Enhancing the Ductility of Mg-(5.6Ti+3Al) Composite Using Nano-B₄C Addition and Heat Treatment

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Received: October 28, 2013; Accepted: December 05, 2013; Published: December 16, 2013

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Abstract

This study investigates the effects of nano-B₄C addition and isothermal heat treatment on the microstructure and mechanical properties of Mg-(5.6Ti+3Al) composite developed through the disintegrated melt deposition method followed by hot extrusion. The developed Mg composites were characterized for their microstructural and mechanical properties in the as-extruded and heat treated conditions. Microstructural studies reveal no significant changes to the pre-existing Al₃Ti intermetallic phases and the average grain size, due to nano-B₄C addition. In the as-extruded condition, mechanical properties measurements showed large improvement in fracture strain without significant changes in strength properties due to nano-B₄C addition. The best combination of strength and ductility observed in Mg-(5.6Ti+3Al)-2.5B₄C composite was attributed to combined presence of nano-B₄C particulates and Al₃Ti intermetallics. In the isothermal heat-treated condition (200°C for 5 hours), all the developed Mg composites exhibit significant enhancement in ductility with marginal reduction in strength due to the stress relaxation at matrix-reinforcement interface.

Keywords: Magnesium Metal Matrix Composites; Nano-reinforcements; Heat treatment; Microstructure; Mechanical Properties

Introduction

Magnesium (Mg) materials with low density (1.74 g/cc) exhibit tremendous application potential in weight critical applications such as in automobile and aerospace sectors. However, the mechanical characteristics such as poor elastic modulus and low absolute strength (especially at high temperature) restrict its utilization in critical engineering applications [1-4]. To overcome these limitations, high strength and high modulus ceramic reinforcements such as Al₂O₃, SiC etc. are conventionally incorporated into Mg-matrix to form Mg-metal matrix composites [2,5]. In addition to the above mentioned conventional ceramic reinforcements, hard, high modulus and high strength intermetallics are also being introduced into Mg matrix as reinforcements and these intermetallic reinforcements were prepared either in-situ or through external processing [6-8]. In this regard, our recent work on the development of Mg-(5.6Ti+3Al) composite containing (Ti+Al₃Ti) phases also confirms

the possibility of utilizing the externally processed intermetallics as a potential reinforcement to develop new Mg-composites [9]. In this study, the Ti and Al particulates were ball milled for 2 hours to form (Ti+Al₃Ti) composite powder and the resulting ball milled mixture was used as composite reinforcement in Mg matrix. The mechanical properties characterization of the developed Mg-(5.6Ti+3Al) composite showed enhanced strength properties at the expense of ductility [9]. Hence, the current research work is aimed at improving the mechanical response, in particular, the ductility of the Mg-(5.6Ti+3Al) composite.

Available literature reveals that the ductility improvement in Mg materials can be attained through the addition of nanoscale ceramic reinforcements such as Al₂O₃, Y₂O₃, ZrO₂ etc. [10-14]. While the oxide nanoparticulates such as those mentioned above have been widely used as nano reinforcements in Mg and Mg-alloys, research work on carbide nanoparticle addition to Mg (such as SiC, TiC or B₄C) is relatively meager [7,10-13]. Hence in this study, an attempt is made to investigate the effect of nanoscale B₄C particles addition to Mg-(5.6Ti+3Al) composite. The developed Mg composites were then subjected to isothermal heat treatment (HT) at 200°C as it would enhance the ductility by minimizing the matrix-reinforcement residual stresses [15,16].

Materials and Methods

Synthesis

Mg turnings of > 99.9% purity (ACROS Organics, USA), was used as the matrix material. The metallic additions include Ti (< 140 m, 98% purity) supplied by Merck, Germany and Al (< 40 m, 98% purity) particles supplied by Alfa Aesar, USA. Nano-B₄C particles (~ 50 nm, 99 % purity) supplied by Nabond, China was used as the nano-ceramic addition.

