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NANO-INDENTATION FRACTURE OF FUSED SILICA AND GLASSY CARBON WITH A CORNER CUBE AND 45° APICAL ANGLE INDENTERS.

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ABSTRACT:

Nano-indentation with sharp pyramidal indenters provides the ability to measure the elastic, plastic and fracture toughness properties of small volumes of material. In this study we investigate the ability to determine the fracture toughness of fused silica and a glassy carbon material, two isotropic amorphous materials. The behaviour of these materials subjected to 45° and 35.3° apical angle triangular pyramidal indenters is compared. Of particular importance is the difference in the force-displacement behaviour of these two indenters with a definitive discontinuity with the corner cube indenter. This behaviour is examined in an attempt to better understand the fracture response about the indenters. Two approaches are compared to analyse the toughness of the materials. A previous relationship with apical angle dependence is found to fir the data.

KEYWORDS

Indentation fracture, Nano-indentation, Fracture toughness, Silica, Glassy carbon,

INTRODUCTION

The use of pointed indenters to quantify the fracture toughness of brittle materials has a relatively recent history. Prior to which cracking about impressions was seen as an impediment to reliable hardness measurement. Most of the pioneering work in this field used Vickers indenters to generate the median/radial crack system about the impression from which the fracture toughness, K_c, was estimated from an expression of the form;

$$K_c = \Psi P/c^{3/2}, \qquad (1)$$

where P is the indentation load, c is the radial crack size and Ψ a constant related to the modulus to hardness ratio and an empirical constant [1]. There has been considerable discussion on the precise form of the constant Ψ as the remaining terms are precisely as expected for a center loaded penny shaped crack.

The initial work by Lawn and Swain [2] of direct optical observation of the cracking during loading and unloading of glass with Vickers indenters showed that initially a median crack formed beneath the indenter and that upon unloading radial cracks developed because of the residual stresses about the plastic impression. A critical review by Cook and Pharr [3], who also observed cracking about indentations in a wide range of transparent glasses and single crystals, suggested that in most instances the radial cracks formed upon loading. Dukino and Swain [4] compared the influence of indenter geometry, namely Vickers and Berkovich, on the extent of cracking for a number of materials. They found that the cracks were somewhat longer with the Berkovich indenter and this was expected on the basis of fracture mechanics for a center loaded star crack. More recently Harding et al [5] have pioneered the use of nano-indentation with sharper corner cube indenters to generate cracks at much lower loads typically less than 20 mN. This development enables in principle the determination of the complete elastic-plastic-fracture properties of very small volumes of materials. The aim of this current study is to compare the deformation and fracture behaviour of two sharp indenters (corner cube and 45 apical angle) on two isotropic materials namely fused silica and a glassy carbon.

EXPERIMENTAL PROCEDURE

The materials used in this study were fused silica and a glassy carbon. The properties of these materials are listed below and were highly polished with a surface finish of < 2nm.

Indentation tests were conducted with an UMIS (Ultra micro-indentation system, CSIRO Australia). The indenter tips were of diamond and apical angles of 65.3° (Berkovich), 45° and 35.3° (corner cube). Indentation tests were made from 50-1000mN.

Table 1. Properties of Materials

Material	Fused Silica	Glassy Carbon
Elastic modulus	70 GPa	30 GPa
Fracture toughness, K_{1c}	0.8 $MPa.m^{1/2}$	1.15 $MPa.m^{1/2}$

RESULTS AND DISCUSSION

Typical force-displacement data generated with the indenters on the two materials of interest are shown in Figures 1 and 2. A noticeable feature of the results for the corner cube indenter was the discontinuity in the loading portion of the force-displacement curve for both materials. In both instances there were differences in the critical load to initiate this “pop-in” event but upon initiation the curves superimposed for the remainder of the loading and unloading portions. It was also observed that the extent of the “pop-in” was greater the higher the load for the initiation of this event. The load for the onset of this discontinuity was also found to vary with the sharpness of the indenter tip. Another significant feature is the difference in the extent of the residual impression with the sharper indenters for both materials. It is also apparent that the recovery was significantly greater for the glassy carbon materials than the fused silica and decreased with sharpness of the indenter tip.

