Transmission electron microscopy study of SiC/Mg alloy interface in cast SiC particulate-reinforced Mg metal matrix composite

G. J. SHEN*, Y. CAI#, J. Z. SONG*
*Analysis and Testing Center, and #Department of Mechanical Engineering, Southeast University, Nanjing 210018, People's Republic of China

Since the mechanical properties of a metal matrix composite (MMSc) are governed to a large extent by the structures and properties of the reinforcement/matrix interface, it is necessary to characterize the interfacial structure in order to understand the mechanical behaviour of a composite and the interaction between the matrix and reinforcement during processing and service. This usually includes identification of the interfacial precipitates and/or reaction products and determination of crystallographic relationships. Therefore, examination of the interfacial precipitates and/or reaction products has attracted much attention in the study of metal matrix composites.

Several studies [1–6] have shown that, in the SiC/Al system, Al_4C_3 forms during processing at the interface. One study [7] has reported that $MgAlO_4$ was found at the interface as a reaction product after processing. Inem and Pollard [8], however, found that lamella eutectic and fine eutectic particles, $(Mg(Cu, Zn)_2$ and Mg_2Si, precipitated at the SiC/matrix interface in the as-cast magnesium–alloy–matrix composite system. Most of these phases are reaction products.

Recent electron diffraction microscopy contrast studies of the SiC/Mg alloy interface in a cast SiC particulate-reinforcement Mg metal matrix composite showed that most of the precipitates were intermetallic phase $Mg_{17}Al_{12}$ with a few of Cu_5Zn_8, with no other precipitate phase detected. These observations permit reconsideration of the reaction process in the interface between SiC and matrix during processing.

The aim of this work is to study the interfacial microstructure in a SiC/Mg system composite using transmission electron microscopy (TEM). Attention is paid to phase identification of the precipitates at the interface, as they play an important role in controlling the interfacial microstructure and mechanical properties of the composite. A formation mechanism for these precipitates will also be proposed.

AZ80 magnesium-based alloy was selected as the matrix for the composite. The reinforcement particles were high-purity silicon carbide with an average diameter of 10 μm. Composite ingots with 15 vol% SiC particles were prepared using compocasting, consisting of entrapping the reinforcement phase in a highly viscous, semisolid alloy, then remelting and mixing above the liquidus. About 1 kg composite

melt was fabricated in an electric resistance furnace using a steel crucible under Ar gas.

The as-cast composite samples were ground and polished to approximately 60 μm and subsequently argon-ion beam thinned at 5 kV, 0.4 mA, and at angles 30° and 10°. The interface between the SiC reinforcement and the magnesium alloy matrix was examined by JEOL-2000EX transmission electron microscope equipped with a double-tilt holder, operating at 160 kV.

Electron microscopical examination of the interface of the SiC/matrix alloy indicated that the most common type of interfacial structure observed consisted of an essentially featureless interface in which matrix was in intimate contact with SiC particles. There was no evidence of extensive chemical reaction at the interface.

However, two different types of particles have been observed at the interface of the SiC/matrix. Fig. 1 is a typical TEM micrograph, showing the
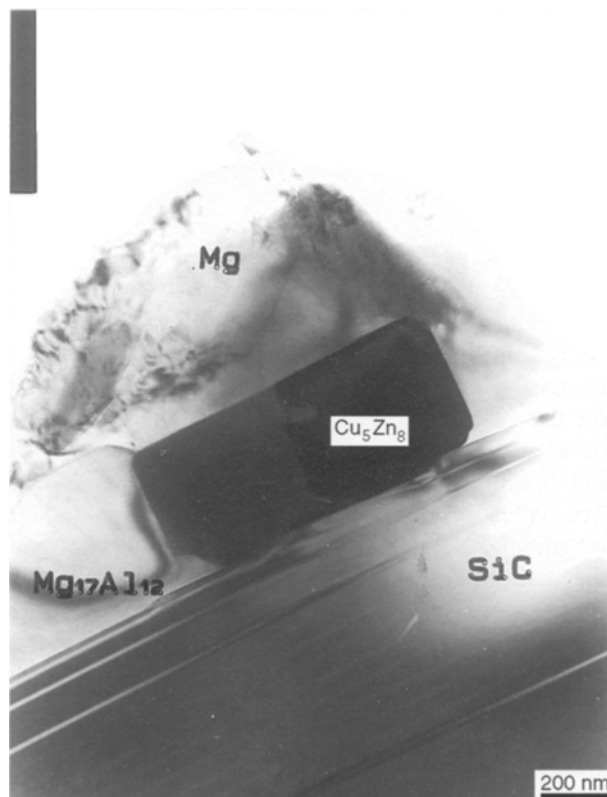

