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Microstructural evolution of pure magnesium under high strain rate loading

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Abstract—The mechanical behavior of extruded pure magnesium was studied experimentally under high strain rate (10^3 s^{-1}) compression loading in the extrusion direction. Electron back scattered diffraction was used to examine the changes in the texture and transmission electron microscopy was used to investigate the dislocation structures in the material. Extensive grain reorientation due to extension twinning is observed. Dislocation activity is observed inside the parent region ($\langle a \rangle$ slip) as well as the twinned region ($\langle c + a \rangle$ slip). The high degree of strain hardening observed is postulated to be due to the texture hardening associated with extension twinning coupled with significant increase in the dislocation density with strain. In compression along the extrusion direction, extension twinning and dislocation activity are both needed to accommodate plastic deformation. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Magnesium; Twinning; Dislocations; High strain rate; Texture

1. Introduction

The interest in the use of magnesium and its alloys in potential automobile, aerospace and other applications has risen significantly over the last decade. The low density and high specific strength of these materials make them attractive in applications where their increased use can lead to significant energy savings [1–4]. In order to successfully use these materials in applications in which they will be subjected to dynamic loading (such as automotive crash), we need to understand their behavior under such conditions. Dynamic loading can induce complex deformation mechanisms in materials which might not be activated under quasi-static loading conditions [5].

Magnesium has a hexagonal close packed (HCP) crystal structure and a slightly less than ideal c/a ratio. The mechanisms that govern the deformation of Mg and its alloys at low strain rates have been widely studied in recent years [6-24]. The dominant slip systems in magnesium are the $(0001)\langle 11\bar{2}0\rangle$ basal slip system and the $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ prismatic slip system [6,25]. Since both of these slip systems have the $\langle a \rangle$ type Burger's vector, they are insufficient to accomodate general plastic deformations [26,27]. Thus, additional mechanisms such as the pyramidal $\langle c + a \rangle$ slip system and deformation twinning must be activated under general loading conditions. The commonly observed deformation twinning modes in magnesium are the

 $\{10\overline{1}2\}\langle\overline{1}011\rangle$ extension twinning mode and the $\{10\overline{1}1\}$ $(10\bar{1}\bar{2})$ contraction twinning mode [8,14,15,18,19,24,28]. The names represent the type of deformation supplied by the twinning mode. The extension twinning mode supplies extension along the crystallographic c-axis whereas the contraction twinning mode supplies contraction along the c-axis [14]. The experimentally measured critical resolved shear stress (CRSS) values for these slip systems in Mg single crystals have been reported by several studies [28-32]. Many authors [28,29,33,34] also define a CRSS for the twin systems, although it is not clear that twinning is controlled only by the magnitude of the shear stress. Recent works suggest that twinning is largely influenced by the local stress concentrations and the grain boundary defects in the case of polycrystalline Mg and alloys [35,36]. Yu et al. [37] reported twin nucleation stresses of ~800 MPa for a nanometer sized sample but suggested that these high stresses can be considered as local or microscopic stresses. Although, looking at the critical stresses for twinning does not provide complete information about the conditions for twin nucleation, it is often helpful to look at the reported critical stress values in comparison with those reported for other mechanisms as presented by the summary from Zhang and Joshi [38]. The lowest CRSS is for basal slip (~0.5 MPa), and this appears to be followed by the so-called CRSS for extension twinning ($\sim 2-5$ MPa) [31,39]. The reported values of CRSS for prismatic slip (~10–45 MPa), pyramidal $\langle c + a \rangle$ slip (~35–80 MPa) and contraction twinning (~30-100 MPa) are higher (and also display significant ranges in value) [28,29,31,32,39]. The activation of the $\langle c + a \rangle$ pyramidal slip system is difficult due to its high CRSS, but it has been observed in some

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quasi-static studies [10,11,32,40-42]. Basal slip and extension twinning are thus the mechanisms that are most easily activated in pure Mg when deformed at low rates. The $\{10\bar{1}2\}\langle\bar{1}011\rangle$ extension twin system has been extensively studied in the low strain rate regime over the last decade [14,15,22-24,35,43-45]. Contraction twinning was observed in some c-axis compression studies in pure Mg single crystals [28,34], and some groups have presented observations of contraction twins in polycrystalline Mg alloys [15,18,46-48].

Strong texture is developed in Mg and its alloys during thermomechanical processing and has been attributed to the underlying anisotropic HCP crystal structure, the very different CRSS for the various slip and twinning systems and the strong reorientation associated with the twinning. The texture of the material relative to the principal loading directions has a significant influence on the deformation of Mg and its alloys. A large anisotropy is observed in this material in mechanical properties such as flow stress, elongation to failure, and strain hardening behavior [6,45,49]. Therefore, it is important to consider the effects of texture when studying the deformation of polycrystalline Mg and its alloys, as noted by previous low strain rate studies [14,15,22–24,43,45].

