



Performance and Control of the Agilent Nano Indenter DCM

Application Note

Introduction

With new materials and material applications come new test challenges. As these new challenges emerge in materials testing, novel test protocols are required for evaluating the performance and predicting the failure of these new materials. Accuracy, precision, and control are all key elements of a test instrument that enable researchers to develop unique test protocols and evaluate the performance of their materials. In this article the three key elements of the Agilent Nano Indenter G200 are evaluated.

The accuracy and precision of the nanoindenter system were evaluated using a calibrated reference material. Given that the system is calibrated correctly, which can be determined by assessing the accuracy, two primary sources of error affect the precision of the instrument: displacement noise and thermal drift. Both of these sources of error were evaluated.

Control of the Nano Indenter system is conducted through the Agilent NanoSuite software. This software allows unparalleled control and test protocol development. Three novel tests were developed to control the nanoindenter and evaluate the response of the transducer to conditions other than load controlled experiments; inherently, almost all of the commercially available nanoindenters are load control instruments. However, NanoSuite allows users to control test protocols based on any hardware signal, calculated formula, or software signal. The three test conditions used for the evaluation

of control were holding the load on sample constant, loading at a constant displacement rate, and performing a constant stress test. Each of these conditions requires real time calculations and high level control of the instrument.

Samples

Corning 7980 (fused silica) was used in evaluation of the Agilent Nano Indenter G200 for load and displacement performance. Fused silica is an amorphous material that is highly reproducible and chemically inert to most elements. Its use as a reference material in verification of nanoindenter performance dates back to the first commercially available nanoindenter system.

The mechanical properties of the fused silica sample used in this article were verified using sonic techniques and a Certificate of Calibration was provided with this sample. Sonic measurements of the reference material provided nominal values of 74.008 ± 0.292 GPa for the average Young's modulus and 0.191 ± 0.002 for the Poisson's ratio.

For testing control of the instrument in conditions other than load control poly(methyl methacrylate) (PMMA), single crystal aluminum, and epoxy were used. These samples were used because they represented samples that would creep at maximum load causing stress relaxation and as a result the load on sample would decrease over a long hold segment.



Test Equipment

All of the tests conducted in this article were performed using an Agilent Nano Indenter G200 equipped with the ultra-low load, ultra-high resolution Dynamic Contact Module (DCM). The DCM is a Nanomechanical Actuating Transducer (NMAT) that is used to apply loads and measure displacements during indentation tests. Its novel design includes decoupled force application and displacement measurement for unparalleled control and flexibility during testing. A cross-section of the DCM-NMAT is shown in Figure 1.

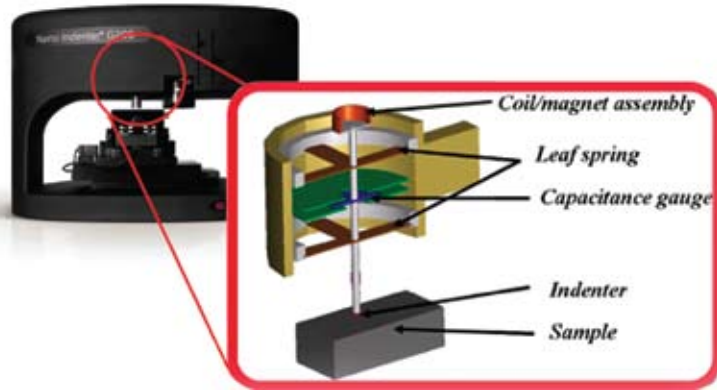


Figure 1. The Agilent Nano Indenter G200 and a cross-sectional diagram of the DCM-NMAT.

Each of the design elements shown in Figure 1 contribute to the repeatable and reliable measurements performed by the Nano Indenter systems.

Control of force is performed using electromagnetic actuation providing three primary advantages:

- 1) High accuracy in force control due to the simple linear relationship between current passed through the coil and the force that is produced.
- 2) Force application over a large displacement range due to the stability of the permanent magnetic field over large distances.
- 3) Flexible force application in both actuating directions because electromagnetic actuation works equally well in both the push and pull directions.

Two leaf springs are used to secure the indentation column for stability and maximum lateral stiffness. The ISO 14577 standard specifies that the surface of the sample should be within one degree of orthogonal alignment with the indenter; in actuality, this is not just a recommendation, it is a must for repeatable and reliable data. As shown in “Indentation Rules of Thumb” errors in orthogonal alignment can lead to larger errors than expected due to finite lateral stiffness in transducer design [1]. High lateral stiffness is a critical design element of the NMAT and is accomplished by the doubly secured indentation shaft that prevents lateral deflection when indenting samples with surface roughness or misaligned samples.

The final critical design component of the NMAT is the capacitance gauge which is used for sensing displacement. All commercially available nanoindenter platforms use capacitance gauges for measuring displacement. However, the capacitance gauge used in the Agilent transducers are specifically designed to allow ultra-high resolution with unparalleled displacement ranges providing users with maximum testing flexibility.

Now, with the release of the Agilent DCM II, flexibility is even greater. Table 1 lists the specifications of the new DCM II Nano Mechanical Actuating Transducer.

Agilent NanoSuite Explorer Software

Ultimate control and flexibility of the Nano Indenter systems is accomplished through the Agilent NanoSuite Explorer software. This software allows control of the system through any hardware or calculated data channel. In addition, formulas can be calculated real-time to use for decision making and control

of the test protocol. In the tests that follow, this control is put into action by creating test protocols that hold the load on sample constant, perform a constant displacement rate test, and hold the stress in a creeping material constant.

The load on sample during a hold segment can change significantly if the material is subject to creep. During standard nanoindentation, the force is held constant for a dwell time prior to unloading the force and measuring the stiffness of the sample. The force that is commonly held constant is the raw force produced by the transducer. This force differs from the load on sample because it does not take into account any force that is used to deflect the springs in the transducer. Therefore, when a material creeps during this dwell time, the load on sample decreases because some of the force is transferred to the springs as they are further deflected.

