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Interface structure and fractography of a magnesium-alloy, metal-matrix composite reinforced with SiC particles

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The interfacial structure in SiC-particle-reinforced, as-cast and heat-treated magnesium-alloy-matrix composites was investigated using analytical electron microscopy. No extensive chemical reactions were observed between the magnesium and the SiC particles or the SiC and the eutectic phase. However, most of the eutectic phase appeared to nucleate at the surface of the SiC particles. In addition to the lamellar eutectic, a fine eutectic and Mg_2Si particles have been identified at the SiC surface using nanoprobe micro-analysis. As with the aluminium base composite, precipitation was observed to take place on dislocations, and dense precipitation was found to occur in the stress fields around the SiC particles. Examination of the fracture surface indicated that the bonding between the SiC/eutectic is stronger than between the SiC/magnesium matrix. Intergranular cracks have been observed both in the fracture surface and also in a polished and etched section. The fracture surface tends to exhibit a more brittle morphology in the composite than is observed in the alloy.

1. Introduction

The properties of a metal-matrix composite (MMC) depend upon the properties of the reinforcement phase, of the matrix and of the interface. A strong interface bonding without any degradation of the reinforcing phase is one of the prime objectives in the development of MMCs. In order to achieve a strong bond between the matrix and the reinforcement, several processing procedures such as using a short consolidation time, minimum possible process temperature, extra alloy element addition, and chemical surface treatment of the reinforcement have been applied. Often degradation of the reinforcement or an unexpected phase formation have been experienced at the interface in different composite systems. In other cases, the formation of small intermetallic phases or the development of a reaction zone provided bonding [1–5], as with the graphite/magnesium cast composite studied in [6–8]. It was observed that Mg_2Si and MgO phases formed due to chemical bonding between the matrix and the SiO_2 fibre coating. However, even without chemical reaction, diffusion of atoms from one component to the other and good wettability of a liquid phase can provide mechanical and physical interactions at the interface, allowing the development of a strong bond.

Additionally, interface regions are characterized by a high dislocation density which is often attributed to the differential thermal contraction which takes place between the matrix and the reinforcement. This contributes to a residual stress and extensive twinning.

The objective of this study is to characterize the interface structure of a magnesium/SiC particle composite by transmission and scanning electron microscopy (TEM and SEM).

2. Experimental procedure

The material used for this study was a magnesium alloy with nominal composition Mg–6%Zn, 3%Cu, 0.5%Mn (ZCM630), provided by Magnesium Elektron Limited (MEL), UK. The alloy was reinforced with either 10 or 15 vol. % SiC particles, with an average diameter of 10 μm. The composite was cast in the form of plates 250 × 250 × 25 mm.

For optical and scanning electron microscopy, the samples were ground through a 1200 grit SiC abrasive and polished on a soft cloth using 6, 3, and 1 μm diamond pastes and then etched. After fracturing, the samples were mounted on aluminium stubs and placed directly in the scanning electron microscope for examination.

ZCM630 composite samples in the as-cast, solution-heat-treated and aged condition were examined using both a Joel 200 CX and a 2010 transmission electron microscope at 200 kV Foil preparation was as follows: after electrodischarge sectioning, the 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°, 15° and 6°. The microstructure was characterized using bright-field and dark-field images, selected-area diffraction (SAD) and nanoprobe energy X-ray spectroscopy (EDX) for microanalysis.

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3. Results and discussion

3.1. Characterization of the microstructure

Microscopical examination reveals that during the solidification process, SiC particles are pushed by the growing primary magnesium grains into interdendritic eutectic regions in which the eutectic formed from the Cu-and-Zn-rich melt having a composition of $Mg(CuZn)_2$ (Fig. 1). It has been observed that the lamellar eutectic tends to nucleate at the particle surfaces. The grain boundaries are decorated with a dense array of SiC particles which are rejected by the primary magnesium grains of the matrix. However, some of the SiC particles are captured within the magnesium grains during the solidification. The difficulty in mixing the SiC particles with molten metal and gas absorption during the composite consolidation causes the particles to agglomerate, and pores were observed to be associated with the particles. Clustering of particles with oxides was also observed around the grain-boundary regions.

Trapping of particles in the grain boundaries terminates the further growth of primary magnesium grains and causes a find grain structure to form. The SiC particles increased the nuclei density in the liquid magnesium by providing many possible sites on the particle surfaces.

