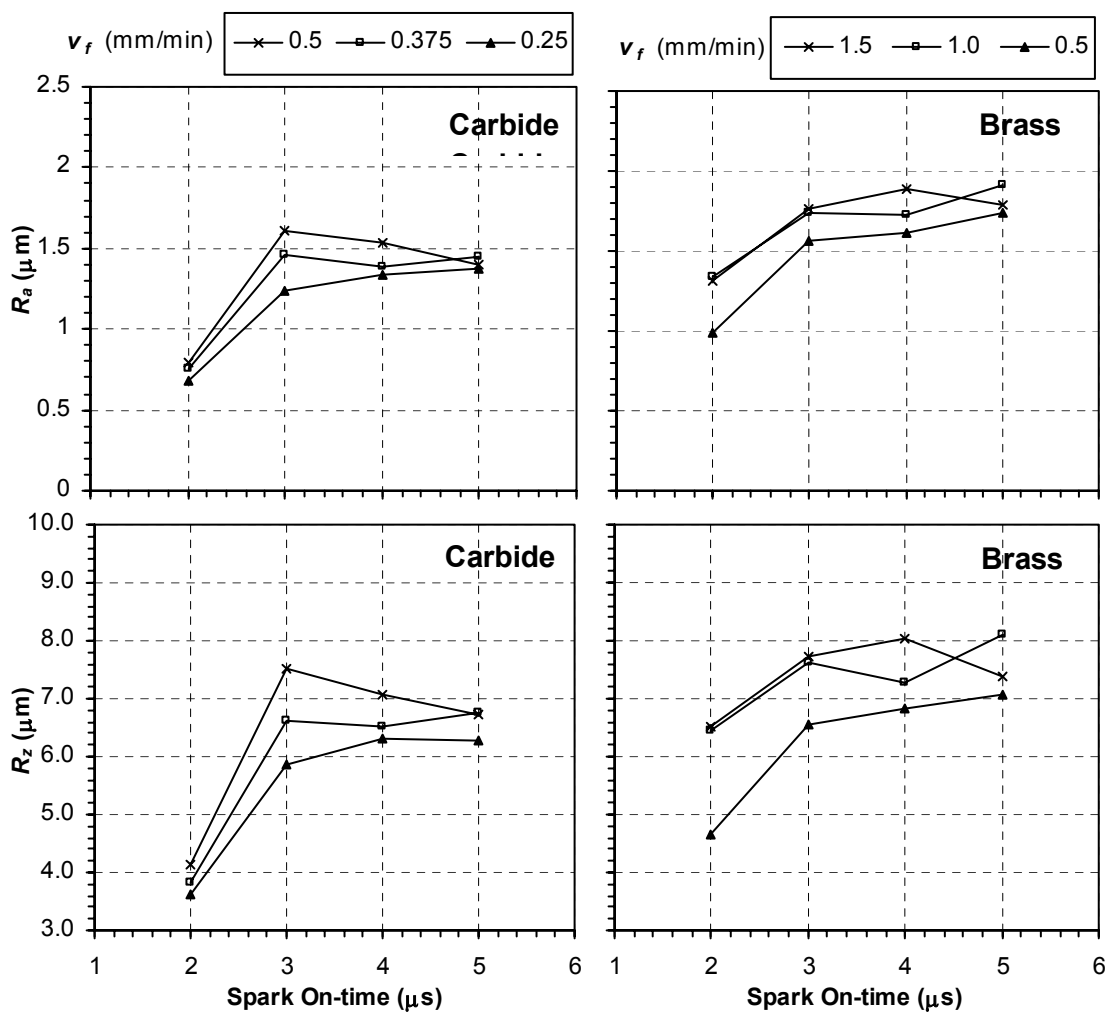


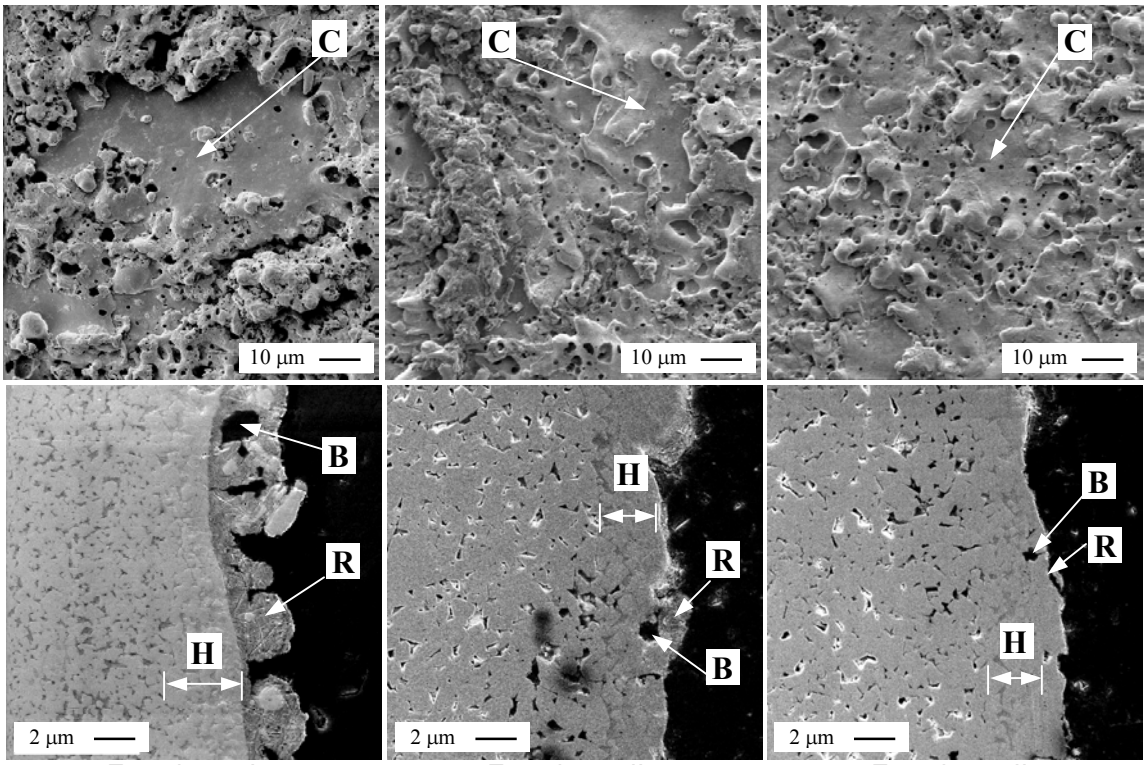
Figure 2. The surface finish of cylindrical wire EDM parts

2. SEM Micrographs of EDM Surface and Sub-Surface

SEM is used to examine the surface and sub-surface of cylindrical wire EDM carbide and brass parts. The cylindrical samples were sliced in the radial direction. Surfaces of sliced cross-sections were polished to observe the sub-surface damage. The craters on the surface, and the recast layers and heat-affected zones in the sub-surface of the cylindrical wire EDM parts are presented in the following two sections.

2.1 Craters

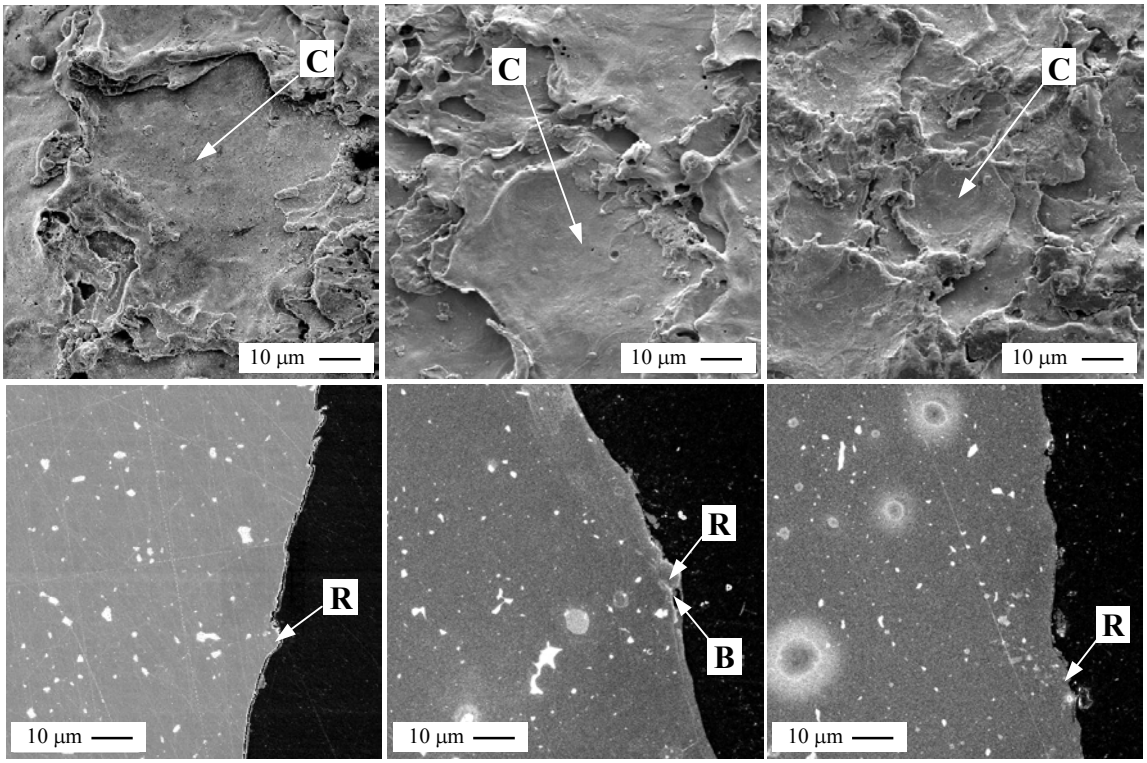
The rough surfaces are observed using the SEM machine to compare the surface texture and crater size. As illustrated in the surface finish results in Fig. 2, the short, 2 μs, pulse on-time generates fine surface finish. Under shorter pulse on-time, electrical sparks generate smaller craters on the surface. For carbide parts, as shown in Fig. 3, the rough estimate of the crater size is about 50, 30, and 20 μm under 14, 5, and 2 μs pulse on-time, respectively. As shown in Fig. 4, slightly bigger craters, estimated as 60, 40, and 25 μm, can be seen on the brass parts machined under the same pulse on-time. On carbide parts, about 1-μm-size tungsten carbide grain can be seen on the SEM micrographs in Fig. 3.



	Experiment I	Experiment II	Experiment II
R_a (μm):	2.53	1.40	0.68
On-time (μs):	14	5	2
ω (rpm):	30	50	25
v_f (mm/min):	1.22	0.5	0.25
MRR (mm^3/min):	13.9	5.70	2.85

Legend – **C**: Crater, **R**: Recast layer, **B**: Bubble, **H**: Heat affected zone.

Figure 3. SEM micrographs of surfaces and cross-sections of carbide parts



	Experiment I	Experiment II	Experiment II
R_a (μm):	3.19	1.78	0.98
On-time (μs):	14	5	2
ω (rpm):	30	150	50
v_f (mm/min):	2.54	1.5	0.5
MRR (mm^3/min):	29.0	17.1	5.70

Legend – **C**: Crater, **R**: Recast layer, **B**: Bubble.

Figure 4. SEM micrographs of surfaces and cross-sections of brass samples

2.2 Sub-Surface Recast Layers and Heat-Affected Zones

The recast layer is defined as the material melted by electrical sparks and resolidified on the surface without being ejected or removed by flushing. Below the recast layer is the heat-affected zone. For the carbide material, the cobalt matrix melts and resolidifies in the heat-affected zone. The molten cobalt fills the pores in the tungsten carbide. This is observed in the SEM micrographs of the carbide cross-section and is used to identify the depth of heat-affected zone. Rajurkar and Pandit [1] have studied the recast layer and heat-affected zones of EDM surfaces and developed a thermal model to predict the thicknesses of damaged layers. For the die-sinking EDM process, the depth of the damaged layer was reported to be from 30 to 100 μm for an AISI 4130 steel workpiece machined with pulse on-time of 100 to 300 μs [2]. Anon [3] has studied the sub-surface heat-affected zones and recast layers of steel and tungsten carbide using the die-sinking EDM and has summarized and explained the possible causes for EDM surface defects.

SEM micrographs of the cross-sections of carbide and brass parts machined under both high and low MRRs (Experiments I and II) are shown in Figs. 3 and 4. On carbide parts, the μm-size carbide grains can be identified. The recast layer, bubbles in the recast layer, and heat affected

zone of three carbide samples are identified in Fig. 3. Under high MRR at 14 μs pulse on-time, the recast layer, about 3 μm thick, can be clearly recognized on the surface. Thinner recast layers, less than 2 μm, exist on samples machined using shorter pulse on-time. Bubbles can be identified in the recast layers of all three carbide samples. Anon [3] has proposed that these micro-bubbles were generated by thermal stresses and tension cracking in the recast layer. As shown in Fig. 3, the depth of the heat-affected zone is estimated to be about 4, 3, and 2 μm on the three carbide samples with 14, 5, and 2 μs pulse on-time, respectively.

As shown in Fig. 4, very thin recast layers, about 1 μm, can be observed on the cross-section of three brass samples. No heat-affected zone can be recognized on brass samples, possibly due to the good thermal conductivity of brass. The heat-affected zone may exist but cannot be identified in brass samples. For carbide, the contrast of porosity filled by the molten cobalt makes the identification of the heat-affected zone easier. For brass material, such a phenomenon does not exist. This has made the heat-affected zone difficult to be identified.

3. Cylindrical wire EDM of Fine Grain-Size Cermet

A preliminary wire EDM test on the fine-grain-size cermet developed by a team led by Dr. Paul Becher has been conducted. Initial test results indicate that this cermet, TiC in Ni_3Al matrix, has a material removal rate comparable to that of carbide for cylindrical wire EDM. A micropin, as shown in Fig. 5, was machined. The diameter of the pin is about 50 μm. This shows the possibility of using the cylindrical wire EDM process to machine the fine-grain-size cermet for micro-mechanical components and micro machine/device applications.

