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Stress Heterogeneity Effect on the Strength of Silicon Nitride

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The experiments reported in this paper demonstrate the causes of the failure of monolithic ceramics. The specimens are made of silicon nitride and tested at room temperature. The stress field within the specimen is different for each of four series of tests that have been conducted. Fractographic observations have also been made to identify the causes of the failures. A size effect analysis is performed. [Key words: silicon nitride, failure, defects, statistics, stress.]

I. Introduction

A LARGE scatter of failure stress is a common feature of all brittle materials (e.g., engineering ceramics, concrete, and glass). For nominally identical specimens, this scatter may be as high as 100% of the mean failure stress. Both the rupture and the scatter are due to initial defects within the material. These defects are either intrinsic (porosities, inclusions) and induced by the manufacturing process of the ceramic (sintering, pressing) or extrinsic and created by grinding of the surface of the specimen.

In this paper, the results of four series of tests, which have been conducted at room temperature on specimens made of silicon nitride, are reported. The stress field within the specimen is different for each of the four series of tests. Fractographic observations have also been made to characterize the causes of the failures. A size effect analysis is performed to study the correlation between mean strength and effective volume.

II. Experimental Study of the Failure of Silicon Nitride

(1) Tensile Tests

The advantage of the tensile test is the homogeneity of the macroscopic stress field within the specimen. Whereas this test is very easy to perform on ductile materials, it is much more complicated for brittle materials. This is because the failure strains of brittle materials are very small (typically 0.1%). Therefore, if no particular caution is taken, spurious bending strains induced by the testing grips can easily reach values of the order of magnitude of the tensile strains.

To avoid such problems, a new testing system has been designed. It is composed of four main parts (see Fig. 1). The role of the elastic joints (4) is to avoid bending strains while allowing compressive loadings. To obtain an equivalent Cardan joint, the elastic joints are constituted of thin elastic plates in two orthogonal directions. Because the specimen shape is axisymmetric, the grips (2) are entirely axisymmetric-like. A cooling device is provided for elevated temperature testing. Two half-shells (3) maintain the specimen in the grip and evenly distribute the loading onto the cone-shaped surfaces of the specimen. To avoid stress concentration, a smooth axisymmetrical specimen (1) without singularities is designed. It is composed of a cylindrical testing volume (1000 mm^3), cone-shaped heads, and torelike connecting parts. Some specimens have been tested with four strain gauges on the central section to verify the stress alignment: the maximal difference between the four gauges is lower than 1.3×10^{-4} (i.e., 7.4% of the tensile strain), and we conclude that the bending strains are negligible (about 11%) with respect to the tensile strains.

Eighteen specimens were tested in tension. They were made of isostatically pressed silicon nitride (SN 220M, Kyocera, Kyoto, Japan; density of 3200 kg/m^3) and machined from rods (18 mm in diameter). The value of the effective roughness (R_a) of the specimens was less than 0.6 μm.

First, the silicon nitride behavior appeared as purely linear elastic-brittle. Second, the failure stress varied within the range 374 to 635 MPa with a mean value of 526 MPa (see Fig. 2).

These experimental results can be fitted (with least-squares) to a two-parameter Weibull model (without threshold stress[1]) with a value of the Weibull modulus (m) of 8.0 (the effective volume is 1000 mm^3). The initiation sites observed on the fracture surfaces of the specimens are defects of 20 to 250 μm (see Figs. 3(a) and (b)). These results underline the direct relationship between the initiation defect size and the failure stress level: the smaller the defect size, the higher the failure stress.

In 17 of 18 failures, the initiation defect is located within the volume of the specimen (see Fig. 4) and not at the surface. This seems to be an a posteriori proof of the quality of the experimental setup that has been designed for these tests: the surface flaws do not play any leading role because of the homogeneity of the stress field that is induced by this setup. Moreover, because of this homogeneity, it is probable that the

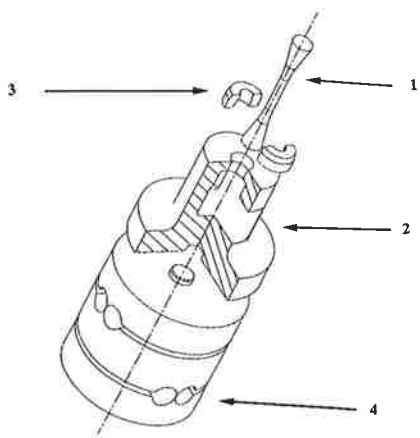


Fig. 1. Gripping system for tensile tests: (1) specimen, (2) grip, (3) half-shells, (4) elastic joint.

