C. Mechanical Property Test Development

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Objective

- Develop mechanical test method standards in support of the Propulsion Systems Materials Program. New methods and sound engineering data will facilitate adoption of new materials in heavy vehicle propulsion systems.

Approach

- Conduct pre-standardization research on test methods that need refinements, or develop new methods.
- Develop draft recommendations for practices or procedures based upon the needs identified by the research.
- Conduct round robins as necessary.
- Standardize procedures in the American Society for Testing and Materials (ASTM) and/or the International Organization for Standards (ISO).

Accomplishments

- Conducted new experiments on zirconia split cylinder flexural strength test specimens using the latest generation material and compared results with full rod flexural strength test results. Conducted extensive fractographic analysis to find the strength-limiting flaws in this commercially important material.
- Neared completion of the comprehensive Guide to Practical Fractography for Ceramics and Glasses.
- Completed a set of papers and made presentations.
- Revised, refined, and improved several current ASTM and ISO standards.

Future Direction

- Finish the split cylinder flexural strength test work and write a paper on the method. Show how a simple modification of ordinary bend fixtures for rectangular bars can do double duty and also work with split cylinders.
- Finish the comprehensive Guide to Fractography.
- Complete prestandardization work on flexure testing of rods and write the first draft of an ASTM standard.

- Resume pre-standardization evaluation of diametral compression strength testing for small round-shaped specimens

Introduction

This project creates new test methods that will facilitate the use of advanced materials in heavy-weight propulsion systems. Much of the work is for brittle materials such as ceramics, for which classical metal mechanical test methods are not suitable. For example, tension strength test specimens of many ceramic materials made in short, stubby cylindrical shapes (e.g., diesel engine fuel injector pins, timing plungers, valves) and classical dog bone shapes are impractical (Figure 1). Our goal is to adapt or refine existing methods or invent new ones that will allow engineers and researchers to measure mechanical properties with good accuracy and precision. They then will be able to construct their own databases with greater confidence. Formal test method standards are our primary objective. Sound test methods and high-quality databases will enhance the credibility of new materials and encourage engineers to use them in advanced heat engines. This purpose of this project is to develop the test methods.

Figure 1. Round ceramic engine components.

Approach

Over the course of this program, we have formulated or contributed to the development of 17 ASTM and ISO standards. We currently are working aggressively on three more:

- Flexural strength of cylindrical rods
- Flexural strength of split cylinders
- Fractographic measurement of fracture mirror sizes

We also plan to tackle the diametral compression strength test method when those three standards are completed in FY 2006.

The standards adopted so far include methods for ceramics such as flexural strength, elastic modulus, Weibull statistical analysis, fractographic analysis, and fracture toughness. As a direct result of this work, there has been a dramatic improvement in test data quality and reliability in the structural ceramics field. We have also contributed to a ceramic material specification for silicon nitride ball bearings. The specification uses several of the test method standards developed in this program. The test method standards have been sufficiently generic that they have even been used in to the biomedical field for materials specifications for ceramic surgical implants.

Considerable energy was put into finishing a Guide to Practice for Fractography of Ceramics and Glasses this year. This is a large document that has been written with a strong practical slant. It will complement the ASTM standard practice for fractography, and it is intended to help engineers and scientists find flaws and fracture origins and help them do their jobs better. The goal is to make fractographic analysis less an art and more an engineering practice.

Results

Flexural Strength of Split Cylinders

Splitting a rod and testing the halves is one way of evaluating the strength of short, stubby cylindrical parts that otherwise might be difficult to test correctly. The break forces for solid full rods are very high and fixtures may be damaged, or high contact stresses could cause Hertzian cracking in the specimen. Splitting the rods makes them much easier to test, as shown in Figure 2. Our goal is to refine this simple procedure and make it available to engineers and scientists as an optional test configuration.

We did a thorough fractographic analysis of the split Coors zirconia rods that we had previously broken. Our goal was to identify the strength-limiting flaws and ascertain whether the new fixture

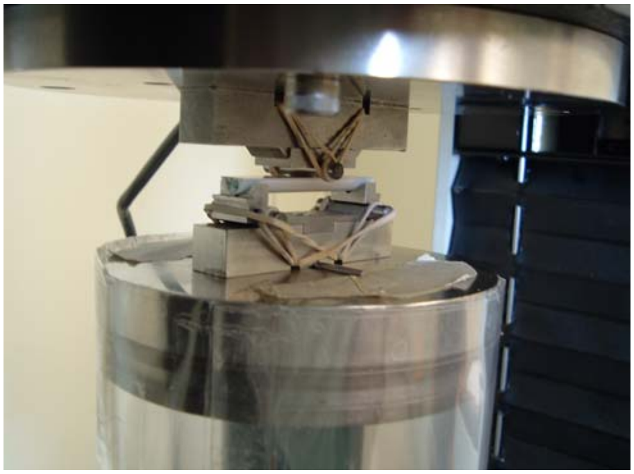

Figure 2. Split zirconia fuel injector pin in the NIST bend fixture for split rods.

scheme produced legitimate test results and breakage patterns. Fractography was very difficult in this material because of its coarse microstructure, but we found that flaw pockets (local clusters of tiny defects), as shown in Figure 3, and grain boundary faults were the origins. A paper on this work is in preparation.

