J Mater Sci (2008) 43:6233–6237
DOI 10.1007/s10853-008-2914-5

Mechanical properties and thermal stability of $ZnAl_2O_4$-coated aluminum borate whiskers reinforced 2024Al composite

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Received: 29 April 2008 / Accepted: 28 July 2008 / Published online: 27 August 2008

Abstract $ZnAl_2O_4$-coated aluminum borate whiskers reinforced 2024Al composite was fabricated by squeeze casting. Interfacial microstructures and tensile properties of the composite were investigated. The results show that $ZnAl_2O_4$ coating of the whiskers can improve the wettability of the whiskers by molten aluminum during squeeze casting, resulting in the increase of tensile properties of the composite. During thermal exposure, $ZnAl_2O_4$ at the interface can effectively hinder harmful interfacial reactions, resulting in the improvement of thermal stability of the composite at high temperatures. Fracture mechanisms of the composite in as-cast and after thermal exposure were also investigated.

Introduction

Aluminum borate whiskers reinforced aluminum composites (ABOw/Al) possess not only quite excellent mechanical properties but also fairly low price, which will promote their widespread industry applications in the future [1–3].

However, in ABOw/Al composites, poor wettability and excessive interfacial reactions are regarded as the major obstacles to synthesize high-performance materials with full use of their potentials [4–7]. During squeeze casting, poor wettablity of ABOw by molten aluminum makes the fabrication difficult and results in incomplete infiltration. In this case, microvoids exist at the interface between the whiskers and matrix or among the whiskers. In addition, before the composite is made into a final component, it often undergoes a T4 or T6 heat-treatment or a secondary process at elevated temperatures. Under such conditions, ABOw can be heavily damaged due to serious interfacial reactions between ABOw and magnesium in Al–Mg alloys [4, 6–8], leading to the deterioration of microstructures and mechanical properties of the composite. Meanwhile, magnesium in the matrix is consumed, which also decreases the solid solution or aging strengthening effect.

All these limit the application of the whiskers to aluminum composites, although its cost is more promising than that of other whiskers, such as SiC, Si_3N_4, and TiC. So, it is necessary to improve the wettability of the whiskers by molten aluminum during squeeze casting and decrease excessive interfacial reactions of the composite at elevated temperatures.

Coating of the reinforcement is one of the most successful techniques adopted to enhance the wettability of the reinforcement by molten metal and prevent interfacial reactions [9–11]. Sol–gel method is an advanced technique to fabricate uniform surface coating of the reinforcement [12]. As a result, interfacial microstructures and mechanical properties of composites can be adjusted using this method. However, available research on coating of the whiskers is less and how to deal with the dispersion of the coated whiskers is crucial. Ding et al. [13] prepared Al_2O_3-coated ABOw by sol–gel method; however, a ball-milling treatment was adopted to disperse the coated whiskers, which resulted in a large amount of whisker fracture and seriously affected the mechanical properties of the composite. Therefore, it is necessary to adopt a better process for the dispersion of the coated whiskers.

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In our previous work [14], nano-$ZnAl_2O_4$ particles were uniformly coated on the surface of ABOw by a sol–gel coating process and ultrasonic dispersion technique. Continuous nano-$ZnAl_2O_4$ particles with large specific areas were coated on the surface of ABOw and the fracture of the coated whiskers can be seldom found, which will establish a basis for the excellent mechanical properties of the composite.

In the present study, the effect of $ZnAl_2O_4$ coating on the surface of ABOw on interfacial microstructures and mechanical properties of $ZnAl_2O_4$-coated ABOw reinforced 2024Al (ABOw/$ZnAl_2O_4$/2024Al) composite are investigated, respectively. Fracture mechanisms of the composite in as-cast and after thermal exposure are also discussed.

