Mechanical Property of M40J_f/5A06Al Composite at Elevated Temperatures

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Abstract In this work, aluminum alloy with a high concentration of magnesium (5A06) was reinforced with 55 vol% unidirectional ultra-high modulus and highly graphitized carbon fiber (M40J) using pressure infiltration method. The effect of temperature on the bending strength of the C_f/Al composites was investigated from room temperature to 500 °C. The experimental results showed that the strength of M40J_f/5A06Al composites was not affected by temperature from room temperature to 200 °C. The bending strength of the composite at 300 °C was decreased by 30% compared with that at room temperature. In order to evaluate the extent of interface weakening, the length of fiber pullout was measured. The results showed that the pullout length reached the maximum at 300 and 500 °C, which indicated weak interface at the corresponding temperature. The DSC curve presented obvious heat absorption peak at around 300 °C, which may be attributed to the dissolution of the interfacial product β (Al₃Mg₂) phases at the C/Al interface. The bending fracture surfaces of the composites after three-point bending tests were observed by SEM, plastic-viscous flow of the matrix were observed at the samples tested at 500 °C. The predominant mechanisms for high-temperature damage of M40J_f/5A06Al composites are matrix softening caused by dislocation recovery and interface weakening caused by the dissolution of interfacial products.

KEY WORDS: Carbon fiber; Aluminum; Metal matrix composites; High-temperature properties; Fiber pullout length

1 Introduction

Because of low density, high strength and stiffness and low coefficient of thermal expansion [1–3], carbon fiber-reinforced aluminum composites (C_f/Al) have been considered

as leading candidates for applications in high-precision space vehicles, where the performance in a wide temperature range is a major design criterion [4]. Early investigations on C_f/Al composites primarily dealt with the interfacial reaction control of these materials. The reaction between C fiber and Al matrix leads to the formation of Al₄C₃ [5–15], which is detrimental to the mechanical and corrosion properties of C_f/Al composites [12–14]. However, there is a paucity of data for the effect of elevated temperature on the properties of C_f/Al composites.

In general, the strength of Al matrix composites or Mg matrix composites decreased monotonously with the increase in temperature [16–23]. Akbulut and Durman [16] found that the strength of short Al₂O₃ fiber-reinforced 7075Al composites was decreased with the increase in temperature, and they concluded that the high temperature performance of short fiber-reinforced composites was

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mainly affected by the properties of matrix alloy at elevated temperature. Lee *et al.* [17] found that the tensile strength, flow stress and work-hardening rates of short C fiber-reinforced 7075Al composites were decreased dramatically with the increase in temperature. Moreover, the descending slope of the strength above 200 °C is greater than that below 200 °C. Furthermore, Kagawa and Nakata [18] suggested that the decreased bending strength of C_f/Mg composites might be due to the insufficient stress transfer between fiber and matrix, originated from the reduction in shear yield stress of the matrix at high temperatures.

Elevated temperature performance of C_f/Al composites has been addressed by Fujita *et al.* [4], it was found that the tensile strength of P100_f/6061Al composites was decreased with temperature, and they found that the residual stress played a significant role on mechanical behavior at high temperature. In our present work, however, the strength of M40J_f/5A06Al composites was slightly affected by temperature up to 200 °C. It is clear that the mechanical behavior of C_f/Al composites varied in different Al matrix and the mechanism has not been fully understood yet. Moreover, the 5A06Al used as the matrix alloy in this paper is a type of Al–Mg alloy with high concentration of Mg. Magnesium can suppress the formation of brittle Al₄C₃ phase, which results in an increase in the mechanical properties of C_f/Al composites at ambient temperature [14]. However, elevated temperature performance of this C/Al system has never been reported.

Accordingly, the aim of this paper is to investigate the effect of temperature (from 20 to 500 °C) on bending strength of M40J_f/5A06Al composites. Moreover, the C fiber/Al interfacial structure was characterized, and the effect of temperature on the microstructure, mechanical properties and failure behavior was discussed.

