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Loading rate sensitivity of nanoindentation creep in polycrystalline Ni films

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Abstract Nanoindentation creep tests in Ni thin films with 3,000 nm thickness were performed with different loading times (5, 10, 20, 30, and 50 s) under the holding load 5,000 μN and holding time 30 s to investigate the dependence of the indentation creep behavior on the loading rate. The results show that significant indentation loading rate sensitivity on stress exponent and hardness was found, which shows that the stress exponent increases with indentation loading rate. In contrast, the elastic modulus decreases slightly (more or less 1%) due to a longer loading time. Based on the experimental results, we infer that the creep phenomena observed were probably induced by plasticity.

Introduction

The nickel electrodeposited carbon steel sheet is an essential type of material for safeguard in engineering. This material possesses good corrosion resistance, attractive toughness, and excellent plasticity, which offer the potential for advanced structural engineering applications. In addition, the strong adhesion between the nickel films and the substrates provides another advance in practical applications. Nanoindentation is now widely used to measure the mechanical properties, such as hardness, Young's modulus and creep, with the emphasis on films due to their nanometer displacement resolution. As the tip of indenter is getting smaller over the past few decades, the tip radius becomes several nanometers, and the creep properties could be examined by nanoindentation technique [1–3], especially for thin film [4].

The mechanical properties of nanocrystalline materials which exhibit essential strain rate sensitivity have been reported recently [5, 6]. However, only a few reports exist in the literature concerning the loading rate sensitivity (LRS) during nanoindentation procedure. Furthermore, to our knowledge, experimental data on LRS of nanoindentation creep in thin metal films have not been reported until now in detail, and this may provide a new area for the research of indentation creep: Does the LRS affect the nanoindentation creep properties?

In this paper, we investigate the effect of LRS on nanoindentation creep properties in ultra fine grained Ni thin films, which were deposited on carbon steel sheet. The creep properties under different loading rate are discussed by the nanoindentation technique.

Experimental

Sample preparation

A carbon steel sheet of 0.3 mm thickness was used as substrate. A uniform nickel film of 3,000 nm thickness was prepared by electrodepositing method on both sides of the steel sheet. The film was obtained with nickel sulfate electrolyte which was composed of 250 g $NiSO_4 \cdot 6H_2O$, 50 g $NiCl_2 \cdot 6H_2O$, and 35 g H_3BO_3 per liter. Pure nickel was used as the anode. The pH value was adjusted with sulfuric acid to 4.0 at 42 °C. A conventional rotating disc electrode was used for electrodeposition. Before electroplating, pretreatments were necessary to get rid of the impurities. The Ni

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thin films' thickness and average grain size were about 3,000 and 25 nm, measured by SEM in our recent study [7].

Nanoindentation creep tests

The nanoindentation tests were conducted using a Tribo-Indenter from Hysitron Inc. with a three-sided pyramidal Berkovich diamond indenter. The load and displacement resolutions of the machine were 100 nN and 0.1 nm, respectively. The Ni thin films were tested under the maximum load $P_{max} = 5{,}000$ μN with varying loading times 5, 10, 20, 30, 50 s, and the holding time was 30 s. In all cases, at least 10 measurements were repeated, and we took the average that could reduce the noises of the creep curves. Before testing, the indenter tip shape function and the machine compliance were calibrated using a standard sample of fused quartz. Figure 1 shows the typical load–displacement curves obtained under the maximum load 5,000 μN with different loading times on the electrodeposited Ni thin films. Significant creep displacements were observed in all the experiments during the hold time.

Theory and data treatment

The strain rate and the stress could be applied as the following equations in depth-sensing indentation technique:

$$\dot{\varepsilon} \sim \dot{h}/h, \ \sigma \sim P/h^2 \tag{1}$$

where P is the indentation load and h is the instantaneous indenter displacement. $\dot{h} = \mathrm{d}h/\mathrm{d}t$ is calculated by first fitting the h–t curve during holding time with the empirical law [8]:

$$h = h_\mathrm{i} + a(t - t_\mathrm{i})^m + kt \tag{2}$$

where h_i, a, m and k are fitting constants, and t_i is the time when the creep process is started. Moreover, the stress

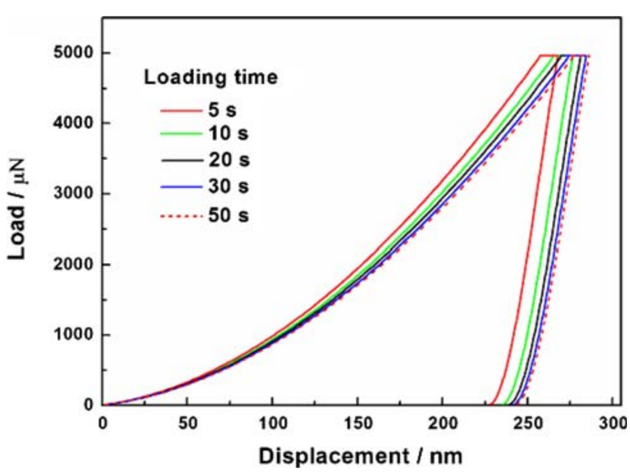

Fig. 1 Comparison of the load–displacement curves for different loading times under the maximum load 5,000 μN with holding time 30 s

