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Characteristic Reaction Products in the AZ91/SiC Composite Fabricated by Pressureless Infiltration Technique

K. B. Lee, J. H. Choi, and H. Kwon*

School of Advanced Materials Engineering, Kookmin University,
861-1, Jeongneung-dong, Seongbuk-gu, Seoul 136-702, Korea

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Since the spontaneous infiltration of molten AZ91 Mg alloy into a powder bed containing SiC particles occurred at 700 °C for 1 h under a nitrogen atmosphere, it was possible to fabricate Mg alloy composites reinforced with SiC particles. Since the fabrication conditions (e.g. temperature, time, and atmosphere) of the composite are different from those of the other fabrication route, reaction products formed during the composite fabrication were investigated in detail using field emission scanning electron microscopy (FE-SEM) and high resolution transmission electron microscopy (HRTEM). From the analysis results, we could identify the formation of $MgAl_2O_4$ and AlN, as well as MgO in fabricated composite.

Keywords: Mg composite, SiC, pressureless infiltration

1. INTRODUCTION

Metal matrix composites (MMCs) reinforced with a ceramic phase have attracted extensive interest because of the combined effects of metallic and ceramic materials relative to the corresponding monolithic alloys. Thus, various fabrication methods have been developed, such as powder metallurgy, stir casting, and the pressure infiltration method, among others, to produce these materials. Over the past several decades, much of the research on MMCs has focused on Al matrix composites, and various aspects of Al matrix composites are well-known, including fabrication methods, mechanical properties, microstructure, and interfacial reaction [1-5].

Recently, there has been growing interest in the development of technologies for the production of Mg matrix composites because of their attractive properties. Mg matrix composites have a low density, a high specific strength, and stiffness at room temperature as well as at elevated temperatures with a superior wear resistance and damping capacity [6-12]. However, the research on Mg matrix composites is still limited compared to that of Al matrix composites. In general, Mg(or Mg alloy) matrix composites reinforced with SiC have been produced by stir casting [6-8], powder metallurgy [9], squeeze casting [10,11], and spray forming [12].

The pressureless infiltration method is an innovative technique for fabricating MMCs by the spontaneous infiltration of a molten Al alloy containing Mg into a ceramic filler or preform under a nitrogen atmosphere, without the aid of a vacuum or externally applied pressure [4,5,13,14]. Although there have been many reports on Al alloy matrix composites fabricated by the pressureless infiltration method, few publications have appeared on Mg alloy matrix composites. The interfacial reaction of the MMCs is known to affect the properties of the MMCs. Since the interfacial reaction in the composites depends on several fabrication parameters (for example, temperature, holding time, atmosphere, and the chemical composition of both the matrix and the reinforcement), the characteristics of fabricated composites vary depending on the fabrication procedure and composite system [15].

The interfacial reaction of the Mg matrix composite is very different from that of Al matrix composites because of the high reactivity of Mg. Furthermore, the fabrication temperature and production time of the MMCs in the pressureless infiltration method are higher and longer than those of other fabrication methods, which may cause severe interfacial reactions in the interface. Therefore, the aim of the present study is to investigate the interfacial reaction of Mg alloy matrix composite fabricated by the pressureless infiltration method.

2. EXPERIMENTAL PROCEDURE

The AZ91/SiC composite used in this study was fabricated by the pressureless infiltration method, and the fabrication

*Corresponding author: hkwon@kookmin.ac.kr

setup was identical to the one used for the fabrication of Al matrix composites [13,14]. The average sizes of Mg, Al, Zn, and SiC particles used in this study were approximately 13 μm, 50 μm, 20 μm, and 22 μm, respectively. After these powders, whose compositions were Mg-9 wt.%Al-1.0 wt.%Zn -30 vol.%SiC, were blended by roll mixing in an alumina jar, the powder mixture was put into a crucible, and an AZ91 Mg ingot was placed on this powder bed. This assembly was heated to 700 °C and held for 1 h under a flowing nitrogen atmosphere in a horizontal tube furnace (1000 cc/min). Then, the assembly was cooled to about 300 °C under the nitrogen atmosphere to inhibit oxidation during solidification and was subsequently removed from the furnace.

