Keysight Technologies
Quantitative Mechanical Measurements at the Nano-Scale Using the DCM II
Application Note

Introduction

Feature miniaturization, especially in the electronics industry, demands knowledge of mechanical properties on the scale of nanometers. Instrumented indentation facilitates such testing, because the area of the contact impression does not have to be measured visually, but can be inferred solely from the relationship between applied force and consequential penetration of the indenter into the testing surface [1]. Instrumented indentation has been used since the 1980's to make measurements at the sub-micron scale, but recent developments allow quantitative determination of mechanical properties using indents of just a few nanometers deep. This article addresses special considerations for such testing, and reports results for seven different materials tested with the DCM II.

The DCM II, shown in Figure 1, is an optional high-resolution actuating transducer for the Keysight Technologies, Inc. G200 Nano Indenter. The DCM II may be used in addition to or instead of the standard indentation head. If both heads are included on a single system, transition from one head to the other is entirely software controlled; the user doesn't have to make any adjustments to hardware, controllers, or calibrations. The range and resolution in displacement (travel) are 70µm and 0.0002nm,

respectively. The range and resolution in force are 30mN and 3nN, respectively. Because the DCM II has a resonant frequency of about 120Hz, measurements of force and displacement are insensitive to environmental noise which occurs at lower frequencies. The DCM II can be used in combination with a variety of indenter tips including Berkovich, cube-corner, and sphero-conical. Changing from one tip to another takes just a few minutes.

Figure 1. The Keysight DCM II.

Experimental Method

Prior to testing, the shape of the diamond indenter was "calibrated" by performing 55 indents on a reference material, fused silica. Then seven different materials, including the fused silica, were tested using the force-time algorithm shown in Figure 2. All materials were tested to the same peak force of 50µN. The materials tested were polycarbonate, Pyrex, fused silica, single-crystal aluminum, silicon (111), nickel, and sapphire. Because these materials are of varying hardness, the indentation depths resulting from the applied force of 50µN varied. The deepest indents of about 100nm were achieved on the polycarbonate, while the shallowest indents of less than 7nm were achieved on the sapphire. Fifteen indents were performed on each sample using the DCM II fitted with a diamond Berkovich indenter. Force and displace-
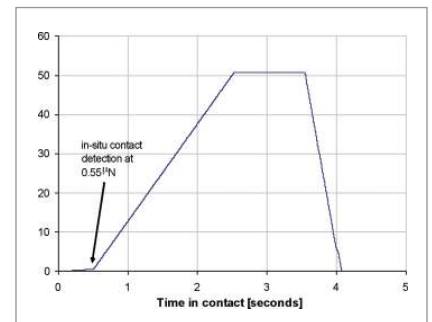

Figure 2. Force-time algorithm for indentation tests.

ment measurements were acquired at a rate of 12.5kHz, averaged in a buffer and reported at a rate of 100Hz. Data were analyzed according to the method prescribed by an international standard for instrumented indentation testing, ISO 14577 [2], which in turn draws heavily upon the landmark article by Warren Oliver and George Pharr [1]. Average modulus and standard deviation were computed using all 15 tests. If a particular test yielded a measure of modulus that was different from the average value by more than two standard deviations, the result for that test was discarded, and the remaining results were averaged again.

The main focus of this work was the determination of quantitative mechanical properties at the scale of nanometers. However, when used in combination with the NanoVision option, the DCM II becomes a profilometer, capable of generating topological images with excellent dimensional accuracy. In this work, a grid for verifying the dimensional accuracy of atomic-force microscopes was scanned. The grid has periodic steps; the steps have a height of 19nm and a period of 3 microns. A square area of 6.5μm on a side was scanned using a scanning force of 1.0μN; the resulting scan was used to select a site for an indentation test. Following this test, the same area was scanned again to reveal the residual indentation impression.

Results and Discussion

Calibration

The process for determining the precise shape of the indenter is automated within the NanoSuite software. The following discussion should not intimidate new users; it is only intended to explain what is done and why.

