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A direct comparison of high temperature nanoindentation creep and uniaxial creep measurements for commercial purity aluminum

P. Sudharshan Phani*, W.C. Oliver

Nanomechanics Inc., 105 Meco Ln, Oak Ridge, TN, 37830, USA

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ABSTRACT

Measuring the uniaxial creep response from nanoindentation has been of great interest to the small scale mechanics community. However, several experimental and modeling challenges pose obstacles to direct comparison of indentation and uniaxial results. In this work, new experimental procedures are developed to improve the precision and accuracy of high temperature nanoindentation tests. Indentation creep experiments are performed on commercial purity aluminum alloy at a number of temperatures up to 550 °C. The activation energy for creep was found to be 140.2KJ/mol/K, matching the value determined with high temperature uniaxial creep experiments extremely well. Uniaxial power-law creep parameters (stress exponent and pre-exponential term) are calculated from the indentation data for direct comparison of results to the uniaxial data. The results are in good agreement with the literature values for uniaxial compression/torsion tests over a wide range of strain rates and temperatures demonstrating the capabilities of high temperature indentation creep experiments. The relative contributions and interplay of indentation size effect, strain rate and temperature on the creep response is also explored.

1. Introduction

Recently, there has been an increased interest in high temperature nanoindentation due to improved instrumentation and better experimental techniques [1–7]. This has further extended the capability of nanoindentation based techniques to study temperature and rate effects on the mechanical properties of small volumes of materials. In this regard, there is great value in measuring the uniaxial creep response from indentation especially with the commonly used Berkovich indenter. However, the complexity of stress fields during indentation often precludes direct measurement of uniaxial constitutive behavior [8]. For instance, in the case of power-law creep, it is relatively simple to measure the uniaxial stress exponents [9] from indentation but not the uniaxial pre-exponential terms. However, Su et al. [8] recently proposed a simple experimental technique to measure the uniaxial power-law creep parameters (stress exponent and pre-exponential term) by instrumented indentation based on a theoretical analysis of Bower et al. [10] and showed good agreement between the indentation and uniaxial results for amorphous selenium at 35 °C. However, the technique has not been validated on more commonly used engineering materials at high temperatures.

Performing nanoindentation measurements at elevated temperatures is challenging on several fronts as elucidated elsewhere [3,7]. These challenges limit the use of the experimental data especially for studying creep at elevated temperatures. In this work, we present new experimental techniques to address some of these challenges and improve the accuracy and precision of the measurements at elevated temperatures. Indentation creep experiments are performed on the widely used commercial purity aluminum alloy (1100 Al) alloy at a number of temperatures up to 550 °C to measure the uniaxial power-law creep parameters (stress exponent and pre-exponential term). These results are compared to the uniaxial compression/torsion data to assess the feasibility of measuring uniaxial power-law creep parameters using high temperature nanoindentation. Recently, several research groups have also explored the temperature dependence of size effects and the interplay between size effects and rate effects [11–15]. Here, we present the relative effects of temperature, strain rate, indentation size effect (ISE) and microstructure on the measured creep response to identify the best experimental procedures to study indentation creep at elevated temperatures.

* Corresponding author.
E-mail address: s.phani@nanomechanicsinc.com (P.S. Phani).

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2. Experimental procedures

2.1. Challenges and solutions

As mentioned earlier, performing nanoindentation measurements at elevated temperatures is challenging on several fronts. Here, we discuss the major challenges specific to indentation creep testing. While some of the issues cannot be completely addressed, we can significantly improve the precision and accuracy of the indentation creep measurements by the following guidelines.

2.1.1. Thermal equilibrium during testing

Maintaining thermal equilibrium between the tip and the sample during testing is very important [4]. However, engineering difficulties in precisely positioning the thermocouples and the associated thermal gradients due to the sample geometry are some of the obstacles to achieving this. To overcome this issue, we use a simple procedure assuming that if the tip and the sample are in thermal equilibrium, the temperature of the tip does not change upon contact with the sample. The tip temperature is initially set to the desired value and contact is established with the sample. The sample temperature (close to the tip temperature) that does not result in any change in tip temperature upon contact with the sample is then determined. Note that, it is still important to have a thermally stable testing system and to position the thermocouples as close to the region of interest as possible to minimize this offset. Fig. 1 shows the change in tip temperature upon contact with the sample at different temperatures, while the tip is nominally being maintained at 250 °C. It is evident from the plot that the sample temperature can be adjusted to minimize the change in the tip temperature upon contact. In this case, a sample temperature of 268.3 °C results in virtually no change in tip temperature upon contact, thereby resulting in thermal equilibrium during testing. This procedure can be repeated once at every test temperature for a given sample to determine the offset. Fig. 2 shows the tip temperature upon contact with the sample at different set temperatures after determining the respective offsets. For a wide range of test temperatures shown in the plot, there is no significant change in the tip temperature upon contact thus ensuring thermal equilibrium during mechanical testing. The offset between the tip and sample temperatures results is some ambiguity in accurately determining the test temperature. However, as the tip thermocouple is closest to the contact, the tip temperature is taken to be the test temperature in this work.

