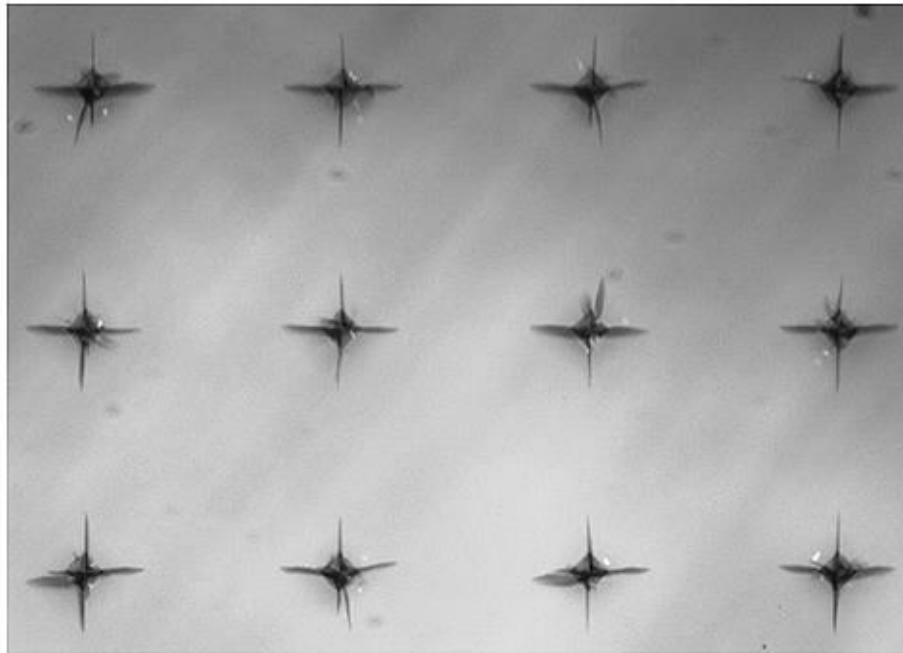


**MICROINDENTATION MAPPING  
WITH ACOUSTIC EMISSION ON GLASS**



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## INTRO

Hardness mapping has been widely used to assess the variation of mechanical properties across a large surface area. However, in the study of brittle materials such as glass, the crack initiation and propagation is of particular interest. Acoustic emission works as a useful tool to monitor the crack initiation and growth during the indentation process.

## IMPORTANCE OF MICROINDENTATION WITH ACOUSTIC EMISSION

The microindentation mapping has proven to be a critical tool for surface mechanics related studies. Fracture has typically been evaluated by measuring the dimension of the cracks of the indent, and the use of acoustic emission during testing has been an overlooked and valuable tool. Microindentation with the measurement of Acoustic Emissions (AE) provides a reliable and user-friendly method to track the fracture behavior and intensity during loading and unloading process.

## MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Microindentation mode, performs a mapping of the mechanical properties such as the hardness, Young's Modulus and the acoustic emission of a glass sample.



**Fig. 1: Micro indenter on the glass sample.**

## TEST CONDITIONS

The Nanovea Mechanical Tester was used to perform a series (4×4, 16 indents in total) of micro indentations on a glass sample using a Vickers indenter. The load, depth and acoustic emission (AE) were measured and recorded during the indentation. All indentations were performed to a maximum load of 1 N. The test conditions are summarized in Table 1.

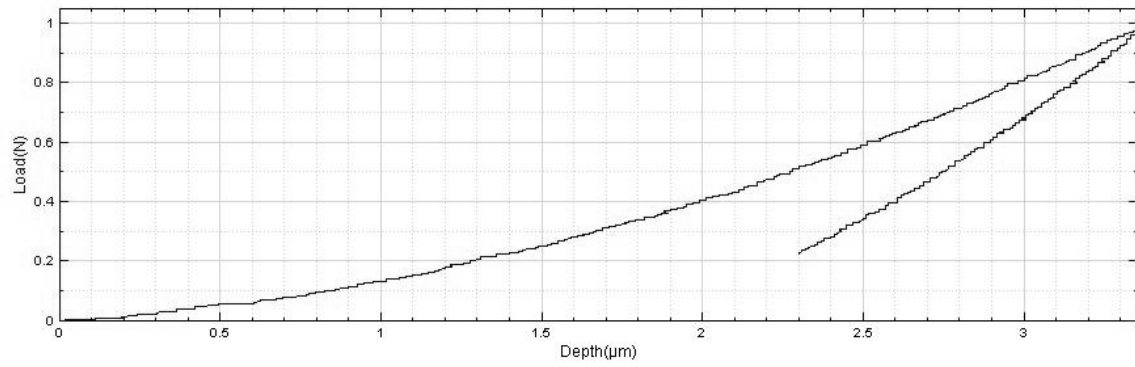
Maximum force (N)	1
Loading rate (N/min)	2
Unloading rate (N/min)	2
Poisson ratio	0.3
Computation method	Oliver & Pharr <sup>i</sup>
Indenter type	Vickers
Mapping	4 by 4 Indents

