

Development of Particulate Treatments and Coatings to Reduce SiC Degradation by Liquid Aluminum

M. Suéry, G. L'Espérance, B.D. Hong, L. Nguyen Thanh, and F. Bordeaux

SiC-particulate-reinforced aluminum alloys are potentially attractive materials for structural applications. Among the fabrication possibilities, the molten metal mixing technique is one of the most promising in terms of cost and production capacity. Degradation of SiC by chemical reaction with molten aluminum is a problem that could be, at least partially, overcome by treatments or coatings of the reinforcements. This article describes the various techniques that have been used to coat particles and presents results obtained during remelting of composites. Prior oxidation of particles, oxide coating by sol-gel, or dry mixing techniques are thus presented. Oxidation of particles and TiO₂ coatings slow the degradation of SiC. Analytical electron microscopy was used to characterize the interfacial reactions that occurred during fabrication and remelting of composites. Possible mechanisms of protection are then presented and discussed.

Keywords

composites, metal matrix, SiC reinforcement

1. Introduction

SiC-PARTICULATE-REINFORCED aluminum composites are attractive for many structural applications and have been studied extensively because they exhibit unusual combinations of mechanical, physical, and thermal properties. Use of these materials, however, also has several drawbacks, particularly low room temperature tensile ductility and poor toughness. Although the causes for low ductility and toughness are believed to be related to several compositional and processing factors, the microstructure of the interface region is an essential parameter.

Because many methods used to manufacture composites involve contact between liquid Al and SiC at some stage of fabrication or processing, one potential problem is the degradation of SiC by liquid aluminum,^[1,2] which leads to the formation of Al₄C₃ according to the reaction:



This phenomenon has several detrimental effects such as reducing the reinforcement and interfacial strength and increasing the corrosion susceptibility of the material. It is then of interest to develop methods to suppress or at least reduce degradation of the SiC particles. A change in the matrix composition by addition of Si is the simplest technique.^[3,4] However, it is applicable to foundry alloys only, due to the large concentration of Si needed to avoid the reaction using current foundry practices.

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Semisolid processing of composites both during fabrication and subsequent forming operations is another possibility. Whereas incorporation of SiC particles into a semisolid alloy is commonplace, with the further advantage of producing homogeneous materials, forming in the semisolid state is still under study.

The third possibility is to treat or coat SiC particles to reduce their degradation susceptibility when in contact with molten aluminum. Several methods involving the presence of oxides around the SiC particles have been proposed recently; these are oxidation of the particles,^[5,6] production of oxide coatings by the sol-gel technique,^[7-9] and deposition of oxide particulates at the surface of the SiC particles by a dry mixing technique.^[10]

The aim of this article is to discuss these various methods and to present the results obtained by the current study. Particular attention will, however, be devoted to the composites produced with particles coated by the dry mixing technique. Transmission electron microscopy (TEM) observations of the particle matrix interfaces will be presented, and the possible mechanisms by which protection of the particles takes place will be discussed.

2. Oxidation of SiC Particles

Heating of SiC particles for sufficient times at high temperature ($\approx 1100^\circ\text{C}$) leads to the formation of a SiO₂ layer around the particles. This layer is, however, not of uniform thickness, so that wet analysis is preferable to characterize the amount of SiO₂ present at the surface of the particles.^[11] Fabrication of composites based on Al-Si-Mg alloys with oxidized particles was performed, and the matrix/particle interfaces were characterized by extensive TEM. This investigation showed that, during fabrication of the composites by compocasting, the SiO₂ layer reacts with the Al(Mg) melt, leading to the formation of a polycrystalline layer consisting mainly of blocky spinel (MgAl₂O₄) crystals with Al-rich areas and Mg₂Si particles.^[12] For high-Mg content alloys, fine MgO crystals are more likely to be formed than MgAl₂O₄.^[13] A

