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Nanoindentation creep of tin and aluminium: A comparative study between constant load and constant strain rate methods

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ABSTRACT

Creep properties of polycrystalline Tin (Sn) and single crystal Aluminium (Al) were studied by two nanoindentation methods, i.e., constant load (CL) test and constant strain rate (CSR) test. The indentation strain rate and stress were calculated as the analogies drawn from uniaxial creep analysis. The stress exponent was expressed as the slope of the *strain rate–stress* curves plotted in the double logarithm scale. Between the two testing methods, the CSR test was clearly shown to be able to detect the creep of Sn in the power-law region, where the grain size had little effect on the creep rate. However, it was found that steady-state creep could not be achieved in the CL test. This has imposed ambiguities in applying the creep analysis developed from conventional creep scheme. The creep displacement from CL test was found unrepeatable for multiple measurements. CL test also has a smaller accessible stress range than that from a CSR test. The gradual variation of the stress exponents, especially for the small grain Sn sample, during holding process in the CL test could be due to the participation of the other rate controlling mechanisms which were closely related to the non-steady-state creep behaviour.

1. Introduction

Nanoindentation technique has been widely used to characterize the mechanical properties of materials. It has indispensable advantages in probing the properties of features down to submicron in size, e.g., solder alloys [1–5] or multi-layer structures [6–9] which are beyond reach by conventional mechanical tests. On this account, it has been involuntarily utilized in creep analysis in materials research [3,10–16]. To establish the external stress and creep rate relationship in an indentation test, analogies are typically drawn between a uniaxial stress, σ, and the indentation hardness, H, from an indentation analysis [14,17]:

$$\sigma = k_1 \cdot H = k_1 \cdot \frac{P}{A_c} \quad (1)$$

where k_1 is a constant, P is the applied load and A_c is projected contact area. The strain rate from an indentation test, $\dot{\varepsilon}_i$, is defined as the instantaneous displacement rate of the indenter, dh/dt, divided by the instantaneous displacement, h:

$$\dot{\varepsilon}_i = k_2 \cdot \left(\frac{dh}{dt}\right) \cdot \left(\frac{1}{h}\right) \quad (2)$$

where k_2 is a constant.

Using these analogies, various types of indentation creep experiment, such as hot press test [18], indentation load relaxation (ILR) test [19], constant loading rate (CLR) test [11], impression test [20–22], constant load (CL) test [17,23] and constant strain rate (CSR) test [14] have been proposed to acquire creep properties. Readers are referred to reference [14,16] for detailed review of each test methods. Assuming that the steady state of creep rate is achievable during the course of indentation creep test, the relationship between indentation strain rate and hardness is established upon the power law equation:

$$\dot{\varepsilon}_i = A_0 H^n \quad (3)$$

where A_0 is a material constant and n is the creep stress exponent. As such, n is defined by:

$$n = \frac{\partial(\ln\ \dot{\varepsilon}_i)}{\partial(\ln H)} \quad (4)$$

The analysis described above has been applied in considerable works to evaluate creep properties. However, the fundamental understanding of the creep mechanism, especially the microstructural evaluation of the creep process under an indenter is very much lacking. The most apparent consequence is that the appreciable discrepancies in the measured properties between the indentation creep and the conventional uniaxial tests. For example, Mayo and Nix [11] measured the stress exponent, n, to be 11.4 for large grain Sn and 6.3 for small grain Sn through the CLR test, while the n values were 7.6 as reported in conventional creep tests [24].

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The reason for the discrepancy in the testing results by indentation and conventional creep test methods lies in the intrinsic difference in the deformation mechanics. In a conventional uniaxial creep test, the deformed material conserves the volume throughout the course of creep deformation. However, in an indentation creep, the deformed volume of material underneath the indenter is continuously expanding to encompass previously undeformed material. The newly deformed materials located near the elastic–plastic deformation boundary are undergoing primary creep process, while those at and close to the centre of the deformed zone are being deformed at a different creep rate. Under such circumstances, the response from an indentation creep test always compromises the contributions from the transient stage as well as steady state, or even post steady-state stages. The objective of present study is to clarify the ambiguous perception in the indentation creep analysis, and to provide insights of an indentation creep process and its effectiveness in interpreting the creep properties of the materials.