2 Experimental Procedure

The fiber used in this experiment was M40J graphite fibers (Toray Industries, Inc., Japan). 5A06 aluminum alloy was chosen as the matrix material, whose nominal composition is shown in Table 1. C_f/Al composites were fabricated by pressure infiltration method. The infiltration temperature and the mold temperature were 750 and 500 °C, respectively. During the infiltration process, a pressure of 5 MPa was applied and maintained for

10 min, and then the composites were solidified in air. The fiber volume fraction of the obtained composites was approximately 55%.

The C_f/Al composites were firstly annealed at 330 °C for 30 min in vacuum. The annealed C_f/Al composites were then cut into 60 mm × 10 mm × 2 mm bars along the 0° direction for the measurement of mechanical properties. Ambient temperature and elevated temperature flexure properties were measured using an Instron 5500R (1186) universal electronic tensile testing machine with a gauge length of 40 mm. The tests were performed at temperatures ranging from 20 to 500 °C, and the samples were allowed to soak at the test temperature for 10 min before the crosshead was actuated at 0.5 mm/min to apply a press load. The furnace temperature was controlled at an accuracy of ±5 °C for elevated temperature tests. In order to improve statistical significance of the results, at least five samples were tested for each result.

Detailed examination of fracture surfaces was carried out using a Helios Nanolab 600i field emission scanning electron microscope (SEM) equipment. The transmission electron microscopy (TEM) investigation of the composites was performed by a Tecnai G2 F30 field emission electron microscope.

3 Results and Discussion

3.1 Morphology

Microstructure anisotropy was observed in the C_f/Al materials in directions parallel to and perpendicular to the fiber. Figure 1 shows a typical metallographic observation of C_f/Al composites in different directions. The C_f/Al composites used in this paper were well infiltrated with Al alloy, had very uniform fiber dispersion and did not have apparent porosity or significant casting defects. This is beneficial to the mechanical properties of C_f/Al composites. Moreover, the fibers are separated by a certain amount of plastic aluminum matrix. This can effectively alleviate the stress concentration at the interface and then prevent the composites from rapid crack propagation during application. The measured density of the C_f/Al composites was 2.23 g/cm³, which was very close to the theoretical density (2.22 g/cm³) of 55 vol% C_f/Al composites.

3.2 Interface

In this work, interfacial structure of M40J_f/5A06Al composites was observed by TEM, as shown in Fig. 2. Rare Al₄C₃ interfacial reaction products were observed at the

Table 1 Chemical composition of 5A06Al alloy (wt%)

Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
0.4	0.4	0.1	0.5	6.5	0.2	0.02	Bal.

Fig. 1 Metallographic photographs of Cf/Al composites: **a** perpendicular to fiber; **b** parallel to fiber

Fig. 2 **a** Interface area between the carbon fiber and the aluminum matrix; **b** bright-field image of β - Al_3Mg_2 phase; **c** SADP of the Al_3Mg_2 phase marked by the circle in Fig. 2b; **d** EDS result of the Al_3Mg_2 phase marked by the circle in Fig. 2b

fiber/matrix interface of this composite system. A large amount of blocky-shaped phases, however, were observed to align uniformly along the fiber/matrix interface, as shown in Fig. 2a, b. The selected area diffraction pattern (SADP) from the area marked by the circle in Fig. 2b and the energy dispersive spectroscopy (EDS) results indicated that the blocky-shaped phases are Al_3Mg_2 phases with a hcp structure, as shown in Fig. 2c, d, respectively.

According to Refs. [14, 15], the formation of the brittle phase Al_4C_3 at the C–Al interface in the $\text{C}_f/\text{Al–Mg}$ composite is inhibited significantly with the increase in Mg content. Increased Mg content in the Al matrix would increase the Gibbs free energy of Al_4C_3 , but decrease that of the Al_3Mg_2 phase. When the Mg content reached a critical value, above which the formation of Al_3Mg_2 would be easier than Al_4C_3 , no Al_4C_3 but blocky β phase (Al_3Mg_2) was observed at the C–Al interface in the $\text{C}_f/\text{Al–Mg}$ composite.

