THE SEGMENTED CYLINDER FLEXURAL STRENGTH TEST

George D. Quinn National Institute of Standards and Technology 100 Bureau Drive Gaithersburg, MD, 20899-8529 Presented at Cocoa Beach Conference of the American Ceramic Society, January 2006. Ceramic Eng. and Sci. Proc., **27** [3] 2006 pp. 295 - 305.

ABSTRACT

Weiler showed in 1987 that the strength of ceramic heat exchanger tubes could be evaluated by flexural strength tests of cylindrical segments or longitudinal slices cut from the tubes. In the present study, a similar approach has been applied to measure the strength of short solid ceramic cylinders. Large loads are normally needed to break these cylinders in flexure and there is a risk of contact stress problems at the loading points. A simple remedy is to cut the rods longitudinally into two halves and test each half. Split zirconia short rods were tested using a conventional bend fixture with simple modifications. Strengths were compared to those of solid round rod parts. Fracture origins were the same type in each case. The split cylinder or segmented cylinder method is a simple, practical, and effective way to measure flexural strength.

INTRODUCTION

Flexure strength testing is the most common mechanical test for ceramics and glasses, with the possible exception of hardness testing. The vast majority of tests on ceramics and many glasses utilize rectangular bars. Methodologies for testing the latter have been optimized and have been standardized across the world.^{1,2,3} Not all ceramics and glasses are available in forms that are conducive to rectangular bar testing, however. Many are available as rods or as components that are fabricated and machined to a cylindrical shape. Cutting a rectangular bend bar out of these shapes is inefficient, costly, and even unsound if the strength test is intended to reflect the actual surface condition of the round part. We are currently engaged in a project to develop a standard flexural strength method for cylindrical rods. An ASTM standard for glass rods already is on the books,⁴ but it is very old and has obvious shortcomings such as the use of fixed loading points for load application. Fixed loading points cause a substantial friction error. The test is described in the standard as being suitable for comparative purposes and limits the specimen size and test configurations. For example, the length to diameter ratio shall not be less than 10. Very little has been done to optimize rod strength testing although Duckworth's seminal paper⁵ in 1951 identified some key factors for obtaining accurate data.

Ceramic and glass parts are often available in the form of short stubby cylinders such as shown in figure 1. Very large test forces are necessary to break such specimens in flexure and usually only three-point loading is used. Four-point loading would require extraordinary forces that would cause contact cracking damage at the loading points and damage to the bend fixtures. Shear stresses (which are negligible for long slender beams and rods in flexure) would be severe in either three- or four-point flexure of short stubby rods.

A simple remedy is suggested by the work of Weiler who measured the strength of 25 mm diameter beta alumina ceramic tubes in 1987.⁶ Weiler cut axial slices from the tubes and tested them in flexure. He coined the expression "cylinder-segment bend test." Volume-distributed

origins of failure were more readily obtained in these specimens than in compressed C-ring specimens or in rectangular bend bars. Recently Wereszczak and colleagues have successfully applied Weiler's approach to evaluate ceramic gun barrel liners.^{7,8,9}



Figure 1 Intact and split zirconia diesel fuel injector pins. The 9 mm diameter portion is only 38 mm long for a length to diameter ratio of only 4.2.

The "segmented cylinder" or "split cylinder" approach is utilized in the present study to measure the flexure strength of short stubby magnesia-stabilized zirconia rod specimens. This work began in 1998 and early preliminary findings were previously published in a limited fashion.¹⁰ The work has been expanded and almost completed. This paper is the first formal presentation of the approach and with new results.

MATERIAL

Twenty magnesia partially-stabilized zirconia (Mg-PSZ) rods from one source were evaluated.^{a, b} The rods were blanks for fuel injector plungers and had not been ground to final shape. They were 9.0 mm diameter by 47.5 mm long and had a fine ground finish probably from by centerless grinding. The rods were sliced in half to make forty specimens. Half were tested in three-point and half in four-point flexure for comparison. A few additional finished plungers from another source^c were tested in 1998 to demonstrate the proof of concept and were mentioned in our preliminary report.¹⁰ These results are not presented here but some of the illustrations in this paper show these alternative pieces which had been ground to final shape and had reduced diameter end shanks.

^a Certain commercial materials or equipment are identified in this paper to specify adequately the experimental procedure. Such identification does not imply endorsement by the NIST nor does it imply that these materials or equipment are necessarily the best for the purpose.

^b Carpenter Advanced Ceramics, Auburn, CA.