The synthesis of Mg-(5.6Ti+3Al) composite involves pre-processing of 5.6 wt. % Ti and 3 wt. % Al particles by ball milling and its subsequent addition to pure Mg as reinforcement. A Retsch PM-400 mechanical alloying machine was used to ball-mill the Ti and Al particulates. Prior to ball milling, the particulate mixture was blended for 1 hour (with 0.3 % as process control

agent) to ensure the uniform mixing of powder particulates. After blending, hardened steel balls of 15 mm diameter were added and the blended mixture was ball milled for 2 h at 200 rpm, with ball to powder ratio of 20:1. This was done to allow the powder particulates to react with each other [9,17,18]. The ball milled (5.6Ti+3Al) mixture was then incorporated into Mg as a pre-processed reinforcement through the disintegrated melt deposition (DMD) technique. It involves the heating of Mg turnings together with the ball milled (5.6Ti+3Al) mixture in a graphite crucible to 750°C in an electrical resistance furnace, under inert argon gas protective atmosphere. The superheated molten slurry was then stirred at 465 rpm for 5 min using a twin blade (pitch45°) mild steel impeller (coated with Zirtex 25) to facilitate the uniform distribution of reinforcement particulates in Mg. The composite melt was then 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 40mm diameter was obtained.

Similar steps were employed to synthesize the Mg-(5.6Ti+3Al)-2.5B₄C composite except that 2.5 wt. % B₄C particulates were also added to Mg turnings, in addition to the ball milled (5.6Ti+3Al) mixture.

The composite ingots thus obtained from the DMD process were then machined to 36mm diameter and soaked at 400°C for 60 min. Hot extrusion was then carried out using a 150 T hydraulic press at 350°C with an extrusion ratio of 20.25:1 to obtain rods of 8mm in diameter. To study the role of heat treatment on the microstructural and mechanical properties of developed Mg-composites, isothermal heat treatment (HT) was carried out at 200°C for 5 h on the test samples prior to characterization of properties. The microstructural characteristics and mechanical properties of the developed Mg composites (before and after heat treatment) were evaluated through the experimental studies conducted on the samples cut from extruded rods.

Materials characterization

Microstructure: The microstructural features (matrix grain characteristics and distribution of reinforcements/second phases) of the developed Mg composites were studied on the as-polished and etched samples using an Olympus metallographic optical microscope (OM) and Hitachi S4300 field emission scanning electron microscope (FESEM). The microstructure of as received Ti, Al, and ball milled (5.6Ti+3Al) were also investigated to determine the mean particle size and morphology. Scion image analysis software was used for this purpose. X-ray diffraction (XRD) analysis was carried out on the ball milled powder and the developed Mg composites using an automated Shimadzu LABX XRD6000 diffractometer to identify the dominant major phases such as Mg, Ti, Al, B₄C and other related minor phases.

Mechanical properties: The mechanical properties of the developed Mg-materials were measured under indentation and tensile loading. A Matsuzawa MXT 50 automatic digital micro-hardness tester was used to measure the microhardness

of the developed Mg-composites. The microhardness test was conducted on the as-polished specimens of extruded Mg materials in accordance with the ASTM standard E3 84-99 with a Vickers indenter under a test load of 25 gf and a dwell time of 15 s [19]. The tests were conducted on three samples for each composition for 10 to 15 repeatable readings. To determine the tensile properties of the as-extruded samples, a fully automated servo-hydraulic mechanical testing machine, Model-MTS 810 was used in accordance with ASTM test methods E8/E8M-08 [20]. The crosshead speed was set at 0.254 mm/min. For each composition, a minimum of 6 tests were conducted to obtain repeatable values. The fractured samples under tensile loading of Mg-materials were analyzed using a Hitachi S-4300 FESEM.

Results and Discussion

Grain characteristics

The matrix grain characteristics (grain size and morphology) of the developed Mg-composites in the as-extruded condition are shown in Figure 1 (a & b) and Table 1. While the composites display refined microstructure when compared to pure Mg [9], no significant changes in the average grain size of Mg composites (considering the standard deviation) were observed after nano-B₄C addition (Table 1). Hence, the influence of nano-B₄C addition is observed to be minimal on the grain size reduction and/or grain coarsening. This can be attributed to the fact that the change in matrix grain characteristics resulting from particle stimulated grain nucleation is less dominant, considering the fine size of nanoscale reinforcements/dispersions. Particle stimulated grain nucleation is reported to occur only for particle sizes greater than 1 µm [21-23]. However, relatively fine grains were seen near the large sized Ti particles and other second phase clusters/agglomerates (Figure 1). This shows that while the clustered/agglomerated particles seem to have an active role

Figure 1: Optical micrographs showing the Mg-matrix grain characteristics of as-extruded (a) Mg-(5.6Ti+3Al) and (b) Mg-(5.6Ti+3Al)-2.5B₄C.

