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DEVELOPMENT OF MAGNESIUM COMPOSITES USING BALL MILLED HIGH ENTROPY ALLOY PARTICLES

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ABSTRACT

The requirement of high performance lightweight materials in various energy saving applications has been catalytic in the development of novel Metal Matrix Composites (MMCs) based on light metallic matrices. Magnesium is the lightest of all metallic materials and possesses one of the highest strength-to-density ratio. Studies were made on magnesium based composites containing ceramic reinforcements, metallic reinforcements as well as hybrid (ceramic + metallic) reinforcements. In the present study, an attempt was made on the development of new magnesium composites containing High Entropy Alloy (HEA) reinforcement. HEA reinforcement used in this study was in the form of ball milled particles. Mechanical properties of the developed composite were determined in terms of microhardness and compression tests. The study showed an increasing trend in the mechanical properties with increasing presence of HEA reinforcement. For microstructure analyses, the size and morphology of the grains, and the size and distribution pattern of the reinforcements were investigated using optical and scanning electron microscopy. The relation between microstructure and mechanical properties are examined to understand the behavior of newly developed Mg-HEA composites.

KEYWORDS: Magnesium, High Entropy Alloy (HEA), Composite, Sintering, Mechanical Properties.

Metal matrix composites (MMCs) have been known as the tailor made materials consisting of two or more materials, with metals as the matrix materials. Tailored properties can be achieved from systematic combinations of different constituent elements/materials. By careful selection of matrix and reinforcing phases, the newly formed composite materials with significant improvements in elastic modulus, strength, ductility and coefficient of thermal expansion can be fabricated. Composite materials are attractive because they can offer the possibility of attaining combined properties which are not obtainable form the monolithic materials. The attractive physical and mechanical properties obtained from MMCs have made them potential candidates for aerospace, automotive and other structural applications [1]. In search of light structural materials, magnesium based metal matrix composites are of great interest due to the fact that magnesium is the lightest metal among all structural materials. Because of some disadvantages of magnesium such as high chemical reactivity and limited deformability at ambient temperature, only limited applications of these materials are realized. Attempts are being made to circumvent some of the limitations of magnesium by reinforcing them with micron and nano size ceramic particulates, carbonnanotubes (CNTs) and the metallic particulates. Furthermore, research work have been done on the magnesium composites containing hybrid (ceramic + metallic) reinforcement as well as bimodal size (micron + nano) reinforcements [3].

A comprehensive examination of the published literature reveals that no attempt has been made to improve the mechanical properties of magnesium using ball milled high entropy alloy particles. Accordingly, in the present study, an attempt was made to synthesize magnesium composites using ball milled high entropy alloy particles. The materials are processed using powder metallurgy route incorporating microwave assisted rapid sintering coupled with hot extrusion. Particular emphasis was placed to study the effect of presence of ball milled high entropy alloy particles in magnesium matrix and the test results were benchmarked against pure magnesium.

EXPERIMENTAL PROCEDURES

Synthesis of Materials

In this study, magnesium powder of 98.5% purity and with a size range of 60-300 μm (acquired from Merck, Germany) was used as the matrix material. Ball milled high entropy alloy particles with an average particle size of 3 μm were used as the particulate reinforcements. The synthesis process for Mg-0.5wt%HEA and Mg-2.5wt% HEA composites involved blending pure magnesium powder with alloy particles in a RETSCH PM-400 mechanical alloying machine at 200 rpm for 1 hour. The blended powder mixtures were then cold compacted using a 100-ton press to form billets that measured 35-mm in diameter and 45-mm in height. Monolithic magnesium was compacted using the same parameters but without blending. The compacted billets were then sintered using an innovative

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hybrid microwave sintering technique [4] for 16 minutes to reach a temperature near the melting point of magnesium using a 900W, 2.45 GHz SHARP microwave oven. The sintered billets were homogenized at 400˚C for 1 hour and subsequently hot extruded at a temperature of 350˚C at an extrusion ratio of 20.25:1.

Characterization

Microstructural characterization studies were conducted to determine the grain size, grain morphology, and presence and distribution of reinforcements. OLYMPUS metallographic optical microscope, Scion Image Analyzer software and a Scanning Electron Microscope (JEOL JSM-6010) were used for this purpose.

Microhardness measurements were performed on the polished samples using Shimadzu-HMV automatic digital microhardness tester with a Vickers indenter. An indentation load of 245.5 mN and a dwell time of 15 seconds was used in accordance with the ASTM standard E384-08.

Room temperature compressive tests were performed on cylindrical monolithic and composite samples according to ASTM E9-89a using an automated servo hydraulic testing machine (MTS810). Extruded rod of 8mm diameter was cut into 8mm length samples for compression tests to provide the aspect ratio (l/d) of unity. Samples were tested at a strain rate of 5×10^{-3} min^{-1} and the compression load was applied parallel to the extrusion direction.

RESULTS AND DISCUSSION

Microstructure

The results of grain size measurement and grain morphology are shown in Table 1. From the measurements, the reduction in grain size was observed in Mg-HEA composite samples. In Mg-0.5HEA composite, the grain size reduction was about 50% than that of pure Mg. However, minimal reduction in average grain size was observed with increasing presence of HEA alloy particles in the composites. It is well known that the grain size reduction in Mg composites is realized through restriction of grain growth by grain boundary pinning due to the presence of reinforcements. Morphology of ball milled HEA alloy particles with an average size of 3 µm used as the reinforcement is shown in Figure 1.

