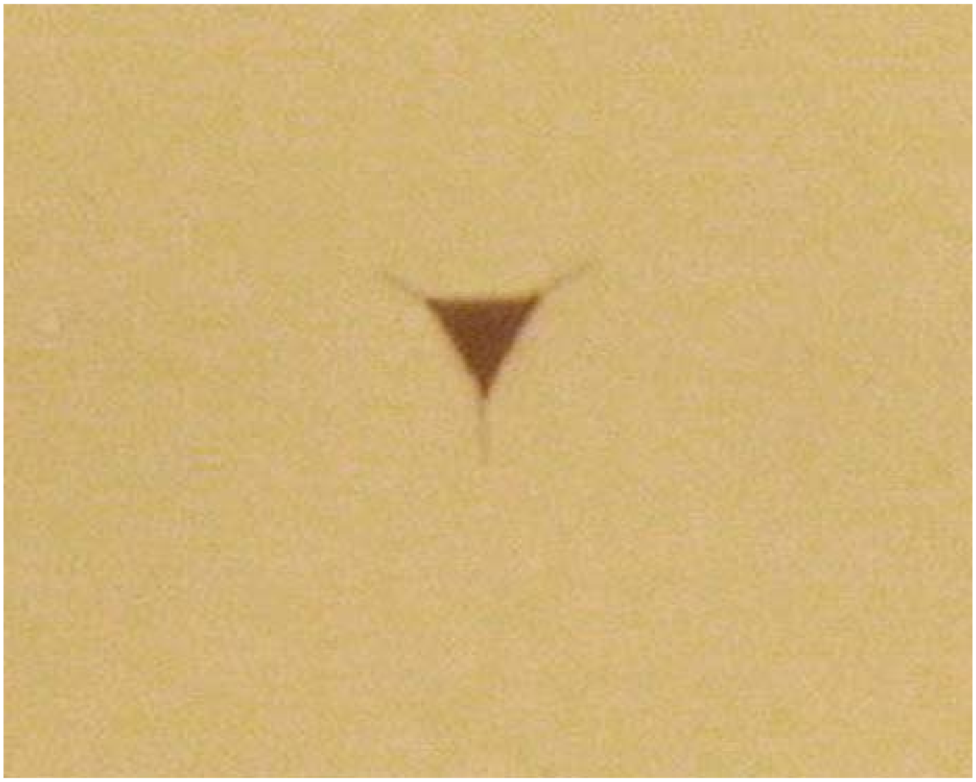
NANOVEA®
A Better Measure.

