LOW LOAD VICKERS HARDNESS MEASUREMENT USING NANOINDENTATION

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INTRODUCTION

Vickers hardness indentation test is a widely used technique that determines the hardness – the resistance to plastic deformation of a material. Compared with other conventional indentation hardness tests such as Rockwell, the Vickers hardness calculation is independent of the size of the indenter test. Therefore, the indenter can be used for all materials irrespective of hardness. Following the ASTM E92 standard[i], the user-defined load is applied by a Vickers diamond tip with a square-based pyramid shape. It is then held constant for a period and removed, leaving a residual indentation imprint on the material surface. The edge and size of the imprint is measured under the optical microscope. The hardness, *HV*, is calculated using the following equation: $HV = P_{max} / A_c$, where P_{max} is the maximum load, and A_c is the projected contact area.

Inevitable user errors are introduced during the measurement of the imprint under the microscope. Especially at low loads, small measurement errors of the indent size will produce large hardness deviations. In comparison, nanoindentation testing evaluates the mechanical properties of a material by driving the indenter tip into the test material and precisely recording the evolution of load and displacement of the tip. It avoids user errors in imprint size measurement.

Nanovea M3 Mechanical Tester provides fully automated Vickers hardness measurement using nanoindentation technique. Its price in the $30K range brings the 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 expand the Vickers hardness testing to low load applications, e.g. thin films.

MEASUREMENT OBJECTIVE

In this application, the Nanovea M3 Mechanical Tester is used to measure the Vickers hardness and Young's modulus of two types of metal samples, aluminum and steel using nanoindentation technique. The measured hardness values are compared with conventional Vickers hardness obtained by measuring the size of the Vickers imprint.

Fig. 1: Vickers indenter on the test sample.

TEST CONDITIONS & PROCEDURES

The Vickers hardness, *HV*, and Young's modulus, *E*, of Steel and Aluminum samples were evaluated using Nanovea M3 Mechanical Tester equipped with a Vickers 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 parameter 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 *HV&E* and creep depth values from the load-displacement curves.

