Aging behaviors of alumina borate whisker reinforced AC8A-AI composite

J. HU, W. D. FEI, C. K. YAO

School of Materials Science and Engineering, P.O. Box 433 Harbin Institute of Technology, Harbin 150001, People's Republic of China E-mail: mzhu@hope.hit.edu.cn

The aging behavior of alumina borate whisker ($AI_{18}B_4O_{33w}/AC8A$ -AI) composite was examined by hardness and differential scanning calorimetry heat flow measurement. Microstructures were investigated by transmission electronic microscope. The results indicated that the interfacial reaction played an important role in the aging precipitation behavior of the composite. As the interfacial reaction between the whiskers and AC8A-AI alloy consumed the Mg atom of the matrix in the composite, the time to the peak aging of the composite was almost same as that of the AC8A-AI alloy. The free precipitation zone can be found nearby some whisker in the composite, duo to Mg content decreased in the matrix. © 2001 Kluwer Academic Publishers

1. Introduction

Aging treatments which can further increase the strength of aluminum matrix composites have been adopted extensively. The aging kinetics and micromechanisms of the composites are attracting attention [1–3]. In silicon carbide whisker (SiCw)/Al composites, studies [4, 5] have indicated that high densities of dislocation can be formed in matrix of the composites because of the difference in the thermal expansion coefficient between the whisker and the aluminum alloy. These dislocations increase the inhomogeneous sites for precipitation nucleation and accelerate the growth of the precipitation phase, so the aging kinetics of the composites are accelerated. These studies on the aging behavior are benefit in optimizing the aging treatment, and can provide important experimental and theoretical information for designing the properties of composites.

Recently, alumina borate whisker (Al₁₈B₄O_{33w}) reinforced aluminum composites have been attracting attention the lower price of the whiskers and excellent properties of the composites [6, 7] Previous studies on Al₁₈B₄O_{33w}/Al composites indicated that interfacial reactions took place between whiskers and some aluminum alloys [8]. In the present work, the effect of interfacial reaction on the aging behavior of Al₁₈B₄O_{33w}/AC8A-Al composite is studied by means of hardness measurement and differential scanning calorimetry (DSC). The tensile properties and fracture mechanisms of the aged composites were also investagated by scanning electron microscope (SEM) in-situ observation of the fracture process.

2. Experiment

The $Al_{18}B_4O_{33w}/AC8A$ -Al composite was fabricated by squeeze casting. The volume fraction of

whisker in the composite was about 25%. The properties of $Al_{18}B_4O_{33w}$ and the composition of the AC8A-Al alloy are listed in Tables I and II, respectively.

The specimens used for Vickers hardness measurements, were $10 \times 10 \times 2$ mm in size, and for DSC heat flow measurements were $4 \times 4 \times 1.5$ mm in size. All specimens were cut from the as-cast ingot. Solution treatment was carried out at 500°C for 5 h. After quenching in water at 80°C, the specimens for DSC were stored in dry ice to prevent natured aging, and the specimens used for hardness measurements were aged at 185°C and 205°C for different times. A perkin-eimer DSC thermal analyzer was employed to measure the heat flow. All DSC specimens were tested from room temperature, to 500°C with a constant scanning rate of 10° C/min.

The microstructures both of monolithic AC8A-Al alloy and the composite were examined using a Philips CM-12 transmission electron microscope (TEM) with energy dispersive X-ray spectrometer (EDS). The specimens for TEM study were thinned by ion milling. The fracture mechanism of the aged composite was investigated by SEM observation. The fracture strengths of the composites at different aged states were measured also.

3. Results and discussion

3.1. The microstructure of as-quenched composites

Fig. 1 shows the microstructures of the as-quenched composite. The high density of dislocations in the matrix of the composite can be seen (Fig. la), which is introduced during quenching as a result of the difference in thermal expansion coefficient between whiskers and

TABLE I Properties of $Al_{18}B_4O_{33}$ whisker

Length (µm)	Diameter (µm)	Thermal expansion coefficient $\gamma (\times 10^{-6}/\text{K})$	Tensile strength (GPa)
10–30	0.1–1	1.9	800

TABLE II Composition of the AC8A-Al alloy (mass%)

