

Measuring fracture toughness of coatings using FIB-machined microbeams

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Abstract

Measuring the toughness of brittle coatings has always been a difficult task. Coatings are often too thin to easily prepare a freestanding sample of a defined geometry in order to use standard toughness measuring techniques. Using standard indentation techniques gives results influenced by the effect of the substrate. A new technique for measuring the toughness of coatings is described here. A precracked micro-beam was produced using FIB (Focussed Ion Beam) machining, then imaged and loaded to fracture using a nanoindenter.

Determining the mechanical properties of coatings can be very difficult due to their thinness (typically a few microns), effects of the substrate, effects of adhesion and residual stresses. Using a nanoindenter, it is possible to determine some properties of coatings (hardness and the elastic modulus)¹. Some other properties, however, are still very difficult to measure. One of them is the fracture toughness. The classical method for measuring fracture toughness is to fracture a pre-notched sample with well-defined geometry. From the critical load it is then possible to determine the fracture toughness K_{Ic} . However, in the case of thin coatings, it is difficult to manufacture a sample of the coating material alone: normal fracture toughness specimens are several 10's of millimeters in size or larger.

In the case of brittle ceramic materials, toughness is very often measured using indentation techniques. The size of cracks around the indent is measured and then related to fracture toughness using a specific model^{2,3}. This method is difficult to use for thin coatings because typical indentation crack sizes are several tens of microns to several hundreds of microns; the effect of the substrate will dominate. If the substrate is not brittle it will be difficult to get a well-defined fracture geometry. This is also the case for fracture toughness testing using Hertzian indentation⁴.

Some other methods more specific for coatings have been proposed. The use of a nanoindenter for toughness determination has been described^{5,6}, and Kim and Nairn⁷ developed a finite element fracture mechanics model to predict the development of multiple cracks in a coating layer in the presence of a substrate. These methods, however, seem to be usable only in a restricted number of cases and the values obtained are not always comparable with those obtained with other techniques. Micro-beams and similar micro-mechanical elements have been prepared for many years in silicon, mostly by etching methods^{8,9,10,11}. These methods are not easily applicable to other materials (though

silicon-etching has been used to prepare Al and Au beams by Son *et al.*¹²). Variants on these micro-beam testing methods have included FIB (Focussed Ion Beam) -machining of cantilevers of Al-coated Si from material polished to 10 μm thickness¹³, and use of FIB to put V-notches in Si specimens produced by etching methods¹⁴.

The technique used here involves the preparation and testing of pre-cracked micro-beams from “bulk” specimens, using FIB¹). The method is first demonstrated using monolithic silicon, and then applied to produce beams of a thin (10 μm) WC coating on a bulk steel substrate. Specimens are imaged and loaded to fracture using a nanoindenter.

The operating principle of a FIB system is similar to that of a SEM, the major difference being the use of a rastered gallium ion (Ga^+) beam instead of an electron beam. The ion beam removes material from the surface of the sample (milling)¹⁵. The secondary electron or ion intensity is monitored and used to generate an image of the surface. Using FIB, cantilever micro-beams can be milled out of the coating material only.

Cantilever beams were milled using a FIB system (See Fig. 1). The first step in the process (Fig. 1a) was to create the shape of the beam by milling two rectangles on the surface, about 4 μm apart and 7 μm deep. These rectangles were created using “staircase milling” and with a current of 3000 pA .

The next stage was to cut one end of the beam (Fig. 1b). A trench 7 μm deep was cut to produce a beam length of 10 μm (Fig. 1c) .

The “staircase” milling did not produce perfectly vertical sides. This was obtained in the next step of the process, milling 7 μm deep vertical sides.

The base of the beam was cut by tilting the sample to 45° (around the long axis of the beam). The base was then milled away (about 4-5 μm) (Fig. 1d) and the operation was repeated on the other side. This process created a beam with a cross section which was symmetrical on a vertical axis but not rectangular (Fig. 2).

