Microstructure and Mechanical Properties of Mg–7.4% Al Alloy Matrix Composites Reinforced by Nanocrystalline Al–Ca Intermetallic Particles

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Abstract Mg–7.6% Al (in mass fraction) alloy matrix composites reinforced with different volume fractions of nanocrystalline Al_3Ca_8 particles were synthesized by powder metallurgy, and the effect of the volume fraction of reinforcement on the mechanical properties was studied. Room temperature compression test reveals considerable improvement on mechanical properties as compared to unreinforced matrix. The compressive strength increases from 683 MPa for unreinforced alloy matrix to about 767 and 823 MPa for the samples having 20 and 40 vol% of reinforcement, respectively, while retaining appreciable plastic deformation ranging between 12 and 24%. The specific strength of the composites increased significantly, demonstrating the effectiveness of the low-density Al_3Ca_8 reinforcement.

KEY WORDS: Intermetallics; Consolidation; Dispersion strengthening; Powder metallurgy; Mechanical property; Electron microscopy

1 Introduction

In recent, past magnesium-based metal matrix composites (MMCs) are being used in automotive, electronics, and aerospace industries due to their low density, high specific strength, and excellent machinability [1–3]. MMCs reinforced with ceramic particles have extensively been used for several light weight and wear resistance applications [2–4]. However, owing to the high abrasiveness, brittleness, and wettability of ceramics [4–7] in recent years, intermetallics as reinforcements in MMCs have emerged as possible substitutes for ceramic reinforcements [8–11]. This appears to be a highly promising option in view of high hardness of intermetallics and their compatibility with the metal matrix. In addition, thermal expansion coefficients of intermetallics

are much closer to the metals than those of ceramic reinforcements.

Among the intermetallics, Al_3Ca_8 (triclinic) and $\text{Al}_{14}\text{Ca}_{13}$ (monoclinic) [12] intermetallic phases of Al–Ca system appear as a promising candidates for the use as reinforcing agent due to their extremely low densities (1.859 and 2.013 g/cm³, respectively). Al_3Ca_8 is one of the important intermetallic phases in the Al–Ca system that has excellent mechanical properties. It is well known that the mechanical properties of intermetallics are closely related to their structures and stability [13]. Al_3Ca_8 phase is known to be hard, brittle, and stable up to a temperature of 852 K [14] making it the most suitable candidate for reinforcement.

For most of the engineering applications, bulk sample is the prerequisite for the performance evaluation [15]. The key challenge in the processing of bulk composite samples is to get homogenous distribution of the reinforcing phases in order to achieve a uniform microstructure. The mechanical properties of an MMCS are strongly dependent on the properties of reinforcements, distribution, size, morphology and volume fraction of the reinforcement, as well as the interfacial strength between the reinforcement,

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and the matrix [16]. Due to their high surface area, nano-size powders will naturally tend to agglomerate in order to reduce their overall surface energy, hence making it difficult to obtain an uniform dispersion by most conventional liquid processing methods. Uniform distribution of reinforcement could be obtained by solid state processing methods. Powder metallurgy is one of solid state processing techniques and is highly flexible and versatile technique for producing metal matrix composites. An important advantage of this method is its low processing temperature compared to melting techniques. Therefore, interfacial reaction between the matrix and the reinforcement phases is prevented. On the other hand, more uniform distribution of the reinforcing particles can be achieved using this technique [17, 18]. Another advantage of powder metallurgy technique is its ability to manufacture near net shape product at low cost.

The present work aims to study the effectiveness of the complex Al_3Ca_8 intermetallic particle as reinforcement in novel Mg–7.6% Al alloy matrix composite. The emphasis has been given to study the effect of volume fraction of the reinforcement on the microstructure and mechanical properties of the composites.

2 Experimental

Mg–7.6% Al (in mass fraction) alloy matrix was prepared by mechanical alloying of the elemental powder of Al and Mg using a Retsch PM400 planetary ball mill with hardened steel balls and vials. The powders were milled for 200 h with a ball-to-powder mass ratio (BPR) of 10:1 and at rotational velocity of 150 r/min. The reinforcement was prepared by crushing of pre-alloyed $\text{Al}_{35}\text{Ca}_{65}$ ingots (prepared by arc melting techniques) followed by 200 h milling in a separate vial. Handling of samples was carried out in a glove box under purified argon atmosphere (O_2 and H_2O volume fraction $<1 \times 10^{-7}$). In order to produce Mg-