3.2. Identification of interfaces

The most common type of interfacial structure observed is as shown in Fig. 2 and consists of an essentially featureless interface in which matrix and eutectic are in intimate contact with the particles. There was no evidence of extensive chemical reaction at the interface.

However, two different particles at the interface of matrix/SiC have been detected [9] and analysed using nanoprobe (EDX) analysis, Fig. 3. One of the particles consists of small eutectic droplets with a size 30–500 nm. This type of eutectic droplet is often apparent on the particle surface at the matrix/SiC interface. It would be the last eutectic liquid present near to the particles and would solidify at the particle surface. The other fine particle was a precipitate analysed as Mg_2Si, having a rectangular shape 10–400 nm in size. In order to characterize them, a 1 nm probe was placed at the interface where there was no precipitate, an analysis carried out and the same size probe was then placed on the precipitate and a further analysis carried out. The results were compared. The difference in X-ray peaks indicates that the precipitates are most likely to be Mg_2Si. Due to the size of the precipitates, difficulty in analysis was experienced. The same phase formation in δ-Al_2O_3/Al–Mg composites with a SiO_2 binder has been reported by other workers [10].

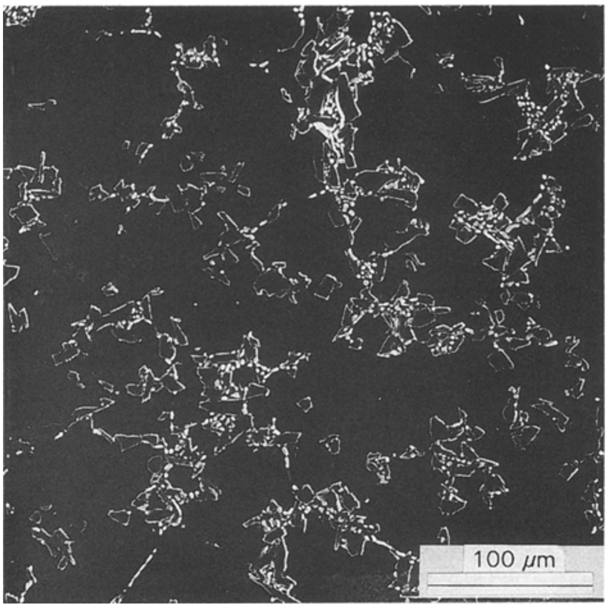

Figure 1 A micrograph showing the microstructure of the ZCM630/SiC composite.

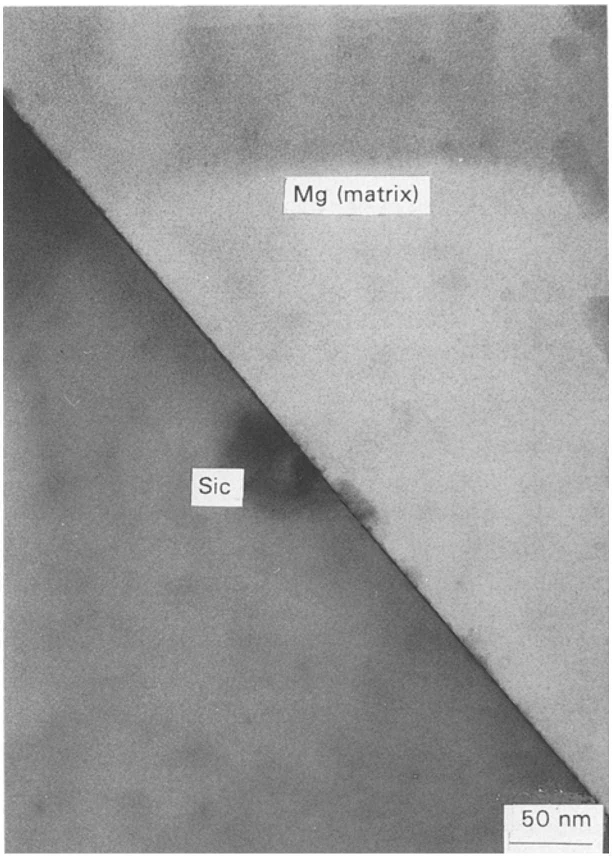

Figure 2 TEM micrograph showing the matrix/SiC interface with fine precipitates.

