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To cite this version:
Y. Nishino, S. Asano. Amplitude-Dependent Internal Friction and Microplasticity in Thin-Film Materials. Journal de Physique IV Colloque, 1996, 06 (C8), pp.C8-783-C8-786. <10.1051/jp4:19968167>. <jpa-00254602>

HAL Id: jpa-00254602
https://hal.archives-ouvertes.fr/jpa-00254602
Submitted on 1 Jan 1996

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Amplitude-Dependent Internal Friction and Microplasticity in Thin-Film Materials

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Abstract: Internal friction in aluminum thin films on silicon substrates has been measured between 180 and 360 K as a function of strain amplitude. The amplitude dependence of internal friction in the aluminum films has been evaluated from the data on the film-substrate composite and further converted into the plastic strain of the order of as a function of the effective stress on dislocation motion. The microplastic stress-strain curves thus obtained for the aluminum films tend to shift to a higher stress with decreasing film thickness and also with decreasing temperature. It is concluded that the microflow stress at a constant level of the plastic strain varies inversely with the film thickness at all the temperatures examined.

1. INTRODUCTION

Recently, internal friction measurements have successfully been applied to the study of thin-film materials. Since the thin-film study was introduced and pursued by Berry and Pritchett [1], the temperature dependence of internal friction has been investigated to date [2-6]: e.g., grain-boundary relaxation and point-defect reorientation. A substantially new area for the thin-film study is the amplitude dependence of internal friction, which is caused by microplasticity owing to dislocation motion in the pre-yield range.

As proposed by Nishino et al. [7], the analysis of the amplitude-dependent internal friction in thin-film materials requires two theoretical procedures. Firstly it is necessary to calculate the internal friction in the film separately from the measured data on the composite system, using the constitutive equation [8]. Secondly it is necessary to convert the amplitude-dependent internal friction in the film into typical mechanical responses such as stress-strain curves in the microplastic deformation. This analysis is based on the microplasticity theory [9,10], which is applicable to various internal friction data not only on metals and alloys [11], but also on ceramics [12] and thin-film materials [13,14] where precise mechanisms are still unknown for the amplitude dependence.

In our attempt to study the mechanical properties of thin films, we have investigated the strain-amplitude dependence of internal friction in aluminum films on silicon substrates. The present analytical approach enables one to evaluate the plastic strain in aluminum films as a function of the effective stress on dislocation motion. In particular, the variations in the microflow stress with the film thickness at various temperatures are examined to clarify the thickness effect on the film strength in the microplastic range.

2. EXPERIMENTAL

Aluminum thin films were deposited onto one face of (100)-oriented silicon wafers 0.5 mm thick by magnetron sputtering at room temperature, using an aluminum target with a purity of 99.999%. The wafers were cut into a rectangular shape 10 mm in width and 80 mm in length.

Internal friction in the composite samples was measured between 180 and 360 K in vacuum by means of a free-decay method of flexural vibration with both ends free and at a frequency around 700 Hz in the fundamental resonant mode. After a steady-state vibration, a free-decay curve was measured using a high-speed level recorder. The logarithmic decrement was determined from a slope of the tangent to the smooth envelope of the free-decay curve as a function of the maximum strain amplitude.
3. DATA ANALYSIS

3.1 Evaluation of internal friction in thin films

When the film of thickness $t$ perfectly adheres to one face of the substrate of thickness $d$, the measured internal friction $\delta_{av}$ in the composite sample is a weighted average of the internal friction in the substrate and the film, $\delta_s$ and $\delta_f$ respectively. The relationship among them is given by the constitutive equation [8] and is expressed for the case of free-free flexural vibration as

$$\delta_{av} = \delta_s + \frac{3t}{d} \frac{E_f}{E_s} \delta_f,$$

(1)

where $E_s$ and $E_f$ denote the Young’s moduli of the substrate and the film, respectively. This relation for the free-free beam is in accordance with that for a clamped-free beam [1]. Further the case of imperfect adhesion at the interface between the film and the substrate has been treated by Wuttig and Su [15]. For the aluminum films of thickness $t > 0.1$ μm, $E_f$ can be regarded simply as that of bulk aluminum [4].

The internal friction $\delta_t$ thus obtained for the aluminum films of thickness $t = 0.5$ to $2.0$ μm at 240 K is represented in Fig. 1 as a function of the maximum strain amplitude $\varepsilon_{max}$. It is noted [14] that the calculated values of $\delta_t$ are almost $10^2$ times larger than the measured values of $\delta_{av}$. The magnitude of the internal friction in the films is found to be of the same order as that in bulk polycrystalline aluminum shown by the broken curve. However, the amplitude-dependent part for the films can be seen only at a strain approximately two orders of magnitude higher than that for the bulk. Within the scope of $\delta_t$, the amplitude-independent part decreases as the film thickness decreases, as in the case for the grain-boundary relaxation [5], while the amplitude-dependent part shifts to a higher strain.

Because of the nondestructive nature of the measurements, the testing temperatures can be changed successively for a given sample. Figure 2 shows the internal friction $\delta_t$ in the aluminum film 1.0 μm thick as a function of the maximum strain amplitude $\varepsilon_{max}$ at temperatures $T = 180$ to 360 K. As the temperature is raised, the amplitude-independent part increases and the amplitude dependence appears more remarkably.