A narrow notch was then milled at about 0.5 μm from the support of the beam by milling successively deeper rectangles in the beam to a total depth of 0.3 μm , followed by milling a line in the centre of the notch (using 1 pA current), with a depth of 1 μm . The result of this process is shown in Fig. 3. At 1 pA the FIB beam is about 8 nm wide; it is reasonable to think that the final notch width would be in the order of 10 nm . This procedure for producing a narrow notch to emulate a “sharp” crack was found to be critical to produce specimens that would give a correct K_{Ic} value, as monitored by testing Si specimens (see below).

In order to measure the fracture toughness of the beams, a nanoindenter²⁾ was used to

¹⁾ www.feicompany.com

²⁾ NanoXP, MTS Systems Corporation, 117-C Flint Road, OaK Ridge, TN 37830, www.mts.com

apply the bending load and measure the critical value at which the beam fails. The nanoindenter can scan the surface using the tip in contact mode, to get a 2D/3D image of the beam (Fig. 4). Before applying the load, a scan of the beam was performed so that the indenter tip could be accurately placed in the centre-line of the beam at a known distance from the notch.

The beam was then loaded to fracture using a loading rate of 20 $\mu\text{N/s}$. Load-deflection curves (Fig. 5) are perceptibly non-linear at low loads, as the indenter initially penetrates the beam; at higher loads, the measured displacement is dominated by beam deflection rather than the indentation process, and becomes effectively linear. Only the fracture load is required to determine K_{Ic} . After fracture, the nanoindenter continues to load the broken beam to a predetermined depth (2000 nm) before unloading.

The plane stress fracture toughness K_{Ic} can be determined using the equation:

$$K_{Ic} = \sigma_c \sqrt{\pi a} \cdot F\left(\frac{a}{b}\right) \quad (1)$$

where σ_c is the fracture stress, a is the crack length and $F(a/b)$ is a dimensionless shape factor dependent on sample geometry. We assume that only small deformations occur, hence σ can be calculated as:

$$\sigma = \frac{PLy}{I} \quad (2)$$

where P is the applied bending force (from the indenter), L is the distance between the crack and the point where the force is applied, I is the moment of inertia of the beam cross section, and y is the vertical distance between the upper surface and the neutral plane. For a beam as described in §2, I and y were calculated as:

$$I = \frac{wb^3}{12} + \left(y - \frac{b}{2}\right)^2 bw + \frac{w^4}{288} + \left[\frac{b}{6} + (b-y)\right]^2 \frac{w^2}{4} \quad (3)$$

$$y = \frac{\frac{b^2w}{2} + \frac{w^2}{4} \left(b + \frac{w}{6}\right)}{bw + \frac{w^2}{4}} \quad (4)$$

where the meaning of w and b are shown in Fig. 2.

The value of F in eq. 1, was estimated by calculating the relation between applied load and

specimen/crack geometry directly using the boundary element method (BEM)³). The equation derived from this is (for $0.3 \leq (a/b) \leq 0.5$) :

$$F(a/b) = 1.85 - 3.38 \left(\frac{a}{b}\right) + 13.24 \left(\frac{a}{b}\right)^2 - 23.26 \left(\frac{a}{b}\right)^3 + 16.8 \left(\frac{a}{b}\right)^4 \quad (5)$$

where b is the height of the parallel vertical face and a is the crack length (Fig. 2) .

K_I values were found to be effectively constant along the crack front for $a/b \approx 0.3$ (short cracks) and to have a weak peak at the centre of the crack front for $a/b > 0.3$ (longer cracks). The $F(a/b)$ value given by eq. 5 is the largest value. In these experiments $a/b = 0.46$ and the value of $F(a/b)$ was 1.58.

In order to verify the technique, silicon cantilever micro-beams were prepared with a (111) fracture plane. Along these (111) cleavage planes the fracture toughness of silicon has been reported as $0.83-0.95 \text{ MPa} \sqrt{m}$ ^{16,17} .

