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Evidence for exceptional low temperature ductility in polycrystalline magnesium processed by severe plastic deformation

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ABSTRACT

An investigation was conducted to examine the mechanical behavior and microstructure evolution during deformation of ultrafine-grained pure magnesium at low temperatures within the temperature range of 296–373 K. Discs were processed by high-pressure torsion until saturation in grain refinement. Dynamic hardness testing revealed a gradual increase in strain rate sensitivity up to $m \approx 0.2$. High ductility was observed in the ultrafine-grained magnesium including an exceptional elongation of ~360% in tension at room temperature and stable deformation in micropillar compression. Grain coarsening and an increase in frequency of grain boundaries with misorientations in the range 15°–45° occurred during deformation in tension. The experimental evidence, when combined with an analysis of the deformation behavior, suggests that grain boundary sliding plays a key role in low strain rate deformation of pure magnesium when the grain sizes are at and below ~5 μm.

1. Introduction

The deformation behavior of magnesium has been studied for several decades but an increasing interest has developed recently because of the potential for using magnesium in a wide range of industrial applications. Nevertheless, the flow behavior of fine-grained magnesium remains an area of some controversy. For example, it is known that twinning plays a major role in the room temperature deformation of magnesium and it was shown that both twinning and dislocation slip are active to accommodate the high strain rate compressive deformation of pure magnesium having a grain size of ~20 μm [1]. Twinning leads to a hard orientation of the crystalline structure and significant hardening is observed in the stress-strain curves [1]. Also, experiments show that the fraction of grains with twinning is independent of the grain area although grain areas smaller than ~21 μm^2, corresponding to grain diameters of <5.25 μm, were not considered [2]. However, a twinning to slip transition was reported in compression testing when reducing the grain size in an AZ31 magnesium alloy [3] and also in pure magnesium [4,5]. Moreover, there is some limited evidence for a grain boundary sliding contribution in plastic deformation at room temperature [6,7]. It was also reported that twinning is suppressed and the contribution of grain boundary sliding increases in magnesium with a grain size of ~60 nm, thereby producing an increased strain rate sensitivity and an inverse Hall-Petch relationship [8]. A Hall-Petch breakdown at low strain rates was demonstrated in magnesium but in samples with grain sizes of several microns [7].

It was shown recently that non-basal slip and twinning are severely hindered during tensile testing at 323 K when the grain size is reduced to ~5 μm and this was interpreted in terms of an increase with grain refinement in the ratio of the critical resolved shear stress for non-basal to basal slip [9]. The accommodation of plastic deformation was attributed to a mechanism of strain propagation through boundaries with misorientations of <35°. Thus, the ductility of a sample with a grain size of 5 μm was lower than in samples with grain sizes of 19 and 36 μm and this was due directly to the limited strain accommodation by this mechanism [9]. Nevertheless, excellent ductilities were recorded in pure

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magnesium with even finer grain structures including an elongation of 230% in a sample with a grain size of 1.2 μm tested at room temperature at a low strain rate of 10^{-5} s^{-1}[7]. Good plasticity at room temperature was observed also in fine-grained magnesium tested at low strain rates [5]. In order to explain this different deformation behavior observed at low strain rates in fine-grained magnesium, a mechanism of strain accommodation was proposed based on basal slip and grain boundary rotation [5].

The present experiments were initiated in order to obtain a better understanding of the deformation mechanisms operating in pure magnesium at low temperatures when using samples with unusually fine grain structures. High-pressure torsion (HPT) [10] was used to produce contamination-free samples with ultrafine-grained structures and the material was tested using dynamic hardness, micro-pillar compression and conventional tensile testing.

2. Experimental material and procedures

The material used in these experiments was commercial purity (CP) polycrystalline magnesium provided by RIMA (Bocaiúva-MG, Brazil). The material was received as a cast ingot with an average grain size of ~300 μm. Billets of 10 mm diameter were machined from the ingot and then discs with 1.0 mm thickness were cut from the billets using a low-speed diamond saw. Finally, the thicknesses of the discs were reduced to ~0.85 mm using grinding papers.

The discs were processed by high-pressure torsion using anvils that gave quasi-constrained conditions. In this processing condition, each anvil contains a shallow depression having a depth of less than one-half of the disc thickness so that some limited material is extruded around the periphery of the disc during processing [11,12]. The samples were processed under a pressure of 6.0 GPa with a rotation rate of 1 rpm at room temperature. The maximum temperature rise in the sample was estimated as ~10 K [13] considering a reasonable flow stress of ~200 MPa. After processing by HPT, the average disc thickness was ~0.72 mm which corresponded to a reduction in thickness of ~15%. Samples were processed to totals of 1/8, 1/2, 2 and 10 turns which correspond to effective strains [14,15] of approximately 1.4, 5.8, 23.1 and 115.5, respectively, at the mid-radius position of $r = 2.5$ mm where r is the disc radius.

