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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A MAGNESIUM-ALUMINIUM-ERBIUM ALLOY

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Abstract

Magnesium alloys with their weight saving advantage exhibit unique application potential in the automotive and aerospace industries. Recent years have seen significant progress in the development of rare earth containing Mg alloys, as rare earth addition is considered a promising route to enhance the mechanical characteristics of Mg. In the present study, a new Mg alloy containing 5.16 at. % aluminum and trace (0.05 at. %) erbium was synthesized using the disintegrated melt deposition technique followed by hot extrusion. The microstructural and mechanical properties of the developed Mg-Al-Er alloy were evaluated in comparison to that of pure Mg. Microstructural investigation revealed significant grain refinement and the presence of $Mg_{17}Al_{12}$ intermetallic phases. Evaluation of mechanical properties under indentation loads showed significant improvement in microhardness by +50%. Under tensile loads, the developed Mg-Al-Er alloy exhibited +86%, +115% and +95% enhancement in yield strength, ultimate strength and ductility respectively. Similarly, an enhancement in yield strength by +115%, ultimate strength by +37% and ductility by +25% were observed under compressive loads. The overall effects of Al and Er addition on the mechanical properties of the Mg are discussed using structure-property relationship.

Introduction

The lightest structural metal, magnesium (Mg) has a density ~1.74 g/cm^3, which is only slightly higher than that of the plastics and only 2/3 that of aluminium (Al), and 1/4 that of iron [1]. Besides lightweight, Mg based materials also exhibit good specific mechanical properties, excellent damping capacity, machinability, castability and weldability. However, the inherent limitations of Mg such as low absolute strength, modulus, and ductility limit its extensive utilization. In order to circumvent these limitations, Mg is usually alloyed with other elements such as Al, Zn, Zr, etc. [2]. Despite considerable efforts, the adoption of magnesium alloys in engineering applications remains limited [1]. One important reason for the under-utilization of Mg is that there are limited Mg-alloys for the designers to select for specific applications, and within these limited choices, the most cost-effective magnesium alloys have inadequate properties such as yield strength, creep-resistance, formability, and corrosion resistance.

Recently, alloying of magnesium with rare earth (RE) elements has gained momentum in the context of improved mechanical characteristics due to RE additions [3]. In these Mg-RE alloys, the precipitation of a dense and fine of RE-containing dispersed phases within the grains, and along the grain boundaries generally strengthens the base metal [4, 5]. Further, the presence of RE phases within the α-Mg matrix also favors the formation of protective films to improve the thermal stability and corrosion resistance of the alloy [4]. In spite, RE additions are widely regarded as an expensive choice and the most commonly used RE containing Mg-alloys are limited to AE42 and AE44 which contains about 4 wt. % Al and 2 to 4 wt. % RE respectively [6, 7]. With this background, an attempt is made in this research work, to synthesize and characterize new Mg-Al alloy containing trace amount of RE element, erbium (Er). For this reason, an aluminium-erbium (AlEr) metal alloy powder was incorporated into pure Mg through disintegrated melt deposition (DMD) technique. DMD is a unique processing method that inherits the combined advantages of stir casting and spray processing techniques [8]. Unlike conventional spray processes, DMD method employs higher super heat temperatures and lower impinging velocity which helps in achieving a bulk material with no-over sprayed powders. Further, the DMD process also offers both the features of (i) fine grain structure of spray process and (ii) simplicity/cost effectiveness of conventional stir cast foundry process.

Experimental

Synthesis of Mg-Al-Er alloy

Mg-Al-Er alloy containing 5.16 at% Al and 0.05 at% Er, required for the current study was synthesized using disintegrated melt deposition (DMD) technique. It involves heating of Mg turnings (> 99.9% purity, supplied by ACROS Organics, New Jersey, USA) together with the aluminium-erbium master alloy powder (94.67% Al and 5.33% Er, supplied by Phoenix Scientific Industries, UK) in a graphite crucible to 750 ºC in an electrical resistance furnace under inert argon gas protective atmosphere. The superheated melt is then stirred (at 465 rpm for 5 min) and bottom-poured into the steel mold (after disintegration by two jets of argon gas oriented normal to the melt stream). Following deposition, an ingot of 40 mm diameter was obtained. The obtained ingot was then soaked at 400 ºC for 1 h and hot extruded at 350 ºC into rods of 8 mm diameter. The synthesis of pure Mg (required for the comparison purpose) also involved similar steps except that no Al-Er master alloy powder were added during the melting and casting.

