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The Effects of Creep on Elastic Modulus Measurement using Nanoindentation

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ABSTRACT

During the unloading segment of nanoindentation, time dependent displacement (TDD) accompanies elastic deformation. Consequently the modulus calculated by the Oliver-Pharr scheme can be overestimated. In this paper we present evidences for the influence of the measured modulus by TDD. A modification method is also presented to correct for the effects of TDD by extrapolating the TDD law in the holding process to the beginning of the unloading process. Using this method, the appropriate holding time and unloading rate can be estimated for nanoindentation test to minimise the effects of TDD. The elastic moduli of three materials computed by the modification method are compared with the results without considering the TDD effects.

INTRODUCTION

Load and displacement sensing nanoindentation is a convenient method for determining the mechanical properties such as hardness and elastic modulus of materials in small volumes [1,2]. The testing methodology given by Oliver and Pharr is most widely used [1]. In this method, the elastic modulus is obtained by analysing the unloading segment as a purely elastic recovery process. However, there should also be time dependent deformation (TDD), including indentation creep [3-6], thermal drift, and viscoelasticity. TDD can affect the accuracy of the measured modulus. Since the indentation creep rate decreases with holding time under the maximum load, the effect of indentation creep can be minimised provided a long enough holding time is used. However, as shown in Fig. 1, we found that, in the materials that we have studied at least, the indentation creep is still not diminished but approaches a constant rate even though the holding time is over 600s. In this paper, we propose a method to correct for the TDD effects, and apply it to analyse data obtained from single crystal Ni_3Al, single crystal copper, and polycrystalline Al.

THEORY

In a typical indentation test to measure the modulus of the specimen, the load schedule would comprise a loading segment during which the indentation load is increased to a maximum value, followed by a holding segment when the load is held constant at the peak value, and then an unloading segment to decrease the load. In the Oliver-Pharr scheme, the reduced modulus E_r is given by

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} \quad (1)$$

where S is the initial unloading contact stiffness, and A_c is the contact area.

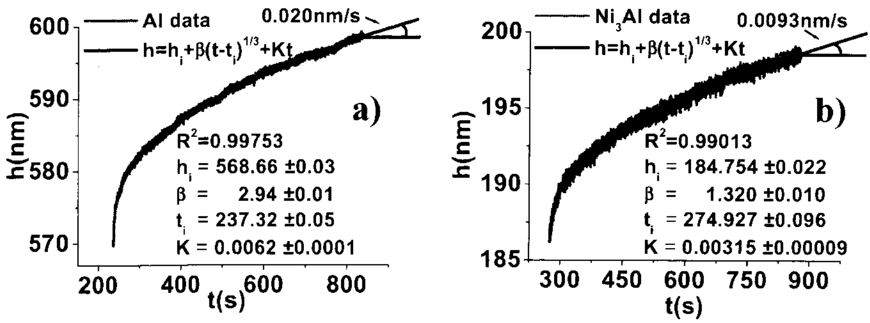

Figure 1. *The displacement-time curve of the holding process after correction of thermal drift for a) Al, load = 2918.1 ± 1.5μN, b) Ni_3Al, load = 4964.6 ± 2.1μN.*

During the unloading process, the elastic recovery process occurs alongside with TDD, so that the actual deformation can be assumed to be the sum of elastic recovery and TDD, i.e.

$$h = h_t + h_e = h^c + h^{th} + h_e \quad (2)$$

where h_t, h_e, h^c, and h^{th} are displacements due to TDD, elastic recovery, indentation creep, and thermal drift respectively, and h is the overall displacement. Here, viscoelasticity is neglected. Eqn. (2) can be differentiated with respect to load P at the onset of unloading to give

$$\left.\frac{dh_e}{dP}\right|_u = \left.\frac{dh}{dP}\right|_u - \left.\frac{dh_t}{dP}\right|_u \quad \text{or} \quad \frac{1}{S} = \frac{1}{S_u} - \left.\frac{dh_t}{dP}\right|_u \,, \quad (3)$$

where the $dh_e/dP|_u$ term is simply the reciprocal of the initial elastic recovery stiffness S, and the $dh/dP|_u$ term is the reciprocal of the observed initial unloading contact stiffness S_u. Here, the $dh_t/dP|_u$ term is the correction term due to the TDD effect that we aim to find. This TDD correction term has to be evaluated from the constitutive law governing material response during the unloading process, but since load and time are both changing during this process, such a constitutive law is not easy to derive.