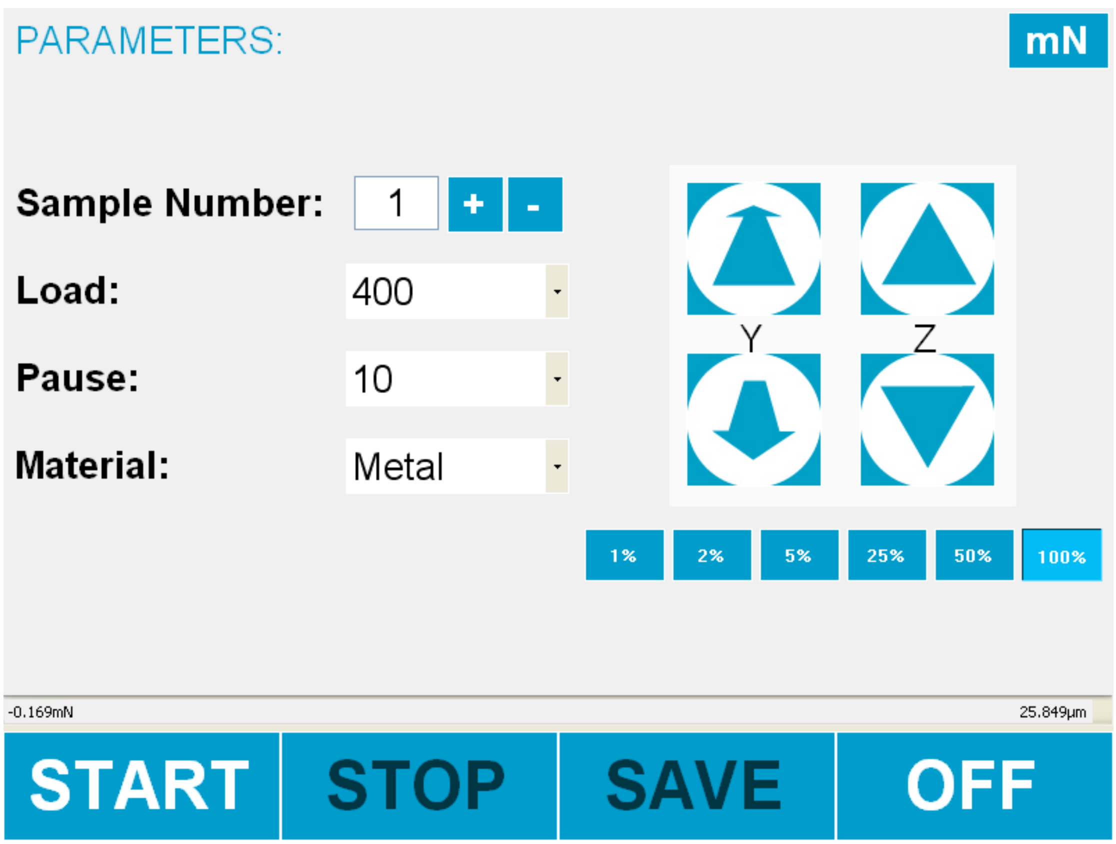

Fig. 2: User interface for test parameter input.

The test conditions are listed in Table 1. Following the indentation tests, the Vickers hardness were automatically calculated by M3 system and displayed on the touchscreen.

Applied Load (mN)	400
Loading rate (mN/min)	800
Unloading rate (mN/min)	800
Pause at maximum load (s)	15

Table 1: Summary of test parameters.

As shown in Fig. 3[ii], conventional 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:

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

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.

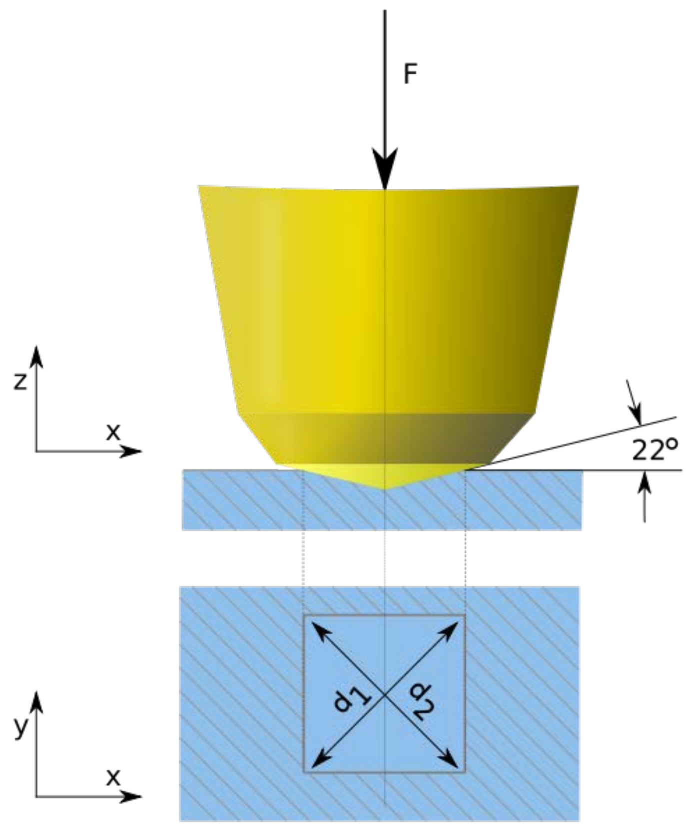

Fig. 3: Vickers hardness scheme.

The imprints of the indenters after the Vickers indentation tests were measured under an optical microscope and the Vickers hardness were calculated. The Vickers hardness obtained by the two methods, namely nanoindentation and conventional Vickers hardness were compared to showcase the consistency of these two test methods.

RESULTS AND DISCUSSION

The test results of the Steel and Aluminum samples were automatically calculated and displayed on the touchscreen of the M3 system. The load-displacement curves of the Steel and the Aluminum samples are shown in Fig. 4, and the Indentation on Steel and Aluminum samples under the microscope are displayed in Fig. 5. Fig. 6 summarizes and compares the Vickers hardness values derived from conventional Vickers hardness test and nanoindentation. It can be observed that the Vickers hardness values calculated from two different methods are in agreement. The Steel sample exhibits a higher hardness of ~330 HV, compared to a Vickers hardness of ~250 HV for the Aluminum.