**Table 1: Test parameters of the indentation mapping.**

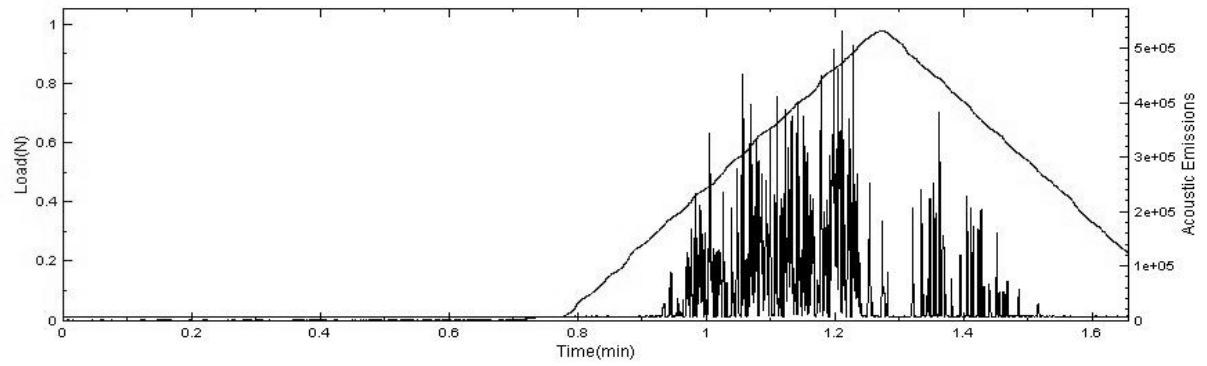
## RESULTS AND DISCUSSION

The load-depth curve and evolution of the acoustic emission during the indentation are shown in Fig. 2 and Fig. 3, respectively. The image of the indentation map is displayed in Fig. 4. The high-precision position control of the sample stage allows users to select the target area for mechanical properties mapping. The acoustic emission during the indentation provides the insight of the development of the cracks during the loading period. It can be observed that the acoustic emission signals start to be detected at a load of ~0.35 N, indicating the initiation of the crack at this point. As the indenter progressively penetrates into the glass, acoustic emission of higher intensity occurs, caused by the propagation of the crack. The Nanovea Mechanical Tester allows quantification of AE in terms of energy released, unlike other systems that only provide a percentage of the intensity with no reference. At 1 N load the maximum AE value is  $5 \times 10^5$  attoJ (0.5 picoJ) during the loading. 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.

