Journal of Engineering Science, Vol. 9, 1–9, 2013

Improving the Mechanical Properties of Mg-5.6Ti-3Al Composite through Nano-Al_2O_3 Addition with Recrystallisation Heat Treatment

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Abstract: *In the current study, the role of nano-*Al_2O_3 *addition to Mg-5.6Ti-3Al composite and subsequent recrystallisation heat treatment in improving the mechanical properties are investigated. The following Mg composites: (i) Mg-5.6Ti-3Al (with 3 wt% Al and 5.6 wt% Ti) and (ii) Mg-5.6Ti-3Al-2.5*Al_2O_3 *(with 3 wt% Al, 5.6 wt% Ti and 2.5 wt%* Al_2O_3*) were synthesised through the disintegrated melt deposition (DMD) technique followed by hot extrusion. Mg-5.6Ti-3Al-2.5*Al_2O_3 *composite was then subjected to recrystallisation heat treatment at 200ºC for 5 h. Mechanical property evaluation of the developed Mg composites indicated a significant improvement in microhardness and tensile properties when compared to pure Mg and Mg-5.6Ti-3Al. Microstructural characterisation revealed a significant grain refinement and uniform distribution of reinforcements/second phases in the developed Mg composites due to hybrid reinforcement additions and heat treatment. In case of as-extruded Mg-5.6Ti-3Al-2.5*Al_2O_3 *composite, the strength improvement occurred at the expense of ductility while for heat-treated composite, the increase in strength properties was accompanied by an increase in ductility. Based on the processing-structure-property correlation, it was identified that the presence of hard reinforcements/intermetallics in Mg matrix contributes to the improvement in strength properties while the stress relaxation during heat treatment contribute to the ductility enhancement.*

Keywords: Magnesium composites, nano reinforcement addition, recrystallisation heat treatment, mechanical properties, scanning electron microscopy

1. INTRODUCTION

The current research and development of magnesium (Mg) based materials are focused more towards making them the best replacement to aluminium (Al), as Mg is ~35% lighter than Al and exhibits properties comparable to that of Al. The advantages of Mg as an engineering material include excellent machinability, damping capacity and castability.[1,2] However, the low elastic modulus and elevated strength properties of Mg restrict its extended usage in critical engineering applications. To overcome these limitations, hard ceramic reinforcements such as SiC and Al_2O_3 are added to Mg which often results in the ductility reduction.[1,2] The available literature reveals that addition of nanoscale particulate reinforcements results in simultaneous improvement in strength and ductility of Mg materials.[1] Similar results are also observed for Mg composites with insoluble metallic reinforcements like titanium and molybdenum.[1] Further, the positive influence of addition of hybrid reinforcements on the mechanical properties of the Mg composites has been recently identified.[3]

In composite development methodology involving secondary processing such as extrusion, the failure of the composite under loading is initiated by the crack formation at the interface owing to the stress attributing to the difference in thermal expansion coefficients between the matrix and reinforcements. In such cases, heat treatment near the recrystallisation temperature results in the stress relaxation at the interface and improves the mechanical properties.[4]

In order to study the role of nano-reinforcement addition and heat treatment, the following Mg composites: (i) Mg-5.6Ti-3Al (with 3 wt% Al and 5.6 wt% Ti) particulates and (ii) Mg-5.6Ti-3Al-2.5Al_2O_3 (with 3 wt% Al, 5.6 wt% Ti and 2.5 wt% Al_2O_3) were synthesised through the disintegrated melt deposition technique (DMD) followed by hot extrusion and Mg-5.6Ti-3Al-2.5Al_2O_3 composite was then subjected to recrystallisation heat treatment at 200°C for 5 h. The physical, microstructural and mechanical properties of the developed composites were evaluated in order to study the effect on the microstructure and mechanical properties.

2. EXPERIMENTAL

2.1 Materials

Mg turnings of >99.9% purity (ACROS Organics, USA) were used as the matrix material. Elemental Ti particulates of particle size <140 μm (purity 98%) supplied by Merck and Al particulates of average particle size ~15 μm (purity 98%) supplied by Alfa Aesar were used as metallic additions. Nano Al_2O_3 particulates of average particle size ~50 nm (purity 99%) supplied by Baikowsi was used as the ceramic reinforcement.

