i Changes have been noted and being incorporated off-site.

CRACKING DURING NANOINDENTATION AND ITS USE IN THE MEASUREMENT OF FRACTURE TOUGHNESS

CONF-941144--106

D.S. HARDING*, W.C. OLIVER**, and G.M. PHARR*
*Department of Materials Science, Rice University, P.O. Box 1892, Houston, TX 77251
**Nano Instruments, Inc., P.O. Box 14211, Knoxville, TN 37914

ABSTRACT

Results of an investigation aimed at developing a technique by which the fracture toughness of a thin film or small volume can be determined in nanoindentation experiments are reported. The method is based on the radial cracking which occurs when brittle materials are deformed by a sharp indenter such as a Vickers or Berkovich diamond. In microindentation experiments, the lengths of radial cracks have been found to correlate reasonably well with fracture toughness, and a simple semi-empirical method has been developed to compute the toughness from the crack lengths. However, a problem is encountered in extending this method into the nanoindentation regime with the standard Berkovich indenter in that there are well defined loads, called cracking thresholds, below which indentation cracking does not occur in most brittle materials. We have recently found that the problems imposed by the cracking threshold can be largely overcome by using an indenter with the geometry of the corner of a cube. For the cube-corner indenter, cracking thresholds in most brittle materials are as small as 1 mN (~ 0.1 grams). In addition, the simple, well-developed relationship between toughness and crack length used for the Vickers indenter in the microindentation regime can be used for the cube-corner indenter in the nanoindentation regime provided a different empirical constant is used.

INTRODUCTION

Nanoindentation is a widely accepted tool for measuring the mechanical properties of thin films and small volumes of material [1,2]. One of the great advantages of the technique is its ability to probe a surface and map its properties on a spatially resolved basis, sometimes with a resolution of better than 1 μm. Nanoindentation has been used to characterize elastic properties such as the modulus, E [3,4], plastic properties such as the hardness, H [3,4], and time dependent properties, such as the stress exponent for creep, n [5]. To date, however, little attention has been given to how the technique may be useful in the measurement of properties important in fracture, such as the fracture toughness, K_c. Here, the results of a study aimed at establishing a method by which nanoindentation can be used in the measurement of fracture toughness are reported.

The method we are pursuing is based on the radial cracking which occurs when brittle materials are indented by a sharp indenter such as a Vickers or Berkovich diamond (see Fig.1). A theoretical description of the mechanics of this indentation cracking has been developed by Lawn, Evans, and Marshall [6] which leads to a simple relation between the fracture toughness, K_c, and the lengths of the radial cracks, c, of the form:

$$K_c = \alpha \left(\frac{E}{H}\right)^{1/2} \left(\frac{P}{c^{3/2}}\right) \qquad (1)$$

Here, P is the peak indentation load and α is an empirical constant which depends on the geometry of the indenter. A particularly attractive feature of using this method in nanoindentation

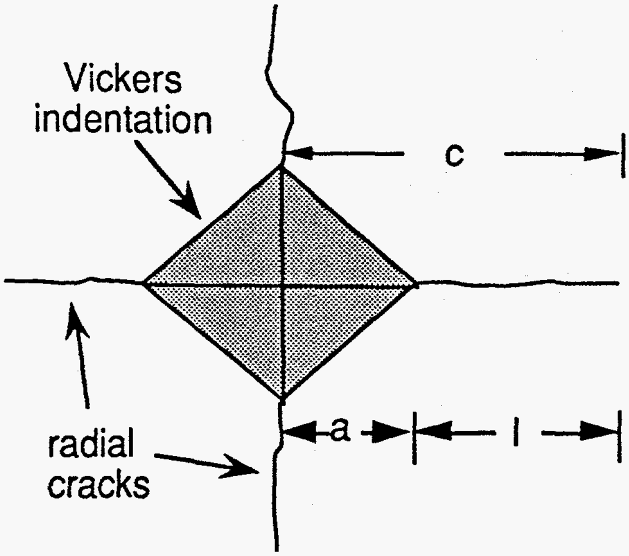


Fig.1. Schematic illustration of radial cracking at a Vickers indentation.

is that both E and H can be determined directly from analyses of nanoindentation load-displacement data. Thus, provided one has a means for measuring crack lengths, implementation of this method using nanoindentation testing techniques is relatively straightforward.