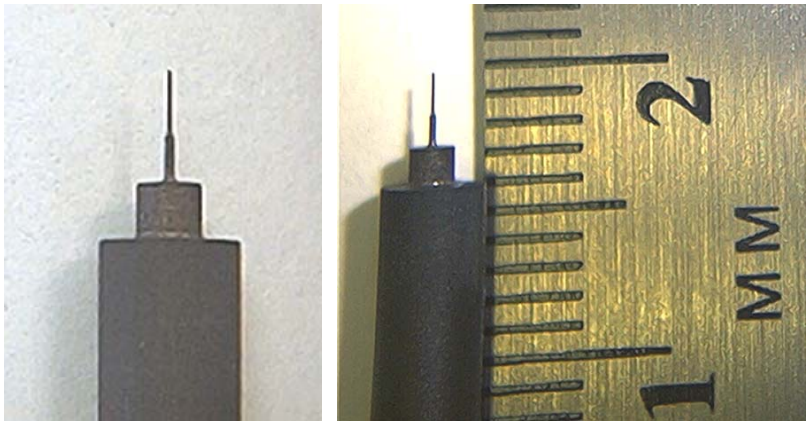


Figure 5. Cermet micropin machined by the cylindrical wire EDM

<u>Status of Milestones</u>

Milestone 1: Develop process technology to achieve high material removal rate in cylindrical WEDM of advanced engineering materials.

Status: Nothing to report. Experiments have been conducted to quantify the maximum material removal rate. Results showed the maximum material removal rate in cylindrical wire EDM is higher than the conventional 2D wire EDM.

Milestone 2: Determine the level of form tolerances and surface finish achievable by the cylindrical WEDM.
Status: Experiments were conducted at the High Temperature Materials Lab at Oak Ridge National Lab to measure the surface finish. The surface finish was as low as 0.68 μm Ra, comparable to rough grinding, for the carbide materials.

Milestone 3: Prototype needles, armature pins, and plungers for diesel engine fuel injectors.
Status: Several prototype diesel injector plungers were manufactured. Exploratory experiments to machine miniature shafts in the size under 0.1 mm diameter were conducted.

Milestone 4: Develop mathematical models for material removal rate and surface finish of the cylindrical WEDM.
Status: Noting to report. The mathematical models for material removal rate and surface finish have been derived. These results have been summarized in a technical paper.

Milestone 5: Characteristics of the recast layer after WEDM and cylindrical WEDM and the surface condition after abrasive blasting.
Status: Experiments were conducted at High Temperature Materials Lab at Oak Ridge National Lab to use the SEM to observe the surface and cross-section of cylindrical wire EDMed brass and carbide parts.

Milestone 6: Development of optical-fiber-based temperature measurement method, with applications for grinding process and diesel exhaust aftertreatment filters.
Status: Adam Curry finished his MS thesis defense and is summarizing his research results in two technical papers, to be submitted to the Journal of ACS.

Communications/Visit/Travel

John Kong, William Clark, and Albert Shih, visited Oak Ridge National Lab and Selee at Hendersonville, NC, on May 21 - 24, 2001.

John Kong is working at the High Temperature Materials Lab, Oak Ridge National Lab. from June 1 to August 1, 2001, to study the infrared-based grinding temperature measurement.

Albert Shih, Ron Scattergood, and three NCSU students (Craig Hardin, Darrin Poirier, and Scott Miller) visited ORNL and Selee on July 2nd and 3rd, 2001.

Publications

1. Qu, J., Shih, A. J., and Scattergood, R., "Development of the Cylindrical Wire Electrical Discharge Machining Process, Part I: Concept, Design, and Material Removal Rate," Submitted to *Journal of Manufacturing Science and Engineering.*
2. Qu, J., Shih, A. J., and Scattergood, R., "Development of the Cylindrical Wire Electrical Discharge Machining Process, Part II: Surface Integrity and Roundness," Submitted to *Journal of Manufacturing Science and Engineering.*
3. Qu, J., Shih, A. J., and Scattergood, R., "Development of the Cylindrical Wire Electrical Discharge Machining Process," *Symposium on Nontraditional Manufacturing Research and*

Applications, 2001 ASME International Mechanical Engineering Congress and Exposition at New York, NY (to appear).

4. Qu, J., Shih, A. J., Scattergood, R. and McSpadden, S. B., "Cylindrical Wire Electrical Discharge Machining Process Development," ASPE 16th Annual Meeting at Arlington, Virginia (to appear).

References

[1] Rajurkar, K. P. and Pandit, S. M., 1984, "Quantitative Expressions for Some Aspects of Surface Integrity of Electro-Discharge Machined Components," Transactions of ASME Journal of Engineering for Industry, Vol. 106, No. 2, pp. 171-177.

[2] Rajurkar, K. P. and Royo, G. F., 1989, "Effect of R.F. Control and Orbital Motion on Surface Integrity of EDM Components," Journal of Mechanical Working Technology, Vol. 20, pp. 341-352.

[3] Anon, 1987, "Controlling EDM Surface Integrity," American Machinist & Automated Manufacturing, Vol. 131, No. 11, pp. 80-83.

NDE/C Technology for Heavy Duty Diesel Engines: Fuel Delivery and Insulating Materials

W. A. Ellingson
Argonne National Laboratory

Objective/Scope

The objective of the work in this task, part of the Testing and Characterization research area of the Heavy Vehicle Propulsion System Materials Program, is to develop enabling nondestructive evaluation/characterization (NDE/C) technology for lower-cost and high-performance materials. Specifically, this project addresses development of advanced NDE/C technology for: a)-advanced fuel delivery systems (including injector nozzles), and b)-insulating materials for reducing heat losses in the combustion zone. Fuel delivery systems for heavy-duty diesel engines are complex, very expensive, and represent a significant portion of the cost of a heavy vehicle diesel engine. High pressures inside these fuel delivery systems contribute to poor fuel delivery and, hence, poor emissions. Materials development is part of Goal 3 of the heavy vehicle propulsion materials program. Insulating materials are also a significant materials development area because of the improvement in engine efficiency if reduced heat losses can be obtained. NDE/C technology that can provide information for reliable cost production and engine component surveillance would be of benefit to reaching the goals.

Technical Highlights

The highlight this period is that we have improved the experimental set up for the polarized, back-scattered laser light for detection of subsurface machining-induced or operational-induced cracking on ceramic fuel metering plungers. We set up an angle-scattering laser lab which incorporated polarization maintaining optical fibers.

Technical Progress

Work this period again focused on the ceramic fuel metering plungers under development by Cummins Engine Company of Columbus, IN.

Fuel Delivery Systems

Fuel delivery systems are critical and impact many engine performance parameters. Higher pressures required for advanced fuel systems are driving new designs and as with any design, material trade-offs become an area for evaluation. Ceramic materials provide many advantages, but only if there is a reduced cost with the same or better reliability as that of a metal component. High pressures and potential for erosion inside fuel metering systems usually call for hard tool steels in the design and, thus, costs immediately become a factor. Ceramics offer a desirable option because they offer high hardness and erosion resistance. As noted last period, Cummins Engine Company has optioned in their CELECT™ fuel injector system to use MgO materials in the metering plunger.

The efforts we have been undertaking on MgO plungers have been to study the potential of non-contact polarized back-scattered laser light methods to detect subsurface machining damage in these plungers.

All initial work was done using normal incidence laser scattering and the results to detect surface-breaking cracks on these very translucent materials were not as successful as we had hoped. Therefore, this period we began an effort to use angle scattering wherein the laser light and the detected scattered light are incident at a grazing angle. This is better shown by looking at Figure 1.

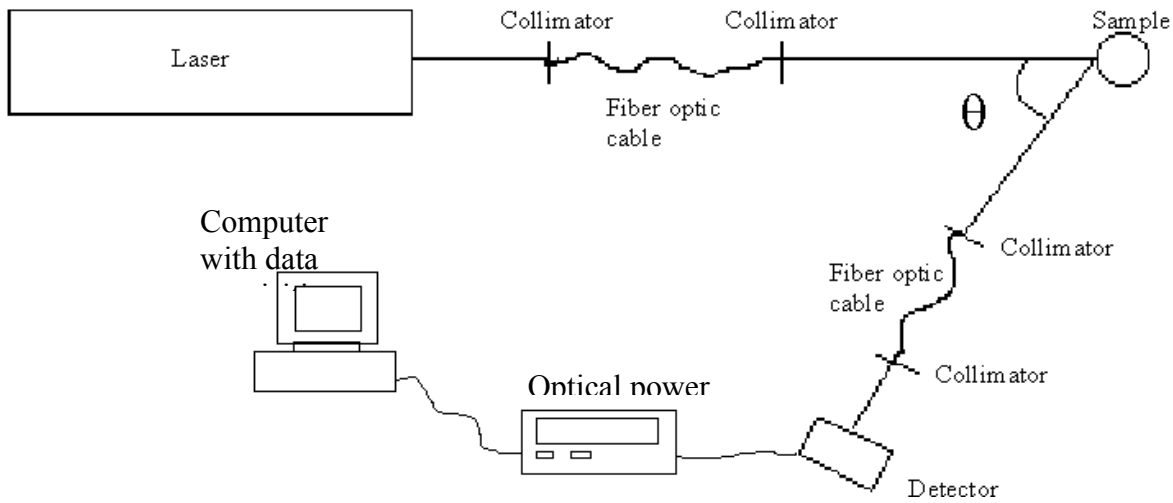


Figure 1. Schematic diagram showing incident angle scattering set up used for studies this period.

In order to establish the best signal level, scattered light intensity as a function of angle between the incident and detected light was evaluated. Figure 2 shows the data which suggest that the included angle of about 20-22 degrees is the best angle.

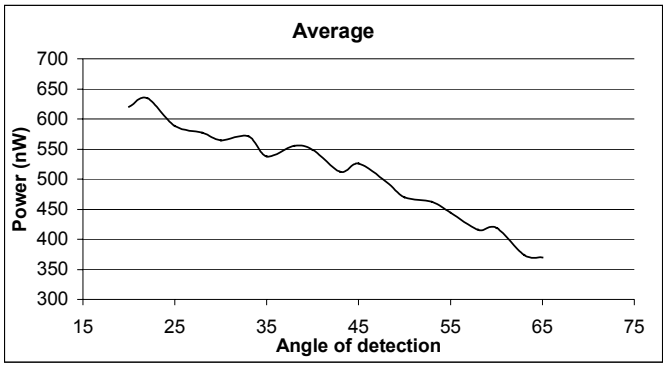


Figure 2. Relationship between detected signal level and included angle between incident light and detector.

Using this data for setting the maximum signal detection, tests were conducted this period on samples received from Cummins Engine Company which had been run in a test cylinder for between 75 and 145 hours. Because of certain internal engine conditions, some of the plungers sustained slight damage which is visible on the surface, but the depth of damage is unknown. We used one of these samples to establish the detection sensitivity to 3 cracks. The results are shown below in Figure 3.

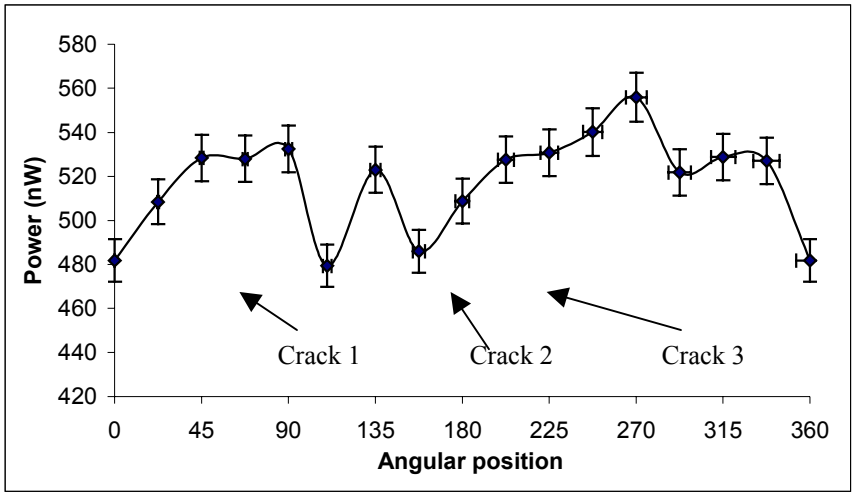


Figure 3. Detection of three surface-breaking cracks on zirconia fuel injector pin using angle-of-incidence laser scatter.

The low power level detected corresponds exactly to the locations of the three cracks. We will continue to pursue this method, including the use of fiber optics, during the next period.

NDE for Insulating Materials

We continue work on developing this aspect of this effort.

Meetings, Trips, Communications

A-Meetings

1. W. A. Ellingson participated in the review held at Cummins Engine Company in Columbus, IN, June 13, 2001.

B-Trips

1. W. A. Ellingson traveled to Columbus, IN, to participate in the Cummins Engine review held June 13, 2001.

C-Communications

1. Discussions were held with staff of Cummins Engine in Columbus, IN.
2. Discussions were held with staff of Enceratec of Columbus, IN.

NDE Development for Ceramic Valves for Diesel Engines

J. G. Sun, W. A. Ellingson,
Argonne National Laboratory
and
M. J. Andrews
Caterpillar, Inc.

Objective/Scope

Emission reduction in diesel engines designated to burn fuels from several sources has lead to the need to assess ceramic valves to reduce corrosion and emissions. The objective of this work is to evaluate several nondestructive evaluation (NDE) methods to detect defects/damage in structural ceramics valves for diesel engines. There are four tasks to be carried out in this work: (1) Establish correlation of NDE data with mechanical properties for fatigue/wear damaged samples. These include rotary fatigue, dynamic fatigue, impact/sliding wear, and possible thermal shock testing. The induced damage will be assessed using various NDE methods including dye penetrant, optical scatter, impact acoustic resonance (IAR), X-ray CT, thermal imaging, and others. NDE data will be correlated with those from characterization tests including mechanical strength, SEM, and others. (2) Develop NDE techniques for ceramic-metal joints. Effort will be focused on developing new methods as well as evaluating existing NDE techniques to characterize quality of ceramic-metal joints. NDE data will be correlated with mechanical data. (3) Conduct NDE studies of full-size engine valves. In this part of the work, full-sized ceramic valves will be produced for testing in a single cylinder test engine. These valves will be examined using NDE techniques developed at Argonne. (4) Finalize correlation of NDE to machining damage. This limited effort will conclude the earlier work to correlate NDE data with mechanical properties of machining-damaged Si_3N_4 ceramics. Elastic optical scatter method will be used to examine these specimens, and the NDE data will be correlated with flexural strength.

Technical Highlights

Work during this period (April-June 2001) focused on correlating laser-scatter data with fracture location for flexure-bar specimens, and on conducting NDE studies for detection of interface cracks in ceramic-metal joints.

