HARDNESS DISTRIBUTION OF HARDENED METAL USING NANOINDENTATION

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INTRO

Surface hardening is a process that hardens the surface of a metal object while maintains the softness and toughness of the core metal deeper underneath. The combination of hard surface and soft interior metal is important for achieving superior resistance to breakage upon impact such as high stress and fatigue, while enhancing the wear protection of the metal surface. Such a unique property is required in parts for numerous industrial applications, such as a cam or ring gear, anti-friction bearings or shafts, turbines and automotive components[i].

IMPORTANCE OF NANOINDENTATION MAPPING FOR SURFACE HARDENED METAL

Surface hardening can be categorized into two approaches in general: (1) buildup or addition of a new layer; (2) surface and subsurface modification without additional layer. Thin films, coatings and overlayers are deposited to provide an enhanced surface in the first case, where the homogeneity and interfacial bond strength play an important role. In the second case of direct surface modification such as carburizing, nitriding and peening, there is usually no clear boundary between the treated surface and the metal beneath, which makes it challenging to determine the thickness of the surface layer. Nanoindentation provides extremely localized precise hardness and Elastic Modulus measurements, making it an ideal tool for hardness mapping to determine the quality, homogeneity and thickness of the surface hardened layer.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester in nanoindentation mode is used to measure the hardness distribution of the hardened surface on the cross section of a surface hardened metal. We would like to showcase the capacity of Nanovea Mechanical Tester in performing nanoindentation mapping with high precision and reproducibility.

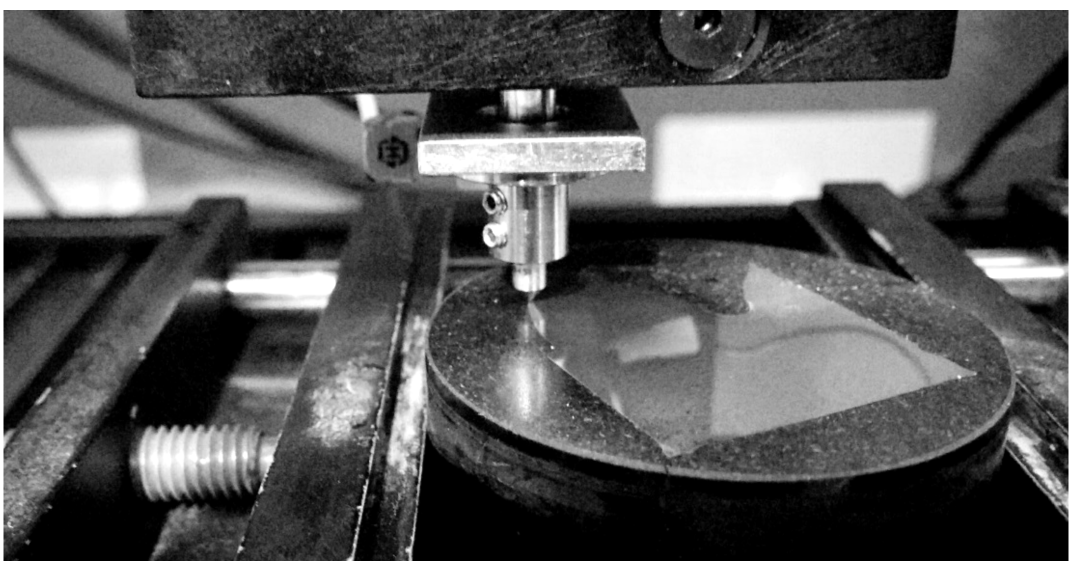


Figure 1: Nanoindentation tip on the cross section of the surface hardened metal.

TEST CONDITIONS

The hardness, *H* and Elastic Modulus, *E*, of 120 points along the distance from the treated metal surface on the cross section were measured spaced 50 µm apart for a total distance of 6 mm as shown in Figure 2. The test parameters are summarized in Table 1.