The Vickers hardness values calculated using nanoindentation method exhibits lower standard deviation, which demonstrates the outstanding repeatability of the M3 system. Compared to conventional Vickers hardness test, Nanovea M3 system performs precise load and displacement measurement in the nanometer range and avoids user errors during observing the imprint and determining its edge and size under the microscope.

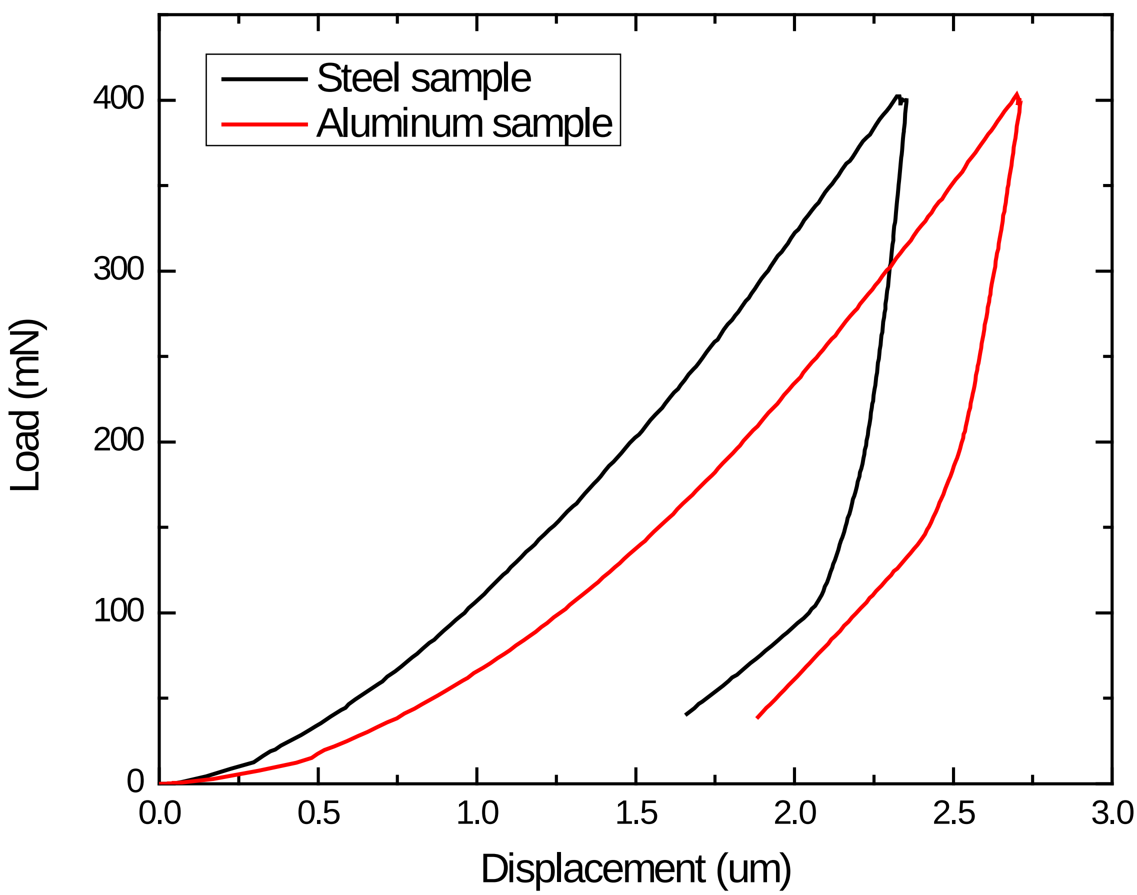

Fig. 4: Load-displacement curves of the Steel and Aluminum samples.

