JOURNAL OF MATERIALS SCIENCE 30 (1995) 395–398

# Mixed-mode high temperature toughness of silicon nitride

P. KHANDELWAL
*Allison Turbine Operations, Indianapolis, Indiana, 46206, USA*
B. S. MAJUMDAR
*Universal Energy Systems, Dayton, Ohio, USA*
A. R. ROSENFIELD
*Rosenfield and Rosenfield, Columbus, Ohio, 43212, USA*

Both opening-mode and mixed-mode fracture toughness tests were carried out at 1200 and 1300 °C on a sinter/HIP grade of silicon nitride. Data for pure opening loading ($K_{Ic}$) agree well with other experiments on the same material, which showed that the toughness was lower at 1000 °C than at room temperature, but increased as temperature increased above 1000 °C. The ratio of $K_{IIc}/K_{Ic}$ was sufficiently insensitive to temperature that it can be considered to be constant. Results are discussed in the context of mechanisms that have been proposed to explain fracture toughness in silicon nitride.

## 1. Introduction

Statistical descriptions of the size and loading-pattern dependence of ceramic strength require knowledge of the orientation dependence of fracture toughness [1]. This requirement arises because there is a finite probability that the critical flaw will be oriented at an angle to the maximum principal stress. As a result, the flaw will be subjected to a combination of opening and shear loadings (Mode I and Mode II, in fracture mechanics terminology).

The objective of this paper is to report the effect of shear loadings on the value of fracture toughness at high temperature for silicon nitride. In contrast, all previous high-temperature toughness data have been obtained using bend specimens and all such data are values for the opening-mode toughness $K_{Ic}$. As part of the research, we have compared the opening-mode toughness values obtained at two different laboratories using two different specimen designs.

The choice of silicon nitride was based on its potential for reducing energy requirements of advanced engines by allowing higher inlet temperatures, which optimize fuel consumption while increasing performance. The specific ceramic was a hot-isostatically pressed commercial grade (PY6 Silicon Nitride, GTE Laboratories, Inc., Waltham MA, USA), containing six percent yttria as a sintering aid. The microstructure [2] typically consisted of one to six micrometre-long acicular grains having diameters of 0.1 to 1.0 μm. The grains were predominantly $\beta$-$Si_3N_4$, separated by partially-crystalline yttrium disilicate grain boundaries.

## 2. Experimental procedures

### 2.1. Opening-mode experiments

Opening-mode toughness was measured in ambient air up to 1400 °C using chevron-notched bend bars. Procedures and results are described elsewhere [3].

### 2.2. Mixed-mode experiments

Mixed-mode fracture toughness measurements were performed to evaluate both opening-mode and longitudinal-shear-mode fracture toughness. Two techniques were used; one technique employed bend bars containing cracked indentations which were formed using a Vickers Hardness tester. The diagonal of each indentation was made at a predetermined angle to the long axis of the bend bar and the bar was loaded to failure at a slow rate in a specially-designed four-point-bend fixture which minimizes experimental errors [4]. Failure originated at the indentations on all specimens, with most cracks running either along the indentation diagonal or very close to the diagonal. Chantikul *et al.*'s equation for strength of an indented specimen [5] was used to calculate toughness.

The diametrally-compressed disc, whose room-temperature application was reviewed recently [6], was used to obtain more detail on the relation between opening-mode and shear-mode fracture toughness (the mixed-mode failure envelope). This test involves compression loading of a thin cylindrical specimen containing a central notch along a diameter. This configuration is inexpensive and simple to use and has the virtue that it can be employed to obtain fracture toughness in tension, in shear, and in any combination of these two modes. At room temperature notches were oriented at several different angles to the compression axis to obtain a complete failure envelope. It has been shown [6] that the tension, or opening-mode toughness, ($K_{Ic}$) measured with this specimen is the same as that measured using alternate procedures and that the shear toughness ($K_{IIc}$) varies from one to two times $K_{Ic}$, depending on the roughness of the opposing crack faces.

