

# EFFECT OF AGING BEHAVIOR ON MECHANICAL PROPERTIES OF AZ91D/ AL<sub>18</sub>B<sub>4</sub>O<sub>33</sub> WHISKER COMPOSITES FABRICATED BY SQUEEZE CASTING

Kouji Maruo\*, Wenguang Wang\*, Nobuyuki Fuyama\*\*, Kazuhiro Matsugi\*, and Gen Sasaki \*

\*Department of Mechanical Systems Engineering, Hiroshima University, \*\* Hiroshima Prefecture Synthesis Institute of Technology Western Industrial Center

> **Keywords**: metal matrix composite, aluminum-borate whisker, AZ91D magnesium alloy, aging, mechanical property, microstructure

### Abstract

 $Al_{18}B_4O_{33}$  whisker reinforced AZ91D magnesium alloy composite was fabricated by squeeze casting. Solution treated composites were aged at 443K. Composites and monolithic alloy had good agehardening behavior due to  $Mg_{17}Al_{12}$  precipitation. Time to reach the maximum hardness of the composites was shorter than that of monolithic alloy. Bending strength of composites was improved by aging treatment, but the obvious decrement of bending strength was observed at over aging. As increasing aging time, the fracture mechanism of the composite changed from inside the matrix to the interface between matrix and whisker. As increasing aging time, the strength of whisker/matrix interface decreased. And then, the strength of composites decreased. Elastic modulus of composites increased before reaching peak-age, but the elastic modulus did not change during over-aging treatment. It indicates that the elastic modulus of composite is a function of quantity of precipitate.

## **1** Introduction

Recently, it is urgent to exploit new structural materials having lightweight, high strength and good recycle ability because of energy and environmental problems. As magnesium is the lightest material in all-structural metals and has many attractive physical and mechanical properties combined with processing advantages, the demand of magnesium alloy is increasing. On the other hand, metal matrix composites are great interest for higher mechanical properties than that of monolithic alloys. In recent years, magnesium alloy (ZK60, AZ91D) matrix composites have received great attention and are

studied widely [1-2]. Discontinuous reinforcements such as short fiber, particle or whisker have been used as the reinforcements for magnesium alloy matrix. Especially, aluminum borate whisker  $(Al_{18}B_4O_{33w})$  receives great attention because of high cost-effective, high strength, high Young's modulus and low coefficient of thermal expansion [1-3]. AZ91D (Mg-9%Al-1%Zn) magnesium alloys and  $Al_{18}B_4O_{33w}$  were adopted as matrix and reinforcement in this study.

As it is well known that aging treatment is usually utilized to increase the mechanical property of magnesium alloys, such as AZ91 and ZK60 [1-2]. Therefore, it is expected that the optimum properties of composites can be improved with aging treatment. The recent years, there are many studies on age hardening of magnesium alloy matrix composites [2-3]. Unfortunately, up to now, there is little information available about the effects of aging treatment on the strength and elastic modulus. Therefore, the microstructures of AZ91D/Al<sub>18</sub>B<sub>4</sub>O<sub>33w</sub> composite were observed in this study. Furthermore, the effects of aging treatment on bending strength and elastic modulus in composites were discussed.

## **2** Experimental procedures

AZ91D magnesium alloy reinforced with  $Al_{18}B_4O_{33w}$  (M-20, Shikoku Chemicals Co.) was fabricated by squeeze casting. Whisker forms 0.5~1.0µm in diameter and 10~30µm in length and its surfaces are smooth in atomic scale. The volume fraction of the whisker in the composites is 30%.

Table 1 shows the fabrication conditions of AZ91D/  $Al_{18}B_4O_{33w}$  composite by squeeze casting. Composite was water-quenched after solution treatment (T4) at 693K for 86.4 ks in argon

atmosphere, and then aged at 443K in oil bath. Monolithic alloy was performed in similar processing for the sake of comparing.

The microstructures of the composites were observed with scanning electron microscopy (SEM) and transmission electron microscopy (TEM). X-ray diffraction analysis was carried out in order to identify the phase in the composite and alloy. In addition, hardness, bending strength and elastic modulus of alloy and composite were estimated.

Table 1 Squeeze casting condition for the composite preparation.

