MICROINDENTATION & FRACTURE OF MINERAL ROCK

Prepared by
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INTRO

The study of the mechanical behavior of rock and rock masses, known as rock mechanics, has endless commercial value being applied in mining, drilling, reservoir production, civil construction (concrete) and many others. With advances in technology and instrumentation precise measurement of mechanical properties is allowing for the improvement of parts and procedures used within theses commercial industries. By understanding rock mechanical properties, at micro scale, necessary steps can be made to insure successful quality control procedures.

THE IMPORTANCE OF MICROINDENTATION REASEARCH AND QUALITY CONTROL

The use of Microindentation has proven to be a crucial tool for rock mechanics related studies. For example, Microindentation has been used to advance studies in excavation by allowing further understanding of rock mass properties and its separation. Microindentation has been used to advance drilling studies to improve drill heads and improve drilling procedures. Microindentation has also been used to study chalk and powder formation from minerals. With the use of Microindentation studies can include Hardness, Young's Modulus, Creep, Stress-Strain, Fracture Toughness, Compression and others with a single instrument.

MEASUREMENT OBJECTIVE

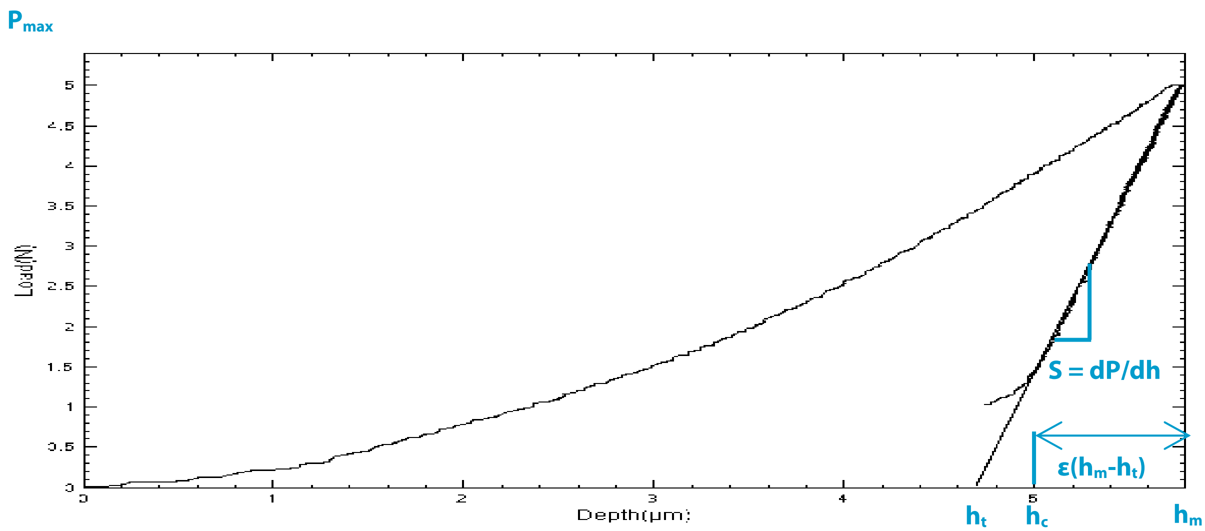
In this application, the Nanovea Mechanical Tester, in Microindentation mode, is used to measure the Vickers hardness (Hv), Young's Modulus (E) and Fracture Toughness of a mineral rock sample. The rock is made up of Biotite, Feldspar and Quartz which form the standard granite composite. Each will be tested separately.

MEASUREMENT PRINCIPLE

Microindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an 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 partial or complete relaxation occurs. This procedure is performed repetitively; at each stage of the experiment the position of the indenter relative to the sample surface is precisely monitored with an optical non-contact depth sensor. For each loading/unloading cycle, the applied load value is plotted with respect to the corresponding position of the indenter. 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. The MHT is especially suited to perform tests of penetration depths in the micrometer scale and has the following specifications: • Displacement measurement: Non-contact optical sensor • Displacement resolution: 10 nm • Maximum Indenter range: 300μm • Load application: Z motor controlled with force feedback loop • Load range: 0–30N • Normal load noise floor resolution: 1.5mN • Minimum load: 10mN • Maximum load: 30N • Contact force hold time: Unlimited.