It is advantageous to control the load on sample during the dwell time when testing materials that creep. The NanoSuite software provides

| | |
|--------------------------------------|------------------|
| Range of indenter travel | 70 μm |
| Displacement resolution | 0.0002 nm |
| Typical leaf spring stiffness | 100 N/m |
| Lateral stiffness | 80,000 N/m |
| Maximum Load | 30 mN |
| Load resolution | 3 nN |
| Thermal drift rate* | 0.05 nm/s |

Table 1. DCM II Specifications.

* Thermal drift rates are dependent on lab environments.

this capability. A common technique for controlling the load on sample is by correcting the raw force for any changes in the load on sample; this is accomplished through a formula that is continuously updated. Equation 1 shows the formula that corrects the load signal for any difference between the load on sample and the target load.

$$\text{Set Point} = \text{Raw Load} - (\text{Load on Sample} - \text{Target Load}) \quad (1)$$

Here, the set point is the value of the commanded force signal to be produced by the transducer, raw load is the current force signal out of the transducer, load on sample is the force applied to the surface of the sample after corrections are made for spring deflection, and target load is the load to be held constant on the sample. Equation 1 corrects the raw force signal for any error detected in the load on sample. The logistics for switching the force control to hold the load specified by Equation 1 is shown in Figure 2.

As opposed to controlling a hardware signal based on a formula that is continuously updated, the NanoSuite software also allows control through a software channel using PID controls. In performing constant displacement rate tests, the electromagnetic force is governed such that the change in displacement occurs at a set speed. The NanoSuite software contains tunable PID settings to control the nanoindenter using data signals other than raw load. Figure 3 details the loading segment of a constant displacement rate test with the PID control settings. This control is unmatched in the industry.

The last control algorithm included in this article is an extension of the use of PID controls in the NanoSuite software to conduct a constant stress test. Instead of using PID controls during a loading segment, this control uses PID controls to hold stress in a material at a constant value during the dwell period of a test. Usually a material will undergo stress relaxation when the load is held constant, but by using the PID controls the stress in the material can be maintained at a constant level. Figure 4 details the hold segment for maintaining a constant level of stress in the material.

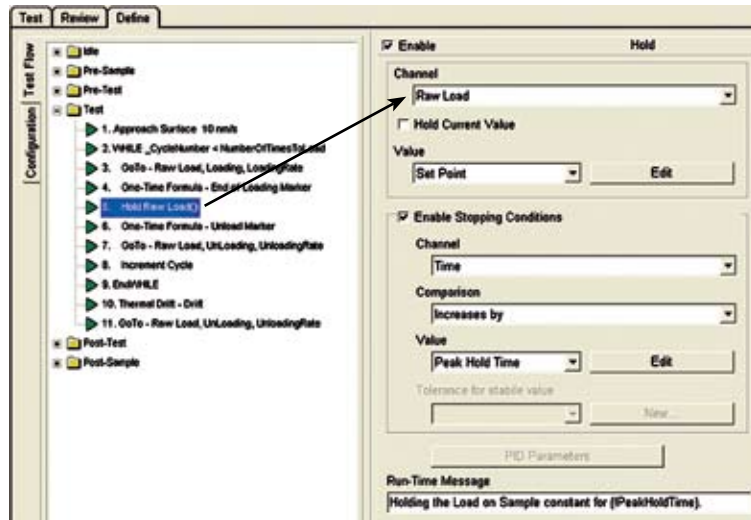


Figure 2. Logistics of controlling the raw load so that it holds the load specified by Equation 1. This hold segment will hold the raw load at the value specified by the hold load (Equation 1).

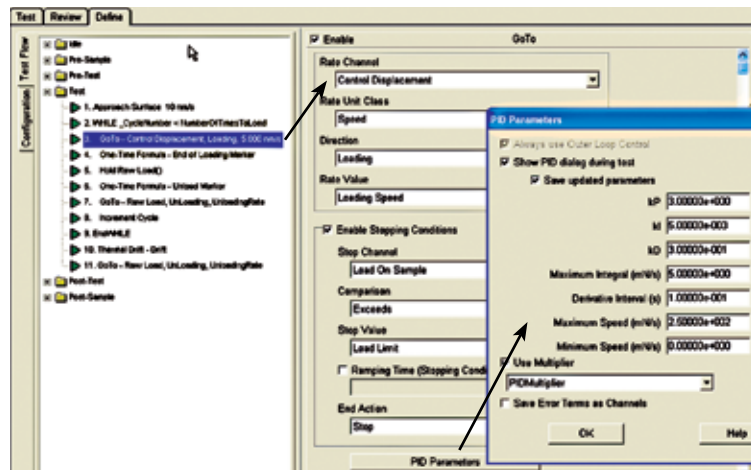


Figure 3. Controlling the loading segment at a constant displacement rate.

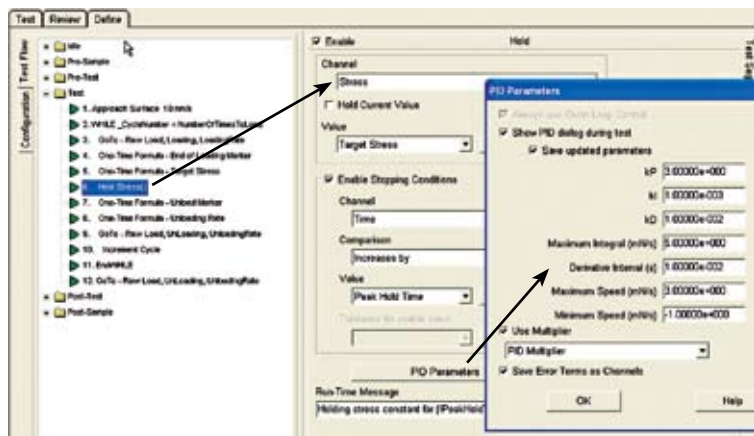


Figure 4. Details of the hold segment during a constant stress test.