2.2 Melting, Casting and Extrusion

The Mg-based materials were prepared using the DMD technique.[3] Mg turnings together with the reinforcements/alloying additions were heated in a graphite crucible to 1023K in an electrical resistance furnace, under inert argon gas atmosphere. The superheated molten slurry was stirred for 5 min at 460 rpm to facilitate a uniform distribution of the reinforcements/intermetallics in the Mg-matrix. Following stirring, the melt was then released through a 0.01 m diameter orifice at the base of the crucible and it was disintegrated by two jets of argon gas. The disintegrated melt slurry was subsequently deposited onto the steel mould to obtain an ingot of 0.04 m diameter. The synthesis of pure Mg was carried out in a similar fashion, except that no alloying elements were added. The obtained ingot was then machined to a diameter of 0.036 m and soaked at 400°C for 1 h to perform hot extrusion at 350°C resulting rods of diameter 8 mm. Further characterisation studies were conducted on the extruded rods. The Mg-5.6Ti-3Al-2.5Al_2O_3 composite is then subjected to recrystallisation heat treatment at 200°C for 5 h.

2.3 Characterisation

Standard tests were conducted on the as-polished samples cut from the extruded rods of developed materials to determine the density.[3] The grain morphology and the distribution of second phases in Mg matrix were studied on the as-polished samples using a Hitachi S-4300 FESEM equipped with EDS, an Olympus metallographic optical microscope and Scion image analysis software. Shimadzu LAB X XRD-6000 diffractometer was used to carry out the X-ray diffraction analysis on the developed Mg materials. The microhardness measurements were carried out on the as-polished samples of developed materials using Matsuzawa MXT 50 automatic digital Microhardness tester based in accordance with ASTM standard E3 84–99. Standard ASTM test method E8M-96 was conducted on the test samples using a fully automated servo-hydraulic mechanical testing machine, to determine the tensile properties of developed materials before and after heat treatment. The fractured surfaces of Mg-materials after tensile test were studied using the Hitachi S-4300 FESEM.

3. RESULTS AND DISCUSSION

The visual observation of the surfaces of the developed as-cast Mg-ingots and extruded Mg-rods indicates no macrostructural defects. From the experimental density values (Table 1), it can be seen that near-dense materials have been developed and the porosity level is relatively low (<0.1%) in all the samples. This confirms the suitability of processing parameters used in the study. The microstructure and X-ray studies reveal the presence of Ti and $Mg_{17}Al_{12}$ intermetallic in Mg-5.6Ti-3Al, and Ti, nano-Al_2O_3 and $Mg_{17}Al_{12}$ in Mg-5.6Ti-3Al-2.5Al_2O_3 and Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) composites as shown in Figure 1 and Table 1. The results from grain size measurements reveal significant grain refinement in the case of Mg-5.6Ti-3Al-2.5Al_2O_3 when compared to Mg-5.6Ti-3Al (Table 1).

The grain refinement as observed in Figure 1 is attributed to the presence of hard reinforcements and other second phase intermetallics acting as sites for grain nucleation to occur during solid-state processing.[5] For post-heat treatment, the observed grain size (Figure 2) of Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) appeared to be larger when compared to Mg-5.6Ti-3Al-2.5Al_2O_3 without heat treatment. The grain growth in Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) attributes to the stress relaxation during heat treatment process, which did not provide the required critical energy to facilitate the grain boundary migration.[6] Also, the increase in the number of equiaxed grains has contributed to the marginal reduction in the average aspect ratio due to the heat treatment.

Table 1: Results of density and microstructural measurements.

S. No.	Material	Th. density (g cc^{-1})	Exp. density (g cc^{-1})	Porosity (%)	Grain size (μm)
1	Mg-5.6Ti-3Al	1.8221	1.7655 ± 0.0064	0.03	8.5 ± 1.53
2	Mg-5.6Ti-3Al-2.5Al_2O_3	1.8474	1.7685 ± 0.0324	0.06	6.3 ± 1.01
3	Mg-5.6Ti-3Al-2.5Al_2O_3 (HT)	1.8474	1.7740 ± 0.0101	0.04	8.2 ± 2.27

The hardness values from microhardness measurements (Figure 2) indicate an improvement in microhardness values in all of the developed composites when compared to pure Mg. An improvement of ~30% occurred in Mg-5.6Ti-3Al-2.5Al_2O_3 and ~20% in Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) when compared to Mg-5.6Ti-3Al. The improvement in hardness value attributes to the presence of harder Al_2O_3 (~2.2 GPa).[7] The hardness value is found to reduce in the case of Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) when compared to Mg-5.6Ti-3Al-2.5Al_2O_3 due to the stress relaxation and grain growth upon heat treatment.[6]