Analysis of Indentation Curve

A typical load/displacement curve is shown below, from which the compliance C = 1/S (which is the inverse of the contact stiffness) and the contact depth hc are determined after correction for thermal drift.



Calculation of Young's Modulus and Hardness

Young's Modulus: The reduced modulus, Er, is given by: $E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} = \frac{\sqrt{\pi}}{2}\frac{1}{C}\frac{1}{\sqrt{A_c}}$ which can be calculated having derived S and Ac from the indentation curve using the area function, Ac 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 Ei and ν i are the Young's modulus and Poisson coefficient of the indenter and E and ν the Young Modulus and Poisson coefficient of the tested sample. Hardness: The hardness is determined from the maximum load, Pmax, divided by the projected contact area, Ac: $H = \frac{P_{max}}{A_c}$

Test conditions and procedure

The following set of conditions was used:

Hardness Parameters	All Samples
Maximum force (N)	2.0
Loading rate (N/min)	4.0
Unloading rate (N/min)	4.0
Creep (s)	5
Poisson Coefficient	0.30
Computation Method	Oliver & Pharr
Indenter type	Vickers diamond

Fracture Toughness Parameters	Biotite	Feldspar & Quartz
Maximum force (N)	20	25
Loading rate (N/min)	75	75
Unloading rate (N/min)	75	75
Computation Method	Oliver & Pharr	
Indenter type	Vickers diamond	

Results

This section includes a summary table that compares the main numerical results for the different samples, followed by the full result listings, including each indentation performed, accompanied by micrographs of the indentation, when available. These full results present the measured values of Hardness and Young's modulus as the penetration depth (Δd) with their averages and standard deviations. It should be considered that large variation in the results can occur in the case that the surface roughness is in the same size range as the indentation.

Summary table of main numerical results for Hardness and Fracture Toughness

Hardness

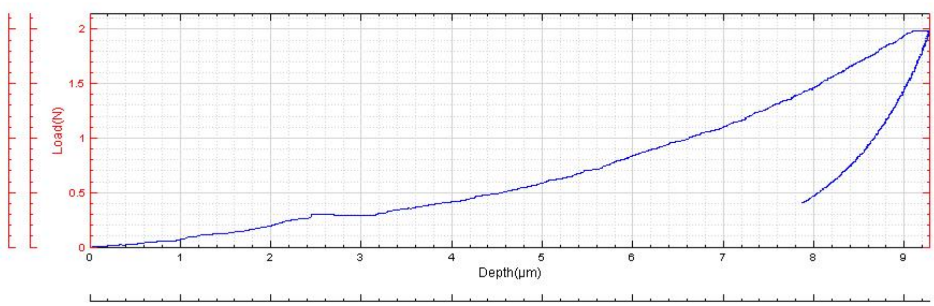
Sample	Hardness [Vickers]			Hardness [GPa]			Young's Modulus [GPa]			Depth [μm]		
	Value		Std Deviation	Value		Std Deviation	Value		Std Deviation	Value		Std Deviation
Biotite-H	94.68	±	10.80	1.00	±	0.11	29.31	±	4.97	9.98	±	0.57
Feldspar-H	850.82	±	48.00	9.00	±	0.51	75.58	±	2.33	4.15	±	0.03
Quartz-H	1258.58	±	31.06	13.32	±	0.33	99.32	±	3.27	3.56	±	0.05

Fracture Toughness

Sample	Fracture Point 1 [N]			Fracture Point 2 [N]			Fracture Point 3 [N]			Fracture Point 4 [N]		
	Value		Std Deviation	Value		Std Deviation	Value		Std Deviation	Value		Std Deviation
Biotite-FT	3.24	±	0.70	9.05	±	0.47	14.93	±	0.27	16.80	±	0.83
Feldspar-FT	2.28	±	0.77	16.62	±	2.39	21.62	±	1.13	23.45	±	0.56
Quartz-FT	1.93	±	0.57	5.83	±	0.58	7.47	±	0.11	21.38	±	1.12

