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Modeling of the effects of athermal flow strength and activation energy for dislocation glide on the nanoindentation creep of nickel thin film at room temperature

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Abstract

Nanoindentation creep behaviour of nickel at room temperature has been modeled based on the obstacle-controlled dislocation glide mechanism. Using the model, the effects of two important materials parameters viz. the activation free energy required by dislocation to overcome an obstacle without any aid from external stress, \( \Delta F \), and the athermal flow strength, \( s_0 \), which is the flow strength of solids at 0 K are systematically studied. It has been found that \( \Delta F \) plays a dominant role in room temperature creep properties of nickel. The role of \( \Delta F \) is particularly dominant in determining the time dependent deformation. On the other hand, role of \( s_0 \) is more crucial in the case of instantaneous deformation.

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1. Introduction

With the increasing downscaling of microelectronic devices and the development of microelectro mechanical systems (MEMS), understanding of the mechanical properties of materials at a small volume is becoming very important. Nanoindentation is a unique technique that can provide information on both elastic and plastic properties from a very small volume, typically several tens of nanometers to several hundreds of nanometers across. Studies show that indentation creep i.e., plastic deformation under a constant load can be more significant at submicroscopic than at macroscopic depths [1]. Therefore creep behaviour of components in microelectronic devices and MEMS has been of interest [2,3]. Creep behaviour of a small volume of material can be assessed by nanoindentation. Nanoindentation creep study at low homologous temperatures allows the investigation of low temperature deformation mechanisms in ultra small structures and thin films [4–6].

Indentation creep has been studied by a number of researchers [1,7–13]. Pollock et al. [1] made experimental as well as theoretical investigations on the nanoindentation creep of nickel thin film at room temperature. It is now generally known that at low homologous temperatures, creep is due to dislocation glide controlled by obstacles [14,15]. The theoretical model followed by Pollock et al. [1] is based on obstacle controlled dislocation glide [14]. Pollock et al. [1] found that reasonable agreement could be found between this simple model and experimental data of nanoindentation creep in nickel film. In this model, the activation free energy required...
by dislocation to overcome an obstacle without any aid from external stress, $\Delta F$ and the athermal flow strength, $\tau_0$, which is the flow strength of solids at 0 K are basic parameters for room temperature nanoindentation creep. Pollock et al. [1] adjusted these parameters to fit their experimental data to the theoretical model. The objective of the present work is to make a systematic theoretical study on the effects of these parameters on the creep behaviour of nickel film at room temperature. Nickel film is considered as an example here as it is an important material for microelectro mechanical systems [16].

2. Obstacle-controlled dislocation glide model for indentation creep

The formalism of indentation creep used in the present study is essentially based on that suggested earlier [14]. It considers that dislocation glide is the dominant mechanism in indentation creep. This applies particularly in the present case where indentation creep is considered at room temperature which corresponds to a homologous temperature ($T/T_m$, where $T$ is the working temperature and $T_m$ is the melting point in K) of 0.17 for nickel. According to the deformation mechanism map [14], at such a low homologous temperature, the deformation type is one of low temperature plasticity where the deformation mechanism is dislocation glide controlled by obstacles. Plasticity is in fact a rate process which is temperature dependant. The rate determining factor for the dislocation glide is the interactions of dislocations with obstacles. The interaction can be with other dislocations, with grain boundaries, and with the periodic friction of the lattice itself. In fcc metals, the lattice resistance is extremely low (<10$^{-5}$) and $v$ is the frequency factor. $\Delta G(\sigma_s)$ depends upon the distribution and type of obstacles. Kocks et al. [18] have given a general expression for $\Delta G(\sigma_s)$ as follows:

$$\Delta G(\sigma_s) = \Delta F \left[ 1 - \left( \frac{\sigma_s}{\tau_0} \right)^p \right]^q$$

(4)

$\Delta F$ is the activation free energy required to overcome an obstacle without any aid from external stress and $\tau_0$ is the athermal flow strength which is the flow strength of solids at 0 K. The factors $p$ and $q$ account for the shape and distribution of the obstacles. Frost and Ashby [14] found that, for discrete obstacle a value of 1 for both $p$ and $q$ gives reasonable agreement with experimental data. Combining Eqs. (1)–(4), one gets

$$\dot{\varepsilon} = v_0 \exp \left[ -\frac{\Delta F}{kT} \left( 1 - \frac{\sigma_s}{\tau_0} \right) \right]$$

(5)

where

$$v_0 = \left( \frac{2}{\mu} \right) \left( \frac{\sigma_s}{\mu} \right) \beta b v$$

(6)