The data used to "calibrate" the shape of the tip are shown in Figure 3 in the form of stiffness squared divided by applied force (S^2/P) as a function of displacement into the test surface, h. We begin by looking at the data in this way, because S^2/P is directly proportional to reduced modulus squared divided by hardness (E_r^2/H), but is independent of contact area (A):

$$\frac{E_r^2}{H} = \frac{\left(\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}}\right)^2}{\frac{P}{A}} = C\frac{S^2}{P}$$

If the machine is working well, then we expect S^2/P to be constant with increasing penetration, having a value of 700GPa ± 50GPa. (As the displacement into the surface decreases to zero, the contact becomes increasingly Hertzian, and we expect S^2/P to increase exponentially, because for a Hertzian contact, the parameter S^2/P goes as $h^{-1/2}$.) Since the trace of S^2/P meets our expectations, we proceed to use this data to determine the precise shape of the diamond indenter. The

relationship between the distance from the apex of the diamond, **d**, and the cross-sectional area at that distance, **A**, is called the "area function". For a perfect Berkovich indenter, the area function is

$$A = 24.56d^2.$$

However, when making nanometer-scale indents, imperfections at the apex of the diamond demand that the area function be determined more precisely. We do this by calculating contact depth and contact area, assuming a value for the reduced modulus of the material. Therefore, each indent on fused silica yields an ordered pair (h_c, A) with contact depth (hc) calculated as

$$h_c = h - \varepsilon\frac{P}{S}$$

and A calculated as

$$A = \frac{\pi}{4}\left(\frac{S}{E_r}\right)^2$$

Figure 4 shows these (h_c, A) data. To determine the refined area function, we curve fit this data to the functional form suggested by Oliver and Pharr [1]:

$$A = m_0 h_c^2 + m_1 h_c + m_2 h_c^{1/2} + m_3 h_c^{1/4} + m_4 h_c^{1/8} + \ldots$$

If we only use the first two terms of this expression to fit the data, the best-fit coefficient of the second term can be used to determine the radius of the tip. By this analysis, the radius of the diamond tip was
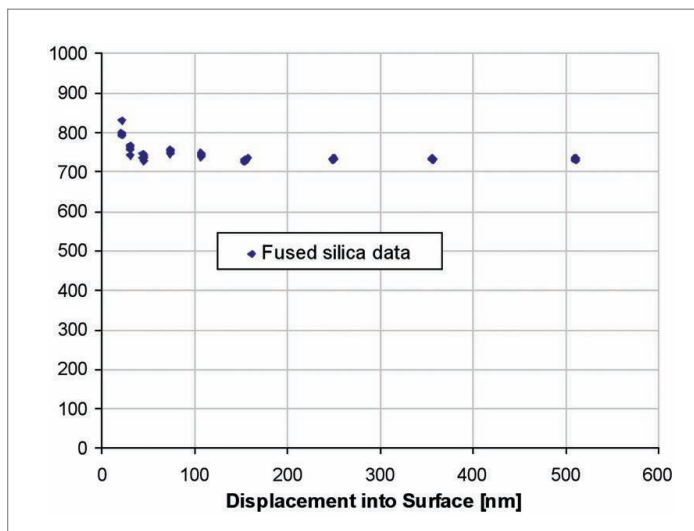

Figure 3. Stiffness squared divided by force (S²/P) vs. displacement (h). For fully plastic contacts, acceptable range for fused silica is 700GPa [6] 50GPa.
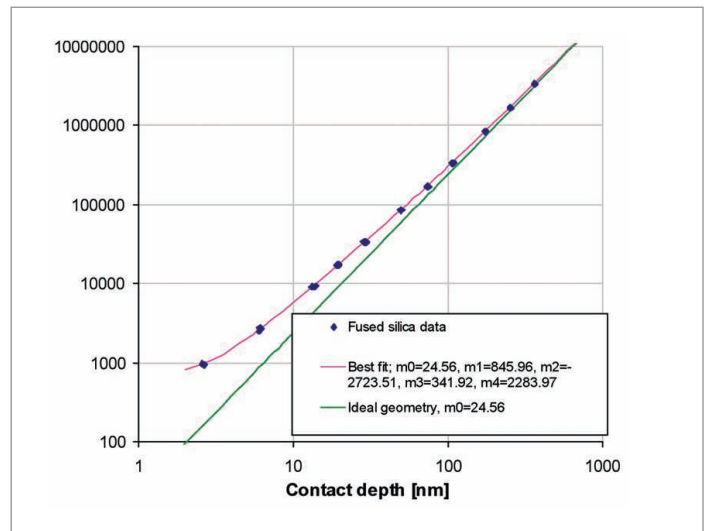
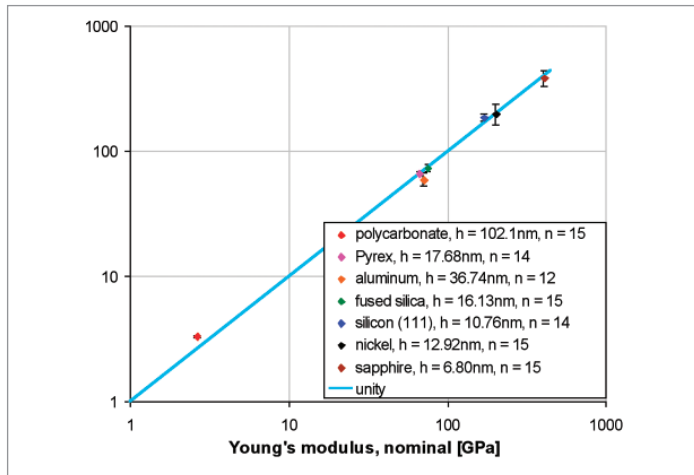Figure 4. Fused silica data (h_c, A) together with 5-term area function.

