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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 by Fett based on the computed crack-opening displacements of cracks emanating from Vickers hardness indentations. To verify this method, experiments have been conducted to determine the toughness of a commercial silicon carbide ceramic, Hexoloy SA, by measuring the crack-opening profiles of such Vickers indentation cracks. Although the obtained toughness value of $K_o = 2.3$ MPa·m$^{1/2}$ is within 10% of that measured using conventional fracture toughness testing, the computed crack-opening profiles corresponding to this toughness display 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 direct observations of lateral subsurface cracks below the indents.

I. Introduction

INDENTATION long has been considered an attractive method for assessing the toughness of ceramic materials because of 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, arresting when the near-tip stress intensity, K_{tip}, equals the material toughness, K_c.[1,2] Measured crack lengths are correlated to K_c through the semiempirical relationship[2]

$$K_c = \chi\left(\frac{E}{H}\right)^{1/2}\frac{P}{a^{3/2}} \tag{1}$$

where P is the applied load, E Young's modulus, H the Vickers hardness, a the radial crack length measured from the center of the indent, and χ an empirically determined "calibration" constant taken to be 0.016 ± 0.004.[2] There are several disadvantages with this method, however. First, there is considerable uncertainty (±25%) in the empirical constant χ, which leads to an inherent uncertainty in the deduced toughness values. Second, the method is problematic for materials that exhibit increasing toughness with crack extension (i.e., R-curve behavior) because of the presence of extrinsic toughening mechanisms, such as crack bridging in the crack wake; here, the indentation toughness test gives an essentially 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}. Third, because of indentation size effects in ceramics, the value of H is not always constant and sometimes depends on the load, P, placing further uncertainty on K_c values computed using this method (i.e., Eq. (1)).[3]

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

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

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

$$A = \left(\frac{\pi a}{2b}\right)^{1/2} \tag{3}$$

$$B \approx 0.011 + 1.8197\ \ln\frac{a}{b} \tag{4}$$

$$C \approx -0.6513 + 2.121\ \ln\frac{a}{b} \tag{5}$$

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

$$u(r) = \frac{K_{\text{tip}}}{E'}\left[\frac{8(a-r)}{\pi}\right]^{1/2} \tag{6}$$

For a ceramic with no R-curve toughening behavior, the intrinsic toughness, K_o, can be determined directly from the crack-opening profile using Eqs. (2)–(5), assuming, as in the traditional indentation toughness method, that the crack arrests when K_{tip} equals the material toughness. Additionally, for bridging ceramics, such as 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 (Eqs. (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 that fractures transgranularly (i.e., no bridging) and has a single-value toughness (i.e., no R-curve behavior) was chosen for

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this study. The reasons for choosing this material are threefold: (i) previous investigations have shown that similar nonbridging silicon carbides produce a well-defined radial crack system;[2,8] (ii) the transgranular fracture mode with no crack bridging simplifies the interpretation of the results; and (iii) grain-bridging silicon carbides can be readily produced,[5,7] allowing for future testing of this method with bridging ceramics while still using nominally the same material, SiC. Results are compared with fracture toughness values measured 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 SiC, Hexoloy SA (Saint-Gobain Advanced Ceramics Corp., Niagara Falls, NY). Before it was indented, 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 39 N 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 using the analysis proposed by Fett,[4] denoted here as the crack-opening profile (COP) method. COP values, $u(r)$, of the radial cracks were measured using field-emission scanning electron microscopy (FESEM), with a maximum resolution of 5 nm for the full crack opening, $2u$. Such results were used to compute the value of K_o using Eqs. (2)–(5). To determine the optimal value of K_o, the least-squares method was used to find the value that gave a calculated crack opening nearest that measured experimentally.

These results were compared with those obtained using a similar method, denoted the near-tip (NT) method, where only the near-tip crack-opening data along with the Irwin solution (Eq. (6)) were used. This was conducted using 5, 10, 15, and 20 μm of crack-opening data, as measured from the crack tip.

