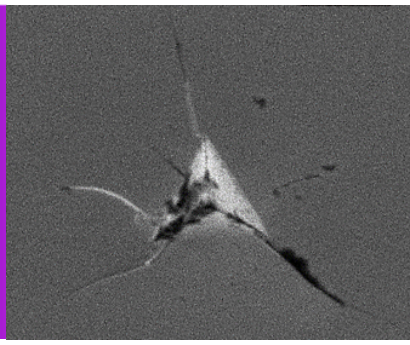
KLA INSTRUMENTS

Fracture Toughness in Bulk Materials



Introduction

Material resistance to crack growth and fracture is known as fracture toughness (K_c), which determines the structural integrity of brittle materials by describing the energy release rate during fracture. The presence of structural defects, e.g., flaws, microcracks, micro-voids, and inclusions, affects the apparent toughness in different ways. Some of these structural defects reduce toughness by increasing the crack growth rate and other defects increase resistance by blunting the crack tip through inducing micro-plasticity and local toughening in materials structure.

Nanoindentation techniques have been used extensively to evaluate the fracture toughness of brittle materials using sharp tip geometries. Developed models rely on the direct measurement of the radial cracks originating at the edge of sharp Vickers (Berkovich) or cube corner indentation marks. Here, various brittle materials were tested using different tip geometries and imaging techniques.

Experimental Method

The KLA Nano Indenter® G200 and iMicro nanoindenter were used to provide a large range of loads required for testing the materials. Fracture toughness was then determined by measuring crack length using optical and scanning features of the nanoindenter instruments.

The iMicro nanoindenter equipped with the InForce 1000 actuator was used for testing a range of very hard materials. The G200 equipped with a high load 10N force actuator was also used to create cracks in extremely hard materials that required higher forces. Indentation and cracking were carried out using the ISO 14577 constant loading rate method. Vickers and cube corner tips were used for cracking. Fracture toughness was then calculated using the well-known equation based on a linear elastic mechanics (LEM) approach:

$$K_C = \alpha \left[\frac{E}{H}\right]^{\frac{1}{2}} \left[\frac{P_{max}}{c^{1.5}}\right] \quad (1)$$

where c is the average crack length from the center of indent, H and E are the hardness and Young's modulus of the material, respectively, and P_{max} is the maximum load during indentation. The value of the coefficient α has been experimentally quantified for a series of brittle (bulk) materials and found to be ~0.016 for the Vickers 4-sided pyramidal indenter and 0.032 for a cube corner indenter. K_C is the critical value of the stress intensity factor at the crack edge necessary to produce catastrophic failure under plane-strain conditions. Lower values of K_C indicate a greater tendency toward catastrophic failure. The residual impressions from the indentations were then imaged by the optical microscope on the G200. For small indentation impressions with fine cracks on extremely hard materials or brittle thin films under very small loading, high resolution imaging is required.

Instead of using high resolution microscopy such as SEM, the Stiffness Mapping technique on the KLA G200 NanoVision Stage enabled the capture of the full length of fine cracks for the most accurate measurements.

The fracture toughness method discussed in this paper is most appropriate for brittle bulk materials where the dimensions of radial cracks are typically much larger than the size of the indentation mark. The geometry of crack systems, indenter tip geometry and material properties must all be considered. It has been shown that the ratio of E/H, or equivalently E/σ_y (where σ_y is the yield stress) plays an important role in crack geometry, which is an indicator of material brittleness (e.g. E/σ_y ~10 for brittle materials). This ratio increases as materials become more ductile, such as metallic material systems where E/σ_y ~100. In more complicated systems such as multilayers and multiple coatings, determining this ratio is challenging because of the underlying effects of substrate material properties and the presence of residual stress during manufacturing processes. In such cases, modified models or energy-based approaches are more appropriate.

Indentation Testing Results – Cube Corner Tip

Figure 1 shows images obtained by using the scanning and stiffness mapping methods on the KLA G200 NanoVision stage. The left image was generated by scanning a silica indent and shows the surface topography of the residual impression as well as cracks at each of three corners propagating across the surface. The right image was generated using stiffness mapping and shows a more visible picture of the indent and cracks along the three corners. Stiffness mapping was also used to determine the crack length for the fracture toughness calculation.

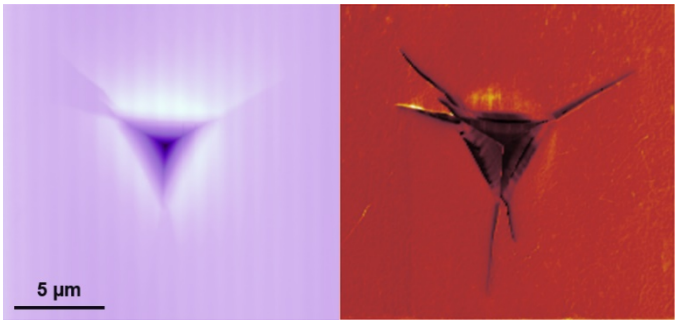


Figure 1. G200 images of a silica indent using a NanoVision stage with the scanning method (left) and the stiffness mapping method (right).