Among the available indentation creep techniques mentioned previously, CL and CSR tests are most widely used to probe creep properties of materials due to their relatively simple experimental procedures. Some test results were found comparable with the ones by conventional creep test. However, the direct comparison of the two methods is scarce [14] especially on the same materials. In fact, most of the studies based on the CL method showed inconsistency in the selection of the time frame within the constant load hold segment for the stress exponent determination. The primary assumption made in the CL test is that the indentation steady-state can be achieved with prolonged holding process, and therefore, the stress exponents are extracted from the linear portion at the rear end of the holding segment. Using such analysis, the reported results are unavoidably subjected to the changes of the initial loading rate as well as the total duration of the holding process. Based on these perspectives, it is one of the tasks of this present study to evaluate the significance of achieving (or not achieving) steady state in the CL test on the measured properties. Both CL and CSR tests were used to analyse single crystal Al and polycrystalline Sn samples. The effect of the grain size on the indentation creep behaviour of the polycrystalline Sn was also presented and discussed.

2. Theory

2.1. Conventional creep analysis

Creep refers to the time dependent deformation of a material at constant stress level lower than yield stress. Conventionally, a creep experiment is conducted with tensile experiment by controlling either the stress or the strain rate to be constant. While monitoring the corresponding strain rate or stress output from the creep experiment, the steady-state is achieved when both strain rate and stress are constant as a result of microstructure balance achieved between strain hardening and relaxation. The steady state strain rate is subjected to factors including steady-state stress, grain size, etc., and can be described by [25]

$$\dot{\varepsilon} = A\left(\frac{D}{\Omega^{2/3}}\right)\left(\frac{\sigma}{G}\right)^{n'}\left(\frac{\sigma\Omega}{kT}\right)\left(\frac{b}{d}\right)^{m'} \quad (5)$$

where $\dot{\varepsilon}$ is steady-state strain rate, A is dimensionless constant, D is diffusivity, Ω is the atomic volume involved in the diffusion, σ is steady-state stress, G is shear modulus, k is the gas constant, T is the temperature in absolute scale, b is the Burgers vector, and d is the averaged grain size. The term $(\sigma/G)^{n'}$ represents the stress dependence of the creep rate. The parameter $(b/d)^{m'}$ demonstrates the grain-size dependence of the creep rate. For the diffusional creep including Nabarro–Herring and Coble creep, the creep rate is grain size sensitive where m' is 2 and 3, respectively. In such cases, the stress has less effect on creep rate since n' is equal to 0. However, for power-law creep, the stress plays a major role in determining the creep rate and n' is found to be in the range of 2–6 for most of the pure metals. Unlike Nabarro–Herring and Coble creep, power-law creep occurs at higher stress range, and the grain size has much less impact on the creep rate and the m' is 0 for most pure metals.

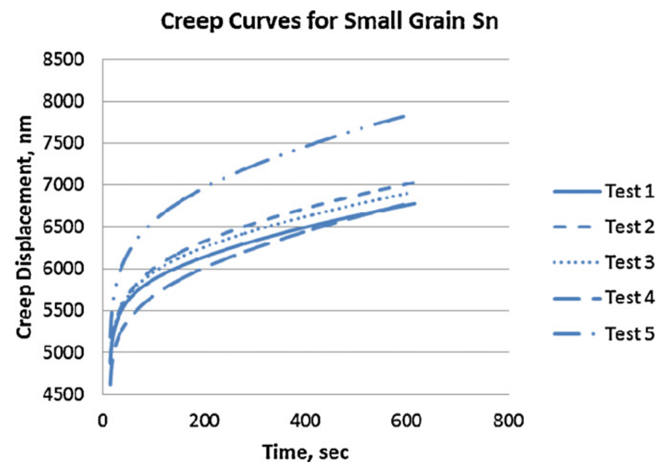


Fig. 1. Five indentation creep curves of the small grain Sn samples.

