

# ISO 14577 Standardized Nanoindentation

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## Summary

Ten different materials are tested with the iNano in accordance with ISO 14577-1, including polymers, metals, glasses, and single crystals. All measured values of Young's modulus are within 10% of reference values. The same test method also automatically determines instrumented hardness and a converted Vickers Hardness Number (VHN).

## Introduction

Instrumented indentation is widely used to measure the Young's modulus and hardness of small volumes of material. Common applications include MEMS, semiconductor components, and protective coatings. ISO 14577 is an international standard that governs instrumented indentation. ISO 14577, Part 1, prescribes the procedure and data analysis, and draws heavily on the seminal work of Oliver and Pharr<sup>1</sup> [2]. Subsequent parts of the standard specify how the test instrument is verified and how reference blocks are manufactured and tested. The iNano nanoindenter includes a test method for testing in accordance with ISO 14577-1. To test in compliance with this standard, the user simply opens this test method (aptly named "ISO 14577 Test Method"), defines the test sites and initiates testing. (If desired, the user may customize the peak test force and loading time, but this is not necessary.) The test method automatically measures and reports Young's modulus, instrumented hardness, Vickers hardness, and the normalized work of indentation.

Relative to other manufacturers of instrumented indentation equipment, the iNano offers many advantages for standardized testing. The iNano is:

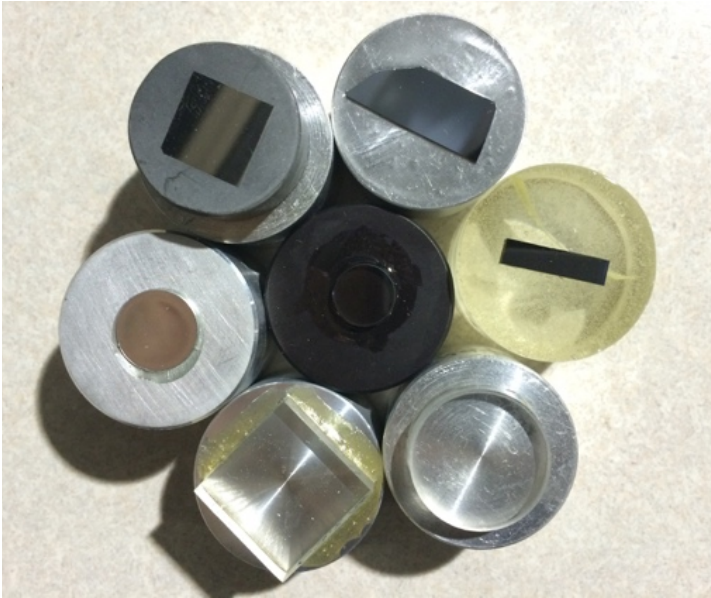
- Inexpensive to own and operate,
- User-friendly and fully automated, and
- Accurate and repeatable.

## Materials and Methods

Ten different materials were tested with the iNano in accordance with ISO 14577-1: sapphire (c-axis), nickel, silicon <111>, silicon <110>, 316L steel, BK7, fused silica, Borosilicate glass, and polycarbonate. Prior to testing, samples were polished to achieve a smooth surface (Fig 1.)

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<sup>1</sup> Dr. Warren Oliver continues his groundbreaking work in nanoindentation as President of Nanomechanics, Inc.



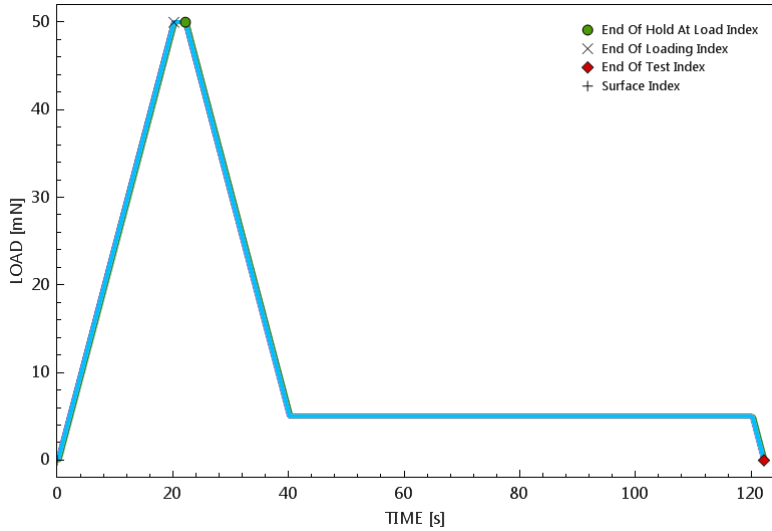
**Figure 1** – Samples mounted to standard aluminum or epoxy metallographic mounts. The diameter of the mount is 1.25" (32mm).