Some shorter split cracks can also be seen in the corners, along with the three primary cracks that occurred by continuous increasing of the applied load during the later stages of loading. The primary crack lengths were used for calculations. The experimental fracture toughness for a maximum load of 100mN averaged over 10 repeated tests was measured as 0.74 ± 0.09MPa·$m^{1/2}$, which agrees well with the reported value for bulk fused silica, 0.79 ± 0.01MPa·$m^{1/2}$.

A series of extremely hard ceramic carbide materials were also tested using the same technique and their fracture toughness values were also calculated. In addition to high hardness and modulus, fracture toughness (resistance to cracking) of these materials is important because of their potential application as candidate tip materials for high temperature indentation. In Figure 2, the left set of SEM images show residual indentation impressions on these materials using a sharp Cube corner tip, with measured crack length between 2-10 µm. The chart at right shows the calculated fractured toughness K_c and applied load for each material, which includes niobium carbide (NbC), sapphire, vanadium carbide (VC), titanium carbide (TiC), zirconium carbide (ZrC) and tungsten carbide (WC). Interestingly, the single crystal WC does not show any visible cracking, even at loads as high as 3N.

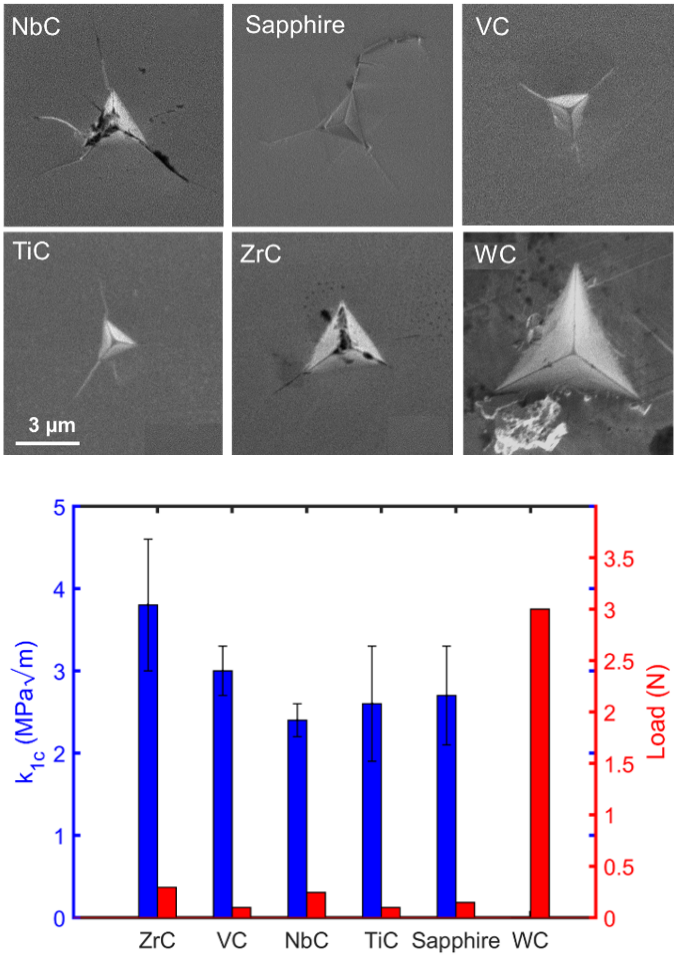


Figure 2. (top) SEM images of residual impressions for six hard materials: NbC, sapphire, VC, TiC, ZrC, and WC; (bottom) calculated fracture toughness K_c and maximum applied load for the six materials.

Indentation Testing Results – Vickers Tip

Schott BK7 (borosilicate) glass and Plexiglas (PMMA) were both indented using a Vickers tip. As shown in Figure 3, long radial cracks appeared along the indentation mark edges for the borosilicate for each of the four corners of the tip. The fracture toughness of Borosilicate was measured as 0.96 ± 0.01MPa·$m^{1/2}$.

The PMMA indentation, shown in Figure 4 (left) does not show any sign of brittle cracking along the edges of sharp Vickers tip. Plexiglas, or PMMA, is a transparent plastic known for its shatter resistance and typically exhibits ductile behavior during deformation. However, if a material is exposed to different environmental conditions, extreme changes in mechanical behavior may occur. Indentation is an effective way to probe such surface changes. When the PMMA was exposed to an acetone environment during the indentation, cracks were

initiated and propagated from indentation corners, clearly showing environmentally assisted crack growth (Figure 4, right). This phenomenon is sometimes called stress-corrosion cracking.