based MMCs reinforced with Al–Ca intermetallic particles, the Mg–7.4% Al alloy powder was mechanically mixed in the planetary ball mill with 20, 40, and 60 vol% of Al–Ca intermetallic powder. The composite powders were consolidated by hot pressing at 673 K and 400 MPa. Structural characterization was performed by X-ray diffraction (XRD) using a Philips PW 1050 diffractometer (CoK_α radiation, $\lambda = 0.17889$ nm) and by scanning electron microscopy (SEM) using a Gemini 1530 microscope. The Rietveld method was applied for the profile-fitting structure refinement using the WinPlotR software package [19]. The thermal stability was investigated by differential scanning calorimetry (DSC) with a Perkin–Elmer DSC7 calorimeter at heating rate of 20 K/min under a continuous flow of purified argon. The density of the hot pressed samples was measured using Archimedes principle. Cylinders having a length/diameter ratio of 2.0 (6 mm of length and 3 mm of diameter) were prepared from the hot consolidated samples for compression testing in accordance with ASTM standard [20]. The specimens were tested at room temperature using universal testing machine (Instron model 8562) under quasistatic loading (strain rate $\sim 1 \times 10^{-4} \text{ s}^{-1}$).

3 Results and Discussion

Figure 1a shows the XRD patterns of the as milled and annealed reinforcement and the consolidated composites with different volume fractions of reinforcement. XRD pattern of the as milled reinforcement powder clearly reveals broad peaks of $\text{Al}_{14}\text{Ca}_{13}$ phase and small peaks of CaO, which could be due to oxidation of highly reactive Ca during handling and sample preparation. The significant peak broadening could be attributed to the reduced crystallite size in nanometer regime. Nanocrystalline phase of the pattern was confirmed by Rietveld structure refinement, which reveals a crystallite size of about 10 nm. Thermal stability of the milled reinforcement powder was also

Fig. 1 **a** XRD patterns for the as milled and annealed reinforcement and composites with 20 and 40 vol% of $\text{Al}_{35}\text{Ca}_{65}$ powder; **b** DSC curve of the 200 h milled reinforcement powders (heating rate is 20 K/min)

Fig. 2 **a** Particle size distribution of 200 h milled $\text{Al}_{35}\text{Ca}_{65}$ powders; **b** corresponding SEM micrograph of the powder

checked by annealing the powder up to 673 K in the argon atmosphere at 20 K/min using DSC apparatus, and the isochronal DSC curve of the same is shown in Fig. 1b. The DSC curve consists of a broad exotherm between 470 and 550 K followed by a small exotherm starting at about 560 K and by a sharp exotherm in the temperature range between 580, and 610 K. Finally, an additional broad exothermic signal occurs at temperature above 620 K. Combined analysis of XRD and DSC result of the annealed powder shows that the $\text{Al}_{14}\text{Ca}_{13}$ phase transforms to Al_3Ca_8 phase at 586 K. The heat released for this phase

transformation corresponding to the sharp DSC peak at 586 K is estimated to 9.6 kJ/mol). This value of heat release is remarkably close to that calculated by Islam *et al.* [21] for same phase transformation (10.4 kJ/mol).

Figure 1a also shows the XRD patterns of the consolidated composite with 20 and 40 vol% reinforcements. They reveal sharp crystalline peaks of pure Mg. Pattern corresponding to the composite containing 40 vol% reinforcement shows higher intensity of intermetallic phases as compared to the pattern of the composite containing 20 vol% reinforcement due to higher volume fraction of the intermetallics. The detection of the only three phases in XRD pattern of the composite shows that no reaction had occurred in the present consolidation conditions (hot pressing 673 K and 400 MPa).

Figure 2a shows the particle size distribution of the 200 h milled $\text{Al}_{35}\text{Ca}_{65}$ powders. Clearly indicates that the particle size ranges between 2 and 18 μm with mean particle size of about 7 μm . Figure 2b shows the SEM micrograph of the 200 h milled $\text{Al}_{35}\text{Ca}_{65}$ powders. This mostly consists of round-shaped fine particles along with agglomerated coarse particles.