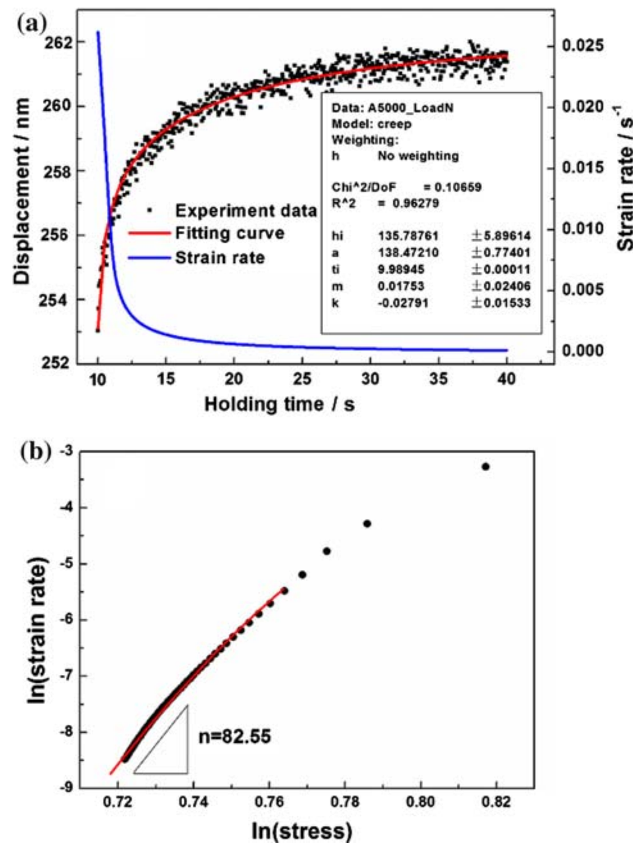

Fig. 2 (**a**) Experimental, fitted, and strain rate curves in Ni films with holding time 30 s. (**b**) The corresponding log (strain rate)–log (stress) plot

exponent n, defined as $n = \partial(\log \dot{\varepsilon})/\partial(\log\sigma)$, is the slope of the double logarithmic plot of $\dot{\varepsilon}_\mathrm{I}$ and σ under isothermal conditions. The fitting protocol in Eq. 2 is found to produce very good fits to most of our results, as can be seen from the example (loading time 10 s) with a correlation coefficient $R^2 = 0.96279$ shown in Fig. 2a. The corresponding creep strain rate $\dot{\varepsilon}_\mathrm{I}$ was calculated by Eq. 1 and also plotted in Fig. 2a. At the beginning of load holding, the penetration deepened at very high strain rate between 10^{-2} and 10^{-3} s^{-1}, being the so-called transient creep [9]. With extending holding time, the strain rate gradually saturated at the order of about 10^{-5} s^{-1}, entering the secondary stage, i.e., representative "steady-state creep" [9]. Figure 2b shows the double logarithmic plot of strain rate vs stress obtained from the data in Fig. 2a.

Results and discussion

The creep curves of Ni thin films under the maximum load 5,000 μN with different loading times are fitted by Eq. 2 and plotted in Fig. 3. The starting points of creep displacement and holding time are aligned for comparison.

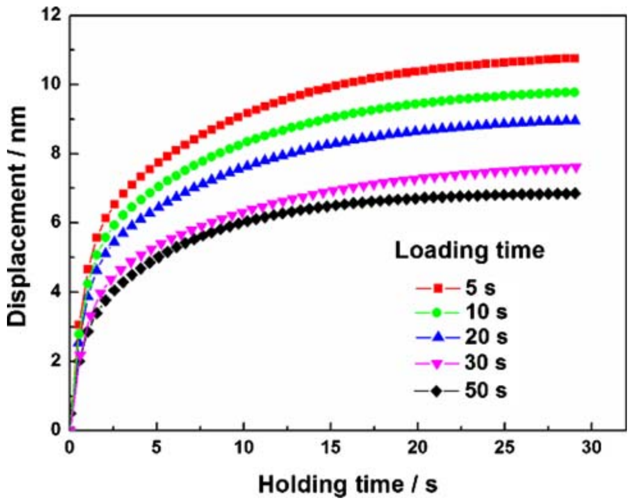

Fig. 3 Comparison of the creep curves with different loading times

The data show that for every experiment there is an initial sharp rise in creep displacement in the early part of the creep segment, followed by a region showing a smaller rate of increase in creep depth. The initial stage in Fig. 3 corresponds to transient creep, and after this initial displacement, the descent of the indenter continues but the rate of descent decreases to attain a steady state value. From Fig. 3 we can also see that with a decrease in loading time (or an increase in displacement rate), the creep deformation is increased; moreover, the trend for the curves for higher loading rates is different from those of the lower loading rates. This may be attributed to two reasons. First, at the lowest loading rate, the strain rate also is lowest and a longer time is needed to reach the holding load, so creep deformation may also occur during the loading time [10], and then the subsequent creep during the holding time will decrease. Second, the dislocation substructure formed beneath the indenter due to the indentation stress may be different at different loading strain rates, and this substructure will certainly affect the subsequent creep behavior [11].

