

INTERFACIAL REACTION IN WHISKERS REINFORCED MAGNESIUM MATRIX COMPOSITES

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SUMMARY: The whisker/matrix interfaces in squeeze cast SiCw/AZ91 Mg composites and $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{Mg}$ composites were studied by transmission electron microscopy (TEM) and high resolution electron microscopy (HREM). For the SiCw/AZ91 composites with silica binders, there existed fine discrete interfacial reaction products MgO at the interface, which were resulted from the reaction between the silica binder and the molten Mg. SiCw did not take part in the interfacial reaction. While in the $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{AZ91}$ composites with silica binder, there existed interfacial reaction products MgO layer at the interface. Besides the reaction between the liquid magnesium and silica binder, the reaction between liquid magnesium and aluminum borate whisker contribute to the formation of MgO. $\text{Al}_{18}\text{B}_4\text{O}_{33}$ whisker was unstable in the $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{AZ91}$ composites.

KEYWORDS : Magnesium alloy, SiC whisker, Aluminum borate whisker, MMC, Interfacial reaction

INTRODUCTION

Discontinuously reinforced magnesium matrix composites have increasing potential in automotive, high performance defense and aerospace application because of their low density, high specific strength, high specific stiffness, high wear resistance, low coefficient of thermal expansion and high damping capacity [1-6]. Discontinuous reinforcements such as short fibers [1,2], particles [3, 4], or whiskers [5,6], have been used as reinforcements of Mg MMCs, because conventional fabrication methods can be applied and the cost is lower than that of continuous reinforcements. Among the composites, whiskers reinforced magnesium matrix composites [5,6] exhibit the best properties due to the better intrinsic properties of whiskers.

SiC whiskers have been widely used as the reinforcement in the magnesium matrix composites, but SiCw/Mg composites are considered to be difficult to be used in civil industry because of the high cost of SiC whisker. Recently, aluminum borate whisker ($\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}$) with low price has been used to reinforce magnesium alloy [7]. The $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{Mg}$ composites are expected to apply to the civil industry, because of their lower cost compared with SiCw/Mg composites.

Magnesium has good wetting or bonding to most of the ceramic reinforcements compared to

aluminum. On the other hand, it may be highly reactive with potential reinforcements, resulting to the interfacial reaction between the reinforcements and the matrix. In general, a frequent problem encountered in the magnesium matrix composites can be the interfacial reaction between magnesium alloy matrix and reinforcement, which can influence the properties of magnesium matrix composites. The nature of the interface in discontinuously reinforced magnesium matrix composites depends on a variety of factors, among them, the nature of reinforcements play an important role on the interfacial nature of the composites[8].

In this paper, the SiCw/AZ91 and Al₁₈B₄O₃₃w/AZ91 magnesium matrix composites were fabricated by squeeze casting process. The aim of the paper is to investigate the interfacial reactions in the two kinds of magnesium matrix composites. Particular attention was paid on the morphology, structure and the formation mechanisms of the reaction products.

EXPERIMENTAL

β-SiC whisker (Tokai Carbon Co. Ltd, Japan) and aluminum borate whisker (Grade M12, Shikoku Kasei, Japan) were employed as the reinforcements, AZ91 magnesium alloy (8.5-9.5%Al, 0.45-0.90%Zn, 0.15-0.30%Mn, 0.20%Si, 0.01%Ni, balance Mg) was employed as the matrix.

The composites were produced by a squeeze casting process. The silica binder was used to prepare the preforms. The mold and the preforms were preheated at 500⁰C. The magnesium melt overheated to 800⁰C was poured into the preform. The pressure exerted by the punch on the molten metal was gradually increased to a pre-determined level of 100 MPa in order to avoid deformation of the preform, and kept at this level for 3 minutes until the molten metal was completely solidified. The volume fractions of the whiskers were about 20% in the composites.

To evaluate the interface of the composites, specimens for TEM were ground mechanically into 30 μ m thick plate. Disc specimens of 3mm in diameter were dimpled on one side with a Gatan dimpler, until the central region was about 10 μ m thick. Final thinning to perforation was carried out with a Gatan Model 600 ion-milling machine. A liquid nitrogen cold stage was used to prevent the heat effect of the ion milling process.

