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Effects of solute Mg on grain boundary and dislocation dynamics during nanoindentation of Al–Mg thin films

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Abstract

Using in situ nanoindentation in a transmission electron microscope (TEM) the indentation-induced plasticity in ultrafine-grained Al and Al–Mg thin films has been studied, together with conventional quantitative ex situ nanoindentations. Extensive grain boundary motion has been observed in pure Al, whereas Mg solutes effectively pin high-angle grain boundaries in the Al–Mg alloy films. The proposed mechanism for this pinning is a change in the atomic structure of the boundaries, possibly aided by solute drag on extrinsic grain boundary dislocations. The mobility of low-angle boundaries is not affected by the presence of Mg. Based on the direct observations of incipient plasticity in Al and Al–Mg, it was concluded that solute drag accounts for the absence of discrete strain bursts in indentation of Al–Mg.

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1. Introduction

Over the last two decades conventional nanoindentation has become a versatile technique for probing the mechanical properties of materials with characteristic dimensions in the sub-micron regime [1,2]. The results obtained from nanoindentation measurements have been limited for a long time to quantitative load–displacement data and ex situ analysis of indented specimens, lacking direct observation of the induced plastic deformation. Recently, direct observation of indentation behavior has been accomplished through the technique of in situ nanoindentation in a transmission electron microscope (TEM) [3,4].

In situ nanoindentation measurements by Minor et al. [5] on polycrystalline Al films have provided experimental evidence that grain boundary motion is an important deformation mechanism when indenting ultrafine-grained thin films. Grain boundary motion in metals typically occurs at elevated temperatures, and is driven by a free energy gradient across the boundary. The boundary mobility is greatly reduced by the addition of solutes [6]. Winning et al. [7] found that both low-angle and high-angle grain boundaries in pure Al move by an external shear stress at temperatures above 200 °C. This type of stress-induced grain boundary motion (known as dynamic grain growth) has been considered by many authors to be the mechanism responsible for the extended elongations obtained in superplastic deformation of fine-grained materials [8]. However, because of the high activation energy required for grain boundary mobility, it is not commonly considered an

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important deformation mechanism at room temperature. The occurrence of grain boundary motion in room temperature deformation of nanocrystalline fcc metals was anticipated recently by molecular dynamics simulations [9] and a simple bubble raft model [10], and confirmed by in situ observations of grain coarsening during indentation of nanocrystalline Al thin films [11] and grain rotation during straining of nanocrystalline Ni thin films [12]. The fact that in situ nanoindentation experiments also showed grain boundary motion at much larger grain sizes (on the order of 0.1 μm) is attributed to the highly inhomogeneous stress induced by the indenter. However, the high purity materials investigated by the in situ nanoindentation technique to date are less attractive for the design of advanced materials. Therefore, in this study we have focused on in situ observations of Al–Mg alloys, probing the influence of solutes on grain boundary motion. This is particularly relevant for understanding temperature effects in superplasticity of ultrafine and coarse-grained aluminum-based materials.

Considerable research effort on Al–Mg alloys has been devoted to understanding the pronounced, repeated yielding that occurs during plastic deformation of these alloys. This phenomenon, known as the Portevin–Le Châtelier (PL) effect or serrated yielding [13], leads to a negative strain rate sensitivity and is caused by interaction between dislocations and mobile solute atoms [14]. In recent years, the PL effect in Al–Mg has been investigated in several deformation modes, including depth-sensing indentation [15,16]. The associated dislocation dynamics have been characterized by in situ straining in high-voltage electron microscopy [17,18] and pulsed nuclear magnetic resonance (NMR) experiments [19].

We have conducted in situ nanoindentation measurements on Al and Al–Mg thin films. Preliminary results from these experiments have shown that solute Mg significantly affects the mobility of high-angle grain boundaries and have also provided direct observation of solute drag on moving dislocations [20]. In this paper, we discuss the influence of solute Mg on the observed indentation-induced plasticity. The observations are qualitatively related to ex situ depth-sensing nanoindentation results from the same specimens.