NANOINDENTATION
FRACTURE TOUGHNESS



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INTRO

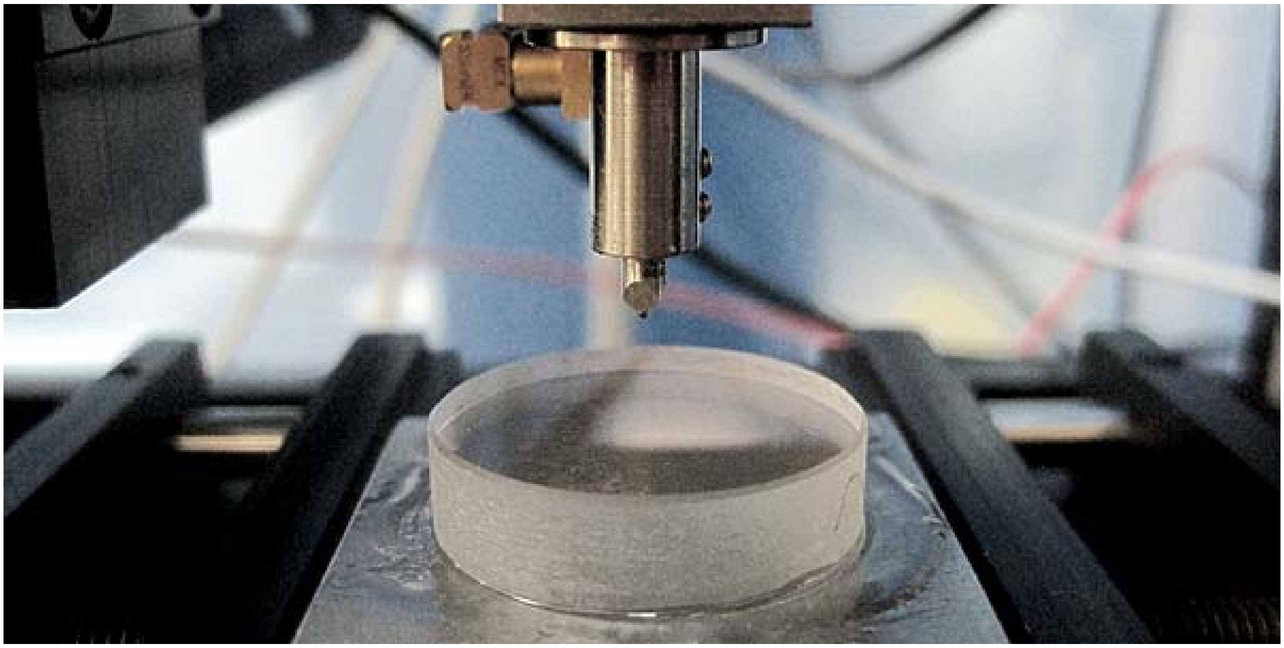
The ability of a material to resist cracking, or fracture, has been vital to the studies of fracture mechanics. Until recently the study of fracture toughness has been analyzed at a macro range using powerful instrumentation applied to large samples. Now using the precise capability of a Nanoindentation system, among many other properties, fracture toughness can be studied with a safe and user friendly system on small and localized areas of samples.

IMPORTANCE OF NANOINDENTATION FOR QC AND R&D APPLICATIONS

Nanoindentation fracture toughness is critical when measurement is limited to a small area or feature, such as thin films and small micro features, although also effective and safer for quickly studying large bulk materials traditional measured at the larger scale. The highly controlled applied loads and positioning of the Nanoindentation system can provide sensitive load/fracture initiation and crack measurement. Nanoindentation provides a reliable and user-friendly method to quickly investigate fracture toughness in addition to an endless list of additional measurements including: hardness, elastic modulus, compression, fracture toughness and many others.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode, is used to study the fracture toughness of a fused silica sample. The sample was chosen for its commonly recognized fracture toughness valuse to display the control and accuracy at the nano scale. A Berkovich tip was used for hardness and a Cube-Corner indenter tip was used for fracture testing