Figure 1 Micrograph showing two types of precipitates at the interface between SiC and matrix in the cast particulate-reinforced Mg metal matrix composite.

presence of both types of precipitate at a SiC/matrix interface, growing from the SiC surface into the matrix. The SiC morphology is readily revealed by the stacking faults. The structure of SiC can be identified as 6H according to its [0 1 0] zone diffraction pattern. The matrix is also indicated. Note that the two types of precipitate have different morphologies, one with a rectangular shape and the other an irregular shape.

A careful tilting experiment with these particles permits determination of the crystal structures of the precipitated particles. Figure 2a and b show a set of single crystal diffraction patterns from the rectangular shaped particle. It was found that they were basically bcc structure with a lattice constant $a = 0.88$ nm. The zone axes of the diffraction patterns are [0 0 1] and [1 1 1], respectively. The diffraction patterns from the irregular particle adjacent to the rectangular one show this crystal is also bcc crystalline phase, with a cell edge of 1.1 nm, as shown in Fig. 3a and b.

To identify the specific phase by electron diffraction, a computed simulation of the electron diffraction pattern was used. A program, DIPA, which considers that the intensity of reflection in the electron diffraction pattern is proportional to the modulus of the structure factor was developed by the author. The calculated diffraction pattern gives good agreement with experimental results. The data needed to be input are the lattice parameter and the coordinates of the atoms for various species of element in a unit cell.
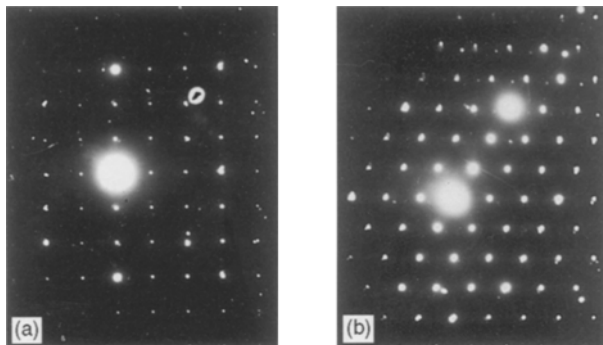
Fig. 4a and b are calculated [0 0 1] and [1 1 1] diffraction patterns which correspond to Fig. 2a and b, respectively, by inputting $D8_2$ structure type data. It is noted that the distribution of dense reflections is in good agreement with experimental results. For example, the {8 0 0} reflections are stronger than {6 0 0} in the [0 0 1] diffraction pattern. Therefore, this particle must be Cu_5Zn_8 which is $D8_2$ type, with lattice constant $a = 0.844$ nm [9].

The irregular shaped particle adjacent to the rectangular Cu_5Zn_8 can also be identified by using the same technique. The calculated [0 0 1] and [1 1 1] diffraction patterns which correspond to Fig. 3a and b are shown in Fig. 5a and b by inputting A12 structure data. In contrast to the Cu_5Zn_8 diffraction pattern, it is noted that {6 0 0} spots are stronger than {8 0 0} spots in this case. Therefore, this particle must be $Mg_{17}Al_{12}$ phase, which has A12 structure type with lattice constant $a = 1.054$ nm [9].

A fixed orientation relationship between Cu_5Zn_8 and SiC can be determined from the composite diffraction pattern. It is of the type

$$[1\,0\,0]_{Cu5Zn8}//[2\,\bar{1}\,\bar{1}\,0]_{SiC}$$
$$(0\,0\,1)_{Cu5Zn8}//(0\,0\,0\,1)_{SiC}$$

This relation can be confirmed by the fact that one of the sides of the rectangular shaped particle is