Observations of the residual impressions following indentation revealed that radial cracks always formed about the indentations in the fused silica over the load range investigated for both indenters. Whereas for the glassy carbon cracks only formed about the corner

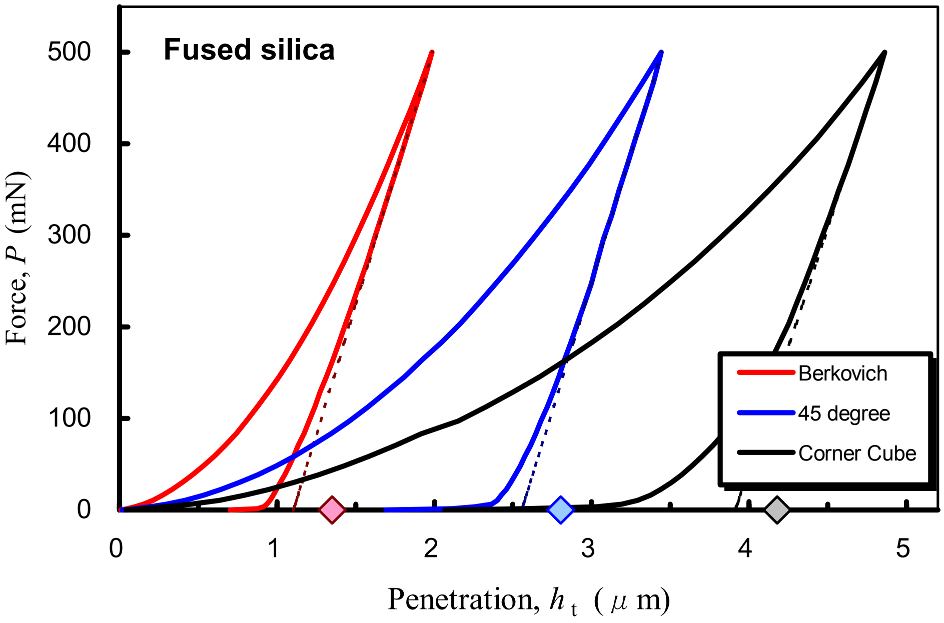

Figure 1. Force-displacement data with, Berkovich, 45˚ and corner cube indenters with fused silica, note the "pop-in" discontinuity with the latter.

