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EFFECT OF BALL MILLED REINFORCEMENT ON MECHANICAL BEHAVIOR OF MAGNESIUM

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Abstract: *In this study, magnesium composites containing as received and ball milled Al particles were synthesized through powder metallurgy route incorporating microwave assisted rapid sintering technique followed by hot extrusion. The results revealed that strength and failure strain of the composite containing ball milled Al particles remained higher compared to the composite containing as received Al particles. Compared to monolithic Mg, Mg/1.626 Al(B) sample showed higher mechanical properties improvement with an increase of 78% in 0.2%YS, 79% in UTS, 87% in failure strain and 225% in WOF while for the Mg/1.626Al sample there was an increase of 51% in 0.2%YS, 53% in UTS, 65% in failure strain and 142% in WOF. The effect of as received and ball milled Al particles contribution on the enhancement of mechanical properties of Mg is investigated in this paper.*
Keywords: *ball milling, magnesium matrix composites, Al reinforcement*

1. INTRODUCTION

The ability of magnesium based materials to exhibit high specific mechanical properties and to offer significant weight savings has significantly fueled research activities in recent times, targeted primarily for their further development. Magnesium based materials are being actively pursued for weight saving applications owing to their low density and high specific mechanical properties [1–2]. Advantages of magnesium include high specific mechanical properties, high dimensional stability, high thermal conductivity, superior damping characteristics, good machinability, good electromagnetic shielding characteristics and recyclability. The limitations of magnesium include its low elastic modulus, rapid loss of strength with an increase in service temperature, poor creep resistance and poor ductility. These limitations are often circumvented by using stiffer and stronger reinforcements based on ceramic and metallic materials [3–4]. In recent years, several attempts have been made to use different types of reinforcement [5–7]. Whilst there have been number of studies to tailor the properties of magnesium using different types and amounts of reinforcement, no attempt has been made to study the effect of reinforcements in ball milled form on the microstructural and mechanical properties of magnesium composites [5-9]. Literature search also revealed that until recently, no studies have been carried out to develop and investigate magnesium reinforced with as received and ball milled Al particles using the powder metallurgy technique incorporating microwave assisted rapid sintering technique.
Accordingly, the present study was aimed at synthesizing magnesium composites containing as received and ball milled Al particles coupled with hot extrusion. Particular emphasis was placed to study the effect of Al ball milling on the mechanical properties of synthesized composites.

2. EXPERIMENTAL PROCEDURES

In this study, magnesium powder (Mg) of 99.5% purity (supplied by Merck, Germany) with particle size range of 60-300 μm was used as the matrix material while aluminum powder (Al) with particle size range of 7-15 μm (supplied by Alfa Aesar, USA) was used as reinforcement. In order to prepare different reinforcements for different composite formulations, the amount of Al particles was varied while 0.3wt% stearic acid was used as the process control agent (PCA). In the first stage, Al particles were blended with stearic acid for 1 hour using

RETSCH PM-400 mechanical alloying machine. In the second stage, steel balls were placed and the blended mixture was ball milled for 2 hours. Ball to powder ratio was kept at 20:1 and the speed of ball milling machine was set at 200 rpm during both blending and ball milling steps. Magnesium matrix composites were synthesized using powder metallurgy technique. The synthesis process involved blending pure magnesium powder with as received and ball milled Al particles in a RETSCH PM-400 mechanical alloying machine at 200 rpm for 1 hr. The blended powder mixture was compacted at a pressure of 97 bar (50-tons) to a billet of 40-mm height and 35-mm diameter using a 100-ton press. The compacted billet was sintered using an innovative hybrid microwave assisted 2-directional sintering technique. The billet was heated for 13 minutes to a temperature near the melting point of magnesium in a 900W, 2.45 GHz SHARP microwave oven. The synthesis of monolithic magnesium was carried out using similar steps without adding ball milled reinforcement. The sintered billets of monolithic and magnesium composites were hot extruded at a temperature of 350°C with an extrusion ratio of 25:1 using 150-ton hydraulic press. Before extrusion, the billets were coated with colloidal graphite and soaked at 400°C for 1 h. Final diameter of the rods obtained after the extrusion was 7mm.