Since the base alloy does not contain Si, it is believed that during the consolidation, free Si or SiO_2 from the SiC particles together with Mg forms Mg_2Si at the matrix/SiC interface. There are basically two possible reactions for formation of a Mg_2Si phase at the interface

$$2Mg + Si \rightarrow Mg_2Si \quad (1)$$

$$4Mg + SiO_2 \rightarrow Mg_2Si + 2MgO \quad (2)$$

In the case of Reaction 2, MgO is the other possible reaction product in the composites. However, MgO has not been observed at the interface. Mg_2Si particles, 20–30 μm in size, adjacent to SiC particles have also been detected by SEM, but an attempt to obtain a

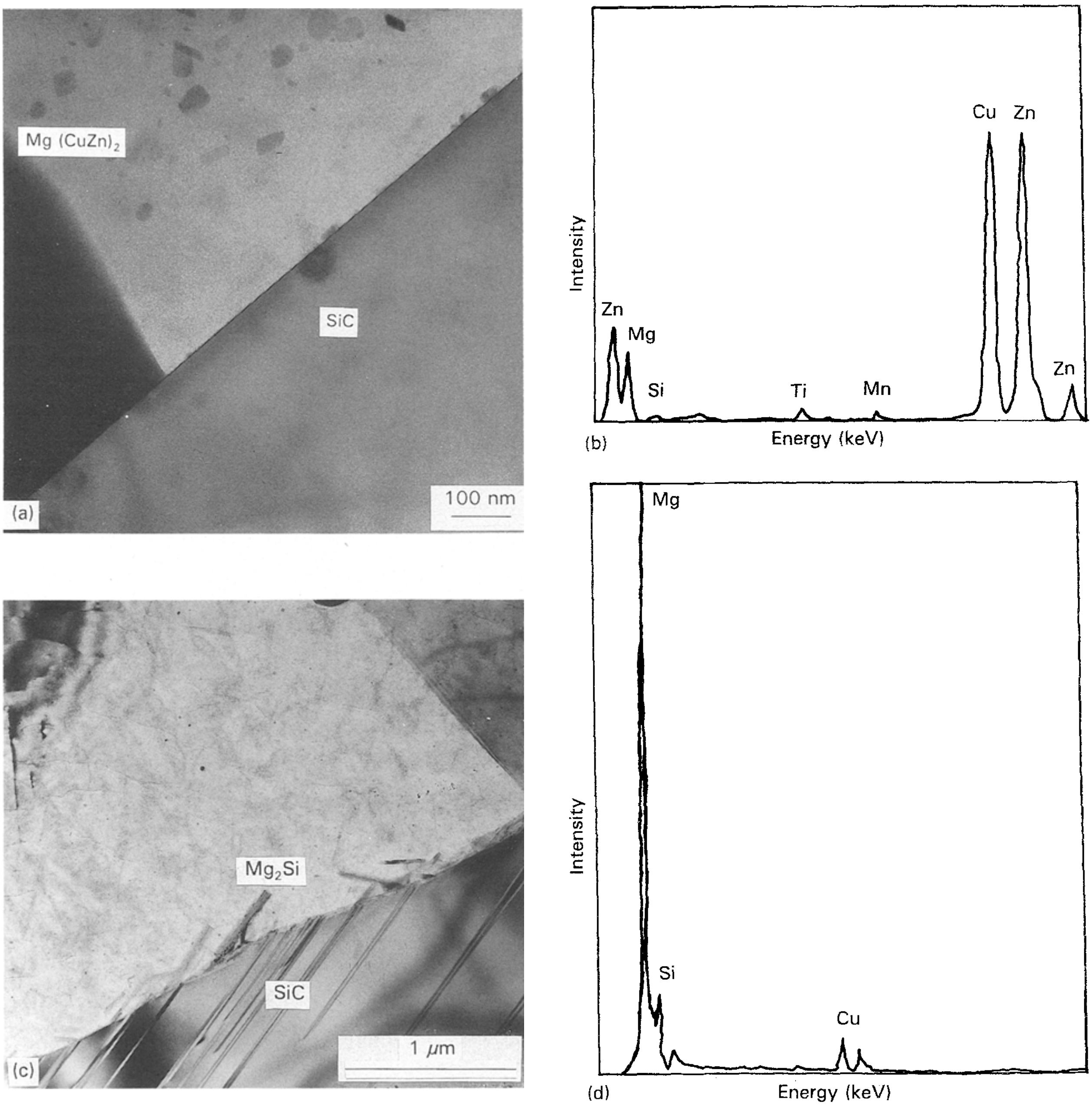

Figure 3 (a) TEM micrograph showing eutectic droplets and massive eutectic phase on the SiC particle, (b) EDX analysis from the eutectic phase, (c) Mg_2Si phase on the SiC particle at the interface, and (d) EDX analysis from Mg_2Si phase.

nanoprobe diffraction pattern from Mg_2Si was not successful.

