Available online at www.sciencedirect.com

ScienceDirect

Acta Materialia 93 (2015) 114–128

Acta MATERIALIA

www.elsevier.com/locate/actamat

Effect of indentation size on the nucleation and propagation of tensile twinning in pure magnesium

R. Sánchez-Martín,[a] M.T. Pérez-Prado,[a] J. Segurado[a,b] and J.M. Molina-Aldareguia[a,*]

[a]*IMDEA Materials Institute, C/Eric Kandel 2, 28906 Getafe, Madrid, Spain*
[b]*ETS Ingenieros de Caminos, Universidad Politécnica de Madrid, 28040 Madrid, Spain*

Received 30 December 2014; revised 7 April 2015; accepted 7 April 2015
Available online 4 May 2015

Abstract—Tensile twinning is a key deformation mode in magnesium and its alloys, as well as in other hcp metals. However, the fundamentals of this mechanism are still not fully understood. In this research, instrumented nanoindentation and crystal plasticity finite element simulations are utilized to investigate twin formation and propagation in pure Mg. With that purpose, several nanoindentations at different indentation depths were performed in pure Mg single crystals with a wide range of crystallographic orientations. A careful analysis of the deformation profile, by atomic force microscopy, and of the microtexture, by electron backscatter diffraction, in areas around and underneath the indents, reveals that twinning is subjected to strong size effects, i.e., that the relative activity of twinning increases dramatically with the indentation depth. Furthermore, the twin volume fraction is found to be related to the pile-up or sink-in areas close to the indentations. A decrease in hardness in orientations where the twinning activity is high was confirmed both experimentally and by crystal plasticity finite element simulations. Finally, our results support the thesis that twin activation is an energetic process that demands a concentration of high stresses in a certain activation volume.

Keywords: Magnesium; Nanoindentation; Crystal plasticity; Twinning; Size effects

1. Introduction

Room temperature deformation of pure magnesium (Mg) is hindered by the limited number of slip systems available. In order to accommodate an arbitrary plastic deformation, especially along the "c" direction, the activation of twinning is necessary [1,2]. Twinning activity, enhanced by large strain rates and low temperatures [3,4], strongly affects the mechanical behavior of Mg and its alloys, specially their yielding [5], work hardening [6,7], ductility [8] and dynamic recrystallization [9].

The main twin planes are $\{10\bar{1}2\}$, $\{10\bar{1}1\}$, and $\{10\bar{1}3\}$ [10]. Twinning along the $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ system is allowed when an extension along the c axis is induced by the applied stress. That is the reason why this deformation mechanism is called "tensile twinning" [11]. On the other hand, twinning on the $\{10\bar{1}1\}$ and $\{10\bar{1}3\}$ planes may be active when a contraction along the c axis results from the application of the external load [12]. These "compression twins" have a significantly higher critical resolved shear stress (CRSS) than their homologous tensile twinning [1], and therefore, are much less commonly activated. In this work, only the tensile mode will be studied, and the term "twinning" will hereafter refer to tensile twinning.

Twin development involves three steps, which take place sequentially: nucleation, propagation and growth [13]. Twin nucleation remains, to date, a very unknown phenomenon. It has been reported that the introduction of a twin embryo is a volume activated process [13,14] that requires quite severe stress states [15]. Indeed, the high energy required to create a twin boundary in pure Mg, γ_{TB}, [16] justifies the requirement of a minimum size of the twin embryo to be stable. These features make twinning a deformation mechanism highly affected by size effects. For example, Barnett et al. [17] reported the inhibition of twinning in the AZ31 magnesium alloy when the grain size drops below one micron. This is related to the decrease in the number of dislocations forming the pile-ups in the fine-grained microstructures, which leads to a reduction in the local stresses at the boundaries to values below those necessary for twin nucleation [14]. Twinning is also profoundly affected by the size of the deformed specimens. Yu et al. [18] investigated this issue by compressing Ti-5 at.% Al single crystal micropillars of different diameters. Their results show that the stress necessary to nucleate and propagate twinning increases drastically as the diameter of the micropillar (D) decreases. For samples with diameters smaller than a critical value ($D \leqslant 0.7$ μm), twinning was completely suppressed. Prasad et al. [19] carried out a similar study in pure Mg single crystals with dimensions of different length scales. In particular, the specimens in the micrometer range showed a critical resolved shear stress

* Corresponding author. Tel.: +34 915493422; e-mail: jon.molina@imdea.org

http://dx.doi.org/10.1016/j.actamat.2015.04.005
1359-6462/

for twinning ($CRSS_{tw}$) several orders of magnitude larger than that of macroscopic samples. The requirement of high local stresses to nucleate twinning in pure Mg microscale specimens has also been reported by Yu et al. [15]. The origin of the great difference between the $CRSS_{tw}$ measured in microscopic and macroscopic samples can be understood as follows. While in the first case the measured stress value is close to the local stress that activates twinning, in the latter, the macroscopically measured stress is significantly lower than the actual twinning activation stress [20].

