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Letter to the Editor

Hoop tensile strength testing of small diameter ceramic particles

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Abstract

A method to measure hoop tensile strength of 1-mm-diameter brittle ceramic spheres was demonstrated through the use of a 'C-sphere' flexure strength specimen. This innovative specimen geometry was chosen because a simple, monotonically increasing uniaxial compressive force produces a hoop tensile stress at the C-sphere's outer surface that ultimately initiates fracture. This enables strength quantification and strength-limiting-flaw identification of the sphere itself. Such strength information is relevant to design optimization and durability assessments of ceramic fuel particles and breeder/multiplier pebbles for fusion when particle surfaces are subjected to tensile stresses during their manufacturing or service.

1. Introduction

Mechanical tests have been developed to measure the mechanical properties of microspheres having a diameter on the order of ~1 mm. These include techniques to measure crushing strength of fully dense brittle spheres [1]. However, if a ceramic or brittle material sphere is subjected to manufacturing or service conditions that cause tensile (i.e., hoop) stresses at its surface, then crushing tests will not reliably produce a valid strength measurement whose value could be used to predict the critical conditions that will initiate fracture of the sphere at its surface.

An innovative test method using a c-shaped (or slotted) coupon called the 'C-sphere' flexure strength specimen was recently developed by the authors to enable the study and measurement of strength and linked flaw size when a ceramic sphere's surface is subjected to hoop tension [2–4]. The C-sphere specimen is produced through the controlled slotting of a ceramic sphere. It is then diametrally loaded or flexed to initiate fracture at the sphere's surface or 'outer-fiber'. The C-sphere is analogous to the 'C-ring' flexure specimen [5], which is produced through the slotting of a ceramic ring. Because of the C-sphere's simple strength testing and geometry, it is experimentally easy to measure hoop strength as a function of temperature, loading rate, and environmental conditions.

Another advantage of the C-sphere flexure strength specimen is that it fractures at low to modest loads (i.e., excessive fragmentation is not caused) so that fractographical analysis can readily be performed and strength-limiting-flaw studies can ensue

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in a practical manner. These advantages significantly contrast with traditional crush testing of whole spheres. During such crush testing, diametrally loading fully dense ceramic sphere produces two Hertzian contacts, causes ring- and cone-crack initiation, and ultimate fracture if the contact stresses are sufficiently high. This fracture evolution makes the study of strength limitation from inherent flaws located at the surface nearly impossible because the high stress concentrations caused by the loading platens initiate the extrinsic fracture. In other words, the test fixturing, rather than flaws in the spheres, causes fracture. Additionally, the fracture of whole brittle spheres from diametral compression occurs at such high loads (i.e., a great deal of potential energy exists in the sphere at fracture) that the test coupon disintegrates into fine rubble [6] rendering fractography nearly impossible, and thus, inhibiting the study of the relationship of strength–flaw size. The above limitations of crush testing whole spheres are typical when large spheres are tested; however, crush testing of small (or microsphere) whole spheres produces exorbitantly high contact stresses such that the spheres, in effect, act as spherical indenters which indent or cause localized fracturing in the loading platens.

The strength–flaw size relationship in ceramic spheres can be readily examined using the C-sphere geometry, and the demonstrative testing of 1 mm diameter silicon nitride ceramic C-spheres was the focus of this study owing to its potential applicability to spherical nuclear fuel particles.

2. Experimental

The 1 mm diameter C-sphere specimen geometry is shown in Fig. 1 and its dimensions were determined using a C-sphere design optimization strategy described elsewhere for a 12.7 mm diameter C-sphere [2]. An important aspect of the geometry is the purposeful non-coincidal positioning of the sphere's center and the radius of curvature of the slot's innermost depth. The introduction of this offset acts to increase the outer-fiber tensile stress through the introduction of an applied bending moment; if it were not introduced, then too much tensile stress could develop on the ground surface of the notch and fracture would undesirably initiate there.