As with the matrix/SiC interface, the eutectic/SiC interface was smooth (Fig. 4). No chemical degradation or intermetallic phases were observed. However, voids 5–30 nm in size were detected at the interface. A 5 nm probe was placed in one of the small voids and an analysis performed. No characteristic peaks other than Mg, Cu, Zn, which were obviously from the eutectic, were detected; this indicates that the area was eutectic with a microvoid at the interface. A considerable stress field existed at the interface which could develop as a result of the contraction between the eutectic and SiC, similar to that observed at the matrix/SiC interface.

TEM showed (Fig. 4) a pronounced nucleation of the eutectic phase at the SiC particle surface. The process is helped by the fact that the wetting angle of the eutectic on the surface of SiC is less than 90°. TEM observation also indicated that wetting took place between the eutectic and the SiC particles. Cu was also detected at the surface of the SiC particles (Fig. 3d). It should be borne in mind that the presence of Cu atoms at the surface might aid the nucleation of the Cu-rich $Mg(CuZn)_2$ eutectic phase, since it has been reported [11] that Cu can form Cu_3Si with free Si in the SiC.

3.3. Dislocation substructure

In MMCs, the large difference between the coefficient of thermal expansion (CTE) of the matrix and reinforcement resulted in sufficient stress to generate a high dislocation density at the interface during the solidification and heat treatment. This high stress and consequent dislocation generation around the reinfor-

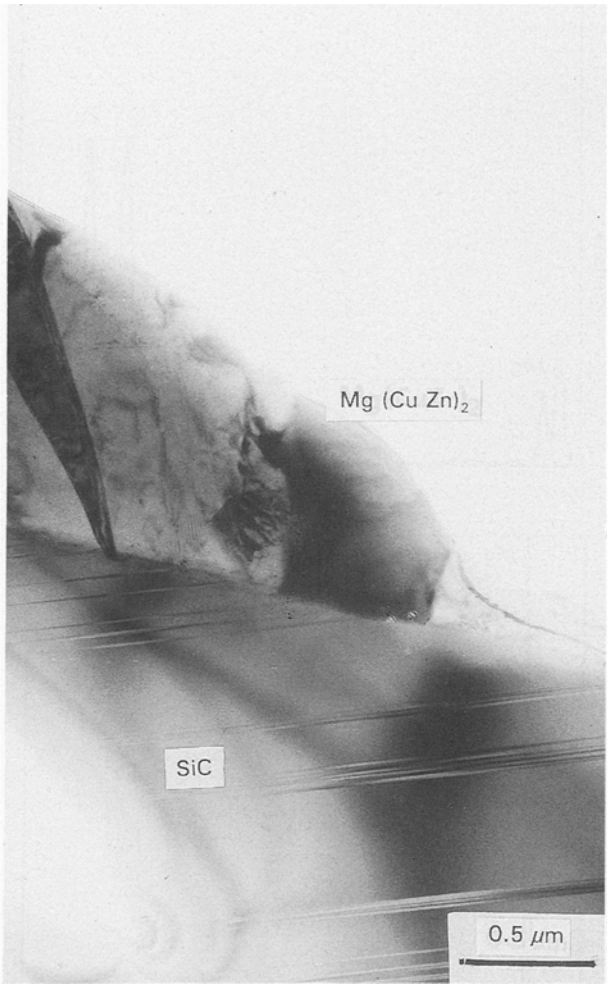

Figure 4 TEM micrograph showing the interface of the SiC/eutectic and nucleation of the eutectic from the SiC particles.

Figure 5 A high dislocation density and heavy twinning around the SiC particles.

cement has often been related to good bonding at the interface [12, 13].

As with other composite systems, a high dislocation density and extensive twinning around the particles has been observed in magnesium-matrix composites with 10% and 15% of SiC particles (Fig. 5). This dislocation substructure and stress field around the particles gives rise to preferential nucleation for precipitation and an accelerated precipitation response. This results in early and rapid ageing of the composite, which has been measured in a previous study [14].

