Aging Behaviors in AZ91D/ $\text{Al}_{18}\text{B}_4\text{O}_{33\text{w}}$ Composite Fabricated by Squeeze Casting

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Aluminum borate whisker ($\text{Al}_{18}\text{B}_4\text{O}_{33\text{w}}$, denoted by ABO_{w}) reinforced AZ91D (Mg-9.1 mass%Al-0.7 mass%Zn) magnesium alloy composite was fabricated by squeeze casting. AZ91D/ ABO_{w} composite and AZ91D alloy were aged at 443 K in oil-bath. The composite exhibited an accelerated age-hardening response comparing with AZ91D alloy. Age-hardening efficiency of composite was better than that of AZ91D alloy. It was attributed to the interfacial reaction between AZ91D alloy matrix and ABO_{w} . The interfacial reaction increased aluminum elements in the matrix and MgO interfacial reaction layer restrained the formation of interfacial precipitate, resulting in more amounts of continuous precipitates and smaller inter-precipitates spacing within the composite matrix. [doi:10.2320/matertrans.MER2007278]

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1. Introduction

Recently, magnesium alloy matrix composites reinforced with discontinuous reinforcements (short fiber, whisker or particle) have received great attention due to low coefficient of thermal expansion, high specific strength and stiffness, and wear resistance comparing with traditional alloys. Therefore, they have a great potential of application in aerospace, automobile, and transport industries.¹⁾

Many discontinuously reinforced metal matrix composites are based on age-hardenable light alloys, and aging treatment can develop the optimum properties of composites.²⁻⁸⁾ Mg-Al binary alloys and Mg-Al-Zn ternary alloys having good castability and mechanical strength are attractive.^{3,8-10)} Many studies⁹⁻¹⁷⁾ on the aging behaviors of Mg-Al-based alloy have shown that the precipitates form either continuously or discontinuously, and continuous precipitate is responsible for most of the age-hardening in Mg-Al-based alloys.

The study on the aging behaviors of AZ91/ SiC_{w} composite³⁾ has indicated that AZ91/ SiC_{w} composite exhibits an accelerated age-hardening response comparing with the AZ91 alloy. And the age-hardening efficiency of the AZ91/ SiC_{w} composite is lower than that of AZ91 alloy. $\text{Mg}_{17}\text{Al}_{12}$ phase precipitates preferentially at clean interface between SiC_{w} and matrix. These interfacial precipitates finally deplete the aluminum elements in the matrix, resulting in less amounts of continuous precipitates in the composite matrix. Badini *et al.*⁴⁾ have investigated the aging characteristics of AZ80/ $\text{B}_4\text{C}_{\text{p}}$ composite by optical microscopy and XRD. The addition of B_4C particles increases the aging rate of AZ80/ $\text{B}_4\text{C}_{\text{p}}$ composite. The nucleation of $\text{Mg}_{17}\text{Al}_{12}$ is greatly enhanced at the interfaces between ceramic and metal matrix in the composite. As a consequence, the hardness increment of composite is lower than that of AZ80 alloy, because of the different precipitate distributions in composite matrix.

In recent years, aluminum borate whisker ($\text{Al}_{18}\text{B}_4\text{O}_{33\text{w}}$, denoted by ABO_{w}) has received great attention as a discontinuous reinforcement for light metal due to some excellent features such as high Young's modulus, high strength, and low coefficient of thermal expansion.¹⁸⁻²⁰⁾

Moreover, magnesium can react to ABO_{w} and forms MgO interfacial reaction layer in AZ91D/ ABO_{w} composite during heat treatment at 693 K.²¹⁾ At the same time, aluminum elements deoxidized from ABO_{w} can dissolve into the matrix of AZ91D/ ABO_{w} composite. In order to optimize the aging treatment and provide the experimental and theoretical information for designing the properties of composites, it is significative to study aging behaviors of AZ91D/ ABO_{w} composite.

The aim of present study is to investigate the aging behaviors and clarify the effects of interfacial reaction on precipitations in AZ91D/ ABO_{w} composite.

2. Experimental Procedures

Commercial AZ91D alloy reinforced with 30%-volume fraction of aluminum borate whisker (ALBOREX M12, produced by Shikoku Chemicals Co.) was fabricated by squeeze casting. The sizes of whisker are 0.5~1.0 μm in diameter and 10~30 μm in length, and the surface of whisker is smooth in atomic scale. The chemical composition of AZ91D alloy is Mg-9.1 mass%Al-0.7 mass%Zn-0.3 mass%Mn-0.08 mass%Si-0.004 mass%Fe.