2.2. Constant load (CL) test

The procedures involved in a CL test include loading the indenter at a high loading rate and then holding the load for certain period of time [17,26]. The strain rate and stress are calculated from each individual data point from the holding segment. As an illustration, creep displacement curves from indenting on polycrystalline Sn sample during the holding segment are shown in Fig. 1. Empirically, such curve can be described by [15]:

$$h(t) = h_i + \beta(t - t_i)^m + kt \quad (6)$$

where h_i and t_i are displacement and time at the starting of holding segment; β, m, and k are fitting parameters. It resembles the creep curve from a uniaxial tensile test, which can be considered as superimposing *transient creep*, i.e., term $\beta(t-t_i)^m$, and *viscous creep*, i.e., kt, after the sudden strain, h_i, resulting from applying the load [27]. As described previously, the strain rate, $\dot{\varepsilon}$, is defined as creep rate divided by instantaneous depth (h). By differentiating equation (6), the strain rate can be expressed as

$$\dot{\varepsilon} = \left(\frac{dh}{dt}\right)\cdot\left(\frac{1}{h}\right) = [\beta\cdot m\cdot(t - t_i)^{m-1} + k]\cdot\left(\frac{1}{h}\right) \quad (7)$$

In the process of creep, the strain rate experiences transition from an initial fast descent stage to a relative stable range. In the meantime, the stress also decreases as the contact area between the indenter and material increases with time. As such, series of strain rate and stress pairs can be obtained from single indentation holding process and the stress exponent, n, is then calculated from Eq. (4).

2.3. Constant strain rate (CSR) test

In CSR test, the constant strain rate is maintained in the load increment stage. Lucas and Oliver [14] demonstrated the means of maintaining an indentation strain rate through controlling the load rate. The concept is developed from equation:

$$H = \frac{P}{A_c} = \frac{P}{ch^2} \quad (8)$$

where c is constant that depends on the geometry of the indenter (24.56 for the perfect Berkovich indenter). By rearranging and differentiating, the strain rate is given by:

$$\frac{\dot{h}}{h} = \frac{\dot{P}}{2P} - \frac{\dot{H}}{2H} \tag{9}$$

where $\dot{h}/h$ is indentation strain rate $\dot{\varepsilon}_i$, $\dot{P}$ is loading rate, $\dot{H}$ is hardness changing rate. The constant strain rate is realized through controlling $\dot{P}/P$ as constant once the steady state value of H is reached.

3. Experiment methodology

3.1. Materials

Pure Sn ingot with purity >99.99% was commercially obtained. Large grain (LG) sample was prepared by annealing the ingot at 100 °C for 3 h followed by oven cooling. To prepare small grain (SG) sample, Sn ingot was first melted at 250 °C for 1 h, followed by quenching in liquid N_2. Both Sn samples were mechanically polished using diamond paste till 1 μm followed a final polishing with 0.1 μm silica suspension. Single crystal Al was provided as a standard calibration sample by Agilent Corporation for the Nanoindenter XP® system. The orientation of the exposed plane of the Al crystal is determined to be (1 1 0) by X-ray diffraction (XRD) analysis.

3.2. Nanoindentation analysis

An Agilent Nanoindenter XP® system is used for this study. The load is applied through an electrical magnetic coil controller attached on top of the indenter shaft. The indenter assembly is supported by a leaf spring which is attached to the fixed frame. Vertical movement of the indenter shaft is monitored by a capacitive gauge. The indentation experiments were performed with a Berkovich indenter, which is a three-faced pyramid diamond with radius about 50 nm at the apex. The roundness at the tip area is accounted by calibrating the tip with a standard material (normally fused silica) with known property. The contact project area, A, can be fitted into a polynomial function of the contact depth, h_c [19]:

$$A = f(h_c) = a_0 h_c^2 + a_1 h_c + a_2 h_c^{1/2} + a_3 h_c^{1/4} + \cdots + a_8 h_c^{1/128} \tag{10}$$