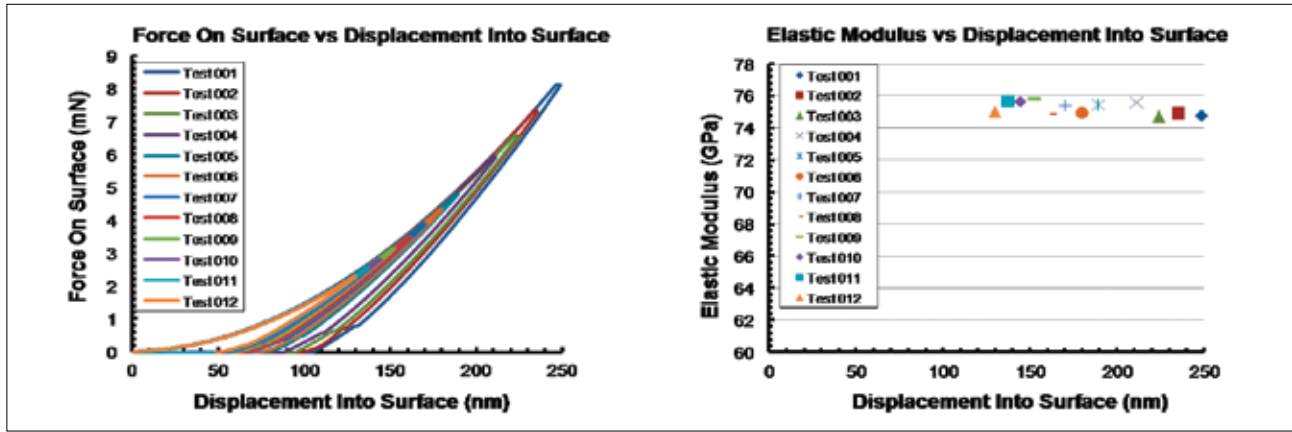


Figure 5. Instrument verification of performance on Corning 7980; the right graph shows the load versus displacement and the left graph shows the results for elastic modulus. The nominal Young's modulus of the reference material was 74.008 GPa.

Test Procedure

Three tests were used in the performance evaluation of the Nano Indenter G200. First, indentation tests were performed over a range of loads on the reference material to verify proper performance of the test instrument and to ensure proper tip calibration. The test method used follows the recommendations outlined by the ISO 14577 standard [2]. These data were analyzed and resulting mechanical properties were compared against the Certificate of Calibration provided with the reference material.

Next, two tests were performed to examine the typical noise floors and thermal drift of the Nano Indenter system. One of the tests included loading to a penetration depth of 90 nm in approximately 30 seconds, holding the load constant for a period of 300 seconds, and unloading in 30 seconds. The other test consisted of loading to 9 mN in 30 seconds, holding the load constant for 300 seconds, and unloading in 30 seconds. At the end of each test following a 90% reduction of load (from the maximum load) a 75 second hold period was conducted to examine the thermal drift rate of the system. The long hold segments at maximum load were analyzed for the displacement noise.

Three test methodologies were also used in evaluating the control of the Nano Indenter. Since flexibility is key in developing test protocols for novel materials, control was evaluated as the instrument's ability to maintain a

setting that did not use force control.

To examine this control the load on sample was held constant on a material that exhibited creep, a constant displacement rate test was conducted to maintain a constant speed during loading, and a constant stress test was conducted on a material that usually exhibits stress relaxation.

Indentation tests were conducted on PMMA to 9 mN of load. During the dwell period of the tests the load on sample was maintained using Equation 1 at a target load of 9 mN while the material continued to creep. These tests were evaluated for the error in load on sample during the hold segment as compared to a standard indentation test where the raw load channel is the only means of control. Each test was loaded to the maximum load of 9 mN in 30 seconds and then the load on sample was held constant for 300 seconds. Following the long hold period the indenter was unloaded completely.

Constant displacement rate tests were conducted at rates of 5 nm/s and 10 nm/s on PMMA. The tests were conducted to a maximum load of 9 mN. Then, the force was held constant for 5 seconds, followed by unloading and withdrawing the indenter from the sample. The displacement rates during the test were examined to evaluate the control.

The last control algorithm used to evaluate the control of the Nano Indenter system was the constant stress test. In this test the software was used to control the ratio of load on sample to the contact area during the dwell time

of the indentation. This was the most complicated control evaluated in this article because it required using the calculations of load on sample, displacement into surface, and the contact area to determine the stress and control the raw force such that the stress remained constant during the dwell time. The contact area was calculated using the displacement into surface as opposed to the contact depth calculated using the Oliver and Pharr methodology; a dynamic indentation test method would allow real-time calculation of the contact stiffness for evaluation of contact depth using the Oliver and Pharr methodology. Both epoxy and aluminum samples were used for the evaluation of control during the constant stress tests. Each test consisted of loading the indenter to a preload of 2 mN followed by a 10 second constant stress hold.