Si	Cu	Mg	Ni	Al
12.0	0.8	1.0	0.9	Balance

matrix alloy [4]. It can also be seen that an interfacial reaction occurs between the whiskers and the AC8A-Al alloy (Fig. 1b). According to the selected area electron diffraction pattern (SADP) shown in Fig. 1c, it can be determined that the interfacial reaction product has a fcc structure and the lattice constant is 0.81 nm which is in agreement with it being MgAl₂O₄. The EDS analysis of the interfacial reaction product also indicated that the product is Mg-rich phase. Therefore, it can be concluded that the interfacial reaction product is MgAl₂O₄ with a spinel structure. This is consistent with the observations of Suganuma *et al.* [6]. The chemical reactions are as follows:

$$Al_{18}B_4O_{33} + 4Al \rightarrow 11\gamma - Al_2O_3 + 4B$$
 (1)

$$\gamma$$
-Al₂O₃ + Mg + [O] \rightarrow MgAl₂O₄ (2)

It is clears that the Mg in the matrix is consumed by the above interfacial reaction, and the Mg content will be less in the matrix of composite than it is in the monolithic AC8A-Al alloy.

3.2. Aging of the materials

The curves of Vicker's hardness vs aging time for monolithic AC8A-Al and Al₁₈B₄O_{33w}/AC8A composite aged at different temperatures are shown in Fig. 2. The hardness of the composite is higher than that of monolithic AC8A-Al alloy because of the reinforcing effect of the whiskers. The time to peak aging of the composite at 185°C is decreased to 4.5 h, compared to 5 h for the monolithic AC8A-Al alloy (Fig. 2a); and the time to peak aging of the composite at 205°C is almost same, as the AC8A-Al alloy (Fig. 2b). These results are different to those repored for a SiCw/Al composite [9]. For a SiCw/Al composite, the aging behavior was accelerated at various temperatures due to an increase in dislocation density. In fact, the thermal expansion coefficient of Al₁₈B₄O₃₃ whisker is lower than that of SiC whiskers [6]. If the effect of thermal mismatch is





[112]_{MgAl2O4}

Figure 1 TEM micographs of the as-quenched $Al_{18}B_4O_{33w}/AC8A$ -Al composite, (a) high density of dislocations in the matrix of the composite, (b) interfacial reaction between the whisker and the AC8A-Al alloy, and (c) selected area electron diffraction pattern.



Figure 2 Curves of Vicker's hardness vs aging time for AC8A-Al and the composite after aging at temperatures of (a) 185°C and (b) 205°C.

considered as the only factor to cause the difference in aging behavior between the composite and the matrix alloy, then the time to peak aging of $Al_{18}B_4O_{33w}/Al$ composite should be accelerated, compared with the matrix alloy. That the time to peak aging of the composite is the same as that of the AC8A-Al alloy at 205°C suggests that there is an other factor affecting the aging behavior of the composite.

3.3. Microstructure of the aged composite and monolithic AC8A-AI alloy

Fig. 3 shows the peak aged microstructures of the matrix alloy and composite at 205°C. It can be observed that the precipitation character in the composite is similar to that in the monolithic AC8A-Al alloy. From the electron diffraction pattern, it can be determined that the precipitate is the β phase. The amount of β phase in



Figure 3 TEM micrographs of the AC8A-Al alloy (a) and the composite (b) aged at 205°C.



Figure 4 Mg content distribution near the whisker in the as-quenched composite. (a) TEM micrograph and (b) Mg distribution.



Figure 5 DSC curves of the composite and AC8A-Al alloy.



Figure 6 Relationship between the tensile fracture strength of the composite and the aging time at 205° C.

the composite is less than that in the monolithic AC8A-Al alloy. This is obviously due to the interfacial reaction consumption of the Mg. From Fig. 3b, a precipitationfree zone (PFZ) "A" around whisker can be seen. To examine the reason for formation of the PFZ, the Mg distribution, as measured by EDS, is shown in Fig. 4. The minimum value in Fig. 4b indicates that the interfacial reaction in the composite could take place during solution treatment.