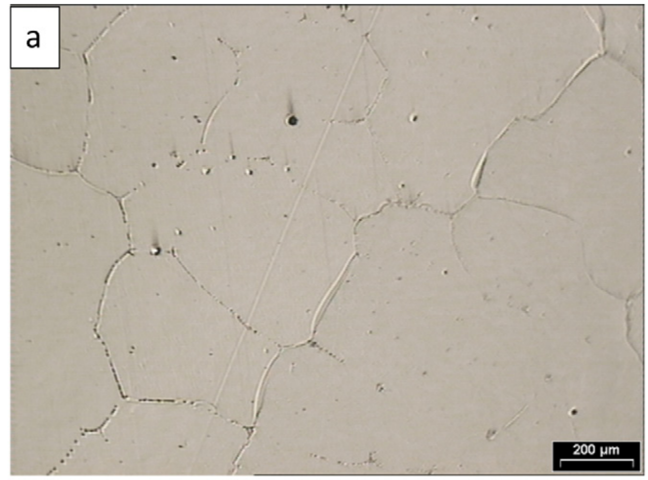
where a_0 is the leading term, which describes a perfect Berkovich indenter; a_1 to a_8 are constants describing deviations from the Berkovich geometry due to blunting at the tip. Two test schemes, namely constant load (CL) and constant strain rate (CSR) tests, were used to evaluate the creep properties of the materials of interest.

3.3. Constant load (CL) test

The materials were loaded to 100 mN in 15 s; the maximum load was held constant for 600 s followed by unloading at the same rate as loading; this is followed by another 60 s holding period at 10% of the maximum load. This lower holding process is to monitor the thermal drift of the material and testing setup. The drift correction is applied to all the data points collected throughout the testing process. At least 5 indents were performed on each sample.

3.4. Constant strain rate (CSR) test

The indenter is pressed into material at constant strain rate by controlling $\dot{P}/P$. 12 different strain rates ranging from 10^{-4} to 0.5 s^{-1} were used in the present study. The maximum depth approached into all samples is 4 μm. The indenter is withdrawn from surface at a rate equivalent to the maximum loading rate. At least 5 data points are recorded and averaged for same test condition.



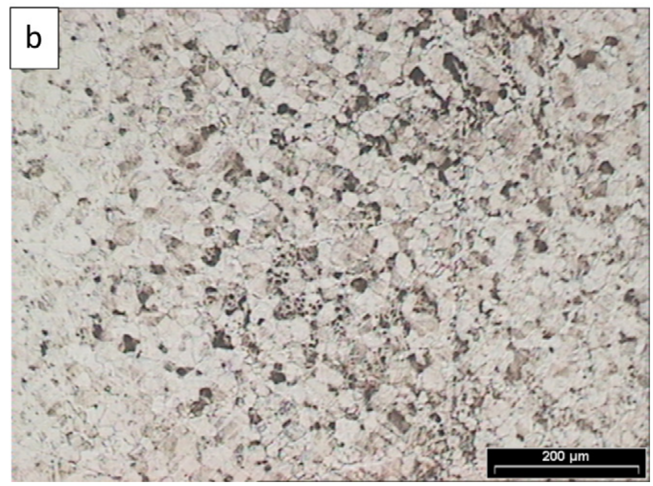


Fig. 2. Optical image of polished Sn samples: (a) big grain Sn prepared by annealing and (b) small grain Sn prepared by melt-quench.

4. Results

Single crystal Al and polycrystalline Sn samples were investigated by CL and CSR methods. The metallurgically prepared surfaces of the Sn samples with two different grain sizes are shown in Fig. 2(a) and (b). The average grain size was approximately 317 μm and 9 μm for the large and small grain samples, respectively. The locations of the indents on large grain sample were chosen to be at the centre of each grain, whereas for the small grain sample, one indent normally covered several grains.