The resulting microstructures and reaction products were investigated using field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). For SEM analysis of the reaction products, specimens were prepared by dissolving the metal matrix in a solution of methanol bromine and examined using a JSM 7401F SEM equipped with an energy dispersive X-ray spectrometer (EDS) operated at 10 kV. Samples for TEM observation were prepared using a conventional procedure (mechanical polishing, dimpling, and finally ion milling) as well as the focused ion beam method (FIB). All samples were examined by JEM 1210 TEM and Tecnai G^2 TEM coupled to an EDS system operated at 120 kV and 200 kV, respectively.

3. RESULTS AND DISCUSSION

The spontaneous infiltration of molten metal at 700 °C for 1 h under a nitrogen atmosphere made it possible to fabricate an AZ91 Mg matrix composite reinforced with SiC. While SiC particles were evenly distributed in the Mg alloy matrix, it was difficult to discriminate the reaction products formed at the interface between SiC particles and the Mg alloy matrix because of the relatively low magnification in the optical microscopy (not shown here). Thus, FE-SEM and TEM investigations were performed in order to identify the reaction products in detail.

Figure 1 shows the reaction products and EDS analysis observed by the SEM after the Mg alloy matrix was dissolved away with a solution of methanol bromine. It can be seen that reaction products of various sizes were formed on the SiC particle. From the EDS analysis, we observed that the composition of the reaction products of the polyhedron morphology is consistent with the stoichiometry of MgO. This phase a possible reaction product in the Mg-SiC system, and several works have reported the formation of MgO in the AZ91/SiC composite [10,11].

Figure 2 shows the SEM micrograph and EDS profile of another reaction product observed on the surface of SiC in the composite. According to the analytical results obtained from EDS analysis, reaction product was identified as $MgAl_2O_4$. It is believed that both the Si and C peaks in the EDS profile were obtained from SiC particle. While this phase may also be formed by an interfacial reaction in the Mg/SiC system, little literature is available on the formation of Mg-based composites. Therefore, TEM observation was performed in order to clearly identify the formation of $MgAl_2O_4$.

Figure 3 shows bright field and dark field images and the selected area diffraction pattern (SADP) of the reaction product observed by TEM. After careful indexing, this reaction product was identified as $MgAl_2O_4$ with a cubic structure and lattice parameter, a = 8.081 nm (theoretical value a = 8.0887 nm, space group: Fd3 m) [17]. In addition, the high-resolution transmission electron microscopy (HRTEM) image of $MgAl_2O_4$ reveals that the interplanar distance between adjacent lattice planes is about 2.89 Å, corresponding to the (220) lattice plane of cubic $MgAl_2O_4$. Fast Fourier transformation (FFT) of the HRTEM image reveals the same result (inset). Therefore, we could identify the formation of $MgAl_2O_4$ through a series of TEM observations. The AZ91/SiC composite in this study was fabricated by the spontaneous infiltration of molten metal into the powder bed containing Mg, Al, Zn, and SiC particles. The surface of constituent

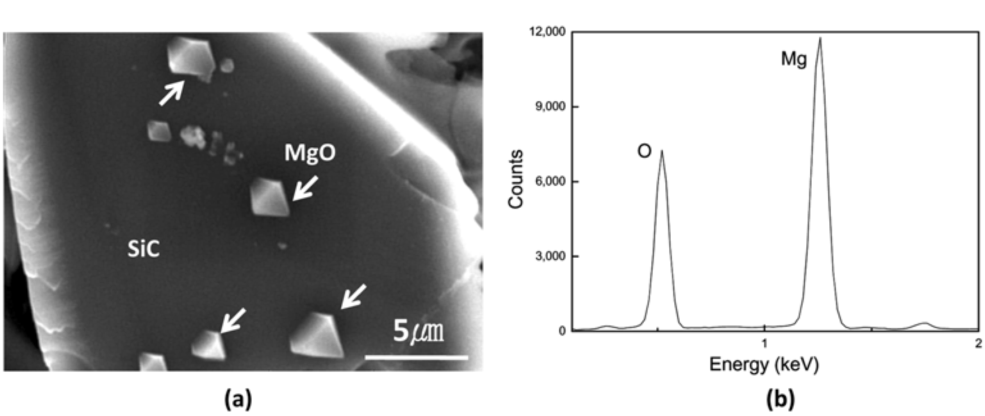

Fig. 1. SEM image and EDS profile showing a reaction product (MgO) formed on the surface of the SiC particle.