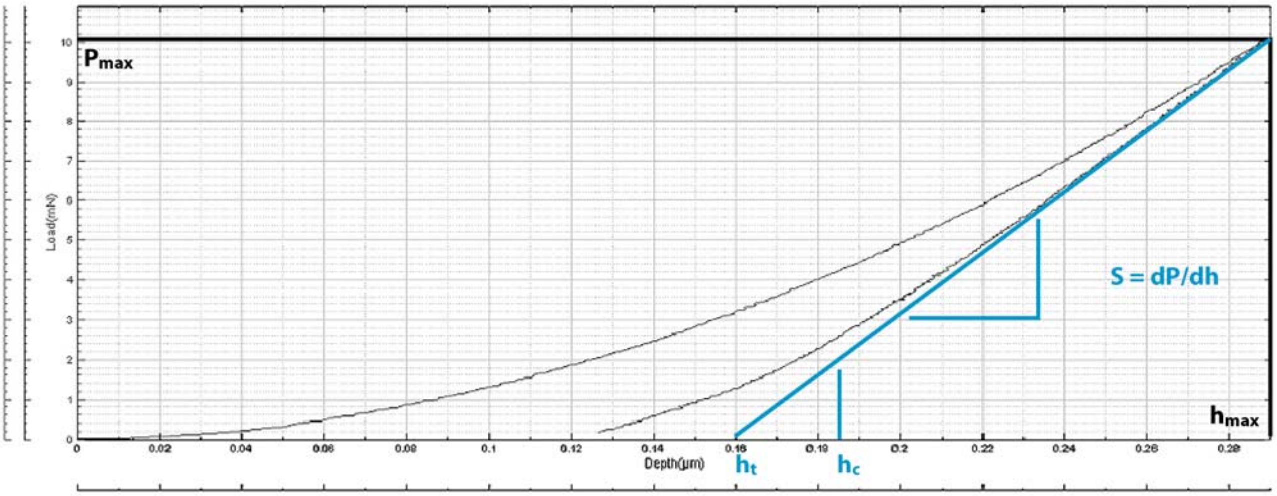
MEASUREMENT PRINCIPAL

Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. The load is applied by a piezo actuator and the load is measured in a controlled loop with a high sensitivity load cell. During the experiment the position of the indenter relative to the sample surface is precisely monitored with high precision capacitive sensor.The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data. Nanoindentation is especially suited to load and penetration depth measurements at nanometer scales and has the following specifications:

Maximum displacement (Dual Range)	: 50 µm or 250µm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 µN
Load Resolution (Noise Floor)	: 1.5 µN

Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.



Hardness

The hardness is determined from the maximum load, P_{max}, divided by the projected contact area, A_c:

$$H = \frac{P_{\max}}{A_c}$$

Young’s Modulus

The reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$

Which can be calculated having derived S and A_C from the indentation curve using the area function, A_C being the projected contact area. The Young’s modulus, E, can then be obtained from:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

Where E_i and ν_i are the Young’s modulus and Poisson coefficient of the indenter and ν the Poisson coefficient of the tested sample.

How are these calculated?

A power-law fit through the upper 1/3 to1/2 of the unloading data intersects the depth axis at h_t. The stiffness, S, is given by the slope of this line. The contact depth, h_c, is then calculated as:

$$h_c = h_{\max} - {}^{3P_{\max}}\!/_{4S}$$

The contact Area Ac is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is $A_c=24.5h_c^2$ For Cube Corner indenter, the area function is $A_c=2.60h_c^2$ For Spherical indenter, the area function is $A_c=2\pi Rh_c$ where R is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant E, given the formula: $\sigma = E\varepsilon$ where σ is the stress, E is the elastic modulus of the material, and ε is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate relationship can be given as,

$$\sigma = \eta\frac{d\varepsilon}{dt}$$

where σ is the stress, η is the viscosity of the material, and dε/dt is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen. Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip might be a better choice.

Other tests possible includes the following:

Stress-Strain & Yield Stress, Creep, Compression strength and Fatigue testing and many others.

TEST CONDITIONS & PROCEDURES

The following indentation parameters were used:

Applied Force (mN)	60
Loading rate (mN/min)	120
Unloading rate (mN/min)	120
Indenter type	Berkovich & Cube Corner

RESULTS

Fracture toughness is calculated using the following equation:

$$K_C = \alpha\left(\frac{E}{H}\right)^{\frac{1}{2}}\left(\frac{P}{c^{\frac{3}{2}}}\right)$$

where α is an empirical constant for a specified indenter, *E* is the Young's Modulus, *H* is the hardness of the fused silica, *P* is the applied load, and c is the crack length.