Fig. 3 shows the typical creep displacement responses of the three samples with CL method as a function of creep time with 0 s representing the beginning of constant load hold segment. The materials were deformed at 100 mN. All curves have been translated to the position where the beginning of the creep displacement is zero for easy comparison. The single crystal Al showed the smallest amount of creep within the 600 s of holding period. For Al, it is understandable that insufficient thermal activation is available to assist diffusion at room temperature, since its melting temperature is high. Under the same condition, Sn crept much faster than Al. The amount of creep of the three materials is summarized in Table 1. Fig. 3 also shows that the LG and SG Sn alternatively led the creep rate in the course of creeping. The LG sample crept slightly faster at the beginning of the holding process, but SG sample took over

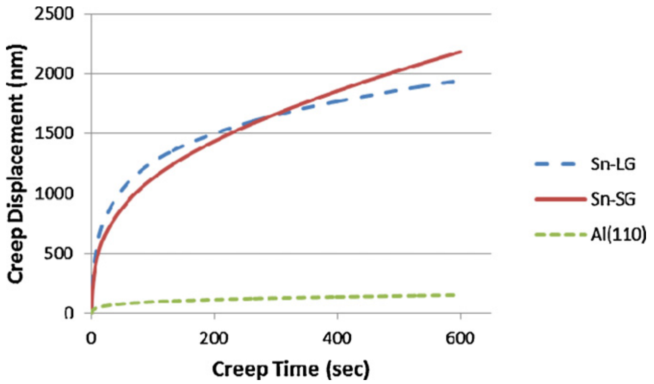


Fig. 3. Creep displacement as a function of time for single crystal Al, large and small grain Sn samples.

Table 1
Indentation response of single crystal Al and polycrystalline Sn samples.

	Al	Sn (LG)	Sn (SG)
Creep depth (nm)	149.82 ± 5.73	1946.96 ± 179.28	2186.62 ± 341.75
n_1 (from CL test)	–	9.90 ± 0.80	9.87 ± 1.06
n_2 (from CL test)	56.02 ± 4.72	8.11 ± 1.93	4.07 ± 0.32
n (from CSR test)	43.64	10.42	9.58

at late stage. The slightly larger deformation found in small grain (SG) Sn could be related to the supplementary role of grain boundary (GB) in accommodating creep deformation by GB rotation and sliding.

Fig. 4 shows the strain rate vs. stress plots of the three samples in double logarithm scale obtained from CL test. The creep process started from the upper right end of each curve where both stress and strain rate are maximum. The gradient of the curve represents the stress exponent, n. It is noticed that the strain rate of single crystal Al responded linearly with stress except slight deviation at the beginning of creep. The deviation is believed to be attributed to the sudden transition from the high deformation rate at loading segment to lower rates at the holding segment. The stress exponent for Al is shown as high as 56.02, which is typical for work-hardening type of deformation for high melting temperature alloys. The accumulated dislocation in the lattice prevents the material to deform further.

As shown in Fig. 4, the stress exponents of *the strain rate–stress* curves of the SG and LG samples were similar at the beginning of creep process. They were found to be 9.87 and 9.90, respectively

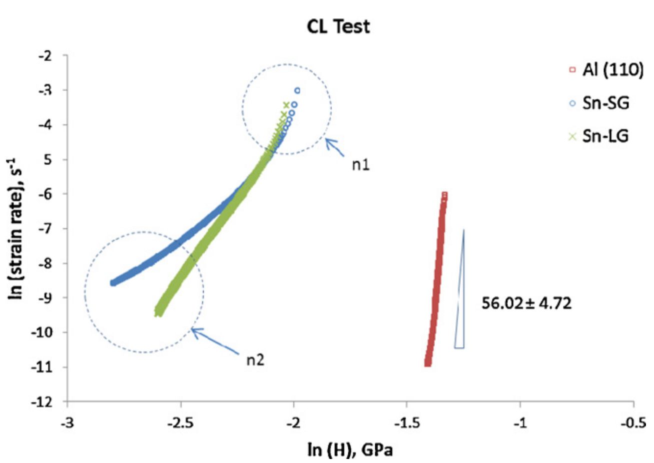


Fig. 4. Double logarithmic plot of stress–strain rate relation for single crystal Al and Sn samples with large and small grain sizes obtained from CL test.