Figure 1: Ball milled HEA particles.

Table 1: Results of grain size and microhardness measurements.

Material	Grain Characteristics		Microhardness (HV)
	Size (µm)	Aspect ratio	
Mg	34 ± 4	1.4 ± 0.3	47 ± 2
Mg-0.5wt% HEA	17 ± 4	1.5 ± 0.3	53 ± 4
Mg-2.5wt% HEA	14 ± 4	1.4 ± 0.3	56 ± 6

With the presence of reinforcement particle size greater than 1 µm in the composites, grain refinement is attainable through nucleation of recrystallized grains at the particles [5, 6] in addition to pinning mechanism. The nucleation of recrystallized grainsis shown in Figure 2. In the microstructure of Mg-2.5HEA composite, the fine grains can be seen in the particle accumulated area (area shown within the circle in Figure 2) and coarse grains can be seen in the particle depletion region (area shown within the rectangle in Figure 2) The morphology of the grain size was analysed through the grain aspect ratio measurement. From the measurement, nearly equiaxed grain morphology was observed in monolithic and composite samples (Table 1).

Figure 2: Coarse and fine grains in Mg-2.5HEA composite.

The distribution of reinforcement particles in the composites is shown in Figure 3. In both composites, the reinforcement particles were found to be mostly located at the grain boundaries. In Mg-2.5HEA composite, as compared to Mg-0.5HEA composite, the increased amount of reinforcement particles are seen to be densely present at the grain boundaries.

Microhardness

The microhardness measurement results are shown in Table 1. The hardness values of composites were higher when compared to monolithic magnesium. A simultaneous improvement in average microhardness was also observed in the composites with increasing presence of HEA particles. Under applied indentation, resistance to localized plastic deformation of Mg matrix was increased due to the presence of harder HEA alloy particles. The observed increment in microhardness in Mg-HEA composites also indicated that HEA alloy particles can be bonded well with Mg matrix [7]. Even though the amount of HEA alloy particles was increased in 0.5wt% to 2.5wt% in Mg matrix, significant improvement in microhardness was not achieved in Mg-2.5HEA composite. This can be explained based on the observation of:

(a) insignificant reduction in grain size in Mg-2.5HEA composite when compared to Mg-0.5HEA composite (Table 1) and
(b) distribution pattern of HEA alloy particles in which HEA particles are mostly decorated at the grain/particle boundaries in both Mg-0.5HEA and Mg-2.5HEA composites (Figure 3).

a

b

Figure 3: SEM micrographs showing reinforcement distribution in (a) Mg-0.5HEA and (b) Mg-2.5HEA composites.

Compressive Properties

The resultant compressive properties of pure Mg and Mg-HEA composites are shown in Table 2. The overall stress-strain curve of Mg and Mg-HEA composites is shown in Figure 4. An improvement in both 0.2% compressive yield strength (CYS) and ultimate compressive strength (UCS) was achieved in both Mg-0.5HEA and Mg-2.5HEA composites. The increased compressive yield strength in composites indicated that HEA alloy particles can effectively bear the applied load and transfer load from the matrix [8, 9]. Improved ultimate compressive strength can be attributed to low twinning deformation and slip dominated flow during compressive loading due to significant grain refinement in composites [10-12]. When compared to pure Mg, compressive failure strain was reduced in Mg-0.5HEA whereas a slight improvement in failure strain was observed in Mg-2.5HEA composite. A simultaneous improvement in strength and ductility with significant strength increment was realized from Mg-2.5HEA composition.

Table 2: Results of room temperature compressive properties.

Materials	0.2%CYS (MPa)	UCS (MPa)	CFS (%)
Mg	91 ± 8	267 ± 8	13 ± 2
Mg-0.5wt%HEA	98 ± 5	358 ± 9	8 ± 1
Mg-2.5wt%HEA	127 ± 5	414 ± 6	15 ± 1

Figure 4: Overall stress-strain curves of Mg and Mg-HEA composites.

Compressive Failure Analysis

Thefracture surface of Mg and Mg-HEA composites are shown in Figure 5. In pure Mg and Mg-2.5HEA composite, similar fracture features with smooth surface can be seen in Figure 5 a and 5 c. This observation conforms with the same level of failure strain exhibited by both materialsunder compression loading (Table 2). In Mg-0.5HEA, formation of short ridges and the presence of small cracks (arrow marks in Figure 5 b), indicated the evidence of brittle failure.

Figure 5: Compressive fracture surface of: (a) Mg, (b) Mg-0.5HEA composite and (c) Mg-2.5HEA composite.

CONCLUSION

The key conclusions from the present research on the synthesis of Mg-HEA composites are highlighted as follows:

1. Monolithic magnesium and magnesium composites containing 0.5wt% and 2.5wt% of ball milled high entropy alloy particles are synthesized using the hybrid microwave sintering approach, which can realize lower production cost for powder metallurgy processed materials.
2. Microstructure characterization revealed a significant reduction in grain size in the composites due to the presence of reinforcement alloy particles in Mg matrix. Reinforcement particles are mostly decorated at the grain/particle boundaries in both Mg-0.5HEA and Mg-2.5HEA composites.
3. Microhardness was increased with increasing presence of reinforcing particles in Mg matrix. The best combination of compressive strength and failure strain was achieved in Mg-2.5HEA composite.

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