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Processing, microstructure and properties of ultrasonically processed in situ $MgO–Al_2O_3–MgAl_2O_4$ dispersed magnesium alloy composites

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ABSTRACT

AZ91 alloy matrix composites are synthesized by in situ reactive formation of hard MgO and Al_2O_3 particles from the addition of magnesium nitrate to the molten alloy. The evolved oxygen from decomposition of magnesium nitrate reacts with molten magnesium to form magnesium oxide and with aluminium to form aluminium oxide. Additionally, these newly formed oxides react with each other to form $MgAl_2O_4$ spinel. Application of ultrasonic vibrations to the melt increased the uniformity of particle distribution, avoided agglomeration, and decreased porosity in the castings. Ultrasound induced physical phenomena such as cavitation and melt streaming promoted the in situ chemical reactions. Well dispersed, reactively formed hard oxides increased the hardness, ultimate strength, and strain-hardening exponent of the composites. Presence of well-dispersed hard oxide particles and stronger interface resulting from cavitation-enhanced wetting of reactively formed particles in the AZ91 alloy matrix improved the sliding wear resistance of the composites.

1. Introduction

Requirement of lightweight materials in automotive and aerospace applications has driven the efforts to improve the mechanical properties of magnesium and its alloys [1]. Magnesium alloys are an attractive material for designers because of their low density which is about two thirds that of aluminium [2]. Because of their high strength-to-weight ratio and low inertia, they find applications in rapidly moving parts such as automobile wheels and parts and sports equipment. It is necessary to pay attention to all the processing aspects of these alloys in order to optimize their properties. Light metal matrix composites (MMCs) provide alternatives for improving the mechanical properties. Numerous trials are done by addition of various hard ceramic reinforcement particles to magnesium and its alloys. Superior mechanical properties, good thermal stability, increased hardness, and stiffness are reported [3].

Ex-situ composite synthesis techniques are widely investigated for aluminium alloys by using different kinds of ceramic reinforcements like SiC, Al_2O_3, MgO and B_4C, whose properties such as refractoriness, high hardness, high compressive strength, and wear resistance make them suitable for use as reinforcement [4–6]. Ex-situ synthesized MMCs exhibit limited improvement in their mechanical properties because of presence of large sized reinforcements and bad interface between the matrix and reinforcements [7]. Further, it involves complex equipments and procedures and requires expensive reinforcement materials. It is challenging to disperse the micron and especially nano-sized particles uniformly in the alloy melts because these have much larger specific surface area and tend to agglomerate [8]. In this context, inexpensive in situ methods offer benefits such as homogeneous distribution of the reinforcing particles, a good reinforcement-matrix interface, and the in situ formed particles are finer [9–11]. Although extensively studied for aluminium matrix composites, this method is less explored for magnesium matrix composites. A recent review reports about Mg_2Si/Mg, MgO/Mg, TiC/Mg, and (TiB_2–TiB)/Mg in situ composites synthesized from Mg–Si, Mg–B_2O_3, Mg–Ti–C, Mg–KBF_4–K_2TiF_6, and MgO systems, respectively [12]. In order to eliminate the difficulties arising from poor wettability, addition of appropriate alloying elements to the matrix metal [12–15], surface treatment of reinforcements [16], and elevation of molten matrix temperature [14] have been widely studied. Porosity, which degrades the mechanical properties, is formed adjacent to the agglomerate particles in the MMCs when ceramic particles are transferred to the melt by stirring [16]. Therefore, in order to overcome the problems arising from poor wettability, novel production method for MMCs is desired. In this context, in situ reaction fabrication is one of the most economical and versatile approaches [17].

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A novel solidification processing technique involving ultrasonic treatment (UST) assisted casting process has been developed [8,17–20]. It is efficient in dispersing the nano particles in to the melts. Poor wettability of reinforcing particles arising because of the intrinsic properties of the material such as the surface energy of the matrix and the reinforcement, and the oxidation and contamination of particle surface can be overcome by the UST. It is one of the simplest and effective physical methods to homogenise and degas the melt [18], and to wet, de-agglomerate, and uniformly disperse the reinforcement particles [17–19].

In view of the aforesaid potential of in situ synthesis approach and the UST, both these techniques are coupled for the fabrication of AZ91 alloy matrix composites. The composites are reinforced with 6.5, 3, and 1 vol.% of oxides formed from the addition of magnesium nitrate that is easily available, low cost and easy to operate with. Formation of hard oxide reinforcement particles by in situ reactions is studied. Microstructure of the composites is characterised for the uniformity in the distribution of reinforcement particles and is correlated with the mechanical properties. By evaluating the dry sliding wear behaviour over a range of loads, the operating wear mechanisms are analysed.

2. Materials and methods

AZ91 alloy used in this study has a nominal composition of 9 wt% Al, 1 wt% Zn and balance Mg. Fig. 1 shows a UST system used for applying ultrasonic vibrations to the melt. It consists of a 1500 W high power ultrasonic probe unit (Model VCX 1500, Sonics and Materials, USA) with a 20 kHz acoustic generator, and an acoustic radiator made of Ti–6Al–4V titanium alloy. During the UST, temperature of the melt and time of application of ultrasonic vibrations were precisely controlled.

