



# Stiffness Mapping and the Calculation of Fracture Toughness of Fused Silica

## Application Note

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### Introduction

The determination of mechanical properties for small volumes of material is becoming increasingly important for many facets of science and technology, both industrial and academic. Nanoindentation equipment from Agilent Technologies has been used in a variety of ways to study the mechanical behavior of materials. Fracture toughness is an important property that can be evaluated with an Agilent nanoindenter. Fracture toughness, represented by the symbol  $K_{IC}$ , is the critical value of the stress-intensity factor at a crack tip necessary to produce catastrophic failure under plane-strain conditions. Lower values of  $K_{IC}$  indicate a greater tendency toward catastrophic failure due to a pre-existing flaw [1]. With the newly released Stiffness Mapping method from Agilent Technologies, evaluating fracture toughness by nanoindentation has never been easier.

### Experimental Procedure

The purpose of the testing was to determine the fracture toughness of fused silica. The first step in evaluating fracture toughness is to indent the material in such a manner as to induce cracking at the surface.

The cube corner was chosen for this purpose due to the high stress it imposes in the vicinity of contact.

Three peak loads were used for this application note 100 mN, 125 mN and 150 mN. Ten indentations were performed at each load. The residual impressions were then imaged by Agilent's new stiffness mapping method. Each indent featured cracking at each of the three corners and these crack lengths were measured. Each crack length was used as an input into the fracture toughness equation.

Once the crack lengths were determined, elastic modulus and hardness of the fused silica were measured with a Berkovich tip. Once the mechanical properties were calculated the fracture toughness was determined according to the following equation [2]:

$$K_{IC} = \alpha \left[ \frac{E}{H} \right]^{\frac{1}{2}} \left[ \frac{P_{\max}}{c^{1.5}} \right], \quad \text{Eq.1}$$

Where  $\alpha$  is a geometric constant whose value is 0.032 for a cube corner indenter [3],  $E$  is Young's modulus,  $H$  is hardness,  $P_{\max}$  is peak load and  $c$  is crack length.



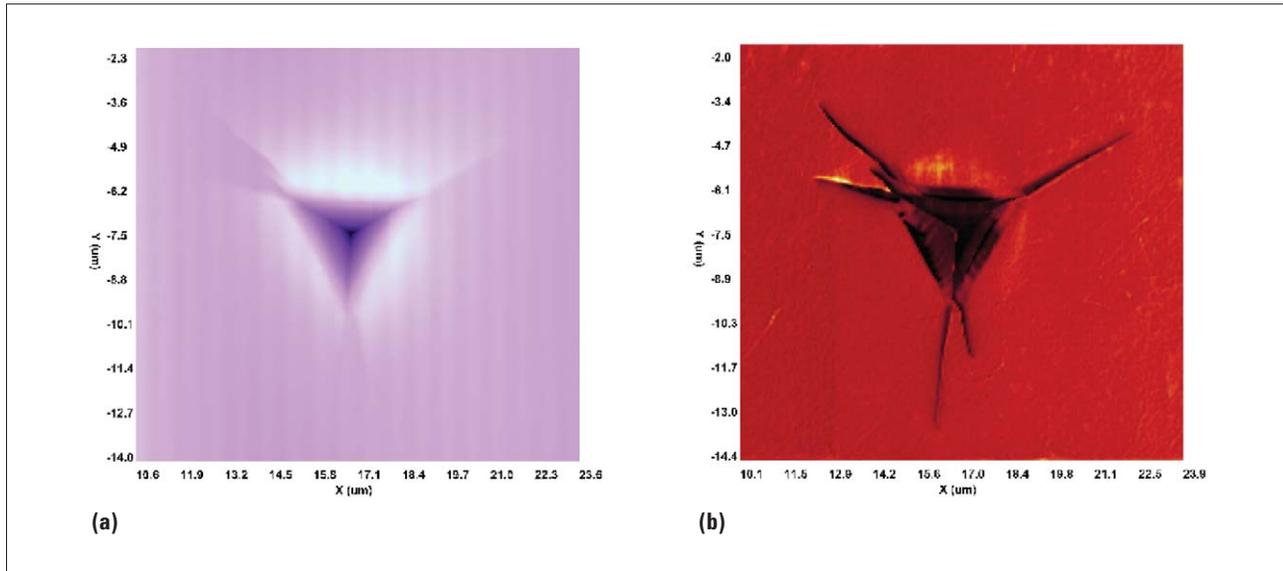


Figure 1. Comparison of (a) topography and (b) stiffness maps.

## Results

An image representative of the scanned indents is shown in Figure 1. Pythagoreans Theorem was used to determine the crack lengths. Some of the indentations produced split cracks which can be seen in Figure 1. When the cracks first begin to propagate, primary cracking, they emanate from the three corners of the pyramidal indenter where stress is highly concentrated. Secondary cracking which produces split cracks may be seen emanating from the corner of the residual impression or from a primary crack. When secondary cracks propagate from the corners it can be difficult to decide which is the primary and which is the secondary crack. In this case it is best to calculate an average length for the primary and secondary cracks at the corner of interest. When secondary cracks propagate from a primary crack they are offset by some angle. Here

only the length of the primary crack emanating from the corner of the residual impression was measured.