Additionally, results were compared with the traditional indentation toughness (TIT) method,[2] where the toughness of the material was assessed using the standard procedure of measuring the indent size and crack length in an optical microscope and computing the toughness with Eq. (1).

Finally, all indentation toughness results were compared with values measured using conventional fracture mechanics methods involving precracked disk-shaped compact-tension samples,[5,7] in nominal accordance with ASTM Standard E-399 ("Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials," ASTM Designation E399–90. *ASTM Book of Standards*, Vol. 03.01. ASTM International, West Conshohocken, PA, 1997) for fracture toughness measurements.

III. Results

Crack-opening profiles for three cracks emanating from Vickers indents, denoted as cracks I, II, and III, are shown in Fig. 1, with details of the near-tip regions shown in the inset. An example indent with subsurface crack profile is shown in the micrograph in Fig. 2. It is apparent from Fig. 1 that the crack openings for cracks II and III are somewhat wider than that of crack I. Based on the results in Fig. 1 and using Eqs. (2)–(5), the intrinsic toughness, K_o, for Hexoloy SA has been calculated using the COP method to be 2.0 MPa·m$^{1/2}$ for crack I and 2.3 MPa·m$^{1/2}$ for cracks II and III. The calculated crack-opening profile corresponding to the toughness value of 2.3 MPa·m$^{1/2}$ for crack II is shown in Fig. 3, together with the experimentally measured opening. Such results appear similar for all three cracks.

Toughness estimates from the NT method, using only the near-tip data (Fig. 1) and Eq. (6), yield different results, depending on how much near-tip data are used to fit the parabola. The estimates of K_o are summarized in Table I with the results using 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 crack II in the inset of Fig. 3.

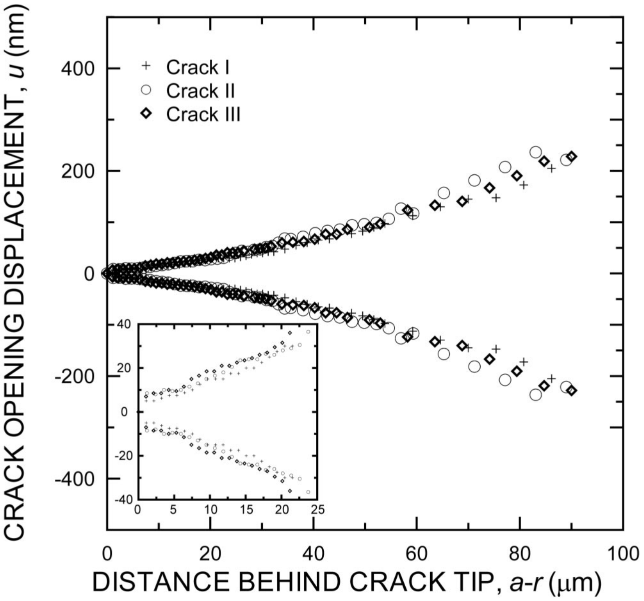

Fig. 1. Measured crack-opening profiles for three indent cracks with inset showing an enlargement of the near-tip region for each crack.

Finally, the toughness of Hexoloy SA has been 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$^{1/2}$.

IV. Discussion

The reported fracture toughness for Hexoloy SA, obtained using conventional fracture mechanics testing with disk-shaped