A critical assessment of the usefulness of Eqn.1 has been provided by Anstis et al. [7], who examined indentation cracking in brittle materials spanning a wide range of toughness. The materials were indented at several loads with a Vickers indenter in a microhardness tester, and the resulting crack lengths and indentation sizes were measured using optical microscopy. Using moduli obtained from the literature and hardnesses obtained from the indentation sizes, Eqn.1 was used to compute the toughness of each material for comparison with values obtained using more conventional methods. The constant α = 0.016 was empirically found to give the best results, and the toughnesses measured from the crack lengths were found to be accurate to within about 40%. This level of accuracy seems to be typical of the method.

To date, fracture toughness measurement by the indentation cracking method has been applied to indentations which, by nanoindentation standards, are fairly large. Typically, the indentations are produced at loads of 1000 grams or greater, for which the cracks are of the order of 100 μm or so in length. Since the size of the cracks sets a limit on the spatial resolution of the technique, much smaller indentations are needed if the method is to be useful in measuring the toughness of very thin films or small volumes using nanoindentation. In this regard, however, a significant problem exists in that there are well-defined loads, called cracking thresholds, below which indentation cracking does not occur. For a Vickers indenter, cracking thresholds in most ceramic materials are about 25 gms or more [8], and since the indentations associated with these loads are relatively large (several microns), the thresholds place severe restrictions on the spatial resolution which can potentially be achieved.

We have recently found that the problems associated with the threshold can be largely overcome by using sharper indenters. Here, we report how one specific indenter can be used to significantly reduce the threshold and how the crack lengths produced with this indenter correlate with toughness.

REDUCING THE INDENTATION CRACKING THRESHOLD

Our approach to reducing the cracking threshold has been to use indenter geometries different from that of the Berkovich, the standard indenter used in nanoindentation testing. The Berkovich indenter is a three-sided pyramid with the same depth-to-area ratio as a Vickers indenter (a 4-sided pyramid), and the cracking thresholds for it are very similar to the Vickers.

DISCLAIMER

DISCLAIMER

Table I. Properties of materials used in this study.

Material	Cube-corner Threshold (mN)	Vickers Threshold (mN)	E [(1)] (GPa)	H [(1)] (GPa)	K_c (MPa$\sqrt{m}$)
soda-lime glass	0.5 - 1.5	250 - 500	76.1	6.1	0.70 [(2)]
fused quartz	0.5 - 1.5	1000 - 1500	69.3	8.3	0.58 [(2)]
Pyrex glass	1.5 - 4.4	500 - 1000	60.5	6.3	0.63 [(2)]
silicon (100)	0.5 - 1.5	20 - 50	185.6	11.5	0.7 [(3)]
silicon (111)	0.5 - 1.5	50 - 100	205.8	11.2	0.7 [(3)]
germanium (111)	0.5 - 1.5	< 10	133.6	10.1	0.5 [(3)]
sapphire (111)	4.4 - 13.3	50 - 100	433.1	25.9	2.2 [(3)]
spinel (100)	4.4 - 13.3	100 - 150	286.2	18.4	1.2 [(3)]
silicon nitride (NC 132)	40 - 120	1000	319.9	21.6	4.7 [(3)]
silicon carbide (SA)	4.4 - 13.3	100 - 150	454.7	30.8	2.9 [(3)]
silicon carbide (ST)	grain pushout	500 - 1000	427	21.8	4.1 [(3)]