Experiments were performed on 400 g of AZ91 alloy melt at 700 °C by employing three different conditions. These are magnesium nitrate additions accompanied with manual stirring, magnesium nitrate additions coupled with ultrasonic stirring, and the control specimen of base alloy without any forced convection. Magnesium nitrate is suitably added to the AZ91 alloy melt for the in situ formation of 1, 3, and 6.5 vol.% oxides. Before stirring, the manual stirrer or ultrasonic radiator was preheated to the processing temperature. Ultrasonic processing was performed isothermally at 700 °C for 180 s by employing a high ultrasonic intensity of 4.3 kW cm^{-2} because it leads to improved microstructure in metals. After processing, the mild steel crucible (70 mm height, 50 mm diameter and 3 mm wall thickness) was immediately cooled in water. Heating and cooling conditions were consistent among all the experiments in order to ensure that the difference in the microstructure and the mechanical properties results exclusively from the difference in process control variables. Material designation used in the present work for different processing conditions is shown in Table 1.

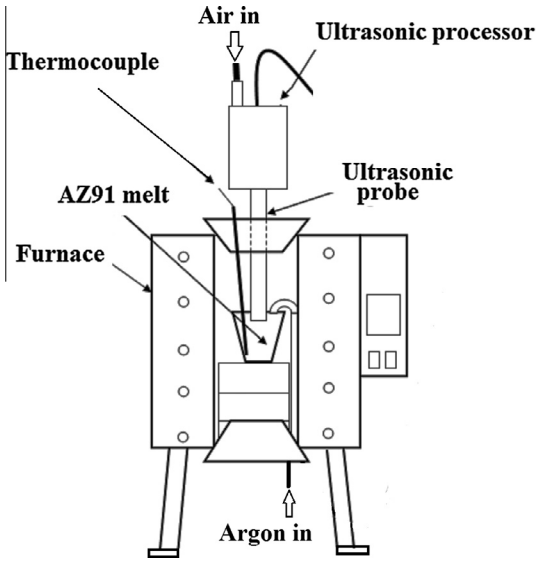

Fig. 1. Schematic diagram showing the experimental setup used in the processing of composites.

Table 1
Material designations used in the present work with AZ91 alloy matrix.

Vol.% of oxide formed	Ultrasonic processing condition	Material designation
0	Unprocessed	AZ91
1	Unprocessed	AZ91-1
1	Ultrasonically processed	AZ91-1-UST
3	Unprocessed	AZ91-3
3	Ultrasonically processed	AZ91-3-UST
6.5	Unprocessed	AZ91-6.5
6.5	Ultrasonically processed	AZ91-6.5-UST

Metallographic specimens were cut from the centre of the longitudinal section of each casting. Specimen preparation involved grinding, polishing with diamond paste, followed by acetic picral etch in order to highlight the contrast between the primary phase and the intermetallic phases. Microscopy was performed using a Leica DMI 5000 M light optical microscope and a FEI-Quanta 200 scanning electron microscope (SEM). The average volume fraction of all the in situ formed oxides is estimated by using Material Plus 4.1 image analysis software using ten micrographs each. Semi-quantitative analysis of dispersed phase particles was performed by using the EDS (energy dispersive X-ray spectroscopy). A Bruker D8 Advance X-ray diffractometer (XRD) was used for the confirmation of the in situ formed phases in the MMCs.

Compression tests were performed on Gleeble®3800 machine at room temperature using a constant strain rate of 9.5×10^{-4} s^{-1}. Test specimens 10 mm in diameter and 12 mm in gauge length were tested for each condition using lubrication to minimize friction at the ends. Yield stress is estimated from the stress–strain data taking the flow stress at 0.2% offset strain while σ_{max} was maximum value of flow stress. A FIE-VM50 PC Vickers hardness tester measured the hardness by employing 5 kg load. At least five hardness readings were obtained and the average is reported along with standard deviation.

For wear testing, a pin-on-disc type wear and friction monitor (Model TR-20 Ducom, India) was used. Pin samples 12 mm in height and 5 mm in diameter were machined from the castings. Wear test parameters are listed in Table 2. Before and after each wear test, test samples were cleaned, dried and weighed. For weighing, a Mettler AJ100 electronic balance having an accuracy of 0.0001 g was used. Separate specimen was used for each test and three tests were performed for each material. After the wear test, mass loss is measured and the average value of mass loss of

Table 2
Pin-on-disc wear test parameters.

Wear disc material	EN32 grade steel
Pin specimen	Cast AZ91 alloy and its composites
Applied force	4.9 N, 9.8 N and 14.7 N
Velocity	1 ms^{-1}
Test length (sliding distance)	500 m, 1000 m and 2000 m
Temperature	23 °C ± 2 °C
Humidity	40%