The interface of the composites was examined using Philip EM420 transmission electron microscope (TEM) and JEOL2000EX-II high resolution transmission electron microscopes (HRTEM), to determine the morphology, structure and bond condition between the reaction products, whiskers and matrix.

RESULTS AND DISCUSSION

Interface in the SiCw/AZ91 composites

Fig.1 shows the TEM micrograph of interface between SiCw and AZ91 in SiCw/AZ91 magnesium matrix composites without any binder. The interface is very flat and clean. It can be seen that SiC whisker is very stable in the magnesium matrix under the squeeze cast condition employed in the present study.

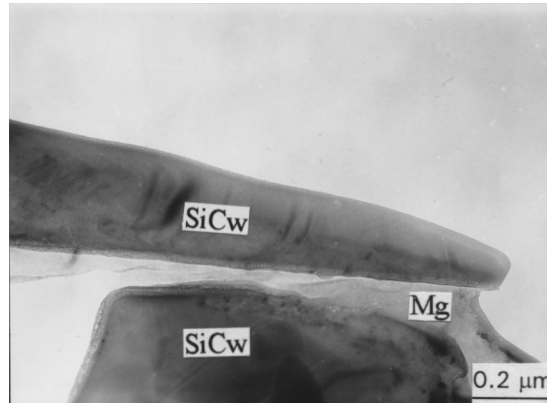


Fig.1 TEM micrograph of interface between SiCw and Mg in SiCw/Mg composites without binder, illustrating clean interface without reaction products.

Fig.2 is the TEM micrographs of the interface in SiCw/AZ91 composite with silica binder. It can be seen that there exist fine discrete interfacial phases at the interface, the size of the phase is about 20-50nm.

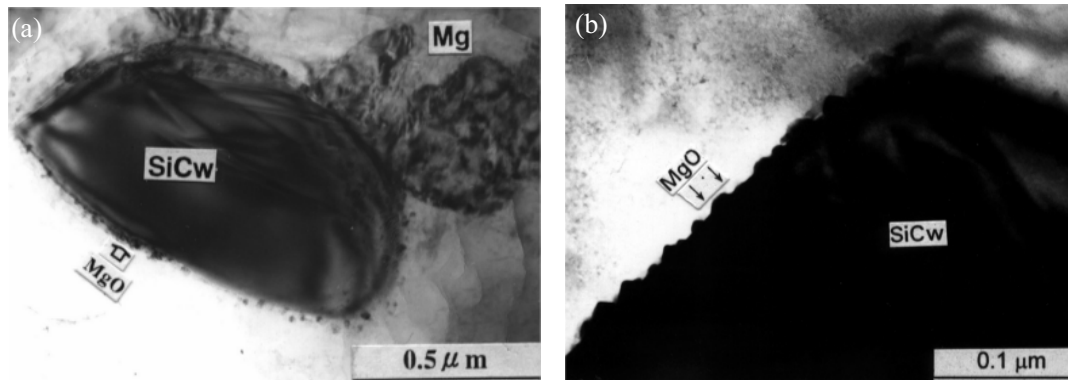


Fig.2 TEM micrographs of the SiCw-Mg interface in SiCw/AZ91 composite with silica binder, (a) low magnification, (b) high magnification.

Because the interfacial particles were too small to be identified by conventional selected area diffraction or micro-diffraction, HREM was performed to identify the interfacial phases. Fig.3 shows an HREM image of the interfacial phase present at the SiCw-Mg interface in the composites with silica binder. Two sets of basic lattice planes with a measured spacing of 0.24nm (labeled (111) and $(1\bar{1}1)$) and an angle of 70 degree were observed. The measured angle between the two lattice planes is 70 degree. The above-measured crystallographic parameters correspond closely to the cubic MgO ($a = 0.42\text{nm}$) phase in $\langle 101 \rangle$ direction.

