NANOVEA®
A Better Measure.

M3 Review
Automated Nanoindentation

Prepared by
Duanjie Li, PhD & Pierre Leroux

6 Morgan, Ste156, Irvine CA 92618 · P: 949.461.9292 · F: 949.461.9232 · nanovea.com

INTRODUCTION

Traditional hardness testing

Surface indentation test is a widely used technique that determines the hardness, the resistance to plastic deformation of a material. Conventional indentation hardness tests such as Rockwell, Vickers, Knoop, and Brinell apply a certain load on the indenter tip (usually made of a hard material) and press it in to the test sample. The user-defined load is then held constant for a period and removed, leaving a residual indentation imprint on the material. The area of this imprint is measured under the optical microscope, and the hardness, H, is calculated using the following equation:

$H = \frac{P_{\max}}{A_c}$ where $P_{\max}$ is the maximum load, and A_c is the projected contact area.

The detailed test procedures of the traditional indentation hardness measurements are documented in the ASTM standards, such as E 10, Test Method for Brinell Hardness of Metallic Materials; E 18, Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials; E 92, Test Method for Vickers Hardness of Metallic Materials; and E 384, Test Method for Microindentation Hardness of Materials, etc.

The conventional indentation hardness tests have several inevitable disadvantages:

1) The strain hardening effect takes place in the indentation tests at a high load and become a major source of error.
2) It may introduce user errors to observe the imprint and determine its edge and size under the microscope.
3) Errors also arise from the recovery of the material underneath the indentation, or piling-up or sinking-in of the surrounding material, which changes the size of the indentation.
4) The large indenter size makes it difficult to locate the area of interest for indentation.
5) The varied tip shapes make it difficult to compare test results using different tips.

Instrumented indentation testing

Instrumented indentation testing (IIT) is a measurement procedure that evaluates the mechanical properties of a material by driving an indenter tip with geometry known to high precision (usually a Berkovich tip) into the test material and recording the evolution of load and displacement of the tip in situ as shown in **Fig. 1**.

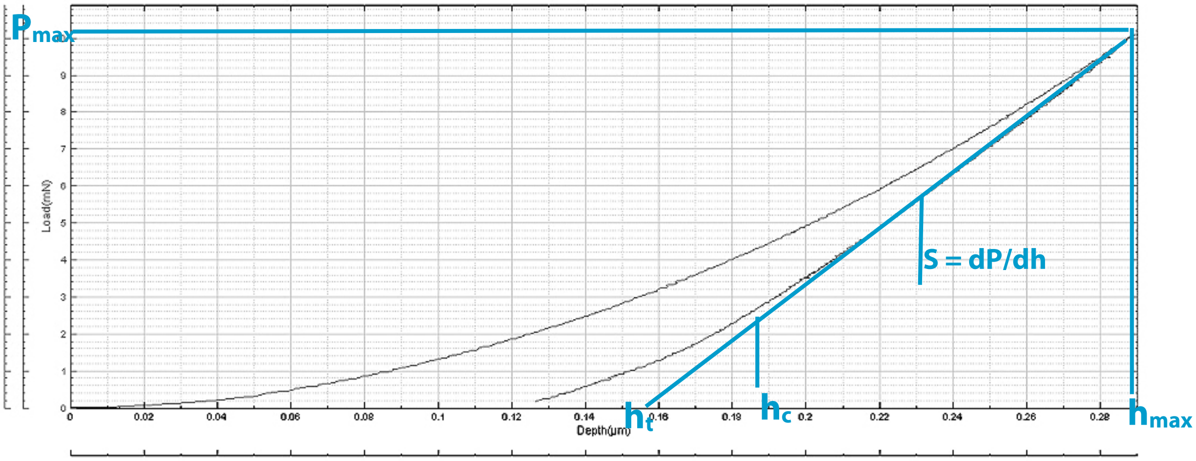


Fig. 1: Load-displacement curve of instrumented indentation.

The hardness and Young's modulus can be determined from the Load-displacement curve in **Fig. 1**.

The hardness is determined from the maximum load, $P_{\max}$, divided by the projected contact area, A_c:

$$H = \frac{P_{\max}}{A_c}$$

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.

As illustrated in **Fig. 1**, 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 projected 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.

Compared to conventional hardness test, IIT can measure other important mechanical properties of the material, such as young's modulus. The detailed in-situ recorded load-displacement curve provides more insight in the mechanical behavior of the material under the load. For example, initiation and progression of cracks of the brittle material can be observed as discontinuities in the loading curve. Creep of the material is reflected as a plateau at the constant maximum load stage of the load-displacement curve.

