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Microstructure, mechanical properties and strengthening mechanisms of Mg matrix composites reinforced with in situ nanosized TiB_2 particles

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ABSTRACT

In this work, Mg matrix composites reinforced with 2.5 wt% nanosized TiB_2 particles are fabricated by adding Al-TiB_2 master alloy prepared by the chemical reaction of Al-K_2TiF_6-KBF_4 system into molten magnesium. The size distribution of TiB_2 particles extracted from the master alloy and their effects on the microstructure and mechanical properties of AZ91 matrix are investigated. The results show that the TiB_2 particles display a dominant number of particles less than 100 nm and a uniform distribution in the composites. Also, the grain size of composites is decreased compared with AZ91 alloy. The high resolution TEM reveals that the interface between nanosized TiB_2 particles and matrix is clear and bonds well. Besides, the yield strength, ultimate tensile strength and elongation to fracture of composites are improved by 49.4%, 25.5% and 51.1% respectively. Among the strengthening mechanisms of nanosized particles reinforced Mg matrix composites, dislocation strengthening and Orowan strengthening play a more important role in increasing the strength. It is noteworthy that the elongation to fracture of the composites is higher than that of AZ91 matrix, which is contrary to other works.

1. Introduction

Magnesium alloys as the lightest structure material are potential candidates in the aerospace and automotive industries due to their low density, good damping capacity, high specific strength and excellent castability [1–3]. However, low strength and ductility limit wide application of magnesium alloys [1]. In recent years, particulate reinforced magnesium matrix composites (PRMMCs) have attracted increasing attentions because of their excellent properties, such as high specific strength and modulus, low density as well as good wear resistance [4,5]. In general, the reinforcements such as Al_2O_3 [6,7], Y_2O_3 [8,9], SiC [10–12], CNT [13] and TiC [14] are used to fabricate Mg matrix composites by disintegrated melt deposition, stir casting and in situ casting methods. Among these methods, in situ casting shows a great potential in PRMMCs due to several advantages, such as good thermodynamic stability and clean interface between the reinforcement and the matrix [15].

In situ micro particles reinforced Mg matrix composites exhibit a considerably improved strength and wear resistance, but a significantly reduced ductility compared with the unreinforced Mg matrix [14,16–20]. In other words, micro particles often lead to an increase in strength of PRMMCs while ductility would be sacrificed. Some reports have shown that nanosized particles reinforced metal matrix composites can achieve excellent mechanical properties with a combination of higher strength and ductility [8,21–24]. Yang et al. [25] obtained in situ nano sized AlN/Mg composites with higher ultimate tensile strength and fracture elongation (16–47%) developed via bubbling nitrogen gas into molten Mg-Al alloy. However, most of the reports in literature are mainly focused on micro sized particles reinforced Mg matrix composites, and only a few works study the effect of in situ nanosized reinforcement on the microstructure and mechanical properties of Mg matrix composites. Moreover, TiB_2 ceramic particles are regarded as suitable reinforcement candidates due to its low density (4.5 g/cm^3), high elastic modulus (565 GPa), good wear resistance and the same crystal structure with magnesium [26]. In previous reports, nanosized TiB_2 particles reinforced Al matrix composites can be synthesized by the chemical reaction of Al-K_2TiF_6-KBF_4 system [27–29]. To the best of our knowledge, few open literatures have been carried out on in situ nanosized TiB_2/Mg composites. Therefore, it is essential to study the microstructure, interface characteristic, mechanical properties and strengthening mechanisms of in situ nanosized TiB_2/Mg composites.

In present study, the size distribution of TiB_2 particles extracted from Al-20 wt% TiB_2 master alloy prepared by the chemical reaction of Al-K_2TiF_6-KBF_4 system at 900 °C is characterized quantitatively. And then in situ TiB_2/AZ91 composites are fabricated by adding Al-20 wt%

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Table 1
Chemical composition of the AZ91 matrix alloy.

Element	Al	Zn	Mn	Mg
wt%	9.45	0.76	0.27	Balance

TiB_2 master alloy into molten magnesium assisted semisolid stirring technique. Moreover, the effect of TiB_2 particles on microstructure and mechanical properties of Mg matrix composites and strengthening mechanisms are discussed in detail.

2. Experimental procedure

2.1. Processing of in situ TiB_2/AZ91 composites

In this study, pure Mg, Al, Zn and Mg-5%Mn master alloy were used to prepare AZ91 matrix alloy, and the chemical composition of AZ91 alloy was given in Table 1. And 2.5 wt% TiB_2/AZ91 composites were fabricated by in situ mixed salts method with two steps. First, Al-20 wt % TiB_2 master alloy was prepared according to the chemical reaction of Al-K_2TiF_6-KBF_4 system, as given in equal (1) [27]. The Gibbs free energy of this reaction could be calculated as $\Delta G^{\theta} = -981990 + 112.8T$ using the thermodynamic data from literatures [30,31]. In order to accelerate the reaction rate, the temperature was selected as 900 ℃ with $\Delta G^{\theta}_{900^{\circ}C} = -849.68$kJ/mol, which means the reaction can take place. Two types salts of K_2TiF_6 and KBF_4 with a high purity of 99.5% were mixed well with a molar ratio of Ti/B = 1/2 and the mass of salts corresponded to the composition of Al-20 wt% TiB_2. When the melt temperature reached 900 ℃, the mixed salts were slowly added into the molten Al in a graphite crucible in an electrical resistance furnace. The Al melt was stirred at 100 rpm for 5 min using a zircon coated graphite rod every 10 min. After 60 min, the slags floated on the top layer of the Al melt was removed and then the melt was poured into a graphite mould.

