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Microstructure and interface characterization of a cast $Mg_2B_2O_5$ whisker reinforced AZ91D magnesium alloy composite

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ABSTRACT

In this study, the microstructure and interface of a $Mg_2B_2O_5$ whisker-reinforced magnesium composite were characterized using optical microscopy, transmission electron microscopy and X-ray diffraction. It was found that the $Mg_2B_2O_5$ whiskers have a twinned structure with the (2 0 2) as the twin plane and growth direction along [0 1 0]. The MgB_4O_7 particles and the globular Mg_2Si particles were observed within the $Mg_2B_2O_5$ whisker, and at the interface between the $Mg_2B_2O_5$ whiskers and Mg matrix, respectively. The MgO and MgB_2 phase formed at the matrix–whisker interface during vacuum-gas pressure infiltration process due to the interfacial reaction. The crystallographic orientation relationships between the $Mg_2B_2O_5$ whisker and the interfacial reaction products were found to be $[010]_{Mg_2B_2O_5}//[1\,1\,0]_{MgO}$ and $[1\,1\,0]_{MgO}//[2\bar{1}\bar{1}0]_{MgB_2}$, and $(202)_{Mg_2B_2O_5}//(0\,0\,2)_{MgO}$ and $(0\,0\,2)_{MgO}//(0001)_{MgB_2}$, respectively. The factors that influence the microstructure of the $Mg_2B_2O_5$ whiskers and the formation of the interfacial reaction products of Mg_2Si, MgO and MgB_2 phases were discussed.

1. Introduction

The needs of high-performance lightweight materials for some demanding applications and the availability of a wide variety of reinforcing materials and new processing techniques have led to extensive research efforts in the development of magnesium matrix composites using cost-effective fabrication technologies [1]. SiC whiskers have attracted much attention in the last two decades because of their superior mechanical properties [2]. However, there are some drawbacks for SiC whisker-reinforced magnesium matrix composite, e.g., the higher cost of SiC whiskers [3]. Recently, metal borate compounds including aluminum borate ($Al_{18}B_4O_{33}$ and $Al_4B_2O_9$) [4–6] and magnesium borates ($Mg_3B_2O_6$ [7] and $Mg_2B_2O_5$) nanowires/nanotubes [8–15] have been synthesized successfully. These metal borates have good mechanical properties, such as high Young's modulus and strength, good chemical inertness, and low thermal expansion coefficient [8,13]. Furthermore, the metal borates whisker is cheaper than silicon carbide whisker [8]. Normally, $Mg_2B_2O_5$ whisker is synthesized from byproducts of seawater desalting or the compounds containing Mg and B of saline, its cost is much lower than that of aluminum borate whisker [13]. The metal borate compounds are competitive alternatives of silicon carbide whiskers as reinforcement in metal–matrix composites (MMCs) and have been used in aluminum-based MMCs [16–22].

It is very important to understand the reinforcement–matrix interface in a MMC which is the region controlling efficiency of load transfer from matrix to reinforcements [3]. The interfacial microstructure in magnesium-based MMCs is different from that in aluminum-based MMCs due to the high reactivity of magnesium [23]. However, comparing with aluminum-based MMCs, few efforts have been made to study the interfacial microstructure in magnesium-based MMCs. In this study, we report for the first time, to our best knowledge, a work on the microstructure of $Mg_2B_2O_5$ whiskers reinforced AZ91D magnesium alloy composite to get insight into the nature of reinforcement–matrix interfaces.

2. Experimental procedure

2.1. Materials selection

The $Mg_2B_2O_5$ whiskers is used as reinforcement, which is supplied by Qinghai Institute of Salt Lakes, Chinese Academy of Sciences, and processed by flux method using magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and borax ($Na_2B_4O_7 \cdot 10H_2O$) as raw materials, and potassium chloride (KCl) as flux [10]. The morphology of as-received $Mg_2B_2O_5$ whiskers was examined using scanning electron microscope (SEM). White acicular $Mg_2B_2O_5$ whiskers, as shown in

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Fig. 1a, are with diameter in the range of 0.2–2.0 μm and length range of 10–50 μm. The selected matrix alloy was the popular AZ91D magnesium alloy with nominal composition of 8.3–9.3 Al, 0.35–1.0 Zn, 0.15–0.50 Mn, ⩽0.01 Si, ⩽0.03% Cu, ⩽0.02 Ni, ⩽0.005 Fe, and balance of Mg (in wt.%).

2.2. Composite preparation

Magnesium composite ingots with 50 vol.% $Mg_2B_2O_5$ whisker were fabricated by vacuum-gas pressure infiltration process. Firstly, $Mg_2B_2O_5$ whiskers were dispersed through a wet process and then pressed to a preform without any binder. The preform was dried in air at room temperature for 3 days, then in microwave oven at 100 °C for 2 h. To increase their strength, the performs in the mould were preheated at 1000 °C for about 3 h, and subsequently high pressure infiltrated by the AZ91D alloy melt (at 600 °C) under the protection of Ar gas. The infiltrating pressure was gradually increased to a pre-set level of 2.6 MPa in order to avoid distortion of the preform. The high pressure was maintained for 30 min until the molten metal solidified completely. The dimension of the specimens produced is 20 mm in diameter and 80 mm in height. The optical micrograph in Fig. 1b shows uniform distribution and random orientation of the $Mg_2B_2O_5$ whiskers in the magnesium matrix. The surface of the $Mg_2B_2O_5$ whiskers is clean and flat, which indicates the successful fabrication of $Mg_2B_2O_5$ whisker reinforced AZ91D matrix ($Mg_2B_2O_5$w/AZ91D) composite using vacuum-gas pressure infiltration technique and whisker preform without binder. However, in comparison with the as-received whisker shown in Fig. 1a, it can be seen that many whiskers have broken after pressure infiltration process.

