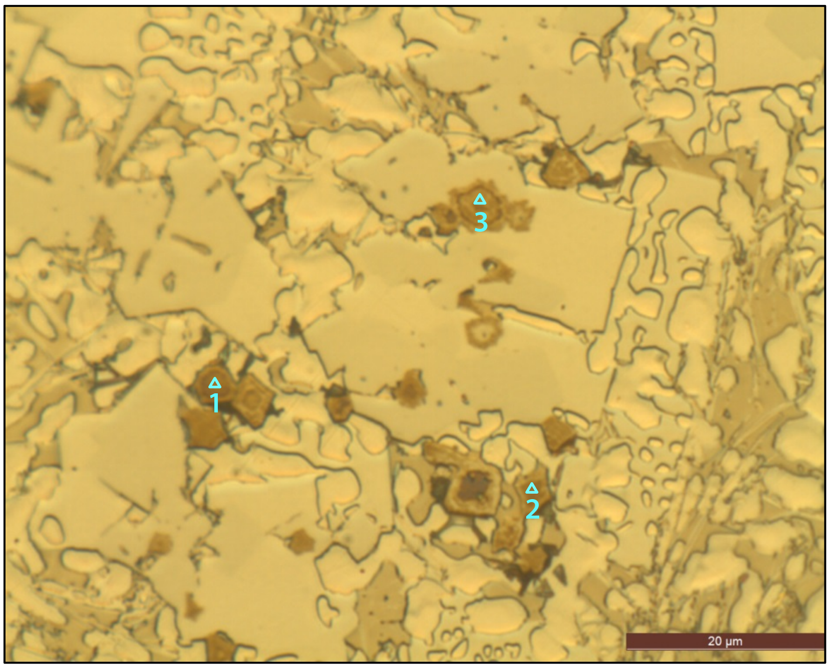
METALLURGY STUDY OF MULTIPHASE MATERIAL USING NANOINDENTATION



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INTRODUCTION

Metallurgy studies the physical and chemical behavior of metallic elements, as well as their intermetallic compounds and alloys. Metals undergoing working processes, such as casting, forging, rolling, extrusion and machining, etc., change their phases, microstructure and texture, which results in varied physical properties including hardness, strength, toughness, ductility, and wear resistance. Metallography is often applied to learn the formation mechanism of such specific phases, microstructure and texture.

IMPORTANCE OF LOCAL MECHANICAL PROPERTIES FOR MATERIALS DESIGN

Advanced materials often contain multiple phases in special microstructure and texture to achieve desired mechanical properties for target application in industrial practice. Nanoindentation is widely applied to measure the mechanical behaviors of materials at small scales[i ii]. However, it is challenging and time-consuming to precisely select specific locations for indentation in a very small area. A reliable and user-friendly procedure of nanoindentation testing is in need to determine the mechanical properties of different phases of a material with high precision and timely measurement.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode is used to measure the mechanical properties of a multi-phase metallurgical sample. We would like to showcase the capacity of Nanovea Mechanical Tester in performing nanoindentation measurement at different locations of a large sample surface with high precision and user friendliness using *advanced position controller* (Patent Pending).