Moreover, when the subject materials are hard thin films, small indentation loads are required to avoid the influence of the substrate deformation during indentation. At low loads, small measurement errors of the indent size under the microscope will produce large hardness deviations. In comparison, IIT can perform precise load and displacement measurement in the nanometer range, making IIT an ideal technique for evaluating the properties of thin films, coatings and surfaces with special heat-treatment or ion implantation.

Being a high precision technique for mechanical properties testing, however, IIT usually requires systematical user training for proper operation of the system and interpretation of the measured results. Many factors have to be taken into account to obtain accurate measurement, such as contact point selection, indenter shape correction, instrument frame compliance determination, etc. An IIT instrument with the capacity of performing nanoindentation generally costs above $100k. The requirement of special expertise and big budget substantially limits the further application of IIT technique in a broader market.

An affordable, user-friendly and fully-automatic IIT system is in need. Nanovea M3 Mechanical Tester has a fully automated measurement procedure and a price in the $20K market, bringing high-end measurement technology to the broader market.

MEASUREMENT OBJECTIVE

In this application, the Nanovea M3 Mechanical Tester is used to measure the hardness, Young's modulus and creep of different types of materials, including metal, glass and polymer, in order to showcase the simplicity, reliability and repeatability of this breakthrough indentation system.

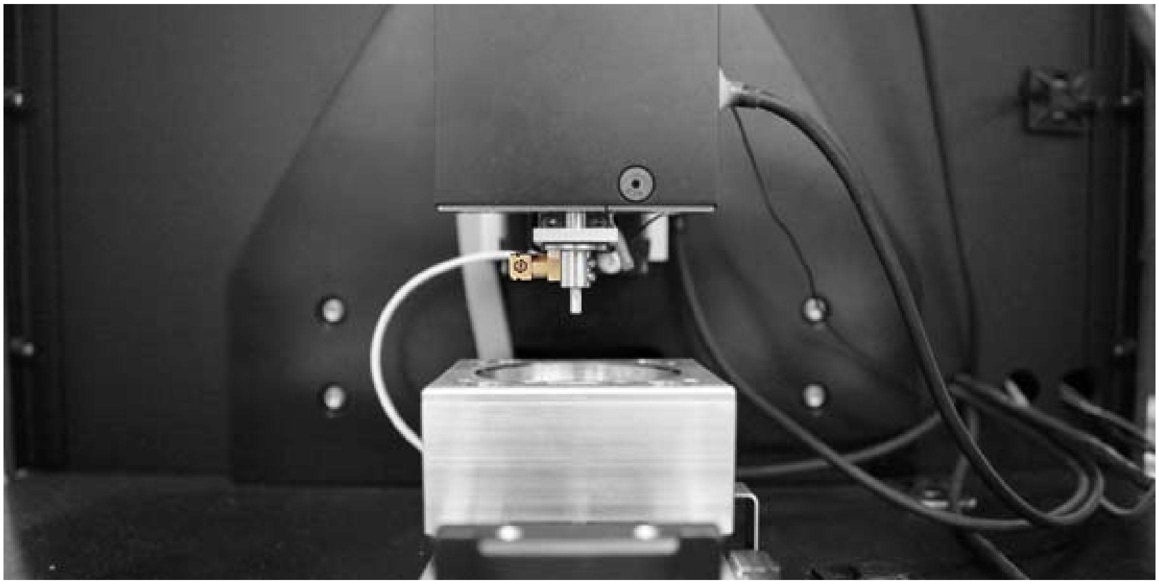


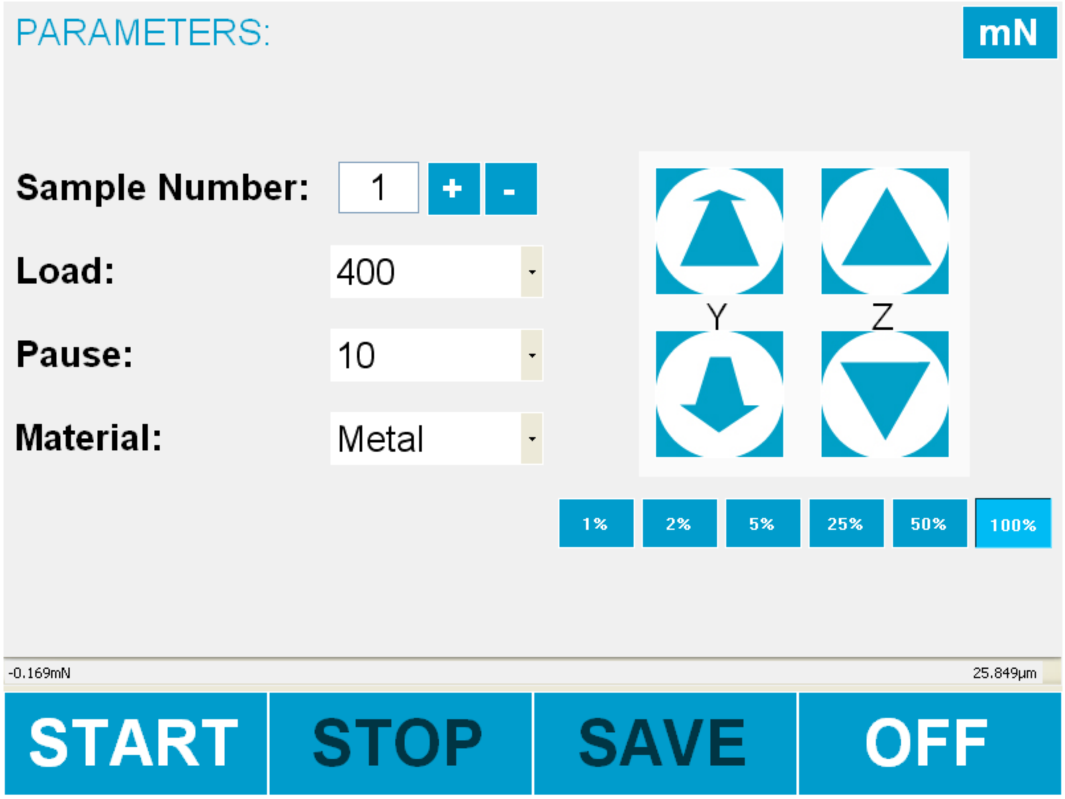
Fig. 2: Sample holder inside the M3.

TEST CONDITIONS & PROCEDURES

The hardness, *H*, Young's modulus, *E*, and creep of several materials were evaluated using Nanovea M3 Mechanical Tester equipped with a Berkovich tip. The test samples possess mirror-like surface finish to avoid the influence of surface roughness. As shown in Fig. 3, the M3 system has a clean and simple user interface for test condition setup displayed on a touchscreen. The drop-down menu allows selection of the maximum load, pause time at the maximum load and the material type, and Y and Z position control allows finding the intended test location on the sample. The test begins after the "START" bottom is clicked. The following procedure will be performed:

1) Automatically engage the indenter to find the sample surface.

2) Perform five indentations near the selected location using the test parameters given by the user and record the depth versus load during the tests.
3) Conduct automated calculation of the *H&E* and creep depth values.



Sample Number: 1
Load: 400
Pause: 10
Material: Metal

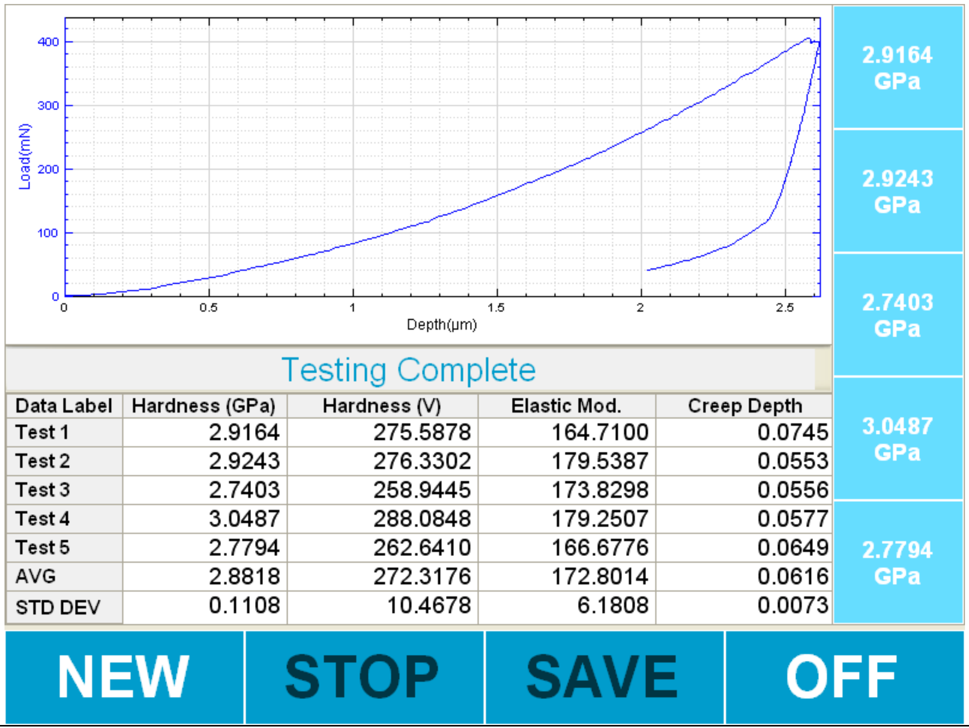
Material	Fe alloy	Al alloy	Fused silica	Acrylic
Applied Load (mN)	400	400	400	200
Loading rate (mN/min)	800	800	800	400
Unloading rate (mN/min)	800	800	800	400
Pause at maximum load (s)	10	10	10	10