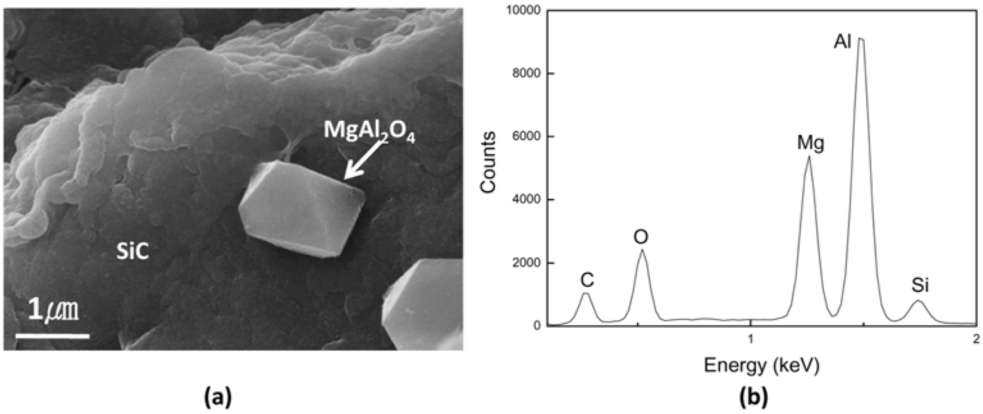

Fig. 2. SEM image and EDS profile showing a reaction product ($MgAl_2O_4$) formed on the surface of the SiC particle.

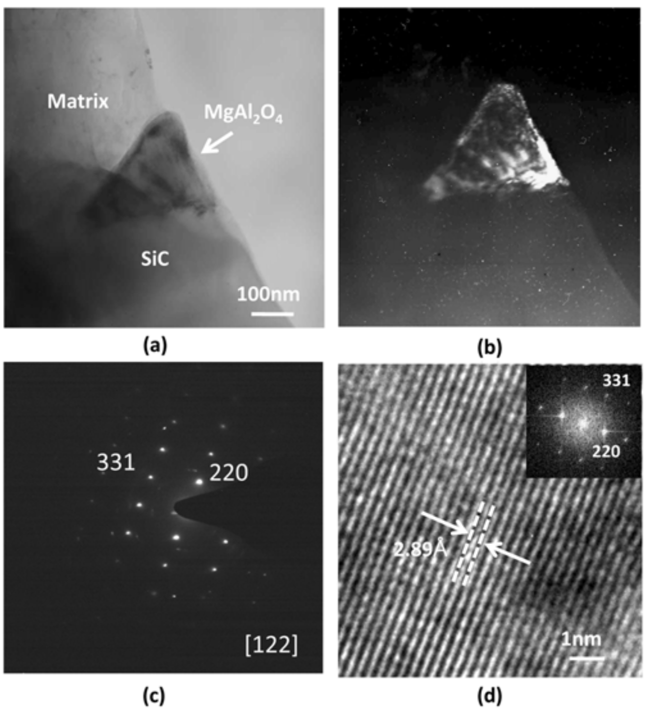

Fig. 3. Bright field, (a) dark field, (b) and SADP (c) images of $MgAl_2O_4$ formed at the SiC/matrix interface. (d) HRTEM image viewed along the [122] zone axis of $MgAl_2O_4$. The inset is the corresponding FFT image.