The porosity of synthesized monolithic and composite materials was determined using experimental and theoretical densities. The densities of extruded samples in the as polished condition were determined using Archimedes principle. Theoretical densities of 1.74 g/cc for Mg and 2.699 g/cc for Al were used, respectively. The samples were weighed in air and when immersed in distilled water using an A&D ER-182A electronic balance with an accuracy of ±0.0001 g. Theoretical densities of the samples were calculated assuming there is no Mg/Al interfacial reaction. In order to calculate theoretical density, rule of mixture was used. Microstructural characterization studies were conducted on metallographically polished monolithic and composite extruded samples with the use of HITACHI FE-4300 Field Emission Scanning Electron Microscope to determine: (a) grain size (b) grain morphology and (c) presence and distribution of reinforcements. X-ray diffraction analysis were carried out on the as received and ball milled Al particles using an automated Shimadzu LAB-X XRD-6000 diffractometer to identify different phases. The samples were exposed to Cu Kα radiation (λ = 1.54056 A°) at a scanning speed of 2 deg/min. Microhardness measurements were made on the polished samples of extruded monolithic Mg and Mg/Al composites rods using a Matsuzawa MXT 50 automatic digital microhardness tester. The microhardness test was performed using a Vickers indenter under a test load of 25 gf and a dwell time of 15 s in accordance with the ASTM standard E3 84–99. Smooth bar tensile properties of the monolithic and composite samples were determined based on ASTM E8M-05 by using automated servo hydraulic testing machine (MTS 810) with a crosshead speed set at 0.254 mm min^{-1}.

3. RESULTS AND DISCUSSIONS

3.1. Macrostructural Characteristics

Macrostructural characterization conducted on the as-sintered billets revealed the absence of macrostructural defects such as circumferential or radial cracks. Following extrusion, no observable macro defects were observed on Mg, Mg/Al and Mg/Al (B) rods. The outer surface was smooth and free of circumferential cracks. This shows the ability of processing parameters and methodology used in this study to synthesize defect free and near dense monolithic Mg, Mg/Al and Mg/Al (B) composites.

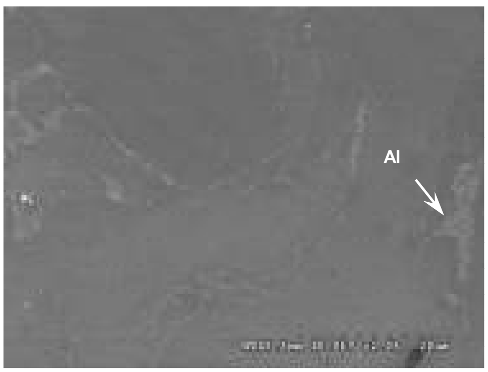
3.2. Microstructural Characteristics

Microstructural characterization results revealed fairly uniform distribution of both ball milled and as received Al particles (Fig. 1). Results also revealed that the average matrix grain size was significantly lower in the case of Mg/1.626Al and Mg/1.626Al (B) composites when compared to pure Mg suggesting ability of Al particles to serve as either nucleation site or obstacles to grain growth during processing (Table 1). However, compared to Mg/1.626Al composite, the extent of grain refinement in the case of Mg/1.626Al (B) composite was higher. No significant change was observed in the aspect ratio of the grains due to addition of either as received or ball milled Al particles. Near-equiaxed grain morphology was observed for both monolithic and reinforced samples indicating that the chosen extrusion temperature was high enough to allow for the recrystallization of strain free grains during extrusion [10]. According to Table 1, compared to monolithic Mg, the porosity of composites reinforced with either as received or ball milled Al particles decreased. However, this reduction in porosity is more evident in the case of Mg/1.626Al (B) composite.

X-ray diffraction studies showed that due to ball milling no second phase formed (Fig. 2). However, it revealed that Al_2O_3 partly exist in both as received and ball milled Al powder.

3.3. Mechanical Properties

The overall results of ambient temperature mechanical properties of the extruded materials are listed in Table 2. The results revealed that the composites exhibited significantly higher hardness compared to the monolithic material. The increase in composites hardness by contribution of either as received or ball milled Al particles as the reinforcement can commonly be attributed to (a) reasonably uniform distribution of Al particles in the matrix [8]; (b) higher constraint to localized matrix deformation during indentation due to the presence of second phase and (c) reduced grain size (see Table 1) [11-12]. Compared to Mg/1.626Al composite, higher hardness of Mg/1.626Al (B) composite can be attributed to smaller average matrix grain size (see Table 2). The results also revealed that compared to Mg/1.626Al composite, strength and failure strain of Mg/1.626Al (B) composite remained higher. Compared to monolithic Mg, Mg/1.626 Al(B) sample showed an improvement in mechanical properties with an increase of 78% in 0.2%YS, 79% in UTS, 87% in failure strain and 225% in WOF while for the Mg/1.626Al sample there was an increase of 51% in 0.2%YS, 53% in UTS, 65% in failure strain and 142% in WOF.



(a)

(b)

Figure 1: Representive micrographs showing distribution of Al particles through the matrix in (a) Mg/1.626Al and (b) Mg/1.626Al (B).