4. Fractography

4.1. Fracture and bonding at the interface

The fracture surfaces of the as-cast and heat-treated composites were microscopically rough with a typical height variation of ~2 mm.

Fig. 6a and b shows the fracture surfaces of as-cast and heat-treated composite samples. The main features of the fracture surface were locally concentrated SiC particles, agglomerates of SiC particles (which were eutectic in the form of the lamellae in the as-cast condition, and spheroidized in the heat treated condition) firmly attached to the SiC particles. Debonding of the particle/matrix interface, cracks on the particles and intergranular cracks in the matrix were also present.

The most interesting feature of the fracture surface was the relationship between the SiC particles and the eutectic phase (Fig. 6a). The deposition of eutectic at the particles' surface indicates some degree of bonding between the SiC particles and the eutectic. The eutectic/SiC bonding is suggested to be stronger than the bonding between the matrix/SiC, otherwise the eutectic would have remained associated with the matrix. The lamellar eutectic at the particle surface could act as a binder at the matrix/SiC interface.

The next type of structure away from the interface was a magnesium layer which covered the particle surface (Fig. 7). The layer has a direct mechanical and physical bonding with the particles. Mg_2Si and small eutectic particles, which were detected at the interface by TEM, may provide a further mechanical bonding.

The other feature was the presence of a crack network (Fig. 8). Intergranular cracks have been observed in the composite, the crack lengths varying between 30 and 100 μm. It is proposed that they were formed by hot tearing, which was observed on the etched surface and which may have been introduced during the solidification. A possible mechanism for the cracking sequence is as follows. During the solidi-

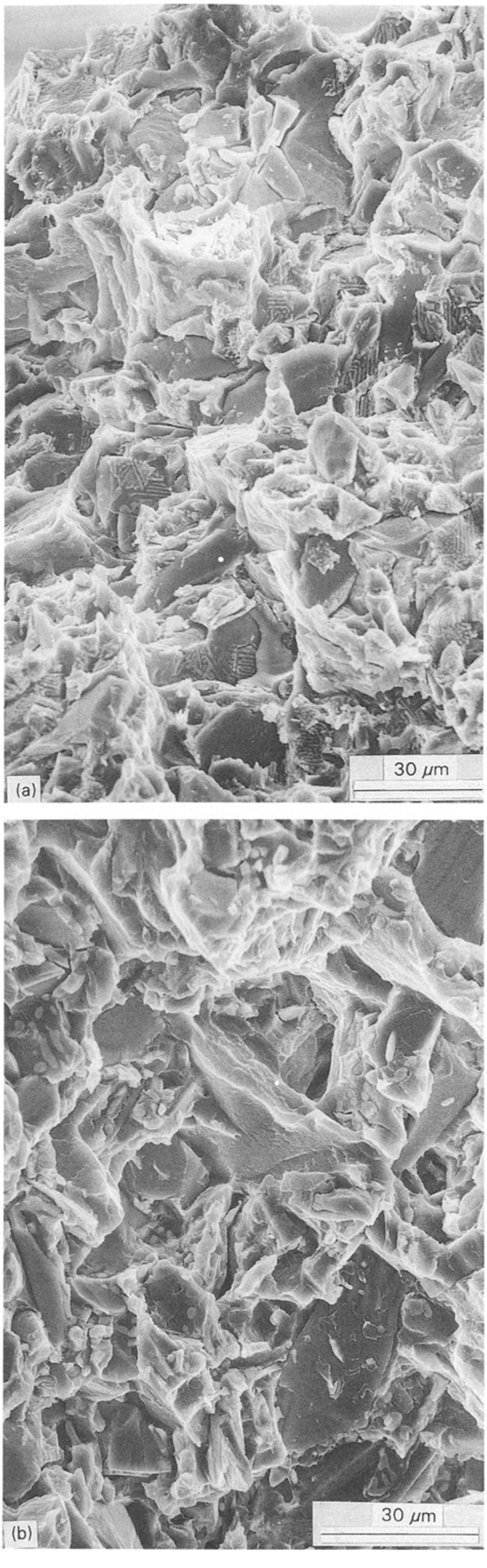

Figure 6 Fracture surface of composite; (a) as-cast condition and (b) heat treated.