Obtaining Hardness and Young's Modulus by testing with Berkovich Indenter

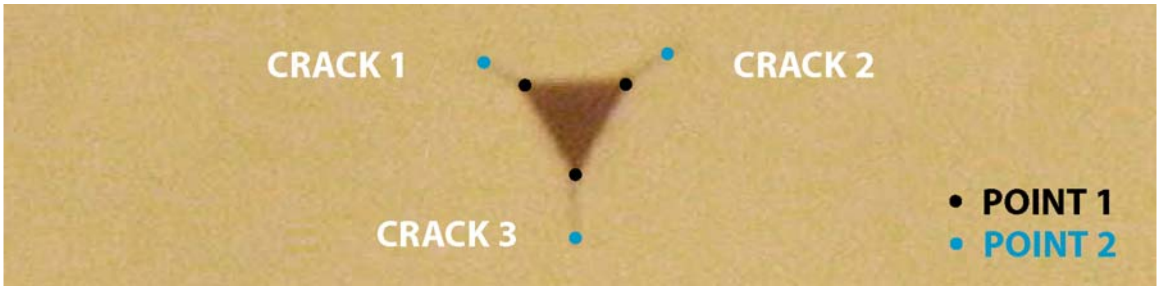
At an applied force P of 60 mN, the fused silica was tested using a Berkovich indenter to obtain the Young's Modulus *E* and hardness *H*.

Test	Young's Modulus [GPa]	Hardness [GPa]
1	74.14	9.16
2	74.61	9.42
3	74.58	9.36
Average	**74.44**	**9.31**
St Dev	**0.09**	**0.05**

Testing with Cube-Corner Indenter and Calculating Crack Length

Using the cube-corner indenter, three tests were performed and the coordinates at Point 1 and 2 for each cracked corner of each indent were obtained as shown in Figure 1. Then the crack length *c* was calculated from these coordinates using the Pythagorean's Theorem.

$$c = \sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2}$$



Test 1					
	Point 1		Point 2		Crack Length c [mm]
	x_1	y_1	x_2	y_2	
Crack 1	64.983	65.309	64.981	65.310	0.002
Crack 2	64.987	65.308	64.991	65.309	0.004
Crack 3	64.985	65.303	64.985	65.299	0.004
				Average	**0.003**

Test 2					
	Point 1		Point 2		Crack Length c [mm]
	x_1	y_1	x_2	y_2	
Crack 1	65.130	65.355	65.127	65.355	0.003
Crack 2	65.136	65.357	65.138	65.359	0.003
Crack 3	65.134	65.354	65.134	65.347	0.007
				Average	**0.004**

Test 3					
	Point 1		Point 2		Crack Length c [mm]
	x_1	y_1	x_2	y_2	
Crack 1	65.191	65.407	65.192	65.409	0.002
Crack 2	65.187	65.405	65.185	65.405	0.002
Crack 3	65.190	65.403	65.190	65.399	0.004
				Average	**0.003**

Calculating Fracture Toughness

For a cube-corner indenter, the empirical constant α is 0.032. The applied force P is 60 mN. The average values of Young's Modulus E and hardness H are 74.44 GPa and 9.31 GPa. Taking these constant values and the average crack length of each indent, fracture toughness K_c was calculated.

Test	Average Crack Length c [m]	Fracture Toughness K_c [MPa√m]
1	3.00×10^{-6}	1.04
2	4.00×10^{-6}	0.68
3	3.00×10^{-6}	1.04
Average	$\mathbf{3.33 \times 10^{-6}}$	**0.92**
St Dev	$\mathbf{5.77 \times 10^{-7}}$	**0.21**

CONCLUSION

In conclusion, we have shown that the Nanovea Mechanical Tester, in Nanoindentation Mode, can be used to quickly provide reliable fracture toughness data. It is to be noted that the length of the cracks could have been better determined using the AFM module integration. Taking the precision measurement of the length of the cracks in account, the value of 0.92+/-0.21MPa/ $m^{1/2}$ falls in the range of the accepted value of about 0.79MPa/$m^{1/2}$. It is to be noted that we believe the Nanovea Nano Mechanical Tester that does a fast control loop feedback on the load to ensure constant loading, even when cracking occurs, leads to increased reproducibility in fracture toughness tests. This may explain why some published papers mention that this technique is not reliably reproducible. Unlike other Nanoindentation systems, that have no feedback loop on the load, the Nanovea Nano Mechanical Tester is equipped with a fast piezo-electric actuator in combination with a high precision load cell to provide a higher level of reliability. To Learn more about Nanovea Nanoindentation.