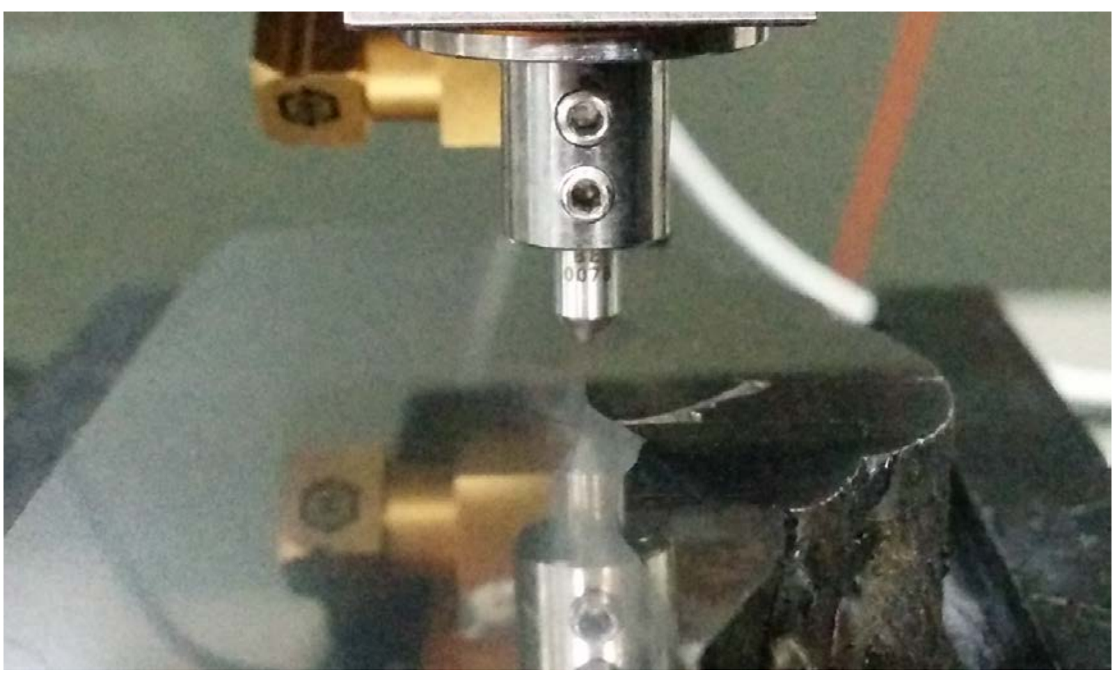

Fig. 1: Nanoindentation tip on the sample.

TEST CONDITIONS

A metallurgical sample with different phases is used in this study. The sample had been polished to a mirror-like surface finish before the indentation tests. Four phases have been identified in the sample, namely Phase 1, Phase 2, Phase 3 and Phase 4 as shown in Fig. 2.