1. Elastic Optical Scattering NDE for Machining Damage

During this period we started to correlate laser-scatter data with fracture locations on machined surfaces of silicon nitride flexure-bar specimens. AS800 specimens were examined first. Most of the AS800 specimens were broken at two locations during the flexure-strength test, as seen in Fig. 1. These surfaces had been scanned by elastic optical scattering before the mechanical tests. It was hoped that the laser scatter technique may detect the fracture initiation spot on the machined surface, by matching the fracture location (or fracture line) with features detected in the laser-scatter data. Figure 2 shows correlations of optical micrographs with laser-scatter sum images at two fracture locations in AS800 flexure-bar specimen #5. The laser-scatter images show no apparent machining damage (i.e., machining lines), because these specimens were machined with optimized standard-machining conditions. At the fracture location 1, the fracture line passes a large subsurface defect as indicated, and at the fracture location 2, the fracture line seems to start from a large subsurface defect at the lower edge. These correlations seem to suggest that these subsurface defects are likely the fracture initiation spots for the AS800

specimen. This result is typical for all AS800 specimens. Examination of the fracture surfaces to identify the fracture initiation spots for AS800 specimens will be carried out in the next period. Correlations for Caterpillar GS44, CFI, and SN235 flexure-bar specimens will also be attempted.

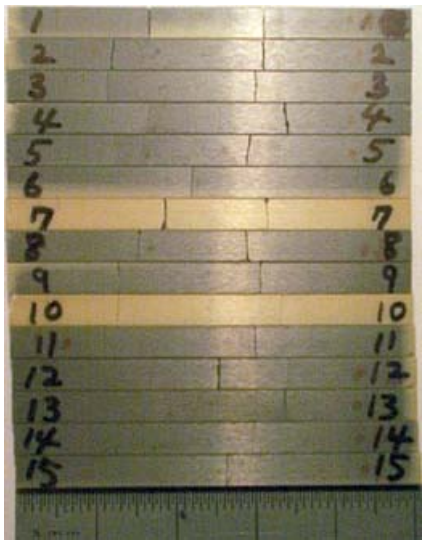


Figure 1. Photograph of fractured tensile surfaces of 15 AS800 flexure-bar specimens.

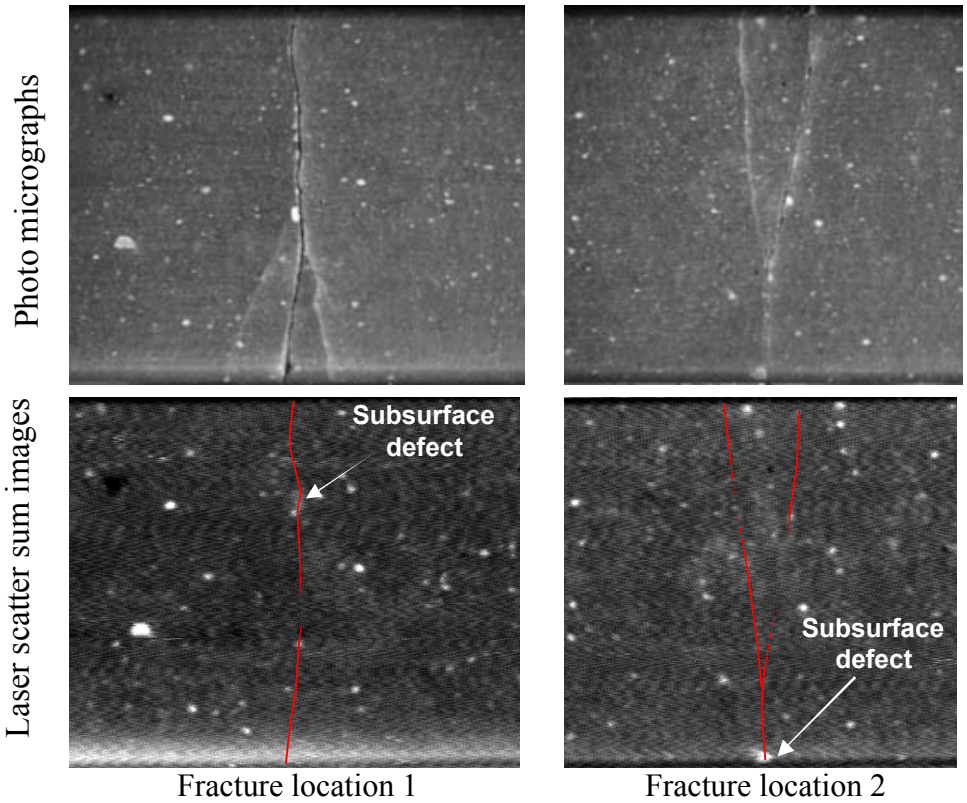


Figure 2. Correlation of fracture lines in micrographs and in laser-scatter sum images at two fracture locations on the tensile surface of AS800 specimen #5.

2. NDE Development for Ceramic-Metal Joints

2.1 Water-coupled ultrasonic scan

We continued water-coupled ultrasonic NDE study of large ceramic-steel joint samples for detection of interface cracks. The experimental setup, an angled pitch-catch configuration for the transmitter-receiver pair, was illustrated in Fig. 3 of the previous quarterly report. Previous tests used transducers of central frequency of 5 MHz and transducer angle of 5°. We now used 10 MHz transducers at angles 3°, 5°, and 7° to determine the optimum detection angle. As a reference, Fig. 3 shows the water-coupled ultrasonic scan image of the ceramic-steel-joint sample #4 at 5 MHz and 5° angle. No apparent features are observed. Figure 4 shows three ultrasonic scan images for the same joint sample at 10 MHz and at angles of 3°, 5°, and 7°. Individual features with higher transmission intensities are clearly seen in each image; the features are also correlated well between the images. These higher-transmission-intensity features are possibly the results of reflection of the angled ultrasonic beam at interface cracks. However, a correlation is yet to be determined. We suggest that, at some stage of this study, some of the joint samples should be destructively examined in order to establish the correlation. This work is been continued for other joint samples with 15 MHz-frequency transducers; results will be presented in the next quarterly report.

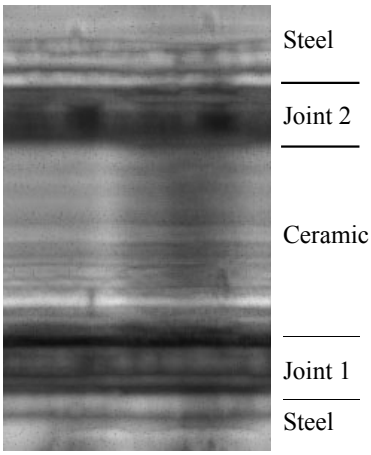
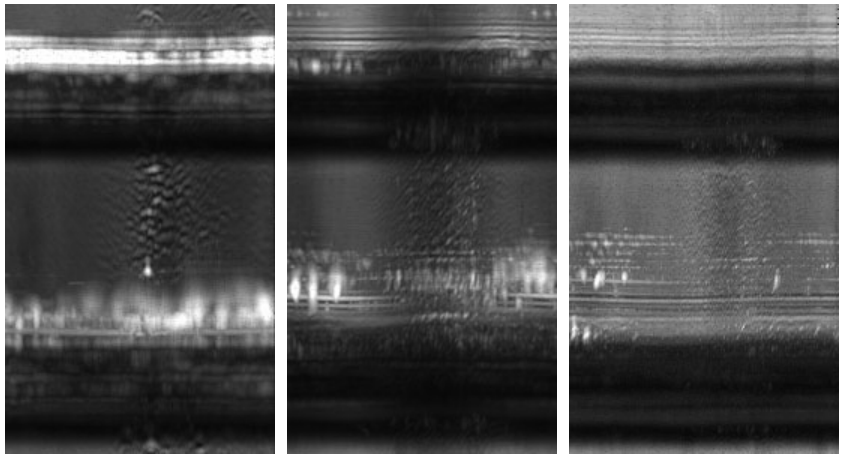


Figure 3. Ultrasonic scan image of large joint sample #4 with 5 MHz transducers at 5° angled pitch-catch transducer setup.



3° 5° 7°

Figure 4. Ultrasonic scan images of large joint sample #4 with 10 MHz transducers at 3°, 5°, and 7° angled pitch-catch transducer setup.

2.2 Infrared thermal imaging

An infrared thermal imaging NDE system was set up to evaluate the metal-ceramic joints. The joint sample is positioned vertically. During the test, the bottom of the joint (steel) is quickly immersed in boiling water, and the resulting upward heat transfer through the joint region is monitored by an infrared, thermal-imaging camera. The thermal imaging data are proportional to surface temperature, and the slope of the temperature profile in the axial direction is calculated at each pixel and mapped into an image. The slope is approximately proportional to the inverse of the thermal conductivity of the material. This heat transfer process was numerically simulated with an 1-D transient heat transfer code for a joint sample with an air-gap of 0.5-µm thickness at one interface. The calculated transient temperature profiles at various time steps are plotted in Fig. 5; it is seen that the crack causes a temperature drop ~0.5°C which should be easily detected by the infrared camera (the camera has a temperature resolution better than 0.01°C). However, cracks in the joint interfaces are typically not extended over the entire cross section and the infrared camera is usually operated at a lower sensitivity because of the large temperature ranges during the test and the low surface emissivity of the joint samples. So a realistic temperature drop across a crack in the joint may be much smaller. Figure 6 shows a thermal imaging test result for a joint. The image is relatively noisy, but shows clearly the different materials in the sample. This technique needs further improvement on both sensitivity and spatial resolution.

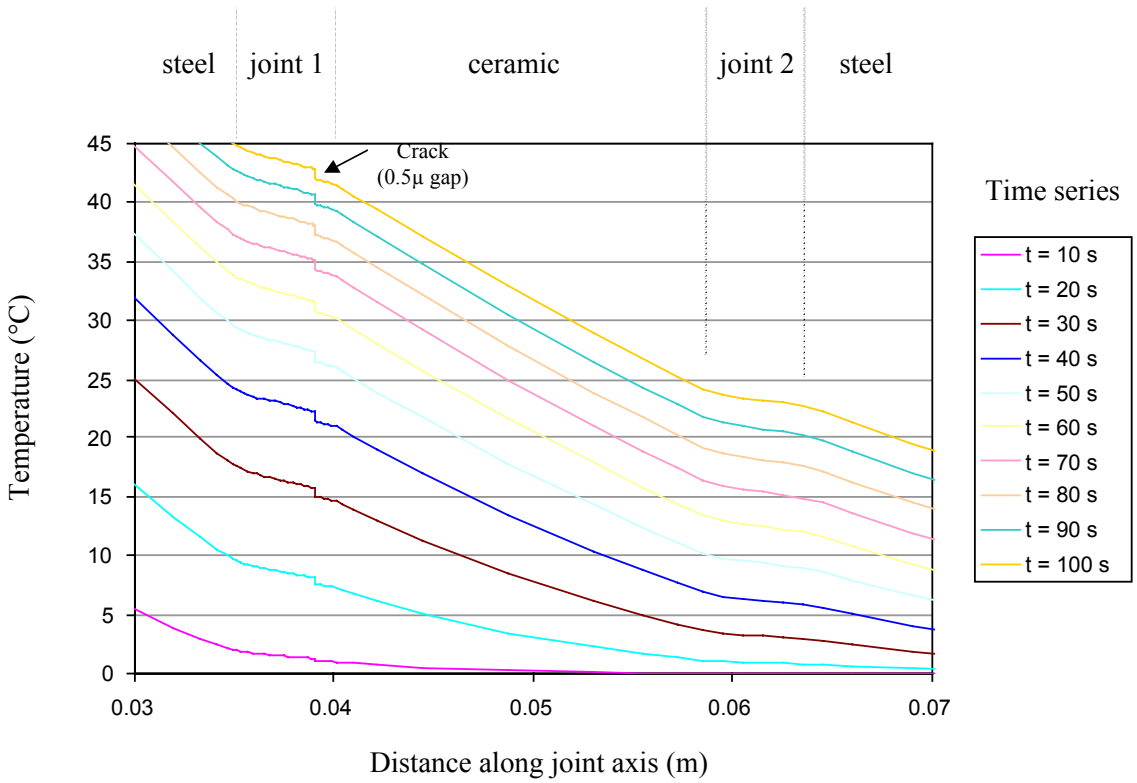


Figure 5. Numerically simulated temperature profiles in a steel-ceramic joint sample with a 0.5-µm air gap at one interface.

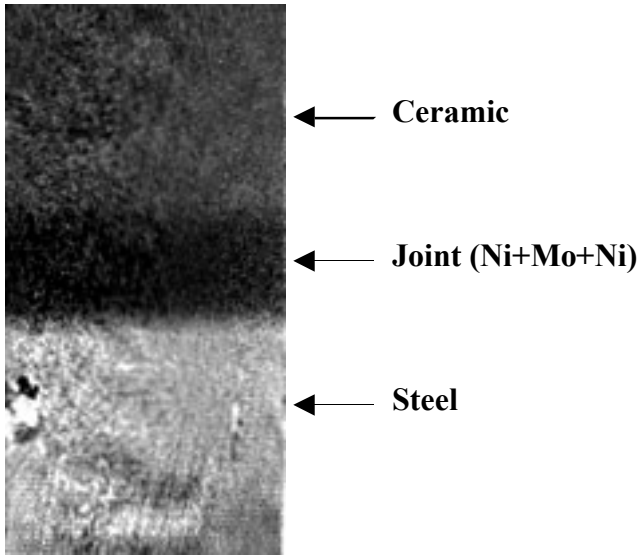


Figure 6. Temperature slope image of a steel-ceramic joint heated at the bottom end.

2.3 X-ray CT

X-ray CT was performed for the small joint sample #6. Figure 7 shows a 2D X-ray image of the scanned region at spatial resolution of 53 µm. The Mo and brazing layers have lower X-ray transmission than the Ni and steel layers. The cross-sectional CT slice images are shown in Fig. 8. These images indicate considerable beam-hardening effects (edge is brighter), but do not reveal any defects at this spatial resolution. Higher spatial resolution is required to examine the details of the interfaces (spatial resolution limit for this X-ray CT system is 20 µm).

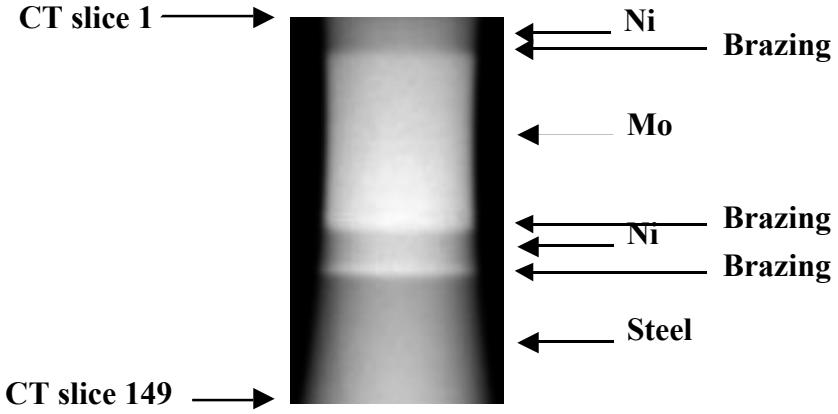


Figure 7. 2D X-ray digital image of the joint section of small joint sample #6. Spatial resolution is 53 µm/pixel.

