General paper

INTERFACIAL MICROSTRUCTURE AND FRACTURE CHARACTERIZATION OF $\text{Al}_{18}\text{B}_4\text{O}_{33}/\text{Al}$ ALLOY COMPOSITES

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Abstract: In order to investigate the relationship between the mechanical properties and the microstructure of aluminum borate ($\text{Al}_{18}\text{B}_4\text{O}_{33}$) whisker reinforced Al-1% Mg alloy composites fabricated by the squeeze casting process, the microstructure and fractured structure were observed by scanning and transmission electron microscopy (SEM and TEM). The interface between the matrix and whisker in an as-cast composite is smooth. By heat treatment at 550°C for 2h, the MgAl_2O_4 reaction layer with a spinel structure is produced at the whisker/matrix interface and appears trapezoidal. The interface between $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and MgAl_2O_4 has a certain preferred orientation with very little mismatch. Cracks tend to propagate along the whisker/matrix and reaction product/matrix interfaces. The interface between the whisker and reaction product seems to have good bonding strength. There are many microcracks in the whiskers in fractured composites, which propagate along the (001) plane in the whisker. Many dislocations and strain fields were observed in the matrix around the reaction products. The bending strength decreases with increasing amount of reaction product. The degradation of the composite strength by heat treatment is caused by weakening of the strength of the interface between the reaction product and matrix or strain concentration at the convexity formed by the interfacial reaction.

Key words: Metal matrix composites, Fracture, Transmission electron microscopy, Mechanical properties, Interface structure

1. INTRODUCTION

$\text{Al}_{18}\text{B}_4\text{O}_{33}$ (aluminum borate) whisker reinforced aluminum composites are candidate lightweight materials because of their superior mechanical properties and high cost performance. The study of the processing and mechanical properties has progressed in recent years [1,2]. However the constant degradation of the mechanical properties of Mg containing Al alloy composites by heat treatment and age hardening has not been studied in detail [1-4]. Although the degradation of the mechanical properties is caused by the reaction between the whisker and matrix, the interaction among the mechanical properties, the microstructure and the effect of heat treatment are still unknown.

In this study, in order to investigate the relationship between the mechanical properties and the microstructure, the fractured structure of $\text{Al}_{18}\text{B}_4\text{O}_{33}/\text{Al}$ -1% Mg alloy composites prepared by a squeeze casting process was analyzed by scanning and transmission electron microscopy (SEM and TEM), respectively. Furthermore, the effect of heat treatment was analyzed.

2. EXPERIMENTAL PROCEDURE

$\text{Al}_{18}\text{B}_4\text{O}_{33}$ whisker (Type M12, Shikoku Chemicals) and Al-1% Mg alloy were employed as the reinforcement and matrix, respectively. Al-1% Mg alloy was fabricated by the mixing of molten pure Al and Al-9% Mg alloy in an electric furnace. The composites were prepared by the squeeze casting process at the preform preheating temperature of 700°C, the melt pouring temperature of 800°C and a pressure of 50 MPa. The volume fraction of whiskers in composites was about 25%. Some composites were heat treated at 550°C for 2h in air in order to investigate the effect of the reaction prod-

ucts. The microstructure and fractured structure were observed by SEM and TEM. SEM and TEMs used were HITACHI/S-800H, JEOL/JEM-4000EX and Tocon/EM-002B, respectively. The whiskers were extracted from the composites using 36% HCl water solution in order to observe the interfacial reaction between the whisker and the matrix by SEM and TEM. A special TEM sample was prepared in the following manner. A micro Vickers diamond pyramid was indented on the surface of the dimple-grained and then ion-thinned sample.

3. RESULTS AND DISCUSSION

3.1. Microstructure of Composites

Figures 1 (a) and (b) show the extracted whiskers from the as-cast and the heat-treated composites at 550°C for 2h, respectively. The surface of the extracted whisker in the as-cast composite was clear and composed of smooth reaction products. For the heat-treated composite, however, the surface had some irregularities. These irregularities were produced by the interfacial reaction between the whisker and the matrix. Figure 2 exhibits the TEM image of the extracted whisker from the heat treated composite and energy-dispersive X-ray spectroscopy (EDS) semi qualitative calculation result at the reaction products. The reaction product appeared a trapezoidal shape on the whisker surface in places. The EDS analysis revealed that the reaction product comprised 64.4 at% oxygen, 25.1 at% aluminum and 10.2 at% magnesium. Its composition is very similar to that of a MgAl_2O_4 compound. X-ray diffraction analysis reveals that the extracted whisker from the heat treated composites consists of $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and MgAl_2O_4 with a spinel structure. Consequently, the trapezoids on the whisker are determined to be the reaction product, MgAl_2O_4 . In the previous study [3], $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and Al-

Mg alloy react at a temperature higher than 517°C . Although liquid Al-Mg alloy is in contact with the whiskers during the squeeze casting process, the reaction products were negligible because of the short reaction time. The reaction products were produced by the heat treatment at 550°C for 2 h.