After the inputs to Eq. 1 were determined the fracture toughness of fused silica was calculated and the tabulated results are shown in Table 1. A graphical representation is provided in Figure 2.

The experimental fracture toughness values from Table 1 agree well with a reported value range of  $0.73\text{MPa}\sqrt{\text{m}} - 0.80\text{MPa}\sqrt{\text{m}}$  for bulk fused silica [3]. The calculated results

were easily acquired using Agilent's Stiffness Mapping method. Stiffness mapping utilizes the Continuous Stiffness Measurement (CSM) technique to measure stiffness as a function of position. As the indenter tip scans the surface of a bulk material the stiffness remains constant. As the tip moves into a crack the contact area of the tip increases resulting in a large increase in stiffness relative to that of the material's surface. This allows for clearly visible cracks in the stiffness mapping image.

	$K_{IC}$ 100mN	$K_{IC}$ 125mN	$K_{IC}$ 150mN
	MPa $\text{m}^{0.5}$	MPa $\text{m}^{0.5}$	MPa $\text{m}^{0.5}$
Average $K_{IC}$	0.660	0.689	0.703
Standard Deviation	0.104	0.147	0.135

Table 1. Fracture Toughness Results

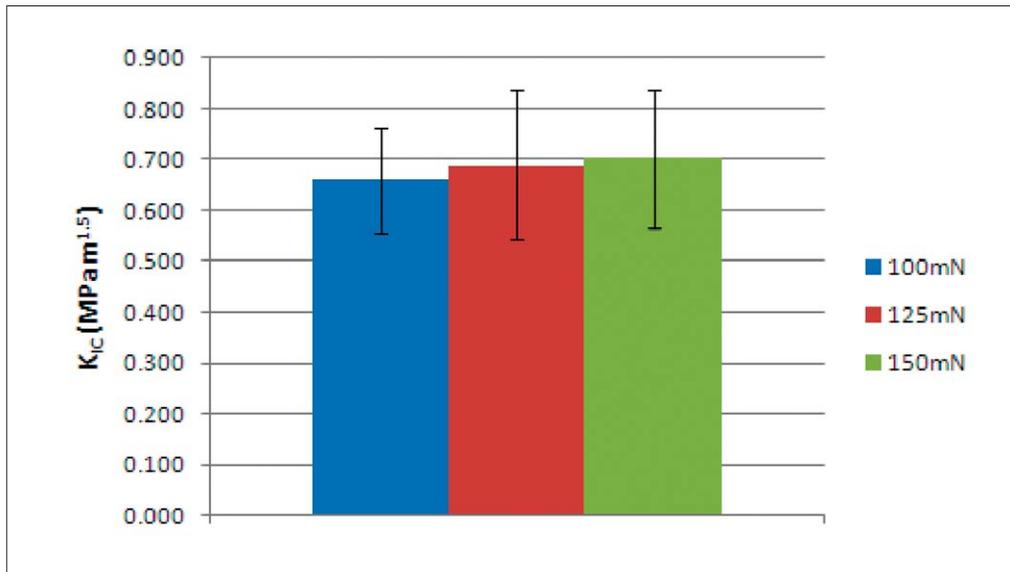


Figure 2.  $K_{IC}$  as a function of peak load.

Fracture toughness may also be calculated using a topological map. However, the cracks are not as visible because the change in topography between surface and crack is not large. The difference in crack visibility between topography and stiffness is illustrated in Figure 1. Because the relative change in stiffness is greater than the relative change in topography, stiffness mapping is the preferred technique when evaluating fracture toughness of materials.

### Conclusion

Topological or stiffness maps may be used to calculate fracture toughness. However, stiffness mapping is the preferred technique due to the clarity of cracks in the resultant image. The crack lengths can be measured using Pythagoreans Theorem. Once the crack lengths and mechanical properties have been determined Eq. 1 may be used to calculate the fracture toughness. The experimental fracture toughness values shown in Table 1 agree well with a reported value range of  $0.73MPa\sqrt{m} - 0.80MPa\sqrt{m}$  for bulk fused silica [3].

## References

1. J.F. Shackelford, *Introduction to Material Science for Engineers*, pp. 330-331, Macmillan Publishing Company, New York, 1988.
2. G.M. Pharr, D.S. Harding & W.C. Oliver, "Measurement of Fracture Toughness in Thin Films and Small Volumes Using Nanoindentation Methods", *Mechanical Properties and Deformation Behavior of Materials Having Ultra-Fine Microstructures*. Pp. 449–61 in NATO ASI Series, Series E: Applied Sciences, No. 233. Edited by M. Nastasi, D.M. Parkin, and H. Gleiter. Kluwer Academic Publishers, Dordrecht, Netherlands, 1993.
3. D. J. Morris and R.F. Cook, "In-Situ Cube-Corner Indentation of Soda-Lime Glass and Fused Silica", *J. Am. Ceram. Soc.*, vol. 87, no. 8, pp. 1494–1501, 2004.

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