Figure 4a shows the variation of the stress exponent at different indentation loading times in Ni thin films. It can be seen that the stress exponent exhibits a strong dependence upon the indentation loading time or loading rate. The maximum indentation depth in this study was more or less 260 nm, or less than 10% of the film thickness. Thus, the influence of the substrate effects should be minimal [12]. Therefore, it is believable to reveal the real creep properties that the stress exponents can be obtained from the log (strain rate) and log (stress) in Fig. 2. In nanoindentation creep tests with different loading times, we consistently observe strong LRS of stress exponent. The LRS in the stress exponent values of the tested samples may suggest there is a difference in deformation

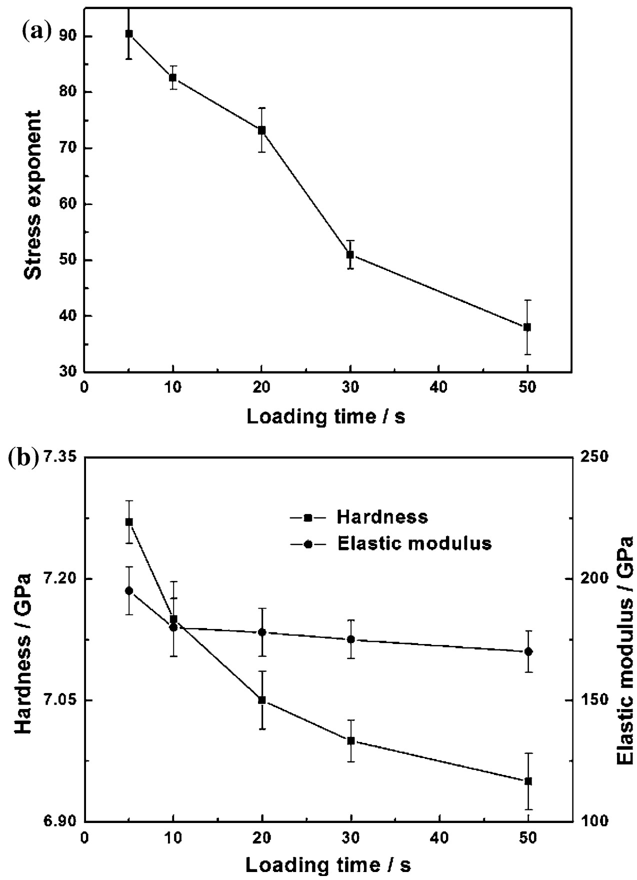

Fig. 4 Variation of (**a**) stress exponent and (**b**) hardness and elastic modulus values with indentation loading time in Ni thin films

mechanism during creep. To understand the phenomena better, we also measured the hardness and the elastic modulus values of Ni thin films, as shown in Fig. 4b. With the initial loading rate increasing, the Ni film has less time to creep to produce a large indent at the end of load-hold. Thus, the hardness increases from 6.95 to 7.27 GPa due to the smaller contact area, indicating a noticeable LRS. In contrast, the elastic modulus decreases slightly (more or less 1%) due to a longer loading time. The increase in the apparent elastic modulus at high initial loading rate is also well understood as an artifact of creep. At a high initial loading rate, significant creep effects will remain during the subsequent unloading, during which the elastic modulus is measured. In the Oliver-Pharr scheme, it is assumed that, during the unloading process, the contact between the tip and the surface is purely elastic. Unfortunately, in many cases, the contact is far from purely elastic. It is well known that creep effects during unloading may cause the contact stiffness to be overestimated [13–15].

The grain size of Ni films tested in this study is much smaller than the indentation size, so the microstructure of each measure point could be considered same. However, the stress exponent changes markedly under different loading conditions. Furthermore, the occurrences of plastic deformation in Ni films were confirmed by the nanoindentation load–displacement (*P*–*h*) curves [16]. Thus, the creep phenomena observed above were probably induced by plasticity. The increased stress exponents with higher holding load were also found by Li and Ngan [8]; however, the increased stress exponent caused by indentation LRS has not been reported and the intrinsic mechanism is not clear and remains to be explored in the future.

Conclusions

Nanoindentation creep tests were carried out on nanocrystalline Ni thin films with average grain size nearly 25 nm. Significant indentation LRS on stress exponent was found which shows that the stress exponent decreases with indentation loading time. The results reveal a common trend of loading condition which affects the nanoindentation creep properties. The studies of the effects of other experimental parameters (such as holding time, frequency imposed on loading) on the nanoindentation creep and mechanical properties of Ni thin films are still in progress.

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