2. Experimental

The design of the in situ indentation stage for a JEOL 200 CX electron microscope used in this study is extensively described in [4]. Briefly, the single-tilt specimen holder contains a Berkovich-type diamond indenter, which is boron-doped in order to be electrically conductive in the TEM. The indenter is actuated by a piezoceramic tube, which controls the movement

in the indentation direction (perpendicular to the electron beam) as well as the fine positioning on the sample. The material to be indented is deposited as a thin film onto a Si substrate with a sharp wedge-shaped protrusion, which is prepared by bulk silicon micromachining. At the top of the wedge, the film is electron transparent and accessible to the indenter, as illustrated in Fig. 1.

Four Al–Mg films with Mg concentrations of 1.1, 1.8, 2.6 and 5.0 wt% and one high purity (5 N) Al film were prepared by thermal evaporation. The substrate was kept at 300 °C to establish a grain size of the order of the layer thickness, which was 200–300 nm for all specimens. After evaporation, the substrate heating was switched off, allowing the specimen to cool down to room temperature in approximately one hour. Because the solubility level of Mg in Al is 1.9 wt% at room temperature, β' and β precipitates were formed during cooling in the 2.6 and 5.0 wt% Mg specimens. Although the attainable image resolution in the indentation setup was not high enough to resolve these precipitates, their presence was confirmed by strain contrast and distorted grain boundary fringes (Fig. 2(a)), which were not observed in the 1.1 and 1.8 wt% specimens (Fig. 2(b)).

On each of the specimens, 3–4 in situ indentations were carried out with maximum depths ranging from 50 to 150 nm. In order to record the grain boundary phenomena during each indentation, the specimen was

Fig. 1. Schematic of in situ indentation setup.

Fig. 2. Bright-field images of evaporated Al–Mg layers with 5.0 (a) and 1.1 (b) wt% Mg.

tilted into such an orientation that both adjacent grains were in a two-beam diffraction condition.

Ex situ nanoindentation measurements were carried out on the same films away from the wedge. As in the in situ experiments, a pyramidal Berkovich tip was used. Load-controlled indentations were executed to maximum depths of 50, 100 and 150 nm at a targeted strain rate of 0.05 s^{-1} , defined as loading rate divided by load. At this strain rate the indenter velocity during loading was on the order of 2 nm/s, which is comparable to the in situ measurements.

3. Results

3.1. In situ observation of grain boundary motion

To confirm the occurrence of grain boundary movement in pure Al as had been reported earlier [5], we performed several in situ indentations near grain boundaries in the pure Al film. Indeed, significant grain boundary movement was observed for both low-angle and high-angle boundaries. Fig. 3 shows subsequent stages of the loading part of an indentation near a high-angle boundary. After initial contact (Fig. 3(a)) and plastic deformation of grain B (Fig. 3(b)), both grain boundaries outlining grain B moved substantially (Figs. 3(c) and (d)), and the volume of grain B increased accordingly at the expense of the volume of the neighboring grains. By comparing dark-field images taken before

and after the indentation, the grain boundary shifts were measured to be $0.04 \mu\text{m}$ for the left boundary and $0.22 \mu\text{m}$ for the right boundary. Qualitatively, the indentations on pure Al show that the grain boundary motion becomes more pronounced with decreasing grain size and decreasing distance from the indenter to the boundary.

Similar grain boundary movement was never found for high-angle boundaries in any of the Al–Mg specimens, even when indented to a depth greater than half of the film thickness. Fig. 4 shows a sequence of images from an indentation on an Al–1.8wt%Mg layer. At an indentation depth of approx. 85 nm into grain B (Fig. 4(c)), plastic deformation was initiated in grain A either by transmission across or nucleation at the grain boundary. However, no substantial grain boundary movement was observed, indicating a significant effect of Mg on high-angle grain boundary mobility in these alloys. Small grain boundary shifts ($\sim 10 \text{ nm}$) that were measured occasionally can be attributed to displacement of the material under the indenter as a whole, with conservation of grain volume, rather than to actual grain boundary motion.

In contrast to high-angle grain boundaries, the mobility of low-angle boundaries in Al–Mg was found to be less affected by the presence of Mg. Figs. 5(a) and (b) show two grains in an Al–5.0wt%Mg layer, separated by a low-angle tilt boundary. The diffraction patterns for both grains are shown in Fig. 5(c). The grains share the same $\langle 112 \rangle$ zone axis, but are in different two-beam

Fig. 3. Series of bright-field images from an in situ indentation on Al, which is accommodated by movement of the grain boundaries (marked with arrows). The approximate indentation depth h is given in each image.