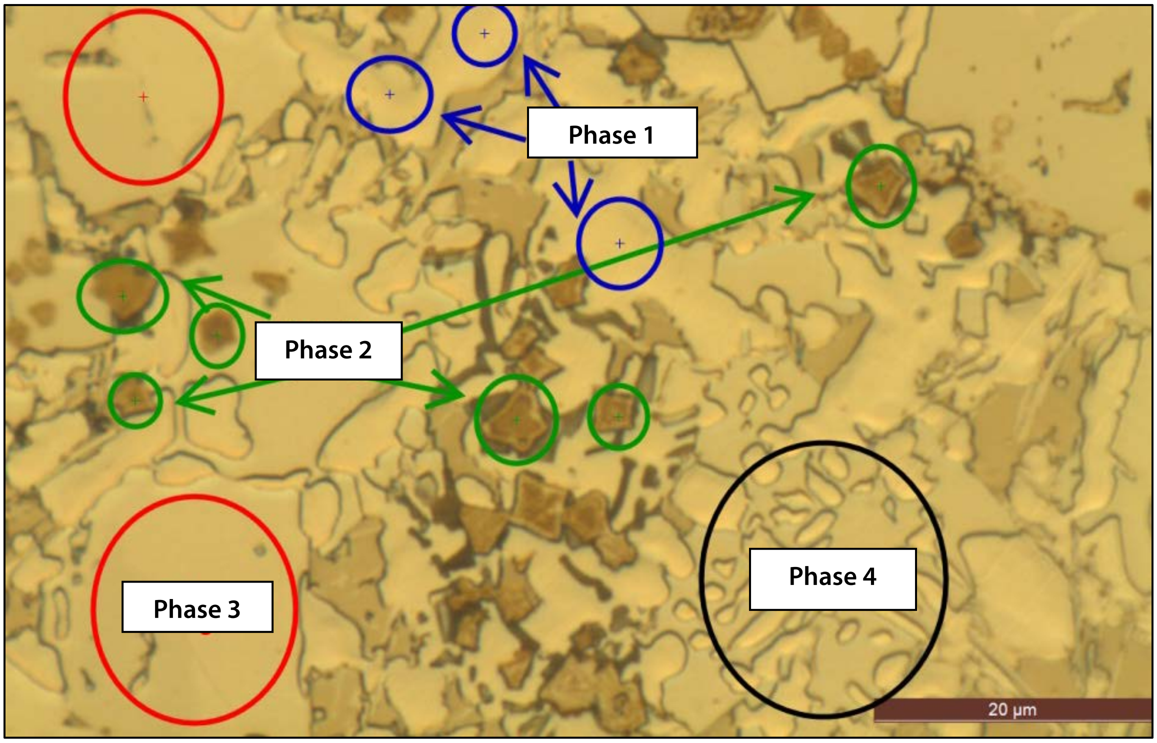

Fig. 2: Metallograph under the optical microscope.

The patent pending *advanced stage controller* is an intuitive sample navigation tool which automatically adjusts the speed of sample movement under the optical microscope based on position of the mouse. The further the mouse is away from the center of field of view, the faster the stage moves toward the mouse's direction. This provides a user-friendly method to navigate the entire sample surface and select the intended location for mechanical testing. The coordinates of the test locations are saved and numbered, along with individual test setup, such as loads, loading/unloading rate, number of tests in a map, etc. Such a test procedure allows user to examine a large sample surface for specific areas of interest for indentation and perform all the indentation tests at different locations in one time, making it an ideal tool for mechanical testing of metallurgical sample with different phases.

In this study, we located the specific phases of the sample under the optical microscope integrated in the Nanovea Mechanical Tester as numbered on Fig. 3. The coordinates of the selected locations are saved, followed by automatic nanoindentation tests all at once under the test conditions summarized in Table 1.