Figure 3 shows the SEM micrographs of the hot consolidated samples taken from the cross-section of the Mg–7.4% Al alloy and the composites reinforced by 20, 40, and 60 vol% Al_3Ca_8 particles. The micrographs of the composites reveal the reinforcing Al_3Ca_8 particles (white regions), which tend to agglomerate around the matrix

Fig. 3 SEM micrographs for the consolidated Mg–7.4% Al alloy **a**, consolidated composites reinforced with 20 vol% **b**, 40 vol% **c**, 60 vol% **d** Al_3Ca_8 particles

Fig. 4 Experimental (*square*) and theoretical (*circle*) densities of the consolidated samples as a function of the volume fraction of Al_3Ca_8 reinforcement

particles (dark regions) forming a cell network throughout the sample. As the volume fraction of the reinforcement increases the particles tend to agglomerate forming clusters and further increment in the reinforcement fraction to 40 and 60 vol%, the cluster regions become more and more interconnected. However, the overall distribution of the reinforcement in the matrix remains fairly uniform. The microstructure of the composite does not show any porosity, corroborating the high density of the samples. The density linearly decreases with increasing amount of reinforcement from about 1.79 g/cm^3 for unreinforced matrix to 1.65 g/cm^3 for the reinforced composite with 60 vol% reinforcement, which corresponds to a reduction of about 8% with respect to the unreinforced matrix (Fig. 4). The decrease in density of the composite shows usual behavior, as the reinforcement being light in comparison with the matrix.

The room temperature compressive stress–strain curves under quasistatic loading of the composites and

Fig. 6 Specific strength of the composites as a function of the fraction of intermetallic reinforcement. For comparison purposes, the values for Mg-based MMCs reinforced with fly ash [3] and TiC [4] evaluated by compression tests are also reported

unreinforced Mg–7.4% Al matrix are shown in Fig. 5a, and the corresponding mechanical data are summarized in Fig. 5b. The addition of the nanostructured reinforcement remarkably improves the mechanical properties of the Mg–7.4% Al alloy matrix. The compressive strength (the maximum compressive stress which the material is capable of sustaining [22]) increases from 683 MPa for unreinforced matrix to 767, 823, and 852 MPa for the composites with 20, 40, and 60 vol% reinforcement, respectively. These results indicate that the addition of the nanostructured reinforcement leads to composite having compressive strengths exceeding that of unreinforced matrix (to the order of 13–24%), while retaining appreciable plastic deformation. This increase in compressive strength could be attributed to well-known factors (pertaining to reinforcement) such as: (1) the dislocation strengthening in the matrix, which is related to the nucleation of additional dislocations in the matrix due to the introduction of the

Fig. 5 **a** Room temperature compression true stress–true strain curves for the consolidated unreinforced Mg–7.4 Al matrix and composites with 20 vol.% ($V = 20$), 40 vol.% ($V = 40$) and 60 vol.% ($V = 60$) of nanostructured Al_3Ca_8 particles, **b** corresponding data of mechanical properties

reinforcement [23, 24], (2) Orowan strengthening mechanism [25], and (3) load transfer from the matrix to reinforcement [24].

The addition of low-density Al_3Ca_8 particles decreases the density of the composites below that of unreinforced matrix (Fig. 4). Figure 6 indicates the variation of specific strength (tensile or compressive strength divided by the density), as a function of volume fraction of the reinforcement. It is very clearly indicated in this figure that the specific strength sharply increases from about 361 kN m/kg for unreinforced matrix to 450 kN m/kg for the composite with 20 vol% Al_3Ca_8 reinforcement and 498 kN m/kg for the composite with 40 vol% Al_3Ca_8 reinforcement. The specific strength further increases to about 526 kN m/kg for the composite with 60 vol of Al_3Ca_8 reinforcement. These values of specific strengths are higher than those reported for AZ92D/Fly ash [3], and AZ92D/TiC [4] composites evaluated by compression tests (Fig. 6). Since a high specific strength is one of the most important aspects of lightweight materials, the composites reinforced with intermetallic particles in the present case could be a better alternative to conventional ceramic-reinforced composites.

4 Conclusions

Mg–7.4% Al matrix reinforced with different volume fractions of nanocrystalline Al_3Ca_8 intermetallic particles has been produced by powder metallurgy, and their mechanical properties have been investigated under compressive loading. The results reveal encouraging room temperature mechanical properties with respect to the unreinforced Mg–7.4% Al matrix. Yield and compressive strength have increased from 556 to 683 MPa for unreinforced alloy matrix to about 677 and 823 MPa for the composite reinforced with 40 vol% Al_3Ca_8 intermetallic particles, respectively, while retaining appreciable plastic deformation. Furthermore, the addition of low-density Al_3Ca_8 intermetallic phase decreased the density of the composite and significantly increased the specific strength of the composites.

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