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Enhancement of strain-rate sensitivity and shear yield strength of a magnesium alloy processed by high-pressure torsion

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A Mg–Zn–Zr alloy was processed by high-pressure torsion for up to 2 turns at room temperature to produce significant grain refinement together with enhanced plasticity and strength. Measurements were performed to determine the strain-rate sensitivity, shear yield strength and activation volume as a function of the processing conditions. The results suggest there is a significant contribution from grain boundary sliding to the flow process and the onset of plasticity is associated with heterogeneous dislocation nucleation.

Keywords: Grain boundary sliding; High-pressure torsion; Magnesium alloy; Strain-rate sensitivity; Yielding

Magnesium alloys have attracted much attention as structural materials for automotive, aerospace and electronics applications because of their very high strength-to-weight ratio. However, there are drawbacks to using Mg alloys due their low ductility at room temperature (RT) due to their hexagonal close-packed (hcp) structure which provides only a limited number of active slip systems and a consequent poor workability and formability. Among various strategies to overcome this deficiency [1], one attractive approach is grain refinement which can improve both strength and ductility.

To date, one of the most recognized methods for achieving excellent grain refinement in bulk metals is through the application of severe plastic deformation in procedures such as equal-channel angular pressing (ECAP) [2] and high-pressure torsion (HPT) [3]. In general, Mg alloys are generally processed by ECAP at relatively high temperatures in order to avoid segmentation and cracking caused by their limited ductility at low temperatures [4,5]. By contrast, processing by HPT introduces a high hydrostatic pressure so that undesirable cracking may be avoided even at RT [6]. By comparison with ECAP, HPT processing also has an advantage in producing higher plastic strains and hence smaller grain sizes [7]. During HPT, the equivalent von Mises strain imposed on the disk, ε_{eq}, is given by $2\pi Nr/(\sqrt{3}t)$, where r and t are the radius and thickness of the disk and N is the number of torsional revolutions [8]. Therefore, in HPT the strain varies locally across the disk and this contrasts with ECAP where the imposed strain is reasonably constant.

Extensive research has examined the evolution of microstructure and hardness of Mg alloys during HPT processing at both RT and elevated temperatures [6,9,10]. Nevertheless, there was no attempt to monitor the changes in strain-rate sensitivity, m, and shear yield strength, τ_y, of Mg alloys processed by HPT although these are essential parameters for estimating the workability of the alloys. The present research was motivated to address this deficiency by using nanoindentation to explore the evolution of m and τ_y and the activation volume, V^*, in a ZK60 Mg alloy. This alloy was selected because it has the best combination of strength and ductility at RT among the common wrought Mg alloys with principal alloying elements of Al, Mn and Zn [11].

An extruded ZK60 (Mg–5.5 Zn–0.5 Zr in wt.%) Mg alloy with a diameter of 10 mm was cut into disks

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~1.5–2.0 mm thick and these disks were carefully polished on both sides to final thicknesses of ~0.8 mm. The HPT processing was conducted at RT under quasi-constrained conditions [12] using a pressure of 6.0 GPa, a rotational speed of 1 rpm and with the disks processed through $N = 1/4$, 1/2, 1 and 2 turns. After processing, the disks were mechanically polished to a mirror-like finish and then etched with a solution of 60 ml ethylene glycol, 20 ml glacial acetic acid, 19 ml distilled water and 1 ml nitric acid for the as-extruded sample and a solution of 1 g oxalic acid, 1 ml nitric acid and 98 ml distilled water for the processed specimens. The microstructures at the edges of the disks were examined using an optical microscope (CK40M, Olympus, Tokyo). The distributions of the Vickers microhardness, Hv, across the diameters of each disk were measured with HMV-2 equipment (Shimadzu, Tokyo) at a peak load P_{max} of 980 mN.

Three different nanoindentation tests were performed at the edge of each disk using a Nanoindenter-XP (formerly MTS; now Agilent, Oak Ridge, TN) which permits constant strain rate (CSR) testing, strain-rate jump (SRJ) testing and "pop-in" testing. The first two tests were performed with a three-sided pyramidal Berkovich indenter in order to estimate m. The pop-in tests were conducted with a spherical indenter to evaluate τ_y. The radius of the spherical tip, R, was estimated by a Hertzian contact analysis [13] of indentations on fused quartz and was determined as 5.75 μm.