Fig. 4. Series of bright-field images from an in situ indentation on Al-1.8wt%Mg. No movement of the high-angle grain boundaries is observed.

Fig. 5. (a) and (b) Dark-field images of two grains of a Al-5.0wt%Mg layer separated by a low-angle $\langle 110 \rangle$ tilt boundary; (c) diffraction pattern showing the $\langle 112 \rangle$ orientation of both grains; the cut-off is due to the in situ specimen geometry; (d) dark-field image after indentation.

conditions due to their slight misorientation ($\sim 0.7^\circ$). Fig. 5(d) shows the grains after an indentation to be both in the same diffracting condition as the grain in

(a). At the onset of plastic deformation, the boundary disintegrated rapidly with the end result of the two grains becoming one.

3.2. In situ observation of dislocation motion

The effect of Mg on dislocation propagation is particularly visible during the onset of plasticity. While in pure Al the dislocations instantly spread across the entire grain (i.e. faster than our 30 frames per second video sampling rate) they advance more slowly and in a jerky type fashion in all observed Al–Mg alloys. In Fig. 4(b) for example, plastic deformation is already visible in the left part of grain B, while the right part of the grain is still undistorted. Fig. 6 shows a sequence of images from an indentation in Al–2.6wt%Mg. The arrows mark the consecutive positions where the leading dislocation line is pinned by solutes. Similar pinning of dislocations is visible during plastic deformation of the grains adjacent to the indented grain, as in Figs. 4(c) and (d): as the indenter is pressed into grain B, dislocations are repeatedly arrested in grain A. From these images, the mean jump distance between obstacles is estimated to be of the order of 50 nm. Due to the single-tilt axis limitation of the indentation stage, the orientation of the slip plane relative to the electron beam is unknown; therefore, the measured jump distance is a projection and a lower bound of the actual jump distance.

3.3. Quantitative ex situ nanoindentation

The extraction of mechanical properties from the ex situ indentation measurements was compromised by

the surface roughness and the grain size at shallow depths and the film thickness at deeper depths. Recent numerical studies [21,22] suggest that for a soft film on a hard substrate, the influence of the substrate may not be appreciable until the depth exceeds one half of the film thickness. Still at these relatively high indentation depths, the probed volume was not sufficiently large to give reliable hardness and modulus data. Most of the films showed considerable surface roughness due to cusps at the grain boundaries as illustrated by the scanning electron micrograph in Fig. 7. This leads to an ill-defined contact area during initial loading. Furthermore, the size of the indents was on the order of the

Fig. 7. Scanning electron micrograph of an indent of depth 150 nm in the as-deposited Al–2.6wt%Mg film.

Fig. 6. Series of bright-field images showing jerky motion of dislocations during indentation of Al–2.6wt%Mg. The time from the start of the indentation is given in seconds. Note the presence of a native oxide layer on the surface.

grain size, causing scatter in the indentation results due to microstructural variations.

Nonetheless, interesting qualitative differences in loading response between the specimens were observed. Many of the indentations of the pure Al film showed abrupt displacement bursts during loading up to a depth of around 70 nm, as illustrated in Fig. 8(a). Between the bursts, the slope of the loading curve increases continuously. No such discontinuities were observed in indentations of any of the Al–Mg films. Fig. 8(b) shows loading curves of the Al–2.6wt%Mg film. The initially “soft”

response of the first tens of nanometers is due to the surface roughness as mentioned above. Only in the Al–5.0wt%Mg specimen, having the highest Mg content, was the serrated yielding characteristic of the PL effect observed (Fig. 8(c)).

4. Discussion

4.1. Grain boundary motion

Ideally, direct measurements of the indenter-induced stress at the boundary during our in situ nanoindentations could be made to quantify the observed behavior. However, due to surface roughness, tip imperfections and the complicated specimen geometry, it is difficult to accurately measure or calculate the local stress fields. Comparisons between different measurements are therefore mainly based on indentation depth. The main driving force for grain boundary motion is the high local stress gradient introduced by the indenter [23]. This view is supported by our observation that grain boundary motion is promoted by decreasing grain size and decreasing distance from the indenter to the boundary, both of which lead to a higher stress gradient across the boundary. Presumably, the grain boundary parameters also play an important role in the mobility of an individual boundary, since the coupling of the indenter-induced stress with the grain boundary strain field depends strongly on the particular structure of the boundary. In the indentations on pure Al, extensive movement of both low-angle and high-angle boundaries was observed.