Experimental

ABOw with a diameter of 0.5–1 μm and a length of 10–30 μm was employed as the reinforcement and 2024Al alloy was selected as the matrix in the present study. The chemical composition of 2024Al alloy is 4.4 wt.% Cu, 1.5 wt.% Mg, 0.6 wt.% Mn, 0.5 wt.% Si, <0.5 wt.% Fe and bal.-Al. In order to obtain $ZnAl_2O_4$ coating on ABOw surface, ZnO was first coated on the whiskers surface by a sol–gel route, then the ZnO-coated whiskers were sintered at 1000 °C for 1 h. During sintering, the reaction between ABOw and ZnO coating took place, so $ZnAl_2O_4$ coating of ABOw was introduced. The initial mass ratio of ZnO to ABOw selected was 1:10. The detailed coating process and reaction mechanism can be found in our previous study [14].

ABOw/$ZnAl_2O_4$/2024Al composite was fabricated by squeeze casting with a melt-pouring temperature of 800 °C and mould-preheating temperature of 500 °C. The volume fraction of ABOw in the composite was about 20%. For comparison, uncoated ABOw reinforced 2024Al (ABOw/2024Al) composite was also prepared.

In order to evaluate the thermal stability of the composites, the two kinds of composites were heat-treated at 490 °C for different time and then cooled in water at room temperature. Then, the composites were preserved at room temperature for 1 day before test.

Tensile experiments were carried out on an Instron-1186 testing machine at the tensile rate of 0.5 mm/min at room temperature. The dimensions of tensile specimens can be found in our previous publication [14]. On considering the strength scattering frequently occurred in composites, five samples from each composite were tested to determine the tensile strength. Tensile fractographs of the composites were examined by an S-3000 scanning electron microscope (SEM).

The interfacial microstructures of the composites were observed using a Philips CM-12 transmission electron microscope (TEM). The specimens used for TEM observation were prepared by mechanical thinning, followed by a conventional argon ion-milling method.

Results

Microstructures of the composites

TEM micrographs of the interface in as-cast composites are shown in Fig. 1. It can be seen that few interfacial products with small sizes sparsely distribute at the interface in ABOw/2024Al composite. The interfacial products are $MgAl_2O_4$ according to the previous research [7]. This indicates that interfacial reactions between ABOw and

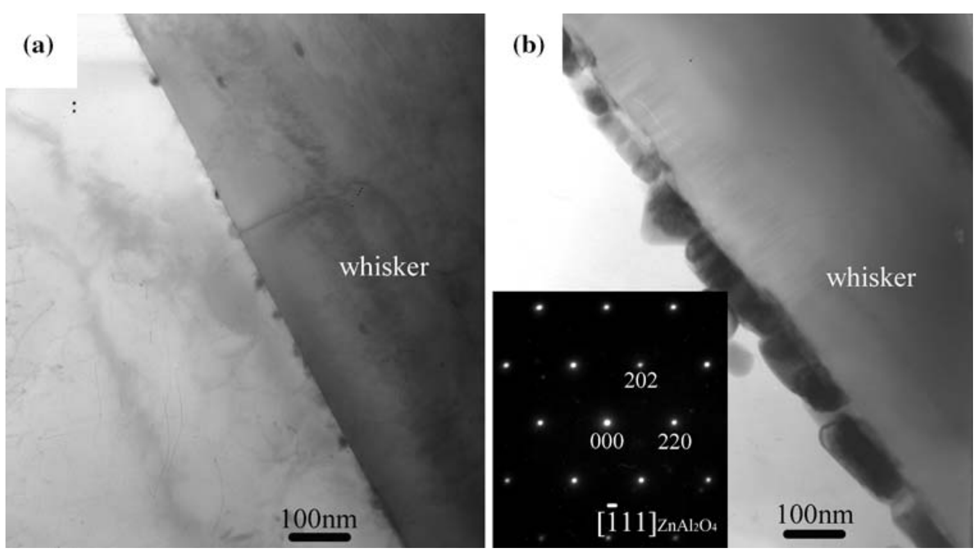

Fig. 1 TEM micrographs of the interface in as-cast composites: (**a**) ABOw/2024Al and (**b**) ABOw/$ZnAl_2O_4$/2024Al

magnesium in 2024Al matrix are slight in the composite during squeeze casting, which may be the shorter stay time at high temperature. However, in ABOw/$ZnAl_2O_4$/2024Al composite, the surface of ABOw at the interface is almost covered by continuous products. By indexing the selected area electron diffraction pattern (SADP), it can be known that the interphase is made up of $ZnAl_2O_4$. So, one can conclude that $ZnAl_2O_4$ on the surface of ABOw does not take part in interfacial reactions during squeeze casting.

