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Effect of long-period-stacking-ordered phases on the microstructure and mechanical properties of carbon fiber reinforced magnesium-gadolinium-zinc composite

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ABSTRACT

In this study, long-period-stacking-ordered (LPSO) phases were introduced to carbon fiber reinforced magnesium-gadolinium-zinc (Cf/Mg-Gd-Zn) composite by adding Gd and Zn with the atomic rate of 2:1 into the matrix, and a new and practical method of design and optimize the interfacial structure was found. The Cf/Mg-Gd-Zn composite was fabricated by pressure infiltration method, and the influence of the LPSO phases on the microstructure and mechanical properties were investigated. LPSO phases were precipitated both at the interface and in the matrix. The LPSO phases at the interface were long rod-like, whose preferential growth direction was almost perpendicular to the axis of carbon fibers. But the LPSO phases formed in the matrix tended to be long and short rod-like, and arranged in a certain angle with the axis of the adjacent carbon fiber. Because of the LPSO phases, the interfacial structure of the composites was significantly optimized. Therefore, the mechanical properties of Cf/Mg-Gd-Zn composite were improved considerably. Specifically, compared with Cf/Mg composite, the interlaminar shear strength and bending strength of Cf/Mg-Gd-Zn composite were improved by 26.4% and 25.7%, respectively.

1. Introduction

Carbon fiber reinforced magnesium matrix (Cf/Mg) composites have been candidates in various industrial fields, especially in the aerospace industry, due to their unique properties, such as high specific strength and stiffness. It is well known that mechanical properties of Cf/Mg composites are influenced remarkably by the Cf-Mg interface. However, the wetting between carbon fiber and liquid Mg is so poor that the interfacial bonding is weak. Consequently, the study on designing and optimizing the interfacial structure is very important and necessary. According to former researches, there are two common methods to improve the interfacial bonding of Cf/Mg composites. One of them is to prepare coatings on the surface of carbon fibers before fabricating composites. Wang et al. [1] used 5.0 mol.% yttria stabilized zirconia (YSZ) to coat carbon fibers by a sol-gel route and then fabricated Cf/Mg composite, the tensile strength of the composite was thus increased to 1.08 GPa. But the main drawbacks of this way are that the coating layer is destroyed easily during the fabrication process of the composite, and the process of preparing coatings is complex and costly. Besides that, adding metal elements to the matrix is another practical method, and the most effective elements are Gd and Y [2,3]. Zhang et al. [3] found that Gd atoms are enriched at the interface and formed Mg-Gd intermetallic, and the interlaminar shear strength was improved slightly. Nonetheless, too much Mg-Gd intermetallic led to the brittle fracture even under low load.

A new and special phase named long-period-stacking-ordered (LPSO) phase with excellent properties was reported in magnesium-rare earth-transition metal (Mg-RE-TM) ternary alloy in 1994. And then, the study about its structure and feature are carried out constantly until now. The thin plate-shaped LPSO phases can boost the strength and plasticity of Mg alloys at the same time. Thus, it has become an important strengthening phase in Mg alloy [4–7]. The most popular study about LPSO phases concentrated on Mg-Gd-Zn and Mg-Y-Zn alloy systems. He et al. [8] introduced LPSO phase to the Mg-Y-Zn-Al alloy and improved the

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tensile yield strength and ultimate tensile strength by about 113–144% and 245–290%, respectively. 14H-type LPSO phase was found in a Mg-13Gd-5Er-1Zn-0.3Zr alloy, and the ultimate tensile strength of the extruded alloy was improved to 410.9 MPa [9]. However, the influence of LPSO phase in the continuous fiber reinforced composites has not been investigated.

In this study, LPSO phase was introduced into the Cf/Mg composite by adding Gd and Zn, and brittle Mg-Gd intermetallic was inhibited at the same time. The effect of LPSO phase on the interlaminar shear strength and bending strength of the Cf/Mg-Gd-Zn composite were investigated.

2. Materials and methods

In this study, carbon fiber reinforced Mg-Gd-Zn (Cf/Mg-Gd-Zn) composite was fabricated by pressure infiltration method. Carbon fiber (M40J) with the diameter of 7 μm was winded to be a unidirectional preform firstly. Ternary alloy was prepared by melting pure Mg (99.97%), Mg-30Gd (wt.%) master alloy and pure Zn (99.95%) in a graphite crucible in the argon atmosphere at 750 °C. Former study have found that it is easier to form LPSO phase, when Gd(Y) and Zn atomic ratio is close to 2:1 in Mg-Gd(Y)-Zn alloys [10,11]. Therefore, the target composition of the matrix was chosen as $Mg_{97}Gd_2Zn_1$. Before the infiltration process, the preform was put into a steel mold and was preheated at 500 °C. During the infiltration process, a pressure of 5 MPa was applied and maintained for 10 min, followed by the solidification of the composite in air. In order to promote the precipitation of LPSO, Cf/Mg-Gd-Zn composite specimens were treated at 480 °C for 8 h followed by water quenching and then were annealed at 225 °C for 16 h in the electrothermostatic blast oven (DGG-9240BD).

The compositions of Gd and Zn in the Mg matrix alloy are 1.75 at.% and 1.34 at.%, respectively, which was detected by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analysis. The volume fraction of carbon fiber is about 55% in the composite, which was measured and calculated by analyzing a large number of scanning electron microscope pictures. The morphology, distribution, and crystal structure of LPSO phases were observed and analyzed by a transmission electron microscope (TEM, Tecnai G2 F30), and the chemical compositions were detected by energy dispersive spectroscopy (EDS) in the TEM. The accelerating voltage of the TEM is 200.0 kV. Specimens for TEM observation were mechanically ground to a thickness of 50 μm and then thinned by ion milling.