S. No.	Material	Grain size (µm)
1	Pure Mg	21 ± 3
2	Mg-(5.6Ti+3Al)	8 ± 2
3	Mg-(5.6Ti+3Al)-2.5B ₄ C	5 ± 3
4	Mg-(5.6Ti+3Al) (HT)	7 ± 3
5	Mg-(5.6Ti+3Al)-2.5B ₄ C (HT)	4 ± 3

^aBased on approximately 80 grains

Table 1: Results of microstructure studies^a.

in grain boundary pinning, the individual nanoparticles exhibit less involvement in the grain boundary pinning mechanism [21-23].

The results of microstructural measurements conducted on the optical micrographs of the developed Mg composites after heat treatment (Figure 2(a & b)) are shown in Table 1. It indicates no significant change in matrix grain characteristics due to heat treatment which confirms that the heat treatment at 200°C did not provide the critical energy required to facilitate grain boundary migration for grain nucleation/growth [14].

XRD analysis and Phase identification

The results of x-ray diffraction analysis conducted on the developed Mg composites (before and after heat treatment) and also that of the ball milled (5.6Ti+3Al) powder are shown in Figure 3. In addition to the prominent peaks corresponding to Ti and Al, few additional peaks (however of low intensity) identifiable with Al₃Ti intermetallic phase were seen in the XRD pattern of the ball milled (5.6Ti+3Al) powder and Mg-(5.6Ti+3Al) and Mg-(5.6Ti+3Al)-2.5B₄C composites. During the ball milling process, the reaction between Ti and Al can result in any of the following intermetallic phases such as Ti₃Al, Ti₂Al₃, AlTi, Al₂Ti and Al₃Ti. Among these transitional phases, the thermodynamic estimation shows that the formation of Al₃Ti is highly favored, as the free energy of formation of Al₃Ti is the least [9,24]. The x-ray analysis results of Mg composites after heat treatment at 200°C for 5 h reveal no new peaks corresponding to the formation of new precipitates. However, the intensity of peaks corresponding to Ti, Al and Al₃Ti intermetallic phase was found to reduce slightly after heat treatment. Further, in the nano-B₄C added composites (before and after heat treatment), the reaction between nano-B₄C and Ti, Al elements could result in the formation of other intermetallic phases such as TiB₂, TiC, Al₄C₃ etc. However, the x-ray diffractograms did not reveal any such peaks which could be attributed to either the absence or the relatively low volume percent (less than 2 %) of these phases in the Mg matrix [25].

Distribution of secondary phases

The distribution of metallic/ceramic particulates and other secondary phases in Mg-matrix before and after heat treatment are shown in Figure 4(a-c) and Figure 5(a-c). In Mg-(5.6Ti+3Al) composite, Figure 4a shows several rod shaped intermetallic

Figure 2: Optical micrographs showing the Mg-matrix grain characteristics of (a) Mg-(5.6Ti+3Al) and (b) Mg-(5.6Ti+3Al)-2.5B₄C after heat treatment at 200°C for 5 hours.

Figure 3: X-ray diffractograms of the (5.6Ti+3Al) ball milled powder and developed Mg composites with and without nano-B₄C, before and after heat treatment.

phases along with the uniformly distributed Ti particulates. The literature study confirms these blocky, rod-shaped or needle-shaped particles as Al₃Ti intermetallic phases [9,24,26]. In the nano-B₄C added Mg-(5.6Ti+3Al) composites, in addition to the Al₃Ti phases (Figure 4(c)), nanoscale B₄C particulates were found in the Mg-matrix (Figure 4(d)). However, an increased number of particulate clusters were seen in Mg-(5.6Ti+3Al)-2.5B₄C composite when compared to Mg-(5.6Ti+3Al) composite. The heat treated composites (Figure 5(a-c)) showed better matrix-reinforcement interface bonding with relatively lesser intermetallic content when compared to the as-extruded Mg composites.