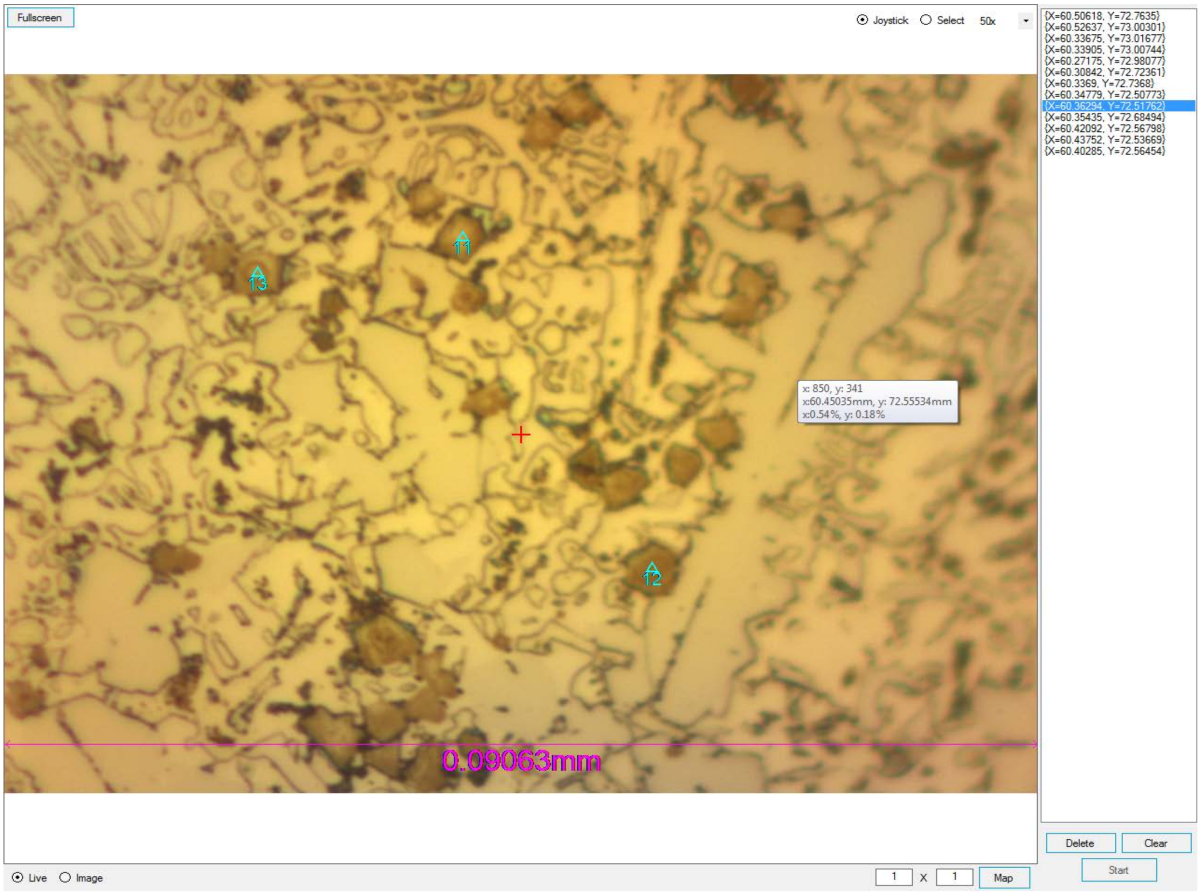

Fig. 3: Selecting nanoindentation location on the sample surface.

Maximum Force (mN)	50
Loading Rate (mN/min)	100
Unloading Rate (mN/min)	100
Creep (s)	5.0
Computation Method	ASTM E-2546 & Oliver & Pharr
Indenter Type	Berkovich

Table 1: Test conditions of the nanoindentation.

RESULTS AND DISCUSSION

The indentations at the different phases of the sample are displayed in Fig. 4. We demonstrate that the excellent position control of the sample stage in Nanovea Mechanical Tester allows users to precisely pinpoint the target location for mechanical properties testing.

