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MICROINDENTATION & FRACTURE OF MINERAL ROCK

Prepared by

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INTRODUCTION

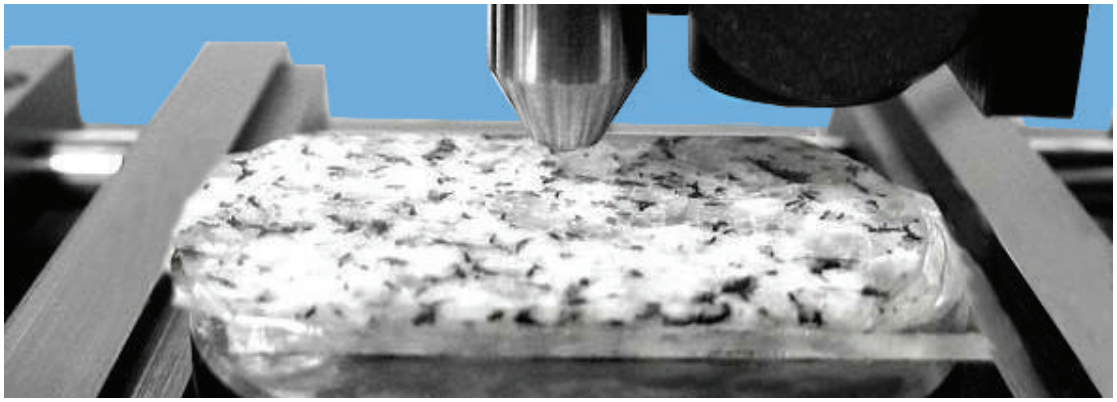
Rock mechanics is the study of the mechanical behavior of rock masses and is applied in mining, drilling, reservoir production, and civil construction industries. Advanced instrumentation with precise measurement of mechanical properties allows for part and procedure improvement within these industries. Successful quality control procedures are ensured by understanding rock mechanics at the micro scale.

MICROINDENTATION RESEARCH AND QUALITY CONTROL

Microindentation is a crucial tool used for rock mechanics related studies. These techniques advance excavation techniques by providing further understanding of rock mass properties. Microindentation is used to improve drill heads which improve mining procedures. Microindentation has been used to study chalk and powder formation from minerals. Microindentation studies can include hardness, Young's modulus, creep, stress-strain, fracture toughness, and compression with a single instrument.

MEASUREMENT OBJECTIVE

In this application the Nanovea mechanical tester measures the Vickers hardness (Hv), Young's modulus, 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 is tested separately.



The following set of conditions was used:

Hardness Parameters	All Samples
Maximum Force	2 N
Loading Rate	4 N/min
Creep	5 seconds
Computation Method	Oliver & Pharr
Indenter Type	Vickers diamond

Fracture Toughness Parameters	Biotite	Feldspar & Quartz
Maximum Force	20 N	25 N
Loading Rate	75 N/min	75 N/min
Creep	0 seconds	
Computation Method	Oliver & Pharr	
Indenter Type	Vickers diamond	

Sample of rock used for testing.

RESULTS & DISCUSSION

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.

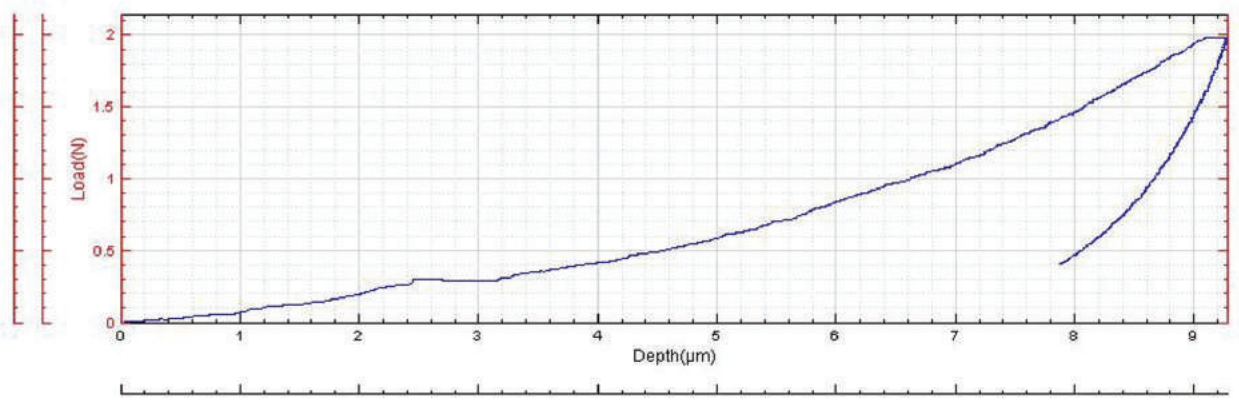
Hardness				
Sample	**Hardness [Vickers]**	**Hardness [GPa]**	**Young's Modulus [GPa]**	**Depth [µm]**
Biotite-H	94.68 ± 10.80	1.00 ± 0.11	29.31 ± 4.97	9.98 ± 0.57
Feldspar-H	850.82 ± 48.00	9.00 ± 0.51	75.58 ± 2.33	4.15 ± 0.03
Quartz-H	1258.58 ± 31.06	13.32 ± 0.33	99.32 ± 3.27	3.56 ± 0.05