3.4. DSC analysis

Fig. 5 shows the DSC curves of the composite and the monolithic AC8A-Al alloy. Two broad peaks can be seen in the DSC curves of both composite and AC8A-Al alloy. The first peak corresponds to the formation of the β' phase (at 200°C for the composite, at 240°C for the AC8A alloy); the other peak corresponds to the β (Mg₂Si) phase (at 281°C for the composite, at 277°C for the AC8A-Al composite). From Fig. 5 it can be concluded that the aging precipitation products in the composite are same as this in the AC8A-Al alloy [10], but the formation temperature of β' phase in the composite is lower than that in the AC8A-Al alloy. Their was attributed to the high density of dislocations. However, the formation temperature of β phase is higher in the composite than that in the AC8A-Al alloy. The main reason may be concerned with the Mg content of the matrix in the composite. From Equation 2, the Mg content of the matrix in the composite is decreased, and is lower in the unreinforced alloy (as shown in Fig. 4). Since the Mg content of the β phase is higher, the β precipitation in the composite lags behind the AC8A-Al alloy, and it takes longer for the β phase to precipitate fully (showing a diffuse feature in the DSC curve). It is suggested that the effect of the decrease in Mg content is greater than that of the higher density of dislocations aged at 205°C, which led to the time to peak aging of the composite being the same as that of the AC8A-Al alloy.

3.5. The mechanism of tensile fracture

According to the above results, it can be concluded that the incorporation of whiskers changed not only the aging behavior of the composite but also the microstructure of the aged composite. To analyze the effect of the aging on the tensile in-situ observations of the composite were carried out. The relationship between the tensile fracture strength and the aging time of the composite is shown in Fig. 6. The tensile strength of the composite decreased sharply as the aging time increased. Fig. 7 shows that cracks can propagate through the matrix or the whiskers. In the over-aged composite, however, the crack propagation is mainly through the



Figure 7 SEM in-situ observation of the crack propagation (a) peak aged at 205°C, (b) over aged 205°C.

matrix. For the peak-aged composite, the strength of matrix is enough high, that whiskers may fracture during the tensile test. For the over-aged composite, the strength of matrix is lower and the crack propagation is mainly in the matrix. In this case, the tensile strength of peak-aged composite is higher than that of the overaged composite.

4. Conclusion

- 1. In the as-quenched Al₁₈B₄O_{33w}/AC8A-Al composite, there is a high-density of dislocations in the matrix and an interfacial reaction between the whisker and the AC8A-Al alloy has occurred.
- 2. The aging kinetics of the composite were accelerated due to the dislocations in the matrix alloy when the composite was aged at 185°C. The time to the peak aging of the composite was almost the same as the AC8A-Al alloy, due to the interfacial reaction, when the composite was aged at 205°C.
- 3. The interfacial reaction changes the microstructure of the aged composite and a PFZ is formed around some whiskers, which results in differences in the fracture paths in the composite aged at different conditions.

Acknowledgement

We would like to thank the National Nature Science Foundation of China for the support of this research.

References

- 1. R. J. ARSENAULT, Mater. Sci. Eng. 64 (1984) 171.
- 2. H. J. PACK, in Proc. Sixth Int. Conf. on Composiet Mater. London, 1987, p. 128.
- 3. I. KOVACES, J. LENDVAI and E. NAGY, *Acta Metall.* **20** (1972) 975.
- 4. A. BADIN, F. MARINA and A. TOMAST, *Mater. Chem. Phys.* **25** (1990) 57.
- 5. L. J. YAO, Master thesis of Harbin Institute of Technology, Harbin institute of Technology, 1991.
- 6. K. SUGANUMA, T. FUJITA, N. SUZUKI and K. NIIHARA, J. Mater. Sci. Lett. 9 (1990) 633.
- 7. J. HU, W. D. FEI, C. LI and C. K. YAO, *Mater. Sci. Tech.* (in Chinese) **1** (1993) 1.
- 8. Idem., J. Mater. Sci. Lett. 13 (1994) 1794.
- 9. K. SUGANUMA, G. SASAKI, T. FUJITA and N. SUZUKI, J. Jpn. Inst. Light Metals (in Japanese) 41 (1990) 297.
- 10. G. J. MAHON and J. M. HOWE, *Acta Metall. Mater.* **38** (1990) 1503.

Received 4 February 2000 and accepted 26 April 2001