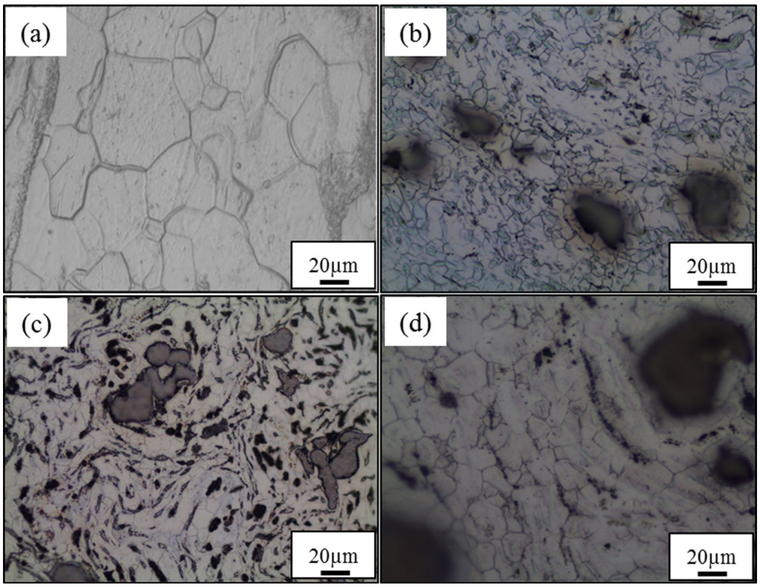

Figure 1: Grain morphology of (a) pure Mg, (b) Mg-5.6Ti-3Al, (c) Mg-5.6Ti-3Al-2.5Al_2O_3 and (d) Mg-5.6Ti-3Al-2.5Al_2O_3 (HT).

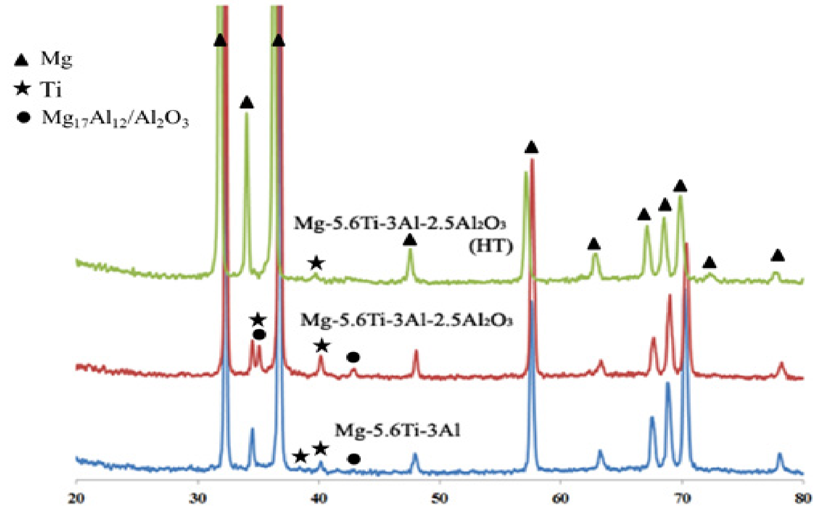

Figure 2: Results of X-ray diffraction studies conducted on developed Mg composites.

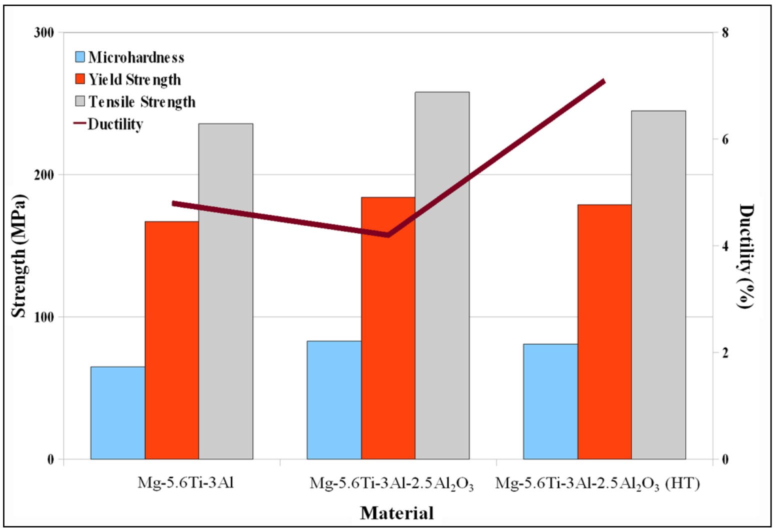

Figure 3: Variation in strength and microhardness of developed Mg-composites.