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

Summary table of main numerical results for Hardness and Fracture Toughness

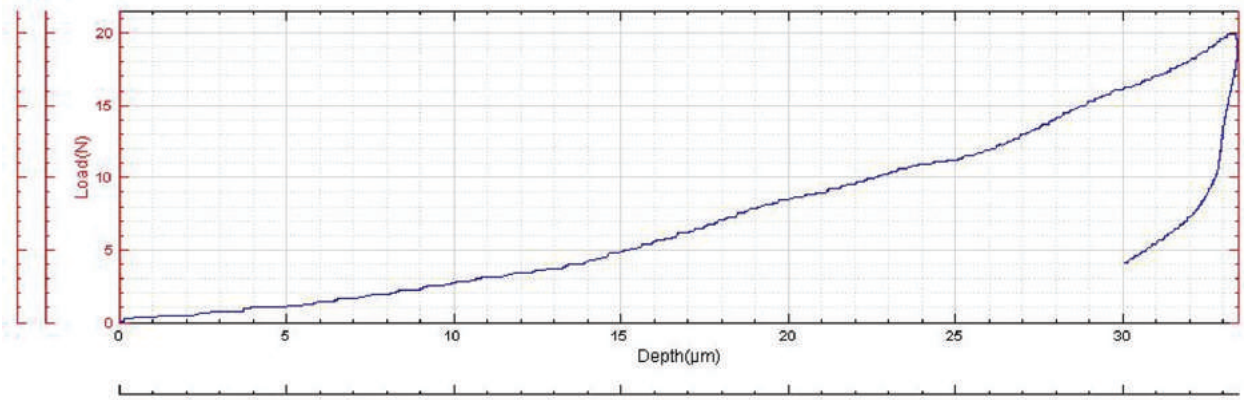
Biotite | Detailed Hardness Results

Test	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Depth [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**



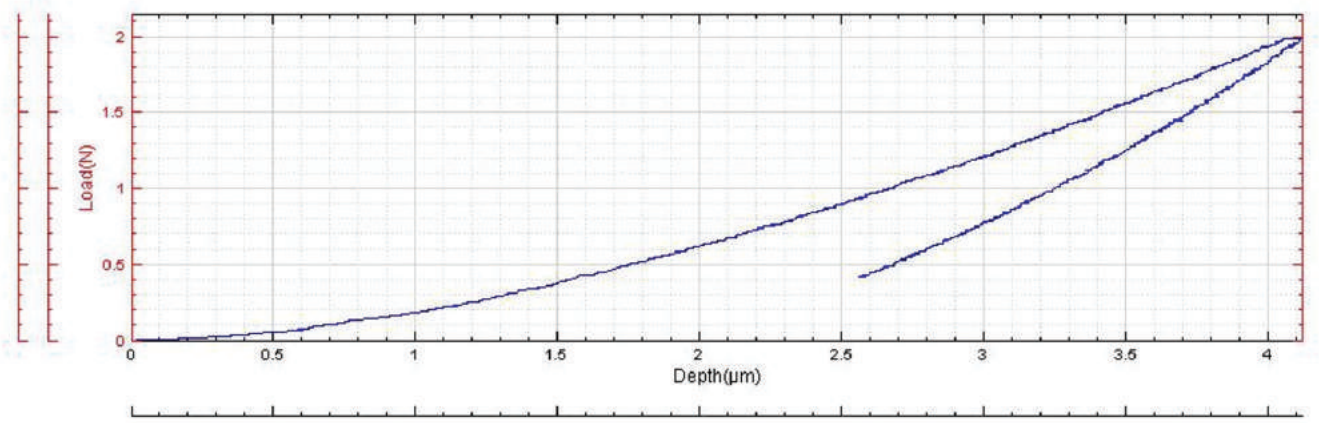
Biotite | Detailed Fracture Toughness Results