Bower et al. [7] have performed an analysis on indentation creep by assuming that i) elasticity is neglected, ii) the creep response follows a power law, and iii) the displacement and strain fields are self-similar. The predicted the following relation

$$\frac{P}{\pi a^2 \sigma_0} = A\left(\frac{\dot{h}^c}{a\dot{\varepsilon}_0}\right)^{1/m} , \tag{4}$$

where σ_0, and $\dot{\varepsilon}_0$ and m are material constants in the power law, a is the contact radius, A is only dependent on the shape of the indenter and the material constant m, and $\dot{h}^c$ is the creep displacement rate. We assume here that eqn. (4) applies to the creep component of the displacement during at least the end of the holding stage as well as the initial portion of the unloading stage. Since the load P and contact radius a are continuous at the onset of unloading, eqn. (4) implies that the creep displacement rate is also continuous in the onset of unloading, i.e.

$$\dot{h}^c\big|_u = \dot{h}^c\big|_{hold} \tag{5}$$

where $\dot{h}^c\big|_u$ is the creep rate at the onset of unloading, and $\dot{h}^c\big|_{hold}$ is the creep rate towards the end of holding. Since thermal drift is independent of load, during time dt just after the onset of unloading,

$$dh_t = (\dot{h}^c\big|_u + \dot{h}^{th}\big|_u)dt = (\dot{h}^c\big|_h + \dot{h}^{th}\big|_{hold})\frac{dP}{\dot{P}} = \dot{h}_t\big|_{hold}\frac{dP}{\dot{P}} . \tag{6}$$

Therefore,

$$\left.\frac{dh_t}{dP}\right|_u = \frac{\dot{h}_t\big|_{hold}}{\dot{P}} = \frac{\dot{h}\big|_{hold} - \dot{h}_e\big|_{hold}}{\dot{P}} \tag{7}$$

During the holding process, $\dot{h}_e\big|_{hold}$ can be neglected, putting eqn. (7) into eqn. (3) then gives

$$\frac{1}{S} = \frac{1}{S_u} + \frac{\dot{h}\big|_{hold}}{|\dot{P}|} . \tag{8}$$

From eqns. (1) and (8), the modified modulus can be found. Eqn. (8) suggests that the importance of the TDD effect can be represented by the ratio of the TDD term $\dot{h}\big|_{hold} / |\dot{P}|$ to the elastic term $1/S$, i.e.

$$TDDF = \frac{\dot{h}\big|_{hold}}{|\dot{P}|}S = \frac{\dot{h}^c\big|_{hold}}{|\dot{P}|}S + \frac{\dot{h}^{th}\big|_{hold}}{|\dot{P}|}S = C + Th . \tag{9}$$

Here, $TDDF$ is the TDD factor, $C = \dot{h}^c\big|_{hold} S / |\dot{P}|$ is the creep factor. and $Th = \dot{h}^{th}\big|_{hold} S / |\dot{P}|$ is thermal drift factor. Eqn. (9) shows that the TDD effect can be minimised by decreasing the TDD rate at the end of the load hold, i.e. increasing the holding time, or increasing the unloading rate.

