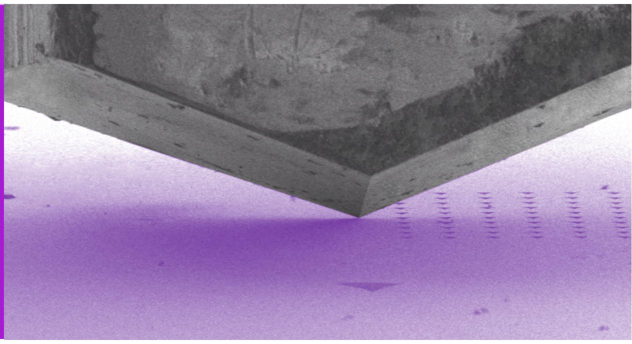
Application Note

KLA INSTRUMENTS

ISO 14577 Standardized Nanoindentation



Summary

Ten different materials are tested with the KLA 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 the 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 [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 (“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 KLA iNano offers many advantages for standardized testing. The iNano is:

- Inexpensive to own and operate,
- User-friendly and fully automated,
- 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, platinum and polycarbonate. Prior to testing, samples were polished to a smooth surface, as shown in Figure 1.

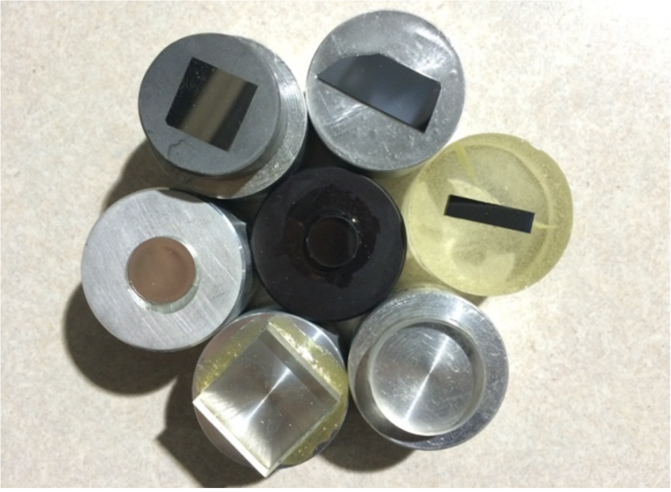


Figure 1. Samples mounted to standard aluminum or epoxy metallographic mounts. The diameter of the mount is 1.25” (32mm).

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

1. The indenter was brought into full contact with the surface with an approach velocity of 100nm/s.
2. The indenter was pressed into the surface of the material using a constant loading rate of 2.5mN/s to a peak force of 50mN.
3. At the peak force, the force was held constant for a dwell period of 2s.
4. The contact force was reduced to 10% of the peak force using an unloading rate of 2.5mN/s.
5. The force on the indenter was held constant for 80s while the displacement of the indenter was monitored (post-test, the data is used to determine the 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[1] and then averaged across all ten tests.

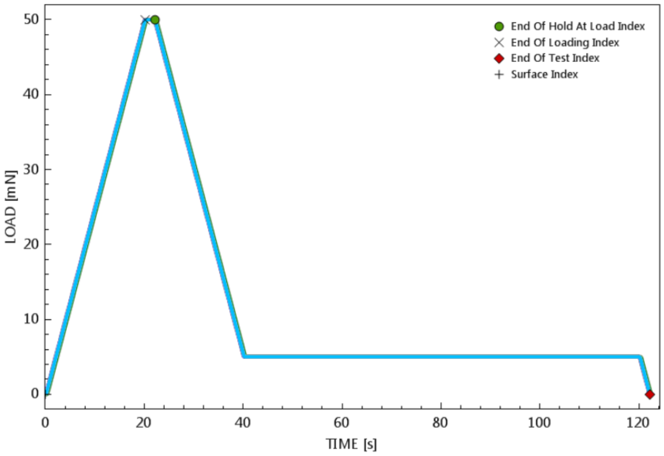


Figure 2. Load time history for the ISO 14577 standardized nanoindentation method. Loading, hold, unloading, followed by a second hold for thermal drift rate measurement.

