



Strain hardening in bent copper foils

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ABSTRACT

A series of systematic tensile and microbend tests were conducted on copper foil specimens with different thicknesses. The specimens were made of a copper foil having almost unidirectional crystal orientations that was considered to be nearly single-crystal. In order to investigate the effects of slip system interactions, two different crystal orientations relative to the tensile direction were considered in the tests: one is close to coplanar double-slip orientation, and the other is close to the ideal cube orientation (the tensile direction nearly coincides to $[001]$) that yields multi-planar multi-slip deformation. We extended the microbend test method to include the reversal of bending, and we attempted to divide the total amount of strain-hardening into isotropic and kinematic hardening components. In the tensile tests, no systematic tendency of size dependence was observed. In the microbend tests, size-dependent kinematic hardening behavior was observed for both the crystal orientations, while size dependence of isotropic hardening was observed only for the multi-planar multi-slip case. We introduce an extended crystal plasticity model that accounts for the effects of the geometrically necessary dislocations (GNDs), which correspond to the spatial gradients of crystallographic slips. Through numerical simulations performed using the model, the origin of the size-dependent behavior observed in the microbend tests is discussed.

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1. Introduction

Metals exhibit size-dependent mechanical responses both under strain-gradient-dominated deformation (e.g. Fleck et al., 1994; Stölken and Evans, 1998; Haque and Saif, 2003; Shrotriya et al., 2003; Motz et al., 2005; Ehrler et al., 2008; Suzuki et al., 2009) and under uniform deformation (e.g. Uchic et al., 2004; Dimiduk et al., 2005; Kiener et al., 2008) at the micron scale. It is known that the plastic strain gradient in a deformed specimen is compensated by *geometrically necessary dislocations* (GNDs) (Ashby, 1970; Fleck et al., 1994). The complete understanding of how the GNDs affect the mechanical response of metals, however, has not yet been established. The interpretation of the size effect in the absence of the plastic strain gradient is also still an issue.

In the current paper, we focus on the size effects under strain-gradient-dominated deformations. In the literature, two main roles of the GNDs in size effects have been postulated. One is that the GNDs in a strain gradient field should augment the amount of slip hardening in addition to that due to statistically stored dislocations (SSDs). This yields size-dependent *isotropic hardening*. The other is that, according to dislocation theory, the configuration of the GNDs should produce an internal stress that acts as a backstress and this internal stress affects the mobility of dislocations that correspond to the

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magnitude of the slip rate in the context of crystal plasticity. The backstress effect should manifest itself as size-dependent *kinematic hardening*. In experiments with monotonic loading, one cannot determine whether the hardening observed is isotropic or kinematic one. To ascertain the amounts of these two types of size-dependent hardening, we need to perform reverse or cyclic loading tests for strain-gradient-dominated deformations.

In the present study, a series of systematic microbend tests as well as uniaxial tensile tests were conducted on copper foil specimens with different thicknesses. The specimens were made of a copper foil with almost unidirectional crystal orientations that was considered to be nearly single-crystal. To investigate the effects of slip system interactions, two different crystal orientations relative to the tensile direction were considered in the tests: one is close to coplanar double-slip orientation, and the other is close to the ideal cube orientation (the tensile direction nearly coincides to $[0\ 0\ 1]$) that yields multi-planar multi-slip deformation. In the tensile tests, no systematic tendency of size dependence was observed. The microbend test method used is that proposed by Stölken and Evans (1998). In this method, a spring-back phenomenon (an increase in the radius of curvature of the foil specimen) after removing the load plays the primary role in determining the applied bending moment. We extended the method to include the reversal of bending, and we attempted to divide the total amount of strain hardening into isotropic and kinematic contributions. It was found that kinematic hardening is size-dependent both for coplanar double-slip and multi-planar multi-slip deformations. That is, thinner specimens exhibit larger amounts of kinematic hardening. Meanwhile, size dependence of isotropic hardening was observed only for the case of multi-planar multi-slip deformation. In the latter half of the paper, a strain gradient crystal plasticity theory is introduced, which accounts for the two types of the GND-dependent hardening mechanism mentioned above. We conduct simulations of the microbend tests and use the computational results to interpret the experimentally observed size-dependent hardening phenomena. Some aspects of the experimental results can be explained by the simulations, while some drawbacks of the theory are found.