Figure 2 shows tensile fractographs of as-cast composites. In the fractograph of ABOw/2024Al composite, many microvoids and microcracks can be found, as shown by arrows in Fig. 2a. They are the unfilled regions of molten aluminum caused by poor wettability of ABOw by molten aluminum during squeeze casting or the interfacial debonding between the whiskers and matrix during tensile deformation. However, in the fractograph of ABOw/$ZnAl_2O_4$/2024Al composite, microvoids and microcracks were rarely seen. In addition, some fractured particles can be observed in the paths of the pull-out whiskers, which can also result in higher ultimate tensile strength (UTS) and elongation to fracture (δ) by consuming grinding force of the whiskers.

TEM micrographs of the interface in the composites after thermal exposure at 490 °C for 10 h are shown in Fig. 3. In ABOw/2024Al composite, interfacial reactions are very serious due to the growth of $MgAl_2O_4$ and the whiskers integrity are also heavily damaged after a long time thermal exposure at 490 °C for 10 h. But in ABOw/$ZnAl_2O_4$/2024Al composite, no interfacial reactions take place. The interfacial phase is still made up of $ZnAl_2O_4$ according to the SADP indexing. After thermal exposure at 490 °C for 10 h, morphologies of the interface are similar to those of as-cast composite. This indicates that interfacial reactions between ABOw and magnesium are greatly diminished due to the existence of $ZnAl_2O_4$ at the interface.

Figure 4 shows the tensile fractographs of the composites after thermal exposure at 490 °C for 10 h. It can be found that in ABOw/2024Al composite, a large amount of whiskers fracture can be seen in the fractograph, as shown by arrows in Fig. 4a. The pull-out of whiskers and matrix

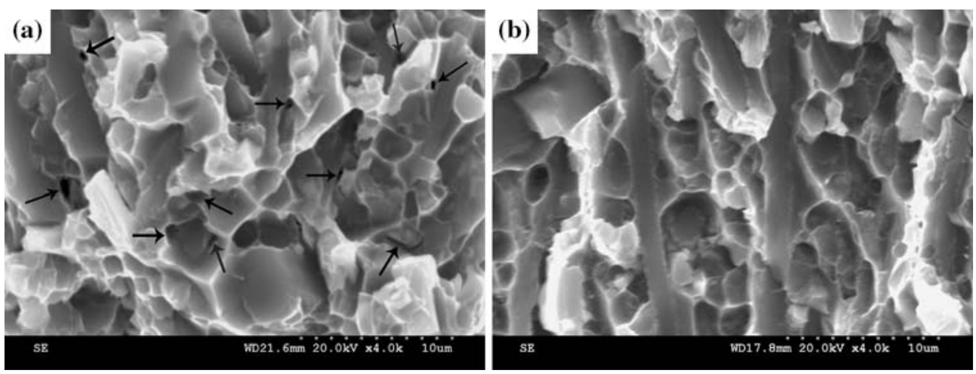

Fig. 2 Tensile fractographs of as-cast composites: (**a**) ABOw/2024Al and (**b**) ABOw/$ZnAl_2O_4$/2024Al

