10/31/2002 7:26 AM
LBNL-51689

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October, 2002

submitted to
*Journal of the American Ceramic Society*

Work supported by the Director, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

# DETERMINING THE TOUGHNESS OF CERAMICS FROM VICKERS INDENTATIONS USING THE CRACK-OPENING DISPLACEMENTS: AN EXPERIMENTAL STUDY[†]

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Recently, a method for evaluating the fracture toughness of ceramics has been proposed based on the computed crack-opening displacements of cracks emanating from Vickers hardness indentations. In order to verify this method, experiments were carried out to determine the toughness of a commercial silicon carbide ceramic, Hexaloy SA, by measuring the crack-opening profiles of such Vickers indentation cracks. While the obtained toughness value of $K_o = 2.3$ MPa√m was within 10% of that measured using conventional fracture toughness testing, the computed crack-opening profiles corresponding to this toughness displayed poor agreement with those measured experimentally, raising concerns about the suitability of this method for determining the toughness of ceramics. The effects of subsurface cracking and cracking during loading are considered as possible causes of such discrepancies, with the former based on evidence observed for secondary radial cracking which affected the crack opening profile and deduced toughness values.

## I. Introduction

Indentation has long been considered an attractive method for assessing the toughness of ceramic materials due to the ease and low cost of conducting experiments. The predominant method to date has involved using a Vickers diamond microhardness indenter to induce radial cracks in the material. Such radial cracks are thought to emanate from the indent as a result of residual tensile stresses that develop during unloading.[1,2] Measured crack lengths are then correlated to the material toughness, $K_c$, through the semi-empirical relationship:[2]

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[†]Supported by the Director, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

$$K_c = \chi \sqrt{\frac{E}{H}} \frac{P}{a^{\frac{3}{2}}} \quad , \tag{1}$$

where $P$ is the applied load, $E$ is Young's modulus, $H$ is the Vickers hardness, $a$ is the radial crack length measured from the center of the indent, and $\chi$ is an empirically determined "calibration" constant taken to be 0.016 ± 0.004.[2] There are several disadvantages with this method, however. Firstly, there is considerable uncertainty (± 25%) in the empirical constant $\chi$, which leads to an inherent uncertainty in the deduced toughness values. Additionally, the method is problematic for materials that exhibit rising toughness with crack extension (i.e., R-curve behavior) due to the presence of extrinsic toughening mechanisms such as crack bridging in the crack wake; here the indentation toughness test gives an essentially a random point on the R-curve, i.e., a toughness value, corresponding to the crack length and geometry of the indentation crack, which lies between the intrinsic toughness, $K_o$, and the steady-state plateau toughness, $K_{ss}$. Finally, due to indentation size effects in ceramics, the value $H$ is not always constant and sometimes depends on the load, $P$, placing further uncertainty on $K_c$ values computed using this method (Eq. 1).[3]

Recently, Fett has made available solutions for the crack-opening displacements of Vickers indentation cracks, which are suggested to provide an alternative, non-empirical, approach to determining the fracture toughness from indentation cracks.[4] The near-tip stress intensity for a linear-elastic indent crack, $K_{tip}$, is related to the crack-opening displacements, $u(r)$, by:[4]

$$u(r) = \frac{4K_{tip}\sqrt{b}}{\pi E'}\left(A\sqrt{1-\frac{r}{a}} + B(1-\frac{r}{a})^{\frac{3}{2}} + C(1-\frac{r}{a})^{\frac{5}{2}}\right) \ , \tag{2}$$

where $E'$ is the plane strain modulus (i.e., $E' = E/1\text{-}\nu^2$, where $\nu$ is Poisson's ratio) while $r$, $b$, and $a$ are the radial position, contact-zone radius, and the crack length, respectively, as measured from the center of the indent. The coefficients are written as:[4]

$$A = \sqrt{\frac{\pi a}{2b}} \quad , \tag{3}$$

$$B \cong 0.011 + 1.8197 \ln(\frac{a}{b}) \quad , \tag{4}$$

$$C \cong -0.6513 + 2.121 \ln(\frac{a}{b}) \quad . \tag{5}$$

Note that the first term in Eq. 2 reduces to the familiar Irwin elasticity relationship for the near-tip crack-opening profile:

$$u(r) = \frac{K_{\mathrm{tip}}}{E'}\sqrt{\frac{8(a-r)}{\pi}} \quad . \tag{6}$$

For an indentation crack growing due to a residual stress field, it is assumed that the indentation crack arrests when $K_{\mathrm{tip}}$ is equal to the intrinsic toughness, $K_o$. Accordingly, for a ceramic with no R-curve toughening behavior, the intrinsic toughness, $K_o$, may be determined directly from the crack-opening profile by using Eqs. 2-5. Additionally, for bridging ceramics such a grain-elongated $Si_3N_4$ and ABC-SiC,[5-7] this method, if successful, has the potential to allow for the determination of the intrinsic toughness, or beginning point of the R-curve, by deconvoluting the displacements due to the residual and contact stresses (Eq. 2-5) and those due to the bridging stresses.[4]

Consequently, the objective of this paper is to present a first experimental study of the method proposed by Fett[4] for the determination of ceramic toughness from Vickers indentation cracks. To simplify the interpretation of results, a commercial SiC material was chosen for this study which fractures transgranularly and has a single-value toughness (i.e., no R-curve behavior). Results are compared to fracture toughness values measured both with precracked compact-tension samples and with conventional indentation toughness methods using Eq. 1.

## II. Experimental Procedures

The ceramic studied was a commercial pressureless sintered silicon carbide, Hexoloy SA. Prior to indentation, the material surface was ground flat and lapped to a 1 μm finish using diamond compounds. Vickers indentations were then placed in the material using a 4 kg load, chosen to maximize the length of the radial cracks while avoiding chipping on the sample surface during indentation.

The intrinsic toughness, $K_o$, was determined three ways:

- *The crack-opening profile (COP) method:* Following indentation, the crack-opening profile, $u(r)$, of the radial cracks was measured in a field-emission scanning electron microscope (FESEM), with a maximum resolution of 5 nm for the full crack opening, $2u$, achieved near the crack tip at magnifications up to 60,000X. Such results were used to compute the value of $K_o$ using Eqs. 2-5. To determine the optimal value, the least-squares method was employed to find the value of $K_o$ that gave a calculated crack opening nearest that measured experimentally.
- *The near-tip (NT) method:* A similar method was used to assess the toughness by using only the near-tip crack-opening data along with the Irwin solution (Eq. 6). This later method was carried out using 5, 10, 15, and 20 μm of crack-opening data, as measured from the crack tip.
- *The traditional indentation toughness (TIT) method:* Finally, the toughness of the material was assessed from the indentation crack using the standard method by measuring the indent size and crack length in an optical microscope and computing the toughness using Eq. 1.

Results were compared to $K_o = K_c$ values measured using precracked disk-shaped compact-tension samples,[5,7] in nominal accordance with ASTM Standards (E-399) for fracture toughness measurements.

## III. Results

Crack-opening profiles for three cracks emanating from Vickers indents, denoted cracks I, II, and III, are shown in Fig. 1, with details of the near-tip regions shown in the inset. Micrographs of these three cracks are shown in Fig. 2. It is apparent from Fig. 1 that the crack openings for crack II and III are somewhat wider than that of crack I; however, there was a secondary radial crack also emanating from the indent near crack I (Fig. 2a). Based on the results in Fig. 1, and using Eqs. 2-5, the intrinsic toughness, $K_o$, for Hexoloy SA was calculated by the COP method to be 2.0 MPa$\sqrt{\text{m}}$ for crack I and 2.3 MPa$\sqrt{\text{m}}$ for cracks II and III. The calculated crack-opening profiles corresponding to these toughness values, together with the experimentally measured openings, are shown in Figs. 3a, b, and c for cracks I, II, and III, respectively.

Toughness estimates from the NT method, using only the near-tip data (Fig. 1) and Eq. 6, yielded different results depending on how much near-tip data was used to fit the parabola. The estimates of $K_o$ are summarized in Table I with the results by the COP method. Additionally, the best fit computed near-tip crack profiles ,obtained using Eq. 6, are shown with the near-tip opening data for each crack in the insets of Fig. 3.