EXPERIMENT

Three materials were used in this study, namely, a single crystal of Ni_3Al, a single crystal of copper, and polycrystalline Al. The composition of the Ni_3Al single crystal is 75 at. % Ni, 16.7 at.% Al, 8.0 at.% Cr and 0.3 at.% B. Prior to nanoindentation, the crystal was annealed at 1250°C for 120 hours. The copper single crystal was 99.99% pure and was annealed for 5 hours at 800°C. The polycrystalline Al was in the as-cast state with grain size approximately 1 to 2 mm. The surfaces of the specimens were electropolished. The indentation surfaces of both Ni_3Al and Cu were both (111) planes. Indentation experiments were performed at room temperature using a Hysitron nanomechanical transducer mounted onto an atomic force microscope (AFM) by Park Scientific Instruments.

Eqn. (8) indicates that to correct for TDD, the overall displacement rate rather than its component creep or thermal drift rate is needed. Eqns. (6) to (8) show that as such, creep and thermal drift can automatically be corrected for without having to know their individual values. Therefore, in eqn. (1), S was modified from the raw displacement data using eqn. (8) directly, after deducting the effect of the machine compliance. However, in eqn. (1), the calculation of the contact area A_c would require displacement data that are net of thermal drift. To evaluate the thermal drift rate, experiments were performed with a low-load holding segment at 15% of the peak load placed towards the end of the unloading stage. During the low-load hold, the drift rate was monitored and was taken to be entirely thermal drift. The holding times of the high-load and low-load processes were both 100s. Because of the relatively long unloading period, the thermal drift calculated this way had only relative accuracy. Nevertheless, the contact area A_c was calculated by subtracting such an estimate of the thermal drift from the overall displacement.

RESULTS AND DISCUSSION

Fig. 1 shows the typical displacement curves recorded during load hold in different specimens. Treating the TDD effects here tentatively as transient creep responses, the following Andrade's law was used to fit the TDD data during holding:

$$h = h_i + \beta(t-t_i)^{1/3} + kt \qquad (10)$$

where β and k are empirical parameters, h_i and t_i are the initial displacement and time at the onset of holding, and they are all obtained by curve fitting. The correction for the contact stiffness can then be obtained by substituting eqn. (10) into eqn. (8).

When the TDD effect is small, the unloading curve can be fitted with a power law [1]:

$$P = A(h-h_f)^m, \qquad (11)$$

where A, h_f and m are empirical parameters, and m is about 2 generally. However, Fig. 2(a) shows that when TDD is large, a "nose" may appear at the onset of unload in the P-h curve. In this case, fitting by eqn. (11) is not satisfactory, and the following equation was applied instead to obtain the correct stiffness S_u at the onset of unloading:

$$h = h_0 + AP^m - BP^n. \quad (12)$$

Here, h_0, A, B, m, and n are fitting constants. The second term, with m about 0.5, represents the elastic recovery, while the third term represents the TDD effect (see Fig. 2(b)).

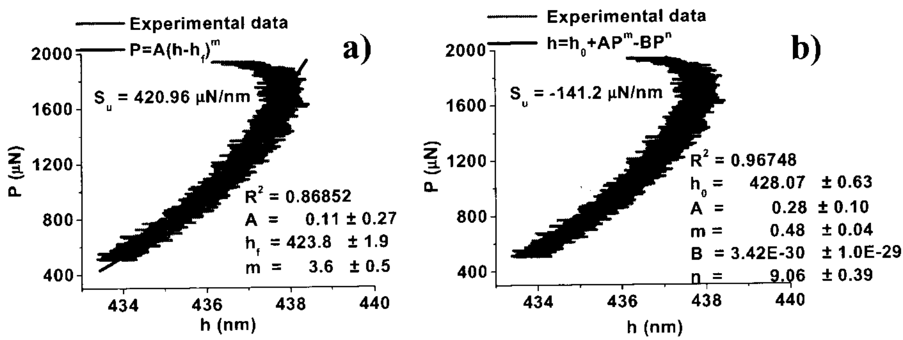

Figure 2. *The curve fitting of unloading for Al with large TDD effect using a) eqn. (11); b) eqn. (12).*