$$3K_2TiF_6+6KBF_4+10Al \rightarrow 3TiB_2+9KAlF_4 + K_3AlF_6 \quad (1)$$

Second, about 1000 g Mg, Zn and Mg-5%Mn master alloy were first melted at 750 ℃ and held at this temperature for 15 min in the mild steel crucible in an electric resistance furnace under an argon protective atmosphere. Subsequently, the Al-TiB_2 master alloy was added into the Mg melt. The melt was kept at 750 ℃ until the Al-TiB_2 master alloy was dissolved. After the melt was cooled to the semi-solid condition, mechanical stirring was carried out at 300 rpm for 15 min by a stainless-steel stirrer to obtain a uniform distribution of TiB_2 particles in Mg matrix. Then, the composites melt was heated rapidly to 720 ℃ and casted into a preheated steel mould. In order to compare with the composites, the AZ91 matrix was also prepared by the same method. At last, the matrix alloy and composites were homogenized heat treatment at 410 ℃ for 24 h followed by quenching in warm water.

2.2. Extraction experiment for obtaining TiB_2 particles

The size distribution and actual weight percentage of TiB_2 particles in Al-TiB_2 master alloy were analyzed by chemical extraction experiment. A small ingot about 10 g cleaned by SiC sand paper was completely dissolved in 10 vol% HCl solution in a breaker for several hours. After dissolution, all particles were deposited at the bottom of the breaker, subsequently the solution of the top was poured out. Residual TiB_2 particles were washed thoroughly for several times until the supernatant reached neutral PH and finally was vacuum-dried at 60 ℃ for 8 h.

2.3. Mechanical properties test

The mechanical properties of specimens were studied by conducting Brinell hardness and tensile tests. The hardness test of materials was carried out in Brinell hardness testing at 250 kgf load for a dwell time of 30 s. The tensile test was conducted at room temperature at the mechanical testing machine (MTS, CMT5305, USA) with a tensile rate of 0.5 mm/min, and three specimens with dimensions of 15 mm length, 4 mm width and 2 mm thickness in the gauge section were tested to ensure the reproducibility of data according to the ASTM-E8 standard.

2.4. Material characterization

Phase identification was carried out by X-Ray Diffraction (XRD, Bruker D8 Advance) using Cu K_{α} radiation (λ = 1.54Å) at a scan rate of 0.2°/s. The size distributions of TiB_2 particles extracted from Al-TiB_2 master alloy were measured using Malvern Zetasizer Nano ZS, which could measure particle size ranging from 0.3 nm to 10 μm. Twelve measurements were conducted for TiB_2 particles diluted with ethanol. Actual weight percentage of TiB_2 particles in Al-20 wt% TiB_2 master alloy was measured for three times to obtain an average value.

Microstructure analysis was carried out using optical microscopy (OM), scanning electron microscopy(SEM), field emission scanning electron microscope (FESEM, Zeiss Gemini500) equipped with energy dispersive spectroscopy (EDS) and transmission electron microscopy (JEM-2100Plus). The specimens for OM and SEM analysis were grinded, polished and etched with 5% nitric acid. The equivalent grain size distribution was measured by Image-Pro Plus software. Quantitative estimations of the volume fraction of TiB_2 particles in the TiB_2/Mg composites were statistically analyzed using 20 different SEM micrographs by Image J software. The morphology of fractured surface of tensile specimen was also analyzed by OM and SEM. The TEM specimens were prepared by mechanical polishing to 50 μm thickness, punching 3 mm diameter discs and then ion-thinned.

3. Results and discussion

3.1. Al-TiB_2 master alloy

Fig. 1(a) shows XRD pattern of Al-TiB_2 master alloy. The existence of diffraction peaks of TiB_2 demonstrates that only TiB_2 phase is synthesized in Al-TiB_2 master alloy. It is also observed that diffraction peaks of intermediate product Al_3Ti or AlB_2 can not be detected, indicating completion of reaction (1). This is in agreement with the results [32] that TiB_2 is the most thermodynamically stable phase due to its lowest Gibbs free energy among these three compounds. Fig. 1(b) shows the SEM micrograph of Al-TiB_2 master alloy. It is clear that TiB_2 particles are mainly distributed along the boundaries of α-Al grains. However, some agglomerations of tiny TiB_2 particles are found as shown in Fig. 1(b).

3.2. Extracted TiB_2 particles

TiB_2 particles were extracted for a further study on morphology and size distribution as well as actual weight percentage of TiB_2 particles in Al-TiB_2 master alloy. Fig. 2(a) shows XRD pattern of extracted TiB_2 particles from Al-TiB_2 master alloy. Only TiB_2 phase peaks are detected, suggesting that there is no other intermetallic compound in Al-TiB_2 master alloy. In addition, the XRD result further confirms the purity of extracted TiB_2 particles, which indicates that the interference of other phases can be eliminated for the following calculation of actual weight percentage and the size distributions of TiB_2 particles in the master alloy. Fig. 2(b) presents FESEM morphology of TiB_2 particles extracted from Al-TiB_2 master alloy. As seen in Fig. 2(b), the particles mainly display rectangular and hexagonal shapes. It is worth to note that the morphology of some tiny particles is not typical hexagonal maybe due to the uncompleted growth for TiB_2 crystal [33]. Moreover, the actual weight percentage of TiB_2 particles in Al-TiB_2 master alloy is about 19.6 wt%.