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

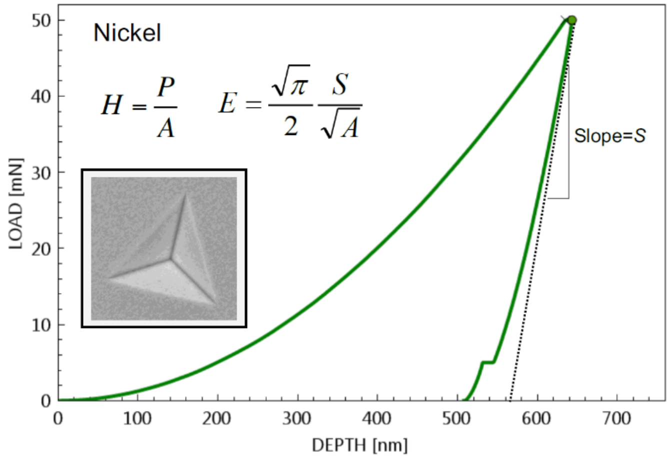


Figure 3. Typical load-depth curve with Berkovich tip at peak load of 50mN of nickel sample (Insert is the residual mark)

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 14kgf/mm^2 while our VHN result shows 17kgf/ mm^2; the reference value of the nickel sample gives 638kgf/ mm^2 and our VHN result shows 577kgf/ mm^2. Note that the validation of hardness is more challenging, because unlike Young's modulus, the hardness of a material depends strongly on the microstructure that results 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, with the dashed line representing unity. The fact that all points lie near this line indicates that all values of Young's modulus measured here are very close to their reference values.

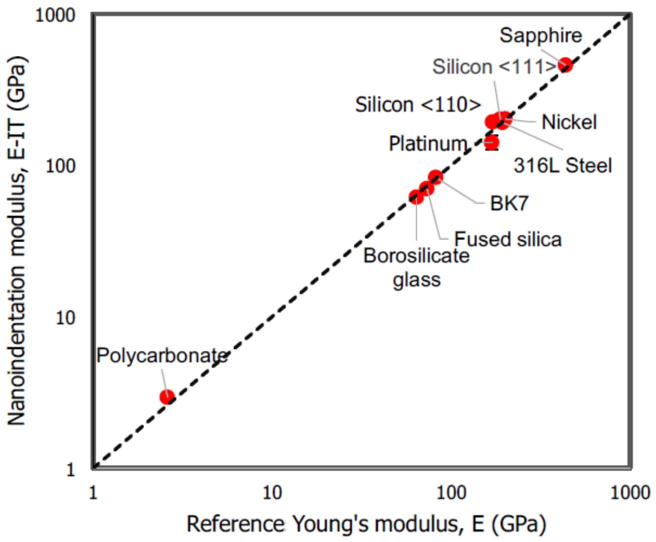


Figure 4. Average Young's modulus measured in this work according to ISO 14577-1, as compared with reference values for the same materials.

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[1] 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.

Table 1. Mean instrumented indentation results for ten tests on each material with maximum load of 50mN.

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

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

Conclusions

Instrumented indentation tests were performed on a variety of materials using a KLA iNano indentation system. The use of the 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 test results show good agreement with reference moduli.

References

1. "Metallic Materials – Instrumented indentation test for hardness and materials parameters," ISO 14577, 2002.
2. Oliver, W.C. and Pharr, G.M., "An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments," Journal of Materials Research, vol. 7(6):1564-1583 (1992).
3. http://www.engineeringtoolbox.com/young-modulus-d_417.html.
4. http://www.azom.com/article.aspx?ArticleID=4765.
5. http://accuratus.com/fused.html.
6. http://www.crystran.co.uk/optical-materials/optical-glass-n-bk7-and-others.
7. https://en.wikipedia.org/wiki/Platinum.
8. http://silicon.mhopeng.ml1.net/Silicon/.
9. http://asm.matweb.com/search/SpecificMaterial.asp?bassnum=MQ316Q.
10. https://www.el-cat.com/silicon-properties.htm.
11. https://en.wikipedia.org/wiki/Nickel.
12. http://www.gavish.com/sapphire-data-table.htm.

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