To obtain the high-temperature failure envelope, the same notched diametral-compression procedures were used as for room temperature, except for fixturing. The specimens were heated by convection and radiation using a Glowbar tube as a susceptor, an arrangement that allowed testing up to 1300 °C. The loading platens were made from silicon carbide. The specimen was placed in the fixture with the notch at either a 0 or 23° angle to obtain either pure opening or pure shear loading, and a slight load was placed on it to maintain the angle during both heating and temperature stabilization, prior to loading to failure. In some cases the specimen was held at load for a period, unloaded, and examined microscopically for crack growth. However, no stable crack growth was detected in any of these cases.

Mixed-mode behaviour was characterized in two ways. Toughness results were fitted to the equation [7]

$$K_{iq}/K_{Ic} + (K_{iiq}/cK_{Ic})^2 = 1 \qquad (1)$$

where $K_{iq}$ and $K_{iiq}$ are the opening and shear fracture toughness components in mixed-mode loading, while $c$ is the ratio of stress intensity at failure in pure shear ($K_{IIc}$) to pure-opening-loading toughness ($K_{Ic}$).

An alternative technique for presentation of mixed-mode fracture-toughness data, which has received attention recently [8], involves relating strain energy release rate ($G$) to a quantity called mixity ($\psi$), where:

$$G/G_{Ic} = (K_{iq}^2 + K_{iiq}^2)/EK_{Ic}^2 \qquad (2)$$

and mixity is given by

$$\psi = \tan^{-1}(K_{iiq}/K_{iq}) \qquad (3)$$

One possible relation between these two quantities is

$$G/G_{Ic} = \sec^2(\psi/\psi_0) \qquad (4)$$

Evaluation of either $K_{Ic}$ and $c$ or of $G_{Ic}$ and $\psi_0$ is sufficient to generate a mixed-mode failure envelope.

## 3. Results

Fig. 1 illustrates the failure envelopes generated at room temperature using the disc specimen. This figure shows that the data do obey Equations 1 and 4. Since the high-temperature data, discussed below, were obtained only at the end points of Fig. 1a, it is assumed that the forms of these equations are temperature independent.

Table I reports the temperature dependence of mean fracture toughness. The standard deviations were 15%, or less, of the means for both uniaxial and biaxial tests. Diametral compression values of $c$ and $K_{IIc}$ were obtained from Equation 1 while $\Psi_0$ was calculated from Equations 2 to 4. The value of $c$ for the surface-cracked bend bars was obtained using the analysis of Chao and Shetty [1].

The data in Table I are believed to be reasonable since the room temperature $K_{Ic}$ values obtained using different specimens are quite similar, while the value of $c = K_{IIc}/K_{Ic}$ is in the mid-range of brittle material values [6]. Similarly, $G_{Ic} = 128\ \mathrm{J\,m^{-2}}$, while $\psi_0$ is found to be 1.78, which is within the range reported for other monolithic ceramics [8] assuming typical values: $E = 300$ GPa, $\nu = 0.24$.

Fig. 2 compares all of the $K_{Ic}$ data and shows that the room temperature values are greater than most of the high temperature data. A dashed line has been drawn through the high-temperature points as a preliminary toughness equation, based on a curve fit which included the room temperature data

