High Temperature Mechanical Characterization of Tin (Sn) Using Nanoindentation

Application Note

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Introduction

Soft metals that exhibit time dependent deformation can be difficult to characterize using quasi-static nanoindentation testing due to creep – causing an increase in the penetration depth – while initially unloading the sample to measure stiffness. The problem is further compounded when testing samples at elevated temperature because traditional techniques of holding the peak force until the creep is minimized is not an option; tests must be completed in a timely manner to minimize the effects of thermal drift. A new test protocol has been developed for the Agilent Nano Indenter G200 that allows dynamic testing to be completed in combination with the Heating Stage option using the Continuous Stiffness Measurement (CSM) technique. This enables accurate measurements of mechanical properties on time-dependent materials at elevated temperatures. This article demonstrates the new test protocol for measuring elastic modulus and hardness on high purity tin (Sn) over the temperature range from room temperature to 150 degrees Celsius. In addition, results from quasi-static testing of the tin sample are compared to results obtained using the continuous stiffness measurement technique.

Sample

The sample was high purity Sn and was mechanically polished to a mirror finish. Figure 1 shows the sample mounted to a puck for testing. Due to the thickness of the sample (approximately 10 mm thick), a thermocouple probe was adhered in a hole located in close proximity

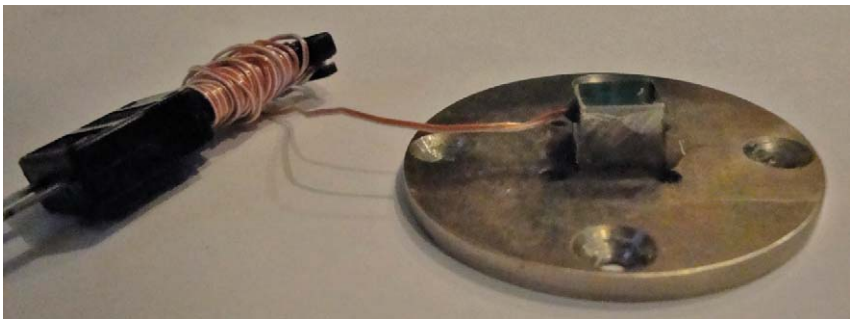

Figure 1. The Sn sample mounted to a puck for testing. A thermocouple is mounted in close proximity to the top surface of the sample.

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to the top surface of the material. This thermocouple was used to control and monitor the temperature during testing. A microscope image of the surface using 250X effective magnification is provided in Figure 2.

Test Equipment

Tests conducted in this article were performed using the Agilent Nano Indenter G200 with an XP transducer, the Continuous Stiffness Measurement (CSM) option, and the Heating Stage option. The CSM option is traditionally used to measure the evolution of mechanical properties as a function of penetration into the surface of the sample. However, when employing the CSM to perform dynamic measurements at elevated temperatures, it is used to acquire measurements of stiffness only during a hold segment at the peak indentation force. The Heating Stage allows nanoindentation testing to be completed over a temperature range from room temperature to 350 degrees Celsius and includes an active system to remove waste heat, a heat shield to block the transfer of heat to the electronics, and an argon supply to reduce oxidation of the surface (primarily for testing above 200 degrees Celsius). Argon, which is heavier than air, is supplied to the sample from underneath the mounting structure and encapsulates the sample in a bubble of argon.

Test Protocol

Five tests were conducted on the Sn sample at temperature settings of room temperature, 50°C, 70°C, 100°C, and 150°C. For these tests, the CSM technique was used to collect stiffness data at the peak indentation force. Traditionally, the CSM technique is used throughout the loading segment of an indentation test; however, the loading rate for conducting standard CSM tests is slower than the loading rates

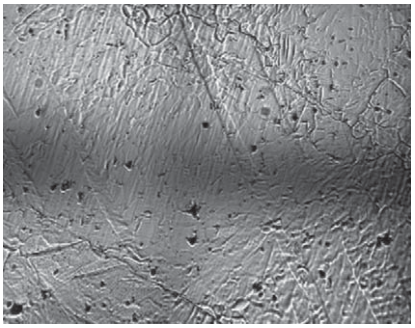

Figure 2. Microscope image of the surface of the Sn sample at 250X magnification.