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

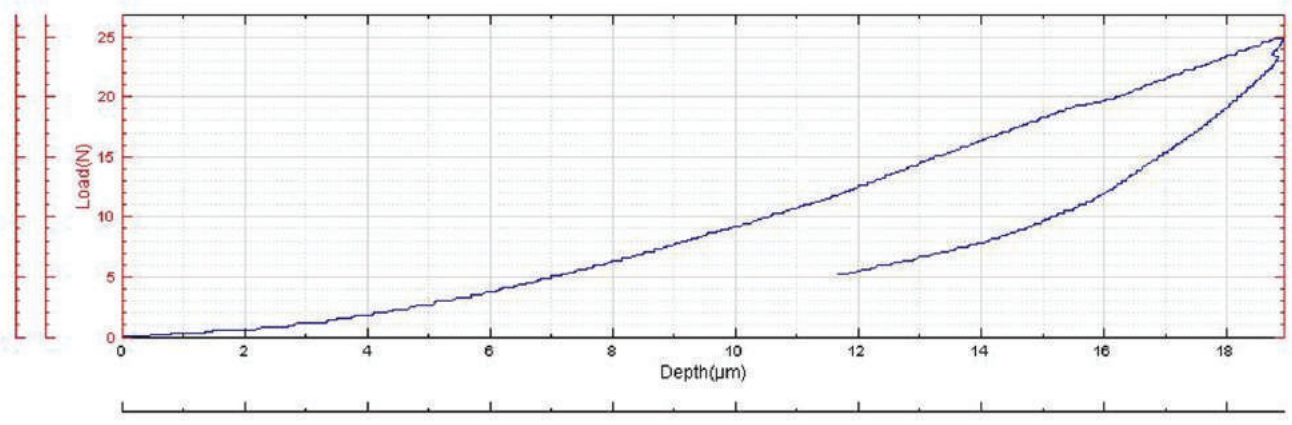
Test	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Depth [µm]
1	900.94	9.53	75.26	4.10
2	763.46	8.08	79.81	4.19
3	887.94	9.40	72.82	4.16
4	850.48	9.00	74.34	4.17
5	851.28	9.01	75.64	4.16
Average	**850.82**	**9.00**	**75.58**	**4.15**
Std Deviation	**48.00**	**0.51**	**2.33**	**0.03**



Feldspar | Detailed Fracture Toughness Results

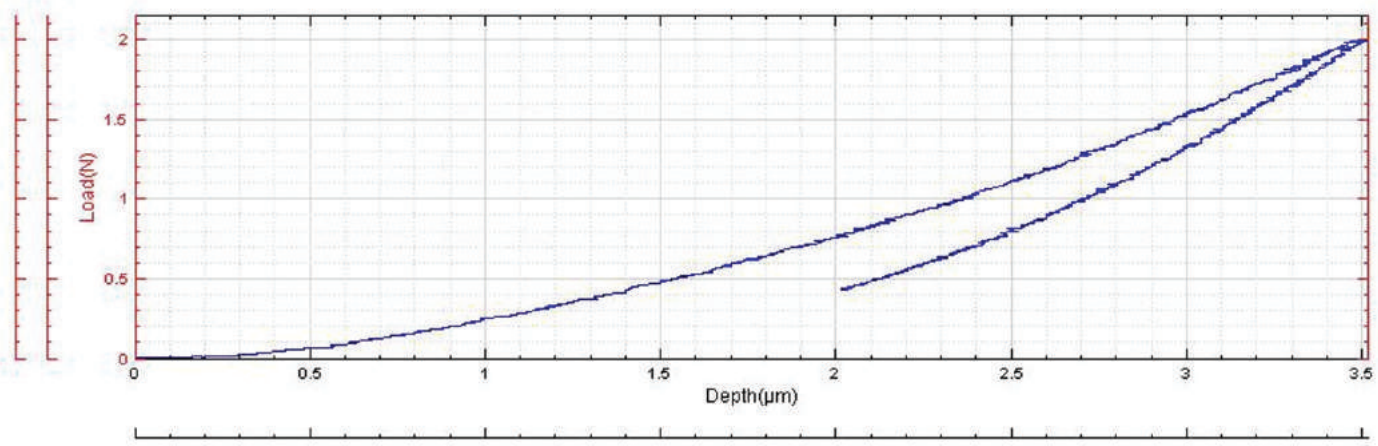
*Note: Fracture Point 3 and 4 are the overall structural fracture points while the other fracture points are local points of fracture.

