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Measurement of fracture toughness by nanoindentation methods: Recent advances and future challenges

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ABSTRACT

In this paper, we describe recent advances and developments for the measurement of fracture toughness at small scales by the use of nanoindentation-based methods including techniques based on micro-cantilever, beam bending and micro-pillar splitting. A critical comparison of the techniques is made by testing a selected group of bulk and thin film materials. For pillar splitting, cohesive zone finite element simulations are used to validate a simple relationship between the critical load at failure, the pillar radius, and the fracture toughness for a range of material properties and coating/substrate combinations. The minimum pillar diameter required for nucleation and growth of a crack during indentation is also estimated. An analysis of pillar splitting for a film on a dissimilar substrate material shows that the critical load for splitting is relatively insensitive to the substrate compliance for a large range of material properties. Experimental results from a selected group of materials show good agreement between single cantilever and pillar splitting methods, while a discrepancy of ~25% is found between the pillar splitting technique and double-cantilever testing. It is concluded that both the micro-cantilever and pillar splitting techniques are valuable methods for micro-scale assessment of fracture toughness of brittle ceramics, provided the underlying assumptions can be validated. Although the pillar splitting method has some advantages because of the simplicity of sample preparation and testing, it is not applicable to most metals because their higher toughness prevents splitting, and in this case, micro-cantilever bend testing is preferred.

1. Introduction

Detailed characterization of the mechanical behavior of thin films and small-scale devices is of paramount importance in understanding their in-service failure mechanisms. To this end, nanoindentation has been widely used in the last two decades as a high spatial resolution micro-probe for measuring mechanical properties of materials at small scales [1,2]. Among the mechanical properties that can be quantitatively evaluated by this technique are: hardness and elastic modulus [1,2], storage and loss modulus [3], strain rate sensitivity [4], yield strength and strain hardening coefficient [5], residual stress [6], adhesive strength of coatings [7] and fracture toughness [8–27].

Numerous methods exist for the measurement of fracture toughness of small volumes of material. Indentation based methods with sharp pyramidal indenters have been widely investigated [8–15] due to the relative ease of testing and sample preparation. In such methods, the fracture toughness is determined from measurements of the lengths of cracks emanating from the residual indentation impression at a given load (Fig. 1). The choice of the model for determining the indentation fracture toughness depends on the type of crack system, e.g., median, radial, half-penny, cone, or lateral cracks [16,17], and the geometry of the pyramidal indenter. Generally, equations relating fracture toughness to applied loads and crack lengths from pyramidal indentation tests have the form of [16,17]:

$$K_c = \frac{P_{\max}}{c^{3/2}} \prod \left(\frac{E}{H}, \upsilon, \psi, \frac{c}{a} \right) \quad (1)$$

where K_c is the fracture toughness, $P_{\max}$ is the maximum indentation load, c and a are the crack length and the contact size (the distance from the center to the indentation to the corner of the contact), respectively, E is the elastic modulus, H is the hardness,

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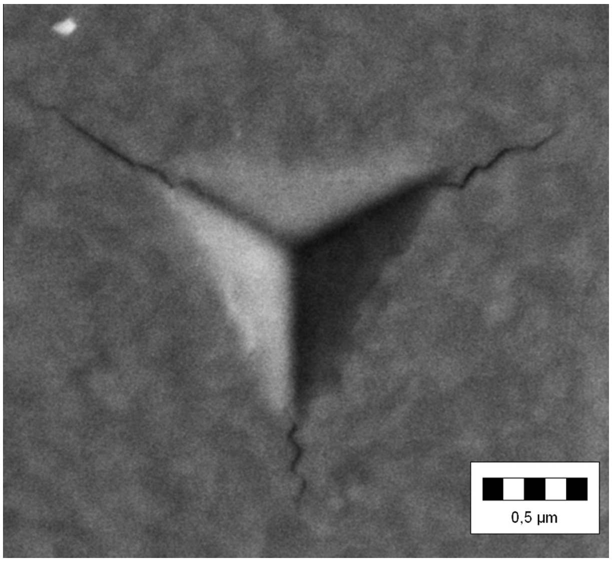

Fig. 1. Radial cracks originating at the edges of a Berkovich indentation for a titanium nitride (TiN) coating material (SEM, 5 kV, ETD SE).

