Grain size and microhardness evolution during annealing of a magnesium alloy processed by high-pressure torsion

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1. Introduction

The techniques of severe plastic deformation (SPD) [1] have attracted significant attention because they are capable of producing significant changes in the material structure and properties. Ultrafine grained structures, defined as structures with average grain size of less than 1 μm, are readily attained in bulk samples. Among these processes, high-pressure torsion (HPT) [2] is one of the most used. It is characterized by the application of high compressive stresses and simultaneous torsion.

HPT has been used to process different metallic materials, including magnesium and its alloys. Earlier papers reported significant grain refinement in these alloys after processing including an average grain size of 100 nm in a Mg–10% Gd alloy [3], 0.4 μm in a Mg–9% Al alloy [4] and ~1.0 μm in a Mg–3% Al–1% Zn (AZ31) alloy [5]. Provided these fine grain structures are stable at high temperatures, these materials may exhibit superplasticity [6]. It has been shown that the refined structure

References

is stable in a Mg–9% Al alloy processed by HPT and it exhibits superplasticity at 473 K [4]. However, the Mg–9% Al alloy is expected to contain second phase precipitates that prevent grain growth. The grain structure stability at high temperatures in a single phase magnesium alloy has not been studied yet. The present paper aims to process a single phase AZ31 alloy by HPT and determine its annealing behavior.

2. Material and methods

The material used in the experiments was an AZ31 (Mg–3% Al–1% Zn) commercial alloy provided by Timminco Corporation (now Applied Magnesium International), Aurora, CO. The material was received as 10 mm diameter extruded rods with an initial average grain size of ~10 μm [7]. Discs of the AZ31 alloy were cut from the rods with thicknesses of ~1.5 mm and ground to a thickness of 0.8 mm. The discs were then processed by HPT for 0.5, 1, 2, 3, 5 and 7 turns at room temperature and under a pressure of 6 GPa using a quasi-constrained equipment operating at 1 rpm (~0.1 rad/s) [8]. It is expected that the temperature rise during processing saturates at ~20 K [9] considering the average flow stress of the AZ31 alloy as 1/3 of its hardness after HPT [5].

The processed discs were cut into 8 parts, wedge shaped, using a diamond coated saw blade operating at low speed. Each sample was annealed for 1800 s at temperatures varying between 373 K, 423 K, 473 K, 573 K and 673 K. After annealing, the samples were polished, tested for microhardness and etched to reveal the microstructure. The microhardness tests were carried out along the radius of the disk from the center to the edge with a minimum of 12 indentations in each sample separated from each other by at least three times the indentation size and the average hardness was determined from all indentations. Representative images of the microstructure were recorded near the center, at the mid-radius and near the edge of the samples. The average grain size after annealing was determined as the average linear intercept length obtained at these different locations.

3. Results

The average hardness of the AZ31 alloy processed by HPT is plotted as a function of number of turns in Fig. 1. It is observed that the initial hardness of AZ31 alloy, ~67 Hv, increases to ~117 Hv after two turns and saturates at ~115 Hv.

Representative images of the microstructures of the AZ31 alloy processed by different number of rotations of HPT and subjected to annealing at different temperatures are shown in Fig. 2.
in Fig. 2. The microstructure of samples processed by 0.5, 1, 2 and 5 turns are shown in different rows and the annealing temperatures of 423 K, 473 K, 573 K and 673 K are displayed in different columns. As expected the overall grain structures coarsen with increasing annealing temperature. It is observed that the general aspect of the grain structures do not change with the number of rotations in HPT before annealing. The microstructure of the alloy processed by HPT and the alloy processed by HPT and subjected to annealing at 373 K could not be clearly resolved by conventional metallography which is attributed to the high density of defects in the crystal lattice.

Fig. 3 shows the evolution of the average hardness (solid lines) and average grain size (dashed lines) of AZ31 alloy processed by HPT as a function of annealing temperature. It is observed that the hardness of all samples follows a similar trend. The only exception is the unexpected increase in hardness of the sample processed by 0.5 turn and subjected to annealing at 373 K. A significant decrease in hardness is observed after annealing at temperature of 423 K and higher. The average grain size increases with increasing annealing temperature as expected. However, there is a larger scatter in the values of grain size compared with the values of hardness.

4. Discussion

These results show that HPT processing leads to significant strain-hardening of the AZ31 magnesium alloy and maximum strength is observed after only two turns. A slight decrease in hardness is observed with further processing. Annealing at 373 K leads to a minor decrease in hardness of the samples processed to 1 turn or more of HPT. This effect is attributed to recovery of the dislocation structure without recrystallization. This is supported by the metallographic examination that failed to resolve the grain boundaries in these samples.

Annealing at 423 K caused a significant decrease in hardness and the grain boundaries of the AZ31 alloy could be resolved using metallography. This suggests that recrystallization takes place at this temperature in all samples. Annealing at higher temperature leads to grain growth and a decrease in hardness.

Fig. 4 shows the collected data of hardness plotted as a function of the inverse of the square root of the average grain size. Datum points from earlier reports of grain size and hardness [7,10–12] are also shown for comparison. A general linear relationship is observed which shows that the Hall–Petch relationship is valid for the range of grain sizes observed. A single point for the smallest grain size (~0.5 µm) [10] seems to stand away from the linear trend. More experiments within the range of grain sizes smaller than 1 µm are needed in order to evaluate a possible breakdown in the Hall–Petch relationship.

The experiments show evidence of recrystallization at 423 K and grain growth at higher temperatures which suggests poor thermal stability of the structure. However, the average grain size grows slowly and is maintained within the range of less than 10 µm up to 673 K. Therefore this material may exhibit superplasticity at high temperatures. In order to clarify the evolution of the grain size with annealing temperature, the present results are plotted in Fig. 5 together with data from

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the literature [13] for a similar alloy processed by other techniques. The average grain size was calculated as the average between the samples processed to different number of turns of HPT and subjected to annealing at similar temperatures. It is observed that the grain sizes in the material processed by HPT are smaller than their counterparts processed by extrusion at all annealing temperatures. The average grain sizes are also smaller than the grain size of the material processed by ECAP when annealing at temperatures higher than ~500 K.

5. Summary and conclusions

1. A magnesium alloy AZ31 was processed by multiple turns of HPT at room temperature and subjected to annealing at different temperatures. The microstructure and hardness evolution were tracked.
2. There is no evidence for a difference in the annealing behavior of samples processed to different numbers of turns of HPT.
3. The material undergoes recovery up to 423 K, recrystallization at 423 K and grain growth at higher temperatures.
4. The AZ31 alloy follows a linear trend of increasing hardness as a function of the inverse of the square-root of the grain size up to grain sizes in the range of 1 µm.
5. The grain size is within the range for superplasticity up to annealing at 673 K and the grain growth during annealing is smaller than other processing techniques.

Conflicts of interest

The authors declare no conflicts of interest.

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