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 Hardness Results

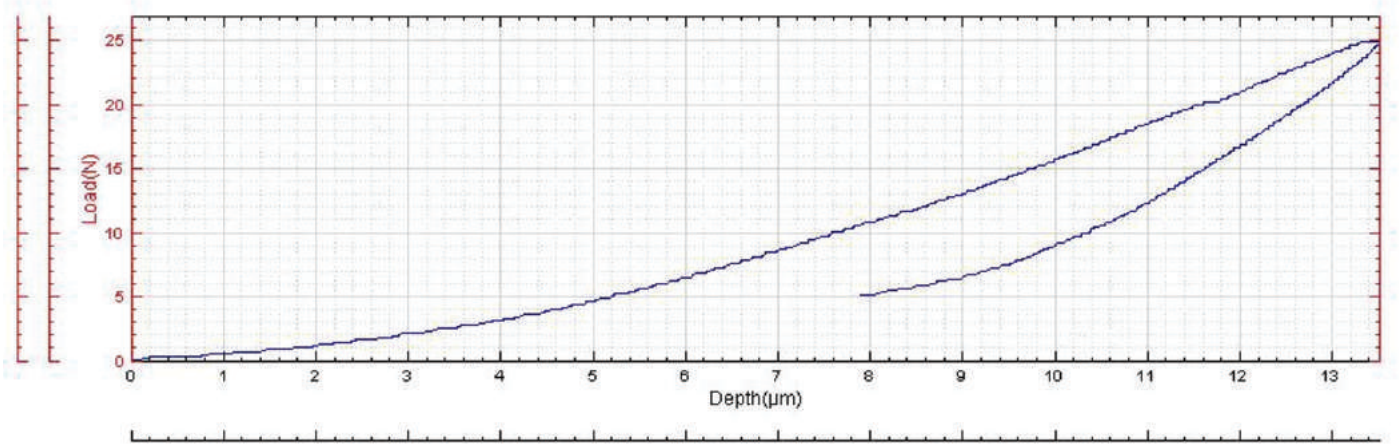
Test	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Depth [µm]
1	1286.32	13.61	103.66	3.50
2	1286.93	13.62	102.30	3.52
3	1251.01	13.24	97.74	3.58
4	1266.07	13.40	98.26	3.56
5	1202.58	12.73	94.62	3.64
Average	**1258.58**	**13.32**	**99.32**	**3.56**
Std Deviation	**31.06**	**0.33**	**3.27**	**0.05**



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

The Nanovea mechanical tester demonstrates reproducibility and precise indentation results on the hard surface of mineral rock. Hardness and Young's modulus of each material forming the granite was measured directly from depth versus load curves. The rough surface meant testing at higher loads that may have caused micro cracking. Micro cracking would explain some of the variations seen in measurements. Cracks were not perceivable through standard microscopy observation because of a rough sample surface. Therefore, it is not possible to calculate traditional fracture toughness numbers that requires cracks length measurements. Instead, we 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 where failures occurred. Unlike traditional fracture toughness tests that simply measure crack length, a load is obtained at which threshold 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.

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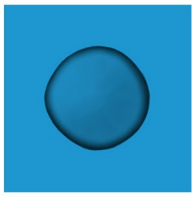
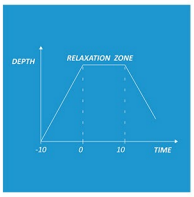
INDENTATION

FRACTURE TOUGHNESS

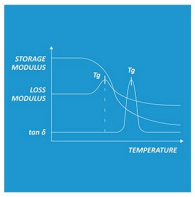
HARDNESS MAPPING



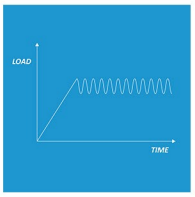
YIELD STRENGTH & FATIGUE



CREEP & RELAXATION



Tg GLASS TRANSITION



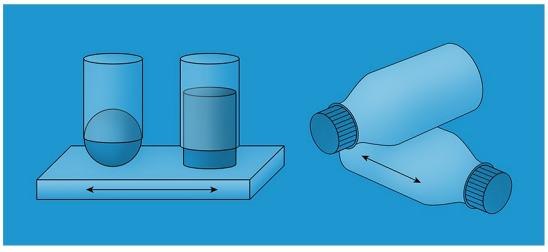
LOSS & STORAGE MODULUS

SCRATCH

COHESIVE & ADHESIVE FAILURE

SCRATCH HARDNESS

FRICTION



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