Figure 5. Young's modulus measured using DCM II at 50 µN. Error bars represent 1σ on n/15 measurements. Solid line indicates unity; i.e. perfect agreement between measured and nominal.
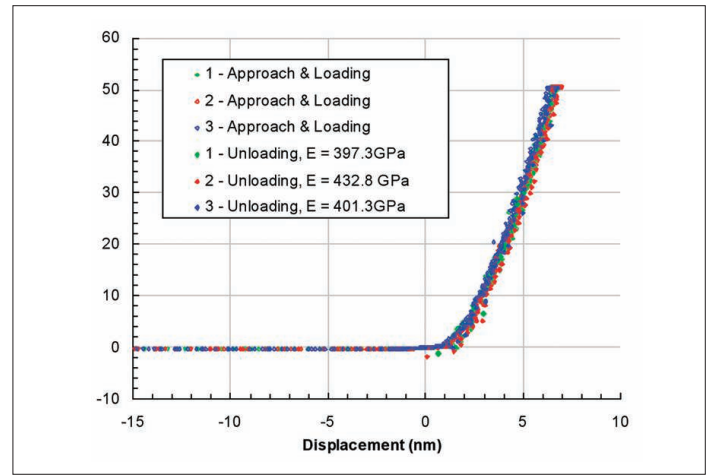

Figure 6. Three consecutive indents on sapphire ($E = 400$ GPa). Loading and unloading curves coincide, indicating that the indents are completely elastic.

determined to be 52 nm. However, two terms rarely provide a fit that is sufficiently good for making nanometer-scale measurements. The fit shown in Figure 4 is a five-term fit. For hc < 100 nm, the maximum difference between this fit and the data is about 4%.

Young's modulus

The results for Young's modulus are summarized in Table 1. The fourth column of this table gives the number of tests (out of 15) that were used in the calculation of results. On all but three materials, all 15 tests were used. In Figure 5, the measured values for Young's modulus are plotted against nominal values, with the ideal plotted as a solid line. For fused silica and Pyrex, the nominal values are what we measured sonically in-house [3]. For sapphire, aluminum, silicon (111), and sapphire, the nominal values for Young's modulus are theoretical values calculated from crystalline elastic constants for the direction normal to the testing surface [4-7]. For polycarbonate, the nominal value is that reported on the website "engineeringtoolbox.com".

Overall, the agreement between nominal and measured values is excellent, especially given the scale of testing. For the Pyrex, fused silica, nickel, and sapphire, measured values for Young's modulus were within 1 standard deviation of the nominal value. Two sets of 15 indents were performed on the fused silica, because it is standard practice to test this reference material before and after the materials of interest. The average maximum indentation depths achieved for both sets of indents on fused silica agreed to 0.005 nm!

For polycarbonate, the measured value of 3.35±0.08 GPa is high relative to the nominal value of 2.6 GPa. This is probably due to the test method that was employed. As a polymer, polycarbonate manifests some viscoelasticity. Thus, obtaining the contact stiffness from the slope of the unloading curve likely results in a stiffness that is too high, because the indenter continues to move into the material even as the force is reduced. It would be better to measure the modulus of this material with our continuous stiffness measurement option (CSM). For the single-crystal aluminum, the measured value of 59.8±6.9 GPa is low relative to the nominal value of 70.0 GPa. However, we have no reason to question the accuracy of the measured value. One possible explanation for the discrepancy between the measured and nominal values may be a surface oxide layer. For the (111) silicon, the measured value of 186.3±11.6 GPa was slightly high, relative to the nominal value of 168.9 GPa. We chose (111) silicon for this testing, because it has the same Young's modulus perpen-