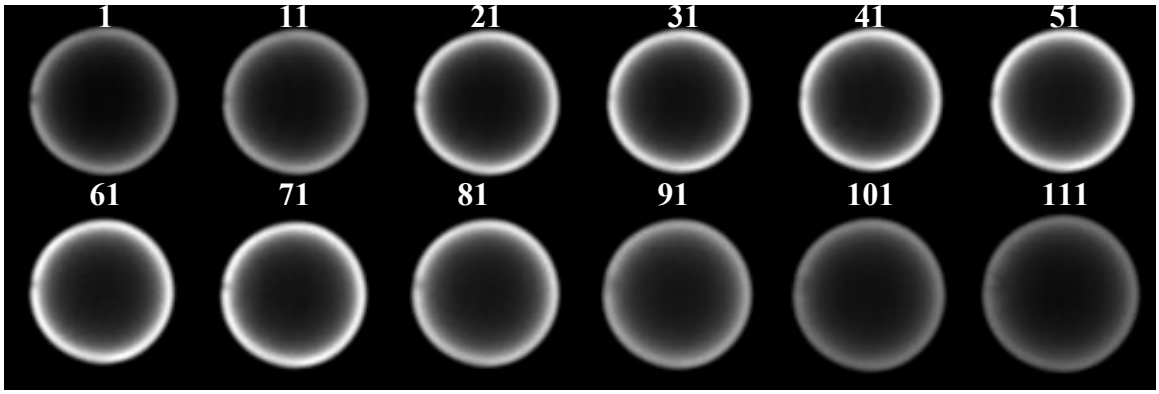


Figure 8. X-ray CT slice images of the joint section of small joint sample #6, corresponding to the slice locations shown in Fig. 7.

Status of Milestones

Current ANL milestones are on or ahead of schedule.

Communications/Visits/Travel
J. G. Sun and W. A. Ellingson plan to visit Caterpillar Technical Center in Mossville, IL, on July 17th to discuss project details.

Problems Encountered
None this period.

Publications
None this period.

Processing and Characterization of Structural and Functional Materials for Heavy Vehicle Applications

J. Sankar, D. Kumar, D. Pai, S. Yarmolenko, and A. D. Kelkar
North Carolina A & T State University

Objective/Scope

The objective of this work is to study properties of novel nanocrystalline metal-ceramic thin film composites.

The required research includes three (3) major tasks:

Task 1. Nanocrystalline metal-ceramic thin film development
Embedding of iron and nickel metal particles in alumina matrix by laser ablation.

Task 2. Magnetic properties of iron and nickel nanocrystalline thin layers
Temperature dependence of the coercivity for Fe-Al_2O_3 metal-ceramic thin films with different Fe particle sizes.

Task 3. Mechanical properties of metal-ceramic thin films
Effect of nanocrystalline metal particles on hardness and Young's modulus of alumina matrix.

Technical Highlights

This investigation focuses on the work on nanocrystalline metal-ceramic thin film composites, which are found to have novel and improved mechanical, electrical, optical, and magnetic properties. The size of nanocrystals was controlled by interfacial energy, substrate temperature, and number of monolayers deposited during pulsed laser deposition.

Inert gas condensation, sputtering, mechanical attrition, aerosol, ball milling, etc., are some of the common methods adopted to synthesize ultrafine magnetic particles. While most of these methods have met with considerable success, producing heterogeneous magnetic materials in a controlled compositional, structural, and reproducible manner is still not satisfactory. One of the most common problems encountered in these methods is the concurrent coarsening of grains in close contact of each other during the order annealing process. The annealing process is usually required to overcome the energy barrier for diffusion and superlattice ordering. The grain coarsening and interaction among the particles will have an adverse effect on the performance of the recording media. It has been reported that physical gaps of 2-5 nm appear to be sufficient to decouple the magnetic grains. It is in this context that we have developed a laser-assisted method to produce nanocrystalline materials, physically well separated from each other, in an amorphous insulating matrix. It is advantageous to have magnetic nanoparticles separated by physical gaps, which can prevent grain coarsening during mutlilayer thin film depositions and eliminate (or greatly reduce) interaction between the particles. The method is generic in nature and can be applied to the synthesis of magnetic, optical,

mechanical, electroluminescent, etc., fine particles. In this paper, however, we will focus solely on the fabrication and properties of magnetic nanocrystalline iron particles in an amorphous alumina matrix.

Nanocrystalline iron and nickel particles were embedded in an alumina matrix by laser ablation. A multitarget pulsed laser deposition system was used where iron and alumina targets are alternately ablated. The depositions were carried out on silicon substrates in a high vacuum environment (~5 X 10^{-7} Torr). The substrate temperature was approximately 500 ^{O}C. The energy density and repetition rate of the laser beam used were 2J/cm^2 and 10 Hz. The size distribution of Fe particles and the crystalline quality of both the matrix and magnetic particles were investigated by cross-sectional high-resolution transmission electron microscopy (HRTEM). The magnetic properties of Fe-Al_2O_3 systems were measured using superconducting quantum interference device (SQUID) magnetometer. The size of Fe nanocrystals was determined by the amount of laser-deposited iron. The zero field cooled (ZFC) magnetization was achieved by applying a small field ranging from 50 to 500 Oe to the sample at 10 K and then warming the sample in the constant field with the magnetization being measured as a function of temperature. The field cooled (FC) magnetization was measured by cooling the sample to 10 K in a (50-500) Oe field and taking the data while heating the samples up to 300 K. The coercivity of each sample was measured at different temperatures by recording magnetization versus field loops at different temperatures. The mechanical properties of thin film composites were measured using a nano indentation system.

Figure 1 shows the variation of the coercivity with temperature for Fe-Al_2O_3 samples having different Fe particle sizes. It is apparent from this figure that the coercivity is strongly dependent on temperature. This is because in order for a particle to reverse its spin, it should have enough thermal energy to surmount the energy barrier $\Delta E = KV$ for the reversal. The energy barrier for the reversal is the difference beween the maximum and minimum values of the total energy *(E)*.

Shown in Figure 2 is the variation of hardness of Ni-Al_2O_3 thin-film composites with four different particle sizes. The Ni particles in these samples were measured to be 3, 4, 6, and 7 nm, respectively, from TEM measurements. The duration of deposition of Ni in these samples with increasing sample code numbers was 20, 40, 60, and 80 s, respectively. For the sake of comparison, we have shown in the figure the hardness data of pure alumina film deposited under identical conditions. The peak value of hardness is taken as the correct value of hardness of each sample. It is clear from the figure that the hardness of alumina is significantly increased (33 %) from 20 GPa to 27 GPa with the increase in particles size of the Ni nanodots from 0 nm to 6 nm in the matrix. Once the Ni particle size exceeds 6 nm, the hardness of the composite decreases. This suggests that there may be an optimum in particle size of dispersed metal to give the highest values in hardness.

The improvement in the values of hardness of Al_2O_3 by embedding Ni nanoparticles may be attributed to the inhibition of grain boundary sliding of Al_2O_3 because of the fine particulates dispersion. The presence of an optimum metal particle size to realize highest hardness values is believed to be caused by the agglomeration of metal dispersion. Once

the metal particle size crosses a certain value, the separation between the adjacent particles reduces to a value where the two particles tend to coalesce, resulting in the disappearance of fine particle characteristics.

Status of Milestones
On Schedule

Communications/Visitors/Travel
None

Problems Encountered
None

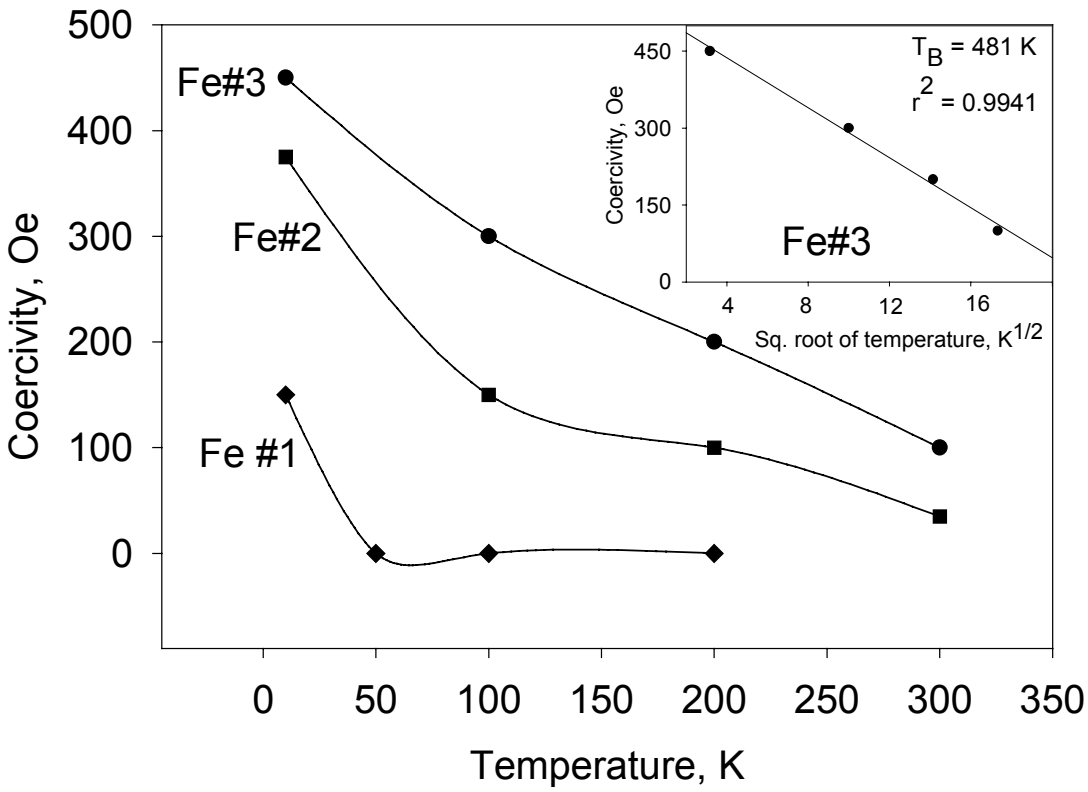


Figure 1. Variation of coercivity with temperature for Fe-Al_2O_3 samples having different Fe particle sizes. The inset shows that the iron particles follow closely $H_c = H_{c,0}\,[1-T/T_B)^{1/2}]$ relationship.

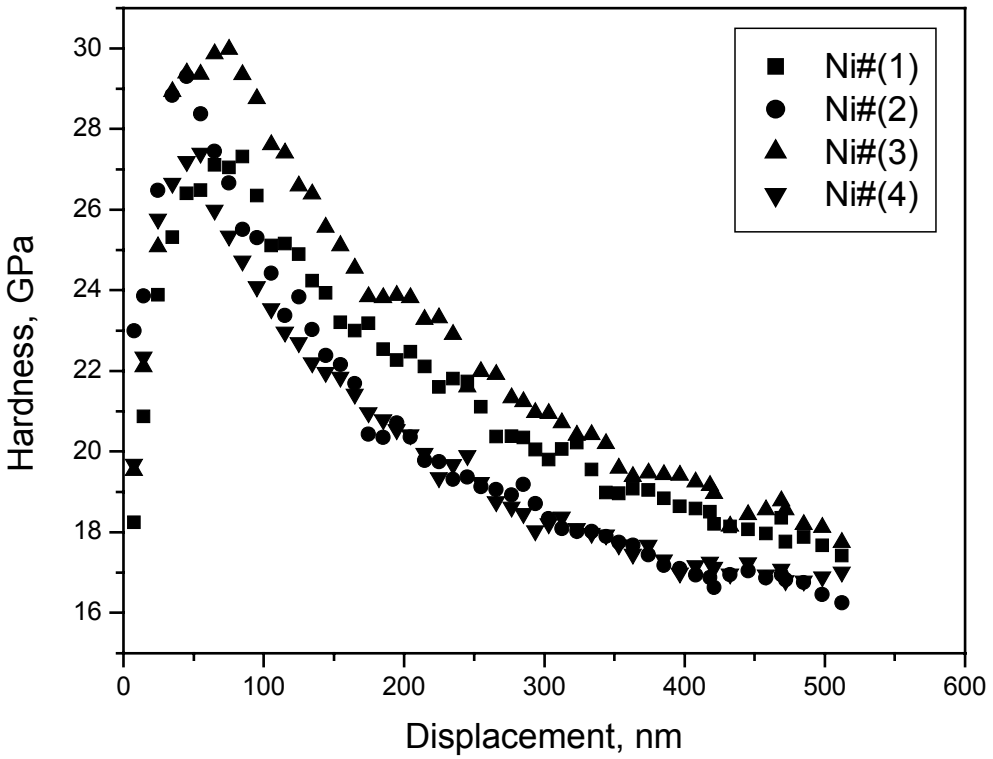


Figure 2. Variation of hardness of Ni-Al_2O_3 thin film composites with four different Ni particle sizes.

Life Prediction of Diesel Engine Components

H. T. Lin, T. P. Kirkland, M. K. Ferber
Oak Ridge National Laboratory
and
M. J. Andrews
Caterpillar, Inc.

Objective/Scope

The valid prediction of mechanical reliability and service life is a prerequisite for the successful implementation of structural ceramics as internal combustion engine components. There are three primary goals of this research project which contribute toward that implementation: the generation of mechanical engineering data from ambient to high temperatures of candidate structural ceramics; the microstructural characterization of failure phenomena in these ceramics and components fabricated from them; and the application and verification of probabilistic life prediction methods using diesel engine components as test cases. For all three stages, results are provided to both the material suppliers and component end-users.

The systematic study of candidate structural ceramics (primarily silicon nitride) for internal combustion engine components is undertaken as a function of temperature (< 900°C), environment, time, and machining conditions. Properties such as strength and fatigue will be characterized via flexure and rotary bend testing.

The second goal of the program is to characterize the evolution and role of damage mechanisms, and changes in microstructure linked to the ceramic's mechanical performance, at representative engine component service conditions. These will be examined using several analytical techniques including optical and scanning electron microscopy. Specifically, several microstructural aspects of failure will be characterized:

(1) strength-limiting flaw-type identification;
(2) edge, surface, and volume effects on strength and fatigue size-scaling;
(3) changes in failure mechanism as a function of temperature;
(4) the nature of slow crack growth; and
(5) what role residual stresses may have in these processes.

Lastly, numerical probabilistic models (*i.e.*, life prediction codes) will be used in conjunction with the generated strength and fatigue data to predict the failure probability and reliability of complex-shaped components subjected to mechanical loading, such as a silicon nitride diesel engine valve. The predicted results will then be compared to actual component performance measured experimentally or from field service data. As a consequence of these efforts, the data generated in this program will not only provide a critically needed base for component utilization in internal combustion engines, but will also facilitate the maturation of candidate ceramic materials and a design algorithm for ceramic components subjected to mechanical loading in general.