Results and Discussion Accuracy and Precision

Before precision and control testing was conducted, verification data was run on the nanoindenter to ensure proper accuracy of the instrument. Twelve indentation tests were performed on the Corning 7980 (fused silica) reference sample and the elastic modulus results were examined for accuracy as compared to the nominal reported value of 74.008 GPa for the reference material. The force curves for all twelve tests are shown in Figure 5 along with the resulting elastic modulus. These results are in good agreement with the nominal value and had an average measured

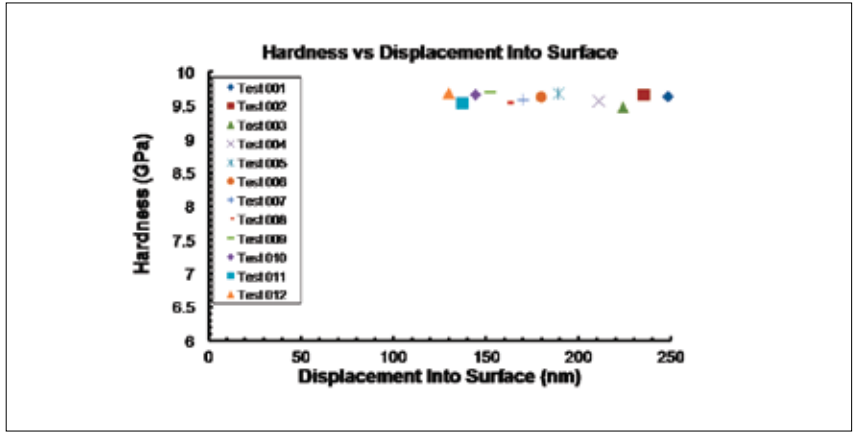


Figure 6. Instrument verification of performance on Corning 7980; results of hardness versus displacement into surface. Acceptable hardness values for fused silica range from 9.0 to 10.0 GPa.

elastic modulus of 75.2 ± 0.4 GPa. Since there is not an independent technique for verifying the hardness of a reference material used in nanoindentation, the hardness results are not compared to a nominal calibrated value. However, common values for the hardness of fused silica are between 9.0 and 10.0 GPa. The hardness results, shown in Figure 6, are in good agreement with common results for fused silica and the average measured hardness was 9.6 ± 0.1 GPa. Results for the elastic

modulus were within 2 percent of the nominal value and the covariance in the results for both elastic modulus and hardness were within 1 percent; this represents excellent accuracy and verified proper operation of the instrument.

Indentation to 9 mN of Load

Four indentation tests were performed on the Corning sample to a load of 9 mN for evaluation of the noise floor associated with the Nano Indenter

G200 DCM transducer and to examine the drift in the system. During this test the sample was loaded to 9 mN in 30 seconds and the force was held constant for 300 seconds. Following the hold period the force was reduced by 90 percent of the maximum force and the drift of the system was measured for 75 seconds. Figure 7 displays the results for the load on sample and displacement versus time for the long hold tests conducted at 9 mN.

To determine the displacement noise, the displacement into surface data was corrected for the drift rate during the hold period at maximum load, and then the standard deviation of the data during the last 150 seconds of the hold period was calculated. The standard deviation of the displacement signal during the hold period at 9 mN was measured to be 0.104 ± 0.036 nanometers. This result provides the measured variation in the displacement signal during steady-state operation of the Nano Indenter DCM transducer. Figure 8 shows a close-up of the displacement into surface versus time in contact data for examination of the displacement noise floor.

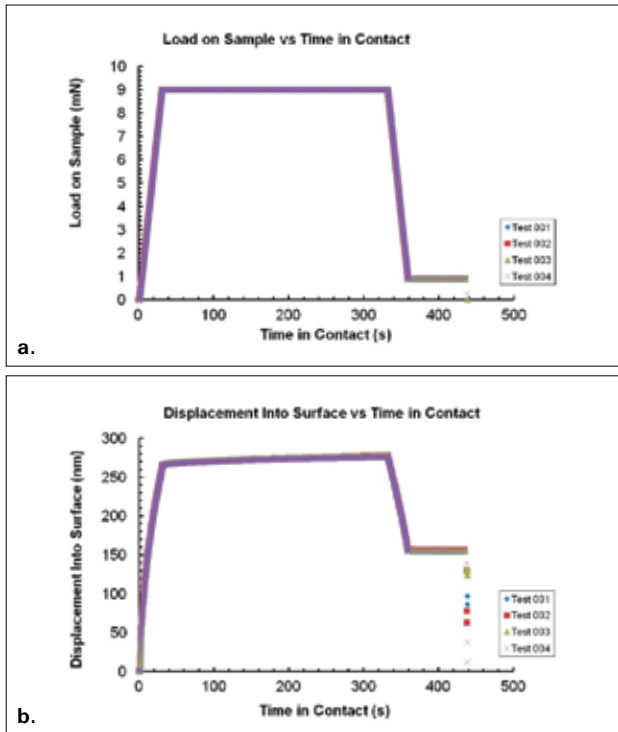


Figure 7. a) Load versus time data for the indentation test to 9 mN of load, b) displacement into surface versus time data.

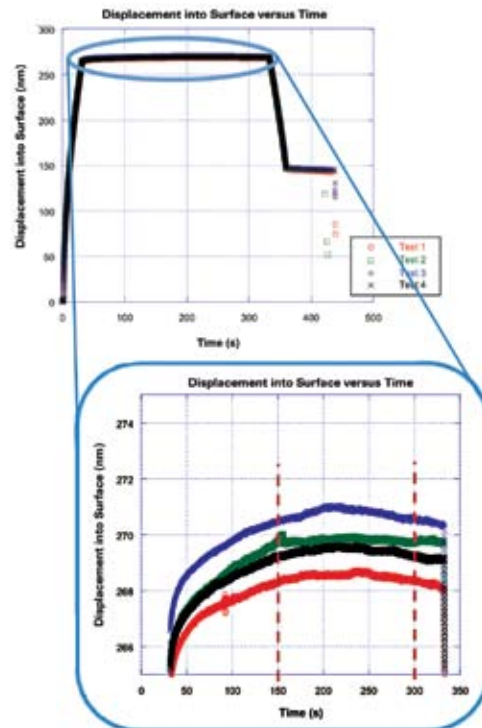


Figure 8. Close up of the displacement into surface versus time for a section of the 300 seconds hold period at 9 mN of load. The average standard deviation in the measurements of displacement during the hold period between the two red dashed lines was 0.104 ± 0.036 nm.

