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Abstract

Some of the important mechanical properties, mainly strength and ductility of magnesium- based structural material can be improved by alloying element; grain size refinement of the alloy and reinforced with nano-particles. Each processing techniques have their own mechanism of strengthening and fracture. In this study, bulk monolithic Mg material and their alloy and composite were synthesised by powder metallurgy technique. The process involved consolidation of powders through iso-static compression followed by sintering and hot extrusion. The microstructural studies were conducted on the extruded sample through XRD test and microscopic observation. In order to measure the strength and ductility, the tensile tests for all the samples were carried out at room temperature. Significant improvement in strength and ductility are observed in the samples of alloys and composite when compared with the sample of pure Mg. Further improvement can also be seen in the refined grain size sample. The possible strengthening mechanisms for each case are discussed. It is speculated that the deformation mechanism in all the cases are controlled mainly by the mechanism of grain boundary movements. The fracture surfaces were examined to identify the possible fracture mode of the samples.

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1. Introduction

The evaluation of mechanical properties of light weight structural materials is an utmost necessity for their appropriate utilization. Magnesium (Mg) is one of the lightest (density $\sim 1.74 \text{gm/cm}^3$) and eighth most abundant metallic material. Their alloys and composites are considered as potential candidates in various engineering and structural applications due to their high specific strength, high damping capacity and good machinability [1, 2]. However, due to the limited slip system available in a hexagonal closed pack (HCP) crystal structure, Mg suffers from poor plastic ability and corrosion resistance. To overcome these limitations, continuous efforts were made towards the development of Mg based alloys or it's composite [3, 4]. Furthermore, the grain refinement enhanced the strength to those of coarse grain counterparts in accord with the Hall-Peach relationship [5]. For attaining better mechanical properties in Mg based materials, researchers have made successful attempts to develop various conventional and novel processing techniques [6, 7, 8, 9]. Among the various synthesis methods, powder metallurgy techniques such as consolidation of powders, and then sintering and extrusion serves as one of the effective approach to produce dense bulk Mg base metallic materials. The powder metallurgy technique is able to control the microstructure during the processing of composite, alloys and further leading to their grain refinement [10]. In this work, the effects of alloying element, reinforcement and grain refinement on the microstructure and their further effects on the mechanical behaviour of Mg based metallic materials are studied. Powder metallurgy approach was employed to synthesis of bulk samples of pure Mg, and their alloy and composite. The results are compared with those of pure Mg prepared by the same route.

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2. Experimental procedures

Commercial elemental powder of Mg with average particle size 60–300 µm and purity 98.5% (supplied by Merck, Germany) was used as base material. For alloy, 3% Al powder of particle size 50-100 µm (USA make) was mixed with Mg. While, 2% nano-sized Y₂O₃ (40-50 nm) particles were used as reinforcement for nanocomposite. Al was chosen as an alloying element due to its potential to improve strength as well as ductility in the Mg. Thermally stable hard nano-sized Y₂O₃ particles were used for composite. The nano-sizeY2O3 particles arrest the grain boundary movement, and thus control the grain growth which provides the additional strength in the Mg material. The mixtures of powders (Mg and Al, and Mg and Y₂O₃) were mixed in a milling machine for one hr. For grain refinement of the alloy, the mixture of Mg and 3%Al were ball-milled for 10 hrs. During ball milling, the micron size particles were crushed continuously by the hard stainless balls and producing near nano-sized particles. The milled and unmilled powders were then cold compacted in a cylindrical die by isostatic pressing (50 tons) to produce 35 mm diameter cylindrical billet. The billets of the alloy were sintered at 450°C for two hrs in an argon furnace, while the billets of composite and pure Mg were sintered using energy efficient microwave sintering process. The sintered samples were then extruded at 350°C with an extrusion ratio of 25:1 to produce 7 mm diameter cylindrical rod. Further details pertaining to the synthesis process of the bulk samples of Mg and their alloy and composite have been presented in ref. [11, 12]. The extruded rods were machined in CNC machine to produce dog-bone shaped tensile specimens with gauge diameter 5 mm and 25 mm gauge length in accordance with ASTM E8M-96 standard. To remove the flaws that occurred during the machining, the specimens were polished using 10 µm polishing cloth followed by ultrasonic cleaning. All the tensile tests were performed at room temperature in an Instron-8874 machine with an initial strain rate of 4×10^{-4} /sec. To identify the constituent phase and crystallographic structure of the mixture of powders and their bulk extruded samples, X-ray diffraction analysis were carried out by using Shimadzu Lab-6000 X-ray diffractometer with CuK_{α} diffraction ($\lambda = 1.5406$ Å). The machine was operated with a scanning speed of 2°/min at 30 kV and 40 Amp. The microstructures of the undeformed and deformed samples were examined using Hitachi S-4300 field emission scanning electron microscope (FESEM).

3. Results and discussion

The crystallographic structure and the phase constituent of the powder and bulk alloy and composite were studied by the XRD analysis. Fig.1 shows the X-ray diffractograms of unmilled and milled powder of Mg-3% Al. In the unmilled powder, the Al peak (1 1 1) and (2 0 0) are distinctly visible as shown in Fig.1(a). This result suggests the presence of Al powder which did not make the solid solution of Mg and Al. The Al peak disappears in the 10 hrs milled powder while the new phase of Mg₁₇Al₁₂ (3 3 0) can be observed in Fig.1(b). This result confirmed that the Al powder completely dissolved into Mg, and the solid solution of Mg and Al formed during milling.



Fig.1. X-ray diffractograms of (a) unmilled and (b) milled powder of Mg-3% Al.

Fig.2 depicts the XRD line profile of bulk sample of (a) pure Mg, (b) Mg/Y_2O_3 nanocomposite, (c) Mg-3%Al alloy and (d) ultrafine Mg-3%Al alloy. In Fig.2(a) only Mg peak was found, while in Fig.2(b) the Y_2O_3 (2 2 2) peak at 29.15° and Mg peaks were observed. This result suggests that during the processing of bulk sample, the nano-sized Y_2O_3 particulates did not make a solid solution but instead acted as reinforcement. On the other hand, Al peak was not identified in Fig.2(c) which revealed that the Al powder completely dissolved into Mg during compaction, sintering and hot extrusion. The Mg₁₇Al₁₂ peak can be identified in the bulk unmilled sample, whereas in the ultrafine sample the formation of oxide particle, MgAl₂O₄, was observed. The formation of MgAl₂O₄ is the result of chemical reactions of the fine particles of Mg, Al and Mg₁₇Al₁₂ along with oxygen during the synthesis process of bulk ultrafine sample. In the extruded sample, it can be observed that the intensity of Mg (0 0 2) peak has

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