2.3. Mechanical properties

To compare the mechanical properties of the as-cast AZ91D alloy and $Mg_2B_2O_5$w/AZ91 composite, the tensile tests were performed using Instron 200 testing machine at a crosshead speed of 0.5 mm/min. For each material, three tests on round-shaped tensile specimens with a gauge length of 30 mm and diameter of 5 mm were conducted at room temperature. The average results presented in Table 1 indicate the dramatic increase of $\sigma_{0.2}$, σ_b and Young's modulus for $Mg_2B_2O_5$w/AZ91 composite comparing with AZ91D cast alloy. However, the elongation to failure decreased significantly.

2.4. Analysis of phases and interfacial microstructures in the composite

Phase analysis of the $Mg_2B_2O_5$ whiskers was identified by means of X-ray diffraction (Cu Kα-radiation). The thin foils for transmission electron microscopy (TEM) observation were prepared using conventional procedure, including cutting 0.2 mm thick discs from the composite, mechanically polishing discs to 50 μm, dimpling to 20 μm and finally ion milling. The TEM observations by bright field imaging (BF), selected area electron diffraction (SAED), elemental analyses of the whisker/matrix interface, and characterization of phases in the composite were performed using Philips CM30 TEM equipped with energy dispersion spectroscopy (EDS) operated at 300 kV.

Table 1
Mechanical properties of the as-cast $Mg_2B_2O_5$w/AZ91D composite and AZ91D alloy.

Materials	E (GPa)	σ_b (MPa)	$\sigma_{0.2}$ (MPa)	ε (%)
AZ91D	45	165	97	7.3
$Mg_2B_2O_5$w/AZ91D	54	265	262	0.98

3. Results and discussion

3.1. Solidification microstructure in the $Mg_2B_2O_5$w/AZ91D composite

In the as-cast composite, no β-$Mg_{17}Al_{12}$ phase was observed by TEM. However, Mg_2Si compound was identified occasionally at interface of $Mg_2B_2O_5$ whisker and matrix. Fig. 2a and b is the TEM image and SAED pattern of the Mg_2Si phase, respectively. Obviously, the Mg_2Si particles are not nucleated on the $Mg_2B_2O_5$ whisker surface since the interlayer between Mg_2Si and $Mg_2B_2O_5$ whisker is observed. The microstructure of the as-cast AZ91D alloy has typically a primary α-phase matrix and a divorced eutectic consisting of large β-$Mg_{17}Al_{12}$ particles and eutectic α-Mg along the α-Mg phase grain boundaries [24]. The eutectic α-Mg phase is supersaturated with Al and can transform, by discontinuous precipitation of the β-$Mg_{17}Al_{12}$ phase below the eutectic temperature, to form a fine lamellar β-$Mg_{17}Al_{12}$ [25]. However, in the as-cast $Mg_2B_2O_5$w/AZ91D composite, no β-$Mg_{17}Al_{12}$ phase was observed by TEM. On the contrary, globular rather than the more common undesirable morphology of coarse Chinese-script Mg_2Si compound [26] was observed at whisker/matrix interface. Although there is almost no Si in the composite, the very small amount of Mg_2Si precipitates at interface during cooling is still possible since the solubility of Si in Mg is extremely low [26]. According to the binary diagram of Mg–Al and Mg–Si alloy, the liquid transformed into α-Mg and β-$Mg_{17}Al_{12}$ or Mg_2Si by the eutectic reaction (L → α-Mg + Mg_2Si (637.6 °C) and L → α-Mg + β-$Mg_{17}Al_{12}$ (437 °C)) [27]. Therefore, the Mg_2Si was formed at higher temperature than β-$Mg_{17}Al_{12}$. By this process, the formation of the low melting point β-$Mg_{17}Al_{12}$ eutectic phase might be suppressed [28]. The Mg_2Si particles formed at whisker/matrix interface are beneficial to the properties of the composite at both low and elevated temperatures [29]. However, the interfacial bond may not be so strong since the Mg_2Si particles are not nucleated on the $Mg_2B_2O_5$ whisker surface.

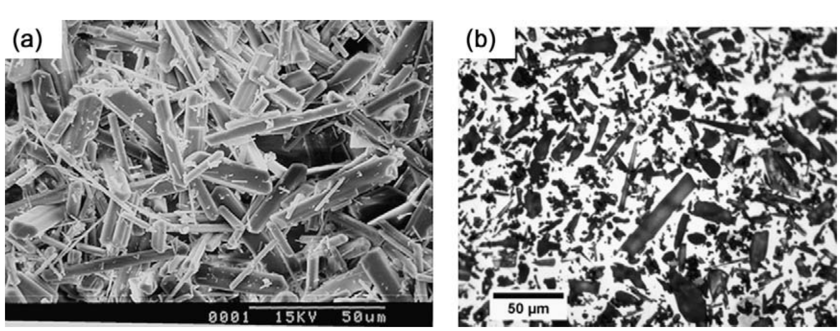

Fig. 1. (a) SEM micrograph showing the morphology of as-received $Mg_2B_2O_5$ whiskers; (b) Optical image showing the microstructure of as-cast AZ91D alloy based composite with 50 vol.% $Mg_2B_2O_5$ whiskers.