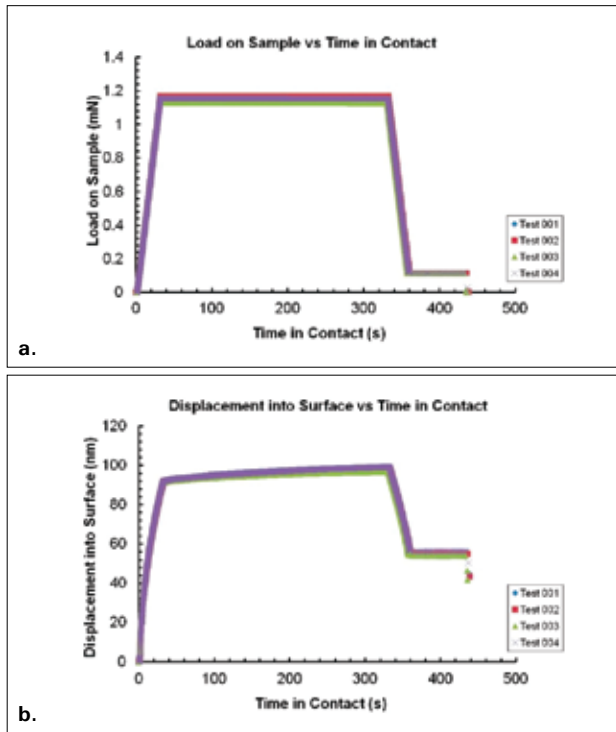


Figure 9. a) Load versus time data for the indentation tests performed to 90nm of penetration, b) displacement into surface versus time data.

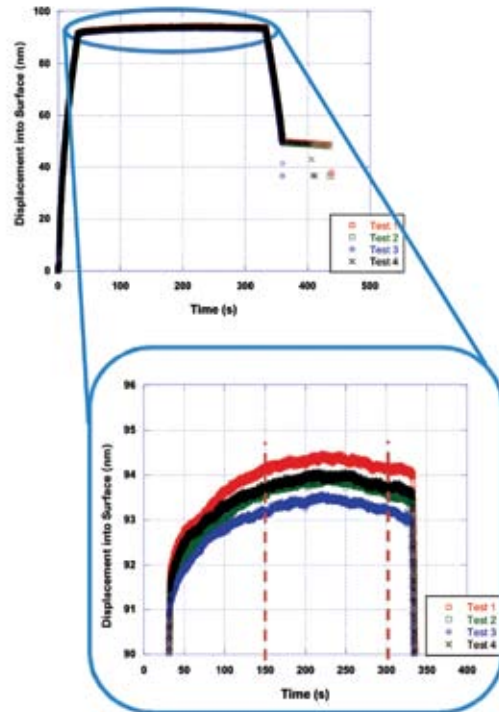


Figure 10. Close up of the displacement into surface versus time for a section of the 300 seconds hold period during the constant load hold at 90nm of initial penetration. The average standard deviation in the measurements of displacement during the hold period between the two red dashed lines was 0.089 ± 0.004 nm.

The drift rate used to correct the data in determination of the displacement noise floor should not be confused with the thermal drift rate of the system; the drift correction above was explicitly to eliminate any steady-state displacement caused by deformation mechanisms or thermal drift. Thermal drift rates should not be calculated at peak indentation force during a test because the contact has experienced just enough plasticity to support the test load—this is inherently an unstable contact. Influences of creep or other deformation mechanisms will cause further plastic deformation that cannot be decoupled from a drift measurement. By measuring drift at a greatly reduced load, this

ensures a fully elastic contact that will only be affected by thermal drift or environmental noise. Therefore, the second hold period was used to evaluate the thermal drift of the sample. Using the data collected during the 75 second hold period an average thermal drift of 0.033 ± 0.002 nm/s was calculated by performing a least squares linear fit to the data.

Indentation to 90 nm of Penetration

In the second performance test, the indenter was loaded to a penetration depth of 90 nm and the load was held constant for 300 seconds to evaluate the displacement noise floor and the drift at low loads. The same

algorithms described above were used to evaluate the displacement noise floor and thermal drift of the system. Figure 9 displays the resulting load and displacement versus time curves for the tests conducted to 90 nm of penetration and held at a constant load for 300 seconds. For the examination of the displacement noise floor, Figure 10 shows the details of the displacement curves and the red dashed line mark the bounds over which the standard deviations were calculated. The average standard deviation of the displacement during the hold segment was 0.089 ± 0.004 nanometers; this represents the variation of the displacement measurement during

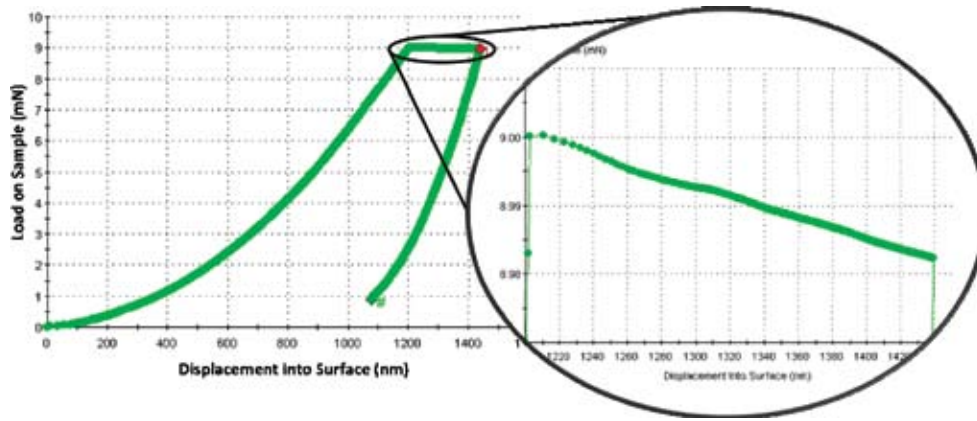


Figure 11. Indentation test on PMMA using the raw load for holding the force constant during the 300 seconds hold period.