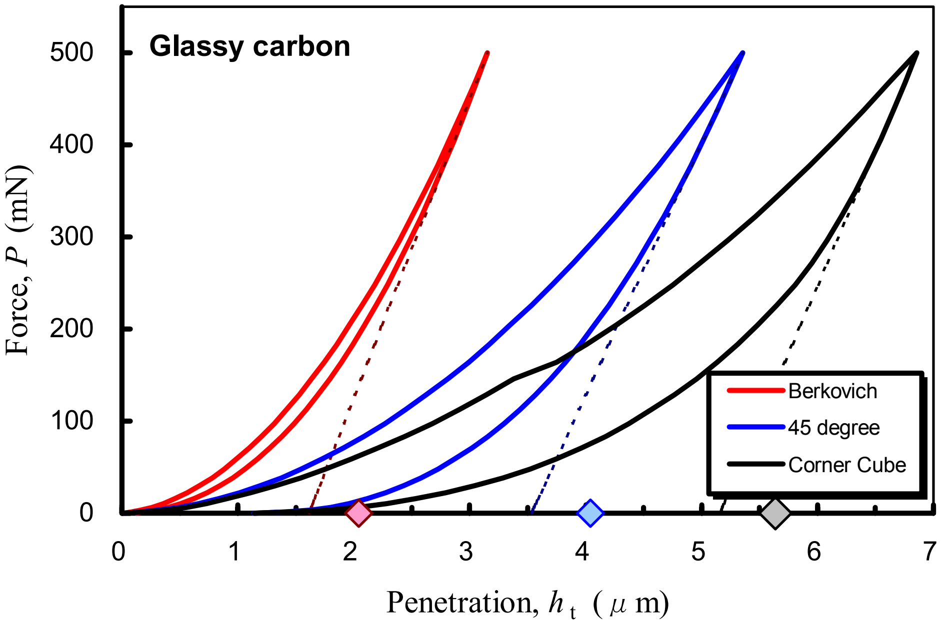

Figure 2. Force-displacement data for a glassy carbon material; comparing, Berkovich, 45 and corner cube indenters.

cube indenter and they were much more defined at loads in excess of the "pop-in" load. The radial cracks lengths were measured and the results plotted in the form expected from equation (1), that is, P versus $c^{3/2}$ and are shown in Figure 3. There is no significant difference in the case of the fused silica for radial crack lengths measured with and without the “pop-in” discontinuity. In the case of the glassy carbon the crack lengths were far more difficult to measure irrespective of whether optical or scanning electron microscopic facilities were used.

