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NANO-INDENTATION STUDIES OF TWINNED MAGNESIUM SINGLE CRYSTALS

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Abstract

Nano-indentation measurements have been performed on {10-12}<10-11> twin and adjacent matrix regions of deformed magnesium single crystals and the hardness values were analyzed by the Oliver-Pharr method. Although the hardness difference between the twin regions and the adjacent matrix was insignificantly small, the hardness values showed orientation dependence regardless of the twins' size and variants. This observation can be interrupted by texture- softening resulting from the lattice reorientation in the twin regions. In contrast, the experimental evidence for the Basinski hardening mechanism in {10-12}<10-11> twins, which is an increase in strength/hardness as a result of dislocation contributions within twin area, was not obtained from this experiment. This presentation provides framework for the discussion of the hardening/softening effect of {10-12}<10-11> twinning on the plastic flow in single crystalline magnesium and quantitative values for hardening parameters used in the crystal plasticity modeling.

Introduction

Hexagonal close-packed (HCP) materials such as magnesium deform by slip and twinning. Basal slip system is the dominant deformation mechanism at room temperature in single crystalline magnesium. However, the basal slip system is insufficient to produce arbitrary deformation of a metal and other deformation mechanism is required. Therefore, non-basal slip systems and twinning becomes important modes of plastic deformation. The {10-12}<10-11> twinning is the predominant twinning mode in magnesium, contributing to the ductility and the strength of the metal, because it provides component of the strain along the c-axis of HCP unit cell and the critical resolved shear stress (CRSS) for the {10-12}<10-11> twinning is the second lowest next to the basal slip. So far, three independent hardening mechanisms due to the {10-12}<10-11> twinning have been suggested: (i) dynamic Hall-Petch effect, arising from the effective grain size refinement and the reduction of the dislocation mean free path due to the twin formations, (ii) the Basinski hardening [1], which predicts an increment in strength/hardness developed as a result of dislocation transmutations within twin area, and (iii) texture hardening/softening, resulting from the lattice rotation in the twin regions (see ref. [2,3] as relevant review papers). However, it is still unclear how much the above hardening mechanisms by the deformation twinning contribute to the overall hardening of the material, even if one considers the hardening behavior of magnesium single crystals.

Nano-indentation is a powerful tool for investigating the mechanical properties and deformation mechanism of materials on the submicron scale, and it has been widely used on a wide range of materials, e.g. thin film materials, bulk materials, and biomaterials over the past few years [4]. The nano-indentation measurements on matrix and twin regions of deformed metals may bring new knowledge concerning the hardening mechanism by deformation twinning, especially for the Basinski hardening and texture hardening/softening due to deformation twinning. {10-12}<10-11> twin regions developed in magnesium single crystals have been reported to be relatively large in size, the order of a few microns in thickness. Therefore, the nano-indentation measurements inside the twin regions should provide information about twin strength without the effect of twin boundaries, because the tip radius of the nano-indentation is very small compared to the width of {10-12}<10-11> twins.

In this study, we deal with the evaluation of the strength of the matrix and the {10-12}<10-11> twin regions in deformed magnesium single crystals by using orientation-imaging microscopy (OIM) and nano-indentation measurements.

Experimental Procedure

Magnesium single crystals of 99.995% purity were used in this experiment. A parent crystal oriented for basal slip was deformed in tension at room temperature, up to a strain of 10%. Smaller secondary samples were cut from the pre-strained parent sample and were subjected to secondary tensile deformation at room temperature. The secondary samples were deformed to a strain of 7% (sample A) and 11% (sample B) and the crystallographic orientation of the samples was such as to induce {10-12}<10-11> twinning during secondary tensile tests. Slip traces and deformation twinning were observed by optical microscopy, and the development of local misorientations and analysis of the type of deformation twins in the secondary samples were characterized by SEM/EBSD measurements. Nano-indentation measurements were carried out on the secondary samples containing matrix and twin regions, with TI 900 Triboindenter™ (Hysitron™) equipped with a diamond cube corner tip with a tip radius of 50nm. During the nano-indentation measurements, the loading force was increased at a rate of 90μN/s to a maximum load of 450μN, held for 2 seconds, and then decreased at an unloading rate of 90μN/s to zero. 30 measurements in 5x6 rectangular arrays geometry separated by 5μm were carried out at one location. The indentation curves that deviate from the behavior of the majority of indentation curves were rejected for the accuracy and reliability of the calibration. The indentation data sets were analyzed by the Oliver-Pharr method [5].