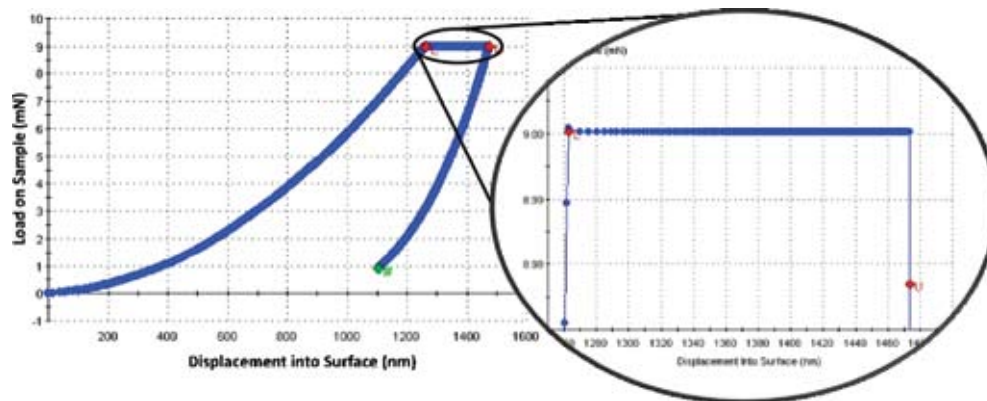


Figure 12. Indentation test on PMMA using Equation 1 to control the load on sample during the 300 seconds hold period.

a hold segment at light loads. Using the data collected in the second hold segment, the thermal drift rate of the system was calculated to be $0.026 \pm 0.004 \text{ nm/s}$.

System Control

Control of Load on Sample

When using a load controlled nanoindenter, the raw load is typically held constant at the peak load for a period of time prior to unloading the sample. This hold period is used to reduce the affects of time dependent deformation on the evaluation of stiffness from unloading the sample. During this hold period it is common for

materials to creep which causes the tip to penetrate further into the material; this added displacement causes the flexural springs in the transducer to deflect further, thereby transferring some of the raw force from the sample to the springs. Figure 11 displays this response on a sample of PMMA that was tested to 9 mN with a 300 seconds hold period. During the hold segment it is apparent that the load on sample drops by approximately $20 \mu\text{N}$; polymers that exhibit more time dependent deformation than PMMA would experience more significant decreases in the load on sample than is shown here. In some situations it is advantageous to control the load on

sample so that this load drop does not occur. The Agilent NanoSuite software allows for direct control through a formula that is calculated at every point. Therefore, Equation 1 (page 3) was used to continuously define, throughout the hold segment, the set point load that the transducer would produce. Figure 12 displays the results for a test that used Equation 1 for holding the load on sample constant during the hold segment of the test. There is no deviation or decrease from the target load on sample during this hold segment.

Displacement Rate Control

Since most (possibly all) nanoindenter systems are inherently force controlled, PID control loops must be used to conduct constant displacement rate tests. The NanoSuite software allows PID controls to be used for controlling the nanoindenter with any hardware or software data channel. A commonly requested loading algorithm for conducting nanoindentation tests is loading at a specified displacement rate as opposed to a loading rate. Figures 13 and 15 show the results from performing indentation tests on PMMA at 5 nm/s and 10 nm/s, respectively. In addition, the errors associated with holding the displacement rates at the set point are displayed in Figures 14 and 16. The indenter started from rest at the surface position and loaded at the respective displacement rates until a maximum load of 9 mN was applied to the sample. The errors in the speed during the indentation tests were, for the most part, well less than three percent. Errors at the surface of the sample for the test conducted at 10 nm/s were a bit larger because the indenter was starting from rest and required a ramp time to achieve stability. However, once this rate was achieved, the error dropped below one percent for the remainder of the test.

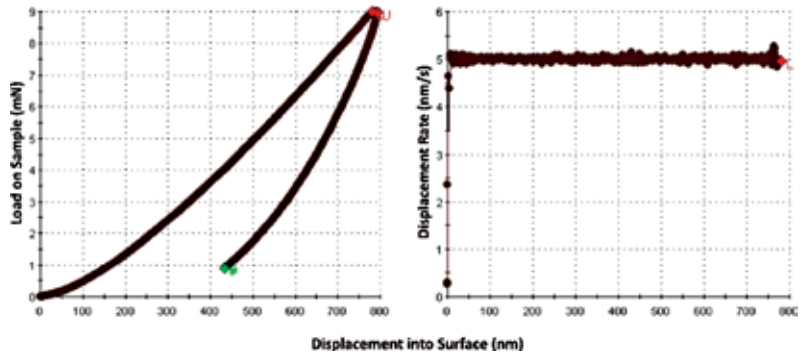


Figure 13. Constant displacement rate test performed on PMMA at 5 nm/s; the right plot shows the displacement rate only during the loading cycle.

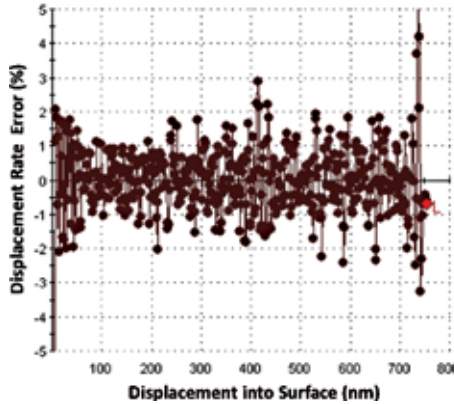


Figure 14. The error in the displacement rate during the test as compared to the target rate of 5 nm/s.