Figure 1 shows representative microstructures of the as-extruded sample and the edges of the disks processed by HPT for $N = 1/4$, 1/2 and 2 turns. There is a noticeable change in microstructure through HPT processing. Thus, the as-extruded condition in Figure 1a consists of a bimodal microstructure with a large area fraction (>50%) of coarse grains with sizes of ~25 μm surrounded by some fine grains with sizes of ~4–5 μm. After processing through 1/4 turn (Fig. 1b), both the fraction and size of the coarse grains was significantly reduced to ~35% and ~20 μm, respectively, and the fine grains were refined to ~2–3 μm with an increased area fraction of ~65%. After 1/2 turn (Fig. 1c), the coarse grains remained unchanged at ~20 μm but the fine grains occupied a high area fraction of ~75% with an average size of ~1.0–1.5 μm. Thereafter, the microstructure remained constant with further increase in N and the microstructural features after 2 turns were similar to those at 1/2 turn as shown in Figure 1d, thereby demonstrating a reasonable level of microstructural saturation up to at least 2 turns.

The microstructural evolution may be explained in terms of dynamic recrystallization (DRX). Thus, although deformation within each coarse grain is insufficient for activating DRX due to the limited active slip systems, the stress concentrations generated at high-angle grain boundaries can trigger DRX leading to the nucleation of new grains preferentially along the original boundaries in a necklace-like pattern with the interiors of the coarse grains remaining unrefined [5]. The bimodal structure introduced after $N = 2$ turns is in agreement with earlier ECAP studies on ZK60 alloys [4,5] and demonstrates that the processed microstructure is essentially dependent upon the initial grain size. In practice, a bimodal grain size distribution is advantageous because of the potential for achieving both high strength and good ductility [14].

The variations in Vickers microhardness vs. distance from the centers are shown in Figure S1 of the online Supplementary material for the as-extruded disk and the processed disks with increasing N. Compared with the as-extruded sample where $Hv \approx 72$, all processed disks exhibit higher Hv throughout their diameters. There is a consistent trend of lower Hv in the centers of the disks and increasing Hv towards the edges where the ε_{eq} is the highest, thereby demonstrating a strain-hardening type of hardness evolution [15]. Although there are distinct scatters in Hv due to the bimodal microstructure, the Hv values are reasonably saturated at points beyond ~2 mm from the centers of all processed disks, and thus there is reasonable microstructural homogeneity around the peripheries of the disks. Accordingly, examinations of m and τ_y were conducted near the disk edges where the gradient in mechanical properties was a minimum and the values of ε_{eq} were the highest.

The values of the nanoindentation hardness H, estimated by the Oliver–Pharr method [16], were obtained at the edge of each disk at four different indentation strain rates from 0.0125 to 0.1 s^{-1} and the results are displayed with increasing N in Figure 2. Also shown are the average Hv values at the edges which were recalculated as the peak load divided by the projected contact area. Although higher values were recorded for H compared to Hv due to the indentation size effect [17], the data sets for H and Hv show a consistent trend which increases rapidly in the early stage of HPT and thereafter saturates towards maximum values. This trend is due to the absence of significant grain

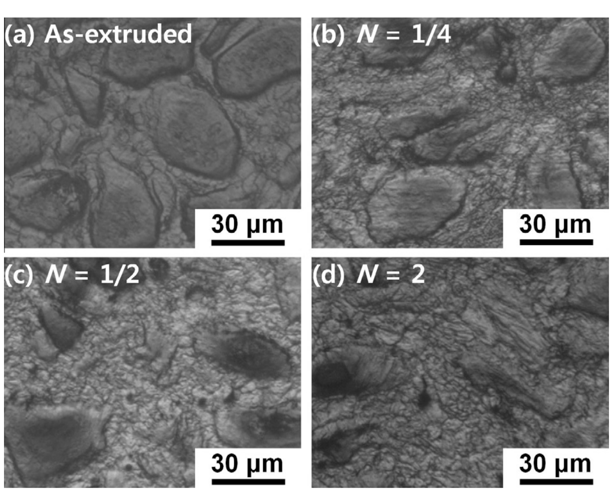

Figure 1. Representative optical micrographs taken at the edges of the ZK60 disks (a) in the as-extruded condition and after HPT for (b) 1/4, (c) 1/2 and (d) 2 turns.

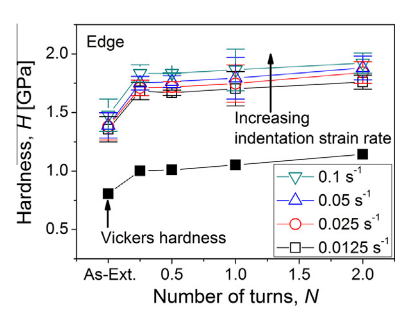

Figure 2. Variations in nanohardness and Vickers microhardness as a function of N: "As-Ext." denotes the as-extruded condition.