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# Compression behavior of Al<sub>2</sub>O<sub>3sf</sub>/Mg-6Al-0.5Nd-1Gd composites fabricated by pressureless infiltration and semi-solid densification



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#### ABSTRACT

Short fiber reinforced magnesium matrix composites containing rare earth elements ( $Al_2O_{3sf}/Mg$ -6Al-0.5Nd-1Gd composites) were fabricated by pressureless infiltration and semi-solid densification. The effect of combined addition of rare earth elements and short fiber on the compression strength and strain hardening behavior of the composites was investigated. The results showed that the refinement and reduction of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phases, the formation of thermally stable Al<sub>2</sub>RE and Mg<sub>2</sub>Si phases, and the moderate interfacial bonding enhanced the compression strength of the composites by 45.8% compared to the Mg-6Al alloys at room temperature. The composites also exhibited good strengthening effect even at temperatures above 200 °C, and the relative improvement in compression strength still reached 48.5% at 300 °C. The strain hardening exponents of the composites (evaluated using Hollomon's equation) were also increased greatly, which range from 0.43 to 0.87, and far greater than that of the Mg-6Al alloys (0.05–0.53).

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#### 1. Introduction

Magnesium alloys have received a great deal of attentions due to their exceptional characteristics including low density, high specific strength, attractive damping capacity and good recyclability, and has broad application prospective in aerospace, automotive, and 3C (computer, communication and consumer products) industries [1]. The Mg-Al series alloys are the most common commercial magnesium alloys due to their good castability and high strength. However, the inferior mechanical properties and poor creep resistance compared with common steel and aluminum alloys restrict their wide application [2]. To extend the application of Mg-Al series alloys, researchers have made great efforts based on the mechanism of matrix strengthening or grain boundary strengthening.

Addition of rare earth elements (RE, e.g. Gd, Y, Nd, Sm, Dy) to Mg-Al alloys can greatly improve their high temperature strength and creep resistance, because the presence of new thermally stable precipitates  $Al_{11}RE_3$  or  $Al_2RE$  phases can suppress the formation of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase, which is known to be the contributing phase for the poor creep resistance, and play an effective role in pinning the

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movement of dislocations during deformation [3–5]. It has been indicated that with addition of multiple RE elements, the properties of the alloy have a significant improvement than that with only one kind of rare earth element because of the interactive effects of multiple elements [6]. However, addition of high content of RE not only increases the cost of Mg alloys, but also increase their density, which weakens the advantage of high specific strength of Mg alloys compared with aluminum alloys. Reinforcing Mg-Al alloys with ceramic particles, fibers or whiskers (i.e. Mg-Al alloy based composites) is another effective approach, which have the advantages of low cost, light weight, and superior mechanical properties [7].

Mg-Al alloy based composites containing small amount of RE not only maintains the advantage of high specific strength, but also exhibits improved creep resistance. Hu et al. [8] fabricated short Saffil fiber reinforced Mg-Al-RE matrix composites by direct squeeze casting technique, and found that the rare-earth alloying elements (La, Ce) were distributed at the interface in the form of Al<sub>2</sub>RE particle phase on the surface of fiber, and Al<sub>11</sub>RE<sub>3</sub> lamellar phase in the matrix, which contributed to the great increase in tensile strength. Nd diffuses in Mg alloys slower than La, Ce, Pr, thus Mg alloys containing Nd have higher heat resistance. Wang et al. [9] prepared Al<sub>2</sub>O<sub>3f</sub>/Mg-9Al composite containing minor Nd by pressureless infiltration method, and found that after adding Nd to the composite, the microstructure of matrix alloy was refined

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obviously, formation and growth of Mg<sub>2</sub>Si were suppressed, which promoted the composite hardness. Moreover, thermally stable precipitates  $Al_{11}Nd_3$  and  $Al_3Nd$  precipitated at grain boundaries, and reduced the mobility of grain boundaries, thus improved the high-temperature creep resistance of the composites [10]. However, Nd hardly dissolves into  $\alpha$ -Mg, and further addition of Nd (above 1 wt %) may impair the tensile properties of Mg-Al alloys [11]. Gd has higher solubility than Nd in Mg alloys, and has strong aging strengthening and precipitate strengthening effect [12]. Zhu et al. [13] reported that with the combined addition of Nd and Gd, highly rigid precipitation  $Al_2Gd$  and  $Al_2Nd$  phase can effectively impede dislocation movement and thus improve the yield strength of Mg-Al alloys. However, there are little literature which discussed the effect of combined addition of Gd and Nd on the mechanical properties of Mg-Al alloy based composites.

In present work, minor Gd and Nd were added to Mg-6Al alloy, and  $Al_2O_3$  short fibers were used to fabricate magnesium matrix composites by pressureless infiltration followed by semi-solid densification. The combined effect of the RE elements and fibers on the microstructure and compression strength of the composites were investigated.