Characterization of Mg-Al-Er alloy

Density and Porosity Measurements: The experimental mass density of the developed Mg-5.16Al-0.05Er alloy was determined using Archimedes' principle which involved weighing the polished samples (~10 mm length) prepared from the extruded rods in air and then in distilled water using an A&D ER-182A electronic balance with an accuracy of ± 0.0001 g. In order to calculate the theoretical density, the relation, ($\rho_{alloy} = \frac{100}{\left(\frac{C_1}{\rho_1}\right)+\left(\frac{C_2}{\rho_2}\right)+\left(\frac{C_3}{\rho_3}\right)}$; wherein 'C' corresponds to the individual elemental concentration in wt.%).

Microstructural Characterization: Microstructural studies were conducted on the polished samples in a direction perpendicular to the extrusion direction in order to determine the average matrix grain size, the presence and distribution of second phases. For this purpose, a small section (8 to 10 mm in length) was cut from the extruded rod and the ends were then ground using 600 and 1200 grit size sand paper in order to remove the large surface scratches and also to produce a flat surface. Once all visible surface scratches and cracks were removed, the sample was polished using a polishing disc with 5 micron diamond slurry, followed by 1 micron diamond slurry and lastly by 0.3 micron alumina slurry. The surface of the polished samples was then etched (to make the grain boundaries visible) with citral (4.2 g citric acid monohydrate in 100 ml of water) and observed under an Olympus optical microscope. Using selected optical micrographs, a total of 120–150 grains were selected to calculate the grain size using the Scion Image analysis software. In order to study the morphology and distribution of secondary phases, the as-polished samples were etched with acetic picral (10 ml acetic acid, 10 ml water, 70ml picral (95% ethanol + 5% 2,4,6-Tri-nitro-phenol)) and observed in a field emission scanning electron microscope (FESEM-S4300, Hitachi Ltd., Tokyo, Japan, SEI mode).

Mechanical Properties: The mechanical properties of the developed Mg-Al-Er alloy were evaluated under indentation, tension and compression loads. Microhardness measurements were carried out on the as-polished samples of developed materials using Matsuzawa MXT 50 automatic digital Microhardness tester in accordance with ASTM standard E3 84-99. Standard ASTM test methods E8/E8M-13a and E9–09 were conducted on the test samples using a fully automated servo-hydraulic mechanical testing machine, to determine the tensile and compressive properties of the developed materials. The fractured surfaces of Mg-materials after tensile and compression tests were studied using the Hitachi S-4300 FESEM.

Results and Discussion

Synthesis

Synthesis of pure Mg and the Mg-5.16Al-0.05Er alloy was successfully carried out by the DMD process followed by hot extrusion. Visual observation of the as-cast ingot and the extruded samples indicates the absence of any macro-defects which clearly designates the suitability of processing parameters used in this study. There was no reaction between the Mg melt slurries with the graphite crucible, which attributes to the inability of Mg to form stable carbides. Also the inert Ar gas atmosphere used during the melting and casting stage has prevented the oxidation of Mg melt.

The results of density measurements conducted on the pure Mg and Mg-Al-Er alloy are tabulated in Table 1. From the tabulated values, it can be seen that near-dense materials have been developed in this study. The results also show an increase in the density due to AlEr addition which can be attributed to the higher density of Al (2.7 g/cm^3) and Er (9 g/cm^3) as compared to Mg (1.738 g/cm^3). The volumetric porosity calculated from the theoretical and experimental density values indicate that the developed Mg-5.16Al-0.05Er alloy exhibit a relatively higher porosity level when compared to pure Mg. However, it is relatively low compared to those materials synthesized using similar technique [8].