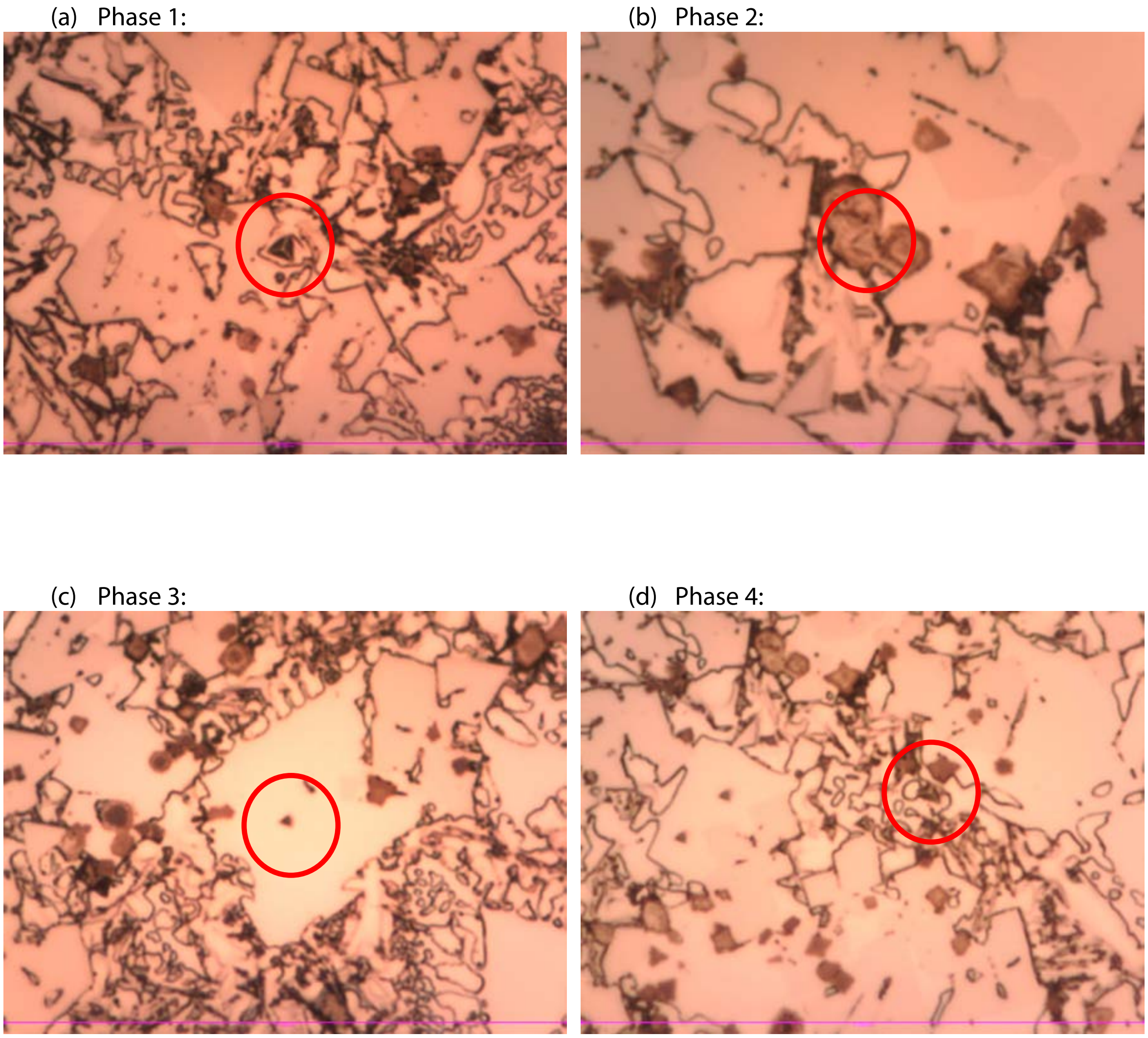

Fig. 4: Nanoindentations on different phases.

The representative load-displacement curves of the indentations are shown in Fig. 5, and the corresponding hardness and Young's Modulus calculated using Oliver and Pharr Method [iii] are summarized and compared in Fig. 6. The Phases 1, 2, 3 and 4 possess an average hardness of ~5.4, 19.6, 16.2 and 7.2 GPa, respectively. The relatively small size for Phases 2 contributes to its higher standard deviation of the hardness and Young's Modulus values.