Figure 2 Electron diffraction patterns of Cu_5Zn_8 taken from the rectangular particle at the interface in Fig. 1. Zone axes are (a) [0 0 1] and (b) [1 1 1].
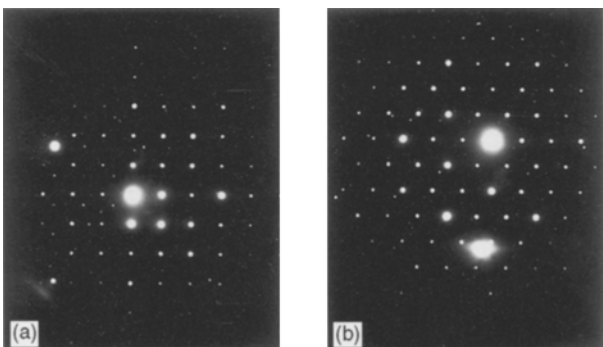

Figure 3 Electron diffraction patterns of $Mg_{17}Al_{12}$ taken from the irregular particle at the interface in Fig. 1. Zone axes are (a) [0 0 1] and (b) [1 1 1].
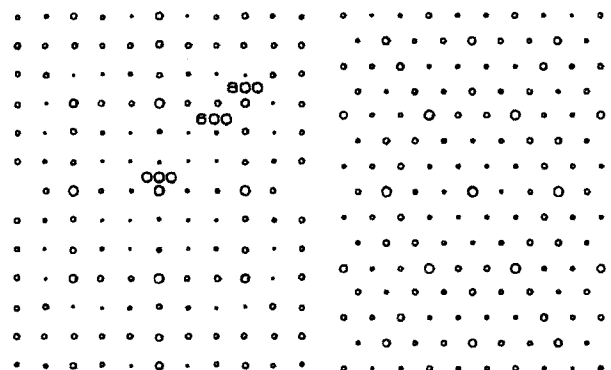

Figure 4 Computed simulation of diffraction patterns in Fig. 2. (a), (b) correspond to (a), (b) in Fig. 2, respectively. Note that the distribution of dense spots is in good agreement with the measured pattern in Fig. 2.
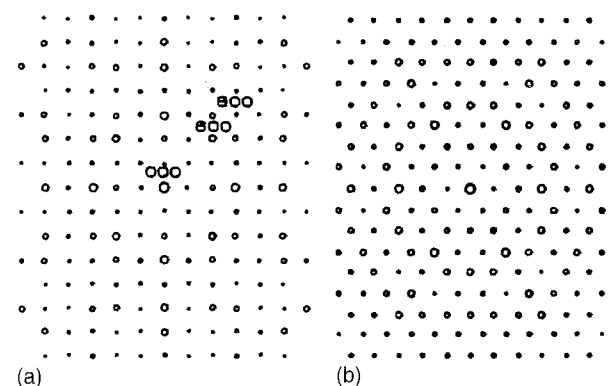

Figure 5 Computed simulation of diffraction patterns in Fig. 3. (a), (b) correspond to (a), (b) in Fig. 3, respectively. Note that the distribution of dense spots is in good agreement with the measured pattern in Fig. 3.

parallel to the stacking faults which are on the $(0\,0\,0\,1)$ plane as shown in Fig. 1.

The orientation relationship between the $Mg_{17}Al_{12}$ and the SiC was also determined to be

$$[1\,1\,1]_{Mg17Al12} // [1\,\bar{1}\,0\,1]_{SiC}$$

$$(1\,1\,0)_{Mg17Al12} // (1\,1\,\bar{2}\,0)_{SiC}$$

From first orientation relationship, we can deduce that during solidification from the melt, Cu and Zn atoms, although the Cu content is low in the alloy, have been driven to the interface to form a Cu- and Zn-rich area. According to the first orientation relationship, it is suggested that Cu and Zn atoms might migrate from matrix to the SiC boundaries and near boundary regions. This Cu and Zn agregation leads to the formation of Cu5Zn8 on the certain surface of SiC particles with the fixed orientation relationship.