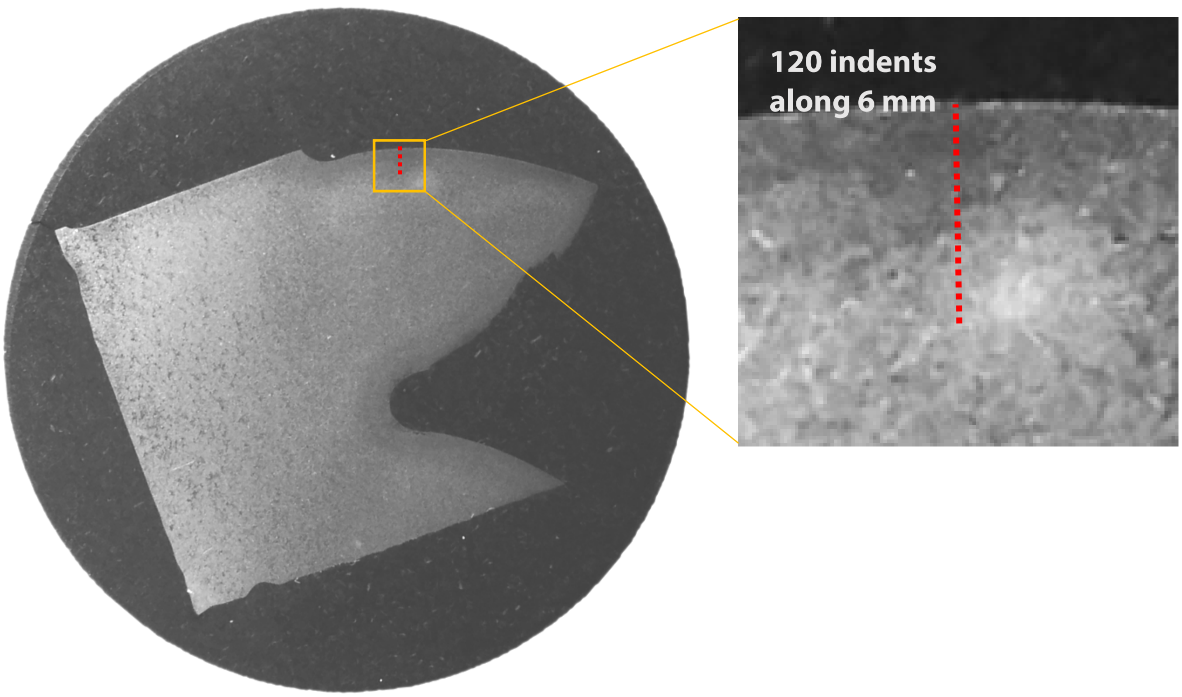


Figure 1: Diagram of test locations.

Maximum force (mN)	100
Loading rate (mN/min)	200
Unloading rate (mN/min)	200
Creep (s)	5
Indenter type	Berkovich diamond tip

Table 1: Test conditions of the hardness measurements.

RESULTS AND DISCUSSION

Surface hardening enhances the surface hardness while maintains the soft, tough nature of the interior metal. Hardness distribution measurement along the thickness of the treated surface ensures the quality and homogeneity of the surface treatment. The *H* and *E* values as a function of the distance from the treated metal surface are plotted in

Figure **3**. It shows that the metal sample possesses the highest *H* value of ~9 GPa at the surface. The *H* value progressively decreases to a value of ~5.5 GPa until a distance of 3 mm from the surface is reached, after which the metal has a consistent hardness of 5.5 ± 0.5 GPa. Despite the hardness enhancement on the surface, the Elastic Modulus maintains a homogeneous value of 225 ± 12 GPa for the surface and interior metal. Figure 4 shows the load-displacement curves measured at the surface and interior of the metal.

