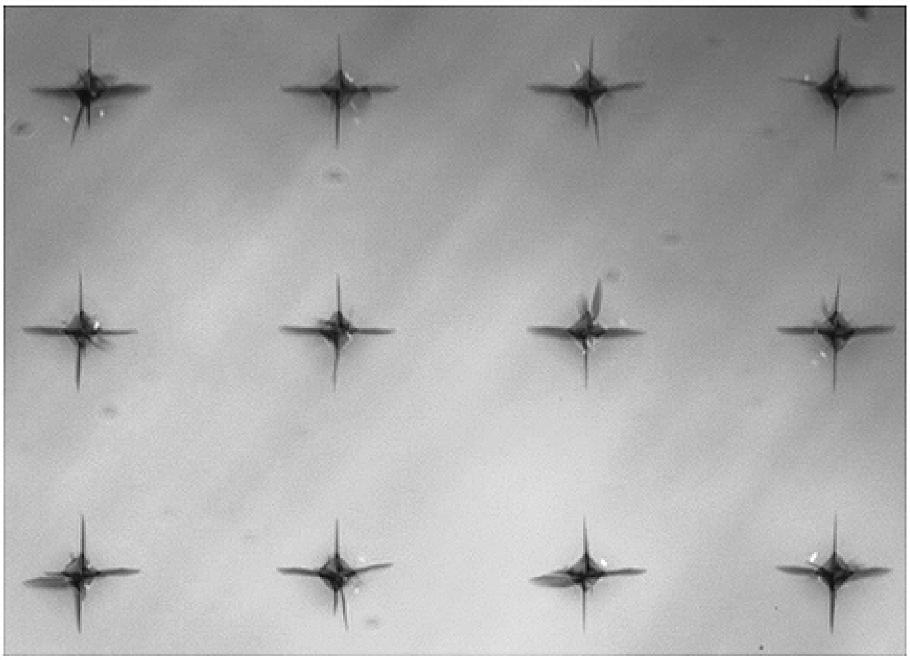
MICROINDENTATION MAPPING WITH ACOUSTIC EMISSION ON GLASS



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INTRO

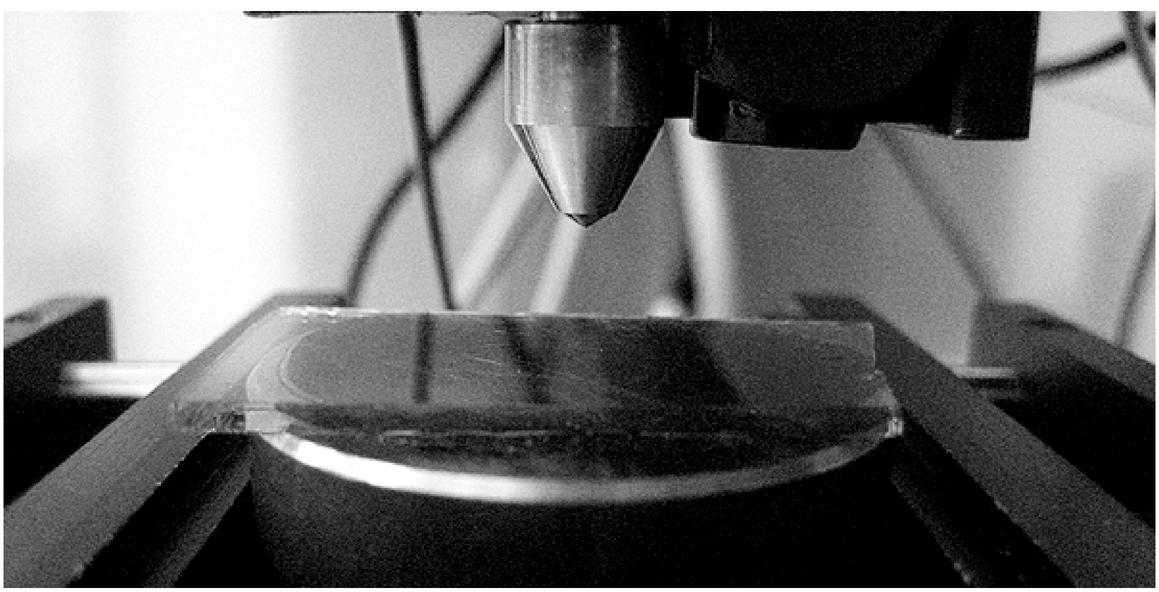
Hardness mapping has been widely used to assess the variation of mechanical properties across a large surface area. However, with the study of applications such as glass it is the crack initiation that is of particular interest. It is for this reason that acoustic emission can be used during the mapping of indents to not only map hardness but also map the crack initiation values over the surface area. Using acoustic emission proves to be a critical tool during the study of applications where micro fracture is of primary concern.