The Al–Mg films used in this study included compositions both below and above the solubility limit of Mg in Al. However, no differences in indentation behavior between the solid solution and the precipitated microstructures were observed. Consequently, the observed pinning of high-angle boundaries in Al–Mg is attributed to solute Mg. The pinning is presumably due to a change in grain boundary structure or strain fields caused by solute Mg atoms on the grain boundaries. Relatively few direct experimental observations have been reported of this type of interaction. Sass and co-workers observed that the addition of Au and Sb impurities to bcc Fe changes the dislocation structure of [1 0 0] twist boundaries of both low-angle [24] and high-angle [25] misorientation. Rittner and Seidman [26] calculated solute distributions at $\langle 1\ 1\ 0 \rangle$ symmetric tilt boundaries with different boundary structures in an fcc binary alloy using atomistic simulations. However, the influence of solutes on the structure of such boundaries has not been experimentally identified. To this end, we are currently conducting a high-resolution TEM study on grain boundaries in Al and Al–Mg [27]. Another effect that may contribute to the pinning of special boundaries is solute drag on extrinsic grain boundary dislocations (EGBDs) as reported by Song et al. [28], who

Fig. 8. (a) Loading curves for indentations in pure Al film, showing discrete displacement jumps; (b) loading curves for indentations in Al–2.6wt%Mg film; (c) serrated yielding during indentation of Al–5.0wt%Mg film.

showed that the dissociation rate of EGBDs in Al alloys is reduced by the addition of Mg. This implies that the indenter-induced deformation is accommodated more easily by these boundaries in pure Al than by those in Al–Mg.

The fact that low-angle grain boundaries were found to be mobile regardless of the Mg content can be explained by their different boundary structure. Up to a misorientation of 10–15°, low-angle boundaries can be described as a periodic array of edge and screw dislocations by Frank's formulae [29]. In such an arrangement, the strain fields of the dislocations are approximated well by individual isolated dislocations and their interaction with an external stress field can be calculated accordingly. Since there is no significant interaction between the individual grain boundary dislocations, the stress required to move a low-angle boundary is much lower than for a high-angle boundary. Low-angle pure tilt boundaries consisting entirely of parallel edge dislocations are fully glissile and therefore particularly mobile. In general, a combination of glide and climb is required to move a low-angle boundary [30].

As a corollary, the structural difference between low-angle and high-angle boundaries also affects the extent of solute segregation. Because solutes generally segregate more strongly to high-angle boundaries [31], the observed difference in mobility may partly be a compositional effect.

4.2. Dislocation dynamics

At the low strains for which jerky-type dislocation motion was observed in situ, solute atoms are the predominant barriers to mobile dislocations, as shown in earlier in situ pulsed NMR experiments [19]. For a review reference is made to [32]. Consequently, the mean jump distance can be predicted by Mott–Nabarro's model of weakly interacting diffuse forces between Mg solutes and dislocations in Al [33]. A calculation of the effective obstacle spacing assuming that the maximum internal stress around a solute atom has a logarithmic concentration dependence yields a value of 30 nm in Al–2.6wt%Mg [19,34]. This is in fair agreement with our experimental observation of a mean jump distance of the order of 50 nm. Indeed also (semi-)coherent β'/β precipitates in Al–Mg alloys can provide significant barriers to dislocation motion. As aforementioned the mean spacing of these precipitates could not be measured very accurately. This has to do with the limited resolution of the microscope combined with the specific indentation stage (Fig. 1). However, from the observed strain contrast in Fig. 2(a) and the low Mg concentration it can be concluded that the mean spacing of the precipitates is larger than the effective solute obstacle spacing, i.e. about twice as much.

