Standardized Nanoindentation: ISO 14577

NANOMECHANICS, INC

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Abstract

Ten different materials are tested with the **iNano** in accordance with ISO 14577-1, including polymers, metals, glasses, and single crystals. Most measured values of Young's modulus are within 5% 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 [1]. ISO 14577, Part 1, prescribes the procedure and data analysis, and draws heavily on the seminal work of Oliver and Pharr[1] [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 instrumented indenters, 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.

Experimental method

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

Figure 1. Samples as prepared for testing.

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

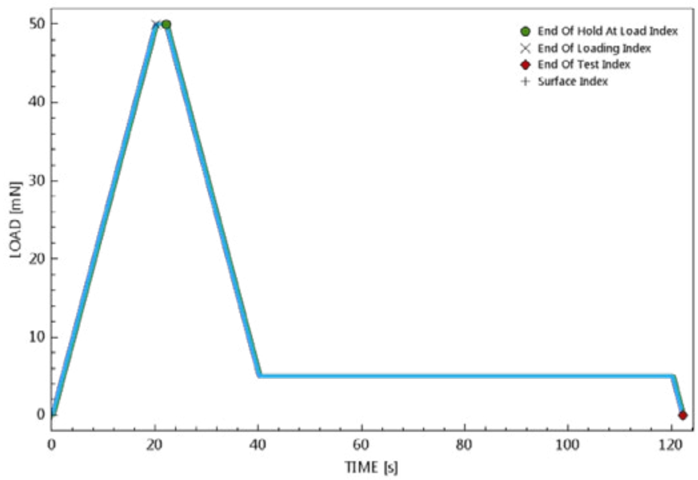

Figure 2. Load vs. time for each indentation test. .

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[2] and then averaged across all ten tests.

Results & 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 (EIT), instrumented hardness (HIT), and Vickers Hardness (VHN).

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 (tensile testing, sonic testing, or analysis of crystalline elastic constants). For most samples, the ISO 14577 values are within 5% of the reference values for Young's modulus.

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 $A = 24.355^{2}_{c} + 165.6h_{c}$*, 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.*

Sample	Poisson's ratio	Depth	Reference Young's modulus	E_{IT}	H_{IT}	Vickers* hardness	Temp
		nm	GPa	GPa	GPa	kgf/mm²	°C
Polycarbonate	0.37	3961	2.6 [5]	2.93	0.18	17.0	26.2
Borosilicate glass	0.02	736	64 [6]	61.5	7.30	690	26.3
Fused silica	0.17	680	73 [7]	69.9	9.15	865	26.4
BK7	0.21	674	82 [8]	82.9	7.71	729	26.1
Platinum <100>	0.39	1504	168 [9]	141.1	0.94	89	25.8
Silicon<110>	0.28	491	170 [10]	192.3	12.90	1219	26.1
Silicon <111>	0.27	511	186 [11]	191.2	11.44	1081	25.8
316L Steel	0.30	744	193 [12]	190.9	4.30	406	26.4
Nickel	0.31	638	200 [13]	202.5	6.11	577	26.1
Sapphire, c-axis	0.30	344	435 [14]	456.7	26.63	2517	26.1

*Vickers hardness values in this table are calculated according to ISO 14577-1, not as prescribed by a microhardness standard.

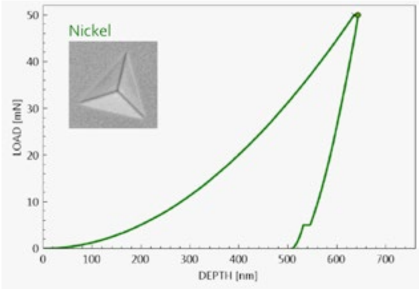

Figure 3. Typical load-depth curve with Berkovich tip at peak load of 50mN of nickel sample (Insert is a residual mark)

Figure 3 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. For the polycarbonate, the discrepancy between measured and reference values is likely due to the higher strain rate of the indentation test. For platinum and silicon, the difference is likely due to the anisotropy of the crystal in combination with the bi-axial nature of the indentation test.

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. However, hardness values do seem reasonable. By the method of ISO 14577-1, we measured the Vickers hardness of polycarbonate to be 17kgf/mm², which compares well with a reference value of 14 kgf/mm² [3]. For nickel, we measured a Vickers hardness of 577 kgf/mm², which compares well with a reference value of 638 kgf/mm² [4].

Conclusion

Instrumented indentation, in conformance with ISO 14577-1 is a valid way to measure Young's modulus and hardness of small volumes of material, whether glass, metal, or polymer. The properties measured in this work compare well with values measured on similar materials by other test methods. The **iNano** system simplifies standardized testing by providing the user with a turnkey ISO 14577 test method.

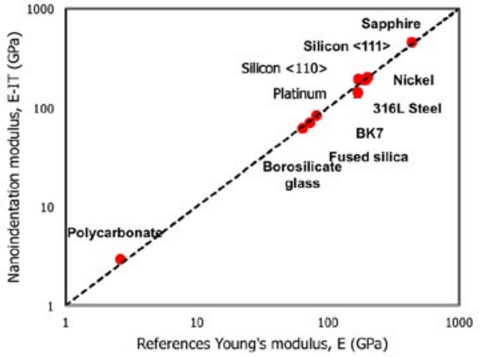

Figure 4. Average EIT measured in this work according to ISO 14577-1, as compared with reference values of Young's modulus for the same materials.

References

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