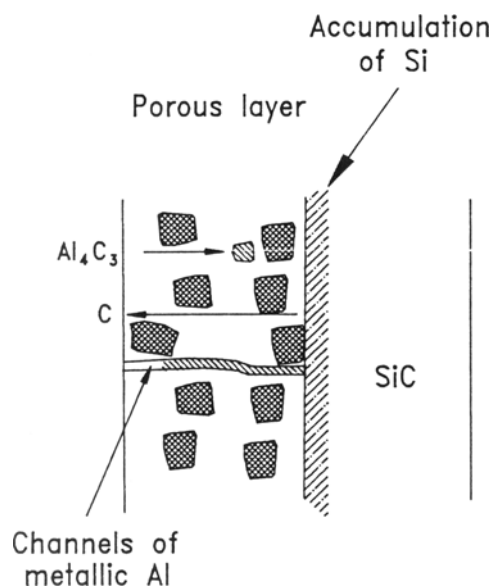


Fig. 1 Proposed mechanism of the protective effect of the oxide layer against attack of SiC by liquid aluminum.

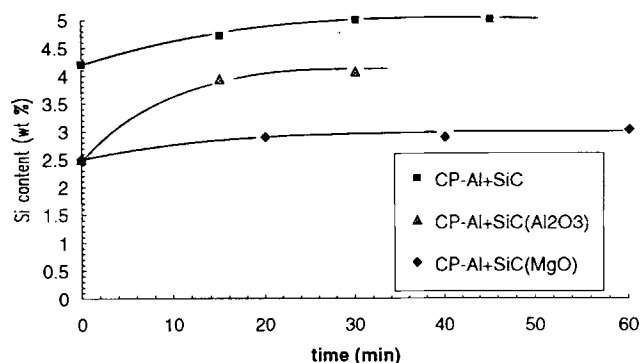


Fig. 2 Silicon content in commercially pure aluminum as a function of exposure time to the melt at 717 °C of coated and uncoated SiC particles. The Si content at time zero corresponds to that measured after fabrication of the composite (From Ref 7).

complex oxide layer thus covers the SiC particles in the as-cast condition.

Remelting of the composites was carried out at 800 °C with the aim at evaluating the role of this oxide layer as a possible barrier against liquid aluminum attack. Observations then revealed that the oxide layer was relatively stable at this temperature and that less degradation of the SiC particles occurred compared to composites reinforced with as-received particles, as shown by the lower Si content released in the matrix.^[5,6]

The only major change in the microstructure was the accumulation of Si at the oxide/SiC interface.^[6] Quantitative energy-dispersive X-ray spectroscopy (EDS) analysis shows that the Si concentration can be as high as 20% in this zone, which tends to suppress SiC degradation. This observation was carried out for Al-1% Mg alloys reinforced with heavily oxidized

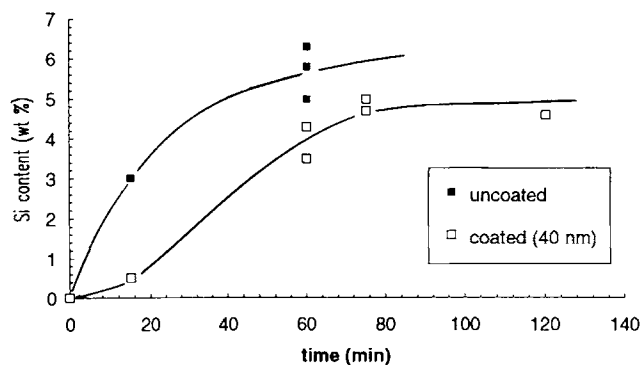


Fig. 3 Silicon content in aluminum matrix as a function of exposure time at 750 °C for Al-10% SiC_w containing uncoated and coated (40nm) SiC whiskers (From Ref 8).

particles, for which the interfacial reaction zone consisted mainly of large MgAl₂O₄ crystals that did not form a fully dense layer, in that the transformation from SiO₂ to MgAl₂O₄ was accompanied by a 27% contraction. For particles with low oxidation levels, the layer is also thinner so that protection is likely to be less effective. In the case of high-Mg content alloys, such as AA5083, the MgO layer is more compact due to the fact that less contraction occurs during the SiO₂ → MgO transformation (13.6%) compared to the SiO₂ → MgAl₂O₄ reaction (27%). Recent experiments using this alloy reinforced with slightly oxidized SiC particles (3%) revealed that protection of the particles was quite effective, although some particles exhibited evidence of severe degradation.^[14] This is probably due to the nonuniformity of the oxidation of the particles, which is particularly obvious for low oxidation levels.