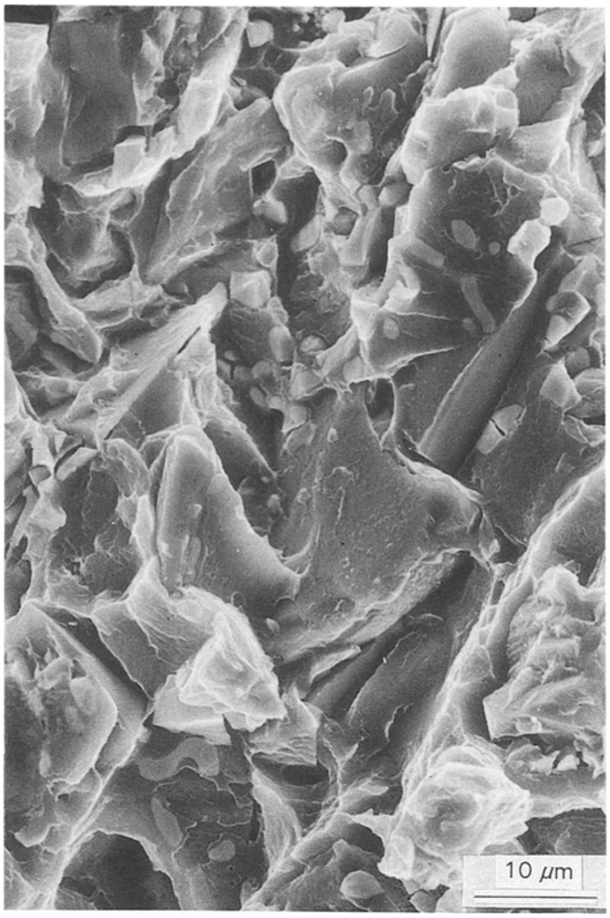

Figure 7 A thin magnesium layer on the SiC particle surface.

fication process, while primary magnesium grains grow, the SiC particles are pushed ahead of the solidification front. During the impingement with the other growing grains these particles coalesce, which limits the relative movement of grains and dendrite arms. This might then restrict the relative movement of both the grains and the viscous liquid phase and, as a result, strains develop during the subsequent cooling. When a critical breaking or tearing stress has then been exceeded, a rift would occur in the network. If there is insufficient liquid remaining to heal the rift, it will then develop until a tear, and the newly formed crack, could then grow to alleviate the shrinkage.

Agglomeration and clustering of particles clearly occurred, and these phenomena can be seen on the fracture surface (Fig. 9). Such particles are indicated in the middle of the agglomerated particle (A, in Fig. 9); the surface appeared to be smooth with no trace of the matrix or eutectic phase. This indicates that during the consolidation absorbed gas may fill the cavities between the particles and hence prevent sufficient infiltration of liquid metal. Similar results have been reported previously [15]. The clustering, agglomeration and intergranular crack formation may play a significant role in the fracture-initiation process. This phenomena has been examined by Hunt *et al.* [16] and reviewed more recently by Lloyd [17].

Microcracking at the matrix and eutectic interface has occasionally been observed by TEM (Fig. 10). Both TEM foil (Fig. 11a) and SEM fracture-surface

