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Nano-ZnO particle addition to monolithic magnesium for enhanced tensile and compressive response

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ABSTRACT

In this study, the effects of nanoscale ZnO reinforcement on the room temperature tensile and compressive response of monolithic Mg were studied. Experimental observations indicated strength properties improvement due to nanoscale ZnO addition. A maximum increment in tensile yield strength by ~55% and compressive yield strength by 90% (with reduced tension–compression asymmetry) was achieved when 0.8 vol.% ZnO nanoparticles were added to Mg. While the fracture strain values under tensile loads were found to increase significantly (by ~95%, in case of Mg–0.48ZnO), it remained largely unaffected under compressive loads. The microstructural characteristics studied in order to comprehend the mechanical response showed significant grain refinement due to grain boundary pinning effect of nano-ZnO particles which resulted in strengthening of Mg. Texture analysis using X-ray and EBSD methods indicated weakening of basal fibre texture in Mg/ZnO nanocomposites which contributed towards the reduction in tension–compression yield asymmetry and enhancement in tensile ductility when compared to pure Mg.

1. Introduction

In recent years, there has been an increasing demand for advanced structural materials with excellent weight saving potential in order to satisfy the growing economic and environmental concerns such as fuel price inflation, greenhouse gas emission [1–3]. Especially in the automotive and aircraft industries, the current research works are more focussed on the extensive utilization of magnesium materials which offers superior combination of properties [1–4]. Magnesium (with density 1.74 g/cc), which is ~75% lighter than steel and ~35% lighter than aluminium attracts extensive research interest in terms of weight saving potential in critical engineering applications. For example, in a V6 engine cylinder block, the replacement of cast iron by magnesium (Mg) would reduce the weight from 86 kg to 30 kg [5]. Similarly, the possible replacement of other structural components by Mg would contribute to ~100 kg reduction in weight which could reduce the fuel consumption by ~500 ml per 100 km and the fuel emission by ~5% [6,7]. Besides weight savings, Mg also exhibit a range of influential properties such as excellent damping capacity, castability, machinability and dimensional stability. Further, the relatively lower working temperature (~250 °C) of Mg when compared to other structural metals also attracts its usage in the perspective of energy conservation and extended die/tool life [1–7].

Being the designers' choice for weight critical applications, the commercial utilization of Mg however demands optimization of mechanical properties, especially the ductility. At room temperature, the ductility of Mg is often observed to be poor owing to the limited slip systems available in hexagonal closed packed (HCP) crystal structure and the recent research efforts are largely focussed on improving the room temperature ductility of Mg [8–11]. In this context, the incorporation of nanoscale reinforcements into Mg has positively influenced the ductility of Mg. The recent review article [12] comprehensively illustrates the beneficial role of various nanoscale ceramic reinforcements on the mechanical properties of Mg and Mg alloys. It showed that the ceramic particles such as SiC, Al_2O_3, SiO_2 and Y_2O_3 were extensively used as nanoreinforcements to improve the strength and ductility of Mg and Mg alloys. The nanocomposites referred to in this review article [12] generally exhibited superior mechanical response and they were prepared using the processing methods such as liquid processing route based disintegrated melt deposition (DMD) method, solid state processing route based

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blend-press-sinter technique, ultrasonic assisted liquid processing, acoustic cavitation and friction stir processing. Hassan and Gupta [10] used both the solid state and liquid state processing methods to develop superior Mg nanocomposites containing various oxide reinforcements such as Al_2O_3, Y_2O_3 and ZrO_2. Choi et al. [13] synthesized nano-SiC particles reinforced Mg composites using the ultrasonic cavitation method and reported that the nanoscale SiC particles have positively influenced the strength properties of Mg without prominent effect on ductility. Various other researchers have also reported similar mechanical property enhancement due to nanoscale reinforcement addition [11,14–17]. In this context, the effect of nanoscale boron carbide particle addition on the microstructural evolution and tensile response of pure magnesium was recently reported and the results showed that the incorporation of nanoscale B_4C particles to Mg have resulted in texture randomization and significantly improved the tensile ductility [18]. From this background, it can be clearly understood that the efficient dispersion of nanoscale reinforcements improves the mechanical properties of Mg.

In this work, an attempt is made to improve the mechanical response of Mg through the incorporation of nanoscale ZnO particles. The Mg/*x*-ZnO nanocomposites for this study were synthesized using liquid-state processing, followed by hot extrusion. The effect of varying volume fractions of nano-ZnO particles on the microstructural and mechanical properties of pure Mg is investigated. Further, the evolution of crystallographic texture in pure Mg due to the incorporation of nano-ZnO particles has been studied in detail using electron back scattered diffraction (EBSD). Structure–property relationship is used to understand the observed mechanical behaviour of the nanocomposites.

2. Experimental procedure

Mg ingot of 99.8% purity supplied by Tokyo Magnesium Company, Japan was used as the matrix material and ZnO particulates (size <200 nm and average particle size – 90 nm) of 99.9% purity supplied by Nanostructured and Amorphous Materials, USA were used as the reinforcing phase. The synthesis of Mg/*x*-ZnO ('*x*' *refers to the vol.% of ZnO nanoparticle*; *i.e. 0.16 vol.%, 0.48 vol.% and 0.8 vol.% in Mg–0.16 ZnO, Mg–0.48 ZnO and Mg–0.8 ZnO respectively*) composites were carried out using the disintegration melt deposition (DMD) technique followed by hot extrusion. As the procedures involved in the synthesis of Mg/*x*-ZnO nanocomposites were identical to those mentioned in earlier work [14], the extensive discussion is hence omitted here for brevity.