$v_0$ can be considered as a frequency term, the value of which was suggested to be $10^6$/s for low temperature plasticity [14]. In an indentation experiment the actual strain rate, $\dot{\varepsilon}$ was shown [19] to be related to the rate of descent, $\dot{\delta}$ of the indenter by

$$\dot{\varepsilon} = C_1 \left( \frac{\dot{\delta}}{\delta} \right)$$

(7)

where $C_1$ is a constant. Replacing $\dot{\varepsilon}$ in Eq. (5) provides

$$\frac{\dot{\delta}}{\delta} = \frac{v_0}{C_1} \exp \left[ -\frac{\Delta F}{kT} \left( 1 - \frac{\sigma_s}{\tau} \right) \right]$$

(8)

Shear stress is related to hardness, $H$ as follows:

$$\sigma_s = C_2 H$$

(9)

It has been shown [4,19] that $C_2 \approx 1/5$ . Hardness is given by

$$H = \frac{P}{C_3\delta^2}$$

(10)

where $P$ is the load applied to the indenter and $C_3$ is a geometrical constant relating $\delta^2$ to the area of the indent. Therefore, putting the value of $\sigma_s$ in Eq. (8) one gets,

$$\frac{\dot{\delta}}{\delta} = \frac{v_0}{C_1} \exp \left[ -\frac{\Delta F}{kT} \left( 1 - \frac{C_2P}{C_3\delta^2\tau_0} \right) \right]$$

(11)

Rearrangement of Eq. (11) gives

$$\ln \frac{\dot{\delta}}{\delta} = -\ln \left[ \frac{C_1}{v_0} \exp \frac{\Delta F}{kT} \right] + \frac{\Delta F}{kT} \frac{C_2P}{C_3\delta^2\tau_0}$$

(12)
The above represents a general model for the indentation creep at low homologous temperatures. Eq. (12) does not have any analytical solution. It was therefore solved numerically to obtain creep curves for nickel loaded under a constant load of 500 μN applied for 1000 s.

3. Results and discussion

Hull and Bacon [20] suggested that the value of $\Delta F$ lies in the range of $0.05\mu b^{3/2}$–$2\mu b^{3}$ depending upon the types of obstacles. This range covers weak obstacles like solute atom to strong obstacles like large precipitates. For nickel, this range equals 36.33–1468 kJ mol$^{-1}$. In the case of pure nickel film, where main obstacles are dislocations, the value of $\Delta F$ is expected to lie in the lower side of the range [14]. Indeed it was found in an earlier work [1] that a value of 52.82 kJ mol$^{-1}$ for $\Delta F$ gave good agreement with experimental nanoindentation results on vapour deposited nickel film. As will be seen in the present work also, higher values of $\Delta F$ yield a flat creep curve which is not consistent with earlier experimental results [1]. Therefore $\Delta F$ values in the range of 36.68–90.0 kJ mol$^{-1}$ have been used in the present calculations.

Frost and Ashby [14] suggested that for medium obstacles like dislocations, the athermal flow stress, $s_{0}/C_{25}$, $l$ is the dislocation spacing. The value of $l$ may vary in a wide range. For annealed metal, $l = 2 \times 10^{-8}$ m [14]. For extremely cold worked metal with high dislocation density, $l$ can be as low as $1 \times 10^{-8}$ m [21]. Using these values of dislocation spacing, the range of value for $s_{0}$ for nickel can be calculated as 0.1–1.97 GPa. This range of $s_{0}$ is used in the present calculations.

Fig. 1 shows a typical nanoindentation creep curve calculated for $\Delta F$ and $s_{0}$ values of 50 kJ mol$^{-1}$ and 1.0 GPa respectively. The curve shows an immediate rise in displacement upon the application of the constant load of 500 μN. This displacement is hereafter referred to as instantaneous displacement. After this initial displacement, the descent of the indenter continues but the rate of descent decreases to attain a steady state value. With the continuation of the load, the displacement increases with time, and this part of displacement is hereafter referred to as time dependent displacement. At any given point in time, the time dependent
displacement is obtained by subtracting the instantaneous displacement from the total displacement of the indenter.

The effect of activation energy for dislocation glide, $\Delta F$, on the creep curve at three different values of athermal flow strength, $\tau_0$, is shown in Fig. 2. It is seen that at all values of $\tau_0$, $\Delta F$ has a great influence on the room temperature creep deformation of nickel. Creep deformation, particularly time dependent deformation increases rapidly as $\Delta F$ decreases. Fig. 3 shows the effect of athermal flow strength, $\tau_0$ on the creep curves at three different values of activation energy for dislocation glide, $\Delta F$.