(1) nanoindentation measurements with Berkovich indenter

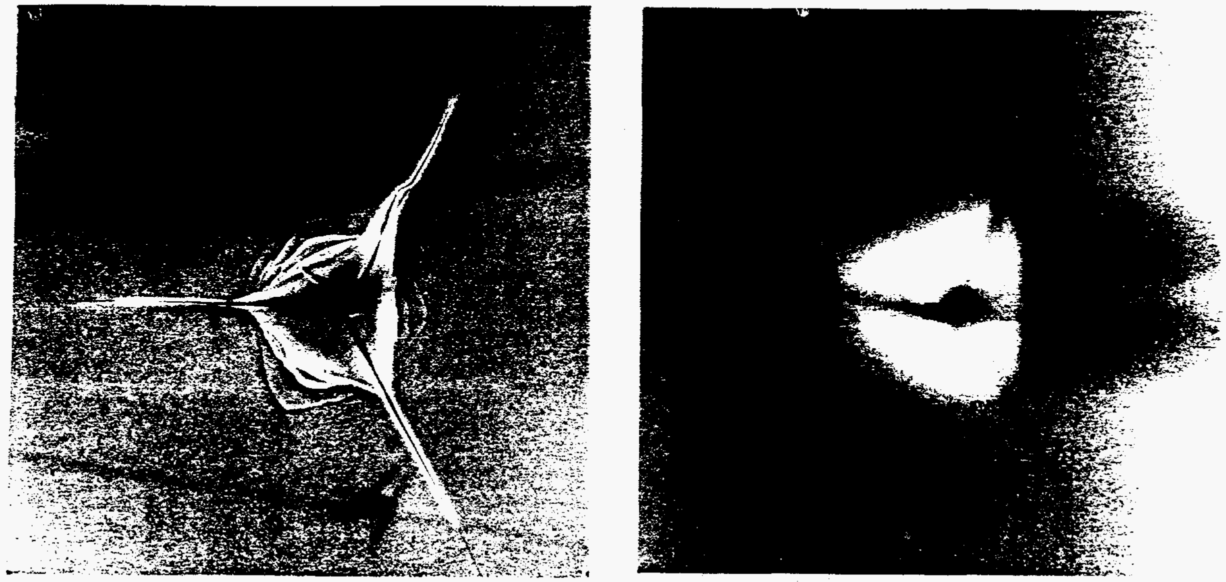
(2) 3-pt bend chevron notch method

(3) from material data sheet or literature

We have found that cracking thresholds can be substantially reduced by employing sharper indenters, i.e., indenters with smaller included tip angles, and the one on which we have focused is a three-sided indenter like the Berkovich but with the geometry of the corner of a cube. For the cube-corner indenter, the angle between the axis of symmetry and a face, 35.3°, is considerably smaller than the 65.3° angle for the Berkovich.

The rational for using sharper indenters like the cube-corner is quite simple. At a given load, the cube-corner and Berkovich diamonds should, to a first approximation, penetrate the material to produce approximately equal projected contact areas, i.e., the hardness measured with the two indenters should be about the same. During the formation of the hardness impressions, however, the cube-corner geometrically displaces more than 3 times the volume of the Berkovich, thus producing greater stresses and strains in the surrounding material. Given that the nucleation and propagation of indentation cracks are processes promoted by stress and strain, one would then qualitatively expect a reduction in the threshold for the sharper indenter.

To establish if and how much the cracking threshold is actually reduced by the cube-corner indenter, a study was undertaken in which nanoindentations were made in the brittle materials listed in Table I to peak loads ranging from 1 to 100 mN. Scanning electron microscopy was then used to image the indentations and determine the loads below which surface cracking could not be detected. The Vickers thresholds for the materials were also determined by optical examination of indentations made at higher loads in a conventional microhardness tester. The observed thresholds are shown in Table I. Clearly, the cube-corner indenter is very effective in reducing the threshold; in comparison to the Vickers indenter, the cube-corner thresholds are at least an order of magnitude smaller in each of the materials. Furthermore, since the indentation size at the cube-corner threshold in most of the materials is less than a micron, the cube-corner indenter can be employed to produce cracks at the micron to sub-micron scale. Examples of small, cracked indentations made with the cube corner indenter in soda-lime glass are shown in Fig.2.



(a) 6250 nm (b) 625 nm

Fig.2. Small cube-corner indentations showing cracks in soda-lime glass: (a) P_{max} = 120 mN; and (b) P_{max} = 1.3 mN.

One special threshold observation concerns the behavior of the material silicon carbide ST. A cube corner threshold could not be established for this polycrystalline material because of a transition at low loads from well-defined radial cracking to a less well-defined cracking behavior involving the pushout of individual grains from around the indenter. This behavior was also observed in other polycrystalline ceramics not included in this report.

Another notable behavior occurs when cube-corner indentations are made at relatively high loads, e.g., those typically used in microhardness testing. When tested in a microhardness tester at loads of approximately 10N (~1000 gms), most of the materials listed in Table I exhibited complete removal of large portions of material from around cube-corner indentations by the formation of extensive lateral cracks. Because of this phenomenon, which is essentially the same as the chipping threshold in Vickers indentation, the cube corner indenter is of little value at large loads. At low loads (< 500 mN), on the other hand, the cube corner indenter performs extremely well. Radial cracks form consistently in all the materials in Table I at loads well below the threshold for the Vickers and Berkovich indenters.