Silicon nitride spheres (NBD200, Saint-Gobain Ceramics, East Granby, CT) were machined using a specially made jig to hold the 1 mm spheres and

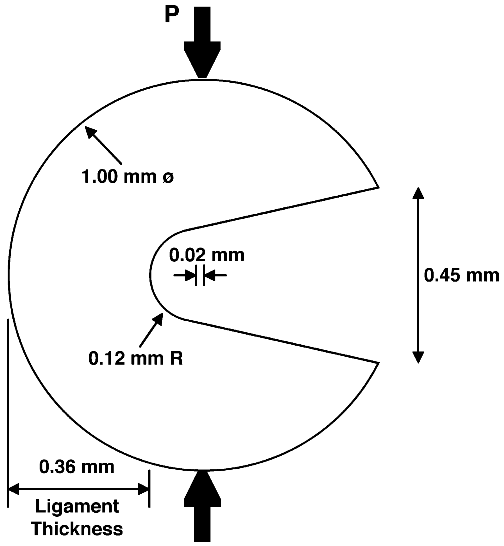

Fig. 1. Nominal dimensions of the 1-mm-diameter C-spheres that were used in the finite element analysis model and in subsequent strength calculations. They were diametrally loaded to fracture as represented by the thick arrows. The centers of the sphere and the 0.12 mm radius are not coincident. The latter is shifted to the left here, and that produces higher outer-fiber tensile stresses (i.e., increases the likelihood that fracture will initiate at the outer-fiber).

a metal-bonded 220-grit (ANSI) diamond-grinding wheel that had a width of 250–300 μm (Bomas Machine Specialties, Inc., Somerville, MA). The spheres were mounted in the jig in a line and then sliced at the same time producing C-spheres with the same slot and ligament dimensions.

After mesh optimization was performed using a one-quarter geometry model, finite element analysis (ANSYS, Canonsburg, PA) was used to determine that 1 N of compressive force produced a 1st principal maximum tensile stress (i.e., maximum hoop stress) of 13.045 MPa for the geometry shown in Fig. 1. This conversion was used in all the strength calculations. Examples of the produced stress field are shown in Fig. 2. High magnitudes of contact stresses were predicted in the FEA model, and though tensile stresses were present and localized in the nearby vicinity of the contact, they did not contribute to fracture of the C-spheres (unlike what occurs during the crushing of whole spheres).

A miniature mechanical test frame equipped with acoustic emission detection capability was used to diametrally compress the C-sphere specimens until they fractured. Specimen alignment was manually produced with the aid of high magnification

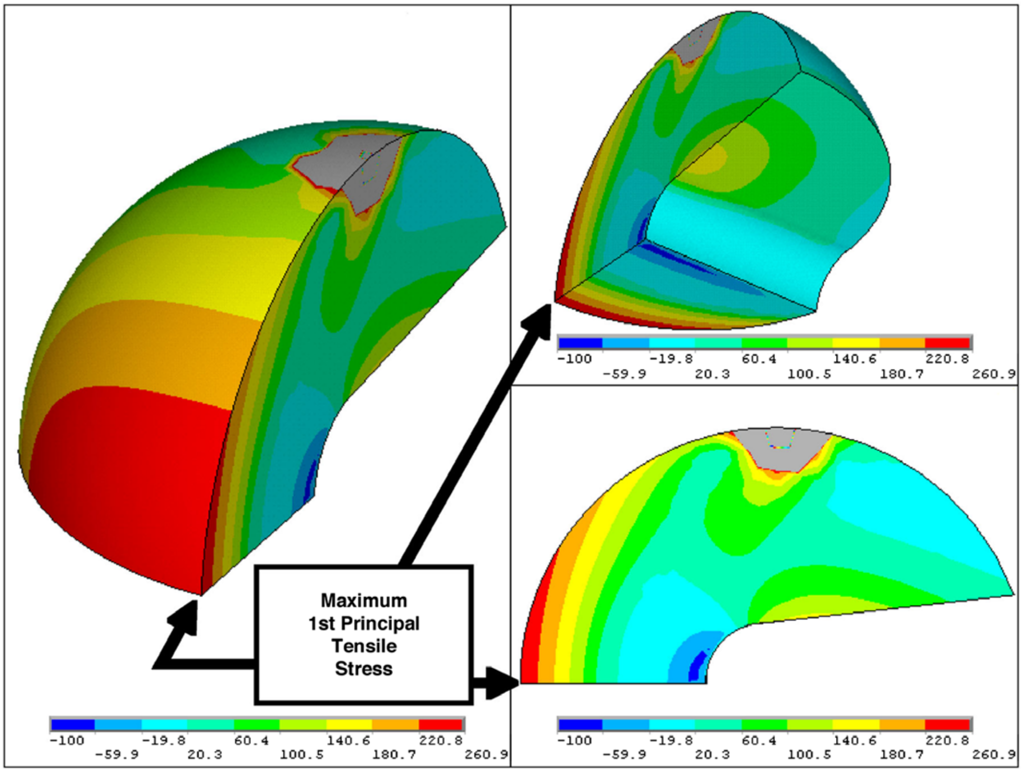

Fig. 2. First principal stress field in one-quarter C-sphere model. 20 N of applied compressive loading equated to a maximum tensile stress of 260.9 MPa. Fracture always initiated in the outer-fiber surface region shown here in red.