In order to avoid damage of composites because of the thermal residual stress caused by the large mismatch of thermal expansion coefficients between reinforcement and matrix, the composites was conserved in stove at 473 K for 2 hours after squeeze casting. AZ91D alloy and AZ91D/ ABO_{w} composite were solution treated at 693 K for 345.6 ks under argon atmosphere and then water-quenched. Furthermore, they were aged at 443 K in oil bath.

The hardness of AZ91D/ ABO_{w} composite and AZ91D alloy were measured using Vickers hardness measurement. Each hardness value was the average of at least ten measurements. Moreover, the relative intensities of precipitates ($\text{Mg}_{17}\text{Al}_{12}$) to magnesium in AZ91D alloy and AZ91D/ ABO_{w} composite were measured by XRD method.

The TEM specimens of both AZ91D alloy and AZ91D/ ABO_{w} composite were prepared by following procedure. At first, disks of 3 mm in diameter, 200 μm in thickness were punched from the foils for subsequent machinery polish and

Fig. 1 Aging-hardening curves of AZ91D alloy, AZ91D/ABO_w composite, AZ91 alloy³⁾ and AZ91/SiC_w composite³⁾ fabricated by squeeze casting.

ion milling. In order to retain the actual estates of materials, these experiments were kept at room temperature. TEM observations were carried out with JEOL JEM-2010 microscope at a voltage of 200 kV and double-tilt stage. In order to obtain the corresponding diffraction pattern of HREM image, Fast Fourier Transform (FFT) was used. Through FFT, the lattice image was translated to the reciprocal space, and the corresponding diffraction pattern of HREM image was obtained.

3. Results and Discussion

3.1 Age-hardening responses

AZ91D alloy and AZ91D/ABO_w composite are aged at 443 K, and the hardness of both materials is measured, as shown in Fig. 1. Additionally, the age-hardening curves of AZ91 alloy and AZ91/SiC_w composite with the whisker volume fraction of 20% were also shown in Fig. 1,³⁾ which were aged at 438 K. The hardness of all composites and alloys increase monotonically before reaching peak hardness. After reaching peak hardness, aging hardness of these materials decline little. With increasing aging time, the hardness of AZ91D alloy and AZ91D/ABO_w composite is enhanced 34 Hv and 38 Hv respectively. And the hardness of AZ91 alloy and AZ91/SiC_w composite is enhanced 32 Hv and 22 Hv respectively. The age-hardening efficiency of AZ91D/ABO_w composite is higher than that of AZ91D alloy. Nevertheless, the hardness increment of AZ91/SiC_w composite is much lower than that of AZ91 alloy. It ought to be noticed that the aging efficiency of AZ91D/ABO_w composite is better than that of AZ91/SiC_w composite, although the chemical composition of AZ91 and AZ91D alloy matrix are similar. Both composites show accelerated age-hardening behaviors. The time to reach the peak aging of AZ91D/ABO_w composite and AZ91D alloy is 115.2 and 216 ks, respectively.

3.2 Relative intensity of precipitates Mg₁₇Al₁₂

Figure 2 shows the XRD relative intensities of precipitate Mg₁₇Al₁₂ to magnesium in AZ91D alloy and AZ91D/ABO_w composite. Because of the XRD intensity is proportion to the phase content, the XRD relative intensity represents the

Fig. 2 Relative intensity of Mg₁₇Al₁₂ for magnesium in AZ91D alloy and AZ91D/ABO_w composite aged at 443 K.

relative content of Mg₁₇Al₁₂ precipitate. After solution treatment, no precipitate is found in both materials. The XRD relative intensity of precipitates to magnesium in peak-aged (at 443 K for 115.2 ks) composite is higher than that in peak-aged (at 443 K for 216 ks) AZ91D alloy. This is attributed to the interfacial reaction between magnesium and ABO_w. According to chemical eq. (1), the interfacial reaction finally increases the aluminum elements in the composite matrix, resulting in more amounts of precipitates within AZ91D/ABO_w composite.