The following mechanism for the protection of the particles is suggested (Fig. 1). The porous oxide layer is permeable to the liquid Al, which can then attack SiC. The reaction leads to an enrichment of Si at the SiC/oxide interface, which slows the degradation. Al diffusion to the particle and Si diffusion from the particle then control the process.

3. Sol-Gel Technique

The sol-gel technique is another process that has been shown to be effective producing oxide-coated SiC particles as well as other reinforcements.^[15] This process uses an alkoxide or mixture of alkoxides dissolved in a solvent. This liquid is used to coat the particles or the fibers, and then it undergoes a chemical reaction to form the final product.

This technique was studied recently by Teng and Boyd.^[7] Al₂O₃ and MgO coatings were produced on SiC particles used as reinforcements for commercially pure aluminum (CP Al). The coating was on the order of 50 nm thick. The composites were fabricated by liquid metal infiltration with a liquid metal temperature of 750 °C. They were subsequently isothermally treated at 717 °C for various times to determine the influence of the coating on the rate of degradation of the particles by liquid aluminum. For this determination, the authors measured the change in the liquidus temperature of the alloy formed by the release of Si in the melt. From this change, it is then possible to

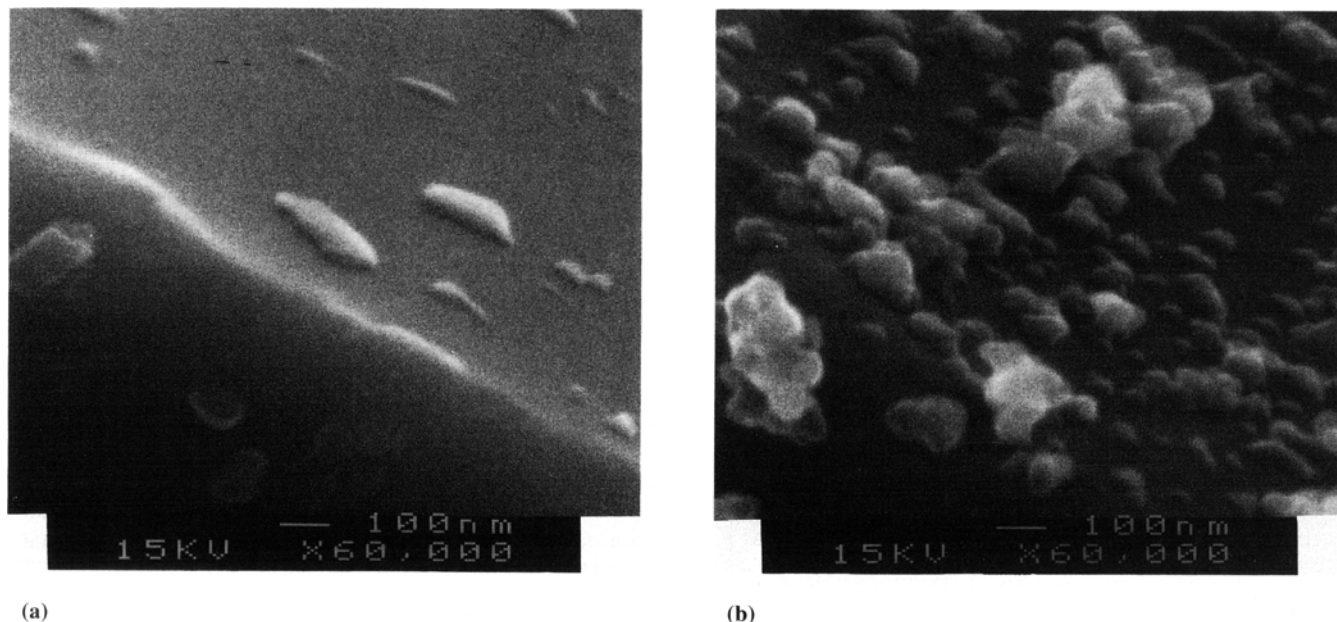


Fig. 4 Scanning electron micrographs of the surface of SiC particles. (a) Uncoated. (b) Coated with 0.3 vol% TiO₂.