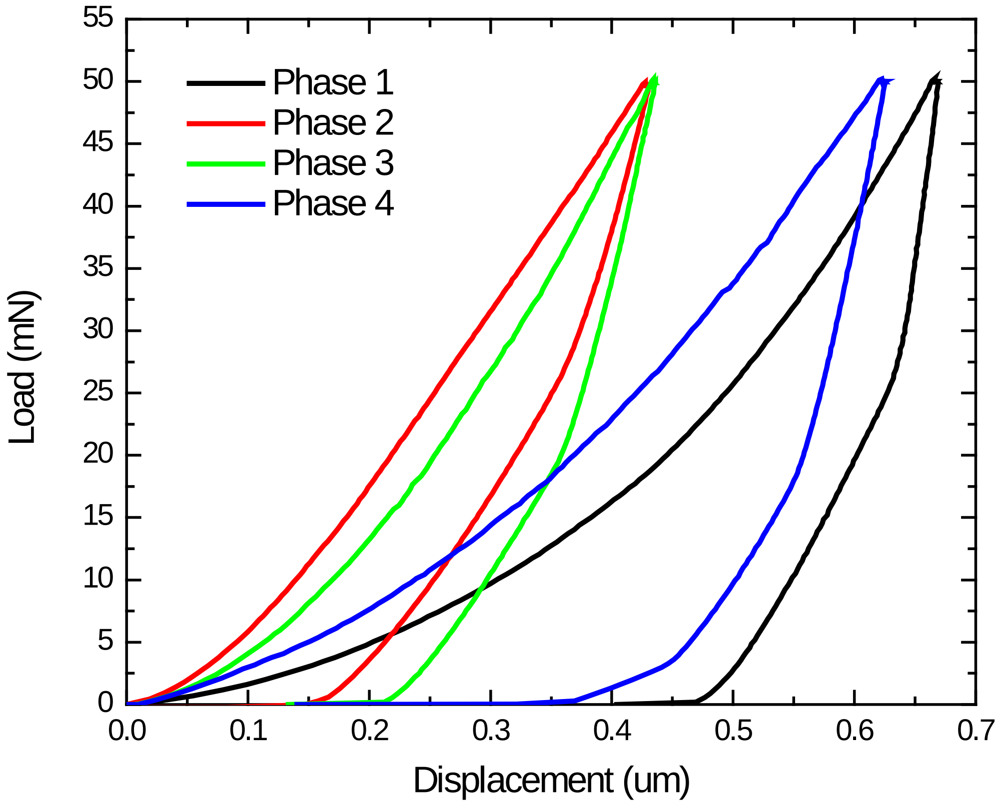

Fig. 5: Load-displacement curves of the nanoindentations.

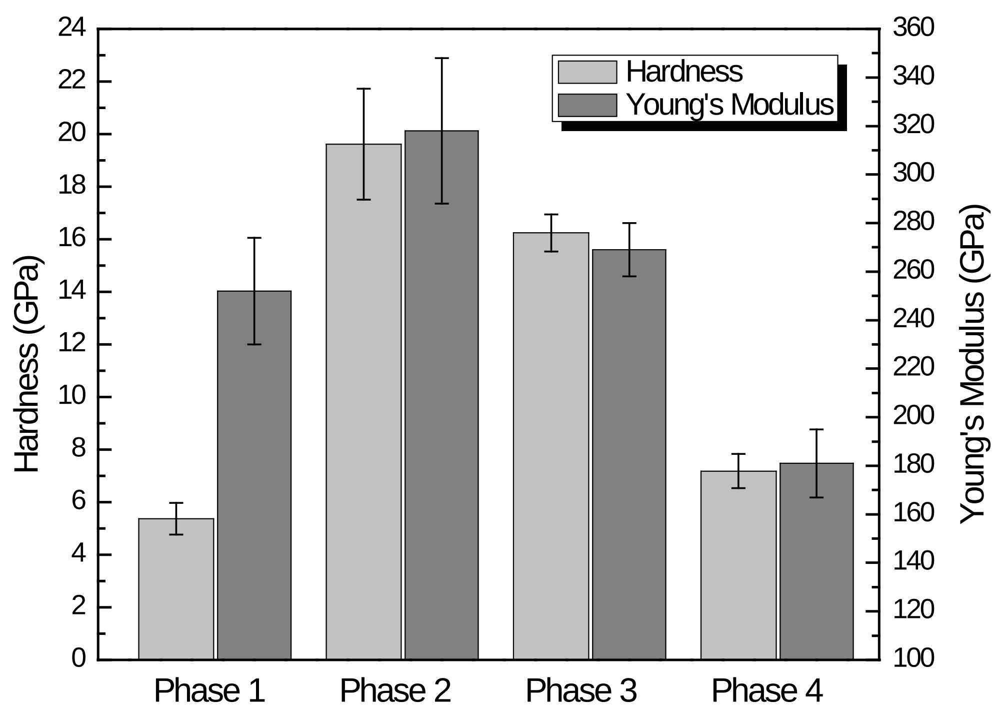

Fig. 6: Hardness and Young's Modulus of different phases.