Microstructure

The microstructural properties of the developed Mg-Al-Er alloy are studied in terms of: (a) grain morphology, and (b) the presence, distribution, and morphology of the second phase particles. Fig. 1 shows the microstructural characteristics of pure Mg and Mg-5.16Al-0.05Er alloy. It reveals the presence of nearly equiaxed grains in both the cases which indicates the complete recrystallization of Mg-matrix during extrusion. Using Scion Image processing software, the average grain size and aspect ratio of Mg-matrix (Fig. 1(a-b)) were calculated and the results are presented in Table 1. It shows that the addition of AlEr alloy to Mg has refined the average grain size of Mg-matrix [9]. It is known that the addition of Al to Mg would result in the formation of $Mg_{17}Al_{12}$ grain boundary eutectic network as shown in Fig. 1c. This shows that the noticeable grain refinement (~ 85%) observed due to AlEr addition could be attributed to the formation of $Mg_{17}Al_{12}$ intermetallic phase at the grain boundaries near the triple point grain edges and its efficacy in inhibiting the matrix grain growth during recrystallization. The incorporation of Er (in the form of AlEr alloy) is also expected to form Er based intermetallic phases that can influence the dynamic recrystallization effects. However, Er based intermetallic phases were not microscopically detected in the developed Mg-alloy. This could be possibly due to the very low amount of Er (0.05 at %) to effectively form any second phases with Mg or Al.

Mechanical behavior

The results of mechanical property measurements conducted on pure Mg and Mg-5.16Al-0.05Er alloy are summarized in Table 2. It reveals that the developed Mg-5.16Al-0.05Er alloy exhibit superior mechanical properties when compared to pure Mg.

The microhardness measurements shown in Table 2 indicate an increase in the mean hardness value (by 50%) when compared to pure Mg. This increase can be attributed primarily to (i) the increase in the presence of harder intermetallic phases $Mg_{17}Al_{12}$ with an increase in amount of Al, and (ii) the reduction in grain size due to AlEr addition [11].

Table 1 - Results of Density and Microstructural Measurements

Material	Density and Porosity			Microstructure	
	Theoretical Density [g/cc]	Experimental Density [g/cc]	Porosity [%]	Average grain size [μm]	Aspect ratio
Pure Mg	1.7380	1.7366 ± 0.0011	0.26	59 ±16	1.55
Mg-5.16Al-0.05Er	1.7786	1.7731 ± 0.0021	0.96	9 ± 2	1.23