Tests were performed on 4 Si samples and the average toughness value was found to be $1.1 \pm 0.016 \text{ MPa} \sqrt{m}$. The value obtained is slightly higher than the expected value, and this could be due to the extra load needed to nucleate a sharp crack at the bottom of the notch. Fig. 6 shows a SEM image of the fracture surface for a silicon specimen: it can be seen that the fracture starts from the base of the FIB-machined precursor flaw. The enlargement of the precursor flaw front in Fig. 6 indicates that fracture begins near the centre of the FIB-machined flaw; i.e. where the BEM calculations give the highest intensity factor for higher a/b values, as used here.

At maximum beam deflection, the horizontal shift in the contact position due to beam curvature is $\sim 4.5 \text{ nm}$; this indenter shift should therefore have negligible influence on the test.

Having validated the method using silicon, tests were performed on a $10 \mu\text{m}$ thick CVD WC-based coating on steel⁴). Beams were machined from the coating material only. The measured fracture toughness (from 4 samples) was $3.2 \pm 0.3 \text{ MPa} \sqrt{m}$.

In summary, a novel technique for measuring the fracture toughness of materials and in particular of coatings is described. The method is based on machining a pre-cracked microbeam using Focussed Ion Beam and then loading to fracture using a nanoindenter with a scanning facility. The method gives accurate results on a reference material (Si) and hence the method can be used on a material with unknown toughness, in this case a CVD WC-based coating. The method could also be used on un-notched samples in order to measure the ultimate bending stress or on cross sections to evaluate the shear strength of an interface.

³)The software packages BES and FRANC3D by Cornell Fracture Group (www.cfg.cornell.edu) were used.

⁴) Produced by Hardide Ltd (Unit 11, Wedgwood Road, Bicester OX26 6UL, UK).

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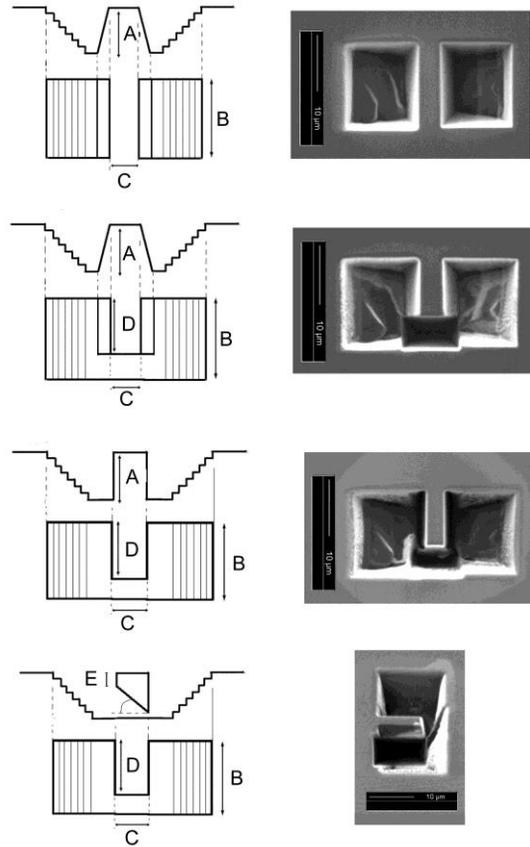


Figure 1: Sample preparation process (silicon specimen). $A = 6 \mu m$, $B = 15 \mu m$, $C = 4 \mu m$, $D = 10 \mu m$, $E = 2.8 \mu m$.

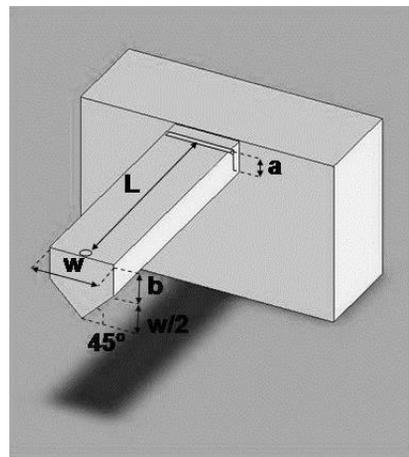


Figure 2: Final result of the sample preparation.