CONCLUSION

In this study, we showcased that Nanovea Mechanical Tester performed nanoindentation measurements on multiple phases of a large metallurgical sample using patent pending advanced *STAGE CONTROLLER*. The precise position control allows users to easily navigate a large sample surface and directly select the areas of interest for nanoindentation measurements. The location coordinates for the indentations are saved and all the indentation tests are performed at once. Such a test procedure makes measurement of the local mechanical properties at small scales, e.g. multi-phase metal sample in this study, substantially less time-consuming and more user friendly. The hard Phases 2, 3 and 4 improve the mechanical properties of the sample, possessing an average hardness of ~19.6, 16.2 and 7.2 GPa, respectively, compared to ~5.4 GPa for Phase 1.

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.

To learn more about Nanovea Mechanical Tester or Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

Nanoindentation 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 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 250 μm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.15 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 μN
Load Resolution (Noise Floor)	: 0.3 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.

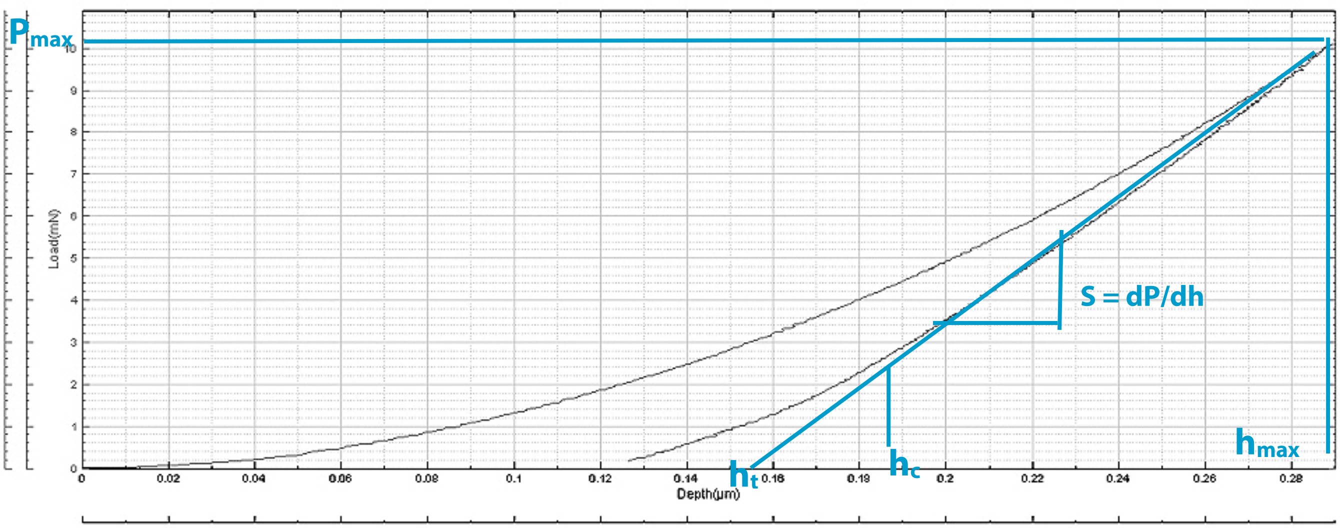

Fig. 7: Load-displacement curve of nanoindentation.

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's ratio of the indenter and ν the Poisson's ratio 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 A_c 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=2πRh_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 d*ε*/d*t* 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 is a better choice.

Other possible measurements by Nanovea Mechanical Tester:

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

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[i] Oliver, W. C.; Pharr, G. M., Journal of Materials Research., Volume 19, Issue 1, Jan 2004, pp.3-20

[ii] Schuh, C.A., Materials Today, Volume 9, Issue 5, May 2006, pp. 32–40

[iii] Oliver, W. C.; Pharr, G. M., Journal of Materials Research, Volume 7, Issue 6, June 1992, pp.1564-1583