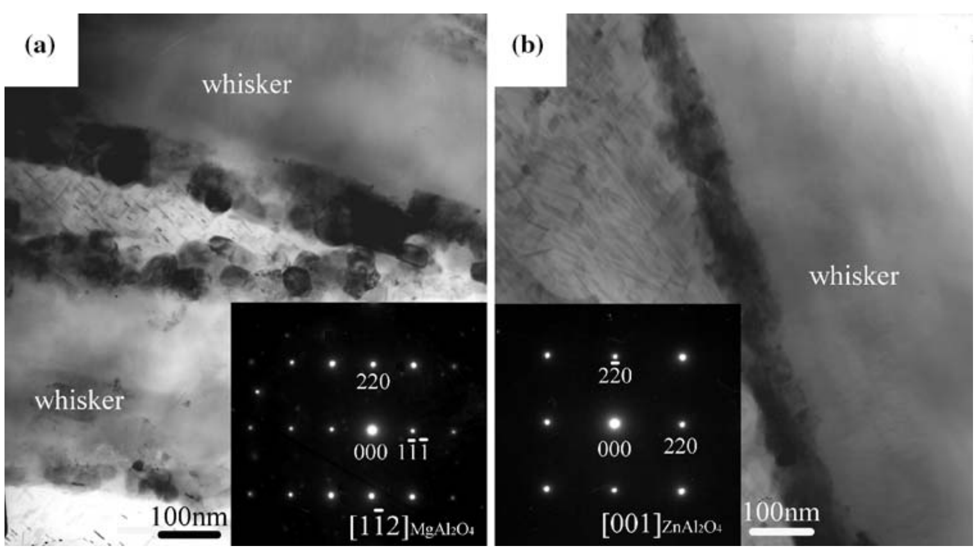

Fig. 3 TEM micrographs of the interface in the composites after thermal exposure at 490 °C for 10 h: (**a**) ABOw/2024Al and (**b**) ABOw/$ZnAl_2O_4$/2024Al

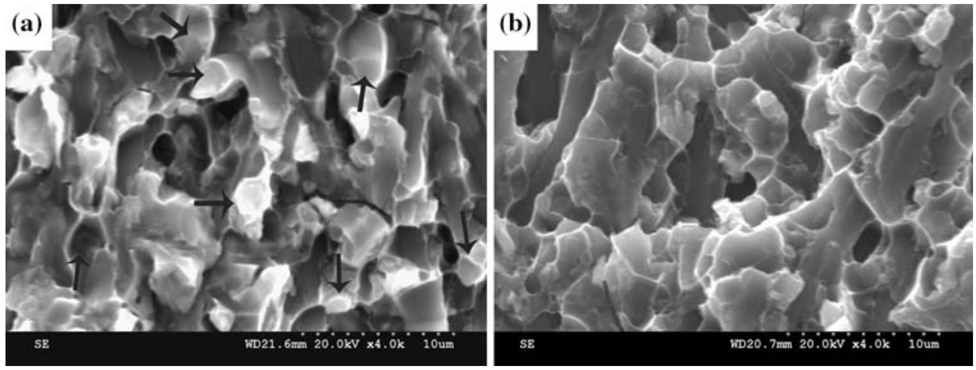

Fig. 4 Tensile fractographs of the composites after thermal exposure at 490 °C for 10 h: (**a**) ABOw/2024Al and (**b**) ABOw/$ZnAl_2O_4$/2024Al

dimples obviously decrease compared with those in as-cast ABOw/2024Al composite. But for ABOw/$ZnAl_2O_4$/2024Al composite, after thermal exposure at 490 °C for 10 h, fractograph morphologies of the composite are similar to those of its as-cast composite.

Tensile properties of the composites

Table 1 shows the tensile properties of the composites in as-cast and after thermal exposure at 490 °C for different time. It can be found that in as-cast ABOw/2024Al composite, the UTS and δ of the composite is separately 310 MPa and 0.9%. However, in as-cast ABOw/$ZnAl_2O_4$/2024Al composite, the UTS and δ of the composite increases to 335 MPa and 1.8%, respectively. This suggests that $ZnAl_2O_4$ coating on the surface of ABOw can obviously improve tensile properties of the composite. After thermal exposure at 490 °C for 1 h, the UTS of the composites rapidly increase compared with that of as-cast composites due to the solid-solution strengthening effect of 2024Al matrix. (After heat-treatment at 490 °C for 1 h, the alloy elements dissolved into 2024Al at high temperature form supersaturated solid solution and reserve in 2024Al after rapid cooling to room temperature, which enhances the UTS of 2024Al.) After thermal exposure at 490 °C for 10 h, tensile properties of ABOw/2024Al composite significantly decrease compared with those of the composite after thermal exposure at 490 °C for 1 h. However, for ABOw/$ZnAl_2O_4$/2024Al composite, variations of tensile properties can be negligible.

Discussion

It is well known that interfacial characteristics play a significant role in determining mechanical properties of metal matrix composites reinforced by the ceramic reinforcement [10, 15].