Table 1: Results of grain size and morphology

Material	Reinforcement (vol %)	Grain Size (μm)	Aspect Ratio	Porosity (%)
Mg	-	19 ± 4	1.5 ± 0.3	0.12
Mg/1.626Al	1.626	13 ± 4	1.6 ± 0.3	0.10
Mg/1.626Al (B)	1.626	9 ± 3	1.7 ± 0.3	0.07

Generally, the increase in tensile strength due to presence of Al particles as reinforcement can be attributed to a number of factors: (a) grain refinement [11-12]; (b) Orowan strengthening (in the case of Mg/1.626Al (B)) [13-15]; and (c) generation of geometrically necessary dislocations to accommodate CTE mismatch between the matrix and the reinforcement due to different CTE value between the matrix and reinforcement [13-15].

Table 2: Results of room temperature tensile properties

Material	0.2% YS (MPa)	UTS (MPa)	Failure Strain (%)	WOF (J/m^3)*	Micro Hardness (HV)
Mg	93 ± 1	153 ± 07	7.9 ± 3.4	12 ± 5	40 ± 2
Mg/1.626Al	140 ± 6	234 ± 11	13.0 ± 1.8	29 ± 3	52 ± 3
Mg/1.626Al(B)	166 ± 6	273 ± 02	14.8 ± 0.9	39 ± 2	55 ± 4

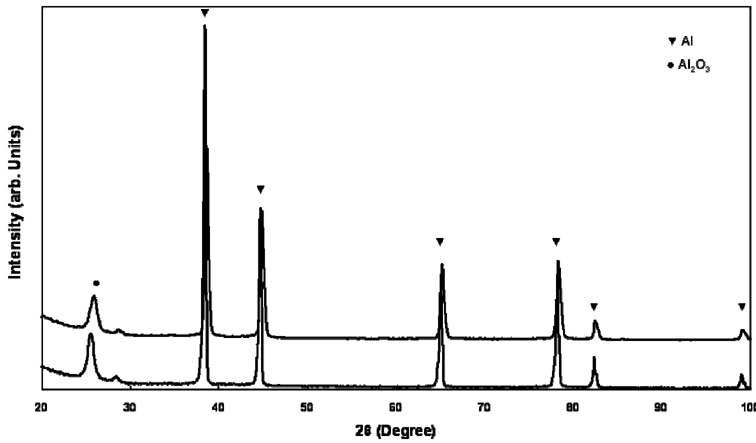


Figure 2: Representative XRD spectra of as received and ball milled Al powder.