Microhardness

Table 2 shows the results of microhardness measurements conducted on the developed Mg materials. On comparison with monolithic Mg, while, the composites exhibit significant improvement in microhardness, the highest value was observed in Mg-(5.6Ti+3Al). In general, the microhardness improvement in composites can be due to (i) high hardness and strength of reinforcement/second phases, (ii) effective load transfer from the matrix to reinforcement, (iii) grain refinement induced due to reinforcement addition and (iv) obstruction to the dislocation movements offered by the reinforcement/second phases [1,11,27,28]. The highest hardness value observed in case of Mg-(5.6Ti+3Al) corresponds to the combined presence of Ti (Hardness of Ti - 0.97 GPa) and Al₃Ti intermetallics (Hardness of Al₃Ti - 4 GPa) and their relatively uniform distribution in Mg matrix [29,30]. However, after heat treatment, the microhardness measurements conducted on both the Mg-(5.6Ti+3Al) and Mg-(5.6Ti+3Al)-2.5B₄C composite samples did not reveal any significant changes in the microhardness value.

Tensile properties

The room temperature tensile properties of developed

Mg composites before and after heat treatment are shown in Figure 6 and listed in Table 3. While a significant improvement in strength properties was observed in all the composites when compared to pure Mg, Mg-(5.6Ti+3Al) composites exhibit the highest strength before and after heat treatment. This can be attributed to the presence and relatively uniform distribution of hard Al₃Ti intermetallic phases in Mg matrix [9]. Further, the strengthening effects arisen from the (i) Hall-Petch relationship due to grain refinement, (ii) Orowan strengthening due to the presence of fine reinforcement/intermetallic phases, (iii) elastic and coefficient of thermal expansion mismatch between matrix and reinforcements and (iv) effective load transfer from matrix to second phases also contribute to the improvement in strength properties of Mg composite [1,11,16,28].

On the other hand, the increase in tensile ductility was observed in composite with nano-B₄C addition as shown in Figure 6 and Table 3. Interestingly, such ductility improvement occurred without significantly affecting the strength properties. The failure strain (ductility) improvement in Mg-(5.6Ti+3Al)-2.5B₄C composite attributes to the following factors pertaining to the presence of nano-B₄C particulates in Mg matrix. Available literature on nanocomposites shows that the nanoparticles provide sites for opening the cleavage cracks ahead of advancing crack front [11,12,31] and alters the local effective stress state from plane strain state to plane stress state in the neighborhood

Figure 4: SEM micrographs showing the distribution of particulate reinforcements and other second phases in the as-extruded (a) Mg-(5.6Ti+3Al) and (b-d) Mg-(5.6Ti+3Al)-2.5B₄C composites.

S. No.	Material	Microhardness [Hv]
1	Pure Mg	48 ± 1
2	Mg-(5.6Ti+3Al)	93 ± 3
3	Mg-(5.6Ti+3Al)-2.5B ₄ C	89 ± 6
4	Mg-(5.6Ti+3Al) (HT)	94 ± 4
5	Mg-(5.6Ti+3Al)-2.5B ₄ C (HT)	81 ± 4

Table 2: Results of microhardness measurements.

Figure 5: SEM micrographs showing the distribution of particulate reinforcements and other second phases in Mg-(5.6Ti+3Al) and (b & c) Mg-(5.6Ti+3Al)-2.5 B₄C composites after heat treatment at 200°C for 5 hours.

Figure 6: Engineering stress-strain curves showing the mechanical behavior of developed Mg materials under tension loading.

of crack tip [32]. This dissipates the stress concentration which would otherwise exist at the crack front [11,12,32]. Further, the activation of non-basal slip due to nanoparticle addition is also reported by various researchers [12,27,32]. Hence, the best combination of mechanical properties (especially ductility) observed after nanoscale B₄C addition was attributed to the combined presence of nano-B₄C particles and Al₃Ti intermetallic phases.