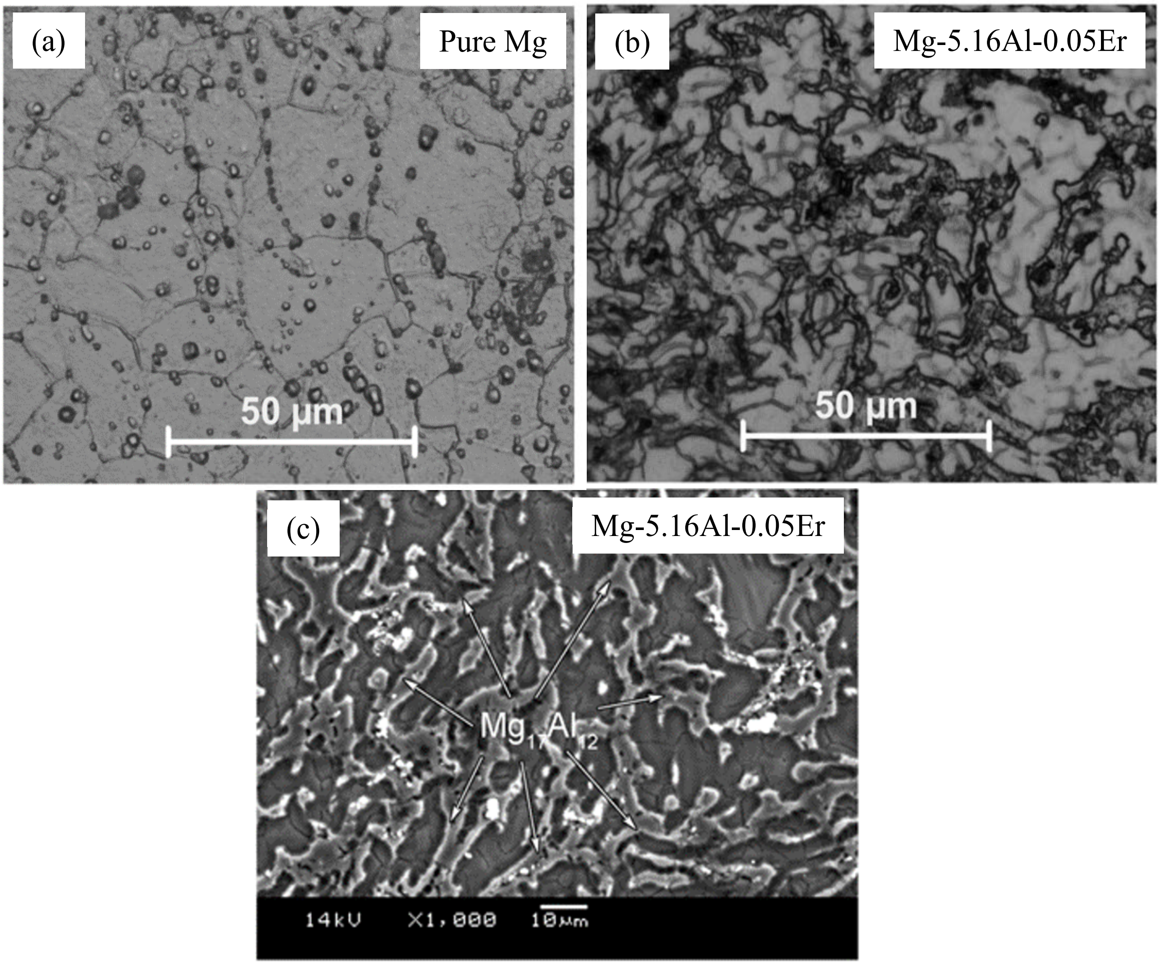

Figure 1 - Microstructures of (a) pure Mg and (b,c) Mg-5.16Al-0.05Er alloy.

The mechanical properties of pure Mg and Mg-5.16Al-0.05Er alloy as listed in Table 2 indicates that the alloying addition of AlEr to Mg has enhanced the tensile and compressive properties of pure Mg. Significant improvement in both yield strength and ultimate strength (under both tensile and compressive loading) was achieved by the addition of Al and Er alloying elements in the form of AlEr alloy powder. Further, it is interesting to note that the developed Mg-5.16Al-0.05Er alloy displayed an increase in tensile and compressive ductility by ~95% and ~25% respectively, when compared to pure Mg. Based on the microstructural analysis; it is evident that the addition of AlEr to Mg has refined the matrix grain size significantly. Hence, in the Mg–Al–Er alloy, the improvement in strength properties under tensile and compressive loads can be attributed to the following strengthening mechanisms:

(i) Strengthening due to the grain size reduction and formation of refined and equiaxed grains [12],
(ii) Strengthening from the reasonably distributed hard grain boundary second phases ($Mg_{17}Al_{12}$) that impedes the stress transfer across the grain boundaries.

The ductility increment due to AlEr addition could be attributed to the activation of non-basal slip systems as suggested by Koike et al. [13] wherein, the non-basal slip activation resulting larger deformation strain (elongation) was observed in the case of fine-grained Mg-alloys.

Table 2 - Results of Mechanical Property Measurements

Materials	Micro-hardness	Tensile Properties			Compressive Properties		
		0.2Offset Yield Strength	Ultimate Tensile Strength	Failure Strain	0.2Offset Yield Strength	Ultimate Tensile Strength	Failure Strain
	[H_v]	[MPa]	[MPa]	[%]	[MPa]	[MPa]	[%]
Pure Mg	69 ± 1	96 ± 8	133 ± 7	8.3 ± 2.9	81 ± 4	368 ± 11	20.1 ± 0.9
Mg-5.16Al-0.05Er	103 ± 7	182 ± 11	285 ± 11	17.5 ± 1.2	175 ± 11	504 ± 8	24.6 ± 0.5