3.3 Bending Properties at Room Temperature

The interface reaction plays a crucial role in the properties of metal matrix composites (MMCs). A large number of

studies show that the presence of Al_4C_3 would adversely affect the mechanical properties of C_f/Al composites [12–14]. According to Ref. [14], with the increase in Mg content, the formation of brittle Al_4C_3 phase is suppressed, which results in an intermediate interface bonding, and therefore improves the mechanical properties of composites. 5A06Al with about 6.5 wt% Mg used in the present work is a type of Al–Mg alloy. Therefore, the interfacial bonding strength and the mechanical properties of M40J_f/5A06Al composites at room temperature should be improved.

Figure 3 shows a typical curve for flexural stress versus displacement of the C_f/Al composites tested at room temperature. It was found that the stress-displacement curve rose up to the maximum value (~ 1000 MPa) linearly and then fell abruptly. It indicated that the composites fractured in a typical brittle mode. A few stress peaks were observed in the descent zones, which corresponded to crack deflection phenomenon. Crack deflection along the interface is an important mechanism which often exists in the composites whose interface bonding is not strong, and would increase the resistance of cracks expanding. In the composite of high interface reactivity of $\text{C}_f/\text{Al}_{\text{pure}}$ (strong interfacial

Fig. 3 Stress-displacement curve of the three-point bending test for Cf/Al composites at room temperature

bonding) [14], the fracture surface is flat and no fiber is pulled out, and the bending strength of the composite is only about 400 MPa. However, in the composite of low interface reactivity of M40J_f/5A06Al (moderate or weak interfacial bonding), the occurrence of crack deflection mechanism promotes the strength of the composites.

When the crack deflection occurs, fibers may be pulled out from matrix before composites fracture. This kind of fiber pullout causes that the fracture surfaces of fibers are not in the same plane, as shown in Fig. 4. Holes were left by the pulled out fibers, as indicated by the arrows. Plenty of tear ridges can be observed at the right side of Fig. 4, which indicates that the fracture mode of Al matrix is ductile fracture.

Fig. 4 Fracture surface of Cf/Al composites after three-point bending test at room temperature

3.4 Effects of Temperature on Bending Properties

Figure 5 shows the stress-displacement curves of annealed M40J_f/5A06Al composites from room temperature to 500 °C. Because electrical resistance strain gages cannot be used at high temperatures, the relationship between stress and displacement instead of stress and strain was described. No change happened to the stress-displacement curves from 20 to 200 °C. At 300 °C, however, more obvious stress peaks were observed in the stress descending area. It implies that the elevated temperature facilitates the crack deflection process during bending tests. At 400 and 500 °C, the composites exhibited obvious matrix softening, which was accompanied by the increase in ductility.

Fracture displacement, which is an index to represent bending properties of brittle and low-plasticity materials, can also evaluate the extent of material softening at elevated temperature. For C_f/Al composites, however, the fracture displacement is very difficult to be confirmed at elevated temperatures. In particular, above 500 °C, C_f/Al composites even cannot be fractured. At this moment, the fracture displacement cannot be offered. In this work, therefore, in order to describe the trend of composite softening with the increase in temperature, $f_{0.5\sigma_{\max}}$, which was defined as the displacement value when the stress value fell to half the fracture strength, was used to evaluate the extent of composite softening instead of the fracture displacement, as shown in the schematic diagram at the upper right corner of Fig. 5.