3.3 Microstructures

Figure 3 shows the TEM micrographs of AZ91D/ABO_w composites that are (a) as-casted, (b) as-quenched and (c) peak-aged (at 443 K for 115.2 ks), respectively. Some precipitates (Mg₁₇Al₁₂) in size of several hundred nanometers form preferentially at the smooth surfaces of ABO_w in as-casted composite, as shown in Fig. 3(a). It is thought that these interfacial precipitates are formed while the composite is conserved in stove at 473 K after squeeze casting. During following solution treatment, interfacial precipitates are dissolved and MgO interfacial reaction layer in thickness of 20 nm is formed,²¹⁾ as shown in Fig. 3(b). With increasing of aging time, plate-shaped continuous precipitates Mg₁₇Al₁₂ are formed in composite matrix aged at 443 K for 115.2 ks, as shown in Fig. 3(c). And interfacial precipitate is found scarcely at MgO interfacial reaction layer. It suggests that MgO interfacial reaction layers restrain the formation of interfacial precipitate in AZ91D/ABO_w composite.

During squeeze casting, although some MgO particles are formed at the interface between AZ91D alloy matrix and ABO_w in AZ91D/ABO_w composite, the surfaces of whiskers are not covered completely with MgO particles due to the short time of squeeze casting and good chemical stability of ABO_w.²¹⁾ Figure 4 shows HREM image of the interface between interfacial precipitate (Mg₁₇Al₁₂) and ABO_w in as-casted AZ91D/ABO_w composite. The surface of whisker is smooth in atomic scale and is not covered with MgO particles. Both the plane spacing of (110)_p and (110)_p are 0.7456 nm. And the plane spacing of (100)_{ABO} and (020)_{ABO} is 0.768 nm and 0.752 nm, respectively. Since the electron

Fig. 3 TEM images of microstructures in (a) as-casted, (b) as-quenched and (c) peak-aged (at 443 K for 115.2 ks) AZ91D/ ABO_w composites, respectively.

diffraction patterns of interfacial precipitate $\text{Mg}_{17}\text{Al}_{12}$ are decorated by that of ABO_w , Fast Fourier Transform (FFT) is adopted to resolve this problem. In present study, the FFT is analyzed with DigitalMicrograph (TM) and taken from the region where is framed with white line, as shown in Fig. 4. The FFT of the HREM image is identical to the diffraction pattern.^{22–24} The image of FFT indicates that interfacial precipitate forms preferentially at the clean surface of whisker obeying a definite orientation relationship: $(110)_P // (020)_{\text{ABO}}$ and $[001]_P // [001]_{\text{ABO}}$. The mismatch between the (110) plane spacing of $\text{Mg}_{17}\text{Al}_{12}$ and (020) plane spacing of ABO_w is 0.86% with the former as standard.

Fig. 4 HREM image of interface between interfacial precipitate $\text{Mg}_{17}\text{Al}_{12}$ and ABO_w in as-casted AZ91D/ ABO_w composite.

Fig. 5 HREM image of interfacial reaction layer in as-quenched AZ91D/ ABO_w composite solution treated at 693 K for 345.6 ks.

After solution treatment, the surfaces of whiskers are covered with MgO particles, and the interfaces between MgO interfacial reaction layers and AZ91D alloy matrix become wave-like. The size of tiny MgO particles is several tens of nanometers, as shown in Fig. 5. And no interfacial precipitate is found at MgO interfacial reaction layer during following age treatment. It is thought that MgO interfacial reaction layer restrains the formation of interfacial precipitate due to higher interfacial energy between precipitate and MgO interfacial reaction layer.

Moreover, it is very important to understand the characteristics of continuous precipitates in AZ91D/ ABO_w composite, since the formation of continuous precipitate is responsible for most of the age-hardening in Mg-Al-based alloy matrix composite.^{3,4} For the sake of comparing, AZ91D alloy is aged at 443 K for 216 ks (peak-aged) and the microstructures are observed with TEM. Figure 6(a) and (b) show TEM and selected area electron diffraction (SADP) images in peak-aged AZ91D alloy observed from different crystal orientation of magnesium. It is indicated that most of continuous precipitates are thin plates lying parallel to the basal plane $(0001)_{\text{Mg}}$. And the orientation relationship

Fig. 6 TEM images of peak-aged AZ91D alloy (at 443 K for 216 ks) observed from different crystal orientation.