After processing, all samples were prepared for electron backscatter diffraction (EBSD) characterization by grinding with abrasive paper #2500 and polishing with 9-, 6-, 3- and 1-μm diamond suspensions and a colloidal silica suspension. Final polishing was conducted using a Vibromet facility with a blue colloidal silica suspension for 1 h. The microstructures of the processed discs were evaluated by EBSD using a JEOL JSM-7001F scanning electron microscope (SEM) operating at 7 kV and the data were collected and analyzed using TSL orientation imaging microscopy (OIM) and appropriate software with a step size of 0.1 μm. All microstructures were observed at the mid-radius positions and Kikuchi patterns were obtained at a working distance of 15 mm with a sample tilt of 70°. The images were cleaned up using grain confidence index (CI) standardization, neighbor CI correlation and grain dilation procedures.

Dynamic hardness testing was performed using a Shimadzu DUH-211S hardness facility equipped with a Berkovich indenter. Tests were conducted using a maximum load of 300 mN, a loading rate of 70 mN s^{-1} and a dwell time of 500 s. The data for the indenter depth, h, and testing time, t, were converted to strain rate, $\dot{\varepsilon}_{eff}$, using the relationship [16–18].

$$\dot{\varepsilon}_{eff} = C \times \left({}^{1}\!/_{h} \right) \left({}^{dh}\!/_{dt} \right) \tag{1}$$

where C is a constant equal to 0.1 [18]. The hardness, H, was converted to flow stress, σ, using [18].

$$\sigma = {}^{H}\!/_{\alpha} \tag{2}$$

where α is a correlation factor equal to 3.3. The strain rate sensitivity was determined as the slope of the curve of flow stress, σ, plotted as a function of strain rate, $\dot{\varepsilon}$.

Tensile specimens with gauge lengths of 1.0 mm and cross-sectional areas of 1.0 × 0.72 mm^2 were extracted from the processed discs at the mid-radius positions using electro-discharge machining. These specimens were tested in tension at room temperature (296 K) using an Instron machine operating at a constant rate of cross-head displacement. The load and displacement data were converted to engineering stress and engineering strain and the influence of the elastic distortion of the testing apparatus was minimized by equating the elastic portion of the stress-strain curves to the theoretical elastic modulus of magnesium. Tests were performed with initial strain rates in the range of 10^{-5}–10^{-1} s^{-1}. Some specimens were also annealed at 373 K for 24 h before testing in tension at 296, 323 and 373 K. The gauge length of a tensile sample was polished prior to testing at a strain rate of 10^{-5} s^{-1}, the test was interrupted at an elongation of 50% and the surface was analyzed by SEM. Further polishing was then conducted to prepare for EBSD. Finally, the gauge lengths of some samples were polished for EBSD analysis near the crack tips after pulling to failure.

Two micro-pillars were fabricated from the material processed through 10 turns of HPT using focused ion beam (FIB) machining [19–21] with the surfaces of these discs prepared using the same procedure as for EBSD characterization. These two pillars had diameters of ~1.77 and ~1.73 μm and lengths of ~6.16 and ~5.19 μm, respectively. The micro-pillar compression experiments were performed using a nanoindenter (Triboscope, Hysitron Inc.) equipped with a custom-made flat punch compression tip. The micro-compressions were conducted at a constant nominal displacement rate corresponding to a prescribed strain rate of 10^{-3} s^{-1}.

3. Experimental results

3.1. Microstructures after HPT

The microstructures of CP magnesium at different stages of processing by HPT are shown in the inverse pole figures in Fig. 1. Inspection shows that significant areas with fine grains are observed even in the sample processed through only 1/8 turn together with areas of coarser grains. Most of these coarse grains exhibit twinning. The area fraction of fine grains increases with increasing deformation and so that fine grains occupy most of the microstructure after 1/2 turn. This gradual refinement of a coarse-grained structure matches a model of grain refinement proposed for magnesium deformed at high temperatures [22,23] and shows that a similar mechanism also occurs at room temperature. After 1/2 turn of HPT, there are only a few coarser grains in the microstructure with sizes of ~1–3 μm. Nevertheless, these coarser grains are also present even after 10 turns of HPT although their area fraction has decreased significantly. Thus, the microstructure is composed essentially of ultrafine grains after 10 turns of HPT which is consistent with earlier reports using TEM analysis [5,24].

3.2. Dynamic hardness testing

The evolution of flow stress as a function of the effective strain rate was determined by dynamic hardness testing at the mid-radius of the discs at different stages of HPT processing. Fig. 2 shows the curves for samples processed by 1/8, 1/2, 2 and 10 turns of HPT. The