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Properties of severe plastically deformed Mg alloys

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Abstract

In this present investigation, mechanical properties of bulk Mg alloys synthesized by mechanical alloying (MA) and equal channel angular pressing (ECAP) were investigated. For mechanically alloyed samples, a dramatic increase in yield strength to 498 MPa has been achieved after 5 h milling. Strain softening accompanied by increased ductility of the longer milled samples is observed through the gradual decrease in yield strength. The lowest yield strength of 200 MPa was observed after 30 h milling while the elongation at fracture was dramatically increased to 45%. The average grain size was about 75 nm for specimens milled for 30 h. In ECAP, the accumulation of redundant shear strain and dynamic recrystallisation refine the microstructure while increasing the material's yield strength. Consequently, ductility is reduced. The mechanical properties of the Mg alloy are also found to be sensitive to the processing temperature at which ECAP is conducted and to the number of ECAP passes.

Keywords: Mechanical alloying (MA); Equal channel angular pressing (ECAP); Severe plastic deformation; Mechanical behaviour

1. Introduction

Mg alloys have received much attention in various engineering applications due to their high specific strength and stiffness. However, Mg alloys suffer from poor plasticity due to their hexagonal close packed (hcp) structure. Currently, most of the commercially available Mg alloys are either die casted or hot worked to minimize production costs. AZ (Mg–Al–Zn) and AM (Mg–Al) series are the most common and popular Mg alloys in which Al serves principally as a solid solution strengthening element to improve strength. However, when the content of Al is above 6 wt.%, the intermetallic $Mg_{17}Al_{12}$ is formed which leads to a further decrease in ductility. Most of the AZ and AM series alloys have yield strengths which are less than 250 MPa and ductility of less than 10%.

The strength of Mg can be improved by solid solution strengthening, particle dispersion strengthening and grain size refinement [1]. Processing methods such as inert gas condensation, electro-deposition, mechanical milling and/or alloying, equal channel angular pressing and rapid solidification have been used in the attempt to improve the mechanical properties of Mg alloys. Of all these techniques, mechanical alloying/milling currently produces the most impressive results in achieving solid solution strengthening, alloy formation, particle dispersion strengthening and grain size reduction. This process has the ability to alloy powder solely in the solid state, avoiding melting and solidification [2]. Many advanced materials such as aluminides, intermetallics [3,4], metal matrix composites and nanocrystalline materials, oxide dispersion strengthened alloys and amorphization of powders [5] have been successfully processed by MA. Nevertheless, there are two major concerns with MA. Firstly, there is contamination during the milling process and secondly, grain growth occurs readily at high temperature during the consolidation of milled powders in conventional pressureless sintering. In addition, post processing is required to produce bulk parts. In most cases, the level of contamination can be controlled to within acceptable level by milling over shorter durations. Grain growth may be minimized by lowering the temperature of sintering or by applying some appropriate pressure. Inter-particle bonding may still be affected as low solid state diffusion rate cause

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poor bonding between the particles and thus limits the ductility. In view of the advantages, limitations and difficulties of MA process, ECAP is another promising method used to process Mg alloys. This processing method effectively fills the gap between MA (nano range grains) and traditional metal working (micron range grains). Some recent advances in ECAP have shown it to be an effective method to reduce grain size of materials like Al and Mg down to sub-micron ranges and to produce a good combination of strength, ductility and even super-plasticity at low temperatures [6–8]. The main advantage of ECAP lies in its ability to process bulk fine grained Mg alloys without external dimensional changes and at a relatively short amount of time. However, the control of the processing parameters is crucial in attaining the desired resultant properties and such parameters are unique to each material system being processed.

In view of the exciting potential of MA and ECAP in the processing of Mg alloys, the paper attempts to investigate and compare the mechanical properties of Mg5%Al processed by MA and Mg3%Al1%Zn (AZ31) by ECAP. The aim is to provide a clearer overview of the benefits and limitations of the two processing techniques to better tailor the processing methods to the materials' intended applications.