Figure 3 shows the atomic arrangement and the schematic illustration of the interface between $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and the reaction product, MgAl_2O_4 , in the composite heat treated at 550°C for 2h. Both the lattices of $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and MgAl_2O_4 bonded directly without any interlayer and relaxation. The interface is parallel to the (020) plane of $\text{Al}_{18}\text{B}_4\text{O}_{33}$, which is the same as the surface of the virgin whisker. The angle between the (020) plane of $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and the (220) plane of MgAl_2O_4 is 82°

Fig. 1. SEM images of the extracted whisker from as-cast composites (a) and heat-treated composites at 550°C for 2h (b).

Element	O	Mg	Al	Si
at %	64.4	10.2	25.1	0.2

Fig. 2. TEM image and EDS analysis of the extracted whisker from composites heat treated at 550°C for 2h.

degrees. The lattice distance of the (001) plane in $\text{Al}_{18}\text{B}_4\text{O}_{33}$ is 0.564nm and is almost twice as large as the lattice distance, 0.286nm of the (220) plane in MgAl_2O_4 . $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and MgAl_2O_4 connect with little mismatch. It seems that the reaction product ' MgAl_2O_4 ' grows epitaxially on the whisker during heat treatment.

3.2. Microfracture of Composites

The bending strengths of composites which were as-cast and heat treated at 550°C for 2h were 465 and 395 MPa, respectively. The bending strength decreased after heat treatment. In previous studies [4-6], the bending strength of $\text{Al}_{18}\text{B}_4\text{O}_{33}$ reinforced with other Al alloy composites containing Mg decreased by heat-treatment. The degradation of the bending strength seems to be associated with the interfacial reaction.

Figure 4 shows the fracture surface of the composites after the bending test. Figure 4 (a) is the fracture surface of the as-cast composite. Dimple patterns caused by the ductile fracture of the matrix were mainly observed and the whiskers were scarce. This implies that the fracture occurred in the matrix. Figure 4 (b) is the fracture surface of the composite heat treated at 550°C for 2h. The

Fig. 3. High-resolution TEM image (a) of the interface between $\text{Al}_{18}\text{B}_4\text{O}_{33}$ and the reaction product ' MgAl_2O_4 ' in the heat treated composite at 550°C for 2h and schematic illustration (b) showing lattice fitness between the array of (001) planes in the whisker and that of (220) planes of the reaction product at the interface

whisker surface was observed in parts, and the reaction products were attached onto the whisker. Although the interface between the reaction product and whisker is strong because of the coherent interface, the reaction product/matrix and the whisker/matrix interfaces are weak.

Figure 5 shows the microstructure of the ruptured composites after heat treatment at 550°C for 2h by TEM. The whiskers were aligned in the same direction. Some cracks propagated in the whisker along the whisker axis. Others propagated along the whisker/whisker and the whisker/matrix interfaces. However, no fractures were observed in the matrix.

Figure 6 shows high-resolution images of the crack tip in the whisker for the composite heat treated at 550°C for 2h. (a) is the low magnification image. The crack propagated along the (001) plane and stopped inside the whisker. This plane appears to be vulnerable to fracture. It is well known that the whisker axis is aligned along the c-axis [2]. In Fig. 5, the whiskers were fractured in the plane perpendicular to the c-axis. Cracks propagate in the whisker by the stress component perpendicular to the whisker axis. On the other hand, cracks propagate along the whisker/whisker and the whisker/matrix interfaces by the stress component parallel to the whisker axis.

Fig. 4. SEM images showing the composites fracture surface of (a) as-cast composites and (b) composites heat treated at 550°C for 2h.

Fig. 5. TEM image showing the ruptured composite after heat treatment at 550°C for 2h.

Figure 7 shows the interfacial structure between the whisker and matrix near the crack for the heat treated composites after fracture. Open circles represent the reaction products, which are trapezoidal in shape. While many dislocations were observed in the matrix around the reaction products, they could not be observed in the whisker and reaction products. These dislocations are induced by the deformation. It implies that the deformation in composites occurred only in the matrix.

Fig. 6. High-resolution TEM images showing the crack tip in the whisker for the composites. Fig. (b) is an enlargement of Fig. (a)

Fig. 7. TEM image of the interface structure and the dislocations in the fractured composite heat treated at 550°C for 2h.

In particular, the deformation around the reaction product was very large. It is assumed that there are strain fields around the reaction products. On the other hand, the convexity formed by the interfacial reaction tended to create a strain concentration field. Such interfacial reaction and the growth of the reaction products lead to the degradation of strength.

4. CONCLUSIONS

(1) The interface between the whisker and matrix in the as-cast composite was smooth. By heat treatment at 550°C for 2 h, the MgAl_2O_4 reaction layer nucleates and grows at the interface between the whisker and matrix with a spinel structure and forms a trapezoid on the whisker. The interface between the whisker and reaction product has a certain preferred orientation with very little mismatch.

(2) Cracks tend to propagate along the whisker/matrix and the reaction product/matrix interfaces. Cracks in the heat-treated composite propagate with many deflections. The interface between the whisker and reaction products appears to be strong. Many microcracks were observed in the whiskers in the fractured composites. Many dislocations and strain fields were observed in the matrix surrounding the reaction products. The bending strength

decreases with increasing amount of reaction products. The degradation of strength by heat treatment arises due to the weak interface between the reaction product and matrix or strain concentration at the convexity formed by the interfacial reaction.

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