This is consistent if we make an estimate of the mean separation of the precipitates based on the solid solubil-

ity of magnesium in Al at RT (1.9%wt [35]). The calculated volume fraction is $f_v = 2.4\%$ for the β phase at 300 K. The mean planar separation, which is a relevant measure for the interaction between a gliding dislocation with a random array of obstacles in its slip plane, is given by [36,37] $\lambda \cong 2r\sqrt{2\pi}/3f_v$, provided the size of the particles is negligible in comparison with their center-to-center separation, i.e. if $\lambda > r$. It is reasonable to assume that the minimum size of the (semi-)coherent precipitates is at least 10 nm to produce sufficient strain contrast in Fig. 2(a). As a result the mean planar separation of the precipitates is calculated 92 nm, i.e. larger than the mean separation between the solutes. Of course in this approach obstacles are assumed to be spherical and as a result we ignore the effect that the precipitation in Al may become discontinuous or continuous depending on the temperature. The latter may generate even a Widmanstätten structure, i.e. needles of Mg_2Al_3 in three crystallographic directions instead of spherical particles. But even in that case the effective separation between needles becomes larger than the effective solute obstacle spacing [38]. Based on the experimental observations in the alloys below and above the solid solubility of magnesium, the strain contrast depicted in Fig. 2 and the theoretical considerations solute atoms are assigned as the main obstacles to dislocation motion.

In situ straining studies in a TEM have related the PL effect to sudden activation, multiplication and coordinated motion of dislocations [17,18]. Such a behavior was not observed in our in situ experiments. Therefore, it is concluded that the conditions for dynamic strain ageing were not satisfied and the observed jerky motion was due to solute drag without appreciable diffusion of solute Mg. It has been established that the PL effect occurs within specific limits of temperature, strain, strain rate and impurity concentration. Based on the theoretical model by Kubin and Estrin [39], Chinh et al. [16] calculated a minimum concentration of 0.62 wt% Mg for plastic instabilities to occur in binary Al–Mg at room temperature. The required strain or equivalent indentation depth at which the instabilities start can be estimated from reported Vickers indentations of bulk Al–Mg [15,16]; at an average loading rate of 0.03 mN/s, the critical depth ranges from 0.10 μm for Al–5.0wt%Mg to 0.19 μm for Al–1.1wt%Mg. Indeed, our ex situ indentation data show serrated yielding starting at approximately 0.10 μm in the Al–5.0wt%Mg specimen as shown in Fig. 8(c). During in situ indentation however, the motion of individual dislocations under the indenter tip cannot be discerned at these large depths due to the geometry of the specimen and the high dislocation density.

The strain bursts observed during loading of the pure Al film (Fig. 8(a)) are separated by regions of Hertzian elastic response. This so-called staircase yielding has been observed in indentation of bulk single crystals

[40] and single crystal and polycrystalline thin films [41]. The observed bursts are attributed to the nucleation of dislocations and their subsequent propagation into the crystal [42]. The absence of these pop-in events during indentation of Al–Mg films (Fig. 8(b)) shows that initial plasticity is significantly affected by solute Mg.

Presumably, solute drag prevents dislocation bursts from propagating through the crystal, i.e. the stored elastic energy is insufficient to push a series of dislocations through the solute atmosphere at constant indentation load. As the load increases further, some of the available dislocations are able to overcome the force associated with solute pinning, thereby allowing plastic relaxation to proceed smoothly. Since there is no collective motion of dislocations as in pure Al, the measured loading response is essentially continuous. This perception is supported by the extensive solute drag observed in situ.

5. Conclusions

The results from the in situ indentation measurements confirm grain boundary motion as an important deformation mechanism in ultrafine-grained Al when it is subjected to a highly inhomogeneous stress field. It is found that solute Mg effectively pins high-angle grain boundaries during such deformation. The proposed mechanism for this pinning is a change in the atomic structure of the boundaries, possibly aided by solute drag on extrinsic grain boundary dislocations. The mobility of low-angle boundaries is not affected by the presence of Mg, which is attributed to their different boundary structure consisting of periodic dislocation arrangements.

Although the conditions for dynamic strain ageing were not satisfied during the in situ indentations, solute drag was observed consistently throughout all Al–Mg films. Based on our direct observations of incipient plasticity in Al and Al–Mg, it was concluded that solute drag accounts for the absence of discrete strain bursts during conventional nanoindentation of Al–Mg.

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