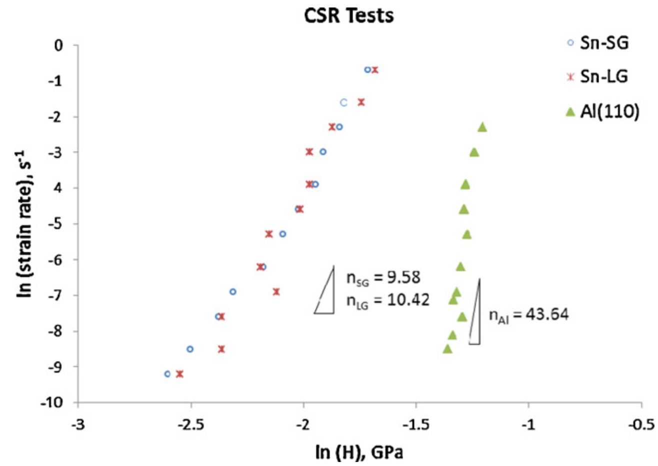


Fig. 5. Double logarithmic plot of stress–strain rate relation for Sn and single crystal Al obtained from CSR test.

in the first 15 s of holding. The values of exponents are summarized in Table 1. n_1 denotes the slope of the linear fit in the first 15 s (stress level around 120 MPa) after the start of holding process. The slopes of both curves progressively decreased with holding time. n_2 denotes the stress exponent obtained from linear fitting of the creep data in the last 300 s of the holding process. Within the time frame studied, n decreased from 9.87 to 4.07 for SG sample and 9.90 to 8.11 for LG sample. The n values obtained for LG Sn sample were close to those reported for dislocation climb controlled mechanism [24]. The little variation of the stress exponents during the load holding process in this sample implied that the operational rate controlling mechanisms has not changed. The low stress exponent of SG Sn found at the rear end of the holding segment is close to those reported for grain boundary related diffusional creep of Sn based solder alloys [4]. We believed that the new rate controlling mechanism such as diffusion in lattice (Nabarro–Herring creep) could start to operate during the end of the constant load-hold process in SG samples.

In addition to the differences in n curve as shown for large and small grain Sn, it is noticed that the n values obtained among large grain samples varied much more than that in small grain ones as shown in Table 1. As the indentations were chosen to be done at the centre of different grains on LG samples, the variation in the results is likely caused by the influence of the crystallographic orientation. In SG sample, one indent normally covers several grains, giving the consistency of the results as an average.

Fig. 5 plots the strain rate as a function of stress from the CSR tests for single crystal Al, SG and LG Sn samples. Linear relationship of the logarithmic stress–strain rate curve was found for all three samples. A stress exponent of 43.64 is identified in the stress range between 0.257 GPa and 0.30 GPa for single crystal Al. SG and LG Sn samples show n values of 9.58 and 10.42, respectively. The discrete data points represent averaged results of at least 5 measurements with the same testing conditions. It is interesting to note that, in contrast to the CL test result, the grain size did not show much effect on the Sn samples in the CSR test. The detailed comparisons of the two methods are discussed in the following section.

5. Discussions

5.1. CL and CSR tests

In the current work, we found that both CL and CSR methods are able to capture the indentation stress and strain rate relations in certain stress ranges. As a nature of the indentation test, the deformed material under the indenter responsively expands its volume to account for the continuous enhancement of the external force. For one particular material, the deformed volume is adjusted to accommodate the variation of the force so that the resulting stress applied on the material, unlike the one in a uniaxial creep test, would not have much room to vary under such test conditions. Even with different loads, the experiment has a limited capacity to cover a broader range of stress for the creep studies. As a consequence, limited range of stress is accessible by the CL test. On the other hand, CSR test provides the opportunity to define the strain rate without constraint from the material. In such test, we are able to evaluate the material creep response in a larger stress range by varying the strain rate. As in the present CL study, the stress ranges, as a response of the external constant force, were 61–137 MPa and 74–130 MPa for SG and LG Sn, respectively. However, a larger stress range from 74 MPa to approximately 180 MPa was obtained from the CSR study. Even higher stress range can be reached by varying the user-defined strain rate if necessary.