From the extruded rods of developed Mg/ZnO composites, dog-bone shaped round samples of 5 mm gauge diameter and 25 mm gauge length were machined and then tested for its smooth bar tensile properties in accordance with ASTM: E8/E8M-13a using a fully automated servo-hydraulic mechanical testing machine, Model-MTS 810 fitted with an Instron 2630-100 series extensometer [18]. The extruded samples of synthesized Mg/ZnO nanocomposites were examined using an Olympus metallographic microscope, a Hitachi S-4300 field emission scanning electron microscope coupled with Energy Dispersive Spectroscopy and an automated Shimadzu LAB-X XRD-6000 X-ray diffractometer (Cu Kα radiation, λ = 1.54056 Å) to determine the average matrix grain size, its morphology and distribution, the presence and distribution of nano-reinforcement and the interface between the matrix and reinforcing phase. The diffraction pattern obtained from the cross section and longitudinal section of the extruded rods were also used for X-ray texture analysis. An FEI Field Emission Gun Scanning Electron Microscope (FEG-SEM) equipped with electron back scattered diffraction (EBSD) detector and EDAX TSL software V6.1 were used for Orientation Image Microscopic (OIM) analysis to calculate the inverse pole figures (IPF), pole figures (PF) orientation distribution function (ODF) and kernel average misorientation (KAM). The EBSD scans were recorded on the surface parallel to the extrusion direction (ED).

3. Results and discussion

The results of tensile and compressive property measurements conducted on the synthesized Mg/*x*-ZnO composites containing different amounts of ZnO nanoparticles are listed in Table 1. Their representative engineering stress–strain curves are shown in Fig. 1(a and b). Under tensile loading, the results indicate an increase in the 0.2 offset Tensile Yield Strength (0.2TYS), Ultimate Tensile Strength (UTS) and ductility values due to nanoscale ZnO particle addition when compared to its monolithic counterpart. While the maximum increase in both the strength values (0.2TYS and UTS) occurred in case of Mg–0.8ZnO nanocomposite, Mg–0.48ZnO exhibit superior tensile ductility (Table 1). Under compressive loading, the incorporation of nano-ZnO particles has resulted in a significant increase in strength without affecting the compressive ductility (Table 1). The significant effect of ZnO particle addition is profoundly observed in case of Mg/0.8ZnO nanocomposite wherein the compressive yield strength was enhanced by ~90% when compared to monolithic Mg.

The matrix grain characteristics of developed Mg/ZnO composites are shown in Fig. 2(a–d). The results of grain size measurements conducted on the optical micrographs (Fig. 2(a–d)) in terms of average grain size and aspect ratio are tabulated in Table 2. It shows that the addition of ZnO nanoparticles resulted in a reduction in the average grain size of pure Mg and the grain refinement was observed to be prominent in case of Mg–0.8ZnO composite wherein the average matrix grain size was reduced from 28 μm to 16 μm. The grain refining effects of ZnO in Mg and Mg–Zn alloy has been recently reported by Fu et al. [19], wherein ZnO was identified as a potential grain refiner in cast Mg alloys. The representative SEM micrographs showing the distribution of nano-reinforcement particles in the developed Mg/ZnO nanocomposites are presented in Fig. 3(a and b). It indicates that the individual nano-ZnO particles were prominently seen inside the Mg-matrix grains and clustered particles (of size few hundred nanometres) were observed near the grain boundaries. The observed relative uniform distribution of either individual nano-ZnO particles in Mg-matrix can be attributed to (i) packing of reinforcement particles inside the machined Mg ingot, (ii) extrusion process which is capable of breaking down the large sized clusters and (iii) argon assisted disintegration resulting in faster cooling [8,9,14,20]. However, the (i) wider size distribution of ZnO particle reinforcement (between 50 nm and 200 nm) and (ii) difference in density values between the Mg-matrix (1.74 g/cc) and ZnO-reinforcement

Table 1
Results of room temperature mechanical properties.

S. No.	Material	Tensile properties			Compressive properties		
		0.2% Offset yield strength (MPa)	Ultimate tensile strength (MPa)	Fracture strain (%)	0.2% Offset yield strength (MPa)	Ultimate compressive strength (MPa)	Fracture strain (%)
1	Pure Mg	95 ± 8	133 ± 7	8.3 ± 2.9	66 ± 8	228 ± 7	22.2 ± 0.9
2	Mg–0.16ZnO	119 ± 11 (~25%)	204 ± 9 (~55%)	15 ± 1.4 (~80%)	98 ± 5 (~50%)	284 ± 6 (~25%)	22.3 ± 0.5
3	Mg–0.48ZnO	131 ± 6 (~40%)	210 ± 8 (~53%)	16.3 ± 1.4 (~95%)	110 ± 2 (~67%)	308 ± 9 (~35%)	22.5 ± 1.2
4	Mg–0.8ZnO	147 ± 9 (~55%)	237 ± 8 (~80%)	11.88 ± 1.7 (~45%)	125 ± 8 (~90%)	345 ± 4 (~51%)	21.7 ± 2.1

* Numbers in bracket shows percentage improvement when compared to pure Mg.