^c CoorsTek, Inc, (then Golden Ceramics), Golden, CO.

TEST FIXTURES

Figures 2 and 3 show the test fixtures with split cylinder specimens in place for three-point loading. This set up is a variation of our normal bend fixtures for testing rectangular specimens. The top inner span has an insert with a V-groove to hold a single roller in the middle for three-point loading. The middle roller does not need to roll, nor is it necessary for it to articulate to match the specimen surface and ensure even line loading since the specimen itself is free to rotate slightly. A simple upper loading piece is adequate. For four-point loading, the insert would not be used and two rollers would be held in place by the shoulder stops and rubber bands.

The lower loading piece for the outer span has a simple removable insert to position the two outer rollers to whatever span is required. Two cradles with V-grooves sit on the lower rollers and apply a distributed load to the specimen. The distributed load is statically equivalent to a concentrated load at the roller provided that the V-groove is symmetric and centered over the roller. The contact length of the cradle V-grooves is exactly the same size as the roller diameter. It is possible that acceptable results may be obtained without the cradles with the split rod sitting directly on the loading rollers, but we were concerned that contact stresses at the two crossed curved surfaces would be excessive. A large base roller on the bottom is used for four-point loading. It allows the lower loading piece to pivot up and down a bit to ensure even loading on the two outer loading rollers. For three-point loading, this pivoting is unnecessary and in fact undesirable. For three-point loading, a dowel pin of a particular size is used to center and hold the outer span roller base in place with no pivoting movement.



Figure 2 Schematic and photo of the test fixtures in the three–point configuration. The schematic shows the fixture with a finished split plunger in place with a short 30 mm span. The photo shows the fixture with a split rod on 40 mm span and slightly redesigned cradles. The extra tab on the bottom of the cradles facilitates their installation and alignment. A few extra parts are shown in the background and foreground for clarity.



Figure 3 Fixture with a split plunger specimen in place in three-point loading with a 30 mm span. Rubber bands hold the rollers in place. The upper loading piece is an articulating design, but we later learned that this was not necessary for three-point loading.

Note the cut surface is loaded in compression and that the original specimen surface is in tension. The three- and four-point outer spans were 40 mm. The inner span for the four-point tests was 20 mm. All testing was in ambient conditions (23 °C to 25 °C) with relative humidity that varied from 55 % to 60 % since testing was done on different days. Cross head rate was 1.3 mm/min for all tests. Break loads were 900 N to 1100 N with break times of 10 s to 15 s for the three-point tests and 1400 N to 2000 N for the four-point tests for times of 15 s to 20 s.



Figure 4 Geometry of the segmented cylinder cross section.

FLEXURAL STRENGTH

The flexural strength for a beam in bending is given by:

$$\sigma = \frac{Mc}{I} \tag{1}$$

where σ is the stress, M is the applied moment at fracture, c is the distance from the neutral axis, and I is the second moment of inertia about the neutral axis. The moment for three-point loading is simply PL/2 where P is the total force and L is the span. For four-point loading, the moment is Pa/2 where a is the distance from an outer to an inner roller. Symmetric cross sections such as a rectangle or a full circle have neutral axes that pass through the middle of the beam cross section in which case c is the half height of the beam. The situation is more complex for the case of the segmented cylinder as shown in figure 4. The geometry is asymmetric from top to bottom and the location of the neutral axis and the moment of inertia are more complicated trigonometric functions as given below. With reference to figure 4, A is the area, R the radius, h the height of the piece, α the angle in radians, and I the moment of inertia about the neutral axis. Equation 6 gives the maximum outer fiber stress which is the nominal stress to be reported for a flexure strength test. (Equation 6 is nearly identical to Weiler's, except that he designated α as the total included angle.⁶)

$$\alpha = \arccos\left(1 - \frac{h}{R}\right) \tag{2}$$

$$A = \frac{R^2}{2} \left(2\alpha - \sin 2\alpha \right) \tag{3}$$

$$c = R - \left[\frac{4R\sin^3\alpha}{3(2\alpha - \sin 2\alpha)}\right]$$
(4)

$$I = \frac{AR^2}{4} \left[1 + \frac{2\sin^3 \alpha \cos \alpha}{\alpha - \sin \alpha \cos \alpha} \right] - \frac{4R^6 \sin^6 \alpha}{9A}$$
(5)

$$\sigma = \frac{M}{R^3} \left[\frac{\left\{ 1 - \frac{4\sin^3 \alpha}{3(2\alpha - \sin 2\alpha)} \right\}}{\left(\frac{\alpha}{4} - \frac{\sin 4\alpha}{16} \right) - \left(\frac{8\sin^6 \alpha}{9(2\alpha - \sin 2\alpha)} \right)} \right]$$
(6)



Figure 5 Flexure strengths for split and solid rods. The two halves of any given solid rod usually had very similar strengths. Three such pairs are highlighted by the curved lines with arrows.