2. Experiments

2.1. Sample and specimens

An oxygen-free copper (99.99% purity) sheet with a thickness of 1.4 mm was fully annealed and then cold-rolled to a thickness of *approximately* 40 μm . Then, the foil was reannealed at 200 °C for 30 min. All mechanical tests in the present study were performed on this oxygen-free copper foil. To measure the crystal orientation, four 10 mm \times 10 mm pieces were cut from the corners of a 100 mm (R.D.) \times 180 mm (T.D.) foil (where R.D. and T.D. stand for the rolling and transverse directions, respectively). The crystal orientations were measured by the electron backscattering diffraction pattern (EBSP) method. The measurement data for the respective four pieces are combined into a single pole figure ($\{1\ 0\ 0\}$ poles) as shown in Fig. 1a. The measurement area for EBSP in each piece was 200 μm \times 433 μm . One of the orientation maps by EBSP is shown in Fig. 1b. All the poles concentrate around a near “cube” orientation, which was rotated clockwise ($\approx 5^\circ$) about the normal direction (N.D.) of the foil and also slightly inclined from N.D. No explicit grain boundary was detected, although scatter of the poles were observed in Fig. 1a. From these data, the foil was considered to be nearly single-crystal. As mentioned above, the material was subjected to a 97%-reduction of the thickness by rolling before annealing. When copper is subjected to such a severe plastic deformation, recrystallization is generally promoted at a rather low temperature (around 200 °C) (e.g. Yamagishi et al., 2006). The preferred orientation of recrystallization is $(1\ 0\ 0)$. Thus, the crystals unidirectionally aligned close to the cube $(1\ 0\ 0)$ orientation observed in our sample are considered to be a consequence of recrystallization. When copper undergoes a smaller thickness reduction, e.g. $\sim 92\%$, such large crystal growth does not occur as reported in Yamagishi et al. (2006).

To investigate the size effect on the mechanical response of the material, the foil thickness was reduced to 22–38 μm by electropolishing at 8 V and a temperature of 15 °C with a solution consisting of distilled water (85 vol%) and phosphoric acid (15 vol%). It was rather difficult to adjust the thickness to a specific value, because we only controlled the duration time of the electropolishing operation. All thickness values indicated in the graphs to be shown are *measured values* (not targeted values).

Effects of oxide layers growing on metal surface have received a bit of attention in the literature. Thickness of oxide layers of Cu_2O on copper surface grows linearly in the logarithm of elapsed time in the atmosphere (e.g. Ohwaki, 2002). We completed the mechanical tests within 2 weeks after finishing the electropolishing process. The thickness of the oxide layer would be an order of 1 nm. The specimens had thickness of about 20–40 μm . Effects of the layers on the mechanical response could be neglected.

After the reduction of thickness, specimens for uniaxial tensile tests and microbend tests were cut from the foil by electric discharge machining (EDM). Two specific crystal orientations relative to the loading axis (the direction of tensile stress in tension and bending) were considered in order to investigate whether the size effect is influenced by change in the combination of active slip systems. Basically, there were inherent limitations on the choice of crystal orientations in the case of the foil specimens because we could only choose an in-plane angle relative to R.D. due to the two-dimensional geometry of the foil.

The first choice was that the longitudinal axis of the specimens was directed counterclockwise at 40° from R.D. Fig. 2a shows the orientations of the tensile direction in stereographic triangle. This inverse pole figure contains all the data for the four pieces cut from the four corners of the foil (they are the same measurement data used for depiction of Fig. 1a).

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