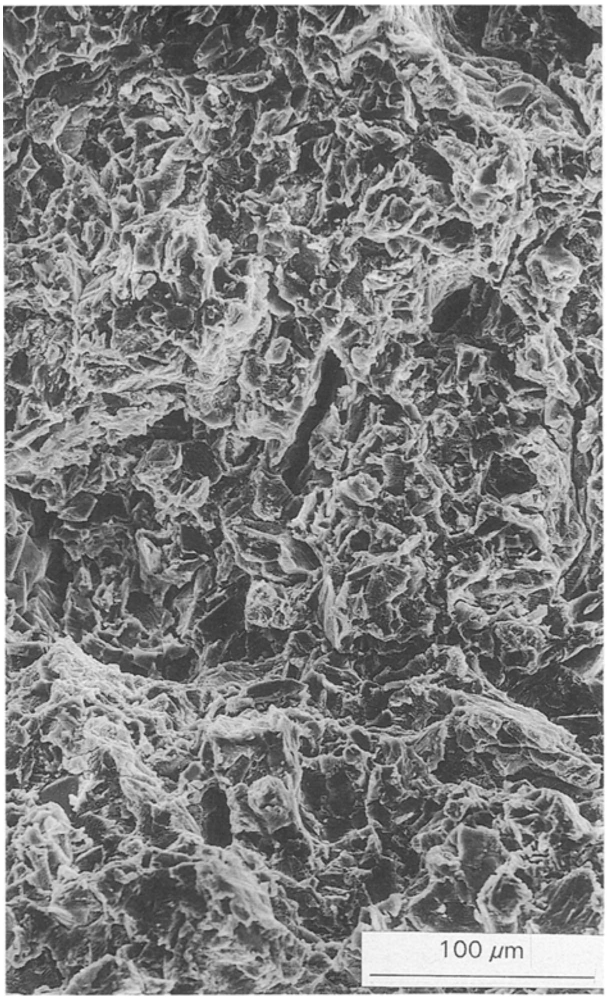

Figure 8 A micrograph of the fracture surface of ZCM630 + 15% SiC particle composite showing the intergranular crack networks.

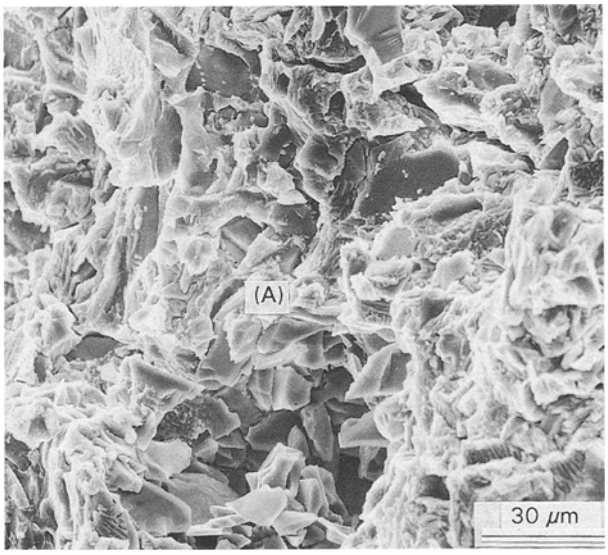

Figure 9 SEM micrograph showing a clean particle surface in the agglomerated region.

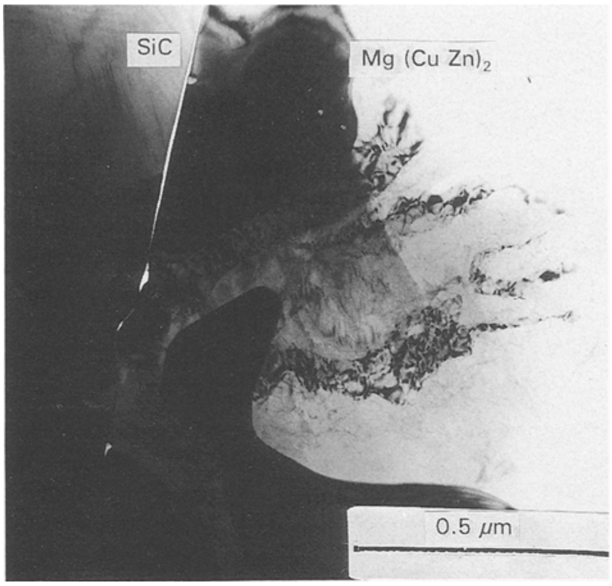

Figure 10 TEM micrograph showing microcracks present at the SiC/eutectic interface.

studies (Fig. 11b) also showed that cracking took place in the SiC particles. The cracking which takes place at the interface and on the particles could be caused by the thermal shock which occurs during solidification or quenching. It is likely that during the SiC particles preparation some crack could be introduced, and the crack caused by such a thermal shock could give rise to particle cracking. An alternative explanation is that particle cracking would have indicated the existence of very strong bonding at the interface, which, due to the nature of the interface, is not likely to be present.

4.2. Fracture morphology

From the fracture-surface study it has been observed that two types of fracture occurred in the composite. In the regions in which the matrix phase was dominant, ductile fracture of the matrix takes place in which void nucleation could play an important role. The regions in which the local concentration of SiC particles is high exhibit brittle fracture. The composite tends to demonstrate a more brittle fracture morphology than the matrix alloy. Fracture of the composite is initiated by debonding at the interface of the matrix and SiC. This would indicate that the overall volume fraction of reinforcement, and the distribution of particles, plays an important role in determining the morphology and mechanism of fracture of these composites.