Technical Highlights

The dynamic fatigue test in four point bending on NGK SN84 silicon nitride at temperatures of 850 and 1000°C at stressing rates of 30 and 0.003 MPa/s was added to the test matrix reported previously, and completed during this reporting period. The objective of the addition of NGK SN84 silicon nitride into the exiting test matrix is to explore more silicon nitride resources for applications of diesel engine components. Note that the NGK SN84 silicon nitride was developed for intermediate temperature applications by NGK Insulators, Ltd., Japan. Two SN84 silicon nitride billets were acquired from Dr. Aihara, NGK-Locke, Inc., Sunnyvale, CA. The SN84 silicon nitride bend bars were longitudinally machined per ASTM C116 standard. Dynamic fatigue tests showed that the SN84 silicon nitride exhibited a comparable characteristic strength to those generated for both Kyocera SN235 and SN235P silicon nitride under the same test conditions employed, as shown in Table 1. Results also showed that the characteristic strength of NGK SN84 silicon nitride tested at 1000°C at 0.003 MPa/s exhibited little strength degradation with respect to those generated at 850 and 1000°C at 30 MPa/s in air, as shown in Table 1 and Figs. 1 and 2. In addition, NGK SN84 silicon nitride exhibited a high fatigue exponent at temperatures of 850 and 1000°C in air, indicative of little susceptibility to slow crack growth process, as shown in Table 2 and Fig. 2. Note that both Kyocera SN235 and SN235P silicon nitride materials revealed a substantial loss in characteristic strength (44-46%) when tested at 1000°C at 0.003 MPa/s in air. The observed strength degradation of Kyocera SN235 and SN235P silicon nitride materials at 1000°C at 0.003 MPa/s was attributed to the onset of slow crack growth and/or creep processes, consistent with the low fatigue exponent obtained, as shown in Table 2. Tests at room temperature and 1200°C for NGK SN84 silicon nitride will be carried out to complete the test matrix and database generation during the next reporting period. Also, the SN84 silicon nitride bend bars will be transversely machined per ASTM C116 standard to understand the effect of grinding orientation.

Studies of stress-rupture behavior of Kyocera SN235 silicon nitride at 1000°C in air were also completed during this reporting period to understand the effect of stress level on the lifetime. Results showed that the SN235 silicon nitride exhibited a stress-dependent lifetime at stress levels above a threshold stress of ~ 350 MPa (defined as an apparent fatigue limit). Note that the static fatigue exponent (N ~15.5) obtained from the stress rupture test was similar to the exponent (N ~ 14) obtained from the dynamic fatigue test at 1000°C, which suggested that the lifetime of SN235 at 1000°C in air could be mostly governed by the slow-crack-growth processes. Note that bend bars, which did not fracture after lifetime < 1000 h, exhibited a permanent curvature, also indicative of the onset of creep deformation processes.

Fifteen SN84 MOR bars were sent to Dr. Andrews at Caterpillar for 1000 h exposure to an oil ash environment at 850°C in air, similar to the exposure tests already carried out for Kyocera SN235 and Honeywell GS44 silicon nitride materials. Dynamic fatigue tests at 850°C at 0.003 MPa/s will then be conducted to evaluate the effect of 1000 h oil ash exposure on the mechanical properties of SN84 silicon nitride.

Status of Milestones
All milestones are on schedule.

Communications / Visitors / Travel

Dynamic fatigue results of Kyocera SN237 were updated and communicated with M. J. Andrews at Caterpillar.

Dynamic fatigue results of SN84 at 850 and 1000°C were communicated with M. J. Andrews at Caterpillar.

Fifteen SN84 MOR bars were sent to Mark Andrews at Caterpillar for 1000 h oil immersion test at 850°C in air.

Discussions of the initial bench test on Norton NT551 exhaust valves were held with M. J. Andrews at Caterpillar.

Problems Encountered
None.

Publications

"Strength and Dynamic Fatigue of Silicon Nitride at Intermediate Temperatures," A. A. Wereszczak, H. -T. Lin, T. P. Kirkland, M. J. Andrews, and S. K. Lee, in preparation.

References

[1] A. A. Wereszczak, M. J. Andrews, M. K. Ferber, and T. P. Kirkland, "Life Prediction Verification," *Heavy Vehicle Propulsion System Materials Program Semiannual Technical Progress Report to DOE Office of Transportation Technologies*, Apr. 1997 - Sep. 1997.
[2] A. A. Wereszczak, and T. P. Kirkland, M. J. Andrews, M. K. Ferber, and R. Tandonl, "Life Prediction of Ceramic Diesel Engine Components," *Heavy Vehicle Propulsion System Materials Program Semiannual Technical Progress Report to DOE Office of Transportation Technologies*, Oct. 1998 - Mar. 1999.
[3] C. R. Brinkman, T. P. Kirkland, H. - T. Lin, R. A. Ott, S. K. Lee, and J. Thiele, "Life Prediction of Ceramic Diesel Engine Components," *Heavy Vehicle Propulsion System Materials Program Bimonthly Technical Progress Report to DOE Office of Transportation Technologies*, Apr. 2000 - May 2000.
[4] J. C. Cuccio, P. Brehm, H. T. Fang, J. Hartman, W. Meade, M. N. Menon, A. Peralta, J. Z. Song, T. Strangman, J. Wade, J. Wimmer, and D. C. Wu, *Life Prediction Methodology for Ceramic Components of Advanced Heat Engines, Phase 1*, DOE Report ORNL/Sub/89-SC674/1/V2, 1995.

Table 1. Summary of uncensored Weibull strength distributions for NGK SN84 (longitudinally machined) and Kyocera SN235 and SN235P (transversely machined) silicon nitride specimens per ASTM C1161.

Material	# of Spmns. Tested	Stressing Rate (MPa/s)	Temp. (°C)	Uncens. Weibull Modulus	± 95% Uncens. Weibull Modulus	Uncens. Chrctstic Strength (MPa)	± 95% Uncens. Chrctstic Strength (MPa)
SN84	18	30	850	25.14	15.58, 37.35	818	799, 836
SN84	15	0.003	850	10.27	6.51, 15.06	761	718, 803
SN84	15	30	1000	20.59	13.49, 29.45	777	757, 796
SN84	15	0.003	1000	14.71	9.63, 20.85	863	829, 896
SN235	15	30	850	26.7	18.0, 36.7	777	760, 793
SN235P	15	30	850	19.3	12.5, 27.6	631	612, 649
SN235	14	0.003	850	18.5	11.8, 26.8	744	720, 767
SN235P	15	0.003	850	18.2	11.5, 26.8	594	575, 612
SN235	15	30	1000	12	8.5, 17.2	711	686, 740
SN235P	11	30	1000	11	7.3, 17.1	560	536, 590
SN235	15	0.003	1000	20.6	14.5, 29.5	395	387, 405
SN235P	10	0.003	1000	22.8	14.7, 36.1	301	295, 309

Table 2. Summary of uncensored dynamic fatigue exponents of NGK SN44 and Kyocera silicon nitride materials as a function of test temperature

Material	Machining Orientation	Temperature	Fatigue Exponent
SN84	Longitudinal	850	88
SN84	Longitudinal	1000	-93
SN235	Transverse	850	164
SN235P	Transverse	850	140
SN235	Transverse	1000	14
SN235P	Transverse	1000	13

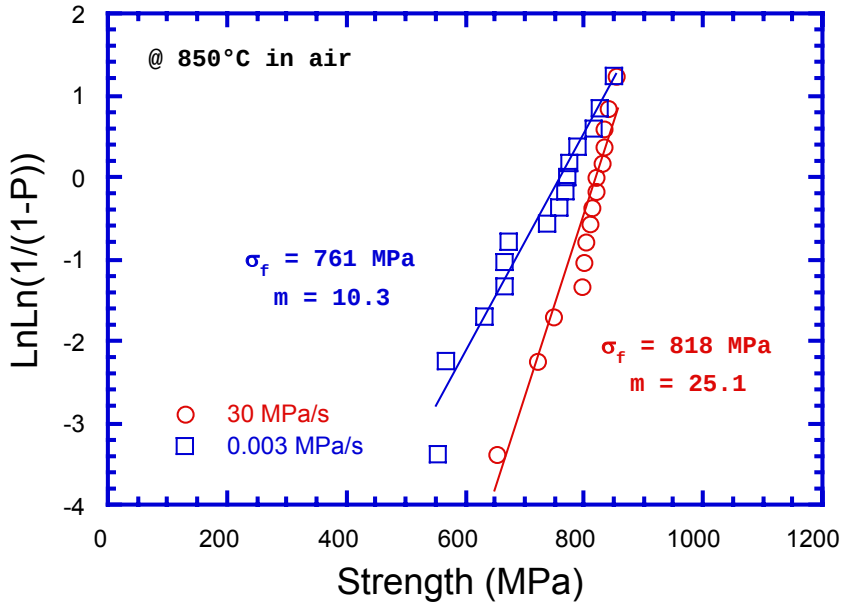


Figure 1. Strength results of NGK SN84 tested at 850°C at stressing rates of 30 and 0.003 MPa/s in air.

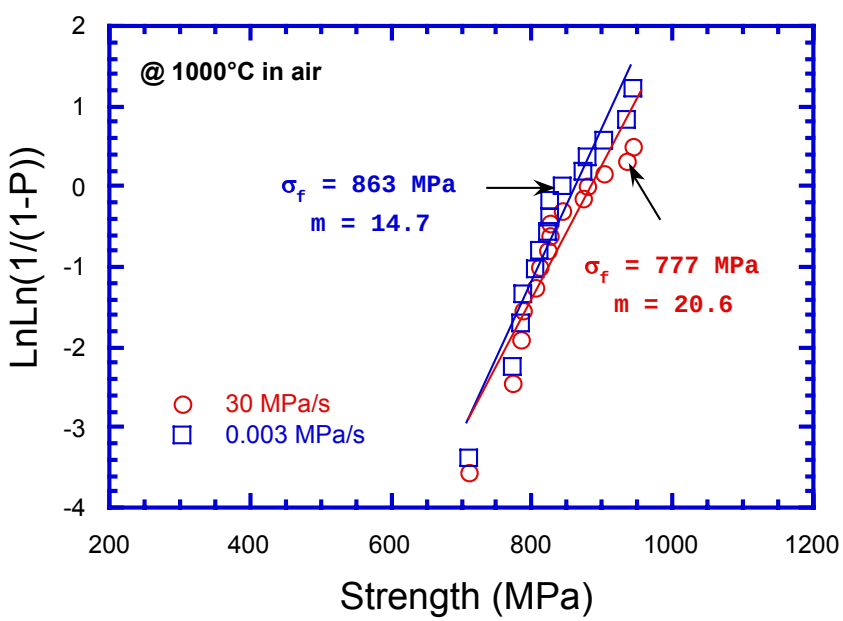


Figure 2. Strength results of NGK SN84 tested at 1000°C at stressing rates of 30 and 0.003 MPa/s in air.

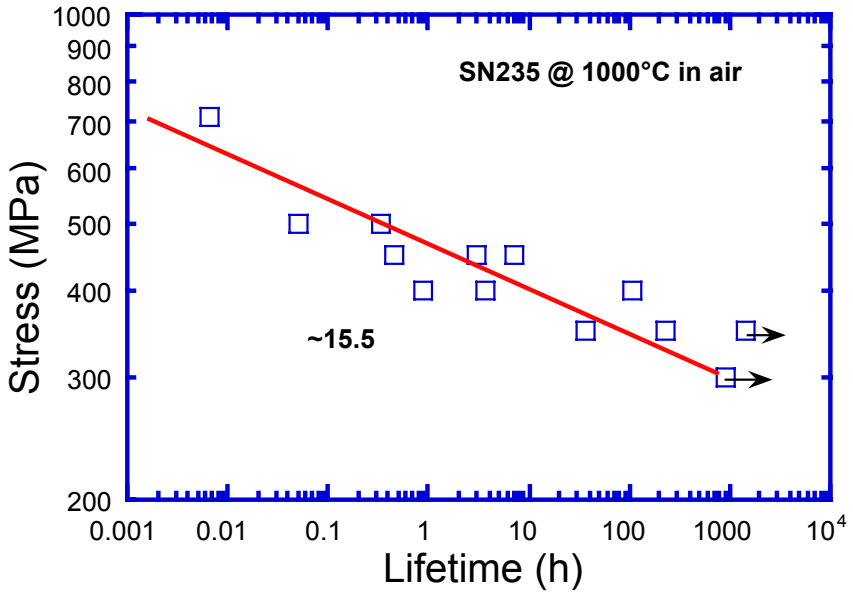


Figure 3. Stress versus lifetime curve of Kyocera SN235 silicon nitride at 1000°C in air.

Durability of Diesel Engine Component Materials

Peter J. Blau and Ronald D. Ott
Oak Ridge National Laboratory

Objective/Scope

The objective of this effort is to enable the development of more durable, low-friction moving parts in diesel engines for heavy vehicle propulsion systems by conducting friction, lubrication, and wear analyses of advanced materials, surface treatments, and coatings. The scope of materials and coatings is broad and includes any metallic alloy, intermetallic compound, ceramic, or composite material which is likely to be best-suited for the given application. Parts of current interest include scuffing-critical components, like fuel injector plungers and EGR waste gate components. Hot scuffing is a primary surface damage mode of interest. Bench-scale simulations of the rubbing conditions in diesel engine environments are used to study the accumulation of surface damage, and to correlate this behavior with the properties and compositions of the surface species. The effects of mechanical, thermal, and chemical factors on scuffing and reciprocating sliding wear are being determined. Results will be used to refine material selection strategies and suggest materials for durability-critical engine components.

Technical Highlights

High-Temperature Scuffing Results. As indicated in the previous two quarterly reports, construction of the high-temperature, oscillating scuffing test system was completed. The basic geometric configuration consists of a horizontal, cylindrical specimen pivoting on a square, flat specimen with a central hole to avoid the center singularity. The shape of the oscillating contact is suggestive of a bowtie, and thus, we decided to call this test method the "bowtie scuffing test." A poster describing this test arrangement was presented at the International Conference on Wear of Materials in Vancouver, Canada, in April.

The results of unlubricated and lubricated baseline tests were given in the previous quarterly report. The current report describes the torque and wear damage results obtained for a series of selected material couples tested at 600°C. Tests were conducted for 3600 cycles of oscillation under a normal force of 10 N, and oscillating rate of 1 Hz. The cylindrical upper specimen diameter was 6.35 mm.