From the second relationship: $Mg_{17}Al_{12}$ can also be imagined to nucleate on the surface of SiC and have the above orientation relationship with SiC, although it is known that $Mg_{17}Al_{12}$ usually precipitates in the matrix and has a crystallographic relation with it. One report [9] states that $Mg_{17}Al_{12}$ can exist in two forms in a Mg–9%Al–1%Zn–0.3%Mn alloy aged at various temperatures: continuous precipitation and discontinuous precipitation. The principal orientation relationship between the precipitate and the matrix is $[\bar{1}\,1\,1]_{ppt} // [2\,\bar{1}\,\bar{1}\,0]_{Matrix}$ and $(0\,\bar{1}\,1)_{ppt} // (0\,0\,0\,1)_{Matrix}$, but small quantity of precipitate displays a relationship of the type $[2\,\bar{1}\,1]_{ppt} // [2\,\bar{1}\,\bar{1}\,0]_{Matrix}$ and $(1\text{-}1\,\bar{1})_{ppt} // (0\,0\,0\,1)_{Matrix}$ for continuous precipitation. For discontinuous precipitation, there are two groups of precipitates; lamellae having the same orientation relationship as the major continuous precipitate, and precipitates displaying no defined orientation relationship with the matrix. Both precipitation and their orientation relationship with matrix can also be observed in our specimen after long time ageing as shown in Fig. 6. This indicates that the surface of SiC should be a priority site for $Mg_{17}Al_{12}$ precipitation during solidification from melt.

Since these precipitates contain no oxygen and silicon which might come from SiC and some oxides, and they cannot be treated as reaction products at the interface, no interaction between matrix and reinforcement has taken place during solidification in the early stage in this cast SiC particulate-reinforced Mg metal matrix composite.
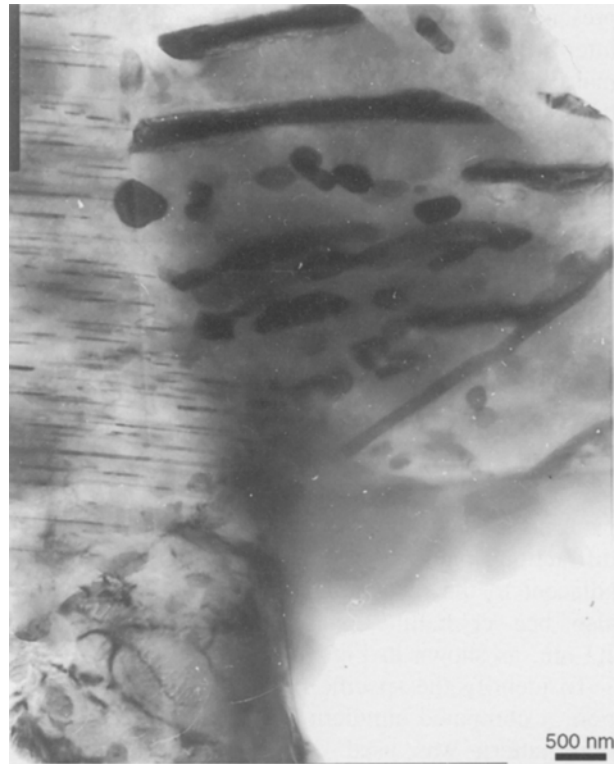

Figure 6 Micrograph showing the precipitated continuous and discontinuous $Mg_{17}Al_{12}$ in the matrix.

References

1. V. LAURENT, D. CHATAIN, N. EUSTATHOPOULOS and X. DUMANT, in *Proceedings of the International Symposium on Advance in Cast Reinforced Metal Composites*, Chicago, IL, Sept. 24–30, 1988, edited by S. G Fisherman and A. K. Dhingra (ASM International, Metals Park, OH, 1988) pp. 27–31.
2. R. WARREN and C. H. ANDERSON, *Composites* **15** (1984) 101.
3. T. ISEKI, T. KAMEDA and T. MARUYAMMA, *J. Mater. Sci.* **19** (1984) 1692.
4. K. KANNIKESWARAN and R. Y. LIN, *J. Met.* **39** (1987) 17.
5. W. C. MOSHIER, J. S. AHEARN and D. C. COOKE, *J. Mater. Sci.* **22** (1987) 115.
6. D. J. LOLYD, H. LAGACE, A. McLEOD and P. L. MORRIS, *Mater. Sci. Engng* **A107** (1989) 73.
7. N. WANG, Z. WANG and G. C. WEATHERLY, *Metall. Trans. A* **23** (1992) 1423.
8. B. INEM and G. POLLARD, *J. Mater. Sci.* **28** (1993) 4427.
9. E. A. BRANDES, "Smithell's metals reference book, 6th edn (Butterworth, London, 1983), pp. 6–11, 13.
10. A. F. CRAWLEY and K. S. MILLIKEN, *Acta Metall.* **22** (1974) 557.