Fig 3 shows the results for the three materials. It can be seen that the modulus calculated without considering creep increases sharply with the creep factor C defined in eqn. (9). The modulus for Al and Cu is dramatically large (over 2000GPa), when the creep factor is larger than about 1.4. After correcting for the creep effect, the value of the modulus tends to be a constant. The corrected modulus is 72.3±7.5GPa for Al, 116.9±11.1GPa for Cu, and 189.9±11.9GPa for Ni_3Al. These compare favourably with the theoretical values of 74.8 GPa for polycrystalline Al, 125.9 GPa for Cu(111) and 201.9 GPa for Ni_3Al(111) [8].

From fig. 3, we can see that the difference of the modulus calculated with and without the correction procedure is small even when the creep factor is as large as 20%. The reason can be seen in fig. 2, which indicates that in the Oliver-Pharr method, the initial unloading stiffness is underestimated because of the poor fit by the power law in eqn. (11). This error fortuitously cancels part of the error introduced by creep. For very large creep factor, the modification method described here will underestimate the modulus a little. This may be because the assumptions leading to eqn. (4) may not be entirely valid.

If creep correction is not to be performed, a very long hold together with a quick unload should be used. However, thermal drift may change a lot during a very long hold. The appropriate holding time to use can be estimated by a criterion such as

$$\left.\frac{\partial^2 h_t / \partial t^2}{\partial h_t / \partial t}\right|_{t-t_i=t_{hold}} \cong 0.5\% \quad \sec^{-1}. \quad (13)$$

The minimum unloading rate to use can be estimated by limiting the creep factor to less than, say, 5%, i.e.

$$|\dot{P}| \geq 20S\dot{h}\big|_{hold}. \quad (14)$$

For the case of Fig. 1, the appropriate holding time is about 200s for both Al and Ni_3Al, and the minimum unloading rates are about 200μN/s for Al and 80μN/s for Ni_3Al.

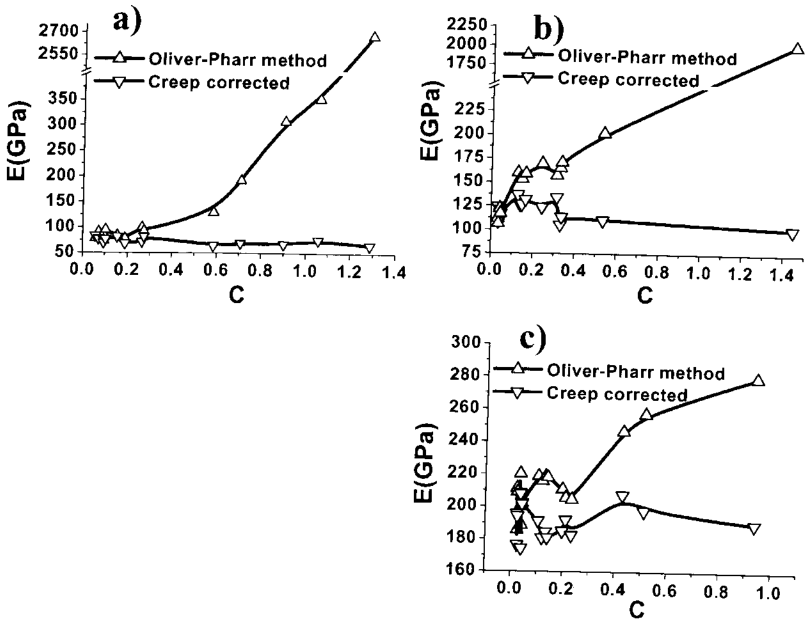

Figure 3. The modulus-creep factor curve for a) Al, b) Cu, and c) Ni_3Al.

CONCLUSIONS

In this paper, the TDD effect is quantified, and a modification method is presented to correct for the TDD effect. Alternatively, if correction is not to be made, the appropriate holding time and unloading rate to use can be obtained by analysing the holding process and the TDD effect.

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