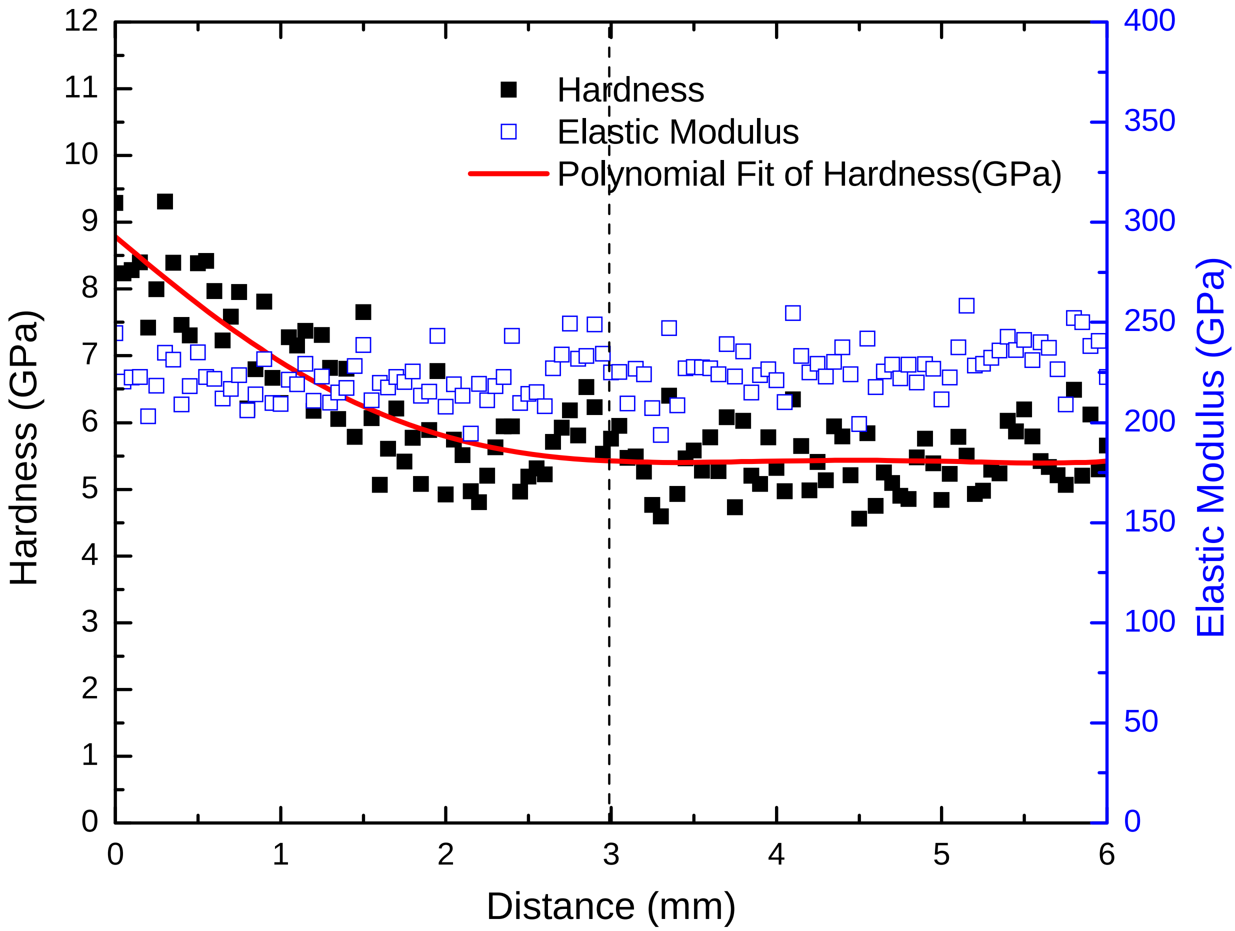


Figure 2: Hardness and Elastic Modulus vs. Distance from the treated surface on the cross section of the test sample.