Ten indentations were performed on each sample using the default test protocol, depicted in Figure 2. Each indentation test had the following steps:

1. The indenter was brought into full contact with the surface with an approach velocity of 100 nm/sec.
2. The indenter was pressed into the surface of material using a constant loading rate of 2.5 mN/sec to a peak force of 50mN.
3. At the peak force, the force was held constant for a dwell period of 2 second.
4. The contact force was reduced to 10% of the peak force using an unloading rate of 2.5mN/sec.
5. The force on the indenter was held constant for 80 seconds while the displacement of the indenter was monitored. (Post-test, these data are used to determine thermal drift rate.)
6. The indenter was withdrawn completely and the sample was moved into position for the next test.

Results for each test were calculated as specified by ISO 14577-1<sup>2</sup> and then averaged across all ten tests.

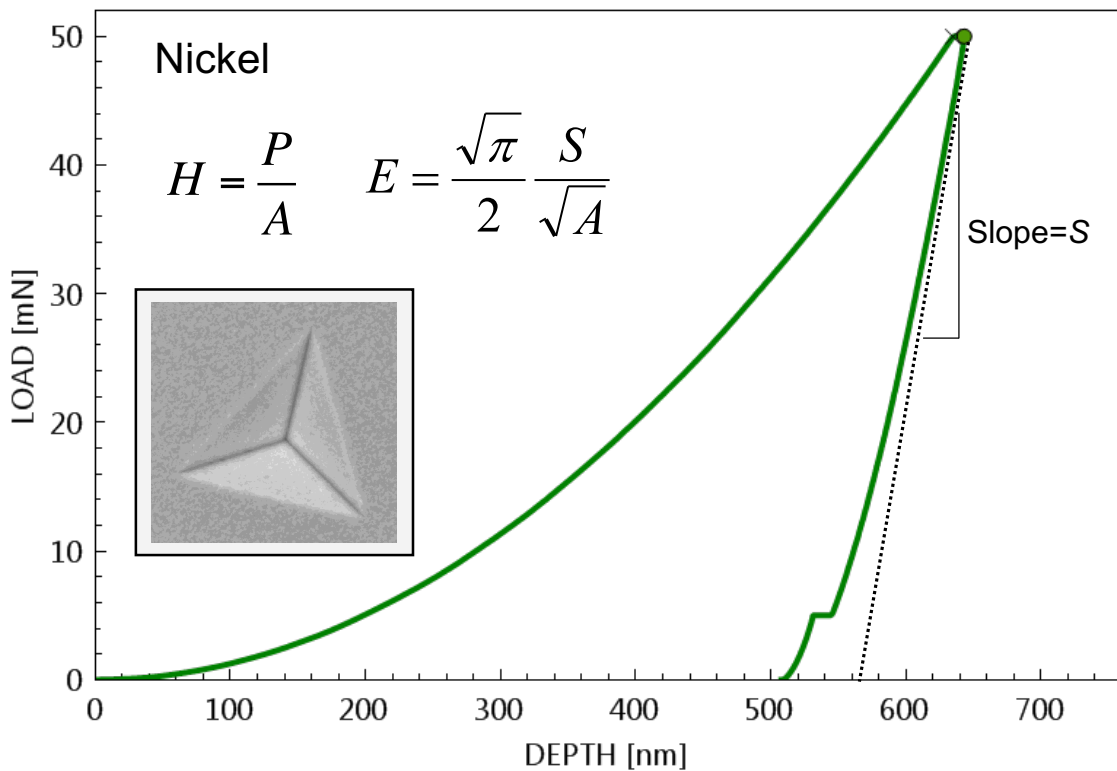
<sup>2</sup> The primary difference between microhardness testing and instrumented indentation is that the contact area is not directly measured, but rather calculated as a function of indentation depth. For this work, the "area function" was  $A=24.355h_c^2+165.6h_c$ , where the first coefficient was calculated by direct measurement of the diamond angles with a laser goniometer. Only the second term, which manifests apical rounding, was calculated by indentation of a reference material.



**Figure 2** – Load time history for the ISO 14577 standardized nanoindentation method. Loading, hold, unloading, followed by a second hold for thermal drift rate measurement.

## Results and Discussion

One load-depth curve for indentation into nickel is shown in Figure 3. Each test on each material produced this kind of curve. This is the fundamental data from which material properties are calculated according to ISO 14577-1, including Young's modulus ( $E_{IT}$ ), instrumented hardness ( $H_{IT}$ ), and Vickers Hardness (VHN).



**Figure 3** - Typical load-depth curve with Berkovich tip at peak load of 50mN of nickel sample (Insert is the residual mark)

Table 1 summarizes the results of all testing. For clarity, only the mean values are tabulated. However, the standard deviation for all reported results is less than 10% of the mean. For Young's modulus, we can compare the value measured by instrumented indentation, in accordance with ISO 14577-1, with reference values obtained by other means (tensile testing, ultrasonic, etc.). For all materials tested in this work, the ISO 14577 values are within 10% of the reference values for Young's modulus. Insofar as we can tell, the hardness values are quite reasonable. For example the reference value of vickers hardness of polycarbonate is 14 kgf/mm<sup>2</sup> while our VHN result shows 17 kgf/mm<sup>2</sup>; Reference value of Nickel sample gives 638 kgf/mm<sup>2</sup> and our VHN result shows 577 kgf/mm<sup>2</sup> (Validation of hardness is more challenging, because unlike modulus, the hardness of a material depends strongly on the microstructure which arises from processing and preparation.) Figure 4 is a graphical depiction of the comparison between Young's modulus measured according to ISO 14577-1 and reference values. The solid line is unity. The fact that all points lie near this line indicates that all values of Young's modulus measured here are very near their reference values.