The flexural strength and $f_{0.5\sigma_{\max}}$ value versus temperature for the C_f/Al composites are shown in Fig. 6. At ambient temperature, the bending strength of the composites was 1020 MPa and the $f_{0.5\sigma_{\max}}$ value of the composites was 1 mm. The composites showed a good retention of bending strength and $f_{0.5\sigma_{\max}}$ value up to 200 °C. Above 200 °C, however, the strength value decreased and the $f_{0.5\sigma_{\max}}$ value increased with temperature. This process was further divided into two stages: (1) From 300 to 400 °C, the

Fig. 5 Effect of temperature on the stress-displacement curve of the three-point bending test for Cf/Al composites

Fig. 6 Effect of temperature on the bending strength and the $f_{0.5\sigma_{max}}$ values of Cf/Al composites

strength value decreased rapidly, and the $f_{0.5\sigma_{max}}$ value increased slightly; (2) from 400 to 500 °C, the descending speed of the strength value slowed down, but the rising speed of the $f_{0.5\sigma_{max}}$ value was significantly increased. At the temperature of 500 °C, the strength value fell to 220 MPa, and the $f_{0.5\sigma_{max}}$ value rose to 11.4 mm. It is apparent that the strength of Cf/Al composites was sensitive to the working temperature. Exceeding a certain temperature, the composites become softer and the mechanical properties were rapidly degenerated. This phenomenon was also found in other metal matrix composites [16–23].

3.5 Fractographic Observation and Fracture Analysis

From the above analysis, the length and amount of fiber pullout have a direct relationship with the process of crack deflection as well as the interfacial bonding strength of the composite. If the interfacial bonding strength is high, the cracks directly cut through the fiber with the increase in load, and that can cause a low-stress fracture. At this moment, the fracture surface is flat and there is no fiber pullout. If the interfacial bonding strength is moderate, the cracks can expand along the fiber direction with the increase in load. At this moment, fracture energy can be consumed on the debonding process of fiber/matrix interface, and fiber pullout is visible on the fracture surface. Therefore, the strength of the composites is excellent. If the interfacial bonding strength is weak, the interface will be debonded under a low stress level. It is difficult for the interface to effectively transfer the load, and the fiber cannot get sufficient play. Therefore, the bending strength of the composites with a weak interface is not high.

Coarse observation of fractured samples tested at different temperatures showed that fibers protrude from the fracture surface at the carbon fiber/Al matrix interface in

these composites, as shown in Fig. 7a. Statistical analysis has been conducted to the longest fiber pullout length of fractured composites. The results showed that with the temperature rising up to 300 °C, the maximum value of fiber pullout length increased obviously. This might be attributed to the dissolution of β (Al_3Mg_2) phases at the interface between carbon fiber and Al alloy. As seen from the typical DSC curve in Fig. 7b, the position of peak A on DSC scans is at about 300 °C. According to the Al–Mg phase diagram in Fig. 8, in 5A06Al alloy containing 6.5 wt% Mg, dissolution of β - Al_3Mg_2 phase into Al solid-solution occurs at about 290 °C that is consistent with the DSC results.

There are a number of defects and microgrooves at the M40J fiber surfaces. These high-energy positions are very beneficial to the formation of precipitation, as shown in Fig. 2a. Therefore, the dissolution of Al_3Mg_2 phases causes that the interfacial bonding strength decreases, and relative slide between fiber and matrix is easy to happen. Moreover, the dissolution of interfacial products can also induce the hinder of cracks expanded along the interface to decrease. Therefore, the pullout length of the fibers increases with the temperature rising up.

Fig. 7 a Effect of temperature on the fiber pullout of Cf/Al composites; b DSC curve for Cf/Al composites

Fig. 8 Phase diagram of binary Al–Mg system (dashed line depicts Mg content in 5A06Al alloy)

When the test temperature arrived at 500 °C, delamination was observed in the samples, which proves again the fact that temperature impacted interfacial bonding strength and interlaminar shear strength of the composites extremely. With the increase in temperature, interfacial bonding strength and interlaminar shear strength of the composites decreased.

