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Fabrication of TiC particulate reinforced magnesium matrix composites

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Abstract

A TiC particulate reinforced magnesium matrix composite (PRMMC) was fabricated by adding a TiC-Al master alloy processed via self-propagating high-temperature synthesis reaction into molten magnesium and using the semisolid slurry stirring technique. The properties of the PRMMC are higher than those of the unreinforced magnesium alloy.

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1. Introduction

Particle reinforced magnesium matrix composites (PRMMCs) have a great potential to be applied in automobile and aerospace industries because of their high specific tensile strength and modulus, as well as their high wear resistance. In general, PRMMCs are produced by stir casting [1– 3], powder metallurgy [4], squeeze casting, and spray forming [5]. In all these methods, the reinforcing second-phase particles (including borides, carbides, and nitrides) are incorporated into molten magnesium by ex situ methods, and the reinforcing particle size is coarse, rarely below $\sim 5 \,\mu\text{m}$. Other main drawbacks that have to be overcome are the interfacial reaction and poor wettability

* Corresponding author. Tel./fax: +86-431-5705592. *E-mail address:* jjanggc@mail.jlu.edu.cn (Q.C. Jiang). between the reinforcements and the matrices due to surface contamination of the reinforcements.

It is well known that the properties of PRMMCs are controlled by the size and volume fraction of the reinforcement as well as the bonding characteristics of the matrix-reinforcement interfaces. The high mechanical properties can be obtained when fine and thermodynamically stable ceramic particles are dispersed homogeneously in the matrix. To meet such demands, PRMMCs have been developed in which the reinforcements were in situ synthesized in the metallic melt by chemical reactions among elements or between an element and a compound during the composite fabrication. In this case, however, the "inert" matrix acts as a diluent, which may make the propagation of the combustion wave unstable owing to the strong heat dissipation in the "inert" metal matrix [6]. The mechanisms responsible for the in situ formation of ceramic reinforcements in

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some reaction systems are still not well understood.

The purpose of the present study is to fabricate a TiC PRMMC by adding a TiC-Al master alloy processed via self-propagating high-temperature synthesis (SHS) reaction into molten magnesium and using the semi-solid slurry stirring technique. It is well established that SHS, also known as combustion synthesis, has many attractive advantages, such as high purity of product, low processing cost, and efficient in energy and time. In this study, TiC particulates were in situ formed in aluminum via SHS reaction. TiC particles in the master alloy are fine ($\sim 5 \mu m$), spherical, and the surface of TiC particles is coated with aluminum and is thus uncontaminated, and this has significantly improved the wetting of the TiC particles in the magnesium alloy. TiC particles can be easily incorporated into the magnesium alloy matrix by the TiC-Al master alloy, and dispersed uniformly in the matrix. Consequently, the properties of TiC PRMMCs have been improved.

2. Experimental procedures

The present fabrication technique consists of the preparation of a TiC–Al master alloy containing pure aluminum and TiC particles, and adding the master alloy into the molten magnesium alloy in an amount according to the desired volume fraction of TiC. The TiC–Al master alloy was processed by the SHS reaction of Ti–C–Al system. TiC particles and aluminum in the master alloy were used as reinforcements and alloying element, respectively.

To prepare the above mentioned master alloy, pure titanium (99.5%, particle size less than 25 μ m), aluminum (98.4%, particle size less than 27 μ m) and carbon (99.9%, particle size less than 38 μ m) powders were used. Powder blends with molar ratio of Ti/C = 1.0 mixed with 30 wt.% Al were prepared by ball milling, and were cold-isostaticly pressed at a pressure of 20 MPa. The cylindrical compact (35 mm diameter and 50 mm height) with about 85% theoretical density was heated to 873 K in a vacuum electric resistance furnace of 3 kW, and held at that temperature for 15 min. The bottom surface of the compact was ignited subsequently by applying a current of about 12 A through a resistance wire. TiC particulates with a size of $\sim 5 \,\mu\text{m}$ were in situ formed in aluminum via SHS reaction, and a master alloy containing pure aluminum and TiC particles is formed.

Processing of the TiC PRMMCs consists of adding the TiC-Al master alloy to molten magnesium, semi-solid slurry stirring, and casting. Based on the chemical compositions of Mg-9 pct Al-1 pct Zn alloy AZ91, commercially pure Mg, Al and Zn were selected to prepare the matrix. About 1 kg of pure magnesium melt was prepared at 1023 K in a steel crucible in an electric resistance furnace of 5 kW under a SF₆/CO₂ protective atmosphere. The desired amounts of the TiC-Al master alloy and alloying elements wrapped by aluminum foils were subsequently charged into the magnesium melt, held at that temperature until the TiC-Al master alloy and alloying elements were dissolved. In order to avoid a large temperature decrease during the addition of the master alloy and alloying elements, they were preheated to 200 °C prior to adding. Stirring was carried out in the semi-solid temperature range for 30-40 min, and then the composite slurry was poured into a preheated steel mould with 55 mm diameter and 180 mm height.