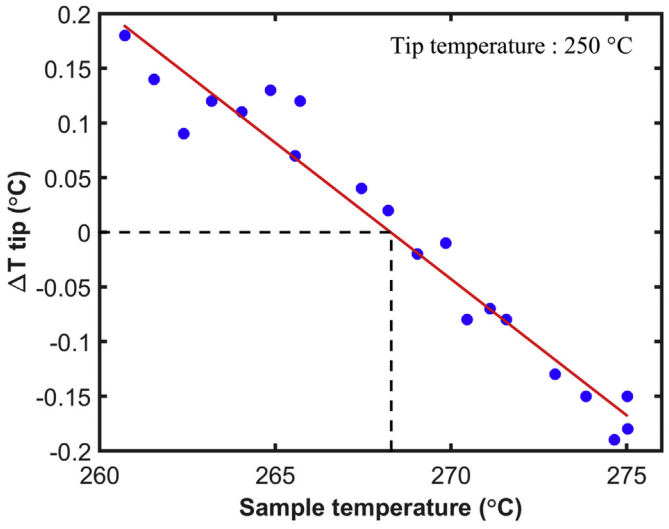

Fig. 1. Change in tip temperature upon contact with the sample at different temperatures. In this case, the tip temperature is maintained at 250 °C.

2.1.2. Thermal drift

Thermal drift is the displacement that results from the dimensional changes caused due to the thermal fluctuations within the testing system. It is one of the major factors that affect nanoindentation measurements especially at elevated temperatures. Performing fast tests while maintaining low drift rates and properly accounting for the thermal drift can help overcome drift issues. This is even more critical for creep testing where the test duration is much longer than usual. Here we describe a simple procedure to determine the thermal drift during high temperature nanoindentation testing where traditional methods to correct for drift cannot be used.

The procedure relies on the fact that in a well-designed high temperature nanoindentation system, thermal drift can be directly correlated to the temperature changes of the tip and the sample. The procedure involves systematically varying the tip and the sample temperatures to result in displacement changes that can be completely attributed to thermal drift. A simple model can then be used to correct the displacement using the known temperature variations. The following equation is used to correct the displacement for static and dynamic variations in tip and sample temperatures.

$$h_{corr}(t) = h(t) + \alpha\Delta T_{tip}\left(t + \tau_{tip}\right) + \beta\Delta T_{sample}\left(t + \tau_{sample}\right), \qquad (1)$$

where, h_{corr} is the drift corrected displacement, h is the uncorrected displacement, α and β are the tip and sample temperature coefficients respectively and τ_{tip} and τ_{tip} are akin to the thermal time constants of the tip and the sample. Fig. 3a shows the temperature fluctuations purposely and systematically induced on the tip and sample. Large variations in temperatures have been deliberating introduced to improve the accuracy of the model. However, the typical temperature variation during a creep test is <0.05 °C. Fig. 3b shows the uncorrected and temperature corrected displacement using Eq. (1). The fact that the corrected displacement is almost zero for any type of temperature variation indicates that the current experimental setup can be precisely modeled by Eq. (1), thereby enabling high temperature measurements without any significant drift. Note that, it is still important to have a thermally stable testing system with small changes in displacement for a given temperature change.

2.1.3. Indentation size effect (ISE)

ISE commonly refers to a phenomenon where the hardness changes as a function of probed volume or impression size [16]. This can lead to misinterpretation of indentation creep data where the desire is to measure strain rate effects directly. This issue can be addressed by performing creep tests at large depths, where the ISE is not only less significant but also the additional creep displacements do not change the contact area as much as at shallower depths. Large indents also minimize the errors due to tip area calibration.

2.1.4. Inhomogeneous sample

Most engineering materials are usually not homogeneous at small length scales and hence can exhibit local variations in mechanical properties at that scale. For example, commercial purity aluminum (1100 Al) has less than 1% of many minor phases of Fe-Al-Si that alter the local mechanical properties. This can lead to inaccuracies in the creep data especially if hardness at different strain rates is measured at different locations. This can be partly addressed by measuring hardness over a wide range of strain rates at the same location. Additionally, performing a large number of tests can provide better statistics for samples with