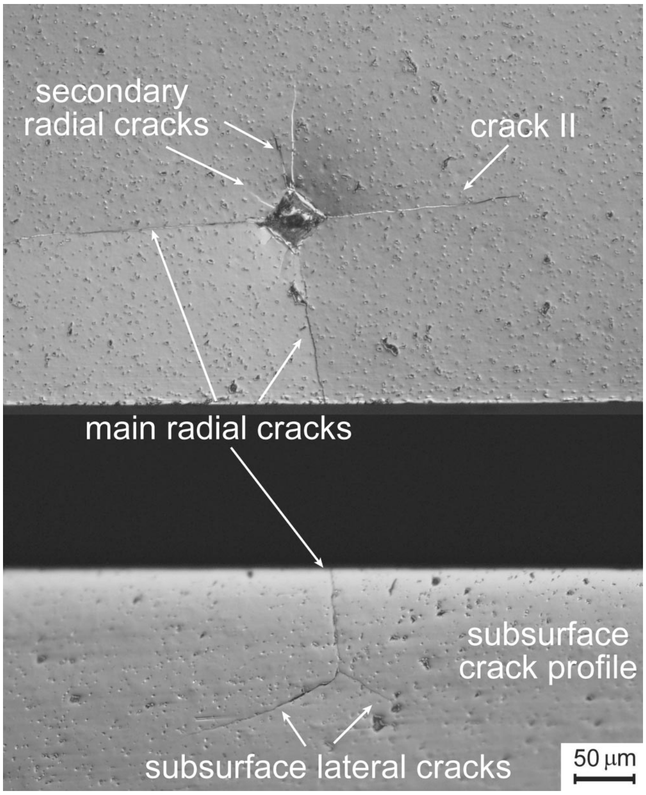

Fig. 2. Optical micrographs showing an example indent with subsurface crack profile. Crack II used for crack-opening profile measurements is shown, along with clear evidence of subsurface lateral cracking and secondary radial cracking.

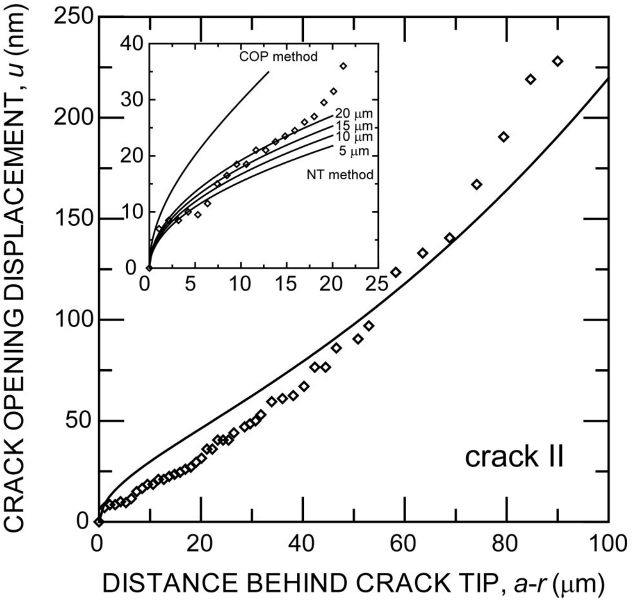

Fig. 3. Representative plot of the best-fit crack-opening profile deduced using the COP method along with the measured data; results were similar for all three cracks. Inset shows the best-fit near-tip profile determined by the NT method along with the near-tip data. Also shown in the inset is the expected crack opening based on the COP method; note the poor fit of this curve to the experimental data in the near-tip region.

compact-tension samples, is ~2.5 MPa·m$^{1/2}$.[5,7] Results obtained using the COP method give values slightly lower than this value; however, for cracks II and III, the deduced value of 2.3 MPa·m$^{1/2}$ is very close to that obtained using conventional fracture mechanics testing. For the case of crack I, a secondary radial crack, similar to those seen in Fig. 2, is observed extending from the indent near the main crack. It is probable that this secondary crack relieves some of the residual stress due to the indent in the vicinity of the main crack, affecting the crack-opening profile and, correspondingly, decreasing the deduced toughness value. For this reason, special care has been taken in choosing cracks II and III such that no (crack II) or minimal (crack III) secondary cracking can be observed in the vicinity of the main crack. Thus, discounting the result obtained from crack I and considering only the results obtained from cracks II and III, one can conclude that a reasonable estimate of the material toughness can be obtained using the COP method, i.e., within ~10% of typical reported values.

Results from the TIT method, i.e., using Eq. (1), cover a wide range of values from 1.4 to 2.8 MPa·m$^{1/2}$. Although the upper range of values overlaps with those obtained by conventional fracture mechanics methods, the lowest values are ~45% below the reported toughness value for Hexoloy SA. This large degree 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 radial cracking is believed to have affected the results, i.e., crack I, the measured value is within 20% of the expected toughness.