3. Results and discussion

SEM micrograph and XRD pattern of the TiC– Al master alloy are shown in Fig. 1(a) and (b), respectively. Because TiC particles were formed by the SHS reaction between the aluminum melt and graphite particles by titanium diffusion to their interface [7], TiC particles are fine (\sim 5 µm) and spherical, and since TiC particles are surrounded by aluminum, the particle surfaces are uncontaminated, which makes the wetting of TiC particles in the magnesium alloy significantly improved.

The presently prevalent combustion reaction mechanism of the Ti–C–Al system has been suggested by Lee and Chung [7] and Choi and Rhee [8]. The reaction between titanium and aluminum to form titanium aluminides $(TiAl_x)$ in the combustion reaction of the Ti–C–Al system occurred



Fig. 1. (a) SEM microstructure, and (b) XRD pattern of the TiC-30 wt.% Al master alloy.

initially, then followed by the $TiAl_x$ -C reaction to form TiC. Through XRD analysis, it was confirmed that the reaction product consists only of TiC and aluminum. The present experimental results agree well with those reported previously [7,8], as shown in Fig. 1(b).

From the SEM micrograph of the PRMMC shown in Fig. 2, it can be seen that TiC particles dispersed uniformly throughout the magnesium alloy matrix, and the agglomeration of TiC particles is not obvious. A high magnification SEM micrograph in Fig. 3(a) clearly shows the good and



Fig. 2. SEM micrograph of as-cast AZ91–10 vol.% TiC magnesium composites.

clean interface between TiC particles and matrix, and EDS line scans in Fig. 3(b) shows no segregation of Al and O atoms occurred at the interface, which indicates that no reaction products such as MgO or Al_2O_3 are formed at the interface. While it is generally reported that compounds like MgO or Mg₂Si are observed at the particle/matrix interface in SiCw/Mg composites [4,9]. It is concluded that magnesium wets TiC well and is a better "host" for particle embedment than aluminum.

Porosity is obviously reduced by inhibiting gas bubble formation, and limiting gas bubble size when stirring in the semi-solid temperature range [9].

Ambient temperature mechanical properties of the TiC/AZ91 composite and the unreinforced AZ91 Mg alloy are given in the Table 1. It is clear that the ultimate tensile strength (UTS) and the hardness (HB) of the AZ91–10 vol.% TiC composites are higher than those of the unreinforced AZ91 Mg alloy.

The sliding abrasive wear rates of the unreinforced AZ91 Mg alloy and its composite were tested under loads ranging from 5 to 35 N using a pin-on-disc apparatus. Both the unreinforced AZ91 Mg alloy and AZ91–10 vol.% TiC composite were used as pin materials with 6 mm diameter and 12 mm height, and 1000 mesh SiC abrasive papers (corresponding to 15 μ m abrasive particles) were used as the counterface. The results are listed in the Table 2. The wear rate of the



Fig. 3. (a) High magnification SEM micrograph of the TiC/AZ91 composite; (b) EDS line scans of C, O, Mg, Al and Ti.

Table 1 Mechanical properties of the as-cast AZ91 and AZ91–10 vol.% TiC composite at ambient temperature

Material	Hardness	UTS	Elongation
	(HB)	(MPa)	(%)
AZ91	60	160	9
AZ91–10 vol.% TiC	83	214	4

Table 2 Wear rates of the as-cast AZ91 Mg alloy and AZ91–10 vol.% TiC composite

Load (N)	Wear rate (10^{-3} g/m)	
	AZ91	AZ91-10 vol.% TiC
5	1.2059	0.4925
15	2.4271	1.1108
25	2.8084	1.5256
35	3.6696	1.8105

unreinforced AZ91 Mg alloy is generally twice as large as that of the PRMMC at all loads. Improvement of wear resistance of the PRMMC must result from the presence of TiC particles.

4. Conclusion

- Magnesium matrix composites reinforced with TiC particles can be fabricated by adding a TiC-Al master alloy processed via SHS reaction into molten magnesium and using the semi-solid slurry stirring technique.
- 2. TiC particles in the master alloy are fine, spherical, and since TiC particles are surrounded by aluminum, the particle surfaces are uncontaminated, which makes the wetting of TiC particles in the magnesium alloy significantly improved. Also by stirring, a PRMMC with dispersed homogenously clean fine TiC particles embedded in a magnesium alloy matrix can be obtained.
- 3. The properties of the PRMMC prepared such as UTS, hardness, and wear resistance are higher than those of the unreinforced magnesium alloy.

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