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Interfacial bond between SiCw and Mg in squeeze cast SiCw/Mg composites

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Abstract

The interfacial microstructure of precipitate- and reaction-free interface in squeeze cast SiCw/AZ91 Mg matrix composites were investigated by High Resolution Electron Microscopy (HREM). Two kinds of crystalline orientation relationships between SiC whisker and Mg are observed, which resulted from the solidification of molten Mg at the different surface planes of SiCw exposed whisker. The interfaces with orientation relationships are semi-coherent interfaces and high interfacial bond strength exist in these interfaces. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: SiC whisker; Magnesium alloy; Composites; Interface; Orientation relationships; Bond

1. Introduction

In recent years, discontinuously reinforced magnesium matrix composites have been receiving great attention as an attractive choice for aerospace and automotive application, owing to their low density, high specific strength, high specific stiffness and high damping capacity [1,2].

The interface between matrix and reinforcement is very important in influencing the physical and mechanical properties of the metal matrix composites, because interface occupies a very large area in composites, and the reinforcement and the matrix will form a system that is not of thermal equilibrium.

For discontinuously reinforced metal matrix composites, the main mechanism of strengthening has not been fully understood. Whether the main mechanism of strengthening is work hardening of matrix due to the relaxation of the thermal residual stress [3] or load transfer from matrix to reinforcement [4], the strong bond at the interface is still very important.

For SiC particle or whisker reinforced Al MMCs, many researches have been focused on the bonding strength [5,6] and the orientation relationships [7-12] between the SiC and Al alloy. However, the interfacial bond mechanism is poorly understood, and there

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is no consensus as to the orientation relationship at the SiC/Al interface.

The orientation relationships (ORs) in SiC/Al MMCs are associated with the fabrication methods of the composites. If the matrix is in the solid state in the P/M fabrication of SiC/Al composites, different results about ORs were reported [7,8]. But if the matrix was in the molten state in the PM fabrication of SiC/Al, consistent orientation relationships (ORs) were found [9,10] for same composite system by different research groups. There exists specific orientation relationship at the interface in order to decrease the interfacial energy. If the composites were fabricated by squeeze casting method, a kind of orientation relationship between SiC whisker and Al matrix were also reported [11,12].

For SiC/Mg MMCs, very few results about interfacial bond mechanism and orientation relationships were reported. No specific orientation relationship between SiC particle and Mg was found at the interface of stir cast SiCp/ZC63 Mg MMCs [13]. Luo [14] thought there should exist some specific ORs at the stir cast SiCp/AZ91 composites, because AZ91 preferentially nuclease at the SiCp surface, but he did not study orientation relationships by TEM.

In our previous work, the interfacial reaction [15] and interfacial precipitation [16] in squeeze cast SiCw/Mg composites have been studied in detail. In this paper, the reaction- and precipitates-free interface, or clean, direct connecting interface between

SiCw and Mg was investigated in order to further understand the interfacial structure and interfacial bonding mechanism of SiCw/Mg composites.

2. Experimental procedure

The composites used in this investigation was fabricated by squeeze cast under CO_2/SF_6 atmosphere; the matrix alloy is 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); the reinforcement is β -SiC whisker; the volume fraction of the whisker in preform is 20%. In squeeze casting, the mold and the preform are preheated to 500°C; liquid magnesium overheated to 800°C was poured into the preform.

The specimens for TEM observation were sectioned from as-cast composites with electrical discharge machine (EDM), ground into 30 μ m thick thin plate. Disc specimens of 3 mm in diameter were dimpled with Gatan dimpling machine on one side until the thickness at the center of specimen was about 10 μ m, then ion milled to perforation with Gatan Model 600 ion-milling machine, at 4 kV and 0.5 mA gun current under an angle of 20° and 8° to the sample. A liquid nitrogen cold stage was used to prevent the heat effect of the ion milling process.

The interface of the composite was examined in detail by JEM 200CX and JEOL 2000EX-II high



Fig. 1. TEM micrographs of interface in the SiCw/AZ91 composites. (a) Morphology of the interface along the zone axis of $[011]_{SiCw}$; (b) SADP across the interface of SiCw and Mg with zone axis of $[011]_{SiCw}$ and $[011]_{Mg}$.

resolution transmission microscope with a point to point resolution of 0.21 nm.

3. Results and discussion

In squeeze cast SiCw/Mg composites, two kinds of crystalline orientation relationships between SiCw and Mg were observed at the interface by TEM and HREM.

Fig. 1(a) shows morphology of the interface between SiC whisker and AZ91 in SiCw/AZ91 composites in the direction of [011] of SiCw. Fig. 1(b) is the SADP across the interface of Mg matrix and SiC whisker in the zone axis of $[011]_{SiCw}$ and $[011]_{Mg}$.

It can be seen that there exists a crystalline orientation relationships (ORs) between matrix and SiCw as follows:

$$(01\overline{1})_{Mg}//(\overline{1}1\overline{1})_{SiCw}$$
, or $(01\overline{1}\overline{1})_{Mg}//(\overline{1}1\overline{1})_{SiCw}$,
 $[011]_{Mg}//[011]_{SiCw}$, or $[\overline{1}2\overline{1}3]_{Mg}//[011]_{SiCw}$.