required when testing on the heating stage. The internal PID controls that maintain a measurable harmonic displacement oscillation (usually 1 to 5 nm) for the CSM measurement do not react fast enough at high loading rates and consequently the harmonic displacement quickly drops to nearly zero displacement. Time is of the essence when testing at elevated temperatures — test should be conducted quickly to minimize adverse effects of thermal drift; therefore, slowing down the loading rate is not an option. To perform dynamic testing in an effective and timely manner a pre-test is used to eliminate the PID controls by determining the appropriate harmonic force to generate measurable harmonic displacement at the peak indentation force. Subsequently, the test protocol has the structure listed below. In addition, the test parameters used are listed in Table 1.

1. The indenter approaches the surface of the sample at a location well removed from the test location (usually a 75 µm offset in the X and Y directions) until contact is detected.
2. Sample is loaded until the peak indentation force is reached.
3. The peak force is held constant until the harmonic displacement becomes equal to the target value; at this point the harmonic force needed to generate the harmonic displacement oscillation is recorded.
4. The indenter is withdrawn from the surface and positioned above the target test location.
5. Once again, the indenter approaches the surface of the sample until contact is detected.
6. The sample is loaded until the peak force is reached and the recorded harmonic force (recorded in Step 3) is applied to generate a measurable displacement oscillation.
7. The peak force is held constant for a short period of time.
8. Finally, the sample is unloaded and the indenter is withdrawn from the sample.

Time to Load	1.5 seconds
Maximum Load	20 mN
Peak Holding Time	3.0 seconds
Time to Unload	1.5 seconds
Harmonic Displacement Target	5 nm
Harmonic Frequency	50 Hz

Table 1. Test parameters for the CSM indentation tests performed on the Sn sample.

For the comparison of techniques, quasi-static indentation tests were also performed on the Sn sample. During these tests, stiffness was determined from the slope of the unloading curve. The quasi-static test protocol consisted of the steps listed below and the test parameters for the quasi-static tests are listed in Table 2.

Time to Load	1.0 seconds
Maximum Load	20mN
Peak Holding Time	1.0 second
Time to Unload	1.0 second

Table 2. Test parameters for the quasi-static indentation tests performed on the Sn sample.