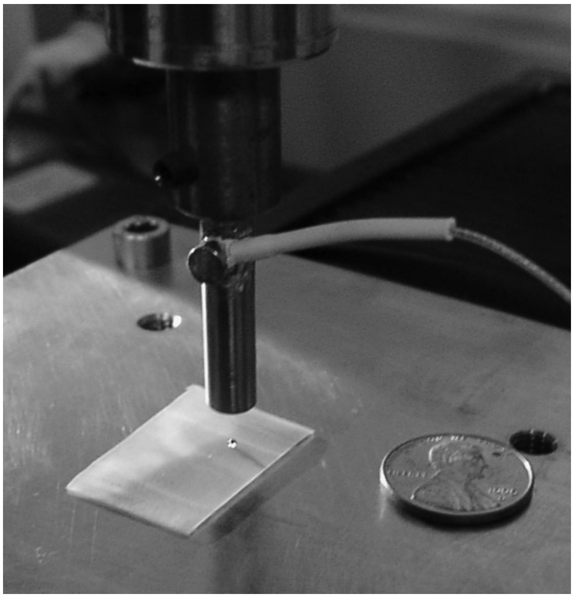

Fig. 3. Positioned and aligned C-sphere ready for compressive loading and strength testing. Load and acoustic emission signal were continuously monitored during testing. Fracture produced an acoustic event whose corresponding compressive load was used to calculate the hoop tensile strength.

imaging and its position was maintained by double-sided tape until compressive loading commenced. Because only failure load was sought, whereas displacement monitoring was not, and displacement controlled testing was used, the compliance added

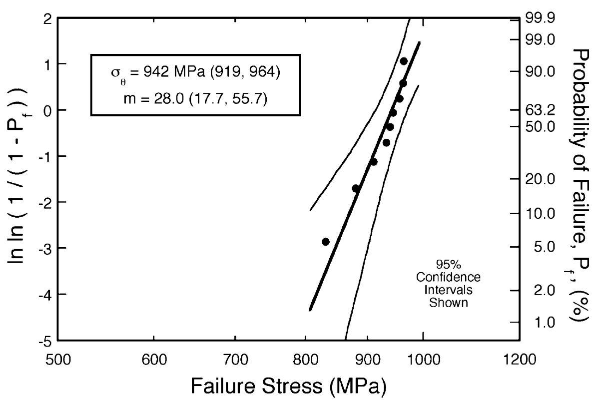

Fig. 4. Uncensored two-parameter Weibull hoop tensile strength distribution of 1-mm-diameter C-spheres. Values in parenthesis correspond to the ±95% confidence bands of the characteristic strength (σ_θ) and Weibull modulus (m).

