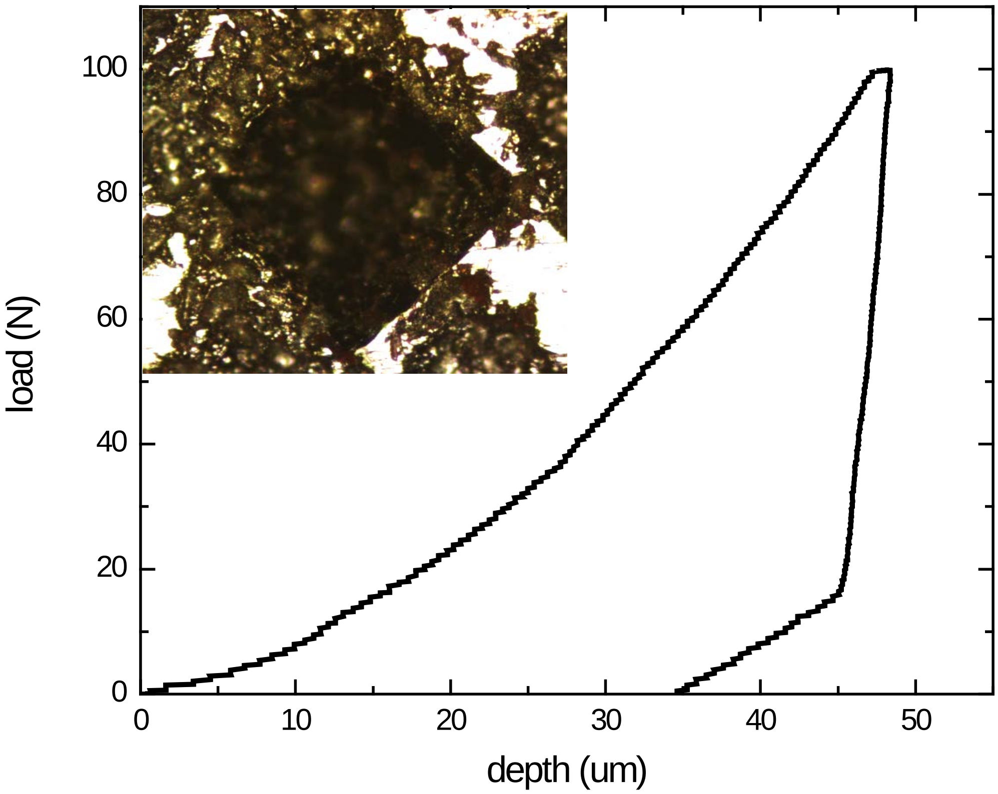
VICKERS HARDNESS VS. MACRO INSTRUMENTED INDENTATION – A COMPARATIVE STUDY



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INTRO

Macroindentation hardness tests are widely used to determine the overall hardness of a material. There are a variety of macrohardness measurements, including but not limited to Vickers hardness test (HV), Brinell hardness test (HB), Knoop hardness test (HK) and Rockwell hardness test (HR). With one of the largest scales among hardness tests, the Vickers test is widely used for measuring the hardness of all metals. As shown in Fig. 1[i], Vickers hardness uses a diamond in the form of a square-based pyramid with an angle to the horizontal plane of 22° on each side. It indents on the sample surface and creates a square imprint. By measuring the average length of the diagonal, *d*, the Vickers hardness can be calculated using the formula:

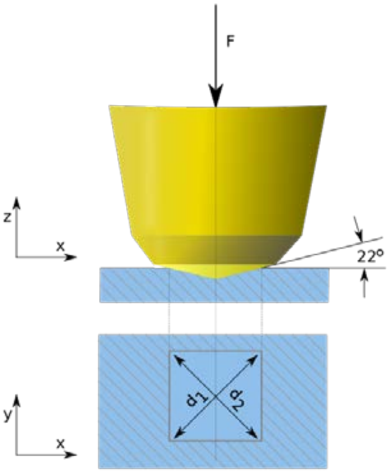


Fig. 1: Vickers hardness scheme.

$$HV = \frac{F}{A} \approx \frac{0.01819F}{d^2},$$

where *F* is in N and *d* is in millimeters. Here, accurate measurement of the *d* value is critical in order to obtain accurate hardness values.