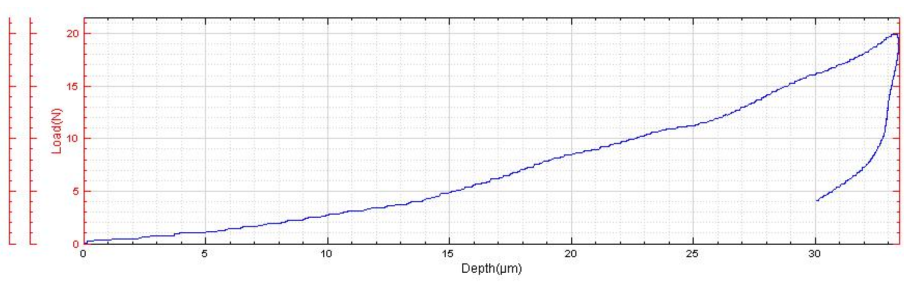
Biotite | Detailed Hardness Results

	Hv [Vickers]	H [GPa]	E [GPa]	Δd [µm]
1	104.89	1.11	33.01	9.44
2	94.25	1.00	34.66	9.85
3	108.68	1.15	30.45	9.32
4	83.09	0.88	22.93	10.68
5	82.49	0.87	25.49	10.58
Average	**94.68**	**1.00**	**29.31**	**9.98**
Std Deviation	**10.80**	**0.11**	**4.97**	**0.57**



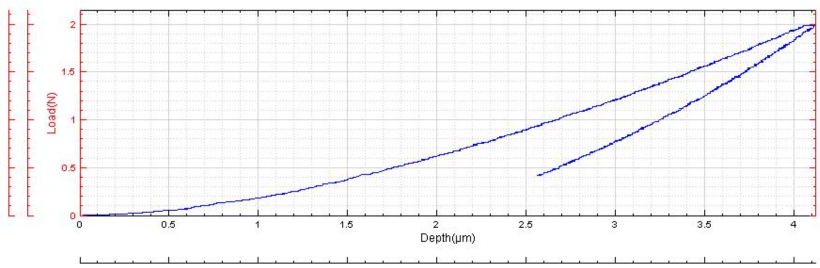
Biotite | Detailed Fracture Toughness Results

Test	Fracture Point 1 [N]	Fracture Point 2 [N]	Fracture Point 3 [N]	Fracture Point 4 [N]
1	3.65	8.60	14.95	16.02
2	3.64	9.02	15.18	16.72
3	2.43	9.53	14.65	16.67
Average	**3.24**	**9.05**	**14.93**	**16.80**
Std Deviation	**0.70**	**0.47**	**0.27**	**0.83**



Feldspar | Detailed Hardness Results

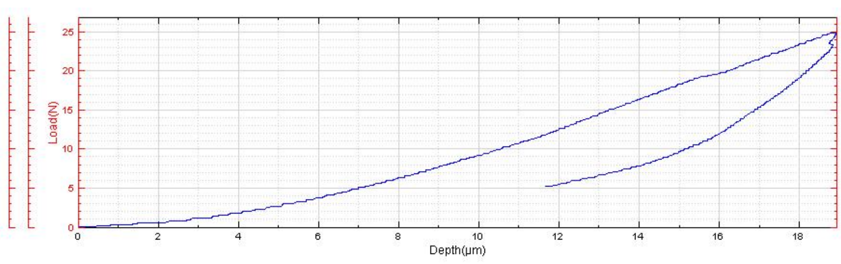
	Hv [Vickers]	H [GPa]	E [GPa]	Δd [μm]
1	900.94	9.53	75.26	4.10
2	763.46	8.08	79.81	4.19
3	887.94	9.40	72.82	4.16
4	850.48	9.00	74.34	4.17
5	851.28	9.01	75.64	4.16
Average	**850.82**	**9.00**	**75.58**	**4.15**
Std Deviation	**48.00**	**0.51**	**2.33**	**0.03**



Feldspar | Detailed Fracture Toughness Results

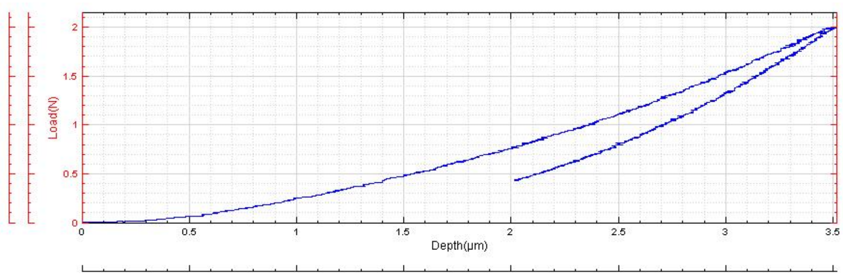
*Note: Fracture Point 3 and 4 are the overall structural fracture points while the other fracture points are local points of fracture.