$$K_{Ic} = 6.59 - T/126 + (T/422)^2 \qquad (5)$$

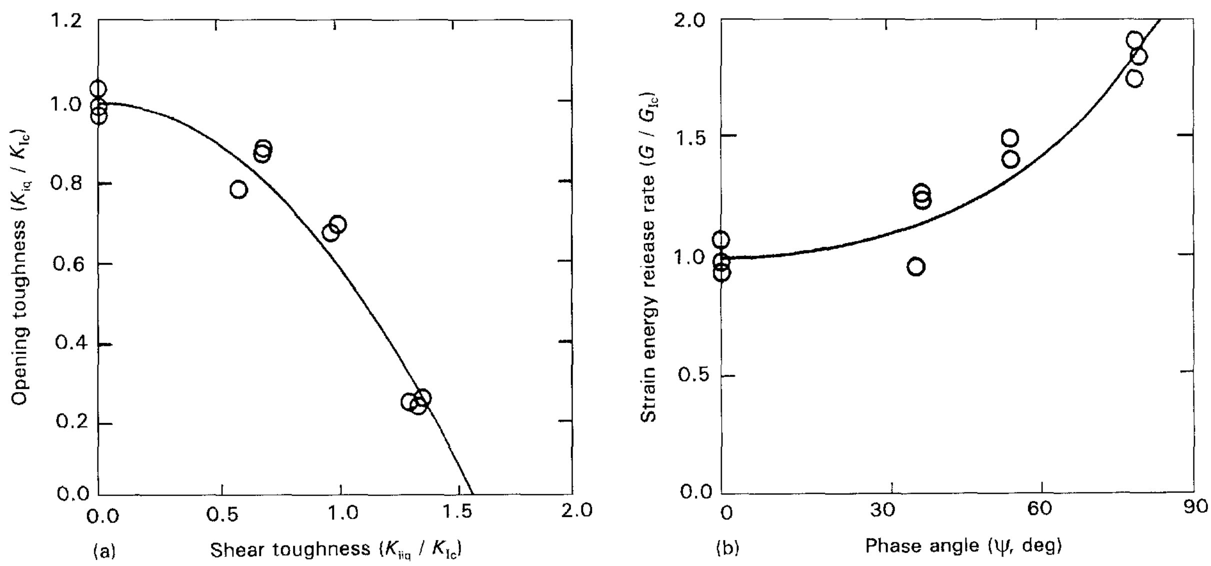


*Figure 1* Mixed-mode fracture toughness of sinter/HIP silicon nitride. (a) Fracture toughness. (b) Strain energy release rate.

TABLE I Opening and shear toughness

| Temperature, °C | $K_{Ic}$, $MPa\,m^{1/2}$ | $K_{IIc}$, $MPa\,m^{1/2}$ | $c = K_{IIc}/K_{Ic}$ | $G_{Ic}$, $J\,m^{-2}$ | $\psi_0$ |
|---|---|---|---|---|---|
| 20[a] | 6.7 | 9.5 | 1.42 | 140 | 2.43 |
| 20[b] | 6.4 | n.a. | n.a. | 128 | n.a. |
| 20[c] | 6.4 | 10.1 | 1.57 | 128 | 1.78 |
| 1000[b] | 4.6 | n.a. | n.a. | 66 | n.a. |
| 1200[b] | 4.5 | n.a. | n.a. | 63 | n.a. |
| 1200[c] | 5.0 | 6.5 | 1.31 | 78 | 2.27 |
| 1300[b] | 6.5 | n.a. | n.a. | 132 | n.a. |
| 1300[c] | 5.4 | 8.0 | 1.47 | 91 | 1.89 |
| 1400[b] | 6.5 | n.a. | n.a. | 132 | n.a. |

[a] Bend bar containing surface crack
[b] Chevron-notched bend bar; n.a. = not applicable
[c] Diametral compression

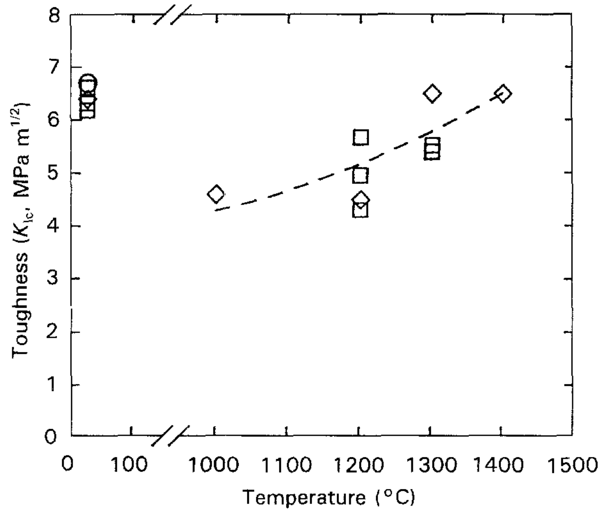


*Figure 2* Mode-I fast fracture of sinter/HIP silicon nitride. ◇, uniaxial through crack, Allison (mean); ○, uniaxial surface crack, Battelle (mean); □, biaxial through crack, Battelle.