In addition to the difference in the accessible stress range from the two test methods, it is noticed that the load–displacement curve at the holding segment of CL test are less repeatable for multiple measurements of the same sample (e.g. Fig. 1). It is known that the CL test consists of two sequential processes, namely, fast loading segment followed by the constant load holding process. The analysis based on the holding process is thus unavoidably subjected to the materials response from the previous loading process. In the present study, the time to achieve 100 mN was set to be 15 s in order to minimize the possible creep incurred in the loading process. However, the material is believed to undergo a large amount of stress/strain rearrangement because of the sudden deformation, where materials are expected to encounter certain uncertainties in the short time frame. Therefore, the total displacements achieved in this 15 s are normally found to be diverse for multiple measurements (as shown in Fig. 1). Due to the fact that the initial displacement at the start of the creep process (h_i as in Eq. (5)) is one of the inputs to generate strain rate from the curve fitting process, variation of the h_i would lead to inconsistency of the strain rate as well as stress exponents measured from a CL test. On the contrary, the strain rate is a direct input for the CSR experiment, and hence the measurements are not subjected to any pre-conditions of the materials.

5.2. Indentation steady state

In a conventional creep process, the material experiences a transient period before the steady-state creep is achieved. In the transient period, strain hardening effect dominates and the deformation rate is very high. The hardening effect diminishes with strain rate slowing down, whereas the dynamic relaxation increases the rate and finally catches up with the hardening rate to achieve a steady-state stage. However, in the indentation CL test, the materials inside the boundary of the expanding cavity always start with the transient creep where hardening dominates which leads to a higher n. The microstructure evolution in the deformed volume is believed to be the combination of the slow rate deformation at the near core region and the transient creep near the boundary. The rate of boundary expansion steadily slows down towards the end of holding segment, which further imposes uncertainties for one to determine the time frame for stress exponent calculation. Carefully examining the stress and strain rate variation during the constant load holding process, as shown in Fig. 6, it is noticed that both quantities go through sudden decrement at the beginning the holding process, followed by a relatively gentle slope. However, neither the stress nor the strain rate truly achieves constant value throughout the holding process. Such situation contradicts the assumptions for applying conventional creep analysis to the indentation data. It thus imposes great ambiguity to apply the analogous conventional creep analysis, which is based on the steady-state process, to the indentation CL test results.

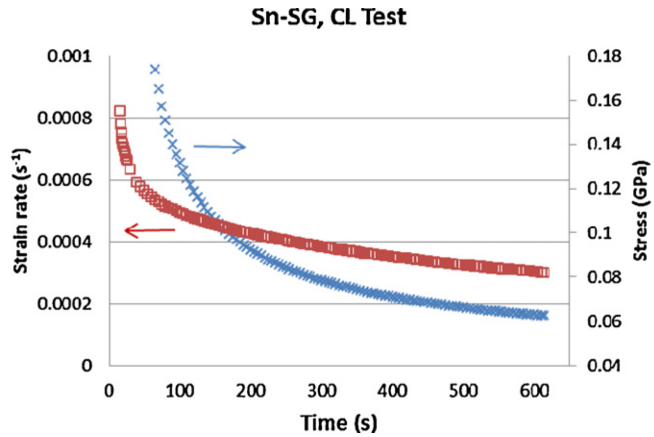


Fig. 6. Stress and strain variation with time in the holding process of a CL test on Sn-SG sample. Both quantities do not achieve steady-state in the course of the time frame studied.