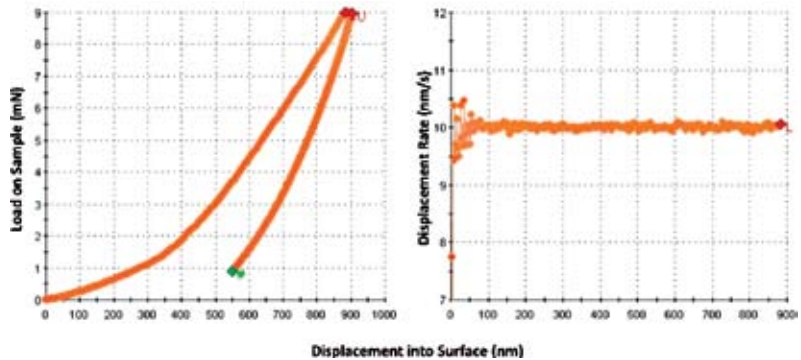


Figure 15. Constant displacement rate test performed on PMMA at 10 nm/s; the right plot shows the displacement rate only during the loading cycle.

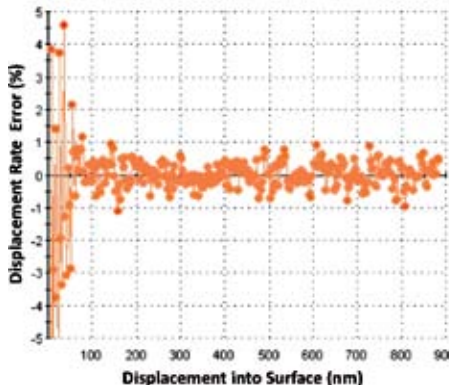


Figure 16. The error in the displacement rate during the test as compared to the target rate of 10 nm/s.

Constant Stress Test

The NanoSuite software allows a variety of controls to be used throughout a test including controls based on software calculations. In some applications, where stress relaxation occurs, it is advantageous to conduct hold segments in which the stress in the material under the indenter is held constant. Two materials, epoxy and single crystal aluminum, were selected for conducting constant stress tests. While epoxy usually exhibits smooth penetration and some slight stress relaxation during hold periods, single crystal aluminum does not exhibit smooth deformation at all. This material usually exhibits many displacement bursts during loading because dislocations are free to run through the material without constraint. This presents control challenges when performing constant stress tests because these displacement bursts serve to spontaneously reduce the stress under the indenter.

Both samples were subjected to a 2 mN pre-load; then, the stress was held constant for 10 seconds or until the load on sample reached 10 mN, whichever came first. The results for the constant stress test on the epoxy sample are shown in Figure 17; markers "L" and "U" are used to show the start and end of the constant stress test. Smooth deformation along with an increase in load is apparent throughout the hold period at a target stress of 163 MPa. Figure 18 displays the error in holding the target stress value during the test. It is clear from this figure that the constant stress test on epoxy exhibited excellent control and the error in maintaining the set point stress was less than 1.5 percent.

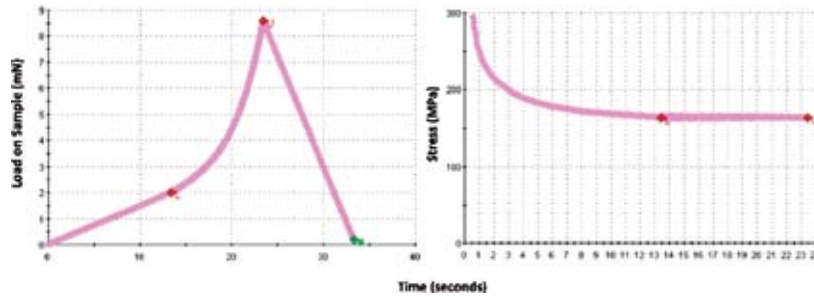


Figure 17. Results for the constant stress test performed on the epoxy sample.

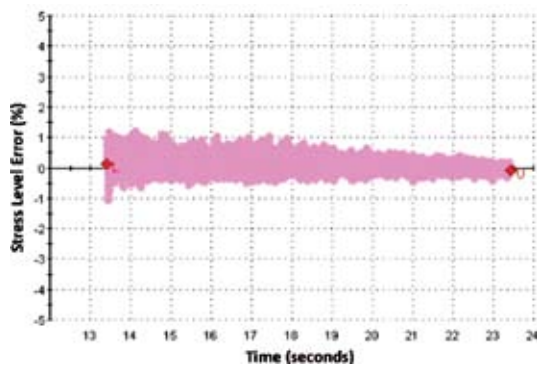


Figure 18. The error in the stress control as compared to the commanded set point.

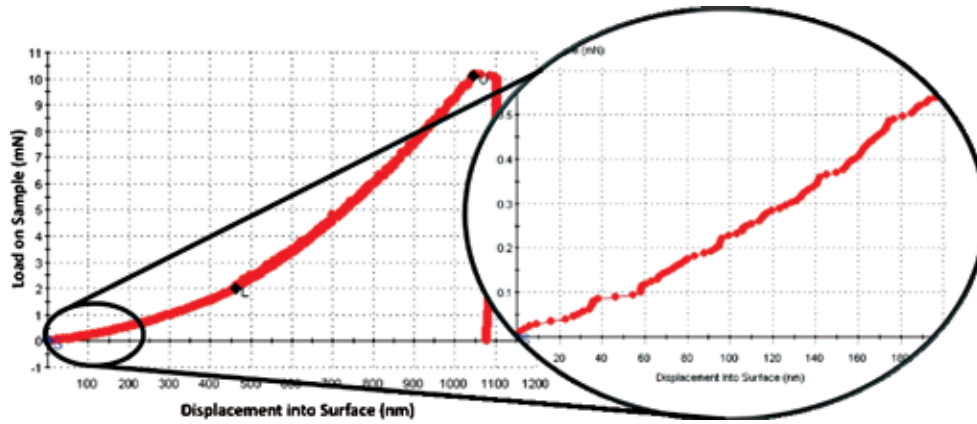


Figure 19. Load versus displacement curve for the single crystal aluminum sample showing displacement bursts during loading.