Test	Fracture Point 1 [N]	Fracture Point 2 [N]	Fracture Point 3* [N]	Fracture Point 4* [N]
1	2.98	19.34	22.67	23.80
2	2.40	15.67	21.76	23.74
3	1.45	14.84	20.42	22.81
Average	**2.28**	**16.62**	**21.62**	**23.45**
Std Deviation	**0.77**	**2.39**	**1.13**	**0.56**



Quartz | Detailed Fracture Toughness Results

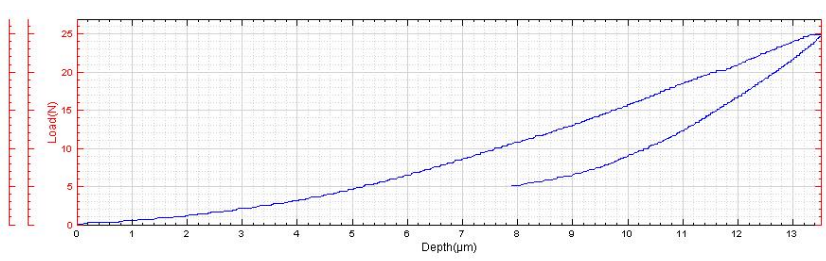
	Hv [Vickers]	H [GPa]	E [GPa]	Δd [µm]
1	1286.32	13.61	103.66	3.50
2	1286.93	13.62	102.30	3.52
3	1251.01	13.24	97.74	3.58
4	1266.07	13.40	98.26	3.56
5	1202.58	12.73	94.62	3.64
Average	**1258.58**	**13.32**	**99.32**	**3.56**
Std Deviation	**31.06**	**0.33**	**3.27**	**0.05**



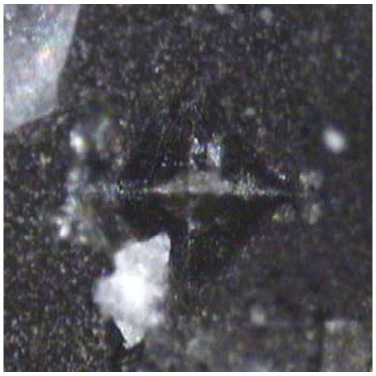
Quartz | Detailed Fracture Toughness Results

*Note: Fracture Point 4 is the overall structural fracture point while the other fracture points are local points of fracture.

Test	Fracture Point 1 [N]	Fracture Point 2 [N]	Fracture Point 3 [N]	Fracture Point 4* [N]
1	2.52	5.24	--	20.78
2	1.89	6.39	7.55	22.67
3	1.39	5.85	7.39	20.68
Average	**1.93**	**5.83**	**7.47**	**21.38**
Std deviation	**0.57**	**0.58**	**0.11**	**1.12**



CONCLUSION:



In conclusion, the Nanovea Mechanical Tester, in Microindentation mode, demonstrates reproducibility and precise indentation results on the hard surface of mineral rock. Not only hardness but also elastic modulus of each material forming the granite was measured directly from the depth versus load curves. The fact that the surface was rough forced testing at higher loads that may have caused micro cracking. Micro cracking would explain some of the variations seen from measurement to measurement. Also, because the surface of sample was rough, cracks were not perceivable through standard microscopy observation. Therefore it was not possible to calculate traditional Fracture Toughness numbers that requires cracks lengths to be measured. Instead, this is why we have used the system to detect initiation of cracks through the dislocations in the depth versus load curves while increasing loads. Fracture Threshold Loads were reported at loads at which failures occurred. Unlike traditional Fracture Toughness tests that simply measure crack length, an actual value is obtained at which threshold load a fracture starts. Additionally, the controlled and closely monitored environment allows the measurement of hardness to use as a quantitative value for comparing a variety of samples.