3.2 Conversion to stress-strain responses

According to the microplasticity theory based on the friction model [9,10], the amplitude-dependent internal friction $\delta(\varepsilon_o)$ or $\delta(\sigma_o)$ can be converted to the plastic strain $\varepsilon_p$ as a function of the effective stress $\sigma$:

$$\varepsilon_p(\sigma) = \frac{\sigma}{E_f} \left[ \frac{1}{4} \delta(\sigma/2) + \frac{1}{2} \int_0^{\sigma/2} \frac{\delta(\sigma_o)}{\sigma_o} d\sigma_o \right].$$

(2)
For the data analysis [7], the δ₁(ε_{max}) curves in Figs. 1 and 2 are approximated by a power function of ε_{max},

\[ \delta_1(\varepsilon_{\text{max}}) = A \varepsilon_{\text{max}}^n + B, \]

where \(A, B\) and \(n\) are constants. For the flexural vibration, the strain distribution in the film is supposed to be uniform through the thickness but inhomogenous along the length. Then, the intrinsic internal friction \(\delta(\varepsilon_0)\), that would be measured under the ideal condition of a homogeneous strain, is expressed as

\[ \delta(\varepsilon_0) = A K(n) \varepsilon_0^n + B, \]

and the correction factor \(K(n)\) for the strain distribution in the film is

\[ K(n) = \frac{\sqrt{\pi}}{2} \left( \frac{n+4}{2} \right) \left( \frac{n+3}{2} \right) \Gamma \left( \frac{n+3}{2} \right), \]

where \(\Gamma\) is the gamma function. Substitution of the amplitude-dependent term of Eq.(4) into Eq (2) yields

\[ \varepsilon_p(\sigma) = \frac{A(n+2)}{2^{n+2}} K(n) \left( \frac{\sigma}{E_s} \right)^{n+1}. \]

This simple expression is applicable to evaluate the microplastic stress-strain responses from the internal friction data expressed by Eq (3).

Figure 3 shows the relation between \(\varepsilon_p\) and \(\sigma\) for the aluminum films of thickness \(t = 0.5 \text{ to } 2.0 \mu\text{m}\) at 240 K, where the effective stress \(\sigma\) is taken on the abscissa. Although \(\varepsilon_p\) is only of the order of \(10^{-9}\) and as low as 0.01 % of the total strain, the microplastic flow indeed occurs in the films and the plastic strain increases nonlinearly with increasing stress. There is a general tendency that the curves shift to a higher stress with decreasing film thickness, thus indicating a suppression of the microplastic flow in thinner films.

Figure 4 shows the relation between \(\varepsilon_p\) and \(\sigma\) for the aluminum film 1.0 \(\mu\text{m}\) thick at temperatures \(T = 180\) to 360 K. These curves shift to a higher stress as the temperature decreases, and the increase in the stress level on cooling is larger at a lower temperature.

![Figure 3: The plastic strain \(\varepsilon_p\) expressed as a function of stress \(\sigma\) in aluminum films of thickness \(t = 0.5 \text{ to } 2.0 \mu\text{m}\) at 240 K.](image1)

![Figure 4: The plastic strain \(\varepsilon_p\) expressed as a function of stress \(\sigma\) in aluminum film 1.0 \(\mu\text{m}\) thick at \(T = 180\) to 360 K.](image2)

### 4. DISCUSSION

In order to make clear the film strength, we define the microflow stress at a certain value of \(\varepsilon_p\) in Figs. 3 and 4. The microflow stress should be regarded as a measure of the resistance to dislocation motion under a steady-state vibration in the microplastic range. Figure 5 shows the relation between the microflow stress \(\sigma\) at \(\varepsilon_p = 1 \times 10^{-5}\) and the film thickness \(t\) at \(T = 180, 240, 300\) and 360 K. At all the temperatures examined, the microflow stress is inversely proportional to the film thickness. Also, \(\sigma_{\text{film}}\) for the films can be directly related with \(\sigma_{\text{bulk}}\) for the bulk at the same value of \(\varepsilon_p\), and then the thickness effect [14] is expressed as

\[ \sigma_{\text{film}} = \sigma_{\text{bulk}} \left( 1 + \frac{C}{t} \right). \]
where $C$ is a constant that depends on the temperature. In Fig. 5, the large increase in $\sigma$ on cooling below room temperature could be caused by the presence of biaxial tension in the film due to the thermal expansion mismatch between the aluminum film and the silicon substrate [14]. It should be pointed out [13] that the thickness effect on the microflow stress, as shown in Fig. 5, qualitatively agrees with the variation in the macroscopic yield stresses as obtained from substrate curvature measurements [16,17] although the respective stress levels are quite different from each other. This qualitative agreement is surprising if one remembers that microplasticity is controlled only by the movement of dislocations under a steady-state vibration, without accompanying the generation and multiplication of dislocations as in macroplasticity. Further, there is actually no grain-size effect on the microflow stress, provided the grain size is larger or at least equal to the film thickness [14], in contrast to macroplasticity [17] where the film strength is attributed to both the effects of the film thickness and the grain boundary. The increase in the microflow stress with decreasing film thickness could be associated with the bowing of dislocation segments whose ends are fixed at the film surface as well as the film-substrate interface [7]. Since a loop length of the dislocation in the films becomes shorter as the film thickness decreases, the stress required to move the dislocation increases, which is consistent with Eq.(7). Although such a model [18] usually underestimates the macroscopic yield stresses measured for aluminum films by an order of magnitude [16,17], it seems to be the most reasonable one for explaining the film thickness effect in the microplastic deformation.

**Acknowledgments**

The authors are grateful to K. Tanahashi, S. Kenjo and S. Tamaoka for their help with the internal friction measurements. This work was supported in part by the Grant-in-Aid for Scientific Research No.05650617 from the Ministry of Education, Science and Culture, Japan.

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