Microstructure and mechanical properties of graphite fiber-reinforced high-purity aluminum matrix composite

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Received: 23 December 2008 / Accepted: 22 May 2009 / Published online: 5 June 2009 Springer Science+Business Media, LLC 2009

Abstract Industrial pure aluminum (0.5 wt% impurity elements) was utilized in many investigations of aluminum matrix composites at home and abroad. However, impurity elements in industrial pure aluminum may influence the interface during fabrication of composite at high temperature. Thereby, it is necessary to use high-purity aluminum (impurity elements less than 0.01%) as matrix to enable study the interface reaction between reinforcement and matrix. In this study, stretches of brittle Al_4C_3 at the fiber/ matrix interfaces in $\text{Gr}_{\mathfrak{g}}/A1$ composite were observed. The fracture surface of the composite after tensile and bending tests was flat with no fiber pull-out, which revealed characteristic of brittle fracture. This was related to Al_4C_3 , as this brittle phase may break before the fiber during loading and become a crack initiation point, while the corresponding crack may propagate in the fiber and the surrounding aluminum matrix, finally resulting in low stress fracture of composites.

Introduction

Metal matrix composites (MMCs) consist of two types of components with different physical and chemical properties. The main factors affecting properties for MMCs are reinforcement, matrix alloy, and interface bonding [\[1](#page-4-0)]. Aluminum alloys are of interest because of their low density, high strength and toughness, and good corrosion resistance for aerospace and automotive applications. More specifically, carbon fiber-reinforced aluminum alloy composites have been receiving considerable attention because of their high specific strength modulus of elasticity, high thermal conductivity, low coefficient of thermal expansion, dimensional stability and designability characteristic. During the processing of composite, the main problems encountered are non-wetting conditions between monolithic phases, and the undesirable formation of aluminum carbides (Al_4C_3) at the interface [\[2](#page-4-0)]. Some common aluminum alloys are chosen as the matrix for the composites, in which the impurity is inevitable during the fabrication. Thus, the effects of impurity of elements on the interfaces and mechanical properties of the composites need discussion [[3,](#page-4-0) [4\]](#page-4-0). In this study, high-purity aluminum alloy is chosen as the matrix of the graphite fiber-reinforced aluminum alloy composites. The microstructure and interfacial bonding, as well as the failure mechanism of the composites are discussed.

Materials and experimental methods

 $Gr_f/A1$ composite was fabricated by a pressure infiltration method by using M40 graphite fiber to reinforce highpurity aluminum (99.99 wt%); the volume content of the M40 graphite fiber in the composites is about 60 vol%. Gr_f/A l composite was fabricated by a pressure infiltration method, the temperature for melted Al alloy and mould was 750 and 500 \degree C, respectively. During the infiltration process, a pressure of 25 MPa was applied and kept for 10 min, and then solidified at the air. The morphology of composite was observed by ZEISS-40MAT optical microscope. The microstructure of interface was investigated by PhilipsCM-12 transmission electron microscopy

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(TEM) with a voltage of 200 kV. The diameter for TEM observation specimen is 3 mm and the thickness is about 10 μm. The final perforation to electron transparency was carried out by argon-ion sputtering in a Duo Mill apparatus. The morphology of fiber was characterized by S-4700 scanning electron microscopy (SEM). The composition and content of M40 fiber and $Gr_f/A1$ composite was verified by X-ray diffraction (XRD); the specimens were subjected to a scanning speed set at 2° min⁻¹. 2θ scans were performed between 20° and 100° .

The measurement standard for tensile specimens is as per ASTM D3352-96, and that for the bending standard ASTM D790 M. Bending tests and tensile tests were conducted using Instron5569 universal electronic tensile testing machine at room temperature with a crosshead speed of 0.5 mm/min. The sample sizes used were 60 mm \times 10 mm \times 2 mm and 120 mm \times 10 mm \times 2 mm for the respective test. Six specimens were tested and an average value was obtained. The fracture surfaces were characterized by S-4700 scanning electron microscopy (SEM).

Fig. 1 Micrographs of Gr_f/Al composite: a transverse to fiber axis; **b** vertical to fiber axis (optical microscope)

Results and discussion

Microstructure

Figure 1 shows typical micrographs of composite indicating that the composite was well infiltrated with good fiber dispersion without apparent porosity, or significant casting defects which seemed to be beneficial to composite properties.

The graphite fibers within the composites were extracted by the 40 wt% NaOH solution at 373 K. As is shown in Fig. 2a, the diameter of M40 graphite fiber was about $5-6 \mu m$, and some grooves parallel to fiber-axis in their surface layer were observed. However, although grooves in extracted fibers of the composite were not obvious, the surface of the fiber was rugged, which may result from interface reaction, and significantly affected the mechanical properties.

XRD analysis

The XRD pattern of M40 graphite fiber (Fig. [3](#page-2-0)) shows that diffraction peak of fiber was narrow, which indicated high

Fig. 2 Surface morphology of M40 fiber: a original fiber; b extracted fiber (SEM)

Fig. 3 X-ray diffraction pattern exhibited by as-received M40 graphite fiber

degree of graphitization, and better lamellar structure orientation than that of common carbon fiber. In addition, the surface of fiber was microcrystalline graphite, having high specific surface area without defects, which resulted in low chemical activity of carbon fiber with high degree of graphitization.