In the recent years, some studies have investigated the high strain rate behavior of Mg alloys [49-57]. Ulacia et al. [50] observed that in AZ31, extension twinning remains the predominant mechanism at high rates and temperatures for loading geometries favoring this mechanism as it does in the low strain rate regime. Tucker et al. [49] have also performed high strain rate tests on a rolled magnesium alloy AZ31B and observed significant rate effects (increase with increasing rate) on the compressive yield, strain hardening rate and ductility in the normal direction but not in the transverse and rolling directions. In a recent study by Dudamell et al. [52] on AZ31, enhanced extension twinning activity was observed at high strain rates. Similar observations of increase in twin volume fraction at higher strain rates were reported by Li et al. [55] in an ultrafinegrained ZK60 alloy. The review of the dynamic behavior of Mg and Mg alloys by Prasad et al. [57] suggests that materials undergoing plastic deformation dominated by extension twinning have relatively rate-insensitive strengths whereas those with dislocation activity as the primary mechanism of plastic deformation exhibit some rate dependence of flow stress. This suggests that high strain rate loading may have a strong influence on some deformation mechanisms in Mg and alloys.

Mg alloys have been the focus of the majority of prior studies, but the behavior of pure magnesium remains less well characterized. It is important to characterize the fundamental deformation mechanisms in pure Mg without the effects of solutes or precipitates. The deformation of pure magnesium under plane-strain compression was studied by a few researchers [6,18,21]. Their observations indicated significant differences in the deformation processes when the material is under c-axis extension as compared to c-axis contraction. Also, suppression of a strong basal texture was observed to impart ductility to pure Mg by Gehrmann et al. [21]. Although there have been some studies on deformation of Mg alloys at high strain rates, a detailed study of the deformation mechanisms in pure magnesium at high strain rates is yet to be performed.

We describe here an experimental approach to investigate the evolution of the fundamental deformation

mechanisms in pure magnesium at high strain rates. We have performed high strain rate compression experiments on pure magnesium in the extrusion direction (ED) and investigated the microstructural evolution through transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD) analyses. In order to investigate the evolution of the microstructure with deformation, controlled strain tests have also been performed under high strain rate loading. The microstructural evolution (of texture, twins and dislocations) is then used to explain the observed mechanical behavior of the material.

2. Experiments

2.1. Initial texture

A hot-extruded Mg rod of commercial purity (99.9%) was used in this study. The microstructure of the material consists of equiaxed grains with an average grain size of about 20 μ m. The initial texture of the material was analyzed using X-ray diffraction (XRD) in the extrusion direction (ED) and is shown in Fig. 1. The initial microstructure was found to be typical for an extruded HCP material with a random in plane orientation spread of the basal (0001) poles perpendicular to the ED [44]. The spread of the basal poles in the radial direction (RD) was observed to be quite uniform.

2.2. High strain rate tests

Uniaxial compression experiments at high strain rates were performed using a conventional Kolsky bar (also known as a Split-Hopkinson pressure bar). The details of this technique have been described in the literature [58]. The specimens were machined using electrical discharge machining (EDM) to dimensions of $3 \text{ mm} \times 4 \text{ mm} \times 4 \text{ mm}$. The loading surfaces were polished with 1200 grit SiC paper before testing to remove any recast layer. Lubrication was used to reduce friction effects. The specimens were compressed in the ED at room temperature at strain rates of the order of 10^3 s^{-1} .

2.2.1. Controlled dynamic strain tests

The nature of dynamic compression tests involving Kolsky bars is such that the total strain imposed is dictated by the strain rate and duration of loading and the tests typically lead to large strains (>0.1). Smaller strains were achieved by using collars of hardened steel surrounding the magnesium sample during testing similar to the work in Ref [59]. Note that care must be taken while performing these experiments to ensure that the collar does not come in contact with the lateral sides of the specimen. If the lateral expansion of the specimen is restricted due to the surrounding collar, the stress state will no longer be uniaxial and effects of lateral confinement will have to be considered in the analysis. These controlled dynamic strain tests were carried out at a strain rate of about $2 \times 10^3 \text{ s}^{-1}$ and plastic strains of approximately 3.5% and 9% were achieved. These values were chosen since at \sim 3.5% strain, the plastic deformation is in the initial stages and the strain hardening rate is rising rapidly while at $\sim 9\%$ strain, the strain hardening rate is at its maximum value. Microstructural investigation at these strains enables us to gain an understanding of the deformation processes responsible for the observed mechanical behavior.

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