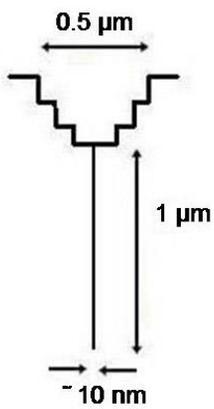


Figure 3: Cross section of the notch.

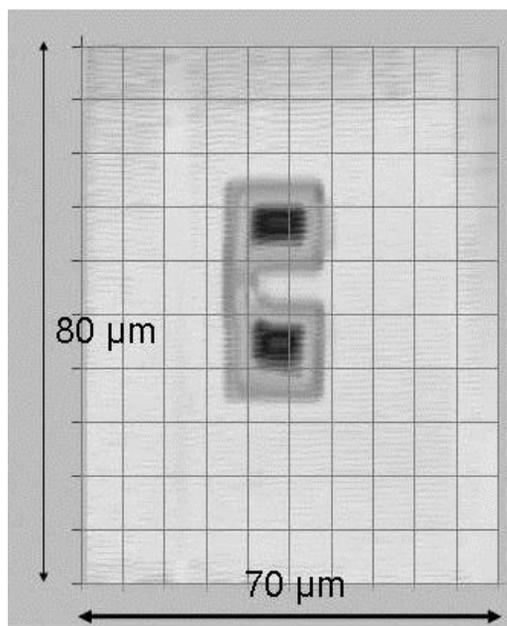


Figure 4: 2D view of the surface scan obtained using the nanoindenter tip (silicon specimen).

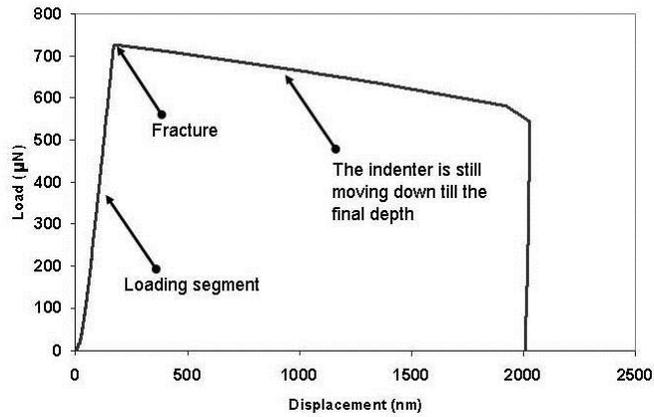


Figure 5: Typical load vs. displacement (beam deflection) graph for a silicon specimen. Only the initial portion up to the critical load value at which the beam fails is relevant for K_{Ic} determination.

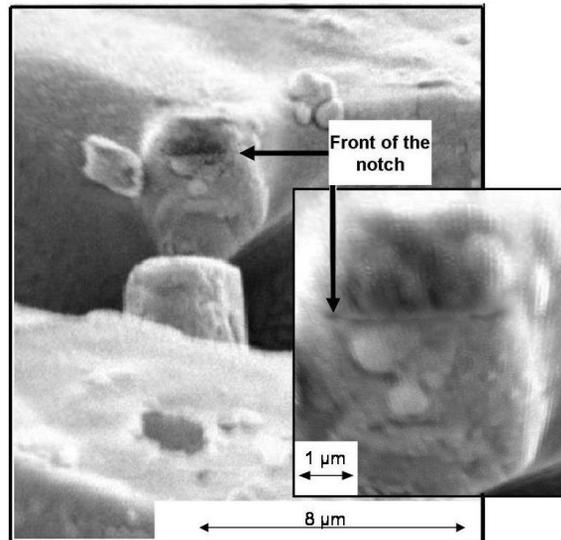


Figure 6: SEM image of the fracture surface for a silicon specimen. The initial notch is indicated.