In comparison, instrumented indentation technique directly measures the mechanical properties from indentation load & displacement measurements. No visual observation of the indent is required; eliminating user error in determining the *d* values of the indentation.

MEASUREMENT OBJECTIVE

In this study, we showcase the repeatability and accuracy of the Nanovea Mechanical Tester in testing macro hardness and Young's modulus of metals using instrumented indentation method compared to conventional Vickers hardness measurement.

Fig. 2: Indentation of the SS304 sample.

TEST PROCEDURE

The hardness and Young's modulus of an unpolished stainless steel SS304 sample were measured using the Macro Mode of Nanovea Mechanical Tester with a high load of 100 N. The load-displacement curve was measured in situ during the test. The Vickers indentation was examined under an optical microscope to determine the Vickers hardness based on the measurement on the indentation. The hardness calculated using the Oliver and Pharr method and conventional Vickers test is compared. The test conditions are summarized in Table 1.

Sample	SS304
Applied Force (N)	100
Loading rate (N/min)	200
Unloading rate (N/min)	200
Indenter type	Vickers diamond

Table 1: Test conditions of hardness and Young's modulus.

RESULTS AND DISCUSSION

The Vickers hardness by conventional Vickers indentation measurement

The hardness of the SS304 sample was tested at different locations using a Vickers diamond tip as shown in Fig. 3. The Vickers tip has a symmetric square shape. The rough surface finish of the steel sample makes it difficult and inaccurate to identify the two diagonals of the indentation, leading to significant inconsistency in the hardness calculation.