derive the Si content of the liquid. Figure 2 shows a plot of the Si content as a function of exposure time at 717 °C for uncoated and coated particles. For the calculation, the liquidus and binary eutectic Al-Si temperatures were taken as 654 and 568 °C, as measured by the authors. The results of Fig. 2 show that there is a significant reaction between the particles and the liquid aluminum during infiltration that continues during isothermal treatments at 717 °C, but ceases after about 30 min exposure to liquid metal. Coating of the SiC particles by Al₂O₃ and MgO thus significantly reduces the rate of reaction, MgO being more effective than Al₂O₃. The mechanism proposed previously to explain such a reduction probably also applies here, the accumulation of Si at the particle/oxide interface being the likely cause of the slowing of the reaction.

The same type of results was obtained using SiC whiskers coated with 40-nm thick Al₂O₃.^[8,9] The composites made with commercially pure aluminum were produced by powder metallurgy. Figure 3 shows the variation in Si content in the aluminum matrix as a function of exposure time at 750 °C for composites containing 10% uncoated and coated SiC whiskers. The curve for the coated whiskers indicates that an incubation time may exist for the attack of SiC_w by Al, which could be explained by the low diffusivity of Al and Si through the Al₂O₃ coating at the beginning of the treatment. A further advantage of the coating is an improvement in the distribution homogeneity of the whiskers in the Al matrix, which improves mechanical properties.

The previous results demonstrate that the sol-gel technique is efficient to coat SiC particles, the coating slowing their degradation by liquid aluminum. However, the reactions are still significant for relatively long isothermal treatments in the liquid state. Note, moreover, that the results were obtained with Al alloys that do not contain a significant amount of Mg. In the presence of Mg, Al₂O₃ will rapidly convert into MgAl₂O₄ or

MgO depending on the Mg content of the alloy, and this transformation will probably influence the rate of degradation of SiC. As already mentioned, restricted diffusion of Al and Si through the oxide layer probably accounts for the slower rate of degradation at the beginning of the process.

4. Dry Mixing Technique

The dry mixing technique is a process in which deposition of fine oxide particles at the surface of the SiC particles is accomplished by dry mixing the two types of particles. With this technique, a discontinuous coating can be produced with volume fractions of oxide particles on the order of a few tenths of a percent.^[10] Figure 4 shows typical scanning electron microscope (SEM) micrographs of the surface of an uncoated SiC particle (Fig. 4a) and a particle coated with 0.3 vol% TiO₂ (Fig. 4b), each TiO₂ particle being about 40 nm in diameter. Figure 4(b) reveals that the oxide particles can agglomerate at the surface to form clusters of about 100 nm in size. Increasing the volume fraction of oxides does not lead to uniform coating of SiC. Large agglomerates are then found that do not fully cover the surface of the SiC particles (Fig. 5).

Various oxide coatings were produced using this technique on 30-μm diameter particles, including TiO₂, Al₂O₃, and SiO₂ treated to be hydrophobic or hydrophilic. They were provided by Lonza, Germany; 10 vol% SiC particles were introduced in Al-1%Mg alloy via a semisolid route. After complete introduction of the particles, the alloy was rapidly heated to 700 °C and solidified under a pressure of 100 MPa. One group of samples was further remelted at various temperatures by induction heating. Both groups of samples, i.e., as cast and remelted, were solutionized at 520 °C, water quenched, and then aged at 175 °C. The amplitude of age hardening (10 h at 175 °C) was taken as a