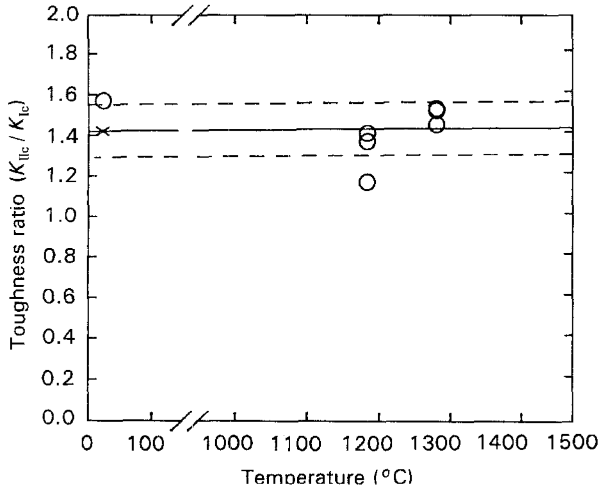


*Figure 3* Shear-mode/opening-mode fracture toughness ratio. ×, uniaxial bending, surface cracks; ○, diametral compression, through cracks.

where $K_{Ic}$ is expressed in $MPa\,m^{1/2}$ and $T$ in degrees C. The standard deviation of the $K_{Ic}$ residuals from Equation 5 is $0.45\,MPa\,m^{1/2}$. This temperature dependence, incorporating a toughness minimum, is in qualitative agreement with the data of Salem *et al.* [9] and of Yeh and Feng [10].

Fig. 3 reports the effect of temperature on the ratio $c = K_{IIc}/K_{Ic}$. The solid line is an averaged value of 1.42, while the dashed lines represent an estimated standard deviation of 0.13. In contrast, Tsuruta *et al.* [11] reported that $c = 1.1$ using asymmetric bending on a sintered, but not HIP'ed, grade of silicon nitride. Whether the difference from the present results is due to the test technique or to the material is not clear. However, material-dependent effects on $c$ can be inferred from a later paper [12]. It is seen in Fig. 2 that $c$ is lower at 1200 °C than at either room temperature or at 1300 °C. However, for simplicity it is recommended that an average temperature-independent value, $c = 1.42$, be employed for modelling, since any error in predicted failure load will be small because failure strength in mixed-mode loading is fairly insensitive to the choice of the value of the $c$ parameter [12].

## 4. Discussion

As noted above, the minimum in the fracture-toughness versus temperature curve has been reported before. The explanation given by Wereszczak *et al.* [3] invokes two mechanisms coming into play as temperature increases. The toughness decrease from room temperature to about 1200 °C is believed to be associated with softening of the amorphous grain boundary phase, leading to a macroscopically-brittle material containing easy fracture paths. As the temperature increases above 1200 °C, the silicon nitride starts to soften, leading to the usual increase in toughness associated with increased ductility. Oxidization of the silicon nitride also occurs at 1300 and 1400 °C, but its influence on toughness is unclear.

The temperature dependence of $K_{IIc}$ is similar to that of $K_{Ic}$, since Fig. 3 suggests that the $c$ ratio is fairly insensitive to temperature. This result implies that shear loadings have the same qualitative effect on toughness as tensile loadings, which is not unexpected for an elastic material, since shear loadings induce an asymmetric tensile stress field at the crack tip whose magnitude is comparable to the opening-mode stress field. These shear-induced tensile stresses also are the cause of the apparent decrease in opening-mode fracture toughness with an increasing shear component of loading.