(a) Steel sample: (b) Aluminum sample:

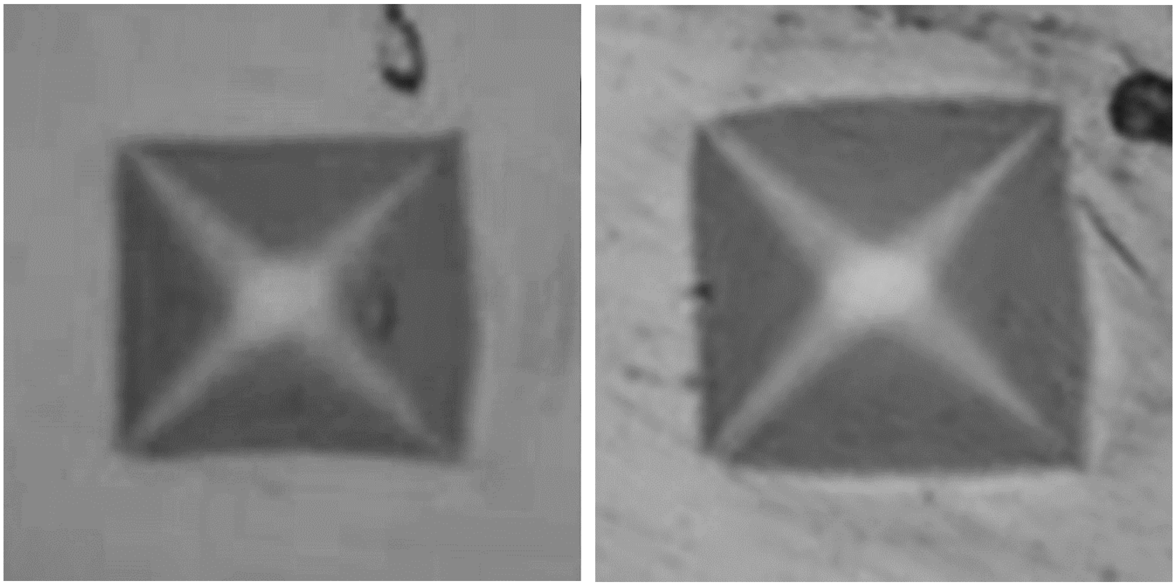

Fig. 5: imprints on Steel and Aluminum samples under the microscope.