2. Experimental procedures

Elemental powders of Mg (purity >98.5%) and Al (purity 99.5%) were used to produce the Mg5%Al alloy. MA was carried out on a Fritsch planetary ball mill operating at 250 rpm using fifty one 15 mm diameter balls with a ball-to-powder weight ratio of about 20:1. A 2–3 wt.% of stearic acid, a process control agent, was added to the powder mixture to prevent agglomeration and excessive cold welding of powders. Prior to MA, the powder mixtures were sealed in milling vials filled with 99.9% pure argon gas. After mixing and MA, the powders were cold-compacted into cylindrical billets of 35 mm in diameter and 35 mm in length. The green compacts were then sintered at 500 °C in an argon furnace for 2 h followed by hot extrusion at an extrusion ratio of 25:1 at 350 °C.

Wrought AZ31 slabs were machined into cylindrical billets 35 mm in diameter. The billets were first directly extruded at 250 °C into 10 mm diameter rods which were sectioned into specimens of 80 mm in length. They were then annealed in an argon tube furnace at 500 °C for 24 h to fully remove defects and release any prior strain caused during fabrication and the direct extrusion process. The fully annealed specimens were then subjected to ECAP according to route B_C [9], respectively, at 150, 180, 220 and 250 °C. The ECAP die has a 10 mm diameter channel with $\Phi = 120°$ and $\Psi = 5°$, where Φ is the included angle at which the two channels meet and Ψ is the angle of curvature of the outside corner of the channel intersection. The die set was heated with a collar heating band and a thermocouple attached in the heart of the die provides temperature feedback and controls the temperature of the die to an accuracy of ±1 °C. Specimens were subjected up to a total of four ECAP passes at each temperature. The total strain of multiple passes is roughly equal to the summation of strains obtained from each individual pass [10,11]. The as-processed rods were transversely sectioned, polished and etched with di-ethylene glycol in nitric acid for microstructural studies.

The grain size of mechanically alloyed (MAed) powders and extruded samples was estimated by X-ray diffraction (XRD) using a Shimadzu Lab XRD-6000 X-ray diffractometer with Cu Kα diffraction. The strains induced broadening of the XRD diffraction peaks was removed using Williamson–Hall analysis. Microstructure of the ECAPed AZ31 was studied using an optical microscope and grain size was measured by linear intercept method, taking account of the high angled grain boundaries only. Hardness measurement was carried out using a Rockwell type micro hardness indentation machine. Tensile test of the specimens was performed using an MTS-810 mechanical testing system in accordance to ASTM E8-9 standard. Elongation was measured using a highly sensitive 25 mm strain gauge. A strain rate of $3.33 \times 10^{-4}\,s^{-1}$ was used throughout the tests.

3. Results and discussions

3.1. Structure and grain size

Structures of the MAed powders and ECAP processed AZ31 were analyzed by XRD. Fig. 1A and B show the XRD spectra of Mg5%Al alloy MAed at different durations before and after sintering, respectively. The Al(1 1 1) diffraction peak can be seen from the XRD spectrum of the un-milled specimen (Fig. 1). After MA, the Al peak receded as Al goes into solid solution with Mg and also formed $Mg_{17}Al_{12}$, as indicated by the (3 3 0) and (3 2 2) diffraction. The $Mg_{17}Al_{12}$ peak was observed in all the milled specimens. After sintering and extrusion, the intensities of the diffraction peaks varied at different crystallographic planes. The intensity of the Mg(0 0 2) peak is lowered after the extrusion. This is caused by the high extrusion ratio where the basal plane rotates preferentially in the direction of [1 0 1] to coincide with the extrusion direction. $MgAl_2O_4$ peaks were also observed in samples after sintering, especially in those milled for 30 h. Broadening of peaks was observed for longer milling durations, indicating a reduction in the grain size. Fig. 2A shows the average grain size of the mechanically alloyed samples before and after sintering. It can be seen that the average grain size obtained after 10 h of milling was below 70 nm and the smallest crystallite size obtained was about 20 nm after 30 h of milling. After sintering, there is a significant increase in the average grain size of the samples. The grain size of 5 h milled sample after post sintering and extrusion increased from 69 nm to an average of about 200 to 300 nm.