Applied pressure / MPa	100
Plunger speed / mm/s	2
Temperature of molten magnesium alloy / K	988
Preheating temperature of perform / K	1023
Preheating temperature of die / K	503

#### **3** Results and discussion

AZ91D alloy and AZ91D/Al<sub>18</sub>B<sub>4</sub>O<sub>33w</sub> composite are solution treated at 693K for 86.4ks in argon atmosphere, and following water-quenched. The results of X-ray diffraction analysis are shown in Fig.1. Precipitate Mg<sub>17</sub>Al<sub>12</sub> is not found in asquenched AZ91D alloy. It indicates that precipitate Mg<sub>17</sub>Al<sub>12</sub> is dissolved completely during solution treatment. Nevertheless, precipitate Mg<sub>17</sub>Al<sub>12</sub> is found in as-quenched composite. Solid solution treatment temperature raised 703K, which is precinct for melting point of AZ91D magnesium alloy. This treatment leads to disappearance of Mg<sub>17</sub>Al<sub>12</sub> in composites. This result shows that the coefficient of thermal conductivity of Al<sub>18</sub>B<sub>4</sub>O<sub>33</sub> whisker (5.6 W/m·K) made cooling rate depress.



Fig.1 XRD patterns of  $AZ91D/Al_{18}B_4O_{33w}$  composite and AZ91D monolithic alloy after solution (T4) treated.

Fig.2 shows age-hardening curves of the monolithic and composite aged at 443K. The time to reach the peak hardness of composite and monolithic alloy is 115.2ks and 180.0ks, respectively. Although precipitate Mg<sub>17</sub>Al<sub>12</sub> is found in as-quenched composite, the composite shows good age-hardening ability as well as the monolithic alloy. The hardness of the composites and alloy increased as a function of aging time before reaching the peak hardness and then gradually decreased. The time to reach the peak hardness of the composites is shorter than that of monolithic alloy. Fig.3 shows TEM image of dislocation nearby the whisker in the matrix of as-When AZ91D/Al<sub>18</sub>B<sub>4</sub>O<sub>33w</sub> quenched composite. composite is cooled from solution treatment temperature to room temperature, large thermal residual stress is applied due to the big difference of thermal expansion between matrix and reinforcement. Further, high-density dislocation is introduced by the large thermal residual stress. Matrix dislocations have been recognized as preferential nucleation sites for  $Mg_{17}Al_{12}$  precipitate [6]. In another words, matrix dislocation can facilitates the formation of  $Mg_{17}Al_{12}$ precipitate. In addition, the dislocation can enhance the diffusion rate of solution atoms. Further, the rate of growth of precipitates is accelerated.

According to the above analysis, the accelerated behaviors of age hardening can be explained by the following mechanism:

First, precipitates remained in as-quenched composite decreases the content of aluminum in AZ91D matrix. Further, the time to arrival peak hardness is shortened. Second, the high-density dislocation can enhance the nucleation and growth of  $Mg_{17}Al_{12}$  precipitate.



Fig.2 Age hardening curves of Al<sub>18</sub>B<sub>4</sub>O<sub>33</sub> whisker / AZ91D alloy composite and AZ91D monolithic alloy aged at 443K.



Fig.3 TEM image of dislocation nearby whisker in composite solution-treated at 693K for 86.4 ks.

Fig.4 shows TEM images of precipitates in (a) as-quenched (b) peak-aged and (c) over-aged composites.  $Mg_{17}Al_{12}$  precipitates are observed in all composites. Before the hardness of composite reaches the peak value, needle-like precipitates grew up. Its average width and length is 0.1µm in 1~2µm, respectively. As increasing aging time, sizes of precipitates do not change obviously, but the shape of precipitates became oval-like from needle-like due the coarsening of precipitate [6].

Fig.5 shows TEM images of precipitates in (a) peak-aged and (b) over-aged monolithic alloy. As well as the result of X-ray diffraction analysis, precipitate is not found in monolithic alloy that is solution treated at 693K for 86.4 ks. Precipitation mechanism is same with that of composite. But the sizes of precipitates are bigger than that of composites, corresponding [6].