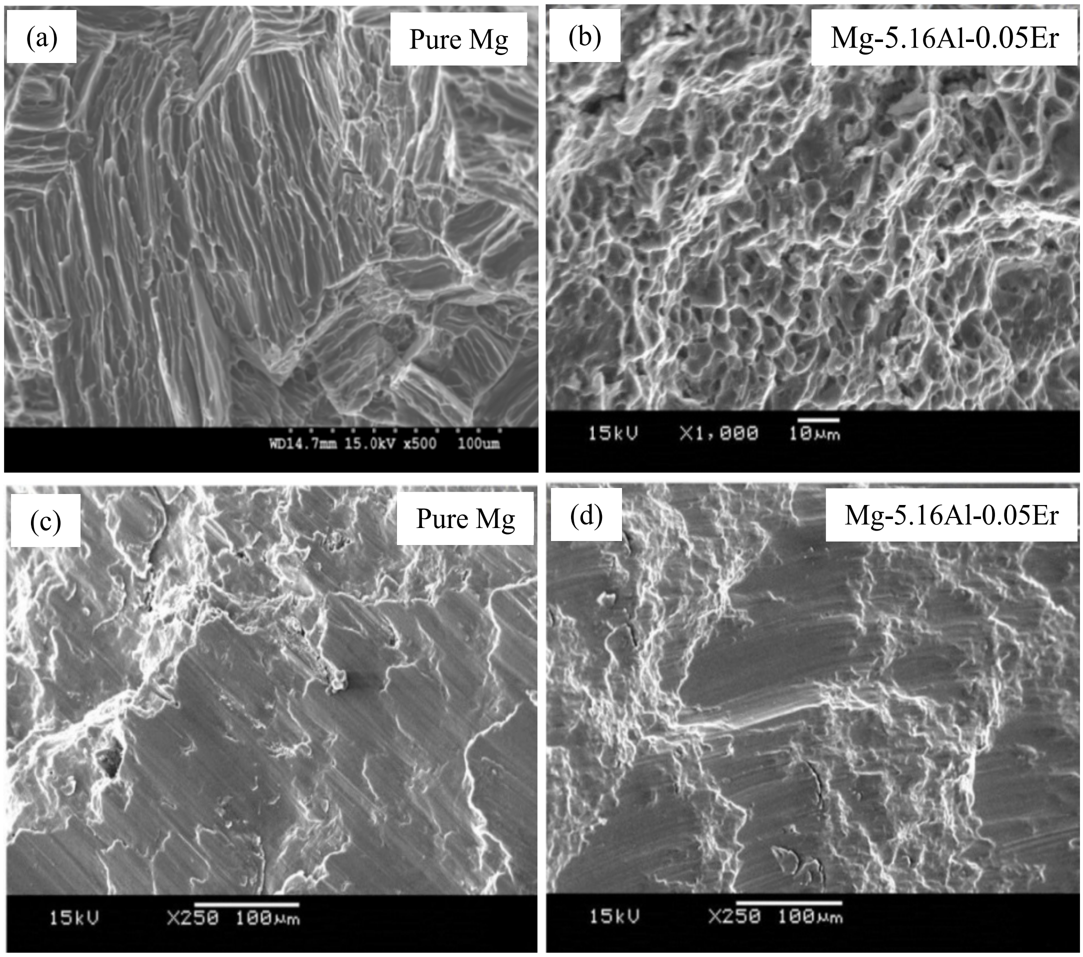

Figure 2 (a-b) Tensile and (c-d) Compressive Fracture Surfaces of Pure Mg (a,c) and Mg-5.16Al-0.05Er alloy (b,d).

The microscopic features of the tension test fractured samples are shown in Fig. 2(a-b). It reveals a typical mixed mode fracture with prominent cleavage features in the case of pure Mg and prominent ductile features in the case of Mg–5.16Al–0.05Er alloy. Typically, the cleavage features observed in magnesium are indicative of the inability to plastic deformation attributed to its HCP crystal structure. The compressive fracture surfaces of both pure Mg and the developed Mg-Al-Er alloy showed shear mode fracture as indicated by the presence of prominent shear bands (Fig. 2(c-d)). However, for Mg–5.16Al–0.05Er, the fracture behavior was observed to be relatively more ductile as indicated by Fig. 2(c-d).

Conclusions

In the present work, a new Mg-alloy containing 5.16 at.% Al and trace (0.05 at.%) Er was produced using the disintegrated melt deposition technique followed by hot extrusion. From the microstructural and mechanical property characterization, the following conclusions can be drawn:

1. The addition of Al and Er in the form of AlEr master alloy powder to Mg resulted in Mg-Al-Er alloy with refined microstructure (~85% grain refinement) containing evenly distributed $Mg_{17}Al_{12}$ intermetallic phases.
2. The developed Mg-5.16Al-0.05Er alloy displayed superior mechanical properties under indentation, tension and compression loads when compared to pure Mg.
3. Microhardness measurements showed ~50% increment in hardness value of Mg due to AlEr addition.
4. Under tensile loads, the developed Mg-Al-Er alloy exhibited +86%, +115% and +95% enhancement in yield strength, ultimate strength and ductility respectively.
5. Under compression loads, an enhancement in yield strength by +115%, ultimate strength by +37% and ductility by +25% were observed.

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