In contrast to the CL test, in a CSR test, the volume of the material involved in deformation expands at relatively high rate. Both hardening and recovering processes are radiating outwards from the deformation core at constant rates, making it possible to achieve a steady state. In the CSR test, the strain rate is controlled to be constant. The responding stress of the LG Sn sample under several strain rate conditions are displayed in Fig. 7. It is noticed that after approximately 2 μm, the steady stress state was able to be achieved, and thus, both stress and strain rate are constant.

5.3. Grain size effects

Another prominent difference between the CL test and the CSR test lies in their responses to the grain size. Both single crystal Al and LG Sn sample showed insignificant difference in the stress exponents measured by the two test methods. However, the SG Sn sample gave significant different creep rates at relatively lower stress range from the two testing methods. From the CSR test, the strain rate and stress demonstrated power law relation for both LG and SG Sn samples. It is known that power-law creep is insensitive to grain size variation as m' is 0 in Eq. (5). Therefore, it is reasonable to obtain fairly similar strain rate response of the two grain-sized samples in the stress range tested by the CSR method,

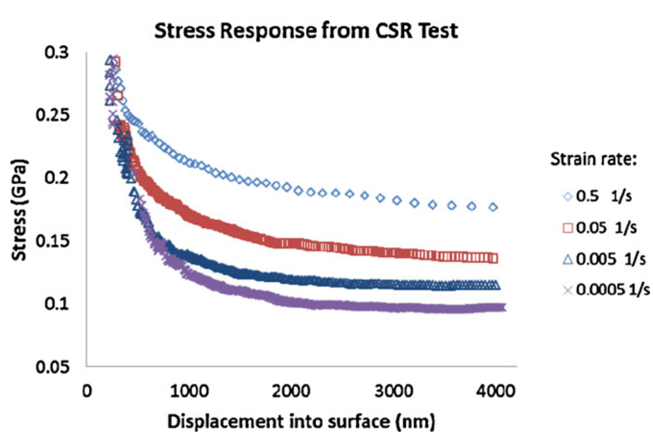


Fig. 7. Stress response of LG Sn sample from CSR test. Steady-state stress can be found after 2 μm of the indentation depth.

which implies that with this method, power-law creep is the only mechanism in operation in current stress range. On the other hand, the stress exponent has deviated to a lower value with deceasing stress from the CL testing. It could be due to grain-size sensitive mechanisms, such as Nabarro–Herring creep, that participates in the deformation of SG Sn sample at such a lower stress level. However, comparing the stress and strain rate behaviour in the same stress range from CL and CSR tests, it is noticed that only power law creep is functioning in CSR test, but multiple mechanisms may exist in CL test. We could not rule out other factors that may have contributed to the deviation of n to a lower value in SG sample, for example, the steady state may not be achieved at lower stress range. In the case of Al, since the test temperature is well below $0.5\,T_m$, the creep mechanism is not expected to change. Both test methods provide comparable n values for this material.

6. Conclusions

Creep properties of single crystal Al and polycrystalline Sn with different grain sizes have been investigated with CL and CSR nanoindentation methods. Through the comparison of these two methods on same materials, the limitation and strength of the two methods have been identified and discussed. Following conclusions can be drawn from present study:

(1) Results from CL test are subjected to the variables from the loading segment, which gives rise to the difficulties in generating repeatable results from CL test. The stress range covered in a constant load hold segment is limited due to the volume of the deformation which is material-determined rather than user-defined.
(2) Genuine steady-state creep may not always be achievable in CL test. On the contrary, CSR test achieves constant strain rate condition through machine control. Steady state stress is then obtained as the output from the experiment.
(3) The insensitivity to the grain size by the CSR test is attributed to the rate controlling mechanism by dislocation climb. The deviation of the stress exponent to a lower value for the SG sample in lower stress range by the CL test could be due to the diffusional creep participating and/or the difficulty to achieve true steady state by this method.
(4) CSR is preferred to obtain power-law creep properties. Due to the unique nature in an indentation experiment, obtaining comparable data in lower-stress creeps, such as Coble and Nabarro–Herring creep, seems to be more challenging than the high stress power-law creep.

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