Finally, the toughness of Hexoloy SA was assessed using Eq. 1 based on measurements of cracks at four indents (TIT method), giving an average toughness of 2.1 ± 0.7 MPa√m.

## IV. Discussion

The reported fracture toughness for Hexoloy SA, obtained using conventional fracture mechanics testing with disk-shaped compact-tension specimens, is roughly 2.5 MPa√m.[5,7] Results obtained by using the COP method give values slightly lower than this; however, for cracks II and III, the deduced value of 2.3 MPa√m falls very close to that obtained by conventional fracture mechanics testing. For the case of crack I, it should be noted that a secondary radial crack was observed extending from the indent near the main crack (Fig. 2a). It is probable that this secondary crack relieved some of the residual stress due to the indent in the vicinity of the main crack, affecting the crack-opening profile, and correspondingly reducing the deduced toughness value. For this reason, special care was taken in choosing cracks II and III such that no (crack II) or minimal (crack III) secondary cracking could be observed. Thus, discounting the result obtained from crack I and considering only the results obtained from cracks II and III, one may conclude that a reasonable estimate of the material toughness can be obtained by using the COP method, i.e., within ~ 10% of typical reported values.

The toughness results obtained using the TIT method (i.e., Eq. 1) cover a wide range of values from 1.4 to 2.8 MPa√m. While the upper values in this range indeed overlap with those obtained by conventional fracture mechanics methods, it should be noted that the lowest values are some 45% below the reported toughness value for Hexaloy SA. This large range of uncertainty represents one disadvantage of using the TIT method to determine the toughness of a ceramic. In comparison, results obtained from the COP method are within 10% of the expected value, and even when secondary cracking is believed to have affected the results, i.e., crack I, the measured value is still within 20% of the expected toughness.

One concern about the present results, however, is the poor correspondence of the best fit crack-opening profile computed using Eqs. 2-5 to the actual data (Fig. 3), particularly in the near-tip region. The poor fit in the near-tip region is further evidenced by the low toughness values obtained using the NT method (Table I), up to 50% lower than obtained using the COP method. While the COP method is expected to be more accurate by taking the entire crack opening into account, such a large discrepancy between the results was not expected; indeed, a far better match of the computed crack-opening shape to the actual data was anticipated. One possible explanation for this difference is subsurface cracking. It is well known that during Vickers indentation, in addition to radial cracking, that lateral cracks also commonly form below the surface, and under high enough load intersect the surface to cause chipping.[8-10] However, none of the methods for determining indentation toughness take into account the possible effects of lateral cracking below the sample surface. In the case of the COP and NT methods, one may expect subsurface cracking to affect the residual stress field, and correspondingly, the crack-opening profiles. Indeed, partially relieving the residual stresses by subsurface cracking would allow the cracks to close partially, resulting in smaller measured crack openings than one would expect; this is the case observed in the near tip in this study. Furthermore, Cook and Pharr[9] have demonstrated that radial cracking does not occur upon unloading in all brittle materials, an assumption which provides a basis for all the indentation toughness methods presented here. The possibility of cracking during the loading portion of indentation raises further concerns about the present level of understanding of the complicated cracking configurations and interactions that occur and may also account for the discrepancies between the measured and computed crack openings observed in this study. Thus, while the crack-opening profile method appears promising as a method to assess ceramic toughness from Vickers indents, it is clear that further investigation in this area is warranted.

## V. Conclusions

Based on an experimental study using the crack-opening displacements from Vickers indentation cracks to determine the fracture toughness of a commercial silicon carbide ceramic, Hexaloy SA (which displays no R-curve behavior), the following conclusions are made:

1. By using the entire crack-opening profile to assess toughness, an intrinsic toughness value of $K_o$ = 2.3 MPa√m was obtained. This value is within 10% of the typical values reported using standard fracture mechanics specimens, demonstrating the viability of using such a method for toughness measurements.
2. Secondary radial cracks are believed to affect the crack-opening profile, and correspondingly the computed toughness values, by relieving some of the residual stresses. Indeed, measured crack openings were smaller, and the deduced toughness was lower, for one crack where significant secondary radial cracking was evident.
3. Even in cases where secondary radial cracking was not present, the computed crack-opening profiles did not correspond well with those measured experimentally, particularly in the near-tip region. Accordingly, toughness values deduced using only the near-tip data were significantly lower than those using the entire crack-opening profile. Possible explanations for these discrepancies include subsurface cracking that relieves some residual stresses and affects the crack openings or cracking during the loading portion of the indentation.
4. While the method of using the crack-opening profiles to determine the fracture toughness of ceramics from Vickers indentations holds promise, it is apparent that there are still unresolved issues that must be addressed before this can be considered as a reliable test method.