Relatively few works have dealt with the occurrence of twinning during indentation of Mg. Shin et al. [21] confirmed that, in addition to $\langle c+a \rangle$ dislocations, twinning is necessary to accommodate the plastic strains developed under an indentation in magnesium single crystals. Moreover, they found that twin formation is influenced by the orientation of the indented plane. Very recently, Zambaldi and Zehnder [22] and Selvarajou et al. [23] studied the active deformation mechanisms of Mg at room temperature using single crystal indentation. They observed different twinning patterns on the surface near the indent depending on the crystal orientation, confirming the orientation dependence found by Shin et al. [21]. Somekawa and Schuh [24,25] studied the effect of grain boundaries on twin formation under indents in Mg and its alloys. While twinning was not found when indenting small grains [24], twinned zones were reported in the case of coarse microstructures [25]. This fact has been explained by the relation between the grain size and the volume affected by plastic strains. While in the first case, the plastic zones developed were larger than the grain size, in the second case these plastic zones were significantly smaller than the grain size, suggesting that grain boundaries play an important role on twin nucleation. Due to the small volumes of material affected by small indentations and to the described nature of twinning as a volume activated process, some kind of indentation size effect (ISE) is expected on twin development. To date, no studies have analyzed this issue and further work is clearly needed in this area.

The objective of this paper is to clarify the incidence of potential ISE effects on the activation and propagation of twinning in pure Mg. With that purpose, several nanoindentations at a wide range of indentation depths were performed in individual grains of a polycrystalline sheet of pure Mg with different crystallographic orientations. The microstructure in areas around and underneath the indents was thoroughly characterized by atomic force microscopy (AFM) and electron backscatter diffraction (EBSD). Moreover, the effect of twinning on the mechanical response of pure Mg single crystals at different indentation depths was analyzed. Crystal plasticity finite element (CPFE) simulations were coupled to experimental mechanical property data in order to determine the incidence of twinning during indentations at different depths. Our results support the thesis that twin activation demands a concentration of high stresses in a certain activation volume.

2. Experimental procedure

The initial material was a rolled and annealed sheet of pure Mg (99.9% purity) with an average grain size of 80 μm and the typical basal texture. In order to obtain a coarser microstructure, which would facilitate the avoidance of boundary effects during indentations at the center of the grains, this Mg sheet was heat treated at 500 °C during 8 h. The annealing treatment was carried out in samples encapsulated in pyrex tubes under an argon atmosphere with the aim of preventing oxidation. The final grain size is in the mm range, as shown in Fig. 1.

Microstructure characterization was performed by electron backscatter diffraction (EBSD) in a focused ion beam field emission gun scanning electron microscope (FIB-FEGSEM) (Helios NanoLab 600i, FEI) equipped with an Oxford-HKL EBSD system and operated at a voltage of 15 kV and using an emission current of 2.7 nA. Sample preparation for EBSD included grinding with 2000 grit size SiC paper, mechanical polishing with 3 and 0.25 μm diamond paste, and chemical etching using a solution of 75 ml of ethylene glycol, 24 ml of distilled water and 1 ml of nitric acid.

With the aim of analyzing the effect of the indentation size on twinning, eight grains with different orientations were selected for this study. In grains 1–7, whose declination angles (angle between the c axis and the indentation axis, δ) are included in Fig. 1, several nanoindentations with maximum indentation depths varying from 200 to 7000 nm were carried out using a Hysitron TI950 triboindenter. A diamond sphero-conical indenter with a tip radius of 2 μm and an apex angle of 70° (Tip 1) was utilized to carry out indentations up to a maximum indentation depth (h_{max}) of 750 nm. A second sphero-conical indenter, with a tip radius of 10 μm and an apex angle of 60° (Tip 2), was used to perform indentations with depths larger than 750 nm. All the tests were conducted in a displacement control mode using a trapezoidal loading curve, with a loading and unloading time of 5 s, and a 2 s hold time at h_{max}. The microstructure and the topography of areas of the free surface in the vicinity of the indents were examined by EBSD and AFM respectively. Grain 8, whose c-axis lies almost perpendicular to the indentation axis ($\delta = 80.1°$), was selected to perform a cross-section EBSD analysis

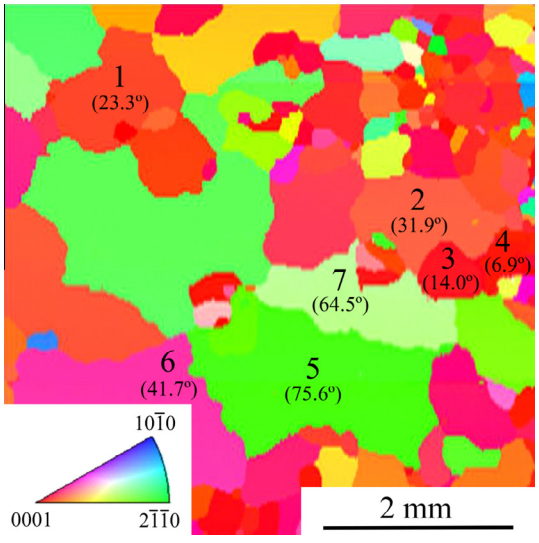

Fig. 1. EBSD inverse pole figure map in the normal direction (ND) of the annealed pure Mg sheet illustrating 7 out of the 8 grains investigated (the declination angle, δ, for each grain is indicated in brackets).