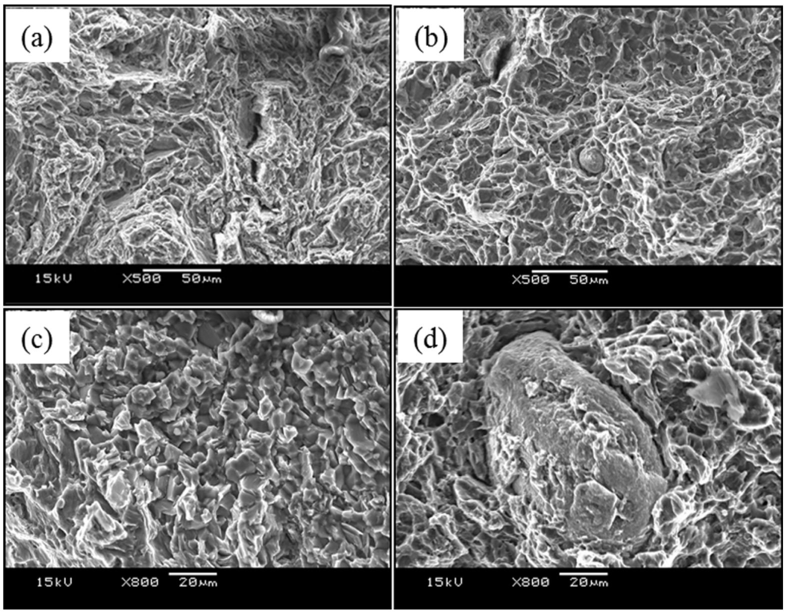

Figure 4: Tensile fractographs of (a) Mg, (b) Mg-5.6Ti-3Al, (c) Mg-5.6Ti-3Al-2.5Al_2O_3 and (d) Mg-5.6Ti-3Al-2.5Al_2O_3 (HT).

The results from tensile tests indicate a significant improvement in the strength properties of Mg incorporated with hard metallic and metallic/ceramic elements (Figure 3). The improvement in strength properties of Mg-composites when compared to pure Mg can be ascribed to the following strengthening effects: (i) thermal misfit between matrix and reinforcements/intermetallics; (ii) grain refinement; and (iii) the morphology of the Ti-particles, wherein the sharp-edged particles contribute to higher dislocation density due to the increased stress concentration at the pointed corners.[8] Also, the presence of harder reinforcements/second phases in Mg matrix eventually increases the load carrying capacity thus improving the strength much significantly.[9]

In case of Mg-5.6Ti-3Al-2.5Al_2O_3 composite, the addition of nano-Al_2O_3 particles is expected to show an improvement in tensile ductility through the activation of non-basal slip systems.[1] However, the positive influence of nano-Al_2O_3 addition is observed to be over-sided by the increased volume fraction and clustering of ($Mg_{17}Al_{12}$/Al_2O_3) secondary phases. Similar negative effect on tensile ductility due to clustered second phases was reported when the nano-Al_2O_3 particulates were directly added together with the sharp edged-Ti particulates to pure Mg.[3]

While the improvement in strength properties in Mg-5.6Ti-3Al-2.5Al_2O_3 occurred at the expense of ductility in Mg-5.6Ti-3Al-2.5Al_2O_3 composite, a significant improvement in ductility with little effect on strength properties took place in the case of Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) composite. The improvement in tensile ductility of Mg-5.6Ti-3Al-2.5Al_2O_3 (HT) could be attributed to the reduction in thermal residual stress upon heat treatment. Under tensile loading, the reduction in thermal residual stress would result in the delay of cavity nucleation and its growth in advance of the crack tip. This in turn will contribute to the improvement in ductility.[4] Thus, the recrystallisation heat treatment at 200°C for 5 h helps in eliminating hot spots of cavitation which lead to a better utilisation of both matrix and reinforcement properties.[4,5]

The fracture surface analysis of composite samples confirms to the ductility values obtained from the tensile testing. The composite samples failed under tensile loading indicating a mixed mode fracture with particulate debonding [Figure 4(b and c)] observed in Mg-5.6Ti-3Al and Mg-5.6Ti-3Al-2.5Al_2O_3, while mixed mode fracture with relative plastic deformation and good interfacial bonding [Figure 4(d)] observed in Mg-5.6Ti-3Al-2.5Al_2O_3 (HT), while the fracture mode of pure Mg under tension is through cleavage [Figure 4(a)].[10]

4. CONCLUSION

It is here concluded that:

1. DMD technique can successfully synthesise magnesium composites.
2. The inherent properties of metallic and ceramic reinforcements such as Ti, Al and Al_2O_3 respectively such as hardness, ductility, structural compatibility with Mg and the good wettability of Ti and Mg result in the enhancement of strength and ductility in Mg composites under tensile loads.
3. Addition of nano Al_2O_3 to Mg-5.6Ti-3Al results in the improvement in strength properties and hardness at the expense of ductility. The stress relaxation, grain growth and uniform distribution of finer intermetallics and second phases contribute to the improvement in ductility in case of Mg-5.6Ti-3Al-Al_2O_3 after heat treatment.

5. ACKNOWLEDGEMENT

One of the authors, S. Sankaranarayanan acknowledges the National University of Singapore research scholarship for supporting this research for graduate program.

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