**Table1** - Mean instrumented indentation results for 10 tests on each material to 50mN

Sample	Poisson's ratio	Max Depth (nm)	Reference Modulus (GPa)	ISO Elastic Modulus (GPa)	ISO Hardness (GPa)	Vickers Hardness (kgf/mm <sup>2</sup> )
<b>Polycarbonate</b>	0.37	3961.3	3 <sup>3</sup>	3.0	0.19	17.94
<b>Borosilicate</b>	0.20	736.5	64 <sup>4</sup>	63.2	7.71	689.57
<b>Fused silica</b>	0.17	680.2	73 <sup>5</sup>	71.8	9.66	912.98
<b>BK7</b>	0.21	674.8	82 <sup>6</sup>	85.2	8.14	769.54
<b>Platinum</b>	0.39	1504.9	168 <sup>7</sup>	141.1	0.91	85.57
<b>Silicon&lt;110&gt;</b>	0.28	491.3	169 <sup>8</sup>	192.3	11.62	1098.50
<b>316L Steel</b>	0.30	744.8	193 <sup>9</sup>	196.0	4.55	429.76
<b>Silicon &lt;111&gt;</b>	0.17	511.2	186 <sup>10</sup>	205.8	12.06	1140.37
<b>Nickel</b>	0.31	638.5	200 <sup>11</sup>	213.9	6.36	600.75
<b>Sapphire</b>	0.30	344.3	435 <sup>12</sup>	466.2	28.00	2646.78

Note: Vickers hardness in this table was converted from indentation hardness

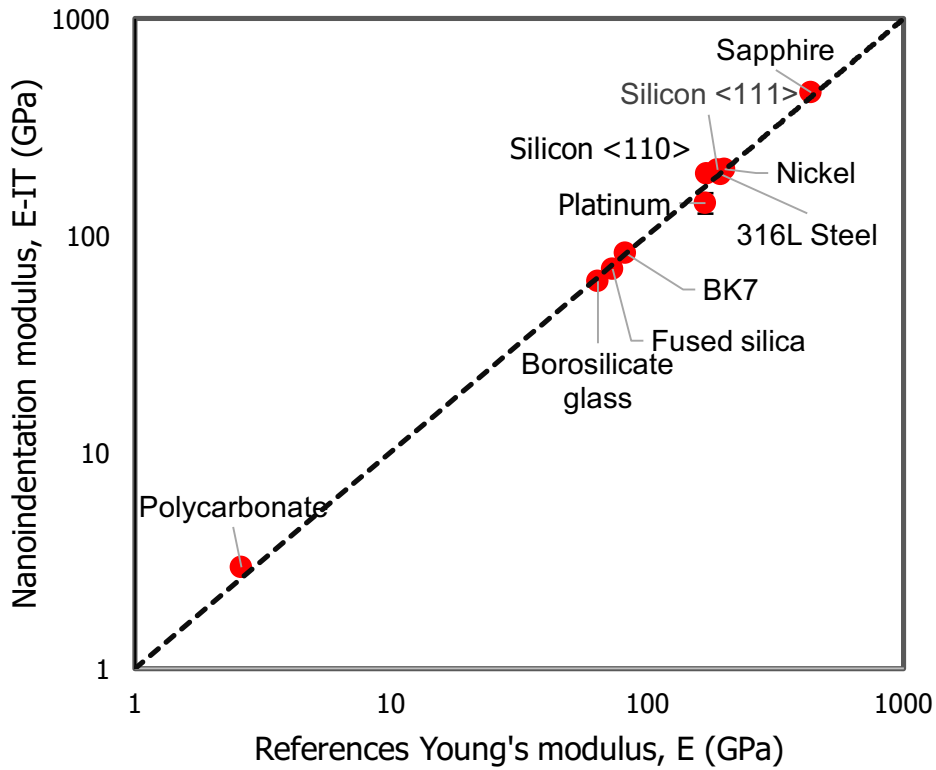


Fig 4. Average Young's modulus measured in this work according to ISO 14577-1, as compared with reference values for the same materials.

## Conclusions

Instrumented indentation tests were performed on a variety of materials using Nanomechanics, Inc. iNano indentation system. The use of iNano testing system with the compliance of ISO 14577 standard allows user to measure material mechanical properties in a fast and less expensive way, at the same time getting accurate and repeatable data. Our indentation tests results show a good agreement with the reference modulus.

## References

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