Fig.4 TEM images of composites aged at 443K. (a) After solution treatment (b) Aging time 115.2ks (peek-aged) (c) Aging time 230.4ks (over-aged)



Fig.5 TEM images of AZ91D alloy aged at 443K. (a) Aging time 180ks (b) Aging time 360ks

Fig.6 shows bending stress and displacement curve of alloy aged for 180ks and composites aged at 115ks. The monoclinic alloy and the composite show ductile and brittle fractures, respectively. The fracture strength of the alloy was improved by combining with the fibers, but the elongation decreased dramatically. In metal matrix composites, as increasing the volume fraction of fiber, the distance between fibers shorten and then the stress of crack propagation becomes to be difficult to relax sufficiently. As the fracture become to be unstable, the composite has brittle fracture. On the other hand, the elongation of composite and alloy decreased as increasing an aging time.



Fig.6 Bending stress and displacement curve of alloy aged for 180ks and composites aged for 115ks.

Fig.7 shows bending strength and elastic modulus of as-quenched, peak-aged and over-aged composites. Bending strength is improved by aging treatment, but the obvious decrement of bending strength is observed at over aging. It seems the increment of strength is caused by the strengthening of  $Mg_{17}Al_{12}$  precipitations. As  $Mg_{17}Al_{12}$  precipitates form and grew up, the strength of composites

increased due to the extra stress required to force dislocations through  $Mg_{17}Al_{12}$  precipitates. It is also considered that big sized  $Mg_{17}Al_{12}$  precipitates lead to the high strength of the composite.



Fig.7 Effect of aging time on bending strength and elastic modulus of composites aged at 443K.

To consider the mechanism of a strength reduction, fracture surfaces for the composites was observed. Fig.8 shows the fracture surfaces of composites by bending test. As increasing aging time, the composite fractures from the matrix nearby whisker/matrix interface. It seems that precipitates at an interface became to coarsen by aging, so that crack occurred at enlarged precipitates. However, the enlargement of precipitates at interface was not observed by TEM observation, even as increasing aging time. On the other hand, as increasing aging time from peak age to over age, the precipitates at interface disappeared and monolithic alloy layer with 10 nm in thickness was observed as shown in fig. 9. It seems that bending stress concentrate in this layer, so that crack propagates along the neighborhood of whisker. Consequently, whisker seems to be pull out from matrix, composite has low strength.

As another cause of the strength reduction, the strength of whisker/matrix interface decreased as increasing aging time. Resultingly, the strength of composites decreased.



Fig.8 SEM images of fracture surface of the composite after bending, which is (a) aged for 0 ks, (b) aged for 115.2ks (c) aged for 230.4ks at 443K.



Fig.9 TEM images of precipitates nearby whisker in composite, which is (a) aged for 115.2ks (b) aged for 230.4ks at 443K.

Moreover, elastic modulus of composite aged for 115.2ks is higher than that of as-quenched composite. Even if the aging time increased to 230.4ks, elastic modulus is constant. In addition, the relative intensity of  $Mg_{17}Al_{12}$  precipitate measured by X-ray diffraction analysis as shown in Fig.10. It shows that the quantity of precipitate increases till reaching the peak-age and then it become top be constant. Taking into account of the curve of elastic modulus of composites, it is seemly that elastic modulus of composite changes as a function of the quantity of precipitate.

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Fig.10 Relationship between aging time and relative intensity of  $Mg_{17}Al_{18}(101)$  for Mg(101) of composite.

#### **4** Conclusions

The relationship between the aging behavior and the mechanical properties in  $Al_{18}B_4O_{33W}/AZ91D$  composites fabricated by squeeze casting were investigated. Followings are conclusions:

- In monolithic alloy, Mg<sub>17</sub>Al<sub>12</sub> was able to disappear by solid solution treatment, but solid solution treatment temperature raised, Mg<sub>17</sub>Al<sub>12</sub> was not able to disappear in composites.
- (2) The time to reach the peak hardness of composite is shorter than that of monolithic alloy.
- (3) Bending strength of composites and alloy were improved by aging treatment, but the bending strength of over-aged composite decreases greatly and is lower than that of as-quenched composite (T4).
- (4) The elastic modulus of composite increases at under-aging, but it does not change after the hardness reaches the peak value.

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