These ORs can be seen more clearly in the HREM image of the interface. Fig. 2 shows HREM image of



Fig. 2. HREM images of the interface between SiCw and AZ91 in SiCw/AZ91 composites along the [011] axis of SiCw, illustrating ORs: $(01\overline{11})_{Mg}/(\overline{111})_{SiCw}, [\overline{1213}]_{Mg}//[011]_{SiCw}.$

the SiCw-Mg interface in Fig. 1(a), the electron beam was along the [011] axis of SiC whisker. The interface is sharp and no voids can be seen at the interface. This part of interface can be defined as clean. It was observed that SiCw and Mg connect directly at the interface with $(01\overline{11})_{Mg}$ plane bond parallel to the $(\overline{111})$ plane of SiCw. For the $(01\overline{11})_{Mg}/((\overline{111})_{SiCw})$, the mismatch generated is about 2.78%, as can be seen below: $\delta = d(\overline{111})_{SiCw} - d(01\overline{11})_{Mg}/d(\overline{111})_{SiCw} \times 100\% = 0.252 - 0.245/0.252 \times 100\% = 2.78\%$. The lattice mismatch is small with little strain at the interface and this kind of interface can be considered as a semi-coherent interface.

Fig. 3(a) shows morphology of the interface between SiCw and AZ91 along the axis zone [101] of SiCw in SiCw/AZ91 composites. Fig. 3(b) is the diffraction pattern of the interface in the direction of $[101]_{SiCw}$ and $[0001]_{Mg}$. Another kind of crystalline orientation relationships between AZ91 and SiCw can be observed as follows:

$(11\overline{2}0)_{Mg}//(111)_{SiCw}[0001]_{Mg}//[10\overline{1}]_{SiCw}$

Fig. 4 shows the HREM image of this kind of ORs at the interface in Fig. 3(a). It can be observed that the (11 $\overline{2}0$) planes of Mg align with the (111) planes of SiCw across the interface directly, and there exists a large mismatch at the interface. The mismatch at the interface between (11 $\overline{2}0$)_{Mg} and (111)_{SiCw} is 36.3%.

From the above observation and analysis, two kinds of different ORs between SiCw and Mg were observed at the interface in the squeeze cast SiCw/Mg composites. At these interfaces, low index planes, or close packed planes of SiCw and Mg align with each other at the interface with small lattice misfit strain, suggesting that the bond of these kinds of interfaces was good.

In the squeeze casting of SiCw/Mg MMCs, SiC whisker surface can act as effective heterogeneous nucleation site for molten Mg alloy. If crystallization of molten matrix occurs according to certain orientation relationships between SiC whisker and Mg, the energy needed for nucleation and growth will be lower, resulting to low strain energy and high adhesion energy at the interface, which is beneficial to the interfacial bond.

The above observation and analysis implies that the ORs is not unique, there seems to exist at least two kinds of ORs at the interface of SiCw/Mg composites, and at some parts of the interface, no ORs exists. This may be concerned with the solidification process of Mg alloy matrix in the squeeze casting of the composites and the surface structure of SiC whisker.

If Mg alloy solidify with the SiCw surface as the nucleation site, ORs will exist. The side surfaces of β -SiCw are mainly composed of {111}, {220}, {200} crystal planes [17]. In the squeeze casting of the composites, SiCw has different exposed surface. If



Fig. 3. TEM micrographs of interface in the SiCw/AZ91 composites. (a) Morphology of the interface along the zone axis of $[10\overline{1}]_{SiCw}$; (b) SADP across the interface of SiCw and Mg with zone axis of $[10\overline{1}]_{SiCw}$ and $[0001]_{Mg}$.



Fig. 4. HREM images of the interface between SiCw and AZ91 in SiCw/AZ91 composites along $[10\overline{1}]_{SiCw}$ direction, illustrating the ORs: $(11\overline{2}0)_{Mg}//(111)_{SiCw}, [0001]_{Mg}//[10\overline{1}]_{SiCw}$.

Mg alloy solidify at different surface of SiCw, the ORs may be different at different sites.

If Mg does not nucleate at the surface of SiCw, OR does not exist, and at these parts of the interface, there only exists mechanical bond between the two phases, and no ORs can be found there.

This can be further illustrated by the in-situ TEM tensile studies of the squeeze cast SiCw/AZ91 composite. Fig. 5 shows the path of crack propagation during in-situ TEM tensile test. The cracks propagate mainly through the matrix, indicating that the bond of the interface between the matrix and the whisker is relatively strong. Occasionally, interface debonding can be observed at the interface, as shown in Fig. 6, in which case, the interfacial bond is weak, the surface of the SiC whisker does not act as heteroge-



Fig. 5. The path of crack propagation during in-situ TEM tensile of SiCw/AZ91 composites.



Fig. 6. Interface debonding at the interface of SiCw/AZ91 composites.

neous nucleation site for Mg alloy, and the matrixreinforcement interface connect only mechanically.

In addition to the ORs between SiCw and Mg, there are other factors that may have influence on nucleation of Mg on the surface of SiCw as well as interfacial bond strength, such as affinity of atoms that come into contact. The interface can be formed between Si and Mg layers or C and Mg layers in SiCw/Mg composites. Although it is difficult to determine the bond strength between Si and Mg or C and Mg, the in-situ TEM tensile results show that the bond strength between SiCw and Mg is greater than that between Mg and Mg.

4. Conclusions

(1) Two kinds of different orientation relationships at the interface were observed in squeeze cast SiCw/Mg composites. There seems to be no unique, specific orientation relationships between SiCw and Mg in squeeze cast SiCw/Mg composites. (2) The molten Mg solidifies at the different surface planes of exposed SiCw, resulting in different ORs between SiCw and Mg.

(3) The interfaces with ORs are semi-coherent interfaces and high interfacial bond strength exists in these interfaces.

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