There was a great amount Al_4C_3 phase observed in the composite according to XRD pattern (Fig. 4), which contradicted with many previous research results [[5,](#page-4-0) [6](#page-4-0)]. Most results showed that M40 graphite fiber with high degree of graphitization has low chemical activity, which makes it suitable as a reinforcement material for aluminum matrix composite.

The formation of Al_4C_3 phase resulted from the interface reaction between fiber and matrix, which is disadvantageous to the mechanical properties. The reaction rates can be reduced by either the reduction or even the

Fig. 4 X-ray diffraction pattern exhibited by composite

elimination to a large extent of the solubility of the reinforcing agent in the matrix. Addition of an appropriate component to the matrix which can decrease the solubility of the reinforcing constituent in the MMC system can fulfill this requirement [\[7](#page-4-0)]. Hence, high-purity aluminum seems to be not a suitable as matrix in composites under this processing method.

TEM analysis of interface

The undesirable formation of aluminum carbides $(A\lambda C_3)$ at the interface is often observed in C/Al composites fabricated at high temperatures. At 670° C, the free energy of formation of Al_4C_3 is -176 kJ/mol [[2\]](#page-4-0).

M40/Al composite (Fig. 5a) shows extents of Al_4C_3 needles at the fiber/matrix interfaces, and there is an orientation relationship between Al_4C_3 and matrix. Al_4C_3 needles were about 830 nm long, 50 nm wide, and with an

Fig. 5 Interface of Gr_f/Al composite: a Al_4C_3 phase; b Al_4C_3 bridging two Gr fiber (TEM)

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aspect ratio about 16.6. In other words, specific surface area of Al_4C_3 is large in two-dimensional space, and the larger the specific surface area, the larger the interface of fiber and aluminum, thus resulting in strong interface bonding which leads to low mechanical strength. The carbide phase Al_4C_3 grows as acicular or lamellar inclusions which embed themselves both in the fibers and in the matrix, and then bridge two fibers in the regions where fibers are close to each other (Fig. [5](#page-2-0)b).

Moreover, despite their limited size, previous results [\[8–10](#page-4-0)] suggest that carbide grains can develop cracks which have the critical size to induce a fiber failure. When a crack notches the reinforcement, because of the turbostratic structure of the carbon and the axial tension at the $\text{Al}_4\text{C}_3/\text{C}$ interface, it will be soon deflected in a direction parallel to the interface. In the case of a bare fiber in this study, the notch will initiate a fiber failure when the tensile stress is high enough.

Table 1 Mechanical properties of matrix and composite

Material	Bending	Tensile	Elastic
	strength (MPa)	strength (MPa)	modulus (GPa)
Al_{pure}	404.8	46	62
Gr_f/Al		372.9	259

Mechanical properties and fracture analysis

Tensile tests and bending tests of composites and matrix were conducted at room temperature. The results indicated that mechanical properties and elastic modulus of composites were improved greatly by reinforcement (Table 1). However, the tensile and bending strength measured is 10% less than the value calculated by rule of mixture. This may be due to Al_4C_3 . During loading, while the reaction is strong, these brittle Al_4C_3 may break before the fiber due to strong interface bonding, and then become crack initiated. The corresponding crack may propagate in the fiber and the surrounding aluminum matrix finally resulting in low stress fracture of composites [\[11](#page-4-0)]. The tensile fracture surface of composite is shown in Fig. 6a, and the magnification in Fig. 6b. Moreover, the bending fracture surface of composites is shown in Fig. 6, and the magnification in Fig. 6d. The fracture surface of composites was flat with no fiber pull-out, which indicated strong interface bonding. Compared to the matrix alloy, the composite exhibits significant increases in modulus and strength. In general, the increase in the composite strength depends on the transfer of stress from the matrix to the carbon fibers [[12,](#page-4-0) [13](#page-4-0)], the differential nature between the composite matrix and the matrix material without reinforcement [[14\]](#page-4-0), the reduction in composite grain size $[15]$ $[15]$, and the generation of a high dislocation density in the matrix as a result of the

Fig. 6 Fracture of Gr_f/Al composite: a and b tensile fracture; c and d bending fracture (SEM)

difference in thermal expansion between the metal matrix and the carbon fibers [16].

In the case of continuous fiber-reinforced alloys in this study, load transfer is the major mechanism affecting the strength of the composite, the other mechanisms being negligible. During loading, while the reaction is strong, these brittle Al_4C_3 may break before the fiber due to strong interface bonding, then become crack initiated, and the corresponding crack may propagate in the fiber and the surrounding aluminum matrix, finally resulting in low stress fracture of composites, which is a result of loss of strength suffered by the fibers [17]. These cracks are likely to have been initiated at the point of maximum stress concentration and propagated along the fiber–matrix interface before the specimen failed, and hence, the stress could not be transferred from the matrix to the carbon fibers, and this finally resulted in lower tensile and bending strength than the value calculated by rule of mixture.

Conclusions

- (1) Composite was well infiltrated with good fiber dispersion, and neither apparent porosity nor significant casting defects was observed.
- (2) M40/Al composite showed extents of brittle Al_4C_3 at the fiber/matrix interfaces, sometimes bridging fibers in the regions where they are close to each other.
- (3) The fracture surface of composites after tensile and bending tests was flat with no fiber pull-out, which revealed characteristic of brittle fracture.
- (4) High-purity aluminum is not suitable as matrix to fabricate $Gr_f/A1$ composite under this processing method.

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