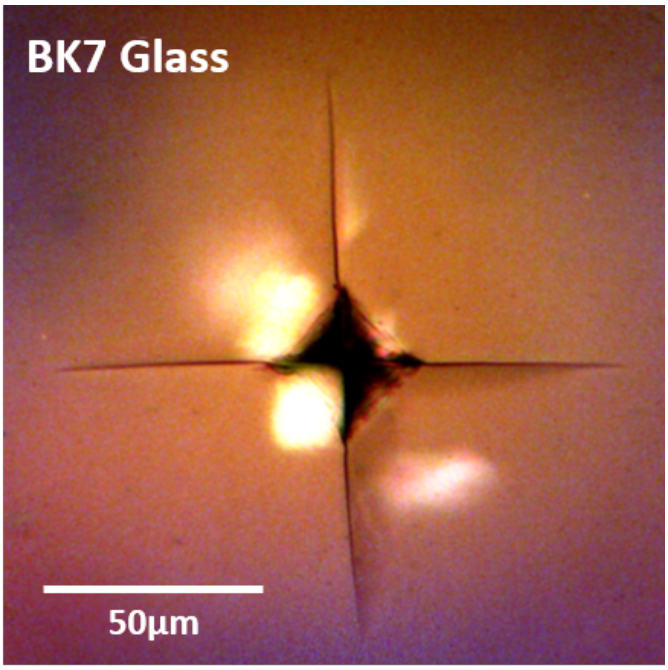


Figure 3. Indentation of borosilicate glass using a Vickers tip.

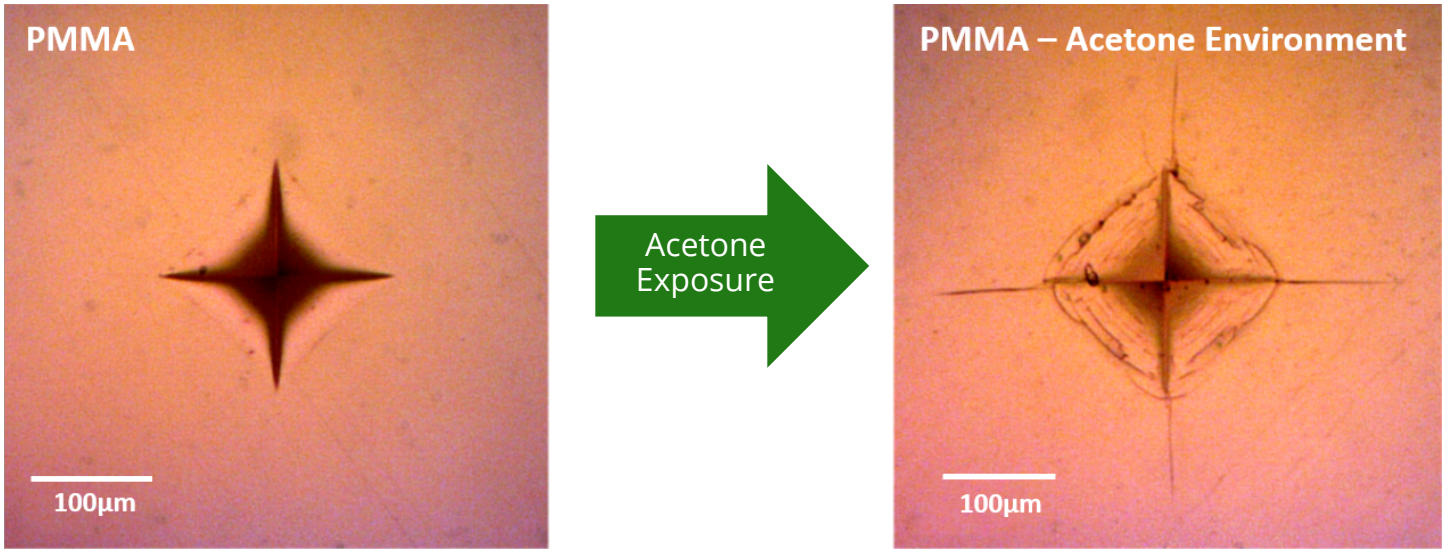


Figure 4. Indentation of Plexiglass (PMMA) using a Vickers tip, under normal conditions (left) and exposed to an acetone environment (right).

Summary

Bulk fracture toughness of various brittle materials was tested using KLA G200 and iMicro Nanoindenters. Fracture toughness was quantified by measuring material property information simply by using the ISO 14577 standard method. Imaging capability of G200 NanoVision stage enabled precise measurements of crack length for calculations. This concept using existing models or other energy dissipation approaches can be used to extend the nanoindentation application in facture toughness measurements for more complex systems.

KLA SUPPORT

Maintaining system productivity is an integral part of KLA's yield optimization solution. Efforts in this area include system maintenance, global supply chain management, cost reduction and obsolescence mitigation, system relocation, performance and productivity enhancements, and certified tool resale.

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