## References


[1]B. R. Lawn, A. G. Evans and D. B. Marshall, "Elastic/plastic indentation damage in ceramics: The median/radial crack system," *J. Am. Ceram. Soc.,* **63** [9-10] 574-81 (1980).

[2]G. R. Anstis, P. Chantikul, B. R. Lawn and D. B. Marshall, "A critical evaluation of indentation techniques for measuring fracture toughness. I. Direct crack measurements," *J. Am. Ceram. Soc.,* **64** [9] 533-8 (1981).

[3]J. Gong, J. Wang and Z. Guan, "Indentation toughness of ceramics: A modified approach," *J. Mater. Sci.,* **37** [4] 865-9 (2002).

[4]T. Fett, "Computation of the crack opening displacements for Vickers indentation cracks," Report FZKA 6757, Forschungszentrum Karlsruhe, Karlsruhe, Germany, 2002.

[5]C. J. Gilbert, J. J. Cao, W. J. MoberlyChan, L. C. DeJonghe and R. O. Ritchie, "Cyclic fatigue and resistance-curve behavior of an *in situ* toughened silicon carbide with Al-B-C additions," *Acta Metall. Mater.,* **44** [8] 3199-214 (1996).

[6]C.-W. Li, D.-J. Lee and S.-C. Lui, "R-curve behavior and strength for in-situ reinforced silicon nitrides with different microstructures," *J. Am. Ceram. Soc.,* **75** [7] 1777-85 (1992).

[7]C. J. Gilbert, J. J. Cao, L. C. De Jonghe and R. O. Ritchie, "Crack-growth resistance-curve behavior in silicon carbide: small versus long cracks," *J. Am. Ceram. Soc.,* **80** [9] 2253-61 (1997).

[8]B. R. Lawn and M. V. Swain, "Microfracture beneath point indentations in brittle solids," *J. Mater. Sci.,* **10** [1] 113-22 (1975).

[9]R. F. Cook and G. M. Pharr, "Direct observation and analysis of indentation cracking in glasses and ceramics," *J. Am. Ceram. Soc.,* **73** [4] 787-817 (1990).

[10]D. B. Marshall, B. R. Lawn and A. G. Evans, "Elastic/plastic indentation damage in ceramics: the lateral crack system," *J. Am. Ceram. Soc.,* **65** [11] 561-6 (1982).

**Figure Captions**

**Fig. 1.** Measured crack-opening profiles for three indent cracks with inset showing an enlargement of the near-tip region for each crack.

**Fig. 2.** Optical micrographs showing the indent cracks used for crack-opening profile measurements.

**Fig. 3.** Plots of the best fit crack-opening profiles deduced by the COP method along with the measured data. Insets show the best fit near-tip profiles determined by the NT method along with the near-tip data. Also shown in the insets are the expected crack openings based on the COP method, note the poor fit of this curve to the experimental data in the near-tip region.

**Table I: Fracture Toughness $K_0$ Results Based on Crack-Opening Profile Data**

| | Crack I | Crack II | Crack III |
|---|---|---|---|
| COP method (Eqs. 2-5) | 2.0 MPa√m | 2.3 MPa√m | 2.3 MPa√m |
| NT method (Eq. 6) 5 μm fit | 0.93 MPa√m | 1.3 MPa√m | 1.4 MPa√m |
| NT method (Eq. 6) 10 μm fit | 1.0 MPa√m | 1.4 MPa√m | 1.2 MPa√m |
| NT method (Eq. 6) 15 μm fit | 1.2 MPa√m | 1.5 MPa√m | 1.4 MPa√m |
| NT method (Eq. 6) 20 μm fit | 1.3 MPa√m | 1.6 MPa√m | 1.5 MPa√m |