We did additional split cylinder testing to fill in a gap in our results. We previously had tested split rods made from the Coors zirconia, but we did not have any solid rod strengths with which to directly compare our results. To get a better sense of whether the split cylinders were giving data comparable to or different from those for solid rods, we obtained a new set of solid zirconia pins from Cummins. These were from a set of Carpenter zirconia rods that had been set aside. Some of these were split into two halves and each half was tested, as shown in Figure 2. The 4-point testing was with effective spans of 20 × 40 mm. The 3-point testing was with a 40-mm span. The strength outcomes are shown in Figure 4. Three-point strength numbers for the same batch of material that were obtained by Cummins are also shown for comparison. The good news is that the 3-point strengths of the split rods are nearly identical to those of the full rods. The 4-point strengths are less, and the shift was in perfect accordance with Weibull size effects.

The fracture origins these specimens were also thoroughly analyzed. An interesting outcome was that most specimens failed from a flaw type not detected in the Coors batch. The Carpenter specimens had lovely fracture surfaces with distinct mirrors and broke from transformed grains right on the outer ground surfaces, as shown in Figure 5. Evidently improved processing in the Carpenter material has eliminated many of the grain boundary and flaw pocket faults that controlled properties in the earlier-generation Coors material.

A remarkable finding was that although the split cylinders had the usual strength variability associated with ceramics, the strengths of the two halves of a particular rod were remarkably consistent. That suggests that the strength-limiting flaws are very consistent within any given rod.

Our work is converging on a very simple solution for this test method. An ordinary bend fixture designed for rectangular specimens may be easily adapted by using simple cradles with alignment parts that enable it to instantly convert to test split cylinders. Engineering drawings of the new single-piece cradles are being prepared.

Flexural Strength of Solid Cylindrical Rods

Work resumed in conjunction with the split cylinder testing described in the next paragraph. As

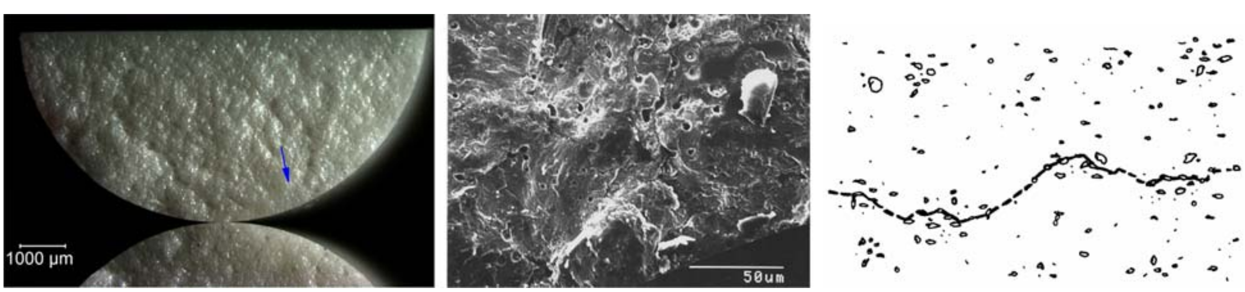

Figure 3. Fracture surface of a Coors zirconia slit cylinder specimen showing the very rough surface. The origin was a region of locally greater porosity coupled with grain boundary faults. The schematic suggests how these pockets could control strength.

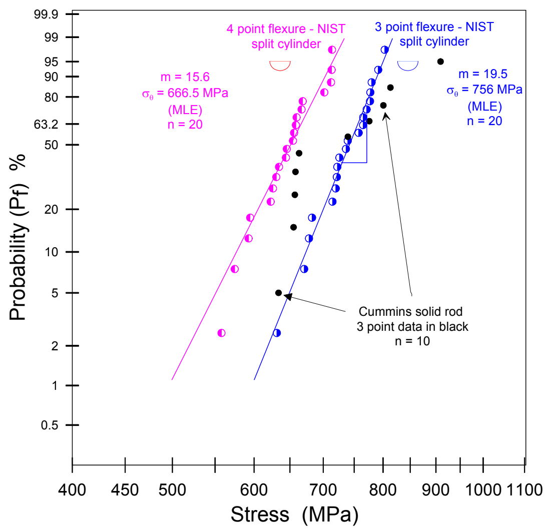

Figure 4. Flexural strengths of split zirconia rods tested measured at NIST. Cummins 3-point data for solid rods is shown in black. Weibull parameter estimates are noted. The 3- and 4-point distributions are simply shifted in accordance with the customary Weibull size effect.