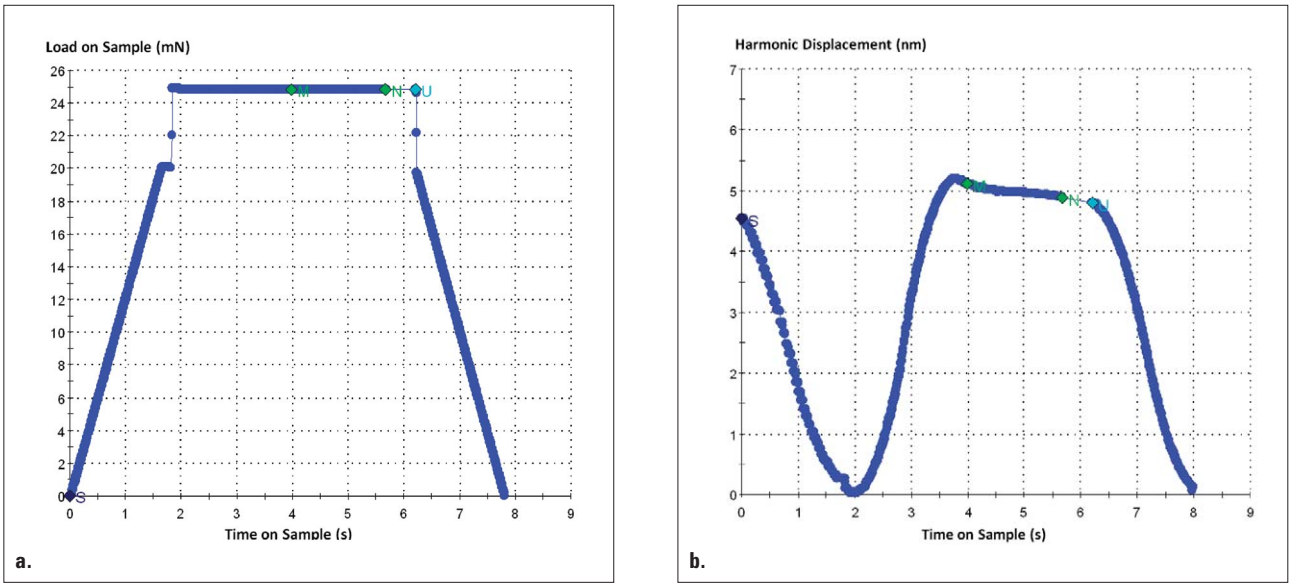

Figure 3. Load-time history for the CSM mode tests performed on the Sn sample (a). At time 1.6 seconds, the harmonic force is instantly increased to generate a measurable harmonic displacement. The resulting harmonic displacement (b) rises quickly in response to the application of the harmonic force.

1. The indenter approaches the surface of the sample at the test location until contact is detected.

2. Sample is loaded to the peak indentation force.

3. The peak force is held constant for a short period of time.

4. Finally, the sample is unloaded and the indenter is withdrawn from the sample.

All of the data from the CSM tests and the quasi-static tests were analyzed using the Oliver-Pharr analysis described elsewhere [1].

Results and Discussion

The CSM technique was implemented for testing the Sn sample because Sn typically exhibits time-dependent deformation. When time dependent deformation occurs, it becomes difficult to decouple creep and elastic recovery during the unloading segment of a quasi-static test for the calculation of stiffness. Therefore, as opposed to measuring the stiffness from unloading the material, the CSM technique was used to impose a 5 nm harmonic displacement oscillation during the hold period at peak indentation force. Typically, the CSM technique is used during the loading segment of the indentation test to provide the evolution of mechanical properties as a function of indentation depth; however, loading rates — which are on the order of 15 mN/s — used on the heating stage are simply too fast to collect continuous stiffness data as a function of penetration.

To employ the CSM technique on the hot stage, a pre-test was performed to determine the harmonic force required to generate a 5 nm harmonic displacement oscillation at the peak indentation force. During the actual indentation test, the sample was loaded to the peak indentation force; then, the predetermined harmonic force was applied to quickly produce the required harmonic displacement. Figure 3 shows the load versus time history for the indentation tests conducted using the elevated temperature stage. The load jump at approximately 1.8 seconds is a result of applying the harmonic force during the hold segment of the test. Load on sample and displacement into surface are both calculated using the contribution of the harmonic force and displacement, respectively, as described by Pharr *et al* [2].

Figure 3 also shows the harmonic displacement versus time. During the load ramp to the peak indentation force, the PID controls did not react quick enough to control the harmonic displacement at the target value — as was expected. A 4.5 nm harmonic displacement oscillation was present upon contact with the surface of the material and as the sample was loaded the harmonic displacement dropped to approximately 0 nm. With the application of the pre-determined harmonic force, the harmonic displacement quickly recovered to 5 nm.

The load on sample versus displacement is shown in Figure 4. A force of 20 mN was applied in 1.5 seconds which created approximately 2800 nm of displacement. The sample immediately started to creep when loading was completed — within fractions of a second the penetration depth had already increased by an additional 200 nm. With the application of the harmonic force, a large increase in the load on sample occurred — this force increase was an instantaneous increase of force and shows up as a step increase in the load on sample. The total creep during the hold segment was approximately 700 nm, yielding an average creep rate of 151 nm/s — the creep rate for the tests conducted at all of the temperatures ranged between 139 nm/s and 151 nm/s.

Results for the elastic modulus and hardness — determined from the dynamic measurements of stiffness during the hold segment — are shown in Figures 5 and 6, respectively. A decrease in the elastic modulus and hardness as a function of temperature is observed. There was a 26% decrease in the elastic modulus as the temperature was increased to 150°C — the melting point for Sn is 231.9°C [3]. The hardness decreased by 36% over the temperature range.