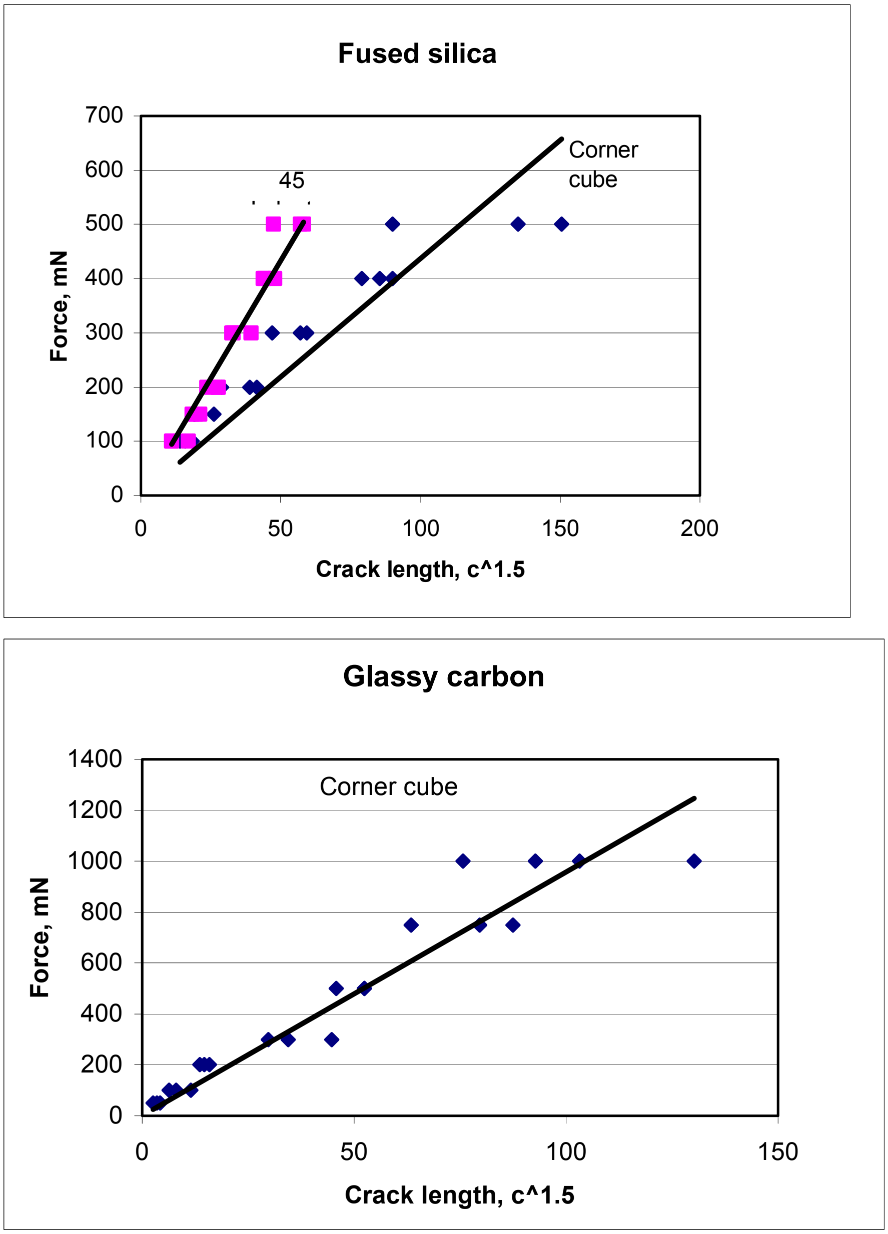

Figure 3. Crack length versus indenter load for the corner cube and 45° indenters.

The observations in Figure 3 show the equation (1) is appropriate but that the slope is dependent upon the indenter angle in the case of the fused silica. Lawn et al [2] in their initial derivation of the above relationship did include an angular dependence of the constant Ψ, namely, $\Psi = \gamma\ (E/H)^{1/2}\ (\tan\theta)^{2/3}$, where θ is the apical angle and γ is a constant. The present observations with the fused silica strongly support this relationship. Using the K_c for fused silica of 0.8 MPa√m leads to a value of γ of 0.023. Using this relationship the toughness of the glassy carbon is 1.1 MPa√m, in agreement with conventional single edge notch bend test (SENB) measurements in Table 1

A major motivation of this work was to understand the behaviour of the materials and also to relate this to the fracture about the indentation. It is well established [6] that the loading curve of a pointed indenter may be described by an expression of the form $P = kh^2$. However for all of the present data a polynomial of the form, $P = kh^2 + bh + c$, was consistently found to be a better fit for all indenters and both materials, provided the load was below the pop-in value for the corner cube indenter. A detailed consideration of why this is a better approach is given elsewhere [7], but relates to the non-perfect form of the indenter tip. The unloading response could be modeled on the basis of an elastic unloading from a residual plastic impression, or elastic reloading of a preformed (pyramidal) conical impression and leads to an expression of the form, $P = (2/\pi)E'h_e^2 \tan\beta$, where E' is the biaxial modulus, h_e the elastic displacement and β is the effective apical angle of the difference between the residual impression and the indenter (conical equivalent) angle. Below the "pop-in" load for the corner cube indenter the fit of such expressions are excellent and the recovered values of hardness and elastic modulus are in good agreement with literature values although we do find that the hardness does increase with sharper indenters. The situation is slightly more complex with the glassy carbon as this material exhibits reversible or hysteretic deformation. For loads above the "pop-in" the above approach is still found to be an excellent fit to the unload portion of the force-displacement data provided the additional depth due to the "pop-in" event is removed. The alternative approach to interpret the data for the fused silica and glassy carbon is to use the classical Oliver and Pharr [8] analysis of a pointed indenter whereby the elastic modulus and hardness can be estimated from the slope of the initial unloading curve, as shown in Figs. 1 and 2. A comparison of the estimates of the hardness and modulus from the two approaches indicated that at loads prior to "pop-in" the values were very similar but decreased slightly at loads above "pop-in".

Comparison of the fused silica and glassy carbon show that for the 45° indenter radial cracks only form with the fused silica which does exhibit a classical "plastic" residual impression and would be expected to also generate residual stresses which would drive the radial cracks to an equilibrium size. In the case of the glassy carbon the near complete recovery of the impression upon unloading would minimize the magnitude of any residual stresses and hence the likelihood for radial crack development. For the corner cube indenter the sharper indenter angle does generate a greater residual impression for both materials and hence a greater residual stress thereby assisting with the extension of the radial cracks. It is unclear whether radial cracks are present during loading with the 45° indenter in fused silica. At "pop-in" we suggest that a radial-median crack or three, quarter penny, cracks are initiated and thereafter with increasing load are wedged further open. The consequence of such a situation is that crack mouth opening displacement develops along the indenter diagonal and hence the "effective" contact bearing area is reduced and also the indenter tip is able to penetrate within the opening median cracks.