THE IMPORTANCE OF MICROINDENTATION REASEARCH AND QUALITY CONTROL

The use of Microindentation mapping has proven to be a critical tool for surface mechanics related studies. Mapping of areas of interest allows more broad studies of slight property changes on the surface. Software is available to simulate stress distribution during the test to better understand crack propagations. Acoustic Emissions (AE) is used to track fracture behavior and intensity during loading and unloading. Mapping of these AE gives insight into the distribution of fracture behavior on the surface. Because fracture has typically been evaluated by crack measurement, the use of acoustic emission during testing has been an overlooked and valuable tool. Microindentation studies can include Hardness, Young's Modulus, Creep, Stress-Strain, Yield Strength, 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 the acoustic emission of a glass sample. The test is set for a 4 by 6 mapping under a 1N and 5N load.

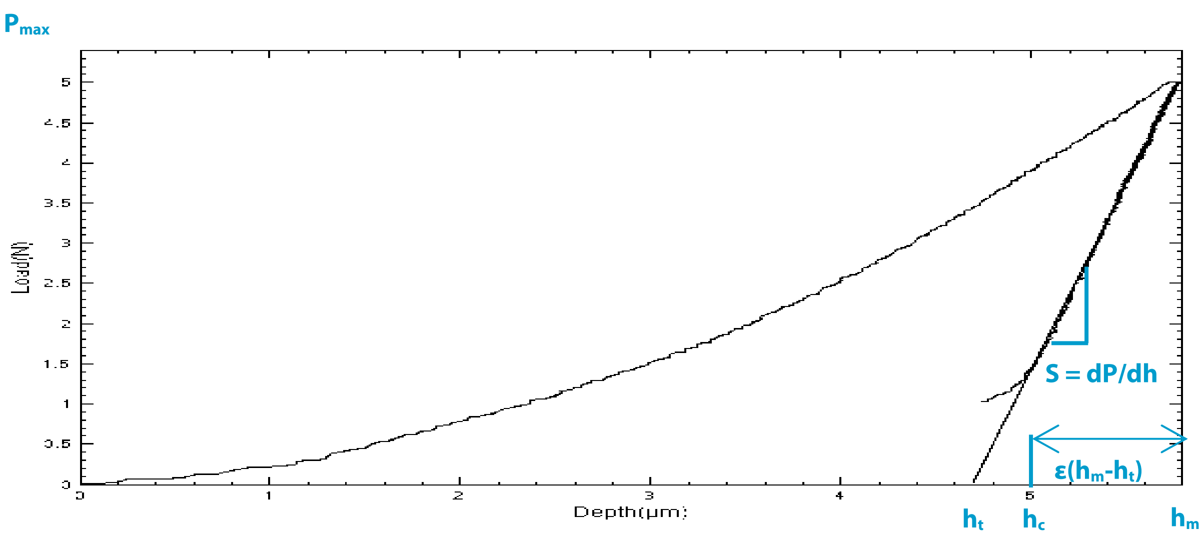


MEASUREMENT PRINCIPLE

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 h_c are determined after correction for thermal drift.