These results scale well with the upper and lower bounds on predicted shear and bulk modulus values as a function of temperature provided by Adams [4]. Adams determined lower and upper bounds for the bulk and shear modulus over the temperature range from –130°C to 150°C using Voigt and Reuss bounds for polycrystalline materials with measured single crystal constants determined by ultrasonic testing. The estimated upper and lower bounds presented by Adams showed

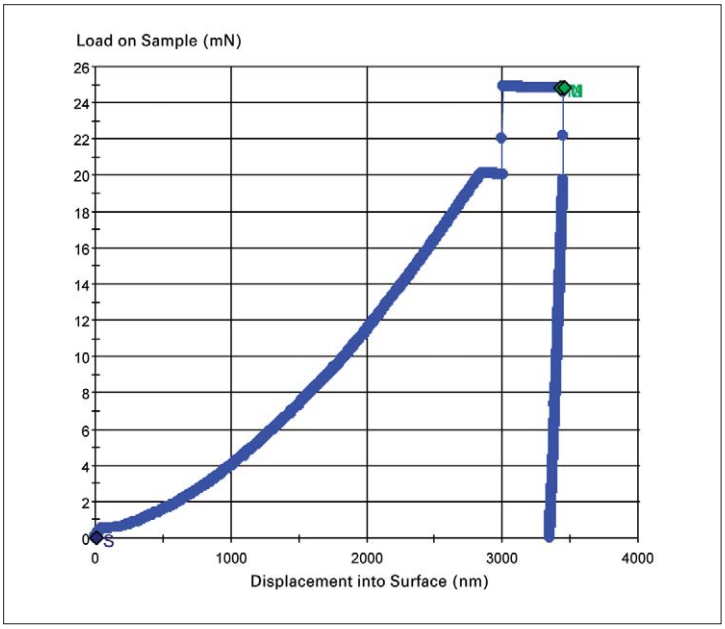

Figure 4. A typical load on sample versus displacement plot for the CSM mode tests performed on the Sn sample. At time 1.6 seconds the harmonic force is instantly increased to generate a harmonic displacement.

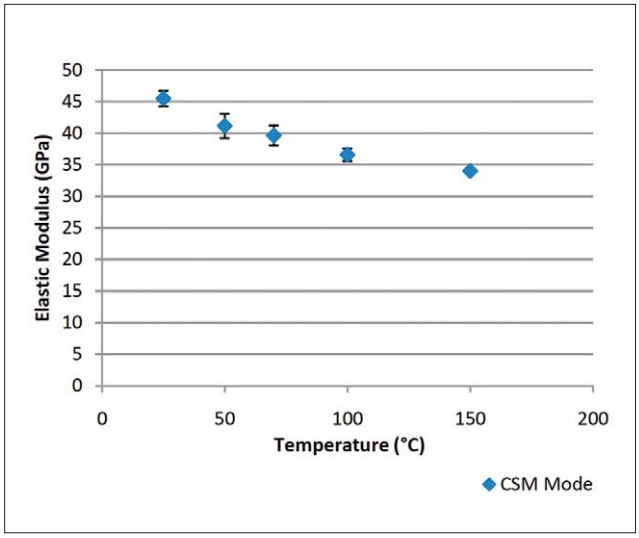

Figure 5. Elastic modulus results as a function of temperature for the CSM mode tests. The melting point of SN is 231.9°C [3]. One standard deviation is represented by the error bars.

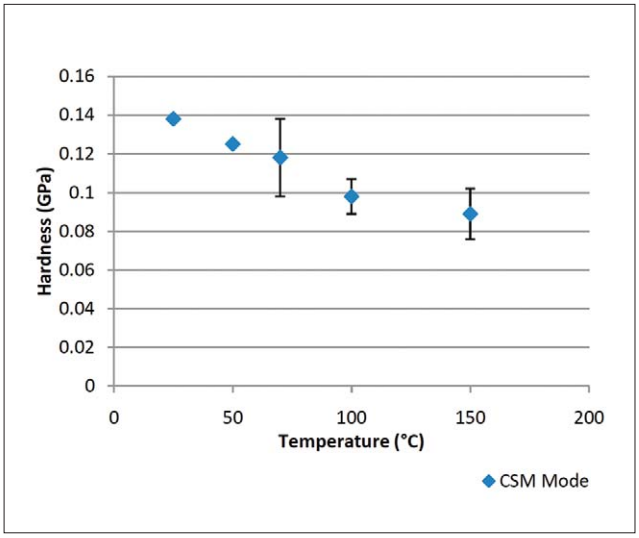

Figure 6. Hardness results as a function of temperature for the CSM mode tests. One standard deviation is represented by the error bars.

a decrease in the shear modulus of pure tin to be 16% to 35% and a decrease in the bulk modulus of 7% over the temperature range from room temperature to 150°C.