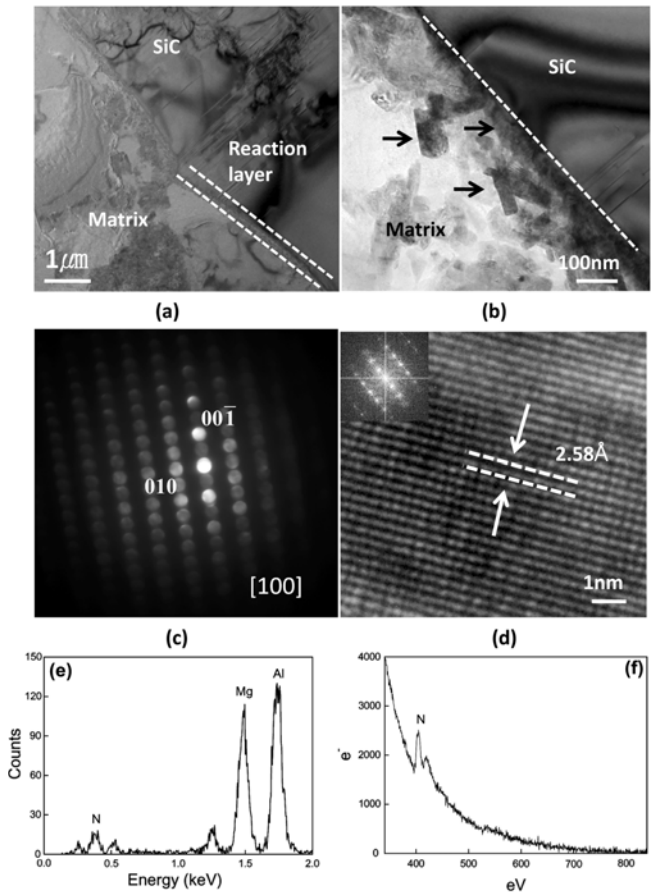

Fig. 4. (a) Low- and (b) high-magnification TEM images showing the reaction layer at the interface. (c) Microdiffraction pattern of the reaction layer. (d) HRTEM image viewed along the [100] zone axis of AlN. The inset is the corresponding FFT image. (e) and (f) are EDS and EELS profiles of the reaction product.

powders in the powder bed inherently contains oxide layers because of the reaction with oxygen. Therefore, oxide films on Al, Mg, Zn, and SiC particles in the powder mixture can provide a possible source of oxygen for the formation of the MgO and $MgAl_2O_4$, in addition to oxygen entrapped in the powder bed and remaining in atmosphere gas.

Figure 4(a) is a low magnification image observed by TEM. In this case, the TEM specimen was prepared by the focused ion beam (FIB) method. It can be seen that a continuous reaction layer is formed at the interface between the Mg alloy matrix and SiC particle. This layer could not be clearly identified with EDS analysis due to the large probe size in the SEM. Figure 4(b) shows a higher magnification image of the same region observed by TEM. From the EDS analysis in TEM (Fig. 4(e)), Al, Mg, and N were detected in the reaction layer. The presence of nitrogen in the reaction layer is also identified with EELS analysis in the TEM (Fig. 4(f)). This reaction product is verified as AlN by micro diffraction pattern, HRTEM, and FFT analysis. AlN has been observed in all Al matrix composites fabricated using the present pressureless infiltration technique; formation of the AlN is related to the fabrication method of MMC used in this study. It is suggested that Mg vapor reacts with nitrogen to form a Mg_3N_2 coating around the particles in the preform or filler [4,5]. This Mg_3N_2 may lead to a spontaneous infiltration of molten Mg alloy via substantial enhancement of wetting between the molten alloy and reinforcement. In addition, this Mg_3N_2 reacts with molten Al, and, thus, AlN is formed as a result of the *in situ* reaction ($Mg_3N_2 + 2Al = 2AlN + 3Mg$) [4,5,14]. Therefore, it is believed that AlN observed in this study may be formed through the same mechanism because it contains the same elements (Mg, Al, and N) in the Mg based composite fabricated by the same pressureless infiltration method. Actually, this AlN is frequently observed in this study by TEM analysis. However, a Mg element was also detected by EDS analysis in the reaction product. Unfortunately, not much is known about the phase containing Al, Mg, and nitrogen. Furthermore, all analysis results (diffraction pattern, HRTEM, and FFT analysis) show the same result, confirming in fact that the reaction product is AlN. As a result, it is believed that the reaction product is AlN containing Mg.

4. CONCLUSIONS

The spontaneous infiltration of molten metal at 700 °C for 1 h under a nitrogen atmosphere made it possible to fabricate an AZ91 Mg matrix composite reinforced with SiC. Since the fabrication conditions (e.g., temperature, time, and atmosphere) of the composite are different from those of the other fabrication route, reaction products formed during the composite fabrication were investigated in detail by FE-SEM and high resolution TEM. From the analysis of reaction products, we can identify the formation of MgO, $MgAl_2O_4$ and an AlN phase containing magnesium.

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