#### 2. Experimental procedures

A RE magnesium alloy Mg-6Al-0.5Nd-1Gd was selected as the matrix alloy, which was prepared from a high purity Mg (>99.95%), Al (>99.98%), and Mg-30Nd (wt. %) and Mg-30Gd (wt. %) master alloys (supplied by Magontec Xi'an Co., Ltd) in a mild steel crucible under a protective flux cover. In order to prevent chemical reaction between liquid magnesium alloy and mild steel crucible, crucible wall was coated with a protective coating (65 wt % water-30 wt % ZnO-5 wt. % sodium silicate solution) before melting. When furnace temperature rises to 750 °C, molten alloy was stirred for about 10 min, and then cast into a permanent mold with 100 m in diameter, 160 mm in height. The as-cast ingot was cooled down at air atmosphere. Al<sub>2</sub>O<sub>3</sub> short fibers supplied by Luoyang refractory ceramic fiber Co. Ltd (3–10  $\mu$ m in diameter, Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>: >99 wt%, Al<sub>2</sub>O<sub>3</sub>:78–81 wt%) were used as reinforcement, and fabricated into cylindrical preforms with a volume fraction of 20% by wet forming method, in which silica sol (containing 10 wt % SiO2, supplied by Xi'an Fu Li Chemicals Plant), ethyl alcohol (≥99.5 wt%, supplied by Xi'an Chemical Reagent Factory) and high purity graphite particles (less than 30  $\mu m$  in diameter, supplied by Shanghai Shan Pu Chemical Co., Ltd.) were added into the wet slurry as binder, solvent and pore generators, respectively. After mechanical stirring and forming the shape of preforms under a pressure, the obtained wet preform was dried at room temperature for 24 h, at 100 °C for 12 h, and sintered at 900 °C for 12 h to burn the graphite particles into CO2.

Al<sub>2</sub>O<sub>3sf</sub>/Mg-6Al-0.5Nd-1Gd composites were fabricated by pressureless infiltration method followed by semisolid densification. Firstly, the preform was placed at the bottom of a graphite crucible, and the prepared Mg alloy blocks were placed on the top of the preform, and then heated in a vacuum Mo wire resistance furnace (10 Pa). Secondly, when the temperature in the furnace reached 660 °C, they were held for 90 min for spontaneous infiltration. Finally, the molten composites were cooled to room temperature inside the furnace. To further eliminate the pores in the composites, the obtained composites were densified under pressure in the semisolid state. In order to determine the solidification interval, differential scanning calorimetry (DSC) was performed on the Al<sub>2</sub>O<sub>3sf</sub>/Mg-6Al-0.5Nd-1Gd composites. According to the DSC curve shown in Fig. 1, the incipient melting temperature of the composites is 432  $^{\circ}$ C, at which the low melting point eutectic phase in the composites starts to melt. The remelting temperature was set

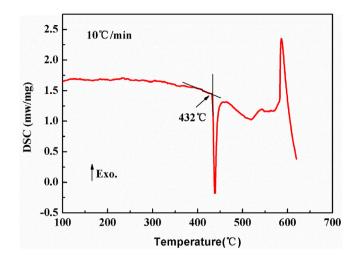


Fig. 1. DSC curve of  $Al_2O_{3sf}/Mg$ -6Al-0.5Nd-1Gd composites showing the incipient melting point.

to 480 °C in densification process. When the composites billet ( $\Phi$ 19 × 15 mm) placed inside a closed die (with 20 mm in inner diameter) was rapidly heated to the preset temperature and held for 30 min, the composites billet was subjected to a compression load of 100 MPa for 10min before furnace cooling.

The metallographic samples of as-cast Mg-6Al alloys, Mg-6Al-0.5Nd-1Gd alloys, and the densified composites were mechanically ground, polished and etched with an acetic-picric acid solution, and then examined by Olympus GX71 optical microscope (OM) and JSM-6700F scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS). Phase identification was confirmed by XRD-7000 type X-ray diffractometer (XRD). Compression tests were performed in CMT5304-30 KN computer-controlled electronic universal testing machine at 20 °C, 100 °C, 150 °C, 200 °C, 250 °C, and 300 °C with a constant crosshead speed of 0.5 mm/min  $\Phi8 \times 12$  mm samples for compression tests were machined from the densified composite ingots. For comparison purpose, compression tests of monolithic Mg-6Al alloys were also conducted under the same condition.

#### 3. Results and discussion

#### 3.1. Microstructure

Fig. 2 shows the microstructure of Mg-6Al alloys, Mg-6Al-0.5Nd-1Gd alloys, and densified Al2O3sf/Mg-6Al-0.5Nd-1Gd composites. It can be seen the microstructure of Mg-6Al alloy exhibits a dendritic microstructure composed of  $\alpha$ -Mg and reticular eutectic comprising of  $\alpha$ -Mg and  $\beta$ -Mg<sub>17</sub>A<sub>12</sub> phases in the interdendritic regions, as shown in Fig. 2(a). After combined addition of minor Nd and Gd, the coarse β-Mg<sub>17</sub>Al<sub>12</sub> phase is refined, and their volume fraction measured by an image analyzer decreases slightly from 12.6 vol % to 11.2 vol %, as shown in Fig. 2(b). From Fig. 2(c), it can be seen that the composites are mainly composed of white  $\alpha$ -Mg, dark-gray network phase, and uniformly dispersed fibers (black acicular or dot-shaped phase) in the transverse section perpendicular to infiltration direction. Most fibers are distributed in a random and isotropic orientation without agglomeration and pores in the matrix, and some long fibers were broken under pressure during the densification. There are some polygonal block-shaped phases near the fibers. Fig. 2(d) shows magnified structure of the composites, it can be seen that there exist a great deal of lamellar phases in the matrix.

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