Quasi-static indentation testing was performed at the elevated temperatures to compare the results to the CSM mode results and demonstrate the challenges associated with performing quasi-static tests on time dependent materials at elevated temperatures. A typical load versus displacement curve for the quasi-static tests is displayed in Figure 7. As anticipated, the creep rate at peak force caused an initial negative stiffness because as the sample was initially unloaded the sample continued to creep at a rate of approximately 150 nm/s. This caused a severe sensitivity to the results of stiffness based on the amount of data that was used to determine the power-law curve fit of the unload data. The more unload data that is used to fit the curve decreases the influence of the initial unloading data; however, the initial unloading data is the most representative data for the stiffness of the material as determined by the Oliver-Pharr analysis. Traditionally, 50% of the unloading data is used in the determination of the power-law curve fit.

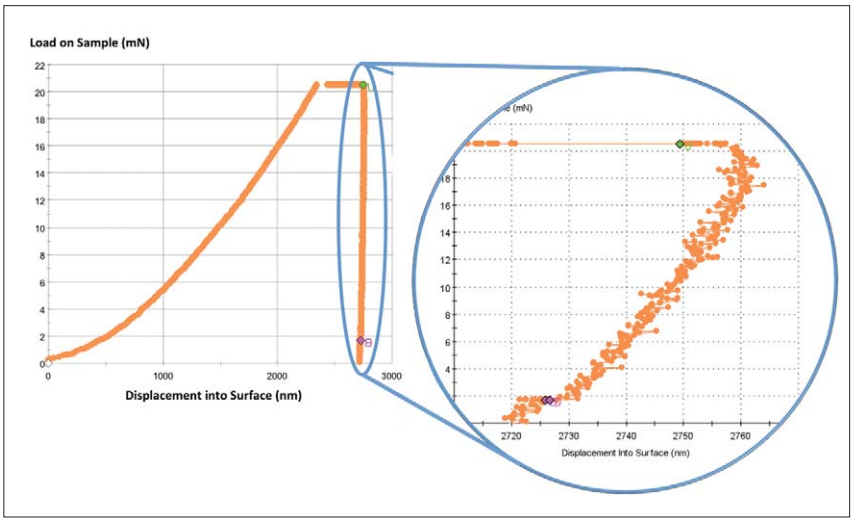

Figure 7. A typical load versus displacement plot for the quasi-static indentation tests. Creep continues in the material even as the load is withdrawn from the sample.

Figures 8 and 9 show the results of the elastic modulus and hardness from the quasi-static mode tests as compared to the CSM mode tests. In these plots, the results for the quasi-static tests were analyzed using 50% and 70% of the unloading curve to determine the power-law curve