Table 1: Summary of test parameters applied.

RESULTS AND DISCUSSION

The test results of the Fe alloy, Al alloy, Fused silica and Acrylic samples were automatically calculated and displayed on the touchscreen as shown in Fig. 4. Table 2 summarizes and compares the measured values of these samples. Among the test samples, Fused silica exhibit the highest hardness of 8.05 GPa and lowest creep depth of 0.0144 µm in 10 s pause. Fe and Al alloys show an intermediate hardness of 2.88 and 1.42 GPa, respectively, and slightly increased creep depth of 0.0616 and 0.0520 µm. In comparison, the Acrylic polymer sample is relatively soft and viscous, showing a low hardness of 0.36 GPa and large creep depth of 0.4436 µm. Fe has the highest elastic modulus of 172.8 GPa among the test materials, compared to that of 3.78 GPa for Acrylic. The low Std Dev in this study demonstrates the outstanding repeatability of the M3 system.

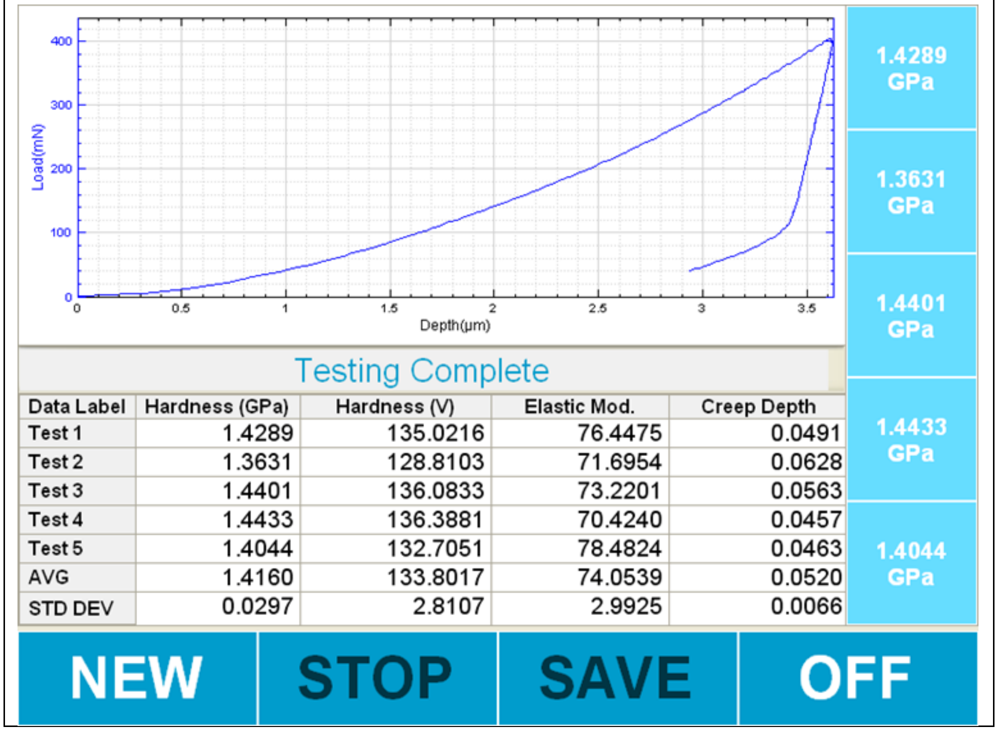
(a) Fe alloy:



Testing Complete

Data Label	Hardness (GPa)	Hardness (V)	Elastic Mod.	Creep Depth
Test 1	2.9164	275.5878	164.7100	0.0745
Test 2	2.9243	276.3302	179.5387	0.0553
Test 3	2.7403	258.9445	173.8298	0.0556
Test 4	3.0487	288.0848	179.2507	0.0577
Test 5	2.7794	262.6410	166.6776	0.0649
AVG	2.8818	272.3176	172.8014	0.0616
STD DEV	0.1108	10.4678	6.1808	0.0073

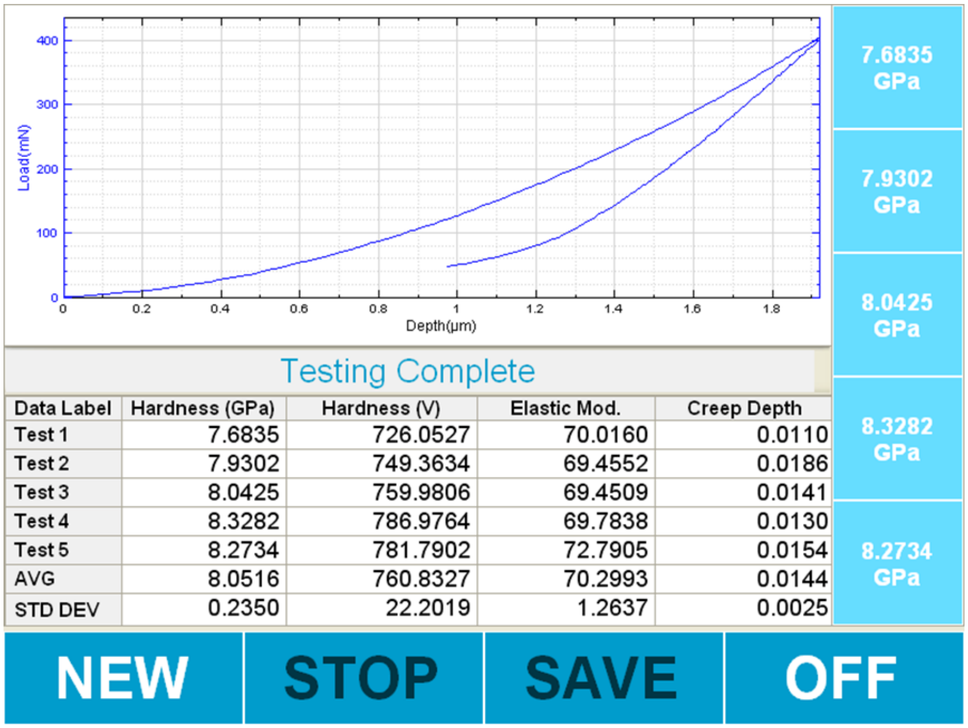
(b) Al alloy:



Testing Complete

Data Label	Hardness (GPa)	Hardness (V)	Elastic Mod.	Creep Depth
Test 1	1.4289	135.0216	76.4475	0.0491
Test 2	1.3631	128.8103	71.6954	0.0628
Test 3	1.4401	136.0833	73.2201	0.0563
Test 4	1.4433	136.3881	70.4240	0.0457
Test 5	1.4044	132.7051	78.4824	0.0463
AVG	1.4160	133.8017	74.0539	0.0520
STD DEV	0.0297	2.8107	2.9925	0.0066

