Letters

Prediction of fracture-surface energy from microhardness indentation in structural ceramics

Recent studies [1, 2] have shown that Knoop-microhardness indentation produces nearly semicircular edge microcracks having highly reproducible dimensions in hot-pressed Si_3N_4 and hot-pressed SiC. These microcracks constitute the "worst flaws" and initiate catastrophic fracture in specimens subjected to tensile stress. Through fracture-mechanics analysis, the fracture-toughness value, K_{IC}, has been computed from values of crack dimensions and fracture stress [1, 2]. The present investigation was undertaken to determine the feasibility of predicting approximate values of fracture-surface energy, γ, for hot-pressed structural ceramics Si_3N_4 and SiC from microhardness loads and corresponding microcrack dimensions without determining the fracture stresses.

The possibility of predicting γ from microhardness data is based upon a recent study by Lawn and Swain [3] who calculated the extent of propagation of microcracks beneath point indentation in brittle solids. Their approach—with slight modification—has been utilized in the present investigation.

A Knoop diamond indentation was made on the 600-grit polished surface of bend bars of HS–130 Si_3N_4 and NC–203 SiC (Norton Co, Worcester, MA, USA). The indentor load was varied from 0.6 to 3.1 kg in increments of 0.5 kg in order to obtain a range of indentation dimensions. These dimensions were measured by light microscopy, and the bars were then fractured in four-point bending. The fracture in each bar was initiated by the microcrack produced beneath the indentation. The dimensions of the microcracks were measured from the fracture surfaces of the bars by light microscopy.

The relevant dimensions of the hardness impression and the microcrack are presented schematically in Fig. 1. L and $2c$ are the lengths of the long diagonal of the Knoop impression and of the

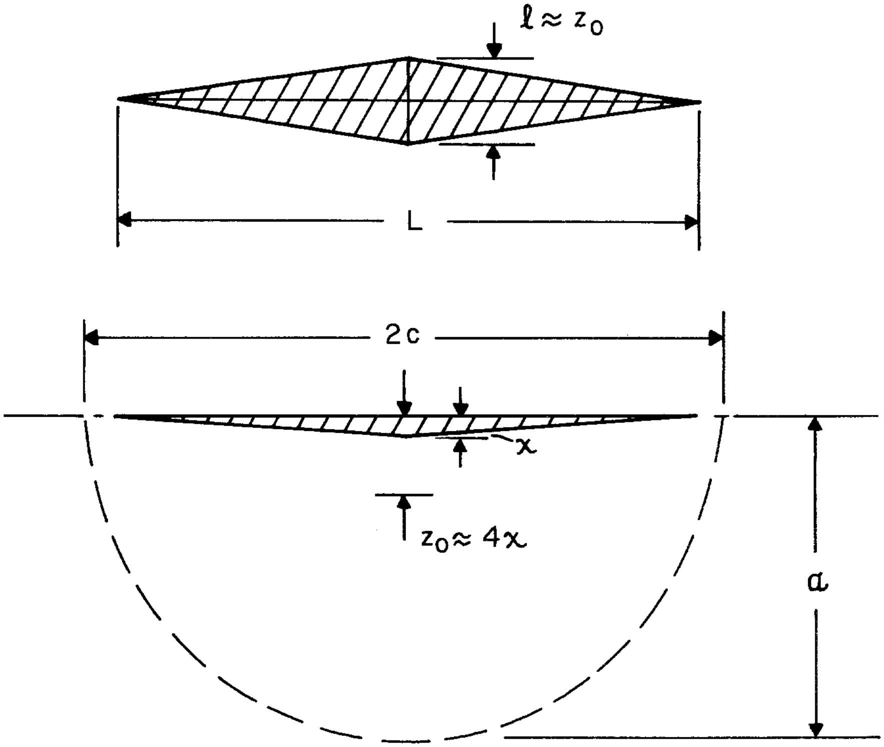


Figure 1 Schematic diagram showing the Knoop-impression and the microcrack dimensions.

microcrack at the surface of the bend bar, respectively; x and a are the depths of the indentation and of the microcrack, respectively, l is the length of the short diagonal of the Knoop impression and z_0 represents the depth of the indentation-induced deformation zone in which no tensile-stress component is present [3]. The significance of the parameter z_0 will be described later in this paper. The measured values of a, $2c$, L, and x in Si_3N_4 and SiC as a function of the indentor load are presented in Table I. The a and $2c$ dimensions for both Si_3N_4 and SiC indicate that for the indentor loads employed, the microcracks are nearly semicircular.