In as-cast ABOw/2024Al composite, there are many microvoids and microcracks caused by the poor wettability of ABOw by molten aluminum during squeeze casting. During tensile deformation, the interfacial debonding between the whiskers and matrix can result in low tensile properties of the composite. However, in as-cast ABOw/$ZnAl_2O_4$/2024Al composite, many matrix dimples and little microvoids can be found because the interfacial wettability can be improved by $ZnAl_2O_4$ particles with large specific area energy according to the wettability mechanism [16]. Therefore, tensile properties of the composite can be obviously improved. Meanwhile, continuous distributed $ZnAl_2O_4$ particles at the interface can serve as an effective barrier layer to hinder the reaction between ABOw and magnesium in 2024Al matrix, which establishes a basis to improve the thermal stability of the composite at high temperatures.

T4 heat-treatment temperature of 2024Al alloy is about 490 °C. After T4 heat-treatment, mechanical properties of 2024Al alloy can be significantly increased due to the solid solution strengthening effect, so mechanical properties of the corresponding whiskers reinforced 2024Al composites can be also improved. For ABOw/2024Al composite, after thermal exposure at 490 °C for 1 h, a part of magnesium in

Table 1 Tensile properties of the composites

Composites	UTS (MPa)			δ (%)		
	As-cast	490 °C, 1 h	490 °C, 10 h	As-cast	490 °C, 1 h	490 °C, 10 h
ABOw/2024Al	310 ± 11	371 ± 10	344 ± 13	0.9 ± 0.2	0.8 ± 0.2	0.5 ± 0.2
ABOw/$ZnAl_2O_4$/2024Al	335 ± 6	403 ± 5	400 ± 6	1.8 ± 0.1	1.8 ± 0.1	1.7 ± 0.1

2024Al matrix is consumed due to the reaction between ABOw and magnesium. However, in ABOw/$ZnAl_2O_4$/2024Al composite, $ZnAl_2O_4$ coating of the whiskers can effectively hinder the reaction, so magnesium in 2024Al matrix can be preserved. Therefore, tensile properties of ABOw/$ZnAl_2O_4$/2024Al composite are higher than those of ABOw/2024Al composite due to the solid solution strengthening effect of 2024Al alloy.

During a long time thermal exposure at 490 °C for 10 h, interfacial reactions between ABOw and magnesium can not only consume magnesium in 2024Al matrix but also seriously destroy the whiskers integrity due to the growth of interfacial products $MgAl_2O_4$ in ABOw/2024Al composite. Therefore, discontinuously brittle $MgAl_2O_4$ is easy to fracture during tensile deformation, resulting in the fracture of whiskers. So the load carrying capacity of whiskers is greatly diminished. As a result, tensile properties of ABOw/2024Al composite obviously decline after a long time thermal exposure compared with those of the composite after thermal exposure at 490 °C for 1 h. However, for ABOw/$ZnAl_2O_4$/2024Al composite, as shown by the interfacial microstructure of thermally exposed composite, $ZnAl_2O_4$ at the interface is stable at high temperatures. It can successfully separate ABOw from the matrix and control harmful interfacial reactions between ABOw and magnesium after subsequent thermal exposure. As a result, there is only a slight decrease of tensile properties after a long time thermal exposure at 490 °C for 10 h.

Conclusion

Based on the above analysis, the following conclusions can be derived. During squeeze casting, $ZnAl_2O_4$ coating of ABOw can improve the wettability of ABOw by molten aluminum, resulting in the increase of tensile properties of the composite. $ZnAl_2O_4$ at the interface can effectively hinder harmful interfacial reactions between ABOw and magnesium in 2024Al matrix. So, thermal stability of the composite at high temperature is improved. After thermal exposure at 490 °C for 10 h, tensile properties of ABOw/2024Al composite significantly decrease compared with those of the composite after thermal exposure at 490 °C for 1 h. However, for ABOw/$ZnAl_2O_4$/2024Al composite, the variations of tensile properties can be negligible.

Acknowledgement The work was supported by the Reward of the Outstanding Young Teacher's Teaching Scientific Research of Institution of Higher Education and National High Technology Research and Development Program of China.

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