Experimental Results and Discussion

Indentation curves

Fig. 1 shows typical load-depth curves obtained from the nano-indentation measurements on matrix and twin regions of the sample A. Loading and unloading curves for matrix and twin regions exhibit very similar behavior. Displacement bursts, i.e. "pop-in" behaviors marked as arrowheads in Fig. 1 were observed on the *P-h* characteristics of both matrix and twin regions. The phenomenon has been observed in several bulk and thin-film materials and it arises from the set-in of plastic deformation on the defect-free regions and the associated nucleation and propagation of dislocations when the shear stress is equal to the theoretical strength of the indented materials [4].

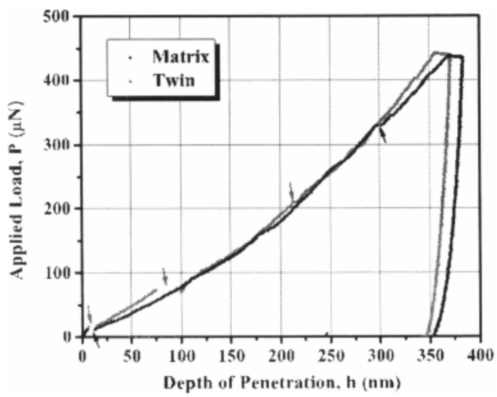

Fig. 1 Typical load-depth curves for matrix and twin regions.

SEM/EBSD analysis and nano-indentation measurements

Fig. 2 shows SEM images of the indentation surface on samples A and B. More complex texture containing various twinning were observed on the surface of Sample B compared to Sample A. Fig. 3 and 4 show inverse pole figure (IPF) maps and 0001 pole figure maps constructed from EBSD data in the location B of Sample A and the locations B and C of Sample B, respectively. SEM/EBSD analysis indicates that all the deformation twins found in the samples were {10-12}<10-11> type.

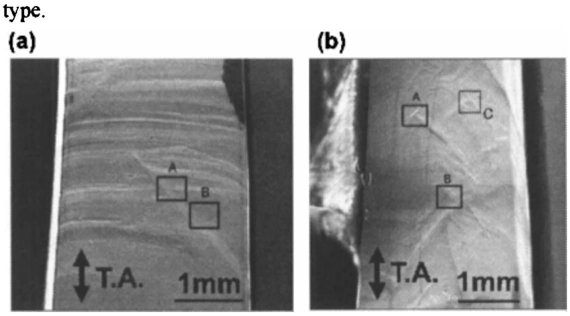

Fig. 2 SEM images of (a) sample A and (b) sample B.

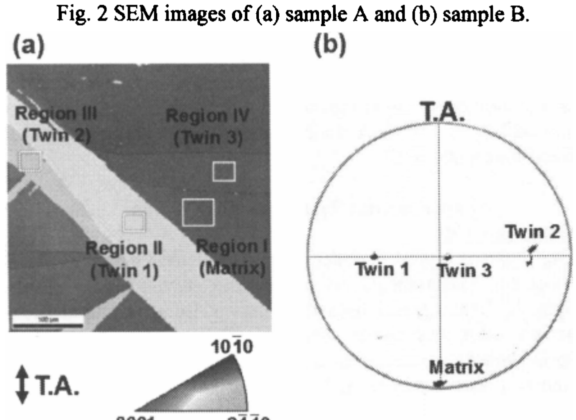

Fig. 3 (a) IPF map and (b) 0001 pole figure on location B of sample A.