The mechanical properties of Cf/Mg-Gd-Zn composite including bending strength and interlaminar shear strength were tested on a universal testing machine (INSTRON-5569), according to ASTM: D2344/D2344M-13 and ASTM: D790-10, respectively. The specimen dimensions for flexural and interlaminar tests were 2 × 10 × 60 mm and 2.5 × 5 × 15 mm, and the span/thickness ratios were 20 and 4, respectively. Both the tests were conducted with a crosshead rate of 0.5 mm/min.

3. Results

3.1. LPSO phase analysis

3.1.1. LPSO phase at the interface

Long rod-like LPSO phases were observed at the interface of Cf/Mg-Gd-Zn composite, as shown in Fig. 1 (a) and (e). The LPSO phases seemed to nucleate on the surface of carbon fibers, whose preferential growth direction was about perpendicular to the axis of carbon fibers. As measured from the TEM pictures, the length of LPSO phases ranged from 200 nm to 1000 nm. The chemical composition of the LPSO phases detected by EDS is $Mg_{79.06}Gd_{19.88}Zn_{1.06}$ (at.%), as referred to Table 1. There is a great difference to the ideal composition of the LPSO precipitate ($Mg_{12}Gd_1Zn_1$) found in Mg-Gd-Zn alloys [12]. Our former research demonstrated that Gd prefers to segregate at the fiber surface during the fabrication process of the composite while Zn tends to distribute in the matrix uniformly [13]. Therefore, the atomic ratio of Gd and Zn in the LPSO phase on the surface of carbon fibers did not match 1:1. Fig. 1 (b) is an HRTEM image taken from the region framed with the red rectangle in (a) along $[2\bar{1}\bar{1}0]_{Mg}$ zone axis, which illustrates the microstructure of LPSO phases and Mg matrix. Fig. 1 (c) and (d) illustrate the fast Fourier transformation (FFT) patterns inversed from the matrix and LPSO phases in (b), respectively. According to Fig. 1 (b)-(d), the LPSO phases have an orientation relationship with Mg matrix, namely $[2\bar{1}\bar{1}0]_{Mg}//[2\bar{1}\bar{1}0]_{LPSO}$, $(0001)_{Mg}//(0001)_{LPSO}$, $d_{(0001)_{Mg}} = d_{(0001)_{LPSO}} = 0.5241$nm, which is consistent with the conclusion drawn in Mg-Gd-Zn alloys [9]. In addition, it is clearly that the c-axis ([0001] direction) of the LPSO phase is about perpendicular to the fibers axis by analyzing Fig. 1 (b) and (d), as shown in the red arrow in Fig. 1 (a). That was because the basal plane of LPSO phases with hcp structure nucleated on the surface of the fiber firstly, and then stacked along the [0001] direction during the formation process.

Another HRTEM image taken from the region framed with the red rectangle in (e) along $[0001]_{Mg}$ is shown in Fig. 1 (f), and relevant FFT patterns inversed from the matrix and LPSO phases are shown in Fig. 1 (g) and (h). No special orientation relationships between Mg and the LPSO phase were observed along this zone axis in Fig. 1 (f), and the boundary is straight. The main strong diffraction spots in Fig. 1 (g) came from Mg matrix while the weak diffraction annulus came from carbon fibers. Streaks clearly observed in Fig. 1 (h) were confirmed to be the disorder of solute atoms in LPSO structure, which is a common phenomenon in LPSO phases in Mg-Gd-Zn alloys [9,14].

3.1.2. LPSO phases in the matrix

A mass of LPSO phases with two different morphologies are also found in the matrix apart from at the interface. One of LPSO phases in the matrix was long rod-like, sharing the same shape and size distribution with those at the interface. These LPSO phases dispersed parallelly to each other and arranged in a certain angle with the axis of adjacent carbon fibers, as observed from Fig. 2 (a). Besides that, numerous short rod-like LPSO phases with the length of around 100–400 nm also distinctly observed in the matrix (Fig. 2 (b)). Fig. 2 (d) and (e) are SAED patterns obtained from the long rod-like LPSO phase in Fig. 2 (a) along $[01\bar{1}0]_{LPSO}$ and from the short rod-like LPSO phase in Fig. 2 (b) along $[2\bar{1}\bar{1}0]_{LPSO}$, respectively. It is indicated that the long and short rod-like LPSO phases shared same structure by calculation of the lattice constants. Interestingly, superlattice spots and streaks were observed in the two SAED patterns, respectively. This result also revealed the LPSO structure with stacking fault and disorder of solute atoms, which is constant with that on the carbon fiber surface. Fig. 2 (c) is a magnified image of the short rod-like LPSO phase in (b), and a HRTEM image was taken from the interface of the LPSO phase and matrix framed with the red rectangle in (c), as shown in Fig. 2 (f). According to the HRTEM image, the short rod-like LPSO phase also share complete coherent boundary with matrix. Moreover, unlike the LPSO phases at the interface, the chemical composition of which in the matrix is $Mg_{84.96}Zn_{15.03}$ confirmed by EDS (shown in Table 2).

3.2. Mechanical properties

Fig. 3 illustrates mechanical properties of Cf/Mg [3], Cf/Mg-Gd [3] and Cf/Mg-Gd-Zn composites. It can be seen clearly from Fig. 3 (a) and (b) that the interlaminar shear strength and bending