RESULTS AND DISCUSSION

Figure 5 shows the strength outcomes for the three- and four-point tests with the 40 mm outer spans. The average strengths for the four-point and three-point specimens were 646 MPa and 737 MPa, respectively, a 14.0% difference. The standard deviations were 45 MPa in each instance. The characteristic strengths (shown in the figure) differ by 13.3 %. The figure also shows limited data for ten circular rods from the same batch of material. They were broken by Cummins Engine Company for quality control purposes. They used three-point flexure with ordinary bend fixtures having 30 mm spans with no special precautions and obtained an average strength of 731 MPa and a standard deviation of 92 MPa. The material manufacturer reports the

four-point flexural strength as 725 MPa but without a standard deviation and without any indication of the specimen type or fixture size.¹¹

The two split cylinder data sets are almost parallel but are displaced relative to each other consistent with expectations based upon Weibull scaling. Closed form solutions are not available for the effective volume or effective surface of segmented cylinders. Weiler grappled with the problem and compared the effective volumes of hoop stressed tubes, compressed C-ring and segmented cylinder specimens for his specific tube geometry. An interesting nuance of Weibull strength scaling with size is that, for specimens of constant cross section shape and size, the relationship between one quarter point, four-point and three-point strengths when the same outer span is used is:¹²

$$\frac{\sigma_{3pt}}{\sigma_{4pt}} = \left(\frac{m+2}{2}\right)^{\frac{1}{m}}$$
(7)

where m is the Weibull modulus. This relationship holds no matter what the cross section shape is: rectangular, round, segmented cylinder, or other. Furthermore, the ratio holds irrespective of whether the flaws are surface or volume distributed. Using an average Weibull modulus of (15.6 + 19.5)/2 = 17.55, the strength ratio is predicted to be 13.9 %, in superb agreement with the actual data shift.

The limited solid rod three-point data had an unusual dichotomy as shown in figure 5. Five of the strengths matched the three-point split cylinder data, but the other five fell between the two split cylinder data sets. It could very well be that the scatter was simply a chance outcome due to the small sample size (n=10).

A very unusual and interesting trend was observed with the split cylinders. The two halves of each rod had very similar strengths. Three examples are highlighted for the four-point data in figure 5. The trend was observed for nearly all the data points and for both three-point and fourpoint. This strongly indicates that most of the strength variability is between parts and not within a part. This should have interesting ramifications for Weibull statistical and reliability analyses.

The breakage patterns for the split rods were as expected. The three-point specimens broke from origin sites near to but not necessarily directly under the middle loading point. The fourpoint specimens broke from sites in the inner span. Fractographic analysis was aimed at identifying the strength limiting flaws and their distribution and, in particular, why the ten solid cylinders had the strength dichotomy.

Fracture origins are notoriously difficult to find in sintered Mg-PSZ. The fracture surfaces are so rough that the classical fracture markings are obscured or absent. This is consequence of the coarse grain size, moderate amounts of microporosity along grain boundaries, and spray dry agglomerates that do not sinter fully together. Patience and perseverance paid off in this case however, and many fracture origins were ultimately identified. Figure 6 shows optical images of uncoated and coated fracture surfaces. Although the origin sites could be detected, the translucency and roughness of the origin area mandated scanning electron microscope (SEM) examination. Nearly all the origins were at or very near to the outer part surface. Figure 7 shows examples of the most common flaw detected: transformed zirconia grains connected to the surface. It is surmised that the grinding preparation led to transformation of the tetragonal precipitates in some large cubic zirconia grains. The typical grain size was several tens of micrometers. Figure 8 shows polished sections of the zirconia. Grain uplift is clearly evident suggesting that even polishing can trigger transformation. Several specimens probably failed

from "flaw pockets", or regions of tiny flaws as shown in Figure 8b. Flaws of this type are extremely difficult to diagnose on the fracture surface since they blend into the natural roughness.