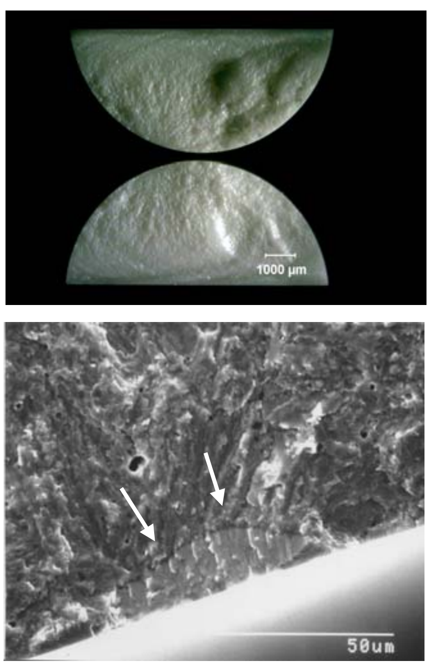

Figure 5. Fracture surface and origin of a Carpenter zirconia split rod specimen. The microstructure is less rough than in Figure 3, and there is a fracture mirror. The second photo shows a typical origin: a transformed grain on the ground surface.

part of our fixture design work, we tested one over-sized steel dowel rod in our flexure fixture that has special cradles to hold round specimens (Figure 6). We did this to ascertain whether minor yielding in the steel cradles (which had been detected after doing some ceramic tests) was due to fixture over-load or an artifact of a transient load pulse when the ceramic specimens fractured. The yielding was only on a tiny portion of an edge of the cradles, but we decided to investigate the source of the deformation to possibly refine our new fixture design. The fixture and hardened steel dowel were loaded to static loads well above those used in the ceramic strength tests and then unloaded for inspection. No deformation whatever was detected in the steel fixture parts or the hardened steel dowel rod. This tends to confirm the notion that a shock–pulse in the fixture when the ceramic test pieces broke was the cause of the observed small deformations.

We have resumed our error analyses work for flexure testing of round specimens. This will be the cornerstone of the standard. Once we have analytical estimates of errors for different specimen and fixture geometries, then we will be able to intelligently select an optimum configuration and set ranges of acceptable configurations and close practical engi-neering tolerances. The error analysis builds upon similar work for rectangular specimens done by Francis Baratta and Quinn in the early 1980s.

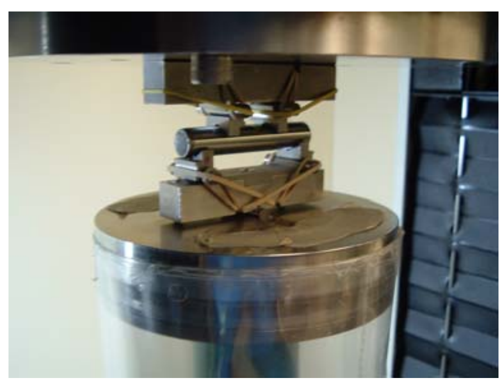

Figure 6. Steel dummy specimen in the same 4-point bend fixtures that were used to test split cylinder specimens. Special cradles hold the round specimens in the fixtures that are normally used to test rectangular bend specimens. Deformations on the cradle edges were observed after ceramic strength tests.

Nuances pertaining to rod shapes must be considered, however, before the analysis can be directly applied to the rod. Most errors are similar or identical, but contact point tangency shift with the new cradles, errors from severe contact loadings from crossed rollers, and wedging stresses are different for rods and rectangular bars. So, for example, in October 2005 we completed a new, simpler analysis for contact point tangency shift for beams in flexure. This error occurs when the specimen deflects and its point of contact with the loading rollers shifts. This in turn affects the moment that is applied to the specimen and alters the stress in the beam. The error is minimal (< 1%) in the standardized rectangular bend bars so long as loading roller sizes are kept small. A similar effect is expected for rods, except that if cradles are used, the error could get as large as 5% or more. The original Baratta analysis of 1982 was cumbersome and required an iterative analysis to solve for a particular geometry. The newer, simpler analysis we derived gives virtually identical error estimates and is far simpler to use. Our goal is to finish the complete error analysis by December 2005, so that we can use the analysis to set reasonable testing limits for an ASTM draft standard.

Guide to Fractography

Work continued on a NIST user-friendly Guide to Best Practice for fractographic analysis. This pamphlet-style document will be about 400 pages long with hundreds of figures illustrating key fractographic markings in ceramics and brittle materials. It will complement ASTM standard C 1322, Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics.