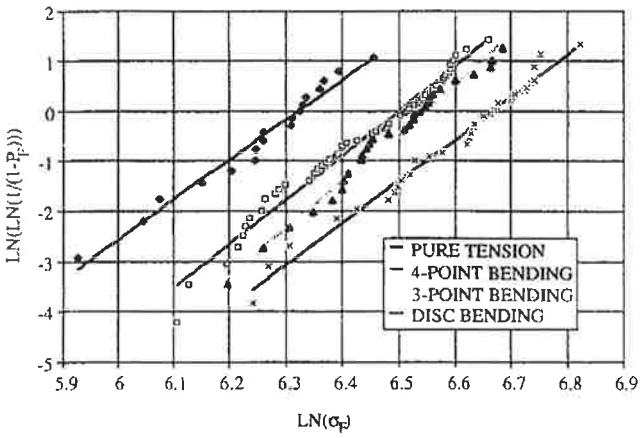


Fig. 2. Experimental database for the rupture of silicon nitride specimens subjected to different loading patterns ((◆) tensile tests, (□) four-point bend tests, (▲) three-point bend tests, (×) disk bend tests (corresponding Weibull correlations are shown)).

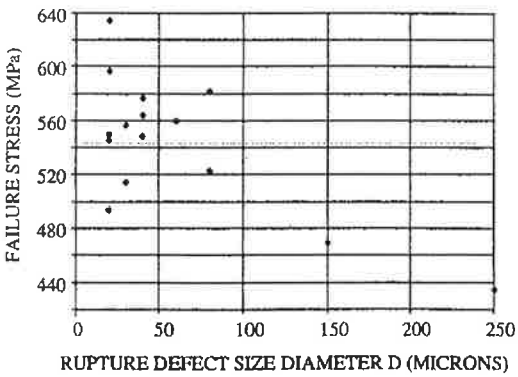
initiation defect observed is, whatever its size, the largest flaw, i.e., the weakest link[2,3] within the specimen.

(2) Four-Point Bend Tests

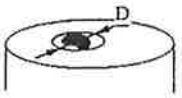
One hundred machined rectangular-type specimens (35 mm × 4 mm × 3 mm) were subjected to four-point bending. They were made of isostatically pressed SN 220M and the R_a value was less than 1 μm. The load and support spans were 12.5 and 24 mm, respectively.

The load–displacement curve has the same linear aspect as the curve obtained in pure tension. The failure stress (defined as the maximum stress over the structure and calculated by beam theory) lies between 448 and 780 MPa (see Fig. 2). The value of m (using a least-squares method) is 8.9.

Compared to the pure tension case, the size of the initiation defects is on an average smaller[4] (15 to 110 μm), and the mean failure stress is higher (629 MPa instead of 526 MPa). Also, note that the initiation sites are located near the surface of the specimen that is subjected to tensile stresses. These observations lead to two conclusions that are valid for this type of loading. First, the highest tensile stress levels play a leading role in the failure process. Second, the initiation defects located near the surface in tension are not in general the largest flaws within the material.



(a)



(b)

Fig. 3. (a) Correlation between the failure stress and the failure defect size in pure tension (silicon nitride). (b) Definition of the defect size.