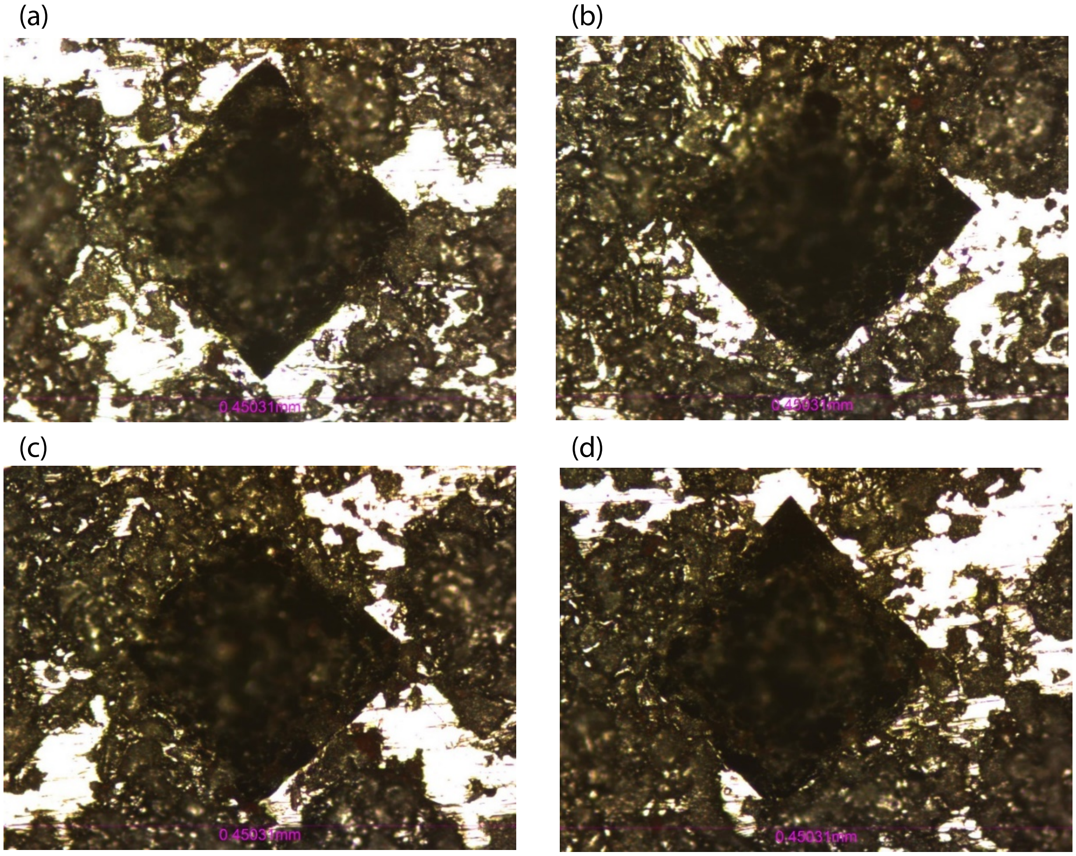


Fig. 3: Indentations at different locations of the SS304 sample (100X).

Hardness and Young's modulus by instrumented indentation measurement

The load-displacement curves are shown in Fig. 4 and the test results are listed in Table 2. It can be observed that the *H* and *E* values calculated using Oliver and Pharr method[ii] at different locations on the sample surface exhibit much lower Standard Deviation. The Vickers hardness is 208.9±5.9 HV, compared to 268.9±35.7 HV for the Vickers hardness calculated using conventional impression measurement.

In addition to hardness measurement, we can also determine the value of Young's modulus *E* at 137.5±14.4 GPa using Oliver and Pharr method. Instrumented indentation allows us to directly determine the mechanical properties from indentation load and displacement measurements without measuring the impression size under the microscope. It eliminates user errors in determining the indention size, which is especially difficult for the rough samples.

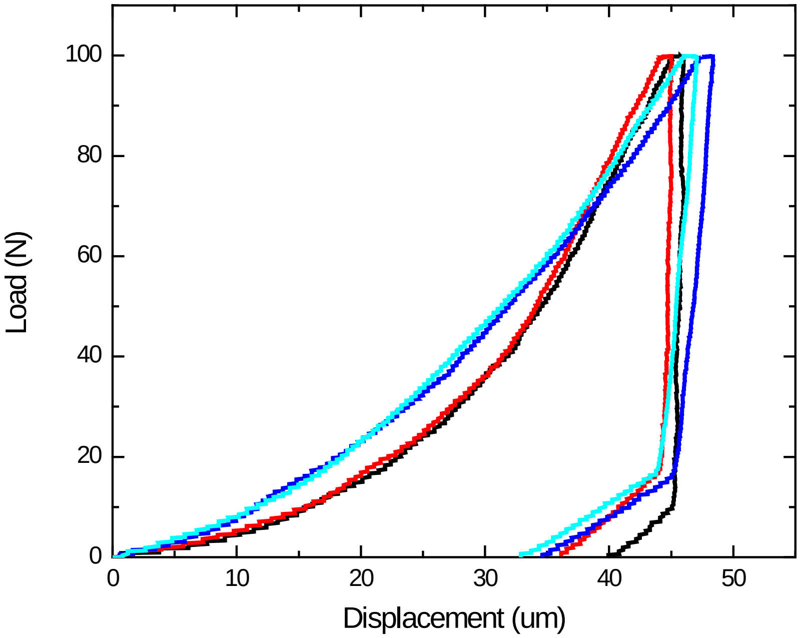


Fig. 4: Load-displacement curves of the indentations at different locations.