Material	Poisson's ratio	hmax	n valid tests	E_{IT}	$\sigma(E_{IT})$	Nominal E	Ref. for nominal E
	–	nm	–	GPa	GPa	GPa	
fused silica	0.190	16.13	15	73.10	3.38	74.6	3
nickel	0.310	12.92	15	199.70	37.53	200.0	4
silicon (111)	0.262	10.76	14	186.30	11.57	168.9	5
polycarbonate	0.370	36.74	15	3.35	0.08	2.6	6
Pyrex	0.209	102.10	14	66.60	2.36	65.8	3
sapphire	0.234	17.68	15	386.10	52.60	403.0	6
fused silica	0.190	6.80	15	73.80	4.54	74.6	3
aluminum	0.330	16.13	12	59.80	6.90	70.0	7

Table 1. Summary of properties measured at 50 µN using the DCM II.

dicular to and parallel to the plane of the wafer; however, the material is not isotropic. Other directions have Young's moduli varying from 130 GPa to 187 GPa [5]. Since indentation is not a uni-directional test, this anisotropy likely accounts for the slight discrepancy between the measured and nominal values.

Figure 6 shows the force-displacement curves for three consecutive indents on sapphire, the material which yielded the shallowest indents. The Young's moduli derived from these curves are included in the legend.

Topography

When the DCM II is used with the profilometry option, NanoVision, the resulting quantitative images can be used to place indents with an accuracy of 20 nm. Figure 7 shows an image of an AFM grid with 19 nm steps, repeating at a period of 3 microns. Figure 7 shows line profiles in the x- and y-directions through the center of the indent shown in Figure 7. With these profiles, the accurate measurements of step height and spacing are easily verified. The residual impression has a depth of 53 nm at its center, and reveals no evidence of pile-up.
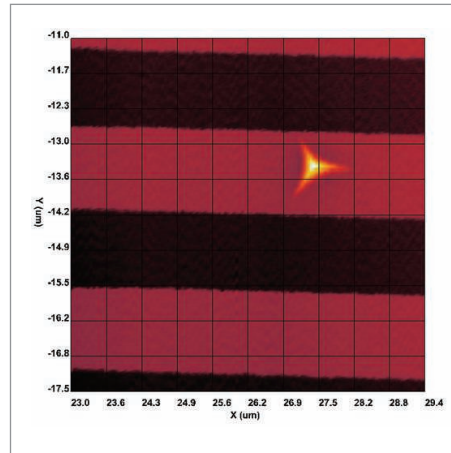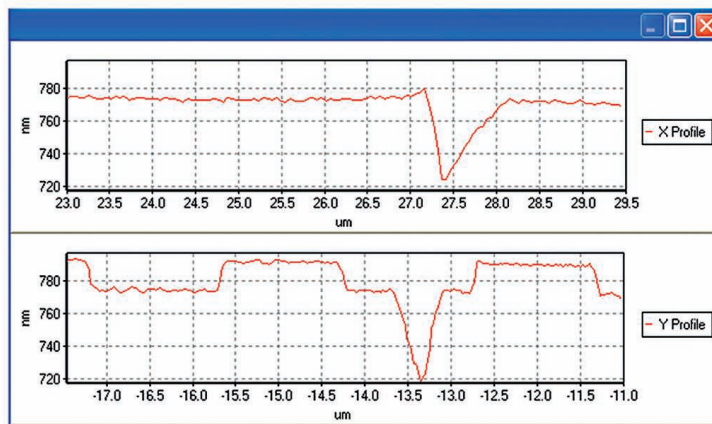

Figure 7. NanoVision scan of a square area (6.5μm on a side) on an AFM-verification grid. Grid steps are nominally 19nm in height with a periodicity of 3 microns. Image size is 100x250. Image generation time is 6.7 minutes.

Conclusions

As an option for the Keysight G200 Nano Indenter, the DCM II was used to measure Young's modulus on seven different materials at the scale of nanometers. For four out of the seven materials, measured values were within one standard deviation of nominal values. For the remaining three materials, discrepancies between measured values and nominal values were attributed to viscoelasticity (polycarbonate), surface oxide (aluminum), and anisotropy (silicon (111).

Figure 8. Line profiles through the center of the residual impression. Step height of 19nm and periodicity of 3μm are easily verified. Residual depth of indentation is about 53nm.

References

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