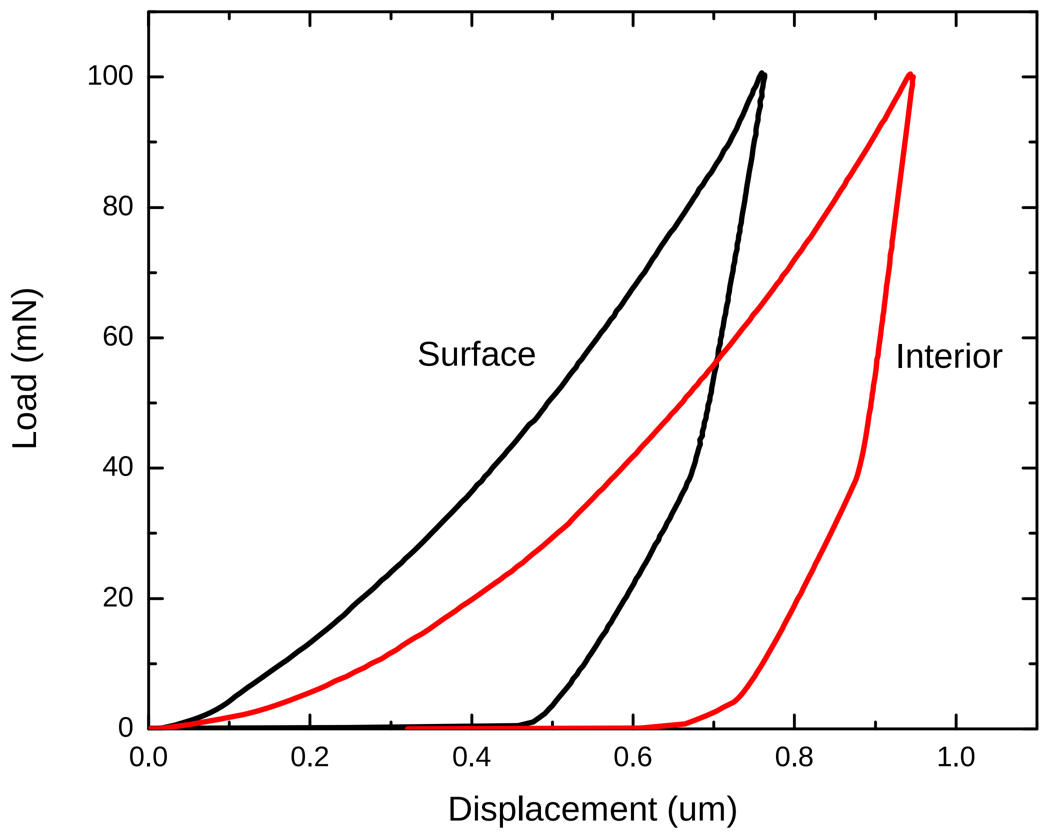


Figure 3: Load-displacement curves measured at the surface and interior of the metal.

CONCLUSION

In conclusion, we have shown that the Nanovea Mechanical Tester in Nanoindentation mode provides reliable measurement of hardness and Elastic Modulus distribution on a surface hardened metal sample. It enables users to assess the quality of the surface treatment and to determine the thickness of the surface layer or coating, which is critical in R&D and quality control for the coatings in a variety of industries, such as cutting tools, aerospace, automotive and many others.

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,

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

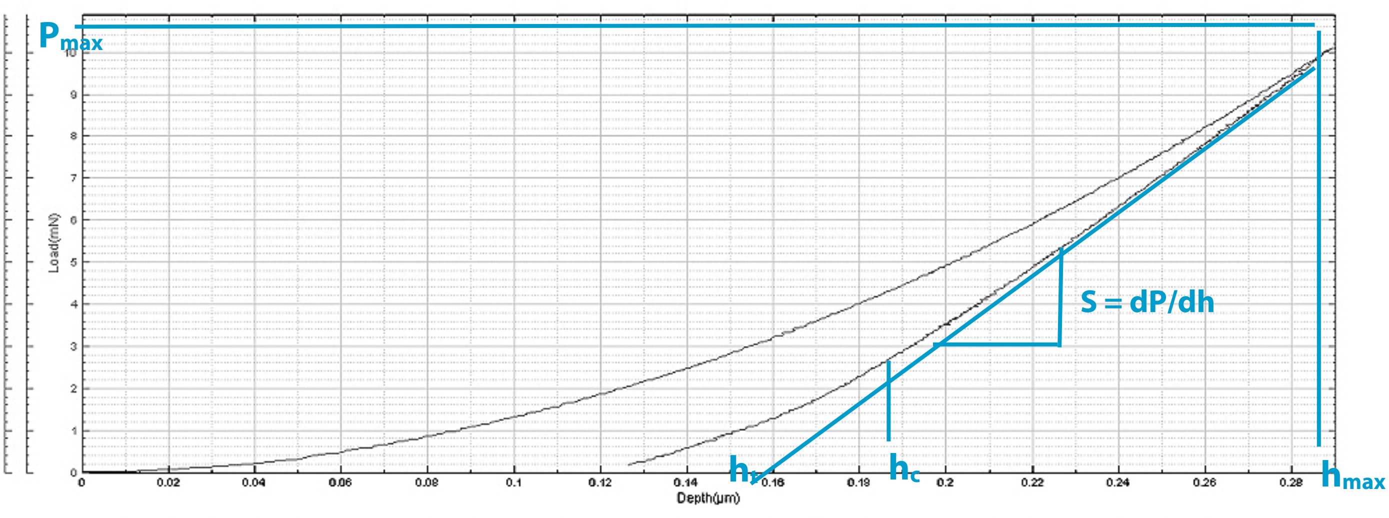
Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set 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 250µm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 µN
Load Resolution (Noise Floor)	: 1.5 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.



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 coefficient of the indenter and ν the Poisson coefficient 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 Ac 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 dε/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 might be a better choice.

Other tests possible includes the following:

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

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[i] Michael J. Schneider, Madhu S. Chatterjee, Introduction to Surface Hardening of Steels, ASM Handbook, Volume 4A, Steel Heat Treating Fundamentals and Processes