Small revisions to five flaw definitions in ASTM C 1322, Standard Practice for Fractography, were balloted. Two negatives were obtained. These negatives were discussed at the Reno, Nevada, meeting of ASTM Committee C-28, Advanced Ceramics, on May 16, 2005, and were found to be unpersuasive. The final revised version 2005b of C1322 was reviewed and has been sent to press. This concludes all our planned revisions to this standard for the time being.

One important byproduct of the Guide to Best Practice is a new set of guidelines for how to measure fracture mirrors. The guidelines are one step away from a formal standard. They will be shown to ASTM Committee C-28 in January 2006 for review and consideration. Fracture mirrors are relatively smooth surrounding a fracture origin in a brittle material, such as is shown in Figure 7. Mirrors are formed when the crack radiates outward from the origin and initially generates a smooth surface, but then creates a rough surface once the crack reaches terminal velocity. A remarkable characteristic of fracture mirrors is that their size is inversely related to the square of stress in the part at the instant of fracture. Thus mirrors may be used to estimate the breakage stress in failed components, even if the engineer has no idea how the parts were loaded or stressed. Mirrors are discussed in the ASTM fractography standard C 1322, which focused on how to characterize fracture origins, but there was no guidance on how to measure them. There are wide variations in how these fractures are currently measured. We are ready to rectify this now with a new standalone standard if the ASTM Committee concurs.

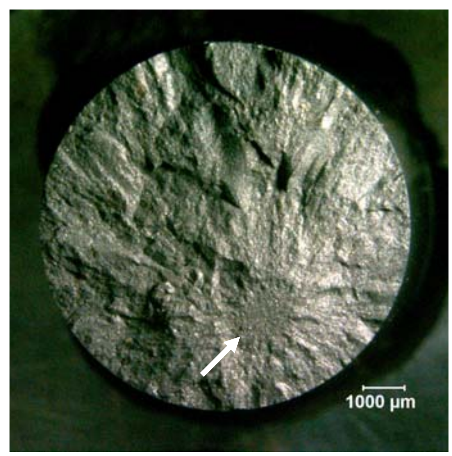

Figure 7. Fracture mirror in a ceramic. The fracture stress can be estimated fairly accurately from the size of the relatively smooth circular fracture mirror.

Other

A review paper, "Design and Reliability of Ceramics: Do Modelers, Designers, and Fractographers See the Same World?" was presented at the American Ceramic Society conference in January. One of the case studies cited in the paper was the excellent work done by the Ford Motor Company in the early 1980s on a model silicon nitride gas turbine rotor that was spun to failure in a hot test rig (Figure 8).

Figure 8. The Ford model turbine rotor was a featured example in a review paper on design and reliability.

Although a high-quality ASTM standard for fracture toughness evaluation has been on the books for 8 years, and although NIST has a Standard Reference Material (SRM) 2100 to support it, people are still using the defective Vickers indentation crack length method to evaluate fracture toughness. Therefore, we have begun some Vickers indentation crack length measurements to estimate the "indentation fracture resistance" of the NIST SRM 2100 in order to demonstrate the shortcomings of the method and the errors that can result. Our goal is to get these findings into the literature as soon as possible before the adoption of indentation crack length methodologies gets codified too far. We advocate the use of genuine fracture mechanics tests for evaluating fracture toughness. The Vickers indentation crack length method evaluates a particular measure of fracture resistance, which crudely approximates fracture toughness. The indentation crack length method may have some utility, but data from it ought to be deemed "indentation fracture resistance" and not "fracture toughness."

We helped to update and refine some existing ASTM and ISO standards. We are continuing to work with W. Mandler of Enceratec/Cummins and B. Mikijelj of Ceradyne as the silicon nitride ball bearing material specification F2094 evolves. Some corrections to data specifications were reviewed and balloted this year. The ASTM specification is also being converted into an ISO international standard. There is some controversy about some of the procedures, and we are working with the stakeholders to ensure that a technically sound specification is prepared. Another example is the new static fatigue test method draft standard that we helped review. Quinn reviewed the history of Committee C-28's awards and furnished a summary report to the current award subcommittee leader. Quinn also reviewed the history of the development of two ASTM powder characterization standards, C 1282 and C 1274, and two nondestructive evaluation standards, C 1212 and C 1336, and sent a summary report to Steve Gonczy, the C-28 chairman. Gonczy wanted to know the backgrounds so that plans could be made to upgrade, refine, or drop these standards as appropriate.

Conclusions

Step by step, we are building a national and international standards infrastructure to facilitate the commercial utilization of new advanced materials in engine applications. The generic test method standards developed to date have proved to be so practical, reliable, and versatile that they are now being used to support a wide range of applications, including surgical implants in humans and even ceramic military body armor.

Publications/Presentations

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