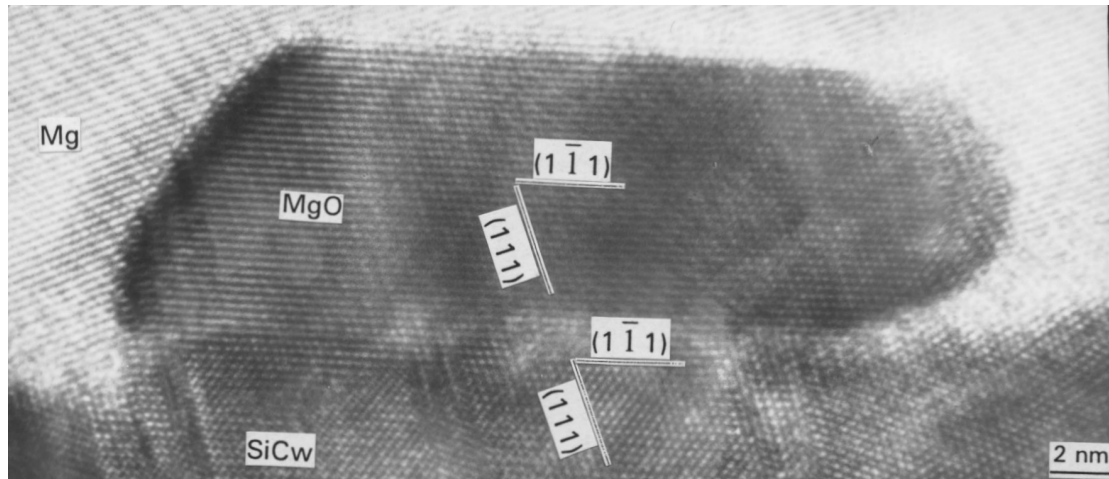


Fig.3 HREM image of interfacial reaction product MgO in the SiCw/AZ91 composite with silica binder. The incident beam is along the $[10\bar{1}]$ axis of the SiC whisker.

The possible reaction products that can form at the interface of SiCw/AZ91 composite are $Mg_{17}Al_{12}$, Mg_2Si , Al_4C_3 , Al_2O_3 , $MgAl_2O_4$ and MgO , etc. Because the Mg-O, Al-O and Si-O bonds are significantly stronger than the Al-C, Al-Mg and Mg-Si bonds [9], oxide phases should form in preference to other possible compounds such as Mg_2Si , Al_4C_3 and $Mg_{17}Al_{12}$ until all of the available oxygen is consumed. Among the possible oxides, the formation free energy for MgO is lower than that of $MgAl_2O_4$ and Al_2O_3 . The study on thermodynamic stability of Al-Mg oxides in Al-Mg alloys [10] shows that the formation of Al_2O_3 , $MgAl_2O_4$ and MgO are competitive processes, with preferential MgO formation for high Mg content. These thermodynamic considerations show that the most stable oxide in Mg MMCs is MgO . On the basis of structure and thermodynamic analysis, the fine interfacial phases are identified as MgO ($a=0.42nm$).

As shown in Fig.3, the interfacial phase MgO is perfectly bonded to SiC whisker without amorphous transition layer at the surface of SiC whisker. There exists a definite orientation relationship between MgO and SiCw, that is: $\{111\}_{MgO} // \{111\}_{SiCw}$, $\langle 101 \rangle_{MgO} // \langle 101 \rangle_{SiCw}$. The lattice matching between the two phases at the interface is good, the mismatch between the two lattice spacing, the $\{111\}$ spacing of β -SiCw (0.25nm) and the $\{111\}$ spacing of MgO (0.24nm), is about 4.0 % with the former as a standard.

Because there exists no fine interfacial phases at the composites without any binder, it can be thought that the interfacial reaction products MgO originated mainly from the reaction between silica binder and Mg in the SiCw/AZ91 composites, that is:



Interface in the $Al_{18}B_4O_{33}w/AZ91$ composites

Fig.4 is the TEM micrographs of the interface in $Al_{18}B_4O_{33}w/AZ91$ composite with silica binder. A layer of reaction products with a thickness of 50-100 μm was observed at the matrix-whisker interface.