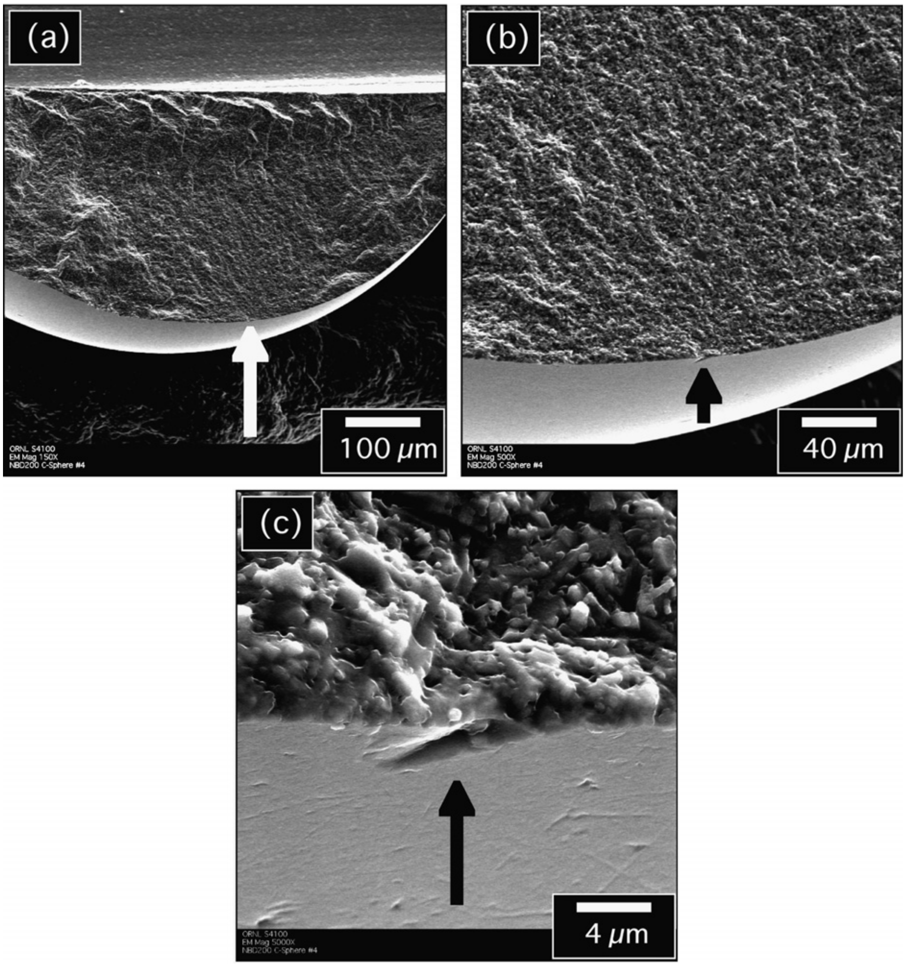

Fig. 5. Example of surface-located strength-limiting-flaw in a C-sphere specimen at (a) low, (b) medium, and (c) high SE-SEM magnifications. Shown specimen had a hoop tensile strength of 880 MPa.

by the tape did not affect the measured maximum load. Nine C-sphere specimens were fractured using a displacement rate of 1 μm/s using the test setup shown in Fig. 3. Fracture occurred within several seconds after loading commencement. Loading response was linearly elastic up to fracture (as evidenced by the constant slope of all the load–time curves and the fact that a constant displacement rate was used during testing) and an acoustic event were always detected at that moment. The failure load and the conversion ratio determined from the FEA were used to calculate the hoop tensile stress. Specimen response was expected to be linear elastic up to the fracture event and it was. Strengths were fitted to a two-parameter Weibull distribution using maximum likelihood estimation using commercial statistical software (WeibPar, Connecticut Reserve Technologies, Cleveland, OH).

3. Strength and fracture

There is a relatively large area subjected to a high 1st principal tensile stress as shown in Fig. 2, and fracture always desirably initiated in this region in all nine C-sphere specimens. An uncensored characteristic strength of 942 MPa and Weibull modulus (m) of 28.0 was determined and the fitted distribution and data are shown in Fig. 4. Effective area[1] was determined as a function of Weibull modulus using CARES/Life (NASA Glenn Research Laboratory, Cleveland, OH) and was calculated to be 0.1084 mm^2 for $m = 28.0$. This information could

[1] 'Effective area' is the area of the specimen that is 'effectively' subjected to uniform tension, and it is a function of Weibull modulus.

be used to predict the reliability and strength–size-scale to other geometries of NBD200 silicon nitride, provided the same strength-limiting-flaw type was operative in both those scaled sizes.

At least two surface-located flaw types were responsible for fracture initiation at the surface, which occurred in all specimens as a consequence of the high hoop tensile stress produced. An example of one strength-limiting surface flaw type, a microstructurally small scratch, is shown in Fig. 5. Surface-located agglomerates containing glassy regions also were strength-limiters in other C-sphere specimens.

Such strength information is relevant to brittle material design optimization and durability assessments of ceramic fuel particles and breeder/multiplier pebbles for fusion whose surfaces are subjected to tensile stresses caused by thermal gradients, thermal transients, swelling, etc., during their manufacturing or service. If the service tensile stresses located at the surface are too high for any arbitrary ceramic sphere, then the user or manufacturer needs to reduce those service stresses, modify the surface quality of the sphere so that its hoop tensile strength is higher valued, or both.

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