TABLE I Measured dimensions of microhardness impressions and microcracks in Si_3N_4 and SiC

Indentor load P (kg)	a (mm)	$2c$ (mm)	L (mm)	x (mm)
HS-130 Si_3N_4				
0.6	0.030 5	0.063 4	0.067 5	0.002 2
1.1	0.040 5	0.096	0.106	0.003 48
1.6	0.049 4	0.012 0	0.120 5	0.003 95
2.1	0.064	0.140	0.139 8	0.004 58
2.6	0.070	0.170	0.157 8	0.005 17
3.1	0.080	0.188	0.165	0.005 41
NC-203 SiC				
0.6	0.033 7	0.077 1	0.060	0.001 97
1.1	0.042 2	0.096 4	0.091 1	0.002 99
1.6	0.065 1	0.133 7	0.103 6	0.003 4
2.1	0.083 1	0.160 2	0.128 2	0.004 21
2.6	0.094	0.188	0.132 5	0.004 35
3.1	0.104 8	0.226 5	0.153	0.005 02

The fracture-mechanics approach utilized by Lawn and Swain [3] consisted of obtaining an expression for the stress-intensity factor K_I for a penny-shaped crack in terms of the inhomogeneous elastic-stress distribution beneath the indentor. They first obtained an expression for K_I for a straight-fronted edge crack and then multiplied this expression by $\sqrt{2/\pi}$ to obtain an approximate K_I value in the case of the penny-shaped crack. Following an analysis by Smith [4], the multiplying factor necessary to convert the K_I expression from the straight-fronted edge crack to a semi-elliptical edge crack (with ratio $a/2c \rightarrow 0.5$ approximating the semi-circular cracks produced in Si_3N_4 and SiC) is $\sqrt{(1/Q)}$, where

$$\sqrt{Q} \simeq \Phi = \int_0^{\pi/2} \left[\sin^2\theta + \left(\frac{a}{c}\right)^2 \cos^2\theta\right]^{1/2} d\theta \qquad (1)$$

for brittle solids. The value of Φ can be calculated readily for a given a/c ratio. The approximate value of K_I for nearly semicircular cracks is then found to be

$$K_I = \left(\frac{1-2\nu}{2}\right) \frac{P}{\pi^{3/2} z_0 a^{1/2} Q^{1/2}} \qquad (2)$$

where P is the indentor load and ν is Poisson's ratio.

The strain energy release rate for the plain-strain condition is given by

$$G = \frac{(1-\nu^2) K_I^2}{E} \qquad (3)$$

where E = Young's modulus. Stable crack growth will occur when

$$G = 2\gamma \qquad (4)$$

where γ = fracture-surface energy per unit area. Combining Equations 2, 3 and 4, the following expression for γ is obtained:

$$\gamma = \frac{(1-\nu^2)(1-2\nu)^2}{8E\pi^3} \frac{P^2}{a z_0^2 Q}$$

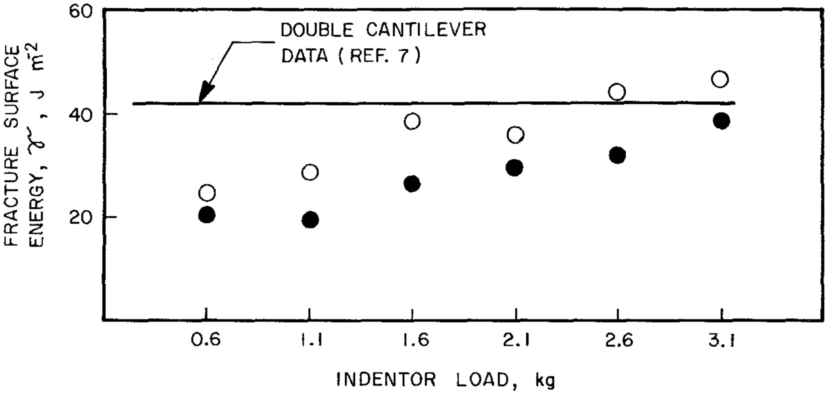
From direct observation by light microscopy of the deformation zone in quartz, Lawn and Swain found z_0 to be equal to the short diagonal length of the Knoop impression. For Si_3N_4 and SiC it was not possible to observe the deformation zone directly; however, an indirect method was employed to obtain values of z_0.

As mentioned previously, the indentation-induced microcrack causes catastrophic failure upon application of a tensile stress normal to it, thereby permitting experimental determination of K_{IC} values. However, it has been shown [1] that for HS–130 Si_3N_4, the K_{IC} values obtained by this method were lower than those obtained from the double-torsion method [5]. This discrepancy has been attributed to the presence of a residual compressive stress immediately beneath the indentation and a residual tensile stress near the microcrack tip; the latter was thought to be responsible for the lower fracture stress and consequently lower K_{IC} value [1]. In a parallel study to be published elsewhere, it will be shown that in HS–130 Si_3N_4, controlled surface removal by careful grinding in small increments of ~0.005 mm subsequent to indentation increases the K_{IC} value as a function of surface removal. K_{IC} attains a constant maximum value (which is in good agreement with double-torsion data [5]) when the surface removal is equal to approximately four times the depth, x, of the Knoop impression (see Fig. 1). It is concluded from these experiments that residual compressive stress is present up to a depth of approximately four times the microhardness-impression depth and that its removal by grinding relaxes the tensile residual-stress component as well. Therefore, it is reasonable to take a value for z_0 equal to four times the microhardness-impression depth. Since the Knoop indentor has a known standard geometry, the impression depth is simply related to the long and short diagonal lengths of the impression by:

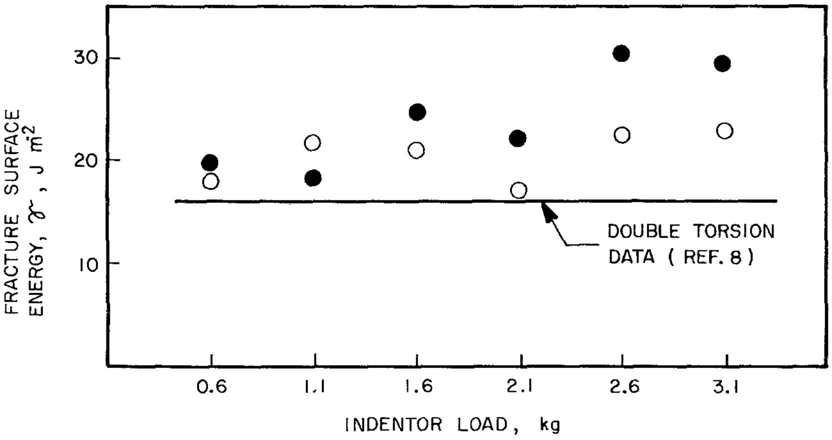
$$\frac{L}{x} \approx 30.48 \quad \frac{l}{x} \approx 4.28. \tag{6}$$

Since $z_0 \approx 4x$, then from Equation 6 $z_0 \approx l$ (the short diagonal length). Interestingly enough, Lawn and Swain also found $z_0 \approx l$ by direct observation of the deformation zone in quartz.

Now, Equation 5 can be utilized to calculate γ as a function of indentation load. For HS-130 Si_3N_4 and NC-203 SiC, the values of E and ν are shown in Table II. The calculated values of γ (open circles) for Si_3N_4 and SiC are shown in Fig. 2a and b, respectively. For comparison purposes, the



(a) HS - 130 Si_3N_4



(b) NC – 203 SiC

Figure 2 Comparison of fracture-surface energy values obtained in the present investigation with those obtained by other techniques.

TABLE II Room-temperature elastic constants of Si_3N_4 and SiC [6]

	Young's modulus E(MN m^{-2})	Poisson's ratio
HS-130 Si_3N_4	31×10^4	0.218
NC-203 SiC	45.5×10^4	0.226

average values of γ obtained by double-cantilever and double torsion techniques [7, 8] (solid lines) are also included in Fig. 2. The agreement between the γ values for Si_3N_4 appears to be quite good (Fig. 2a); the agreement between γ values for SiC also appears to be reasonable (Fig. 2b.)

Since the γ values in Fig. 2 were calculated from microcrack dimensions and indentor loads, it is important to be able to clearly observe the microcracks on the fracture surfaces of the specimens. Since this may be difficult in some cases, further simplification of the calculations was made by assuming the microcracks to be exactly semicircular with $2c$ equal to the long diagonal length L of the indentation (see Fig. 1). Under this assumption the actual determination of the microcrack dimensions is not a requirement and the γ values can be calculated simply (solid circles in Fig. 2) from the dimensions of the hardness impression. Comparison of these values to those represented by the solid line shows that the agreement is not particularly good; however, these simple modifications permit the prediction of γ values to within 30% of those obtained by more sophisticated techniques.

In conclusion, this study has shown that the approximate values of fracture-surface energies of hot-pressed Si_3N_4 and SiC can be predicted from microhardness-indentation loads and dimensions.

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The surface tension of Bi_2O_3-based fluxes used for the growth of magnetic garnet films

Thin films of rare earth iron garnet are commonly grown from solution in a $PbO–B_2O_3$ flux onto non-magnetic garnet substrates by the liquid-phase epitaxy (LPE) dipping process [1]. Such films are important for magnetic bubble domain, magneto-optic and microwave applications. For magneto-optic applications [2] it is desirable for the film to have a large Faraday rotation (θ) and a low optical absorption coefficient (α), the ratio θ/α defining the magneto-optic figure of merit at a given wavelength.

It is well known that the partial substitution of Bi in the garnet lattice enhances θ and that to obtain appreciable Bi substitution in garnet films, low growth temperatures are required [3]. However, at such temperatures divalent Pb from the flux is readily incorporated into the garnet, substantially increasing α. Although the $BaO-BaF_2–B_2O_3$ flux system may be used for garnet LPE, the combination of its high viscosity [4] and high surface tension [5] prevent a clean separation of the film from the flux on termina-