The material pairs chosen for this work were selected based on a review of the literature and discussions with material suppliers and colleagues. Material pairing included materials developed for galling resistance, alloys developed for high-temperature wear resistance, an intermetallic alloy of Ni –Al, and two ceramics. The selected material pairs are listed in Table 1, along with their measured Vickers microindentation hardness numbers. Compositions of these materials will be given in a subsequent report.

Several criteria were used to analyze the results:

a) Average frictional torque during a 30 second interval at the beginning and end of each run
b) Standard deviation of the frictional torque at the beginning and end of each run
c) The wear of the upper (cylindrical pin) specimen
d) The post-tested roughness of the flat specimen tile

The wear volume of the pin was estimated based on the length of the contact along the cylindrical surface, and the average wear scar width.

Table 1.
Material Pairs Used for Scuffing Tests at 600° C

Pair No.	Upper Specimen (cylinder) Material	HV* ave (std dev) (GPa)	Lower Specimen (flat tile) Material	HV* ave (std dev) (GPa)
1	Type 303 stainless steel	4.63 (0.07)	Type 304 stainless steel	3.89 (0.12)
2	Stellite 6B	5.47 (0.37)	Type 304 stainless steel	3.89 (0.12)
3	Stellite 6B	5.47 (0.37)	Ni_3Al alloy IC-221M	4.15 (0.18)
4	PS Zirconia ceramic	12.88 (1.51)	Type 304 stainless steel	3.89 (0.12)
5	PS Zirconia ceramic	12.88 (1.51)	Type GS-44 Si_3N_4 ceramic	22.58 (1.24)
6	PS Zirconia ceramic	12.88 (1.51)	Ni_3Al alloy IC-221M	4.15 (0.18)
7	Gall Tough™ stainless steel	3.23 (0.13)	Gall Tough ™ stainless steel	4.70 (0.26)
8	Tribonic 20 specialty steel	2.74 (0.08)	Ni_3Al alloy IC-221M	4.15 (0.18)

*Vickers microindentation hardness of the test surface prior to testing. Load = 200 gf. Average and standard deviations for at least 4 indentations. Data were also obtained on the surface after exposure to 600° C, but are not reported here.

The complete set of the test data is too large to include here, but the non-weighted ("ordinal") ranking of material pair performance, based on each criterion listed above, are in given in Table 2. Two tests were performed on each pairing. Where the numerical values of a test parameter were close enough to be statistically indistinguishable, the material pairs were given the same ranking. Therefore, not every column has a full set of values from 1 to 8. The summation of rank orders across each row in Table 2 is indicated, and serves as a means to rank the composite scores of each pair based on both friction and wear metrics. The composite rankings fall into two groups: those with values above 20 and those with values below 14. Interestingly, zirconia on silicon nitride (pair 5), had a relatively poor ranking even in light of the fact that the pair had the lowest wear damage. This was primarily because of the pair's high and erratic frictional torque. The top three combinations in overall ranking were zirconia on IC-221M (pair 6), self-mated GallTough (pair 7), and Stellite 6B on IC-221M (pair 3). Tribonic 20 on IC-221M (pair 8) was only slightly worse than these.

Table 2.
Non-Weighted Ranking of Performance Metrics for Material Pairs Tested at 600° C
(Lowest value ranked #1, equal values given the same ranking)

Pair	Ave. Initial Torque	Std. Dev. Initial Torque	Ave. Final Torque	Std. Dev. Final Torque	Pin Wear Volume	Tile Roughness (Ra)	Sum (Order) of Rankings
1	4	4	3	2	4	4	**21 (6)**
2	6	5	4	3	3	5	**26 (8)**
3	3	2	2	1	2	2	**12 (3)**
4	5	4	5	4	3	4	**25 (7)**
5	4	3	6	5	1	1	**20 (5)**
6	2	1	2	1	1	2	**9 (1)**
7	1	2	1	1	3	3	**11 (2)**
8	2	2	3	3	2	2	**14 (4)**

In general, the pairs containing tiles of the intermetallic Ni_3Al alloy IC-221M performed very well in our scuffing tests, as did most pairs that involved zirconia as the cylindrical pin. When the zirconia was mated with IC-221M (pair 6), the best results were obtained.

Self-mated ceramics were excellent in terms of wear, but not friction. The probable reason why friction was not as low for the ceramic pair was that both specimens wore so little that a cushioning layer of wear particles did not form and could not act as a granular lubricating layer. Scanning electron microscopy revealed how granular patches comprised of wear particles formed on most of the metallic specimens. (See Figure 1.) A publication describing these results in detail is in preparation.

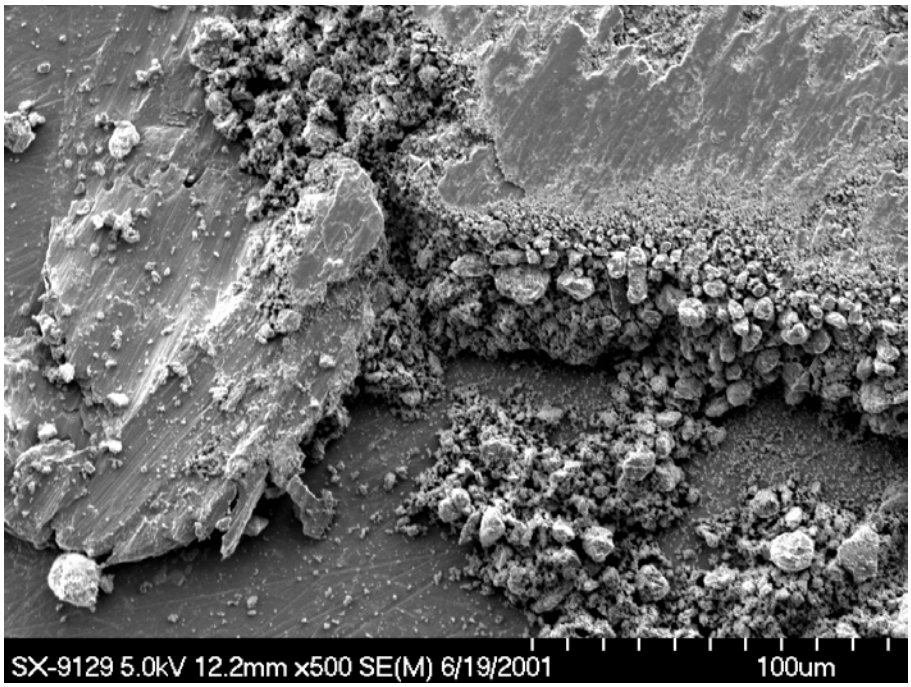


Figure 1. Scanning electron micrograph of the edge of a patch of compressed wear debris that formed on pair 4 (Zirconia on Type 304 stainless steel).

Future Plans

1. Complete metallurgical studies of scuff-tested specimens and complete preparation of a journal article on these results.

2. Continue work on the redesign of a current ORNL wear testing system to evaluate the durability of fuel injector plungers.

Travel

P. Blau and R. Ott attended the 2001 International Conference on Wear of Materials, Vancouver, Canada, April 22-26, 2001. Both travelers made presentations. Blau presented the results of friction and wear studies on amorphous metals (reported to OHVT last FY). Ott and Blau also

presented a poster titled: "Development of a High-Temperature, Bowtie Scuffing Test for Diesel Engine Applications."

Status of Milestones

The following milestones were developed for FY 2001.

1) Complete baseline scuffing tests at room and elevated temperature. Submit report. (February 28, 2001 – report completed and submitted for clearance for publication and distribution as an ORNL Technical Report.)

2) Complete elevated temperature scuffing tests of leading candidate materials. Submit report. (March 31, 2001 – extension to June 30 was requested due to the late receipt of machined test specimens of the specimen materials. Testing completed, report in preparation.)

3) Complete design and construction of a computer-controlled fuel injector materials testing system capable of studying the effects of varying injector motions and loading cycles on material response. (September 30, 2001)

Presentation

(1) P. J. Blau and R. D. Ott, "Development of a High-Temperature, Bowtie Scuffing Test for Diesel Engine Applications," poster presented at the International Conference on Wear of Materials, Vancouver, BC, Canada, April 23-26, 2001.

Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications

M. K. Ferber
Oak Ridge National Laboratory

Objective/Scope

The International Energy Agency (IEA) was formed via an international treaty of oil consuming countries in response to the energy crisis of the 1970s. A major objective of the IEA is to promote secure energy supplies on reasonable and equitable terms. The governing board of the IEA, which is composed of energy officials from each member country, regularly reviews the world energy situation. To facilitate this activity, each member country provides energy experts who serve temporary staff assignments at IEA headquarters. These staff or secretariat support the governing board by collecting and analyzing energy data, making projections in energy usage, and undertaking studies on specialized energy topics. The governing board is also assisted by several standing groups, one being the committee on energy research and technology (CERT), which encourages international cooperation on energy technology. Implementing agreements (IAs) are the legal instruments used to define the general scope of the collaborative projects. There are currently 40 active implementing agreements covering research topics such as advanced fuel cells, coal combustion science, district heating and cooling, enhanced oil recovery, fluidised bed conversion, fusion materials, solar heating and cooling, pulp and paper, hydropower, heat pumping technologies, hybrid and electric vehicles, high temperature super conductivity, wind turbines, and high temperature materials. A complete listing can be found at the IEA website, www.iea.org.

This progress report summarizes recent activities in the implementing agreement entitled, "Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications." This implementing agreement currently consists of one active annex entitled, "Annex II: Co-Operative Program on Ceramics for Advanced Engines and Other Conservation Applications." The motivation for this IA is the development of new and improved ceramic materials, brittle material design methods, and life prediction methodology. The objective of Annex II is coordinated R&D on advanced ceramics leading to standardized methods for testing and characterization.

The Executive Committee for the IA on Advanced Materials is also exploring the possibility of adding a new effort (Annex III) focusing on the characterization of materials for diesel engines. Possible topics include characterization of contact damage (fatigue) and novel methods for the assessment of the mechanical reliability of coatings.

Technical Highlights

General

Planning for the next executive committee meeting was initiated this reporting period. The United States will host this meeting to be held in conjunction with the PAC RIM IV meeting which will be held 4-8 November 2001 in Wailea, Maui, Hawaii, at the Outrigger Wailea Resort.

The major objectives of this meeting are to discuss progress in Annex II (Subtasks 11 and 12) and to propose new activities.

Annex II

Annex II currently consists of two active efforts, Subtasks 11 and 12. In Subtask 11, techniques for the measurement of thermal and mechanical fatigue of silicon nitride ceramics are being examined. National efforts in Japan and the United States focus on the development of procedures for evaluating the mechanical fatigue behavior of silicon nitride ceramics using either uniaxial flexure or biaxial test specimens. The national effort in Germany consists of the development of thermal fatigue procedures using the laser thermal shock equipment evaluated in Subtask 9. The national effort in Sweden will focus on the evaluation of the fracture surfaces of specimens tested in Japan, the United States, and Germany.

Subtask 11 will also include an international effort in which the thermal fatigue behavior of a single silicon nitride will be compared with the mechanical fatigue data generated at a temperature which is the same as that at the fracture point in the thermal fatigue test. Germany will be responsible for the thermal fatigue testing while both Japan and the United States will conduct mechanical fatigue testing. Sweden will perform fractographic analysis of the specimens.

The effort in Japan was initiated this reporting period. Samples for the testing in Germany were machined at ORNL and sent to Rolf Wäsche who will be conducting the thermal fatigue tests.

Annex III

To promote pre-competitive standard testing and characterization methods for diesel engine materials, DOE is proposing to start an international collaborative effort under the umbrella of the International Energy Agency. This effort, which would be in the form of a new annex under an existing Implementing Agreement (Advanced Materials For Transportation Applications), would focus on development and verification of advanced characterization techniques for diesel engine materials.

To better define the needs in this area, a subcontract was initiated with Gateway Materials Technology. During this reposting period a contact list for the diesel engine and ceramic coating community (engine manufacturers, coating suppliers, and testing, government, and academic research agencies) was created. A survey requesting information on characterization needs with respect to ceramic coatings was sent to these contacts via e-mail. The results of this survey will be discussed in the next quarterly report.

Status of Milestones

All milestones are on track.

Communications/Visits/Travel

None

Publications
None

References
None

Standards For Reliability Testing Of Heavy Vehicle Propulsion Materials

Said Jahanmir, James F. Kelly, and William Luecke
Ceramics Division
National Institute of Standards and Technology

Objective/Scope

The objective of this project is to develop international standard test methods for assessing the reliability of ceramic components used in diesel engines and other heavy vehicle propulsion systems. Advanced ceramics such as silicon nitride offer a unique combination of properties that include light weight, excellent high-temperature strength, and resistance to wear and corrosion. These properties make them particularly attractive for diesel engines, where their use as key engine components will allow the higher operating temperatures that lead to higher thermal efficiencies and environmentally cleaner propulsion systems. Reliability and cost-effectiveness are critical issues in implementing ceramics in the valve train of diesel engines. Ceramic valve train components are subjected to demanding conditions that include high contact loading, elevated temperatures, and corrosive environments. To ensure a reliable service life, standard test methods are needed to evaluate the performance of potential ceramics in highly loaded rolling and sliding contacts. This project will develop test methods for evaluating the contact damage behavior of ceramics under rolling and sliding conditions that simulate the cam roller followers, valves and valve seats. In support of this goal we will pursue four research thrusts: 1) fundamental issues in the relation between ceramic microstructure and performance reliability, 2) basic mechanisms that lead to formation and propagation of contact damage and the effect on reliability of residual stresses developed during machining and by contact, 3) effect of machining damage on contact reliability as well as the interactions between machining and contact damage that may lead to premature failure, 4) development of international standards for assessing contact damage.

Technical Highlights

Standards for Rolling Contact Fatigue

On May 3, 2001, NIST hosted a workshop to solicit input from and encourage collaboration with academic and industrial organizations for a new project on contact damage and reliability assessment of ceramics for diesel engine applications. Seventeen participants from Caterpillar, Cummins, Ceradyne, DOE, ORNL, NIST, and Colorado State University attended. Representatives from Kyocera, Ford, Detroit Diesel, and Chand Associates were also invited, but could not attend. The participants from industrial and academic organizations presented their views on the needed measurements and standards related to the focus of the workshop. Brief presentations from NIST staff on NIST capabilities followed these presentations. After extensive discussions, the participants developed a three-part, tentative work plan devoted to writing a practice guide for evaluating contact damage, developing standards and standard test methods for evaluating contact damage and reliability, and initiating work in areas needing fundamental research. The group agreed to meet again in six months to review progress and articulate the work plan.

