



In-situ and Small-Volume Fracture Toughness Measurement via Nanoindentation

For hardness testing via nano/micro indentation, any cracking caused by sharp indenter tip and excessive load is undesirable and will lead to questionable data. However, such phenomenon and capability has been proven to be very useful in evaluating one of the critical mechanical properties of materials: fracture toughness. Fracture toughness is a measure of the materials ability to resist crack propagation and fracture under stress. Commonly used methods for evaluating fracture toughness of materials include bending, tension and impact tests of a specimen with a sharp crack or a defined notch. As regulated and recommended by many ASTM and international testing standards, these methods require the specimen with sufficient thickness and dimensions to ensure measurement validity. In many industrial and technical applications that involve small volume of materials, however, these requirements could not be practically met, for example in thin films, coatings, welds and miniaturized devices. The unmet needs by the conventional fracture toughness measurement methods have offered an excellent opportunity for the nanoindentation based techniques that are developed for mechanical characterization of small volume materials at nanoscale. Benefited from the established model and in-situ scanning probe microscopy (SPM) imaging capability, fracture toughness measurement via nanoindentation has become a preferred technique for in-situ and small-volume fracture behavior study of materials.

To measure fracture toughness of small volume of materials, a relative high load is chosen for a nanoindentation routine with the goal of creating cracks at the corners of the indent. Then the indented surface is imaged using the nanoindenter's in-situ SPM imaging function to capture the fine features of corner cracks of the indent as shown in Figure 1. The fracture toughness of the material is calculated using the following equation:

$$Eq. 1 \quad K_c = \alpha \left[\frac{E}{H} \right]^{1/2} \left[\frac{P}{c^{3/2}} \right]$$

Where K_c is the indentation fracture toughness; E is elastic Young's modulus, H is the hardness, P is the peak load and c is the average crack length from the center of the indent to the tips of the cracks. The constant,

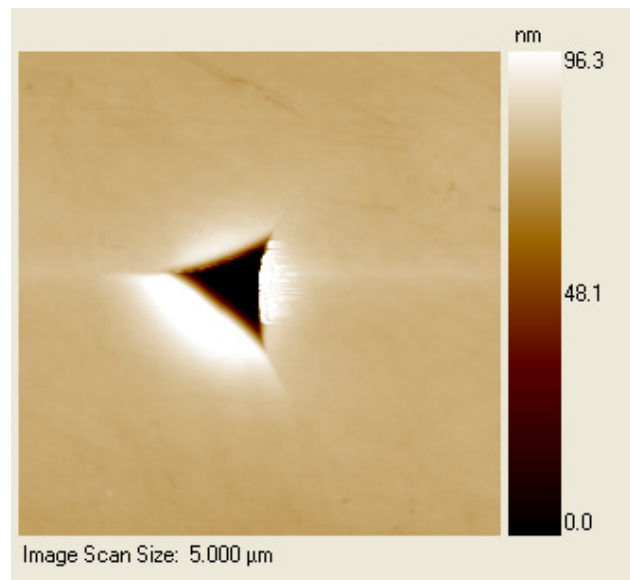


Figure 1. In-situ SPM image of an indent with cracks propagating from each of the corners for fracture toughness measurement.



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α , is a value related to the tip geometry and its values are known for cube cornered, Berkovich and Vickers tips.

Very conveniently, the hardness values required for determining the fracture toughness can be easily measured by using a smaller load and performing the normal nanoindentation test without causing any cracks. The Young's modulus is derived from the reduced elastic modulus determined through nanoindentation and the Poisson's ratio of the material. The crack length is measured from the center of the indent for all formed cracks using imaging software. Table 1 presents indentation fracture toughness values of a few specimens made of different materials using nanoindentation method.

Table 1 Fracture Toughness Determined using Nanoindentation Method

Specimen	Load (mN)	H (GPa)	E (GPa)	Crack Length (m)	K_c (MPa*m ^{1/2})
Gelatin Capsule	7.5	0.33	6.70	4.08E-06	0.13
Fused Quartz	8.0	9.52	71.96	1.06E-06	0.64
Silicon Nitride	16.0	12.69	188.48	1.17E-06	1.57
Silicon Wafer	3.0	10.69	142.88	5.11E-07	0.96