The strength dichotomy of the ten solid rods could not be traced to a flaw type shift. Every specimen was examined on the SEM and the origins were: four transformed grains, two flaw pockets, one subsurface crack caused by a scratch that linked with agglomerate, and surprisingly, three specimens with shallow (5 μ m, 7 μ m, and 8 μ m deep) grinding cracks that linked with material flaws. The latter were surprising since Mg-PSZ is reputedly insensitive to surface damage due to its high fracture toughness, reported by the manufacturer to be 8 MPa \sqrt{m} to 12 MPa \sqrt{m} .¹¹ The strength dichotomy is probably caused by the same part to part strength variability noted above in the discussion of the split rod test results.



Figure 6 Uncoated (a) and gold coated (b) optical images of the same fracture surface of a fourpoint segmented cylinder specimen. The arrows mark the origin. 575 MPa



Figure 7 Fracture origins of two segmented cylinder specimens. Surface connected transformed grains acted as the strength limiting flaws.



Figure 8 Polished sections of the Mg-partially stabilized zirconia showing outlines of uplifted grains and, in (b), a flaw pocket of microporosity and other microstructural irregularities.

CONCLUSIONS

The segmented cylinder flexural strength test is a simple procedure for measuring the strength of short stubby cylinders. Specimen preparation costs are minimal and the cut surface is inconsequential. Two strength data points are obtained for each test piece and the original surface condition is retained. Only simple adaptations to ordinary bend fixtures need be made. Further work will address whether the load distribution cradles are even necessary.

ACKNOWLEDGEMENTS

Support for this work was furnished by the U. S. Department of Energy, Heavy Vehicle Materials Program with Oak Ridge National Laboratory. Brian Sparenberg assisted with the testing and Sandra Claggett with the specimen polishing. The author thanks Thomas Yonushonis and Randall Stafford for consultations and for furnishing the rods and the solid rod data.

REFERENCES

- 2 ISO 14704 (2000) "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) Test Method for Flexural Strength of Monolithic Ceramics at Room Temperature," International Organization for Standards, Geneva, 2000.
- 3 G. D. Quinn and R. Morrell, "Design Data for Engineering Ceramics: A Review of the Flexure Test," *J. Am. Ceram. Soc.*, 74 [9] (1991) pp. 2037 2066.
- 4 ASTM C 158-02, Standard Test Methods for Strength of Glass by Flexure (Determination of Modulus of Rupture), Annual Book of Standards, Vol. 15.02, ASTM, West Conshohocken, PA, (originally adopted in 1940, the current version is dated for 2002).
- 5 W. H. Duckworth, "Precise Tensile Properties of Ceramic Bodies," *J. Am. Ceram. Soc.*, 34 [1] (1951) pp. 1 -9.
- 6 L. Weiler, "Cylinder-Segment Bend Test: A New Strength Measuring Technique for Ceramic Tubes and Cylinders," pp. 621 625 in *Science of Ceramics 14*, ed. D. Taylor, The Institute of Ceramics, Stoke on Trent, United Kingdom, 1988.
- 7 A. A. Wereszczak, J. J. Swab, S. F. Duffy, and E. H. Baker, "Strength of a Sectored Flexure Specimen," presented at the 28th International Cocoa Beach Conference on Advanced Ceramics and Composites, Cocoa Beach, FL, 27 January 2004.
- 8 S. F. Duffy and E. H. Baker, "Size Effects in Ceramic Materials: Computational Issues Associated with Parameter Estimations," Technical Report ARL-CR-0560, US Army Research Laboratory, Aberdeen, MD, April, 2005.
- 9 J. J. Swab, A. A. Wereszczak, J. Rice, R. Caspe, R. H. Kraft, and J. W. Adams, "Mechanical and Thermal Properties of Advanced Ceramics for Gun Barrel Applications," Technical Report ARL-TR-3417, US Army Research Laboratory, Aberdeen, MD, February, 2005.
- 10 G. D. Quinn, "Ceramic Mechanical Property Test Method Development," pp. 133 142 in Heavy Vehicle Propulsion System Materials Program, Semiannual Progress Report for October 1998 through March 1999, Oak Ridge National Labs Technical Report TM-1999/95, June 1999.
- 11 Product literature data table, MS grade Mg-PSZ, www. cartech.com, Carpenter Advanced Ceramics April 2004.
- 12 G. D. Quinn, "Weibull Strength Scaling for Standardized Rectangular Flexure Specimens," J. Am. Ceram. Soc., 86 [3] (2003) pp. 508-510.

¹ ASTM C 1161-02 "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature," Annual Book of Standards, Vol. 15.01, ASTM, West Conshohocken, PA, (originally adopted in 1996, the current version is 2002).