Summary of Workshop Presentations:
Mark Andrews (Caterpillar) presented an overview of contact-damage related research at the Caterpillar Technical Center that focused mainly on sliding wear issues evaluated using a reciprocating ball-on-flat, ball-on-inclined-flat, and commercial block-on-ring test machines. He listed a number of important research areas in which collaboration could occur: 1) development of generalized test methods for selecting materials prior to rig simulation testing, 2) extension of current room-temperature tests to elevated temperatures, 3) assessment of the relation between microstructure and machining conditions as it impacts wear under sliding and rolling, 4) validation of the use of accelerated tests, 5) research on the effect of thermal gradients on wear and failure, and 6) effect of using different material pairs in wear testing. He suggested that in a comprehensive program these elements must be integrated into life prediction methods.

Biljana Mikijelj (Ceradyne) made a presentation directed specifically at issues central to rolling contact fatigue testing, including specimen preparation, test conditions, and failure definitions. She suggested several critical areas for collaboration: 1) developing a recommended practice for machining/finishing of the test rods, 2) developing methods for failure diagnosis, including identification of failure origins, 3) evaluating the potential causes of scatter in the data and possible elimination of early failures from statistical evaluation, 4) understanding the influence of finishing methods on test results, and 5) assessing of the current test conditions used in industry with the goal of increasing the contact stresses to reduce testing time.

Paul Wilbur (Colorado State University) described his work using the NTN RCF tester and echoed the sentiments of Mikijelj on needs in RCF testing. He was also concerned about selecting appropriate test conditions (steel vs Si_3N_4 balls and their surface roughness). He outlined possible research in several areas: 1) fundamental assessment of failure mechanisms of silicon nitride, including identification of failure origins, and 2) comparison of damage initiation and propagation in steel and silicon nitride. He also discussed possible directions that a research plan might take and how they could yield improvements in RCF performance both quickly and cost effectively. In this approach he singled out damage-accumulation analysis and analysis of the vibration spectrum of the rod/test head during the test as fruitful areas to pursue.

Tom Yonushonis (Cummins) reviewed some of the knowledge of materials issues in RCF, stressing that removal of all porosity is the primary way to improve RCF performance. He identified a number of important issues in future RCF research including developing tests at lower, more realistic stresses, testing larger volumes of material than the current RCF test, and including the possibility of sliding in the test. During the discussion he emphasized the need for tests that included edge effects as well as tests and research on zirconia, in addition to silicon nitride.

Said Jahanmir (NIST) gave an overview of the NIST Ceramic Machining Consortium as an example of how the Contact Reliability Working Group (CRWG) could interact. He distributed a draft Research Agreement that detailed how the group could function and asked the participants to review it and reply with comments as to its suitability. The CRWG would meet twice per year and be open to all US-based industries, universities, and government laboratories. NIST would be the central body for organization and coordination. To encourage participation, there would be no membership fees, and no proprietary information would be exchanged. All results would

be shared freely among the members and published in the open literature. He also suggested developing collaboration with international organizations such as IEA and VAMAS for pre-standards research that would lead to harmonization of standard test methods for assessment of contact damage in ceramics.

Brian Lawn (NIST) reviewed his recent work on the relation between microstructure of silicon nitride and dental ceramics and contact damage by ball-on-flat (both single and cyclic loading). He is using ball-on-flat static and cyclic loading to introduce damage in these ceramics. In terms of damage modes, fine microstructures develop cone cracks under the ball, and the strength of such materials is degraded precipitously by the damage. In contrast, coarser microstructures develop a quasi-plastic zone under the ball and are flaw-tolerant; their strength degradation is much smaller.

Bill Luecke (NIST) reviewed the types of RCF tests that have been used in the past. Compared to other methods, three-ball-on-rod (NTN machine) tests have the advantage that the specimen is easier and less costly to fabricate, but suffer from the fact that the load drops if the specimen wears. Ball-on-ball tests are appropriate for bearings, but fabricating test balls of candidate materials is not trivial. These tests also suffer from load drop if wear occurs. Cylinder-on-cylinder tests allow variable sliding, but the specimens are expensive. No rolling contact fatigue test standards exist for any of these geometries.

The group finished the afternoon with a wide-ranging discussion of potential research areas that the group might pursue and developed the following tentative work plan, which encompasses three separate areas. The work plan should be viewed as tentative and can be changed to reflect the interests of other members who may join in the future or to accommodate insights and discoveries that arise during the research. Suggestions were made to invite manufacturers and suppliers of test equipment, as well as other heavy equipment manufacturers (including Deere and International) to participate.

1. Development of a Practice Guide

One area that all members of the group agreed to participate in is the development of a practice guide for evaluating contact damage, and rolling contact damage in particular. The definition of a practice guide was deliberately left vague. Participants agreed to send references and data in the near future to NIST, who will evaluate it for inclusion in such a guide. At the next meeting in six months, participants will articulate the structure of this guide.

Specific Action Items

· All Participants: Submission of relevant data, test, and analysis methods for evaluation and possible inclusion in a practice guide. Completion: 3 months.

2. Development of a Rolling Contact Fatigue Test Method/Standard

This is a long-term goal, and should not be pursued before more information is developed on whether such a test can be standardized. Because several participants own NTN three-ball-on-rod test machines, we will focus initially on this machine. Work will include articulation of the test procedure, better definition of what failure in RCF means, and further research into appropriate test conditions. It will certainly incorporate an interlaboratory study (round robin) involving at least six laboratories. Preliminary work could involve analyzing the variability of the M50 steel

certification bars supplied with the NTN RCF tester. Other items that must be addressed are the development of a machining specification for the test bars as well as verifying surface roughness and roundness measurements. Ceradyne can supply material for the bars, but another supplier (possibly Kyocera) should be identified. The group will contact other organizations and participants (e.g., NTN and Falex) and invite them to participate.

Specific Action Items

· Cummins, CSU: Submit M50 qualification data for NTN-RCF machines as a first step toward understanding interlaboratory variability. Completion: 3 months.

· NIST: Identify and invite other potential interlaboratory study participants (NTN, Falex, other users of NTN RCF Tester). Completion: 6 months

· NIST: Explore the possibility of international collaboration through IEA or VAMAS

· NIST: Develop understanding of the impact of the definition of failure in the RCF Test on the results.

· NIST: Identify and contact potential materials suppliers for an interlaboratory study.

3. Fundamental Studies on Contact Damage

The participants also identified a number of fundamental research areas in contact damage on which to focus. Special tests are needed for probing the damage initiation and propagation of contact damage. Participants were also interested in mechanics-based modeling, possibly using OOF (Object-Oriented Finite element analysis, developed at NIST) to study the damage process. Spectral analysis of the vibration signals from the RCF tester to study the initiation of spalls during the test is another research area. Also of interest were projects to link more basic studies of fatigue crack propagation rate to the development of spalls in RCF. Parallel studies on sliding wear and impact damage were also mentioned.

Specific Action Items

· CSU: Develop methods to analyze the vibration signals from the RCF tester to identify spallation failure in silicon nitride.

· NIST and CSU: Model the failure process using a mechanics approach.

· CSU, Ceradyne, NIST: Develop special tests for studying damage initiation and propagation in rolling contact fatigue.

Moisture and Binder Content of Alumina

The national representatives to IEA subtask 12 are currently reviewing the final report. Recently we have conducted thermogravimetric analysis (TGA) of the alumina/binder samples to follow the moisture and binder loss as a function of temperature. The results agree with the laboratory mean values for both moisture and binder loss. An error of up to 30% in moisture measurement can arise if the bottle is sealed at 105°C before weighing, since the trapped air is less dense. An error of up to 0.2% in absolute binder content can arise if the specimen is not weighed promptly after the test, because powder exposed to room air will adsorb moisture.

Status of Milestones

1. Complete final report for the first year effort on Subtask 12 (December 30, 2000) – Completed: Currently being reviewed within NIST and awaiting comments from national representatives.

2. Initiate a new project direction and develop collaboration with diesel engine companies in the U.S. and IEA countries (February 28, 2001) – Completed.

3. Evaluate the feasibility of possible test methods for standardization (September 30, 2001) – On schedule.

Communication/Visits/Travel

Said Jahanmir attended the IEA Subtask 12 Meeting in Berlin on May 31, 2001. Attendees discussed the recent round robin data on binder and moisture content. The final report on this activity is expected soon after incorporation of comments from National Representatives. The Subtask Committee nominated and approved Kevin Ewsuk (Sandia National Laboratory) as the new chair of the subtask. His name will be submitted to the Chair of the Executive Committee (Sid Diamond) for approval. The next activity of the subtask will be focused on green body characterization. Said Jahanmir proposed a new activity related to diesel engine materials that may be incorporated under a new annex. There was a general agreement that a new subtask should be formed under Annex 3 with participants from Germany, Japan, and US. It was also indicated that instead of round robin testing, the new subtask should aim at annual exchange of information. Other non-IEA countries should be invited to participate and share information. NIST hosted a workshop on Contact Damage and Reliability (described above). Table 1 lists the participants.

Publications

None

References

None

Table 1: Participants in the Contact Damage and Reliability Workshop

Name	Company	E-mail
Mark Andrews	Caterpillar	Andrews_Mark_J@cat.com
Lou Balmer-Millar	Caterpillar	Balmer-Millar_Lou@cat.com
Biljana Mikijelj	Ceradyne	bmikijelj@ceradyne.com
Paul Wilbur	Colorado State Univ.	pwilbur@engr.colostate.edu
Carl Musolff	Cummins	Carl.F.Musolff@cummins.com
Tom Yonushonis	Cummins	Thomas.M.Yonushonis@cummins.com
Sid Diamond	DOE HQ	Sid.diamond@ee.doe.gov
Ray Johnson	ORNL	johnsondr@ornl.gov
Steve Gonzcy	Gateway Mat'ls	gatewaymt@aol.com
Sandy Dapkunas	NIST Ceramics Div.	Stanley.Dapkunas@nist.gov
Steve Freiman	NIST Ceramics Div.	Stephen.Freiman@nist.gov
Said Jahanmir	NIST Ceramics Div.	Said.Jahanmir@nist.gov
Lew Ives	NIST Ceramics Div.	Lewis.Ives@nist.gov
Brian Lawn	NIST MSEL	Brian.Lawn@nist.gov
Bill Luecke	NIST Ceramics Div.	William.Luecke@nist.gov
George Quinn	NIST Ceramics Div.	George.Quinn@nist.gov
Shelley Wiederhorn	NIST MSEL	Sheldon.Wiederhorn@nist.gov

Mechanical Property Testing and Standards Development

George Quinn
Ceramics Division
National Institute of Standards and Technology

Objective/Scope

This task is to develop mechanical test method standards in support of the Propulsion Systems Materials Program. Test method development should meet the needs of the DOE engine community but should also consider the general USA structural ceramics community as well as foreign laboratories and companies. Draft recommendations for practices or procedures shall be developed based upon the needs identified above and circulated within the DOE engine community for review and modification. Round robins will be conducted as necessary. Procedures will be standardized by ASTM and/or ISO.

Technical Highlights.

1. General

Attention was focused on standards maintenance in this quarterly period. Several standards needed attention of one sort or another. The key flexural strength standard ASTM C 1161 will be significantly revised after 11 years on the books. It is due for a normal ASTM 5th year review and, since the ISO standard has been recently adopted, it is timely to go back and change C 1161. Over 24 changes, some major, will be made. A second round of major changes is underway for the fracture toughness standard, C 1421. Other revisions to the fracture toughness standard C 1421 are in a formal ASTM ballot. Other ASTM and ISO draft standards were revised.

2. Fracture Toughness

a. ASTM standard C 1421 revised again

The 40 revisions to the ASTM fracture toughness standards that were balloted in the spring of 2001 were all approved.

In the last quarterly report, we reported on how lateral cracks could interfere with determination of fracture toughness by the surface crack method. This problem, which occurs only in very brittle materials, came as a surprise to us. Nonetheless, we investigated the problem and determined a simple solution. If vestiges of lateral cracks are on the polished specimen's surface after removal of the customary amount of material, then additional materials should be

polished away. This new refinement to the SCF method, and the new data collected for alpha sintered silicon carbide, will now be incorporated into a revision to the ASTM standard C 1421.

A short paper on the lateral crack interference in the SCF method will be presented at the European Ceramic Society conference in the fall of 2001. A full paper was submitted to the Journal of the American Ceramic Society.

We are pleased that the three methods in the ASTM standard (surface crack in flexure (SCF), single-edged precracked beam (SEPB), and chevron notch in bending (CNB)) produce test results that concur within ~5% for the silicon carbide. We previously had obtained agreements within 1% for silicon nitride. All told, nearly 20 corrections or refinements to C 1421 will be balloted in the fall of 2001.

b. Standard Reference Material 2100 paper revised

A paper on the SRM 2100 for fracture toughness was revised. This paper was presented at the ASTM symposium on fracture in November 2000 and a number of changes were made in response to reviewer's requests. SRM 2100 supports the ASTM test method standard C 1421.

3. Revisions to ASTM Standard C 1161, Flexural Strength of Advanced Ceramics

After 11 years on the books, ASTM standard C 1161 is due for an overhaul. This is especially true since many key elements in it have been incorporated into the new International Standards Organization standard 14704. Nevertheless, it would be wise to change C 1161 as necessary to make it more harmonious with the new elements incorporated in the ISO standard.

Several figures and illustrations in C 1161 will be changed and replaced by figures from ISO 14704. There are no technical changes. The other changes to C 1161 include:

1. Numerous small editorial changes
2. References to new ASTM standards:
   - C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
   - C 1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
   - C 1368 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature[3]
   - C 1495 Standard Test Method for Effect of Surface Grinding on Flexure Strength of Advanced Ceramics
3. New definitions and terms:
   - inert flexural strength
   - inherent flexural strength
   - inner gage section , complete gage section
   - slow crack growth

4. Deletion of the 1/8" x ¼" x 2" specimen on 0.75" x 1.5" fixtures, Configuration "D."
5. New clarifications on what to do if fractures occur under a loading point, or outside the inner gage section.
6. A new schematic on how to interpret fracture patterns in broken bend bars.
7. New schematics and text to clarify the meaning of "semiarticulating" and "fully articulating" fixtures.
8. A new appendix on how to correct flexural strength results for oversized chamfers.
9. A new appendix explaining differences between the older MIL STD 1942 (MR) from 1983, and the newer ASTM standard C 1161. (MIL STD 1942 was cancelled by the U.S., Army 2 years ago.)
10. A *major revision* to the "standard specimen" preparation section.

The substantial amount of work that has been done at ORNL, NIST, and elsewhere in the last few years on the topic of machining ceramics will contribute to a revision of the machining requirements in C 1161. There is a trend to use finer grit wheels in preparation of both components and laboratory test pieces. Mr. Quinn visited Chand, Norton-Higgins, and Bomas in Massachusetts in April to get the latest information from and the perspectives of these leading shops. It was learned that many customers were having components finished with 400-grit or finer grinding wheels. Some customers were uncomfortable with flexure specimens prepared with only a 320-grit finish. They were concerned that the strengths may not adequately represent the finer-grit-finished components. So although a 320-grit final finish may be perfectly adequate for longitudinally ground bend bars; in most cases, the trend has been to finer grits.