Calculation of Young's Modulus and Hardness

Young's Modulus: The reduced modulus, E_r, 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 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 E and ν the Young Modulus and Poisson coefficient of the tested sample. Hardness: The hardness is determined from the maximum load, P_{max}, divided by the projected contact area, A_c: $H = \frac{P_{max}}{A_c}$

TEST CONDITIONS & PROCEDURES

Hardness Parameters	1N	5N
Maximum force (N)	1	5
Loading rate (N/min)	2	10
Unloading rate (N/min)	2	10
Creep (s)	n/a	n/a
Poisson Coefficient	0.3	0.3
Computation method	Oliver & Pharr	Oliver & Pharr
Indenter type Mapping Mapping time*	Vickers 4 by 6 Indents 22min	Vickers 4 by 6 indents 22min

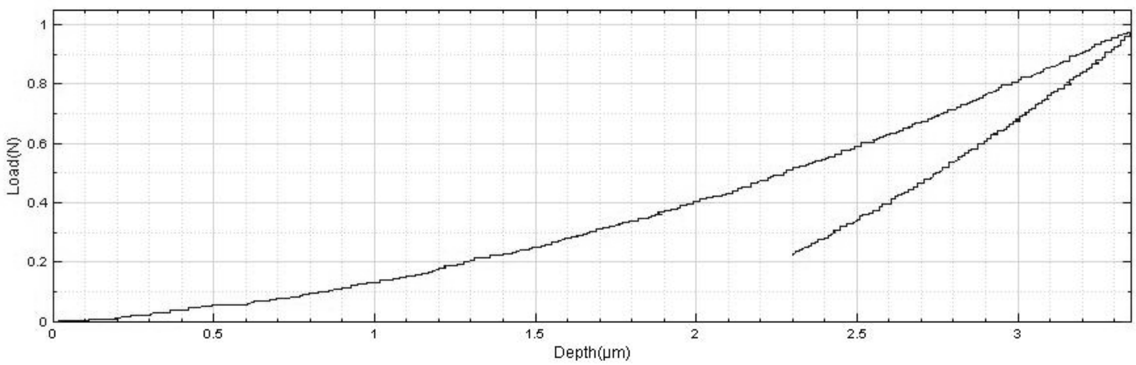
**Mapping time can be adjusted based on application & parameters*

RESULTS

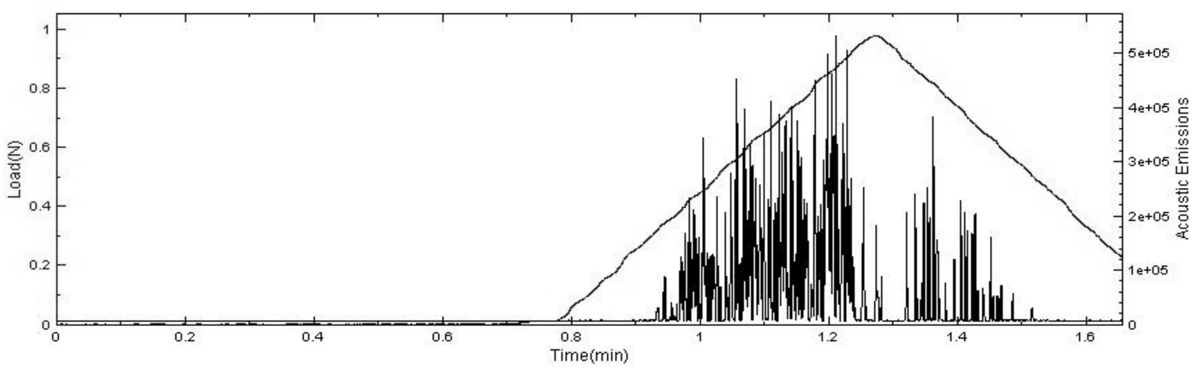
Below is the measured values of Hardness and Young's modulus of 1N & 5N loading as the penetration depth (Δd) with their averages and standard deviations. It should be considered that large variations in the results can occur in the case that the surface roughness is in the same size range as the indentation. One indent from each mapped load is highlighted to preview acoustic emission and optical image.