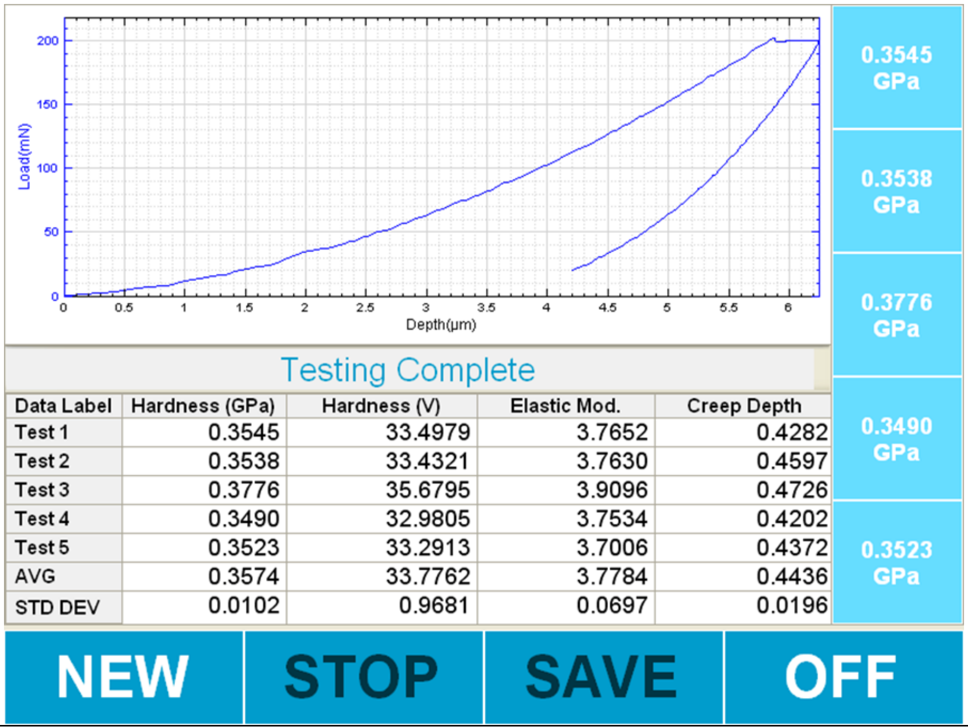
(c) Fused silica:



Testing Complete

Data Label	Hardness (GPa)	Hardness (V)	Elastic Mod.	Creep Depth
Test 1	7.6835	726.0527	70.0160	0.0110
Test 2	7.9302	749.3634	69.4552	0.0186
Test 3	8.0425	759.9806	69.4509	0.0141
Test 4	8.3282	786.9764	69.7838	0.0130
Test 5	8.2734	781.7902	72.7905	0.0154
AVG	8.0516	760.8327	70.2993	0.0144
STD DEV	0.2350	22.2019	1.2637	0.0025

(d) Acrylic:



Testing Complete

Data Label	Hardness (GPa)	Hardness (V)	Elastic Mod.	Creep Depth
Test 1	0.3545	33.4979	3.7652	0.4282
Test 2	0.3538	33.4321	3.7630	0.4597
Test 3	0.3776	35.6795	3.9096	0.4726
Test 4	0.3490	32.9805	3.7534	0.4202
Test 5	0.3523	33.2913	3.7006	0.4372
AVG	0.3574	33.7762	3.7784	0.4436
STD DEV	0.0102	0.9681	0.0697	0.0196

Fig. 3: Result screens of (a) Fe alloy; (b) Al alloy; (c) Fused silica and (d) Acrylic.

Material	Hardness (GPa)	Hardness (HV)	Elastic modulus (GPa)	Creep Depth (µm)
Fe alloy	2.88±0.11	272.3±10.5	172.8±6.2	0.0616±0.0073
Al alloy	1.42±0.03	133.8±2.8	74.1±3.0	0.0520±0.0066
Fused silica	8.05±0.24	760.8±22.2	70.3±1.3	0.0144±0.0025
Acrylic	0.36±0.01	33.8±1.0	3.78±0.07	0.4436±0.0196

Table 2: Comparison of hardness, Elastic modulus and Creep depth of the Fe alloy, Al alloy, Fused silica and Acrylic samples.

Nanovea M3 system provides fully automated indentation measurement of mechanical properties while maintaining a competitive price of $20K. It makes high-end nanoindentation technology available and affordable to the broader market such as smaller R&D units and quality control lines. The M3 system automatically takes into account the factors such as contact point, indentation shape correction and frame compliance, and calculates the hardness, elastic modulus and creep. Its fully automated and user-friendly test procedure provides simple and direct result presentation, making complicated nanoindentation technique more accessible to broader average users.

Compared to conventional micro Vickers hardness testers, M3 system does not require visual observation of the indent and eliminates user error in determining indent size. Its capacity in low load hardness measurement enables broader test material range, including ceramics, polymers, and metals, as well as thin films and coatings.

CONCLUSION

In this study, we showcased the capacity of Nanovea M3 Mechanical Tester in evaluating mechanical properties of various materials in a reliable and repeatable manner following ASTM E2546 standard. The simple experimental setup and full automation of this system make nanoindentation technique accessible to more users. It is designed to price in the $20K market in order to bring high-end measurement technology to the broader market. The combination of competitive price and fully-automated high-precision measurement makes M3 system an ideal next-generation technology to replace the traditional standard hardness-testing equipment, such as visual micro Vickers hardness testers.

To learn more about Nanovea M3 Mechanical Tester or Lab Services.