It is fascinating how this has evolved over the years. When MIL STD 1942 was adopted in 1983, a bold innovation was a specification that final finishing had to be done to controlled depths with 200-500 grit wheels. ASTM C 1161 took this a step further by narrowing the range to 320-500 grit. The time has come for a further refinement. Grinding wheel manufacturers and machine shops have become more skilled with ceramic grinding, and whereas 400-grit or finer grinding was costly and difficult in the past, finer grit wheels are more routine and practical.

The proposed revision to C 1161 will call for finish grinding to 400-grit or finer finishing wheels. Another major change will be the inclusion of a simple, master blueprint for flexure specimens. An engineering drawing based on the current edition of C 1161 is enclosed here as Figure 1. The proposed specimen preparation revisions to C 1161 are listed on the two pages following Figure 1. Comments or opinions on these changes should be addressed to Mr. Quinn at NIST, or a formal ballot may be cast in Committee C-28 in the fall of 2001.

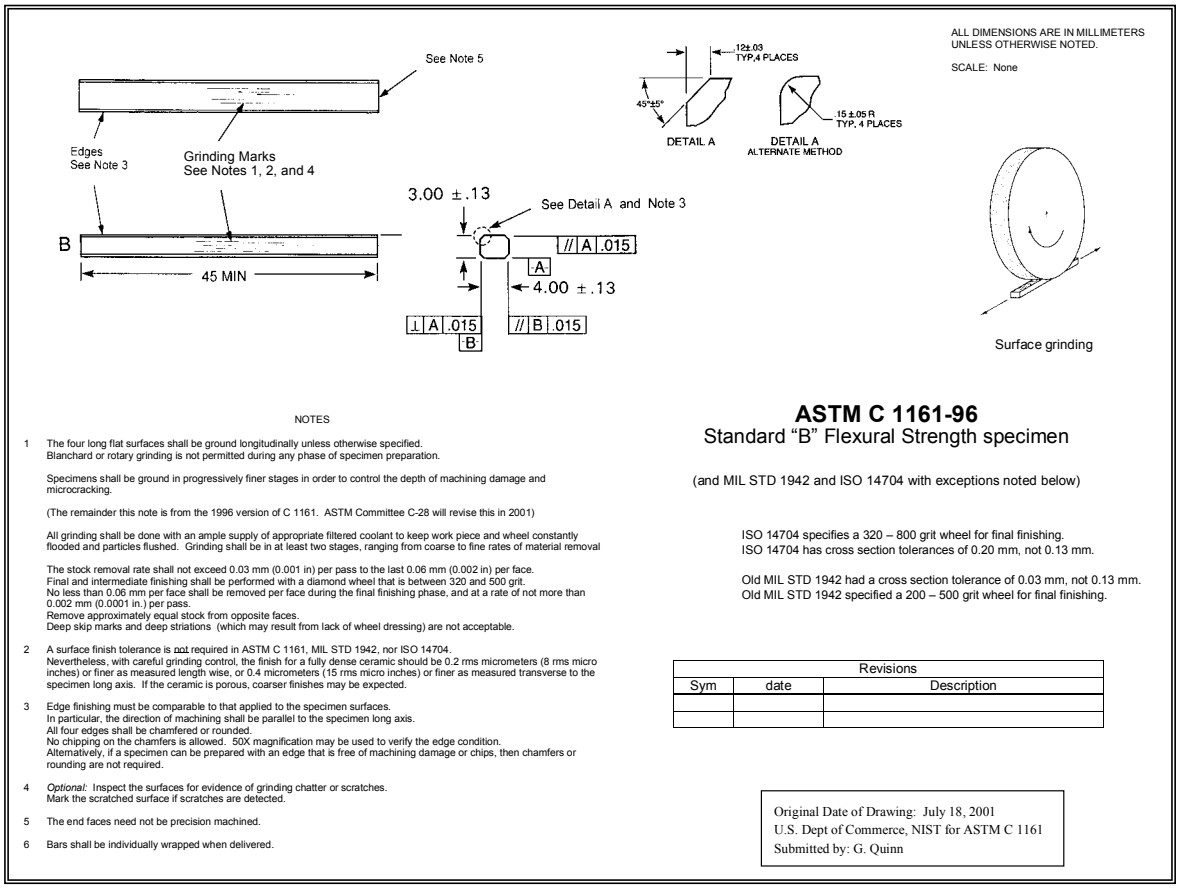


Figure 1. Proposed engineering drawing for "B" type flexural strength specimens in accordance with ASTM C 1161. The flexure strength specimen procedure <u>may be revised</u> in the Fall of 2001. If so, this drawing **<u>may be rendered obsolete</u>**.

OLD-Current C 1161-1996

7.2.4 Standard Procedures – In the instances where 7.2.1 through 7.2.3 are not appropriate, then 7.2.4 shall apply. This procedure shall serve as minimum requirements and a more stringent procedure maybe necessary.

7.2.4.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep work piece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode, and shall be parallel to the specimen long axis as shown in Figure 5. No Blanchard or rotary grinding shall be used.

7.2.4.2 The stock removal rate shall not exceed 0.03 mm (0.001 in) per pass to the last 0.06 mm (0.002 in) per face. Final and intermediate finishing shall be performed with a diamond wheel that is between 320 and 500 grit. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm (0.0001 in.) per pass. Remove approximately equal stock from opposite faces.

Proposed Revisions - OPTION # 1c

7.2.4 Standard Procedures - In the instances where 7.2.1 through 7.2.3 are not appropriate, then 7.2.4 shall apply. This procedure shall serve as minimum requirements and a more stringent procedure maybe necessary.*(unchanged)*

7.2.4.1 All grinding shall be done with an ample supply of appropriate filtered coolant to keep work piece and wheel constantly flooded and particles flushed. Grinding shall **be in two or three** stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface-grinding mode, and shall be parallel to the specimen long axis as shown in Figure 5. No Blanchard or rotary grinding shall be used. **Machine the 4 long faces in accordance with the following paragraphs. The two end faces do not require special machining.**

7.2.4.2 Coarse grinding, if necessary, shall be with a diamond wheel no coarser than 150 grit. The stock removal rate (wheel depth of cut) shall not exceed 0.03 mm (0.001 in) per pass to the last 0.060 mm (0.002 in) per face. Remove approximately equal stock from opposite faces.

7.2.4.3 Intermediate grinding, if utilized, should be done with a diamond wheel that is between 240 and 320 grit. The stock removal rate (wheel depth of cut) shall not exceed 0.006 mm (0.00025 in) per pass to the last 0.020 mm (0.0008 in.) per face. Remove approximately equal stock from opposite faces.

7.2.4.4 Finish grinding shall be with a diamond wheel that is between 400 and 600 grit. The stock removal rate (wheel depth of cut) shall not exceed 0.006 mm (0.00025 in) per pass. Final grinding shall remove no less than 0.020 mm (0.0008 in.) per face. The combined intermediate and final grinding stages shall remove no less than 0.060 mm (0.0025 in) per face. Remove approximately equal stock from opposite faces.

7.2.4.5 Wheel speeds should not be less than 25 m/sec (~1000 inch/sec) . Table speeds should not be greater than 0.25 m/sec (45 feet/min) .

7.2.4.6 The procedures in 7.2.4 address diamond grit size for coarse, intermediate, and finish grinding but leaves the choice of bond system (resin, vitrified), diamond type (natural or synthetic, coated or uncoated, friability, shape, etc.), and concentration (percent of diamond in the wheel) to the discretion of the user.

NOTE: The sound of the grinding wheel during the grinding process may be a useful indicator of whether the grinding wheel condition and material removal conditions are appropriate. It is beyond the scope of this standard to specify the auditory responses, however.

OLD - Current C 1161-1996

7.2.4.3 Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

7.2.4.4 The four long edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12 ± 0.03 mm as shown in Fig. 4. They can alternatively be rounded with a radius of 0.15 ± 0.05 mm. Edge finishing must be comparable to that applied to the specimen surfaces. In particular, the direction of machining shall be parallel to the specimen long axis. If chamfers are larger than the tolerance allows, then corrections shall be made to the stress calculation (1). Alternatively, if a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required.

Proposed Revisions - OPTION # 1c continued

7.2.4.7 Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates. *(unchanged)*

7.2.4.8 The four long edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12 ± 0.03 mm as shown in Fig. 4. They can alternatively be rounded with a radius of 0.15 ± 0.05 mm. Edge finishing must be comparable to that applied to the specimen surfaces. In particular, the direction of machining shall be parallel to the specimen long axis. If chamfers **or rounds** are larger than the tolerance allows, then corrections shall be made to the stress calculation ~~(1).~~ **No chipping is allowed. Up to 50X magnification may be used to verify this.** Alternatively, if a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required.

7.2.4.9 *Optional:* Visually inspect the surfaces for evidence of grinding chatter, scratches, or other extraneous damage. A 5X – 10X hand loupe or a low power stereo binocular microscope may be used to aid the examination. Mark the scratched surface with a pencil or permanent marker if scratches are detected.

> **Note: Damage or scratches may be introduced by handling or mounting problems. Scratches are sometimes caused by loose abrasive grit.**

7.2.4.10 Very deep skip marks or very deep single striations (which may occur due to a poor quality grinding wheel or due to a failure to true, dress, or balance a wheel) are not acceptable.

Committee C-28 is also contemplating revising the definition of flexural strength. The current definition in C1161 is:

Flexural strength a measure of the ultimate strength of a specified beam in bending.

The new definition, already included in the slow crack growth standard 1368, is:

Flexural Strength a measure of the ultimate strength of a specified beam in bending *determined at a given stress rate in a particular environment.*

The history of how the former, shorter definition was derived in the early days of Committee C-28 was researched and a position paper on the merits of the two definitions was prepared and sent to the C-28 terminology chairman. In a curious development, the European Community prepared an alternative definition:

Nominal Flexural Strength The maximum nominal stress at the instant of failure supported by the material when loaded in elastic bending.

An office thesaurus equates "nominal" with "so called," "in name only," "ostensible," or "supposed." These are unnecessarily weak and probably attest to confusion in Europe over whether the maximum outer fiber stress is a "correct number" to use for flexural strength. It is. Nonetheless, a better explanation why the maximum outer fiber stress is the correct value to use will be incorporated as one of the revisions to C 1161. Quibbling over definitions may seem trivial, but the disagreements often reflect concern or confusion over important technical details.

4. Upgrade Fractographic analysis equipment

During this quarterly period, new digital cameras were installed on the stereo binocular and research metallographic microscopes. A new computer was installed. These upgrades were accomplished with NIST depreciable equipment funds, but the benefits will be felt in this DOE supported program. We will be able to capture superior images of ceramic fracture surfaces. Some time was spent with the equipment installation, software upgrades (and headaches), and programming tutorials.

5. Flexure Testing of Cylindrical Ceramic Specimens

There was no work on cylindrical rod testing this period. Dr. Klaus Morgenthaler of Daimler-Chrysler I Germany was contacted about Daimler's experiences with machining cracks damage depths.

6. Upgrade Microhardness machine

An old Leitz Miniload II microhardness machine was acquired on loan from the U.S. Army Materials Research laboratory. Indenter asymmetry and misalignment problems were solved with the aid of a summer student. New optical calibration factors were determined with the aid of a certified stage micrometer.

7. Other

a. Design of new Semi articulating flexure fixtures for rectangular specimens

No activity this period

b. ISO TC 206, Working Group 16, Fracture Toughness by SCF method

The new draft 3a of this standard was finished and submitted to the ISO secretariat for formal distribution. The standard includes major changes in light of the lateral crack problems and new research findings as discussed in the last quarterly report. More input from the Korean delegation was received, which showed that slow crack growth can seriously interfere with the SCF method, and to a lesser degree, with the SEPB method. The data furnished by the Koreans was for a 99.5% alumina.

c. ISO TC 206, Working Group 2, Flexure Strength at Room Temperature

The document has been approved as an ISO standard.

d. ISO TC 206, Working Group 8, Flexure Strength at Elevated Temperature

No activity this period.

e. ASTM Committee G-02 *scratch hardness standard*

A new scratch hardness standard prepared by Peter Blau was reviewed and an affirmative with comment ballot was filed.

<u>Status of Milestones</u>

Milestones must be revised.

<u>Communications/Visits/Travel</u>

G. Quinn gave a talk on international fracture toughness standards at the American Ceramic Society annual conference in May 2001.

A review of allowable asymmetries in Knoop and Vickers hardness indentations was made in response to a request from the National Physical Laboratory in England.

A draft position paper on the future of ASTM committee C-28 was prepared and sent to the new chairman, Prof. M. Jenkins of the University of Washington.

Notes on prior activities within C-28 on gravity sedimentation powder test methods were sent to Dr. S. Gonczy of Gateway Associates. Dr. Gonczy has agreed to lead a small task group in the preparation of a test method standard or guide for powder characterization by the gravity sedimentation method. Mr.

Quinn searched through his files on the history of ASTM C-28 and IEA round robin activities on this method, and furnished a summary for Dr. Gonczy.

Mr. Quinn contacted Dr. Klaus Morgenthaler of Daimler-Chrysler in Ulm, Germany, by phone and mail in connection with machining of ceramics. Daimler had learned a great deal about machining ceramic valves, bend bars, and other components over the years. Mr. Quinn contacted Morgenthaler to inquire about Daimler's preferences for wheel grits, etc., for the revision to the ASTM C 1161 standard. Over 10 years ago, Morgenthaler prepared an intriguing schematic showing the depth of damage created during the machining of silicon nitride. Mr. Quinn contacted Morgenthaler to learn more background information about that intriguing and perceptive illustration.

Reports on flexure fixtures were furnished to engineers at the University of North Dakota and the American Dental Association. These groups will probably copy the quasi-standard ASTM semi-articulating flexure fixture design.

Information on testing thin ceramics was furnished to Dr. G. Crosbie of the Ford Motor Company.

Publications

1. G. D. Quinn and J. A. Salem, "Effect of Lateral Cracks Upon Fracture Toughness Determined by the Surface Crack in Flexure Method," accepted by J. Am. Ceram. Soc., July 2001.
2. G. D. Quinn, "Refinements to the Surface Crack in Flexure Method for Fracture Toughness of Ceramics," subm. to Proceedings of the 7th European Ceramic Society Conference, Brugge, Belgium, Sept. 2001.
3. G. D. Quinn and J. A. Salem, "Effect of Lateral Cracks Upon Fracture Toughness Determined by the Surface Crack in Flexure Method," accepted by J. Am. Ceram. Soc., July 2001.