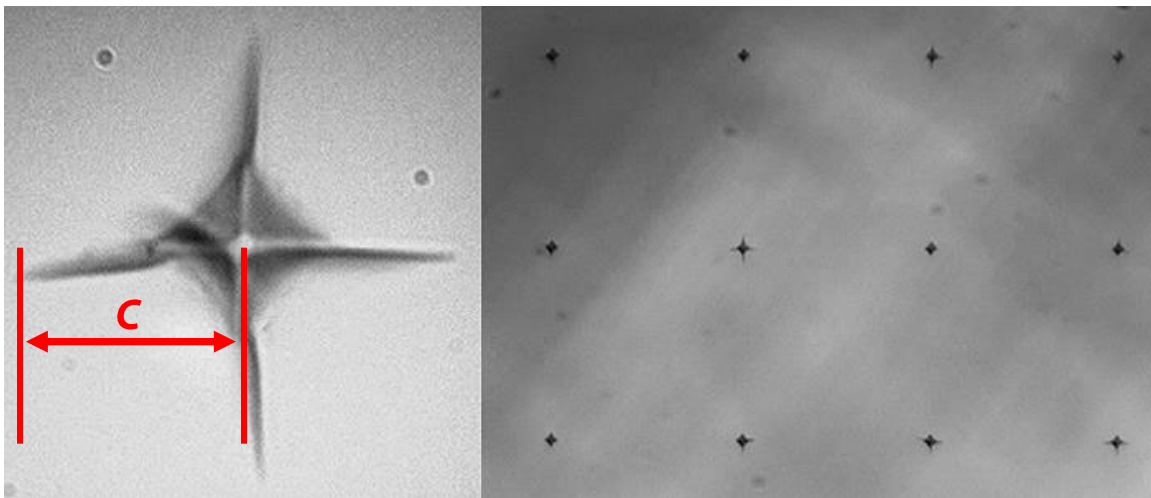
The hardness and Young's Modulus are summarized and compared in Fig. 5 and Fig. 6. The glass sample exhibits a homogenous mechanical properties – it possesses an average hardness of ~6.6 GPa and Young's modulus of ~56.7 GPa throughout the test area. Fig. 6 provides a useful tool to observe the distribution of hardness and Young's modulus across the tested sample surface. Such a 4×4 indentation matrix was finished within 20 minutes with superior precision and repeatability, thanks to the accurate position control and the direct load/displacement measurement of the Nanovea Mechanical Tester. The mapping mode in this study enables speedy quantitative mapping of mechanical properties including Young's modulus and hardness, which is critical for quality control of a variety of materials.