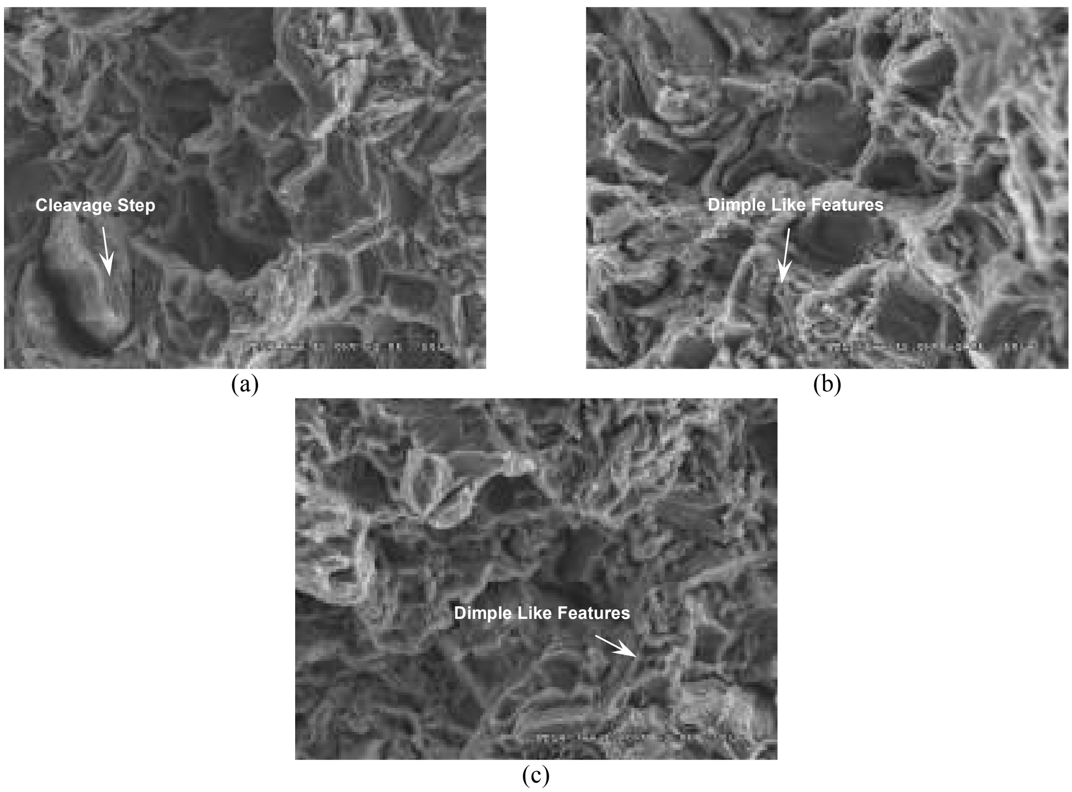


Figure 3: Representative FESEM micrographs taken from the tensile fracture surface showing (a) cleavage steps in pure Mg and mixed fracture mode in (b) Mg/1.626Al and (c) Mg/1.626Al (B) composites.

Regarding (a), the strengthening of composites from grain size reduction fundamentally comes from the mutual disturbance of slip among the grains. Here, the motion of dislocations across the grain boundary is impeded and the yield stress can be estimated by the Hall-Petch equation [9-10]. The contribution of this strengthening mechanism in the case of Mg/1.626Al (B) composite due to having smaller average matrix grain size compared to Mg/1.626Al composite (Table 1) is manifested. Thus, higher strength increase in the case of Mg/1.626Al (B) composite can be mostly assigned to its smaller average matrix grain size. Regarding (b), a dislocation line is known to loop around a particle in the way of its advancement, provided the particle is sufficiently formed and has an atomically non-coherent interface with the matrix [12]. In the case of Mg/1.626Al (B) composite, due to size of ball milled Al particles (0.460-0.688μm), Orowan strengthening can be also responsible in increased strength. Regarding (c), the difference in coefficient of thermal expansion (CTE) between the matrix and reinforcement (28.9×10^{-6} $K^{-1}$for Mg, 26.49×10^{-6} K^{-1} for Al and 7.4×10^{-6} K^{-1} for Al_2O_3 [16]) contributes to

generation of geometrical dislocations near the matrix-reinforcement interface. The pile up of these dislocations is responsible for the increased strength of composites [14-15].
The increase in failure strain due to the presence of either as received or ball milled Al particles when compared to pure Mg can be attributed to: (a) grain refinement (see Table 1) [17] (b) presence of reasonably uniformly distributed Al particles (Fig. 1) [18] and (c) activation of a non-basal slip system due to presence of either as received or ball milled Al particles [19]. The results of fracture surface analysis revealed a typical brittle fracture in the case of pure Mg sample (Fig. 3a). This can be attributed to the hexagonal close packed crystal structure of magnesium that restricts the slip to the basal plane. The presence of cleavage steps indicates the inability of magnesium to deform significantly under uniaxial tensile loading. However, the fracture surface in the case of Mg/1.626Al and Mg/1.626Al (B) composites had a higher occurrence of small dimple-like features compared to that of monolithic Mg. The involvement of shear and formation of dimples during deformation and fracture can be attributed to shear localization around: (a) second phase particles and (b) voids in the deformed matrix surrounding the second phase particles, in Mg/Al and Mg/Al (B) composites [20].
The tensile work of fracture (WOF) of monolithic Mg, Mg/1.626Al and Mg/1.626Al (B) composites are listed in Table 2. WOF express the ability of a material to absorb energy up to fracture under a load. The significantly high increment in tensile WOF exhibited by Mg/1.626Al and Mg/1.626Al (B) show their potential to be used in damage tolerant design.

3. CONCLUSIONS

1. Conventional solid state powder metallurgy technique using rapid microwave sintering and hot extrusion can be successfully used to synthesize near dense Mg composite containing as received and ball milled Al particles.
2. Reinforcement ball milling led to significant improvement in composites strength. Compared to monolithic Mg, tensile strength of Mg/1.626Al composite was enhanced with an increase of 51% and 53% in 0.2%YS and UTS, respectively while for Mg/1.626Al (B) composite the improvement were 78% and 79%, respectively.
3. Reinforcement ball milling led to improvement in composites failure strain. Compared to monolithic Mg, failure strain of Mg/1.626Al was enhanced by 65% while for Mg/1.626Al (B) composite the failure strain improvement was 87%.
4. Compared to monolithic Mg, Mg/1.626Al and Mg/1.626Al (B) exhibited significant improvement of 142% and 225%, respectively, in WOF due to their simultaneous increase in strength and failure strain.

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