Fracture surface of C_f/Al composites tested at ambient and elevated temperatures is shown in Fig. 9. It is obvious that from room temperature to 300 °C, little difference in fracture morphology is observed (Fig. 9a–d). More obvious tear ridges have been observed in the fracture morphology of the composite tested at 400 °C, as shown in Fig. 9e. It indicated that the plasticity of Al matrix increases, but the fracture mode—ductile fracture has not change. Furthermore, the evidence of plastic-viscous flow was obviously observed at the fracture surface of the composite samples tested at 500 °C, as shown in Fig. 9f, which indicated that the extent of matrix softening was further aggravated. Moreover, plenty of debris adheres at the fiber surface, as shown in Fig. 10a. These debris formed shaped patterns at the fiber surface. In order to confirm composition of the debris, EDS was used. The results showed that the debris contains a large number of Al element and a little Mg element, as shown in Fig. 10b. Combining with the TEM results in Fig. 2, these debris maybe attributed to that the dissolved interfacial products Al₃Mg₂ adhered to the fiber surface. This conclusion, however, still needs to be further determined.

Mechanical property of composites is mainly affected by the performance of reinforcement, matrix and interface. In the temperature range of this study (20–500 °C), the properties of carbon fiber changed little. However, a

Fig. 9 Fracture surface of C_f/Al composites after test at different temperatures: **a** room temperature; **b** 100 °C; **c** 200 °C; **d** 300 °C; **e** 400 °C; **f** 500 °C

Fig. 10 **a** Fiber surface of the composites tested at 400 °C; **b** EDS result of the debris

temperature change in this range will have significant effect on the interfacial state and the matrix properties, and this effect can be further separated into two stages with the increase in temperature: (1) When the temperature rose to 300 °C, the β (Al_3Mg_2) phases at the C/Al interface began to dissolve, and that caused the decrease in interfacial bonding strength; (2) when the temperature rose to 400 °C, the extent of matrix softening was aggravated, and at the temperature of 500 °C, the plastic-viscous flow of matrix would happen under load.

Therefore, three reasons were considered to explain the phenomenon that the strength of the composites decreased monotonically after 300 °C in this work: (1) When the temperature rose to 300 °C, the interfacial bonding strength between the fiber and matrix also becomes weak due to the dissolution of the interfacial products; (2) with the increase in test temperature, the alloy matrix will soften, work hardening in matrix and piling up of dislocations become weak, and dynamic recovery and dynamic recrystallization of matrix material are easier to happen; (3) ability of the matrix to transfer load to the fiber decreases, and the strengthening effect of fiber was gradually weakened as the test temperature increases.

4 Summary and Conclusions

The effect of temperature on the mechanical properties of M40J carbon fiber-reinforced 5A06 aluminum alloy composite was investigated. The following conclusions can be arrived in this investigation:

1. The strength of M40J/5A06Al composites was not affected by temperature up to 200 °C. However, as the temperature was raised up to 300 °C, bending strength of the composites was decreased by 30% and then decreased monotonically with the increase in temperature.

2. The $f_{0.5\sigma_{\max}}$ value was introduced to evaluate the extent of composite softening at elevated temperatures for low-plasticity material. The $f_{0.5\sigma_{\max}}$ value of M40J_F/5A06Al composites was slightly affected by temperature up to 200 °C. However, the $f_{0.5\sigma_{\max}}$ value monotonically increased after 200 °C, and the ascending slope of the $f_{0.5\sigma_{\max}}$ value after 400 °C is greater than that before 400 °C.
3. Bending fracture of composites after three-point bending test at high temperature observed by SEM showed that: (1) As the test temperature rose up, the pullout length of fibers increased; (2) at the temperature of 500 °C, the plastic-viscous flow of matrix alloy was observed.
4. Differential scanning calorimetry results and Al–Mg alloy phase diagram analysis indicated that the interface weakening of C_F/Al composites at 300 °C might be attributed to the dissolution of the interfacial product β (Al_3Mg_2) phases.
5. The predominant mechanisms for high-temperature damage of M40J_F/5A06Al composites are matrix softening caused by facility of dislocation movement as well as dynamic recrystallization, and interface weakening caused by the dissolution of interfacial products.

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