After heat treatment, all the composites showed significance enhancement in ductility over their non-heat treated parts (~140% and 75% for the composites without and with nano-B₄C

S. No.	Material	0.2 Yield Strength [MPa]	Ultimate Tensile Strength [MPa]	Failure Strain [%]
1	Pure Mg	125 ± 9	169 ± 11	6.2 ± 0.7
2	Mg-(5.6Ti+3Al)	194 ± 2	265 ± 2	4.8 ± 0.6
3	Mg-(5.6Ti+3Al)-2.5B ₄ C	183 ± 6	255 ± 4	7.6 ± 0.3
4	Mg-(5.6Ti+3Al) (HT)	181 ± 1	258 ± 2	11.8 ± 1.3
5	Mg-(5.6Ti+3Al)-2.5B ₄ C (HT)	174 ± 4	244 ± 5	13.5 ± 2.4

Table 3: Results of room temperature tensile testing.

respectively). The heat treatment process in Mg composites resulted in internal stress relaxation at the matrix-reinforcement interface. This eventually results in a less-stressed interface (with relatively less interfacial residual stress) and such a less-stressed interface would delay the nucleation and growth of cavities in advance of crack tip and hence improve the ductility. Thus, the heat treatment at 200°C for 5 hours assisted in eliminating hot spots of cavitation and lead to a better utilization of both matrix and reinforcement properties [15,16]. Further, the increase in failure strain can also be attributed to the reduction in size of the secondary phases after heat treatment as seen in Figure 5.

Fractography

The fractographic evidences of as-extruded and heat treated Mg composites under tensile loading are shown in Figure 7(a-c) and Figure 7(d-f), respectively. The fractographs confirm to the ductility results of tensile testing. At microscopic level,

the developed Mg composites showed mixed mode fracture features in contrast to the cleavage mode brittle fracture in case of pure Mg [9,11,]. In general, micro-cracks are generated in the composites due to the interfacial stresses between the matrix and reinforcement [11]. Such matrix/particle cracking was prominently observed in composites without heat treatment while the particle fracture behaviour with lesser number of cleavage steps were observed in the case of heat treated composites. This suggests decrease in the degree of brittle fracture behaviour due to relief of stresses at the matrix-reinforcement interface [1,16]. In case of Mg-(5.6Ti+3Al), microvoids coalescence forming cracks extending into the matrix indicating good interface bonding at the particle/matrix interface was observed as seen in Figure 7(a) [9]. In this case, the residual stresses at Mg/Ti interface would be relatively high due to the presence of Al₃Ti intermetallic phase which would limit the load transfer across the interface. However, such void nucleation and interface cracking were not seen in the heat treated composites (Figure 7d). This observation also confirms the decrease in strength alongside ductility enhancement as observed in Table 3. Similar observations were also seen in Mg-(5.6Ti+3Al)-2.5B₄C composite (Figure 7(b & e)) which also confirms the fracture strain values given in Table 3. In addition, the nano-B₄C particles can simultaneously facilitate the activation of non-basal slip systems as indicated by the uneven lines in Figure 7c supposedly due to combined effect of basal and non-basal slip in contrast to the straight lines due to slip in the basal plane in Mg [12,32].

Conclusions

The effects of nano-B₄C addition and heat treatment on the microstructure and mechanical properties of Mg-(5.6Ti+3Al) system were studied and the following conclusions were drawn from the present investigation.

- (1) Nano-B₄C addition did not cause significant changes to the pre-existing Al₃Ti phases and the matrix grain characteristics.
- (2) Addition of nano-B₄C particulates to Mg-(5.6Ti+3Al) provided improved ductility and strength retention.
- (3) The best combination of strength and ductility of Mg-(5.6Ti+3Al)-2.5B₄C was attributed to the combined presence of nano-B₄C and Al₃Ti intermetallic phases in Mg matrix.
- (4) Heat treatment played a beneficial effect in significantly

Figure 7: SEM fractographs showing the fracture surfaces of developed Mg materials tested under tensile loading.

enhancing the ductility of all the composites. The large improvement in ductility was attributed to the interfacial stress relaxation between the matrix and the other secondary phases/reinforcements.

Acknowledgements

One of the authors, Mr. S. Sankaranarayanan, sincerely thanks the NUS research scholarship for supporting this research for his graduate program.

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