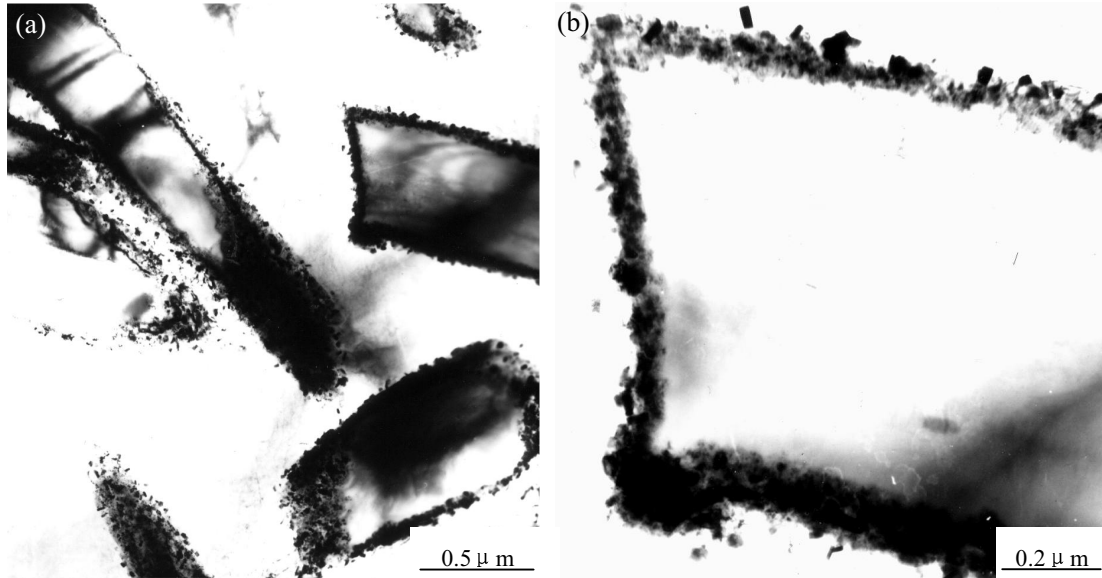
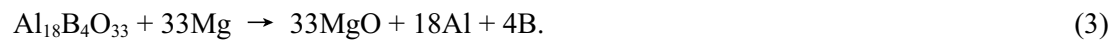


Fig.4 TEM micrographs of the $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}$ -Mg interface in $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{AZ91}$ composite with silica binder, (a) lower magnification, (b) higher magnification.

TEM and EDAX analysis show that the layer was composed of polycrystalline MgO particles. The reaction products formed a layer, other than discrete interfacial reaction products in the SiCw/AZ91 composites with silica binder. Furthermore, the size of the reaction products was larger. It also can be seen that the surfaces of the whiskers become slightly rougher, indicating that the aluminum borate whiskers take part in the reaction.

Besides the reaction between the liquid magnesium and silica binder, the reaction between liquid magnesium and aluminum borate whisker contribute to the formation of MgO. The main interfacial reaction process in the $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{Mg}$ composites are as follows: firstly, liquid magnesium alloy reacts with silica binder at the surface of the $\text{Al}_{18}\text{B}_4\text{O}_{33}$ whisker, then the liquid magnesium alloy reacts with $\text{Al}_{18}\text{B}_4\text{O}_{33}$ whisker. Both of the reactions lead to the formation of MgO. The reaction equations are as follows:



From the above results and analysis, it can be seen that the interfacial stability of magnesium matrix composites depends on the reinforcement employed. In the SiCw/AZ91 composites, the surface of SiC whisker was very flat and clear, SiCw did not take part in the reaction. While in the $\text{Al}_{18}\text{B}_4\text{O}_{33}\text{w}/\text{Mg}$ composites, the $\text{Al}_{18}\text{B}_4\text{O}_{33}$ whisker surface became slightly rougher, the $\text{Al}_{18}\text{B}_4\text{O}_{33}$ whiskers took part in the reaction, although the interfacial reaction was slight, not very serious.

CONCLUSIONS

1. There existed fine discrete interfacial reaction products MgO at the interface of the SiCw/AZ91 composites with silica binder. Interfacial reaction products MgO were resulted

from the reaction between the silica binder and the molten Mg. There existed a definite orientation relationship between MgO and SiCw.

2. There existed interfacial reaction products MgO layer at the interface of the $Al_{18}B_4O_{33}w$ /AZ91 composites with silica binder. Besides the reaction between the liquid magnesium and silica binder, the reaction between liquid magnesium and aluminum borate whisker contribute to the formation of MgO.

3. The interfacial stability of magnesium matrix composites depended on the reinforcements employed. In the SiCw/AZ91 magnesium matrix composites, the interface is very stable, SiCw did not take part in the interfacial reaction. While in the $Al_{18}B_4O_{33}w$ /AZ91 composites, the interface is unstable, $Al_{18}B_4O_{33}$ whisker reacted with AZ91, leading to the formation of MgO.

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