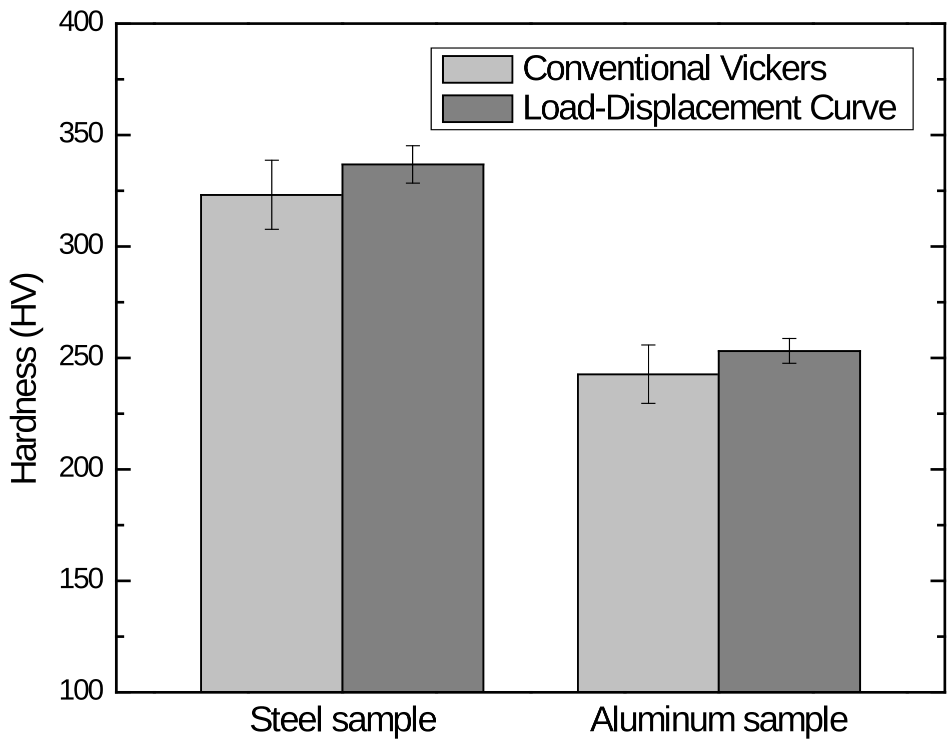

Fig. 6: Comparison of Vickers hardness values derived from conventional Vickers hardness test and nanoindentation using load-displacement curves.

Compared to conventional Vickers hardness test, Nanovea M3 system using nanoindentation method can measure other important mechanical properties of the material, such as young's modulus as shown in Fig. 7. In addition, the detailed in-situ recorded load-displacement curve may provide 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.

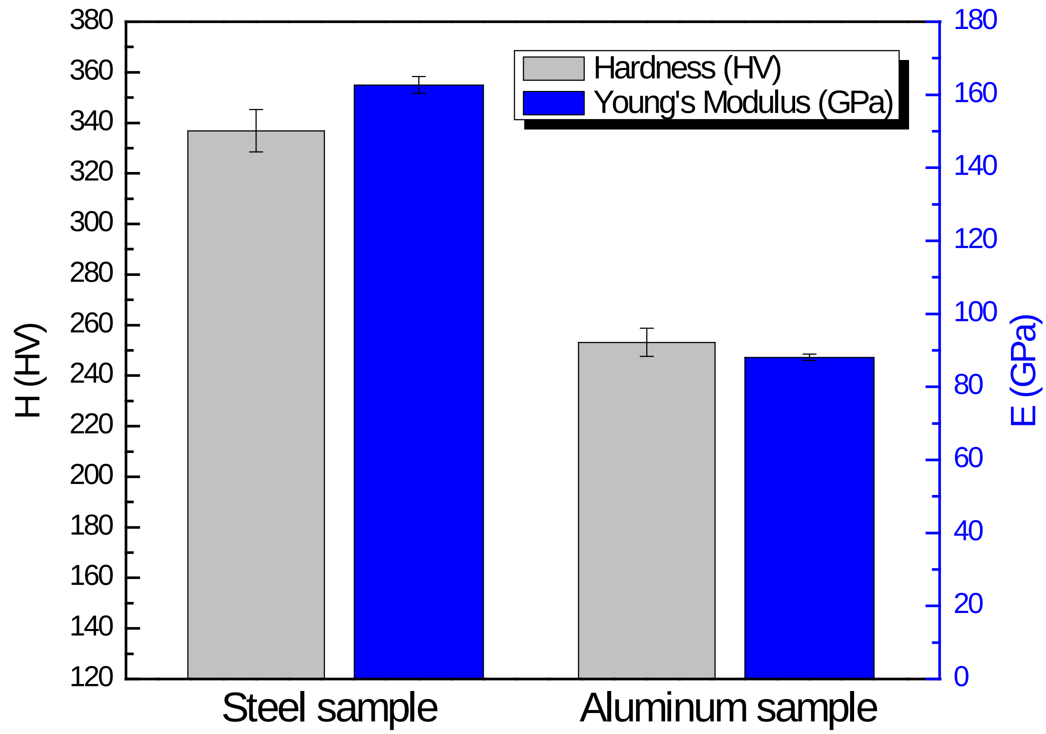

Fig. 7: Hardness and Young's Modulus of the Steel and Aluminum samples.

CONCLUSION

In this study, we showcased that Nanovea M3 system provides fully automated Vickers hardness measurement in a reliable and repeatable manner. The Vickers hardness values provided by M3 system is in a good agreement with that by conventional Vickers hardness measurement. 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.

Nanovea M3 system provides fully automated mechanical properties measurement while maintaining a competitive price of $30K. 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.

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

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[i] **http://www.astm.org/Standards/E92.htm**
[ii] **https://en.wikipedia.org/wiki/Vickers_hardness_test#/media/File:Vickers-path-2.svg**