ν is the Poisson's ratio, and ψ is the axis-to-face angle of the pyramidal indenter. It is clear from this equation that the selection of an appropriate model for indentation fracture toughness requires a knowledge of the elastic and plastic properties of the material, the lengths of the cracks, and the indenter geometry. A simplified analysis can be performed in the case of long cracks ($c/a \gg 1$), which leads to the classic Lawn–Evans–Marshall (LEM) model [8,9]:

$$K_c = \alpha \cdot \sqrt{\frac{E}{H}} \cdot \frac{P_{\max}}{c^{3/2}} \tag{2}$$

The value of the LEM coefficient, α, has been experimentally calibrated over a number of bulk, brittle materials and found to be ~0.016 for the Vickers 4-sided pyramidal indenter. However, recent studies of indentation cracking with cohesive finite element calculations [16,17] have shown that α depends significantly on the ratio of E/H, Poisson's ratio, and indenter geometry. This is because median type cracking dominates at low E/H ratios and Palmqvist type cracking at higher ratios [17], and this changeover in mechanism has significant implications for how the toughness is related to the crack length. Consequently, the choice of the most appropriate model for toughness evaluation from radial crack measurements is complex. In addition, the LEM model requires accurate measurements of crack lengths, which can sometimes be difficult at small length scales. Critical issues associated with indentation cracking methods based on observations from cohesive zone finite element simulations have recently been discussed in detail [16,17].

In case of thin films, the measurement of fracture toughness is made even more complex by influences of the substrate, which may enhance or inhibit plastic zone development and crack development, especially as the size of the contact approaches the thickness of the film. In addition, the large residual stresses that can exist in thin films can substantially alter the cracking behavior, making the use of indentation-based techniques unsuitable in practice unless the residual stresses are precisely known by other methods [26,36]. The establishment of a correlation between measured quantities and actual in-service failure modes is even more complicated, since it arises from a complex interaction between the intrinsic resistance to fracture and the residual stress field present in the film [26]. Microstructural features (e.g., grain size and distribution, defect density, and the substrate/coating interface) further contribute to the complexity of the problem.

A new class of techniques has recently been developed to resolve some of the issues associated with indentation based fracture toughness measurements of films. The methods generally use a nanoindentation system to apply force to and measure the displacements of micro-scale mechanical test specimens of various geometry produced by focused ion beam (FIB) milling.

The specimen geometries used in these tests include single cantilever beams [18,20–22,49,50], clamped beams [19,27], double cantilever beams [23], membranes [24,25], and pillars [26]. In case of the single-cantilever beam specimens, a pre-notched micro-specimen is deformed in bending until crack propagation is induced, as shown in Fig. 2. An analytical model is then used to calculate the fracture toughness from measured values of the critical loads for crack extension, crack lengths, and specimen sizes using:

$$K_c = \sigma_c \cdot \sqrt{\pi a} \cdot F\left(\frac{a}{b}\right) \tag{3}$$

where σ_c is the fracture stress, a is the crack length and $F(a/b)$ is a dimensionless shape factor that depends on sample geometry, which is illustrated in Fig. 2 [18]. In addition to monolithic materials and thin films, cantilever-bending methods have been used to test the interfacial toughness of grain boundaries [21] and the adhesion of coatings [22].

Double cantilever beam testing at small scales can be conducted in a simple compression experiment using a specimen with a special geometry like that shown in Fig. 3 [23]. Such tests are typically performed in situ (i.e., inside a scanning electron microscope) on FIB-milled, pre-notched double-cantilever beams, like that shown in Fig. 3. The fracture toughness of such specimens is evaluated from the compression load at crack extension using:

$$K_c = \sqrt{3}\,\frac{(e - \mu h)}{l d^{3/2}} P_c \tag{4}$$

where l is the width of the specimen, e is the distance between the line of action of the load P and the neutral axis of the beam, P_c is the critical load for crack extension and μ is the friction coefficient between the flat punch indenter, used to apply the compression to the specimen [23]. Multiple load–unload cycles are usually performed in order to study the influences of friction and plasticity on the toughness measurements. One of the drawbacks of cantilever-based methods is that specimen preparation by FIB milling may induce structural damage, especially at the root of the stress-concentrating notch, where it may influence the crack propagation load. In addition, for the double cantilever method, a tedious calibration procedure is required to estimate the friction coefficient between the indenter and the specimen. In a research environment,

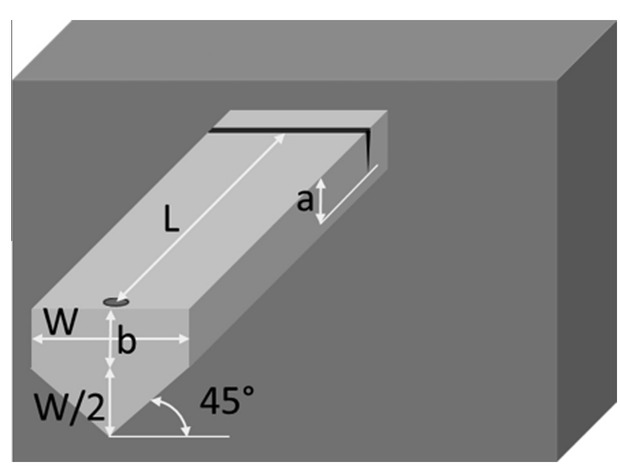

Fig. 2. Schematic representation of the micro-cantilever bending geometry (reproduced from [18] with permission of the authors).