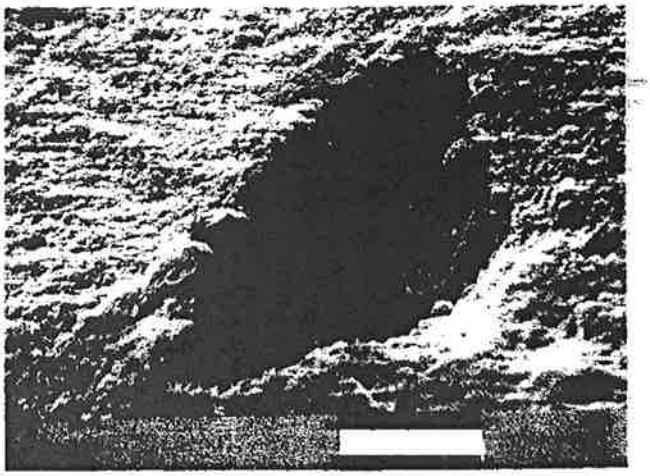


Fig. 4. Fractographic observations for tests in pure tension (silicon nitride) (bar = 100 μm).

(3) Three-Point Bend Tests

Fifty machined rectangle-type specimens (70 mm × 4 mm × 3 mm) were subjected to three-point bending. The material and the R_a value were the same as for the four-point-bending specimens. The support span was 60 mm.

The load–displacement curve has the same linear aspect as the curve obtained in pure tension. The failure stress (defined as the maximum stress over the structure and calculated by beam theory) lies between 491 and 801 MPa (see Fig. 2). The value of m (using a least-squares method) is 9.6.

Compared to the four-point bend case, the size of the initiation defects is on an average similar,[4] and the mean failure stress is higher (660 MPa instead of 629 MPa).

(4) Disk Bend Tests

To understand the behavior of silicon nitride when subjected to a multiaxial stress field, a set of 50 disks were subjected to punching. The specimens were disks 30 mm in diameter and 3 mm in thickness. They were made of uniaxially pressed SN 220M and the R_a value, after machining, was less than 1 μm. The disk bend-testing setup consisted of loading and supporting rings 2.5 mm in diameter and 24 mm in diameter, respectively.

The failure stress, defined as the maximum principal stress over the structure and derived from a finite element analysis, lies between 513 and 918 MPa (see Fig. 2). The value of m (using a least-squares method) is 8.4.

Compared to the three-point bend case, the mean failure stress is higher (721 MPa instead of 660 MPa).

III. Effective Volume Analysis

Using the results of Davies,[5] an effective volume analysis can be preformed to correlate mean strength and effective volume. A Weibull model leads to a linear correlation between the two quantities in a ln–ln plot with a slope of $-1/m$.

The effective volume in disk bending is computed by considering the maximum tensile stress at each integration point.

The correlation between mean strength and effective volume (see Fig. 5) leads to a slope of -0.04 ($-1/25$) with a coefficient of correlation (ρ^2) of 0.995. The value of the slope does not correspond to the experimental Weibull parameter.

Contrary to Katamaya and Hattori,[6] for this set of experiments, a correlation in terms of effective volume is not satisfactory. Also, a correlation in terms of effective surface is not satisfactory since it leads to a slope of $-1/17$ with $\rho^2 = 0.983$.

IV. Conclusions

A series of tensile tests has been conducted on silicon nitride specimens. The mean strength in tension is less than the

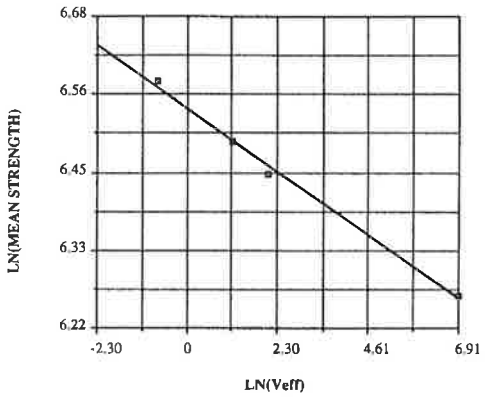


Fig. 5. Correlation between mean strength and effective volume.

mean strength in four-point bending, three-point bending, and disk bending. The Weibull parameter in tension is of the same order as in the different types of bending.

A correlation between the size of the defect leading to rupture is given. Seventeen of eighteen failures initiated in the volume of the specimen.

The size effect analysis performed for the present results leads to a good correlation in a ln–ln plot. However, the slope does not correspond to the experimentally determined Weibull parameter.

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