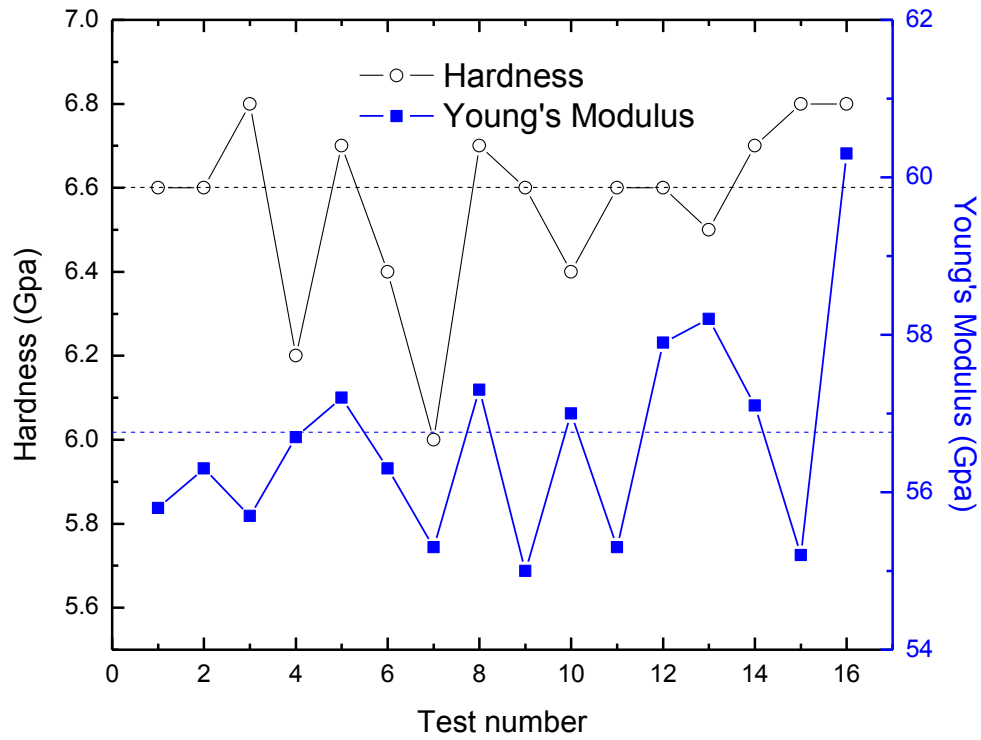
**Fig. 2: Load vs Depth Curve.**



**Fig. 3: Evolution of Acoustic Emissions during the loading.**

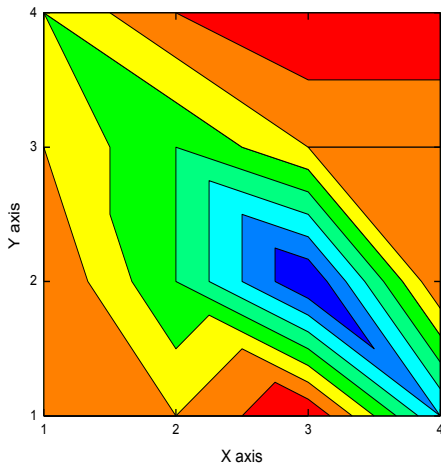


**Fig. 4: Indentation Map.**

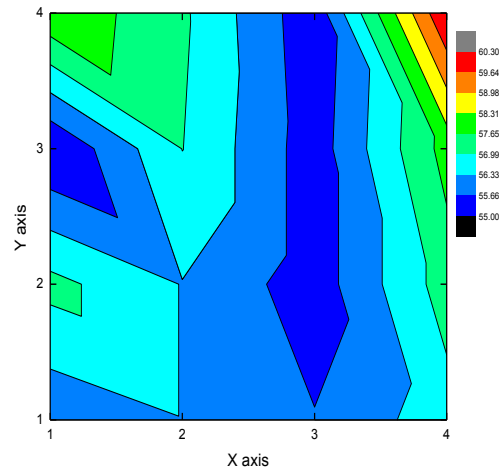


**Fig. 5: Hardness and Young's modulus of the glass sample.**

**(a) Hardness distribution:**



**(b) Young's Modulus distribution:**



**Fig. 6: Distribution of the Hardness and Young's modulus.**

The fracture toughness of the glass can also be calculated based on the length of the cracks at the corners of the indents as shown in Fig. 4. 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  $\alpha$  is an empirical constant for a specified indenter,  $\alpha = 0.016$  for the Vickers tip,  $E$  is the Young's Modulus,  $H$  is the hardness,  $P$  is the applied load, and  $c$  is the crack length.

## CONCLUSION

In this study, we has demonstrated that the Nanovea Mechanical Tester, in Microindentation mode, provides reproducible and precise mapping of mechanical properties across a glass sample. The sample possesses homogenous hardness and elastic modulus in the measured area. The evolution of acoustic emission recorded during the indentation works as an important indication of the fracture process.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young's modulus, fracture toughness, adhesion, wear resistance and many others.

In addition, optional 3D non-contact profiler and AFM Module are available for high resolution 3D imaging of indentation, scratch and wear track in addition to other surface measurements such as roughness.

To learn more about [Nanovea Mechanical Tester](#) or [Lab Services](#).

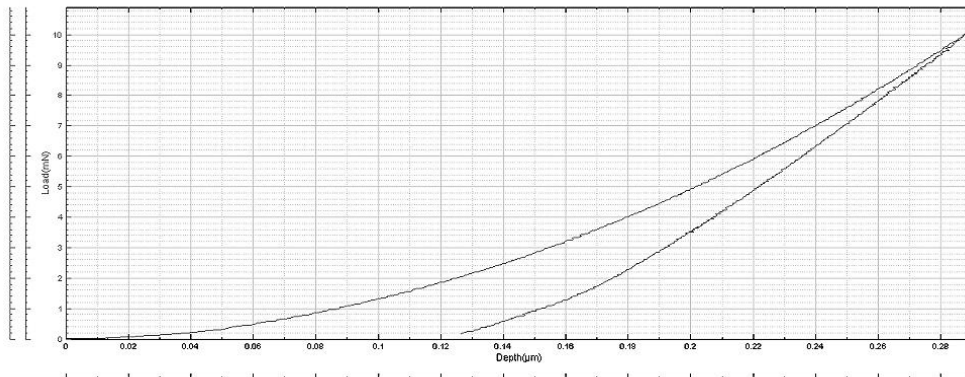
## APPENDIX: MEASUREMENT PRINCIPAL

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  $\nu_i$  are the Young's modulus and Poisson

coefficient of the indenter and  $E$  and  $\nu$  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,  $A_c$ :  $H = \frac{P_{max}}{A_c}$

<sup>i</sup> Oliver, W. C.; Pharr, G. M., Journal of Materials Research, Volume 7, Issue 6, June 1992, pp.1564-1583