CORRELATION OF CRACK LENGTH WITH FRACTURE TOUGHNESS

Studies were also undertaken to determine the extent to which the lengths of cube-corner radial cracks can be correlated with fracture toughness through the use of Eqn.1. To this end, indentation testing with the cube-corner indenter was performed in both a conventional microhardness tester at loads ranging from 1000 to 10000 mN and in a nanoindentation system for loads in the range 1 - 100 mN. The average crack lengths were determined by imaging the indentations in an SEM (nanoindentations) or an optical microscope (microhardness indentations).

To test the applicability of Eqn.1, crack lengths for the cube corner indenter are plotted as a function of load in Fig.3. To be consistent with the form of Eqn.1, the crack lengths are plotted in normalized form as $K_c[H/E]^{1/2}c^{3/2}$, where the values of E, H, and K_c are those in Table I. The elastic moduli and hardnesses were measured by standard nanoindentation techniques using

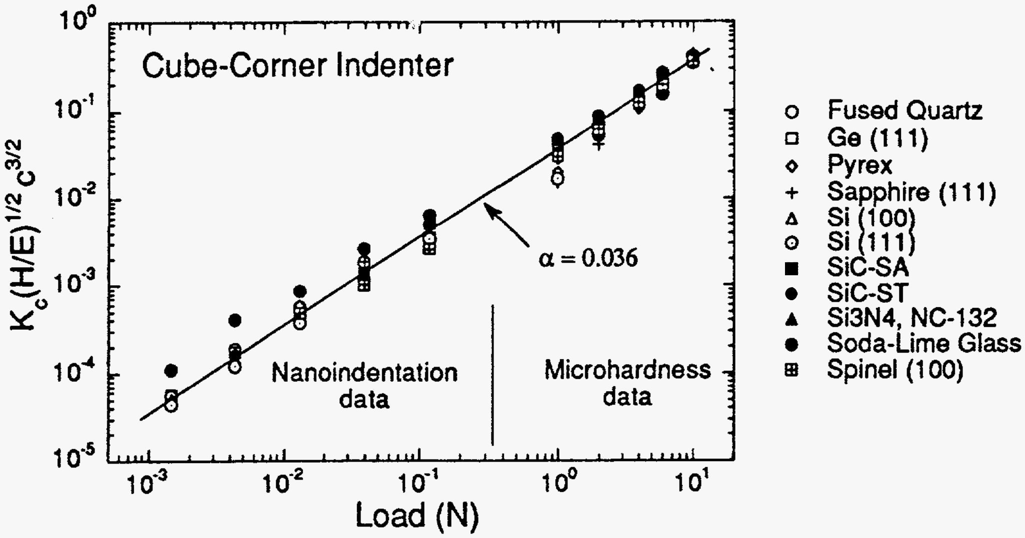


Fig.3. Plot of normalized crack length vs. applied load for the cube corner indenters.

a Berkovich indenter, and the fracture toughnesses were either measured using the 3-point bend chevron-notch method or extracted from reliable sources in the literature. If Eqn.1 is valid for the cube-corner indenter, then data from all the materials should group about a single line, the slope of which is related to the geometric constant α.

Fig.3 demonstrates that this is indeed the case. When fitted to a power law relation of the form $y = ax^n$, the data are best fit with a power law exponent $n = 1.05$, thus implying that the assumed relation between P and $c^{3/2}$ works well for the cube corner indenter over a wide range of loads. The value of the geometric constant resulting from the fit is $\alpha = 0.036$. The only material which appears to deviate in a consistent way is soda-lime glass, for which the normalized crack lengths obtained in the nanoindentation tests are slightly higher than the rest of the data. A possible explanation for this behavior is that soda-lime glass is known to exhibit environmentally-assisted, post-indentation crack growth [7,9]. Because the crack lengths for the soda-lime glass nanoindentations were measured by SEM examination several days after the indentations were produced, significant post-indentation growth may have occurred.