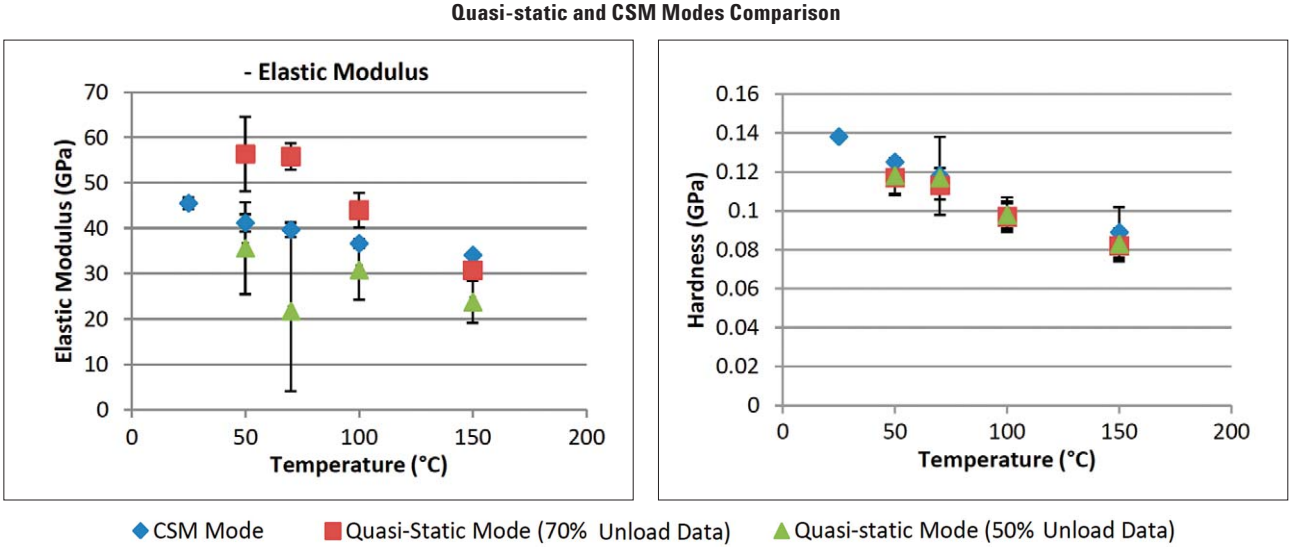

Figure 8. Elastic modulus results as a function of temperature for the quasi-static mode tests compared with the CSM mode tests. The scatter in the quasi-static mode tests is a direct result of time-dependent deformation occurring during unloading. The quasi-static results show high sensitivity around how the data from unloading is analyzed.

Figure 9. Hardness results as a function of temperature for the quasi-static mode tests compared with the CSM mode tests. The results for hardness show that the elastic deformation in the contact is almost non-existent and the contact depth is almost equal to the total penetration depth.

fit and, consequently, the stiffness. The results for elastic modulus clearly show that the stiffness measurements, as determined from the slope of the unloading curve at the initial point of unloading, are very sensitive to the amount of unloading data that is used in the curve fit when testing Sn. In contrast to the results for the elastic modulus, the hardness results agree well with the CSM results. This is because the contact area is approximately equal to the total penetration depth — there is very little elastic deformation in the contact between the tip and sample. When using the Oliver-Pharr analysis, the contact depth (h_c) is determined by Equation 1 [1].

$$h_c = h - \epsilon \frac{P}{S} \quad (1)$$

Where h is the maximum penetration depth, ϵ is a constant that depends on indenter geometry, P is the maximum load, and S is the stiffness of the contact at the maximum load. For the tests on the Sn sample, the unloading curve was almost vertical and exhibited very little elasticity creating a contact stiffness that approached infinity, as is typical for high stiffness materials that have low yield strength. This effectively reduced Equation 1 to

$$h_c = h \quad (2).$$

In fact, when the total penetration depth was substituted for the contact depth, the results for elastic modulus and hardness changed by less than 4%. This points to the determination of stiffness by quasi-static indentation techniques as the error in the measurement of elastic modulus in Figure 8.

Conclusions

Mechanical properties of elastic modulus and hardness were determined for Sn over a temperature range from 25 degrees Celsius to 150 degrees Celsius; both the elastic modulus and hardness of the Sn sample decreased by 26% and 36%, respectively, over the temperature range. The decrease in elastic modulus measured by the CSM technique scaled well with the estimated decrease in the elastic constants of pure tin presented by Adams [4]. Average creep rates during the hold segment at peak force were measured to be 139 nm/s at room temperature and 151 nm/s at 150°C. This caused great difficulty in the measurement of stiffness from the unloading curve using quasi-static indentation testing. The CSM technique proved to be essential in the appropriate determination of the elastic modulus of metals that exhibit time-dependent deformation such as Sn. Measurements of hardness on the Sn sample provided statically equivalent results when measured using quasi-static and CSM mode indentation testing. This was due to the very limited amount of elasticity present in the contact between the tip and the sample; the contact depth was shown to be approximately equal to the total penetration depth.

References

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