Data extracted from Figs. 2 and 3 are re-plotted in Figs. 4 and 5 which respectively show the effect of $\Delta F$ and $\tau_0$ on both zero time displacement and time dependent displacement. It is seen that the effect of $\Delta F$ on zero time displacement depends upon the value of $\tau_0$ (Fig. 4(a)). At low values of $\tau_0$, $\Delta F$ has a large affect on instantaneous displacement. However at higher values of $\tau_0$, the effect of $\Delta F$ on instantaneous displacement is not that prominent. One the other hand, $\Delta F$ has a very large effect on the time dependent displacement, which is almost independent of $\tau_0$ (Fig. 4(b)). In Fig. 5(a), it is seen that athermal flow strength greatly influences the instantaneous displacement at all values of $\Delta F$. The effect of $\tau_0$ on time dependent displacement is however less pronounced (Fig. 5(b)). It is thus seen that $\Delta F$ plays a dominant role in room temperature creep properties of nickel. The role of $\Delta F$ is particularly dominant in determining the time dependent deformation. On the other hand, $\tau_0$ is more crucial in the case of instantaneous deformation. Thus while fitting experimental data to

Fig. 3. Effect of athermal flow strength ($\tau_0$) on the creep curve of nickel at three different values of activation energy for dislocation glide ($\Delta F$). (a) $\Delta F$: 42.5 kJ mol$^{-1}$; $\tau_0$: (m) 0.1, (n) 0.33, (o) 0.66, (p) 1.00, (q) 1.33, (r) 1.66 and (s) 1.965 GPa; (b) $\Delta F$: 52.82 kJ mol$^{-1}$; $\tau_0$: (m) 0.1, (n) 0.33, (o) 0.66, (p) 1.00, (q) 1.33, (r) 1.66 and (s) 1.965 GPa ($\tau_0$ increases from top/left to bottom/right).

Fig. 4. Variation of (a) instantaneous displacement and (b) time dependent displacement as a function of activation energy for dislocation glide, $\Delta F$. 
the model, appropriate value of \( \tau_0 \) may first be selected to adjust instantaneous deformation while the selection of appropriate value of \( \Delta F \) will allow the time dependent deformation to be fitted to experimental value.

The model used in the present study was applied to nanoindentation creep of vapour deposited nickel film by Pollock et al. \[1\]. The model was found to show reasonable agreement with experimental data when two key parameters viz \( \tau_0 \) and \( \Delta F \) were adjusted. Value of \( \Delta F \) used by Pollock et al. was 52.82 kJ mol\(^{-1}\). Pollock et al. used a value of \( C_3\tau_0 = 95 \) GPa, where \( C_3 \) is a constant relating \( \Delta F \) to area of the indent. Since the value of \( C_3 \) used by Pollock et al. \[1\] is not available, the exact value of \( \tau_0 \) used cannot be found out.

Both these parameters \( \tau_0 \) and \( \Delta F \) are material dependent. Athermal yield strength, \( \tau_0 \) depends upon obstacle type and density, grain size, alloying etc. \[22–24\]. These in turn depend upon the processing condition of the material. Deposition processes of interest to thin films and MEMS applications are physical vapour deposition, sputtering, electrodeposition etc. which can provide a wide variety of microstructure and obstacle type and density \[8,25–27\]. Thus the values of \( \tau_0 \) and \( \Delta F \) will be different depending upon processing conditions. Using this model, the values of \( \tau_0 \) and \( \Delta F \) can be found out from experimental data which will lead to better understanding of the mechanical properties of thin films/ultra small structures.

Implicit in the present model is the assumption that the nature of the obstacle does not change with deformation. However, with deformation at room temperature the obstacle density do change \[28\]. This may be taken into account in a more sophisticated model.

### 4. Conclusions

Obstacle controlled dislocation glide model has been used to investigate the effects of activation free energy required by dislocation to overcome an obstacle without any aid from external stress, \( \Delta F \) and the athermal flow strength, \( \tau_0 \), which is the flow strength of solids at 0 K on the nanoindentation creep behaviour of nickel at room temperature. Range of values of \( \Delta F \) and \( \tau_0 \) used are 36.68–90.0 kJ mol\(^{-1}\) and 0.1–1.97 GPa, respectively. The room temperature creep deformation of nickel can be divided into instantaneous deformation and time dependent deformation. It has been found that \( \Delta F \) plays a dominant role in room temperature creep properties of nickel. The role of \( \Delta F \) is particularly dominant in determining the time dependent deformation. On the other hand, role of \( \tau_0 \) is more crucial in the case of instantaneous deformation.

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