Fig. 7 TEM images of AZ91D/ABO_w composite aged for 115.2 ks at 443 K. The incident beam parallels to (a) $[1\bar{2}1\bar{3}]_{\text{Mg}}$ and (b) $[01\bar{1}0]_{\text{Mg}}$, respectively.

between continuous precipitates and magnesium matrix is $(110)_{\text{CP}} // (0001)_{\text{Mg}}$ and $[\bar{1}11]_{\text{CP}} // [\bar{1}210]_{\text{Mg}}$. This orientation relationship is proposed by previous researches.^{10,15–17)}

The microstructure of peak-aged AZ91D/ABO_w composite is observed with TEM. Figure 7(a) and (b) show the TEM images of same area observed from different incident beam orientation which is parallel to $[1\bar{2}1\bar{3}]_{\text{Mg}}$ and $[01\bar{1}0]_{\text{Mg}}$, respectively. It indicates that most of continuous precipitates are thin plates lying parallel to the basal plane $(0001)_{\text{Mg}}$. Slip in magnesium, like most h.c.p. metals, occurs predominantly on the close-packed basal planes (0001) . Since the continuous precipitation consists of dispersed plates lying parallel to the basal plane, there are many opportunities for dislocations to glide between the precipitates. Therefore, the age-hardening response of the AZ91 alloy is poor.¹⁰⁾ In the AZ91D/ABO_w composite, the disadvantages remain unaltered.

Figure 8(a) shows that continuous precipitate $\text{Mg}_{17}\text{Al}_{12}$ grows with the basal plane $(0001)_{\text{Mg}}$ as the growth habit plane in AZ91D/ABO_w composite matrix aged for 115.2 ks. The thickness of plate-shaped continuous precipitate is about 16 nm. Figure 8(b) shows HREM image of the interface

between magnesium alloy matrix and continuous precipitate. The FFT is analyzed with DigitalMicrograph (TM) and taken from the interface between continuous precipitate and magnesium matrix, as shown in Fig. 8(c). The image of FFT illustrates that continuous precipitate forms and grows in the matrix of AZ91D/ABO_w composite obeying Burgers orientation relationship: $(110)_{\text{CP}} // (0001)_{\text{Mg}}$ and $[\bar{1}11]_{\text{CP}} // [\bar{1}210]_{\text{Mg}}$. Figure 7 and 8 indicate that most of continuous precipitates are thin plates lying parallel to the basal plane $(0001)_{\text{Mg}}$ and this characteristic remains unaltered in AZ91D/ABO_w composite. Moreover, comparing with peak-aged AZ91D alloy, the width and thickness of continuous precipitate in peak-aged composite are not altered obviously. Nevertheless, it should be noted that the length of continuous precipitate in peak-aged AZ91D/ABO_w composite is shorter obviously than that in peak-aged AZ91D alloy. This is attributed to the narrow spacing of inter-whiskers in AZ91D/ABO_w composite.

According to the result of XRD measurement, the XRD relative intensity of precipitates to magnesium in AZ91D/ABO_w composite is higher than that in AZ91D alloy under same aging condition. It is attributed to interfacial reaction

Fig. 8 (a) HREM image of continuous precipitate in peak-aged AZ91D/ ABO_w composite, (b) high magnification image of interface between $\text{Mg}_{17}\text{Al}_{12}$ and magnesium matrix, and (c) FFT image of the interface analyzed by DigitalMicrograph (TM).

between magnesium and ABO_w . Furthermore, the interfacial precipitate is restrained due to the formation of MgO interfacial reaction layer, and the sizes of continuous precipitate in peak-aged AZ91D/ ABO_w composite are not bigger than that in peak-aged AZ91D alloy respectively. It is thought that the amount of precipitates per unit volume in peak-aged composite matrix is higher than that in peak-aged AZ91D alloy. This is supported by age-hardening curves. The amount of precipitates in composite matrix is increased, resulting in smaller inter-precipitates spacing. As a result, the age-hardening efficiency of AZ91D/ ABO_w composite is higher than that of AZ91D alloy.

Fig. 9 The high density dislocations in as-quenched AZ91D/ ABO_w composite solution treated at 693 K for 345.6 ks.

Additionally, grain boundary is not observed clearly in the matrix of AZ91D/ ABO_w composite. It is probable that the grain boundaries distribute along whiskers. During the solidification of squeeze casting, surfaces of ABO_w serve as heterogeneous nucleation sites for matrix grains, the growth of magnesium grains are restricted markedly by the high volume fraction of ABO_w . As a result, the grain boundaries are decorated by the interfaces between matrix and whisker. According to previous studies,¹⁰⁾ discontinuous precipitate ($\text{Mg}_{17}\text{Al}_{12}$) forms at grain boundaries and grows behind a moving grain boundary in earlier aging process for Mg-Al-based alloy. Two mechanisms for discontinuous precipitation have been proposed previously. One ascribed to Tu and Turnbull^{25,26)} dominates at low temperature, and another proposed by Duly dominates at high temperature.^{13,14)} However, independent of the exact mechanisms, both the mechanisms require the migrations of grain boundary to serve as a reaction front, the movability of the grain boundaries being a crucial factor for the nucleation and growth of discontinuous precipitation.^{12,14)} In AZ91D/ ABO_w composite, the grain boundaries are bounded by the ABO_w and have limited movability. It is disadvantage for discontinuous precipitation. Therefore it is very difficult for the formation of discontinuous precipitates at the grain boundaries in the composite. The result of present observation is similar to that in AZ91/ SiC_w composite.