Two other mechanisms affecting toughness should be mentioned: rubbing and bridging. Both effects decrease the crack-tip stress intensity. Rubbing occurs when the crack-opening displacement is smaller than the asperities on the opposing crack faces [13]. This effect comes into play at low Mode-I/Mode-II ratios and causes $K_{iiq}$ values associated with low $K_{iq}$ levels to be higher than they would be in the absence of rubbing. If rubbing were absent, the $K_{IIc}/K_{Ic}$ ratio would be closer to unity than the values reported here. The occurrence of bridging at 1000 °C, and above, has been suggested by Wereszczak *et al.* [3] to account for rising $R$ curves. This mechanism occurs when the crack bypasses some grains, leaving behind an island of unbroken material, which restrains crack opening and further crack advance. Like rubbing, this mechanism reduces the stress intensity and increases the apparent toughness.

## 5. Conclusions

1. The $K_{Ic}$ fracture toughness of silicon nitride is lower at 1000 °C than at room temperature, but increases as temperature is raised above 1000 °C. This behaviour is associated with successive softening of the grain boundary phase and the silicon nitride grains.

2. The ratio $K_{IIc}/K_{Ic}$ is sufficiently insensitive to temperature that it can be considered to be constant for practical purposes over the temperature range investigated.

## Acknowledgements

The authors are grateful to G. T. Wall and P. R. Held of Battelle for experimental assistance. This research was sponsored by the DOE Ceramic Technology Office under Contract No. DE-AC05-84OR21400 to Allison from Martin Marietta Energy Systems.

## References

1. L-Y. CHAO and D. K. SHETTY, *J. Amer. Ceram. Soc.* **74** (1991) 333.
2. M. K. FERBER and M. G. JENKINS, *J. Amer. Ceram. Soc.* **75** (1992) 2453.
3. A. A. WERESZCZAK, M. K. FERBER, R. R. SANDERS, M. G. JENKINS and P. KHANDELWAL, "Fracture Toughness ($K_{Ic}$ & $\gamma_{wof}$) of a HIPed $Si_3N_4$ at Elevated Temperatures", in Proceedings of 17th Annual Conference and Exposition on Composites and Advanced Ceramic Materials, Cocoa Beach FL, USA, January (1993).
4. R. G. HOAGLAND, C. W. MARSCHALL and W. H. DUCKWORTH, *J. Amer. Ceram. Soc.* **58** (1976) 189.
5. P. CHANTIKUL *et al.*, *J. Amer. Ceram. Soc.* **64** (1981) 539.
6. A. R. ROSENFIELD and B. S. MAJUMDAR, *ASTM STP 1172* (1992) 63.
7. A. R. ROSENFIELD, "Mixed-Mode Fracture of Steel and Ceramics" in "Fracture Behaviour and Design of Materials and Structures", edited by D. Firrao (EMAS, Warley, UK 1990) pp. 5091.
8. J. AHMAD, "Combined Mode I and Mode II Fracture Toughness of Brittle Materials and Interfaces", submitted to *J. Eng. Mater. Tech.*
9. J. A. SALEM, S. R. CHOI, M. R. FREEDMAN and M. G. JENKINS, *J. Mater. Sci.* **27** (1992) 4421.
10. H. C. YEH and H. T. FENG, "Improved Silicon Nitride for Advanced Heat Engines", NASA Contractor Report 182193, Allied-Signal Aerospace Co., April, 1991.
11. H. TSURUTA, N. YAMADA, Y. NAKASUJI, M. MASUDA and M. MATSUI, *J. Ceram. Soc. Jpn. Internat. Ed.* **99** (1991) 558.
12. Y. NAKASUJI, N. YAMADA, H. TSURUTA, M. MASUDA and M. MATSUI, in "Fracture Mechanics of Ceramics", Vol. 10, edited by R. C. Bradt *et al.* (Plenum, New York, 1992).
13. A. R. ROSENFIELD, *Inter. J. Fracture* **49** (1991) R47.

*Received 30 September 1993*
*and accepted 6 July 1994*