1N Loading

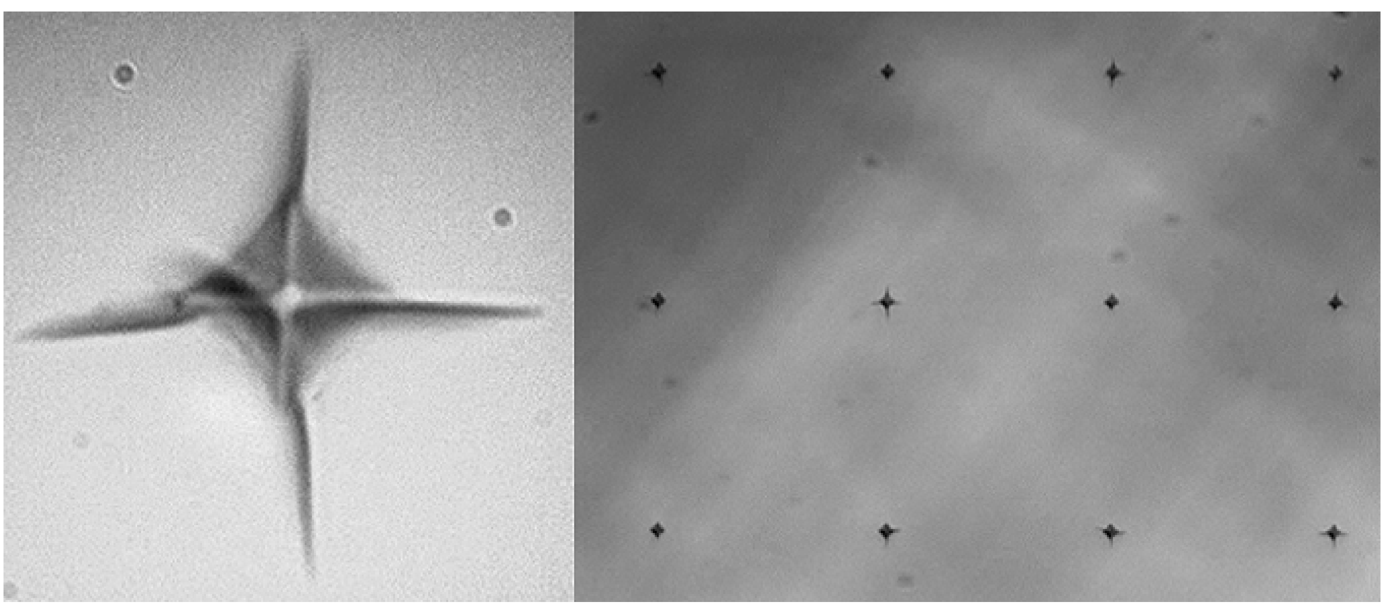
Test	Max Load	Max Depth	Hardness (Gpa)	Hardness (HV)	Modulus
Test 1	979.84	3147.30	7.50	708.35	65.84
Test 2	978.51	3734.12	8.73	825.05	36.27
Test 3	977.85	3359.82	6.76	638.89	55.73
Test 4	981.84	3420.40	6.20	585.59	56.73
Test 5	978.51	3339.57	6.75	637.74	57.20
Test 6	979.18	3390.13	6.44	608.70	56.34
Test 7	983.84	3470.93	6.01	568.16	55.29
Test 8	979.18	3349.67	6.66	628.95	57.32
Test 9	978.51	3390.16	6.60	624.12	55.01
Test 10	978.51	3380.04	6.44	608.48	56.95
Test 11	980.51	3390.09	6.60	623.91	55.25
Test 12	983.84	3359.65	6.56	620.15	57.94
Test 13	981.84	3359.70	6.50	614.38	58.21
Test 14*	**970.53**	**3329.70**	**6.73**	**635.87**	**57.10**
Test 15	975.19	3359.90	6.80	642.58	55.17
Test 16	978.51	3288.98	6.81	643.31	60.28
AVG	**979.14**	**3379.38**	**6.76**	**638.39**	**56.04**
STDev	**3.21**	**116.84**	**0.62**	**58.13**	**5.90**