Figure 9 shows the high-density dislocation arrays in the matrix of AZ91D/ ABO_w composite after solution treatment and water quenching. As well known, matrix dislocations have long been recognized as the preferential nucleation sites for $\text{Mg}_{17}\text{Al}_{12}$ precipitates.^{14,16,17)} In addition, the diffusion rate of solution atoms will be enhanced along the dislocations pipe.³⁾ Clark¹⁴⁾ indicates that introduction of dislocations by cold work prior to aging has the same effect of enhancing precipitation of $\text{Mg}_{17}\text{Al}_{12}$ in Mg-9 mass%Al alloy. High-density dislocations resulting from the cold work serving as preferential nucleation sites of $\text{Mg}_{17}\text{Al}_{12}$, markedly increasing the rate of age-hardening of AZ91D alloy, similar to that observed in the present composite. As a result, rate of precipitation in AZ91D/ ABO_w composite is accelerated.

Fig. 10 Sketch maps of precipitation behaviors in (a) AZ91/SiC_w³⁾ and (b) AZ91D/ABO_w composite, respectively.

3.4 Comparison of aging behaviors in AZ91D/ABO_w and AZ91/SiC_w composite

In order to evaluate the effects of interfacial reaction on age-hardening efficiency in Mg-Al-based alloy matrix composite exactly, the behaviors of precipitation in AZ91D/ABO_w composite are compared with that in AZ91/SiC_w composite. Zheng *et al.*³⁾ have studied the aging behaviors in AZ91/SiC_w composite fabricated by squeeze casting. No interfacial reaction is found in solution treated AZ91/SiC_w composite. Precipitates preferentially nucleate and grow at the clean interface between matrix and whisker. These interfacial precipitates finally deplete the aluminum element in the matrix, resulting in less amounts of continuous precipitates within composite matrix. As a result, age-hardening efficiency of AZ91/SiC_w is lower than that of AZ91 alloy. Badini *et al.*⁴⁾ find that the precipitation characteristics in the AZ80/B₄C_p composites are similar to that in AZ91/SiC_w composites. As shown in Fig. 10(a), no interfacial reaction is found in AZ91/SiC_w composite, and the amount of continuous precipitates is depressed because the aluminum element in the matrix is depleted by the interfacial precipitation. There are more opportunities for dislocations to glide between the precipitates comparing with that in AZ91 alloy.

On the other hand, magnesium reacts to ABO_w and forms MgO interfacial reaction layer during solution treatment. As products of interfacial reaction, aluminum is dissolved into composite matrix. The concentration of aluminum in AZ91D/ABO_w composite matrix is enhanced. Moreover, MgO interfacial reaction layer restrains the formation of interfacial precipitate. As a result, the amount of continuous precipitates is enhanced in AZ91D/ABO_w composite matrix and the inter-precipitates spacing is reduced, as shown in Fig. 10(b). So the age-hardening efficiency of AZ91D/ABO_w composite is not only higher than that of AZ91D alloy, but also higher greatly than that of AZ91/SiC_w composite.

Under these investigations, the effects of reinforcements on precipitates behaviors in discontinuously reinforced Mg-Al-based alloy matrix composites begin to be clarified. It would be worthwhile to reduce precipitation at the ceramic-metal matrix interfaces with suitable methods, such as modification of the matrix-reinforcement interface.

4. Conclusions

AZ91D/ABO_w composite exhibits an accelerated age-hardening response comparing with AZ91D alloy.

The age-hardening efficiency of AZ91D/ABO_w composite is higher than that of the AZ91D alloy. This is attributed to the interfacial reaction. The interfacial reaction increases aluminum elements in the matrix and MgO interfacial reaction layer restrains the formation of interfacial precipitate, resulting in more amounts of continuous precipitates, smaller inter-precipitates spacing within the AZ91D/ABO_w composite matrix.

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