The constant stress test on the single crystal aluminum sample proved to be more difficult to control than on the epoxy sample due to the displacement bursts that occurred during testing. These displacement bursts are clearly seen in Figure 19 during the first loading segment of the test while the force was increased to 2 mN. Figure 19 displays the load versus displacement curve for the test performed on the aluminum sample with a zoomed view of the first 200 nm of penetration to provide more detail on the displacement bursts. Displacement bursts are seen at 20, 40, 85, 100, 145, and 180 nanometers of displacement into surface; in fact, these bursts continue throughout the test. The combination of the displacement bursts and the low yield stress of this material – low yield stress in a material creates a contact during indentation with very little elasticity – provided a challenge in controlling the stress in the material. Figure 20 displays the results of the constant stress test on the single crystal aluminum sample. The set point stress for the hold segment was 322 MPa; this was the stress that was present in the material at the instant when 2 mN of load was applied during the loading segment of the test. It is apparent that the loading rate required to maintain a constant stress in the aluminum sample was almost double the rate for the epoxy sample.

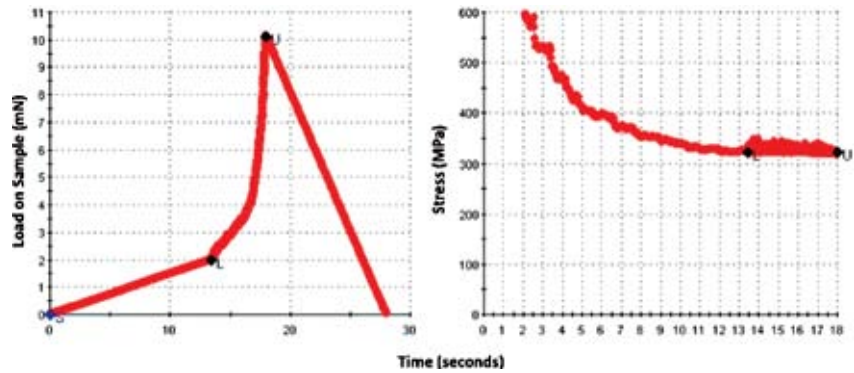


Figure 20. Results for the constant stress test performed on the single crystal aluminum sample.

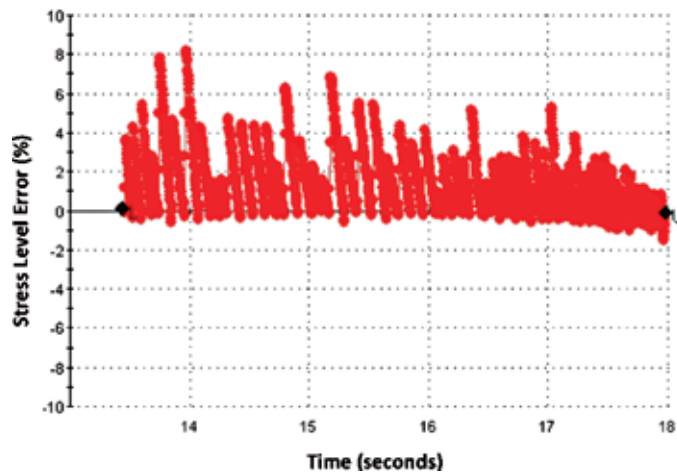


Figure 21. The error in the stress control as compared to the commanded set point.

Errors associated with holding the stress at the set point in the aluminum sample, shown in Figure 21, were larger than the errors displayed for the epoxy sample. One large difference in the response of these two materials is that the aluminum sample shows very little negative error; while the positive error was as high as 8 percent, the negative error was only about -1 percent. The error in maintaining the stress set point during the test on epoxy showed balanced positive and negative error. During the constant stress test of the aluminum sample, it was preferential to have more stress in the material rather than too little because of its extreme plastic nature—if the stress was too low, a further small decrease in force would cause loss of contact. Commonly the control of stress was within 5 percent of the set point, but there were a few occurrences in which the stress exceeded the set point by as much as 8 percent.

Conclusions

The accuracy, precision, and control of the Nano Indenter system with the DCM option have been demonstrated. Accuracy of the system was tested at multiple forces using a verified reference material and the results showed excellent agreement as compared to the nominal calibration values. In addition, the covariance in the results for elastic modulus and hardness were less than 1 percent.

Given that the calibrations of the instrument are correct, then there are two primary sources of precision error: displacement noise and thermal drift. The precision of the DCM transducer was evaluated at two force levels, approximately 1 mN and 9 mN of force. Results for the displacement noise floor of the DCM transducer were determined by eliminating the steady-state deformation or drift that occurred at the maximum load and evaluating the standard deviation in the displacement

data over 150 seconds; the results of the displacement noise floor were 0.089 ± 0.004 nm and 0.104 ± 0.036 nm for the 1 mN and 9 mN holds, respectively. Thermal drift rates during these tests were less than 0.04 nm/s.

Three test protocols were developed to evaluate the control of the Nano Indenter system by means other than force control. The test protocols included controlling the load on sample, displacement rate, and stress during the tests. Each control was implemented using either a formula that was continuously updated or PID controls. All of the tests showed the power of the Nano Suite software when flexibility and control are needed.

References

- [1] B. A. Crawford. Indentation Rules of Thumb. Agilent Technologies, Application Note, 2010.
- [2] "Metallic Materials – Instrumented indentation test for hardness and materials parameters." ISO 14577, 2002.

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