Table I. Fracture Toughness, K_0, Results Based on Crack-Opening Profile Data

Method	Fracture toughness (MPa·m$^{1/2}$) Crack I	Crack II	Crack III
COP (Eqs. (2)–(5))	2.0	2.3	2.3
NT (Eq. (6))			
5 µm fit	0.93	1.3	1.4
10 µm fit	1.0	1.4	1.2
15 µm fit	1.2	1.5	1.4
20 µm fit	1.3	1.6	1.5

One concern about the COP method that results, however, is the poor correspondence of the best-fit crack-opening profile computed using Eqs. (2)–(5) to the experimentally measured profiles (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 that obtained using the COP method. Although the COP method is expected to be more accurate by taking into account more of the crack opening, such a large discrepancy between the results is not expected; indeed, a far better match of the computed crack-opening shape to the actual data is anticipated. One possible explanation for this difference is subsurface lateral cracking, which is shown in Fig. 2. It is well-known that, during Vickers indentation, in addition to radial cracking, lateral cracks commonly form below the surface, and, under high enough load, intersect the surface to cause chipping.[9–11] Although such subsurface cracking certainly affects the residual stress field and, accordingly, the crack-opening profiles, none of the methods presented here for determining indentation toughness take into account these effects. In the case of the COP and NT methods, relieving some of the residual stresses by subsurface cracking allows the cracks to close partially, because there is less stress keeping them open. This scenario results in smaller measured crack openings than one expects, as observed in the near tip in this study. Furthermore, Cook and Pharr[10] have demonstrated that radial cracking does not occur on unloading in all brittle materials, an assumption that provides a basis for all three indentation toughness methods compared here. It is currently unknown whether radial cracks form in SiC during unloading (as assumed), because only transparent materials can be used in the method of Cook and Pharr.[10] However, 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; such factors may contribute to discrepancies between the measured and computed crack openings and place further uncertainty on indentation toughness methods in general. Therefore, although Fett's[4] COP 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 SiC ceramic, Hexoloy SA (which displays no bridging/*R*-curve behavior), the following conclusions are made.

(1) Using the entire crack-opening profile to assess toughness, an intrinsic toughness value of $K_o = 2.3$ MPa·m$^{1/2}$ is obtained. This value is within 10% of the typical values reported using standard fracture mechanics samples, 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 are smaller, and the deduced toughness is lower for one crack where significant secondary radial cracking is evident.

(3) Even in cases where secondary radial cracking is not present, the computed crack-opening profiles do not correspond well with those measured experimentally, particularly in the near-tip region. Accordingly, toughness values deduced using only the near-tip data are significantly lower than those using the entire crack-opening profile. Possible explanations for these discrepancies include observed subsurface lateral cracking that relieves some residual stresses and affects the crack openings or cracking during the loading portion of the indentation.

(4) Although 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 remain

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] 533–38 (1981).

[3] J. Gong, J. Wang, and Z. Guan, "Indentation Toughness of Ceramics: A Modified Approach," *J. Mater. Sci.*, **37** [4] 865–69 (2002).

[4] T. Fett, "Computation of the Crack Opening Displacements for Vickers Indentation Cracks," Rept. No. FZKA 6757, Forschungszentrum Karlsruhe, Karlsruhe, Germany, 2002.

[5] C. J. Gilbert, J. J. Cao, W. J. MoberlyChan, L. C. De Jonghe, 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] J. J. Petrovic and L. A. Jacobson, "Controlled Surface Flaws in Hot-Pressed SiC," *J. Am. Ceram. Soc.*, **59** [1–2] 34–37 (1976).

[9] B. R. Lawn and M. V. Swain, "Microfracture beneath Point Indentations in Brittle Solids," *J. Mater. Sci.*, **10** [1] 113–22 (1975).

[10] 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).

[11] 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] 561–66 (1982). ☐