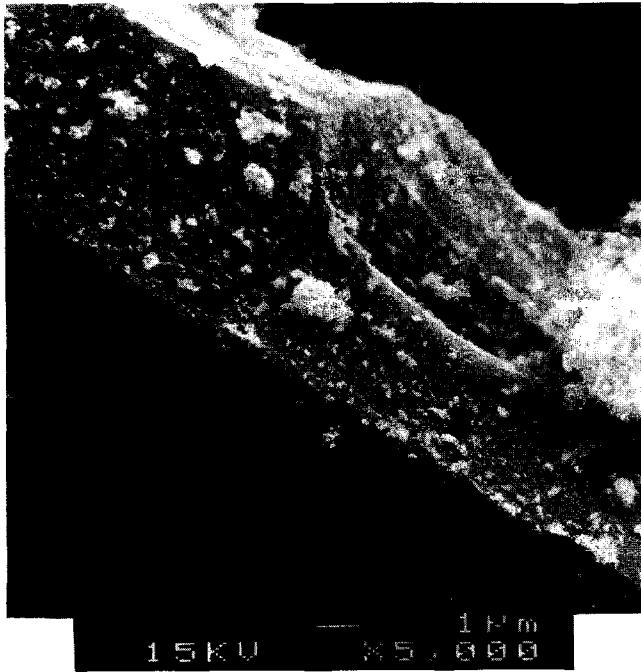


Fig. 5 Scanning electron micrograph of the surface of a TiO_2 -coated (2 vol%) SiC particle.

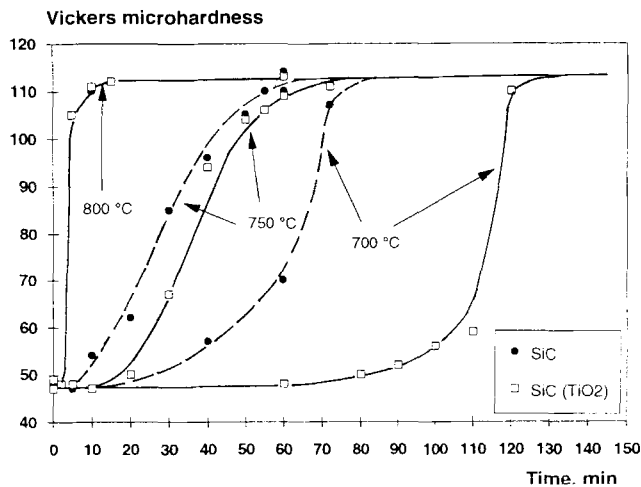
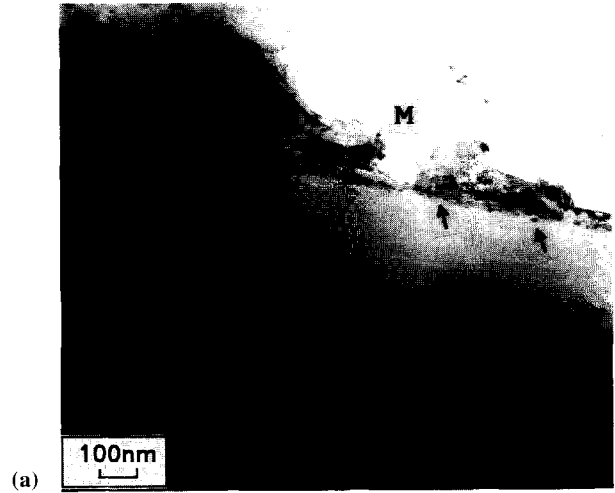
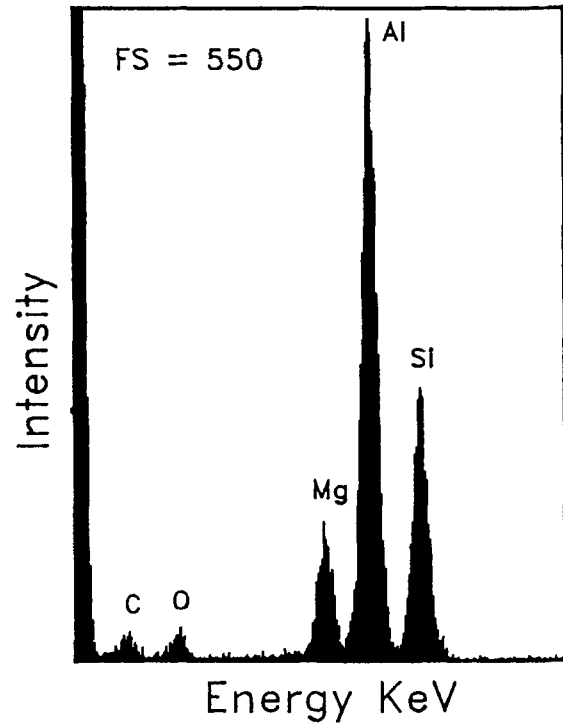