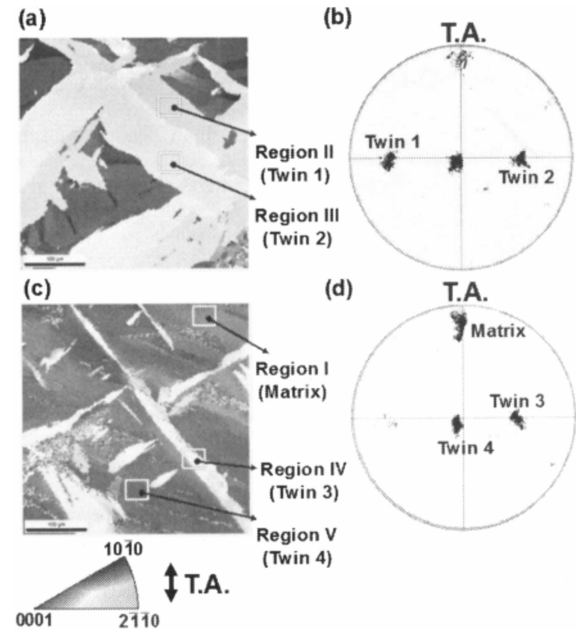

Fig. 4 (a) IPF map and (b) 0001 pole figure on location B; (c) IPF map and (d) 0001 pole figure on location C in sample B.

In the present nano-indentation experiments, the tip radius of 50nm is significantly narrower than the width of the twins, even for higher order twins with very fine size. Therefore, the measurements can be carried out at the area of a homogeneous twin phase far from twin boundaries. The hardness from the Oliver and Pharr method [5] is defined as:

$$H=P_{max}/A \qquad (1)$$

,where P_{max} is the maximum indentation force and A is the resultant projected contact area at that load. Nano-indentation experiments were carried out on regions I-IV (sample A) and regions I-V (sample B), which were located at matrix and several {10-12}<10-11> twins. The hardness data for samples A and B are summarized in Table I. The standard deviation and the basal Schmid factor values calculated by the pole figures constructed from the EBSD data are also included in the table. The hardness values clearly depend on the orientation of the region. Deformation twinning causes a lattice rotation of the twinned region. Twinned regions rotated to softer orientation such as twins 1 and 2 in sample A and twins 1, 2 and 3 in sample B exhibited relatively smaller hardness values, while twins having harder orientation such as twin 3 in sample A and twin 4 in sample B showed higher hardness values. Matrix region was originally in a harder orientation and therefore, showed higher hardness values. Thus, the present nano-indentation measurements show that the hardness of the matrix and the twin regions is strongly dependent upon crystallographic orientation and the effect of the structural transformation on the increase in the hardness of twin regions is negligible. Previous nano-indentation studies on polycrystalline α-titanium [6] showed that deformation twins were always harder than the matrix regardless of the twins' size, variants and orientations. The authors argued that the hardness increment in twin regions arises from structural transformation of dislocations of matrix to the twin lattice, which is known as the Basinski hardening [1]. In contrast, the present results did not confirm this strengthening effect of the twin. This may be due to low deformation applied both in primary and secondary tensile tests to induce meaningful dislocation densities responsible for this effect. Also the nano-indentation experiments may not fully probe the three-dimensional distribution of dislocations in the sample tested. Therefore, further studies on the type and distribution of dislocations in matrix and twin regions by TEM are required and are currently in progress.

Table I. Nano-indentation results obtained from matrix and {10-12}<10-11> twins.

Sample	Locations	Hardness [MPa]	Std. Dev.*	S.F. for Basal slip**
A	Matrix	838	40	0.06
	Twin1	824	33	0.49
	Twin2	747	28	0.42
	Twin3	888	23	0.07
B	Matrix	832	47	0.08
	Twin1	774	28	0.46
	Twin2	752	31	0.49
	Twin3	751	24	0.49
	Twin4	798	28	0.05

*Std. Dev.=Standard Deviation, **S.F.=Schmid factor.

Summary

Nano-indentation measurements on the deformation matrix and several {10-12}<10-11> twin regions have been carried out in deformed magnesium single crystals. The hardness values show orientation dependence, following Schmid law in both matrix and twins and no significant increase of the hardness of twin regions is observed.

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