Another significant observation in Fig.3 is that indentation toughness measurement with the cube-corner indenter appears to work well for fused silica and Pyrex glass. This is somewhat surprising since it is well known that these materials behave anomalously when tested with a Vickers indenter; specifically, the computed toughnesses are usually too high [10]. We speculate that the better performance of the cube-corner indenter has to do with a difference in cracking mode. It was observed that the cracks which form first in fused quartz with a cube-corner indenter are radials, while those with a Vickers indenter are cone cracks. This is important because cone cracks interfere with the formation of the radials and thus influence their lengths. Thus, in addition to reducing the thresholds, there are other reasons to prefer the cube corner indenter in the measurement of fracture toughness at low loads.

Finally, to show the predicative capability of Eqn.1 in measuring fracture toughness with the cube-corner indenter, the toughness of most of the materials in Table I has been computed from Eqn.1 using $\alpha = 0.036$ and the values of E and H given in the table (note that E and H were also determined using nanoindentation methods). These toughnesses are plotted as a function of the conventionally measured toughness (see Table I) in Fig.4. For each material, the indentation toughness was determined for each of the indentations produced in the nanoindenter (loads ranging from 1 to 100 mN), and the scatter bars indicate the range of the resulting values. The figure shows that, as in the case of toughnesses measured from Vickers indentation cracks at higher loads, the fracture toughnesses computed from the cube-corner data are accurate to within approximately ±40% of the conventionally measured values.

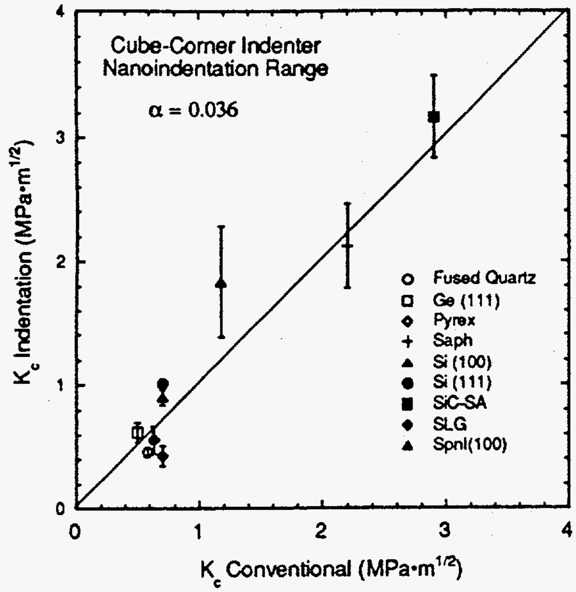


Fig.4. A comparison of the fracture toughnesses determined from cube-corner indentation crack lengths with values measured using conventional tests.

CONCLUSIONS

1. By using an indenter with the geometry of the corner of a cube, indentation cracking thresholds can be reduced relative to the Vickers and Berkovich thresholds by more than an order of magnitude, thus making it possible to produce sub-micron indentations with radial cracks in many brittle materials.

2. The crack lengths resulting from cube-corner indentations correlate with fracture toughness in a manner very similar to those for the Vickers indenter. The relation established in this work, $K_c = 0.036\,(E/H)^{1/2}\,(P/c^{3/2})$, can be used in the estimation of fracture toughness for indentations made in the micron to sub-micron range.

ACKNOWLEDGMENTS

This research was sponsored in part by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc., and by the Texas Advanced Research Program (Advanced Technology Program) under Grant No. 003652-123.

through the SHaRE program ...

REFERENCES

1. G.M. Pharr and W.C. Oliver, MRS Bulletin **17**, 28 (1992).
2. W.C. Oliver, MRS Bulletin **11**, 15 (1986).
3. M.F. Doerner and W.D. Nix, J. Mater. Res. **1**, 601 (1986).
4. W.C. Oliver and G.M. Pharr, J. Mater. Res. **7**, 1564 (1992).
5. M.J. Mayo and W.D. Nix, Acta Metall. **36**, 2183 (1988).
6. B.R. Lawn, A.G. Evans, and D.B. Marshall, J. Am. Ceram. Soc. **63**, 574 (1980).
7. G.R. Anstis, P. Chantikul, B.R. Lawn, and D.B. Marshall, J. Am. Ceram Soc. **64**, 533 (1981).
8. J. Lankford, and D.L. Davidson, J. Mater. Sci. **14**, 1662 (1979).
9. K. Hirao and M. Tomozawa, J. Am. Ceram. Soc. **70**, 497 (1987).
10. A. Arora, D.B. Marshall, B.R. Lawn, and M.V. Swain, J. Non-Cryst. Sol. **31**, 415 (1979).