	Conventional Vickers Hardness	Instrumented Indentation Hardness		
	Vickers Hardness (HV)	Vickers Hardness (HV)	Hardness (GPa)	Modulus (GPa)
Test 1	262.4	205.8	2.2	157.1
Test 2	243.4	213.5	2.3	123.1
Test 3	321.1	214.1	2.3	137.7
Test 4	248.6	202.2	2.1	132.0
Average	268.9	208.9	2.2	137.5
St Dev	35.7	5.9	0.1	14.4

Table 2: Comparison of results based on Conventional Vickers Hardness and Instrumented Indentation Hardness methods.

CONCLUSION

In this indentation study, we show that Nanovea Mechanical Tester equipped with a Vickers tip is an ideal tool for analyzing the macro mechanical properties of the metal materials. Compared to conventional macro Vickers hardness measurement, the instrumented indentation method used by Nanovea Mechanical Tester does not require visual observation of the indent and eliminates user error in determining indent size. It significantly increases the repeatability and accuracy of the hardness and Young's modulus measurements, and therefore improves consistent mechanical properties measurement on rough samples.

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. The Nanovea Tribometer offers precise and repeatable wear and friction testing using ISO and ASTM compliant rotative and linear modes, with optional high temperature wear, lubrication and tribo-corrosion modules available in one pre-integrated system. 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 and Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

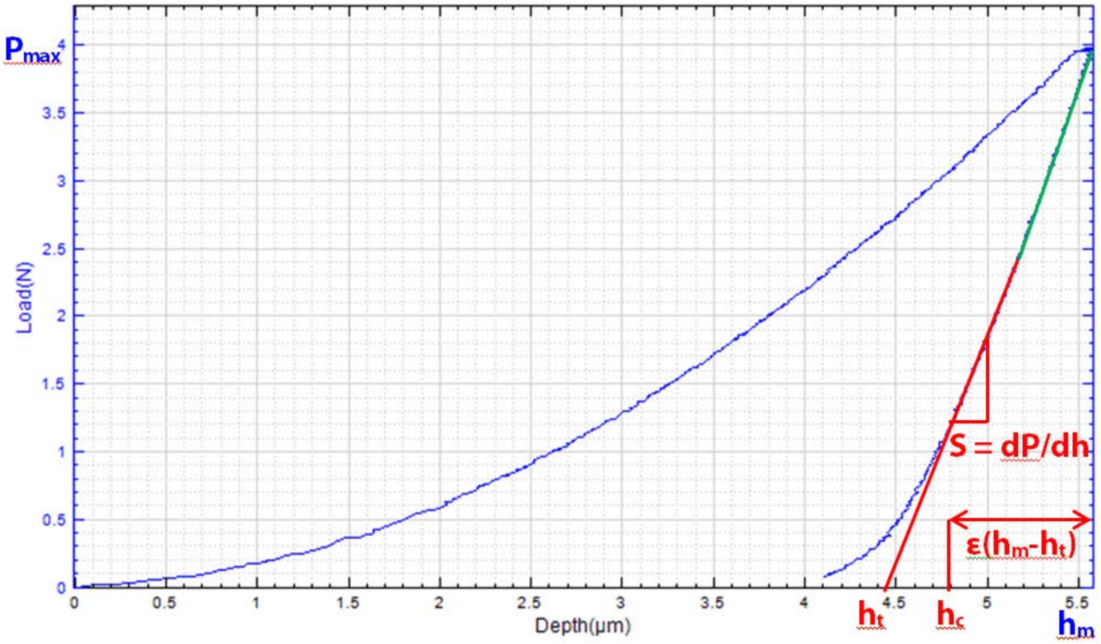
PRINCIPLE OF INSTRUMENTED INDENTATION TEST

The indentation test 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. During the experiment the position of the indenter relative to the sample surface is precisely monitored.

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.

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.



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 $A_c=2\pi Rh_c$, 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 $d\varepsilon/dt$ 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.

[i] https://en.wikipedia.org/wiki/Vickers_hardness_test#/media/File:Vickers-path-2.svg

[ii] W.C. Oliver and G.M. Pharr, J. Mater. Res. 7, 1564 (1992)