The extent of "pop-in" displacement, that is extra displacement from the simple quadratic expression for the initial loading curve, with the corner cube indenter for both the glassy carbon and fused silica scales directly with the radial crack size and indentation load above the "pop-in" threshold, Figure 4. Another noticeable feature is the energetics of the indentation. There are two components; the irreversible work expended with permanent deformation and the reversible elastic strain energy. A comparison of the energy increment per unit volume of displaced material (Δv) ($W_p = p\Delta v$, with p the contact pressure) is shown for a corner cube indentation of fused silica in Figure 5. The results are compared with a model corner cube indentation without "pop-in". Both show that initially the curves asymptote to a value of ~9 GPa and only at the onset of "pop-in" does the value jump to ~10 GPa. Other results for the 45º indenter asymptote to 9 GPa.

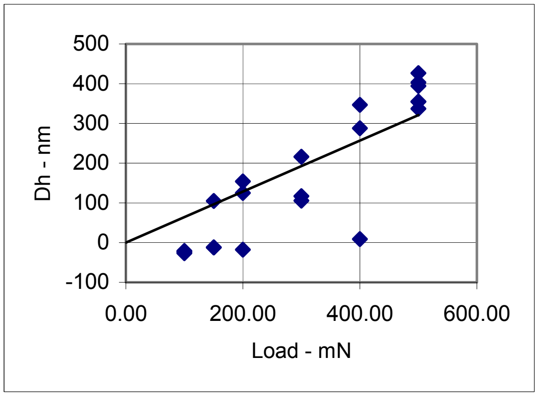

Figure 4. "Pop-in" depth versus indenter load for fused silica with corner cube indenter.

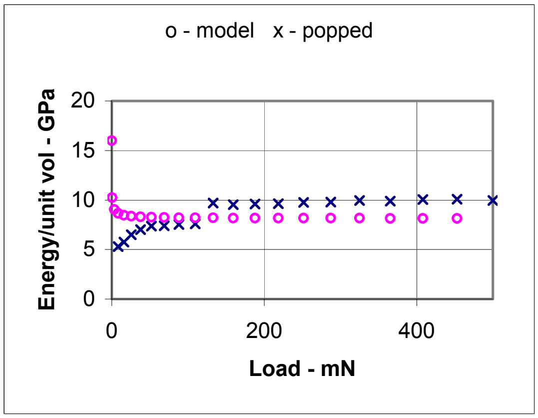

Figure 5. Change in energy per unit volume of fused silica with corner cube indenter and compared with model.

CONCLUSIONS

The present observations support the approach developed by Harding et al [5] to determine the fracture toughness of brittle materials using a corner cube indenter. There are a number of specific conclusions we can make:

1. Comparison of the determined fracture toughness using both the corner cube and 45º apical angle indenters suggests that there is an indenter angular dependence which is of the form predicted by Lawn et al [1], namely
   $$K_c = \gamma (\tan \theta)^{2/3}(E/H)^{1/2}P/c^{3/2} \quad \text{with } \gamma = 0.023.$$
2. The force displacement curves with the corner cube indenter suggest "pop-in" occurs during loading and appears to be associated with the development of a median crack (three quarter penny cracks) beneath the indenter. For the glassy carbon only with "pop-in" do radial cracks form about the impression, whereas for fused silica cracks are formed with both indenters. The hysteretic recovery of the glassy carbon and absence of residual stress associated with permanent set are considered responsible for the non-occurrence of cracking in this brittle material.
3. Although clear associations were established between 'pop-in" displacement, radial crack length and the change of the energetics, no non-optical measure of crack length from analysis of the force-displacement curves has been found.
4. It is possible to determine the intrinsic elastic and plastic properties of the materials using corner cube indenters by analysis of the force-displacement curves by removal of the additional displacement associated with "pop-in".

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