Fig. 6 Peak microhardness (10 h of aging at 175 °C) as a function of exposure time at various temperatures for Al-1%Mg/SiC composites containing uncoated and TiO_2 -coated SiC particles.

measure of the extent of degradation of SiC by Al because the formation of Al_4C_3 leads to Si released in the melt, thus rendering the Al-1% Mg matrix alloy age hardenable. This technique is thus used only at the beginning of the reaction process, the degree of hardening of the matrix reaching saturation when the Si content exceeds the solubility limit in Al-1%Mg. This is about 0.8 wt% for a solutionizing temperature of 520 °C.



(a)



(b)

Fig. 7 (a) TEM micrograph of interface region showing small precipitates (arrows) in the as-cast composite reinforced with uncoated SiC. (b) Typical X-ray spectrum of the precipitates indicating the presence of MgO.

The results showed that no protection was obtained at 800 °C, whereas at 700 °C significant reduction in the rate of SiC attack was observed with the TiO_2 coating compared to uncoated particles.^[10] Figure 6 shows the change in the peak microhardness of the matrix (measured after 10 h of aging at 175 °C) as a function of the prior exposure time of the SiC particles to the melt for the composites containing uncoated and TiO_2 -

coated particles. TiO_2 significantly increases the incubation time of the reaction to about 60 min at 700 °C, after which the rate of degradation appears to be similar to that of the composite with uncoated particles.

To better characterize the materials that have undergone remelting treatments, TEM observation and analyses were carried out on both the as-cast and remelted composites. Remelting for 1 and 2 h at 700 °C was chosen, and composites reinforced with uncoated and TiO_2 -coated particles were examined. Thin foils for TEM were prepared by cutting small sections from the bulk samples. These sections were punched into 3-mm diameter disks after grinding to a thickness of about 0.2 mm. The disks were mechanically dimple ground to produce a profile with a thickness of about 20 μm in the center. Finally, the disks were milled using a 4- to 5-kV and 2-mA argon ion beam and an incident angle of 25°. This angle was reduced to 14° as soon as a hole appeared in the middle of the disk for the final thinning stage. To avoid overheating of the disks during ion thinning, a liquid nitrogen-cooled sample holder was used. The microstructure was characterized by SEM and TEM using diffraction contrast imaging, microdiffraction, and EDS. The microscopes used were a JEOL S-840, a JEOL 2000 FX, and a Philips CM30 equipped with a LINK ultra-thin window X-ray detector allowing the detection of light elements down to boron.

4.1 Composites with Uncoated SiC Particles

The SiC/matrix interface in the as-cast composite reinforced with uncoated SiC particles typically exhibited numerous small precipitates of 10 nm or less in size, as shown in Fig. 7(a). The EDS analysis of the small precipitates and of the nearby matrix showed the presence of relatively high levels of magnesium and oxygen (Fig. 7b) in the precipitates. They were identified as MgO using microdiffraction and EDS quantitative analysis. Similar results were reported on 2124 Al/ $\text{Si}_3\text{N}_{4\text{w}}$ composites^[16] and on 2124 Al/ SiC_{w} composites,^[17] where small crystallites of MgO were found individually and in clusters along the Al/whisker interfaces in all samples. The MgO precipitates may be formed as a result of Mg and O segregated at the interface through the following reaction:



Indeed, extensive Mg segregation at the interface was observed in all samples characterized in this study (Fig. 8), as well as in many other investigations reported in the literature.^[16-18] Oxygen most probably originated from the surrounding air and was entrapped in the semisolid melt during fabrication of the composite.