1N Depth vs Load Curve



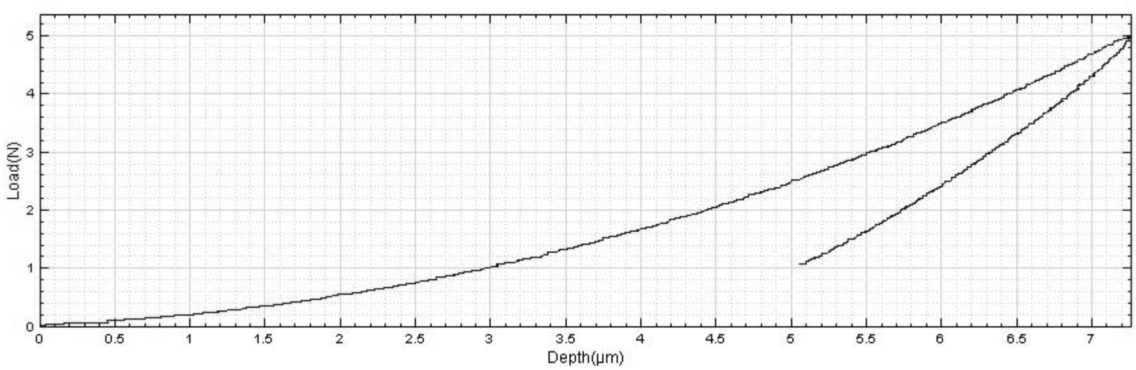
1N Loading Curve With Acoustic Emissions