5. Conclusions

1. The interface structure, with which the matrix and eutectic are in intimate contact with SiC particles, is essentially featureless, there was no evidence of extensive chemical reaction or degradation of reinforcement.

2. Fine eutectic particles and Mg_2Si were detected at the matrix/SiC interface. Microvoids were observed at the eutectic/SiC interface. Some SiC particles appeared to be partly covered by a thin layer of magnesium.

3. The eutectic phase tends to nucleate at the interface of the SiC particles surface. Good wettability of

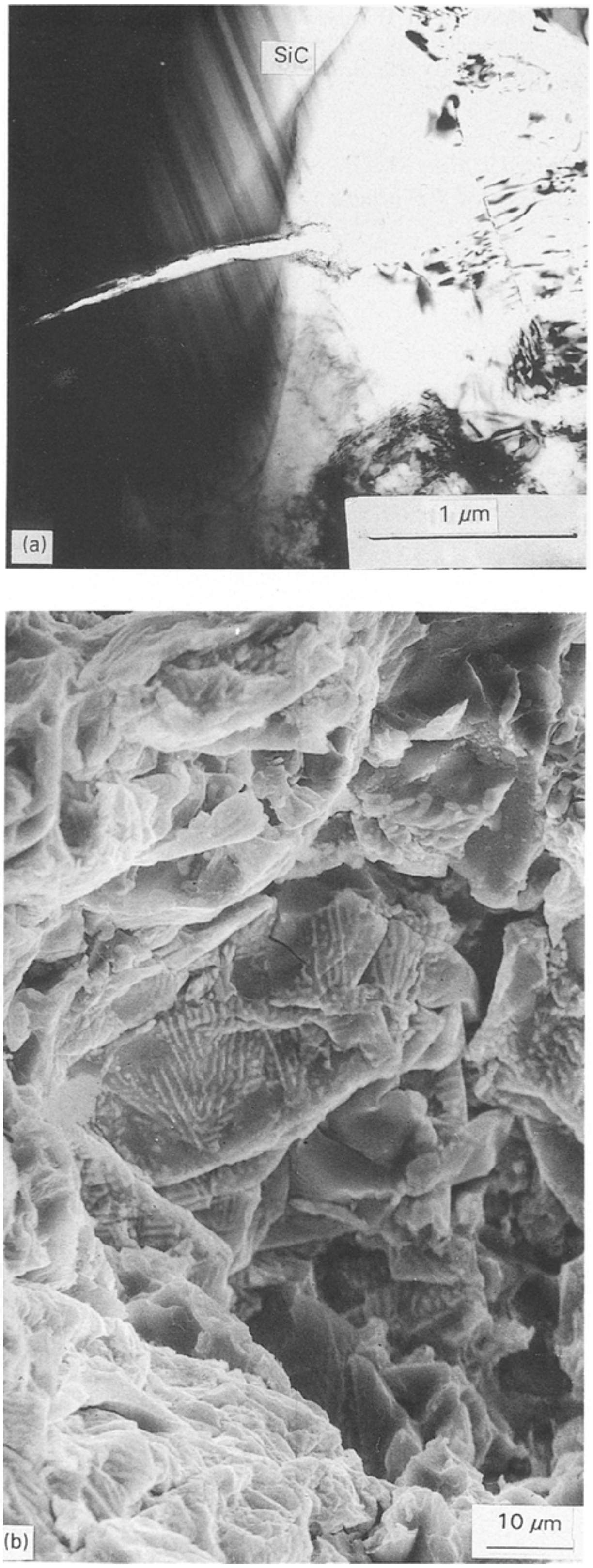

Figure 11 (a) TEM micrograph showing particle cracking in the composite, and (b) SEM micrograph showing particle cracking in the composite.

the eutectic and the presence of Cu at the SiC surface could give rise to the nucleation site of the eutectic.

4. The large difference between the CTE of the matrix and the reinforcement particles generates sufficient stress to form a high density of dislocations and twinning at the interface. This stress field, the resultant dislocation distribution, and twin substructure at the interface, give rise to preferential nucleation sites for precipitation. As a result, fine precipitation occurs around the SiC particles in the stress fields.

5. At the fracture surface an intergranular crack network occurs.

6. Occasionally interfacial cracking between the matrix/SiC and eutectic/SiC and particle cracking was observed.

7. In the composite, the fracture surface tends to exhibit a more brittle morphology than the matrix.

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