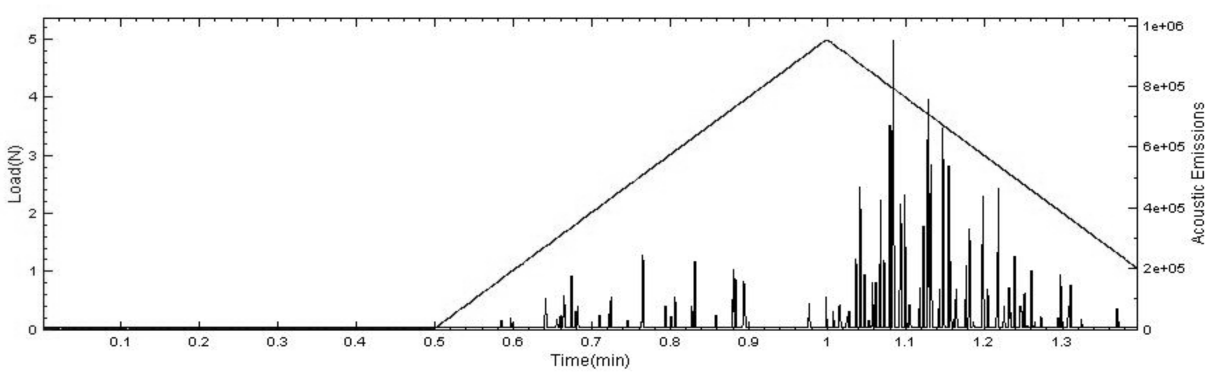
1N Load Indentation Map

5N Loading

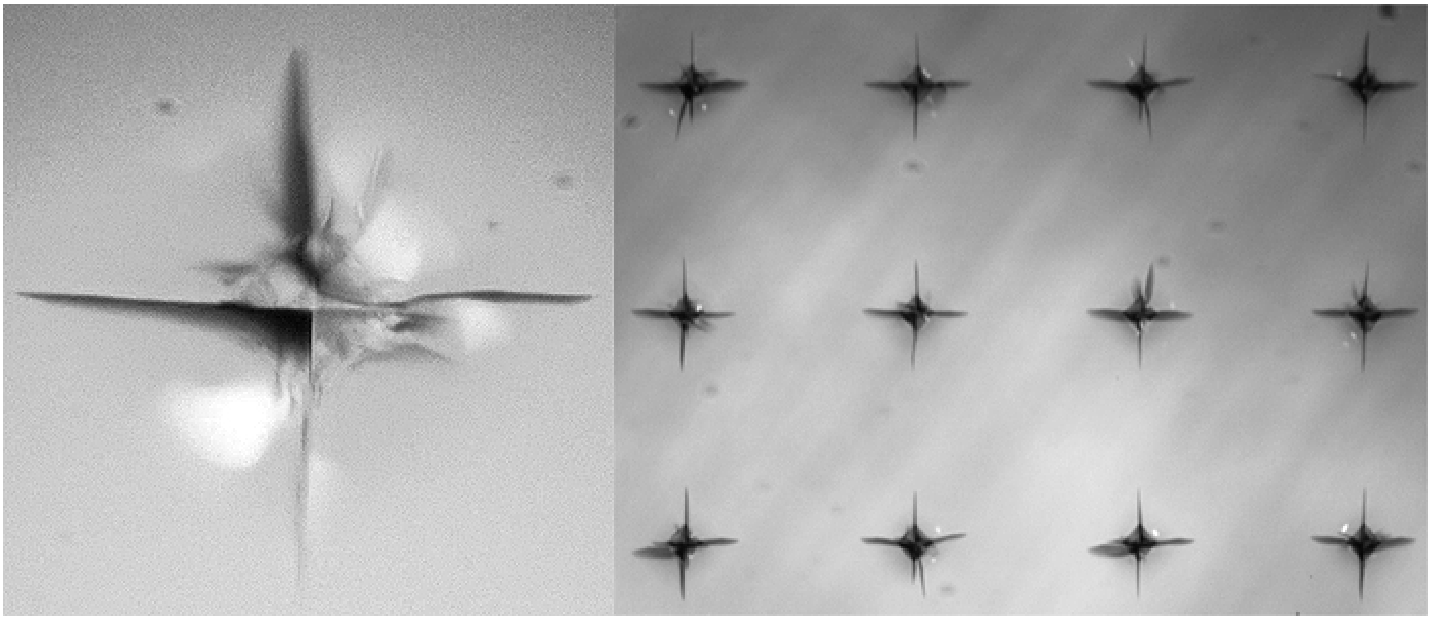
Test	Max Load	Max Depth	Hardness (Gpa)	Hardness (HV)	Modulus
Test 1	4943.79	7368.53	6.29	594.45	66.61
Test 2	4948.45	7418.97	6.22	588.01	65.57
Test 3	4949.11	7368.37	6.36	601.27	65.86
Test 4	4957.76	7398.46	6.31	596.43	65.54
Test 5	4951.11	7469.49	6.14	580.03	64.72
Test 6	4953.77	7418.81	6.29	594.84	64.82
Test 7	4947.12	7358.31	6.38	603.02	65.97
Test 8	4952.44	7509.91	6.09	575.57	63.78
Test 9	4948.45	7398.74	6.30	595.57	65.37
Test 10	4953.10	7348.02	6.47	611.83	65.44
Test 11	4943.79	7378.65	6.32	597.35	65.80
Test 12	4941.13	7429.32	6.23	588.56	64.86
Test 13	4948.45	7378.52	6.38	602.52	65.27
Test 14	4945.79	7247.07	6.68	631.04	66.93
Test 15	4945.12	7348.26	6.44	609.00	65.55
Test 16	4943.13	7348.32	6.42	606.79	65.77
AVG	**4948.28**	**7386.73**	**6.33**	**598.52**	**65.49**
STDev	**4.51**	**58.74**	**0.14**	**13.11**	**0.74**



5N Depth vs Load Curve



5N Loading Curve With Acoustic Emissions



5N Load Indentation Map

CONCLUSION:

The Nanovea Mechanical Tester, in Microindentation mode, has demonstrated reproducibility and precise Microindentation mapping results on a glass application. It is to be noted that cracking will reduce the hardness and the elastic modulus measured. Hardness and elastic modulus were monitored using acoustic emissions during each indentation. The ability to measure acoustic emission during the test can help identify the fracture process. The Nanovea system also allows quantification of AE in terms of energy released, unlike other systems that only provide a % of intensity with no reference. At 1N load the maximum is $5X10^5$ attoJ (0.5 picoJ) during the loading. At 5N load, with a faster loading process, the maximum of $1X10^6$ attoJ (1picoJ) is during the unloading part of the curve. It is worth to note that the system can measure energy level orders of magnitude lower than what is shown here and orders of magnitude higher.