It is also possible, however, that MgO was formed as a result of the reaction between Mg segregated at the interface during fabrication and the residual and thin SiO_2 layer found on the surface of as-received uncoated particles. Auger spectroscopy of as-received particles carried out in a previous study showed this layer to be typically 5 nm thick.^[11] The reaction is then:

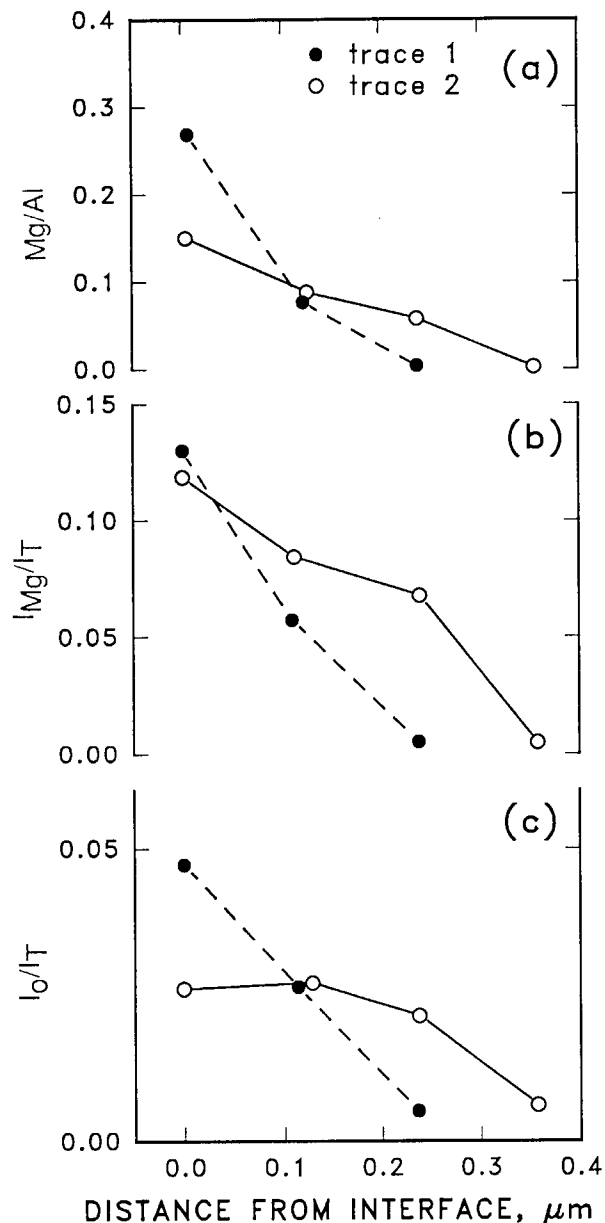
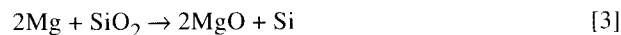


Fig. 8 Concentration ratios (in at.%) of Mg to Al (a) and X-ray intensity ratio of $I_{\text{Mg}}/I_{\text{T}}$ (b) and $I_{\text{O}}/I_{\text{T}}$ (c) along two traces perpendicular to the matrix/SiC interface, where I_{T} is the total net intensity from O, Mg, Al, and Si.

which is favored over the reaction producing MgAl_2O_4 for relatively high Mg content, as is the case here due to extensive segregation at the interface.^[17,19]

The matrix/SiC interface in the as-cast composite reinforced with uncoated SiC particles therefore consists of small MgO particles embedded in Al with some Mg in solution (which did not react with O at the interface). The presence of Mg in solution in Al is indicated by the variation of the O/Mg ratio measured along the interface and the fact that very small values of this ratio (close to 0) were obtained in some areas along the interface. Thus, the Mg/Al and O/Al ratio are expected to vary along the interface because the small MgO par-

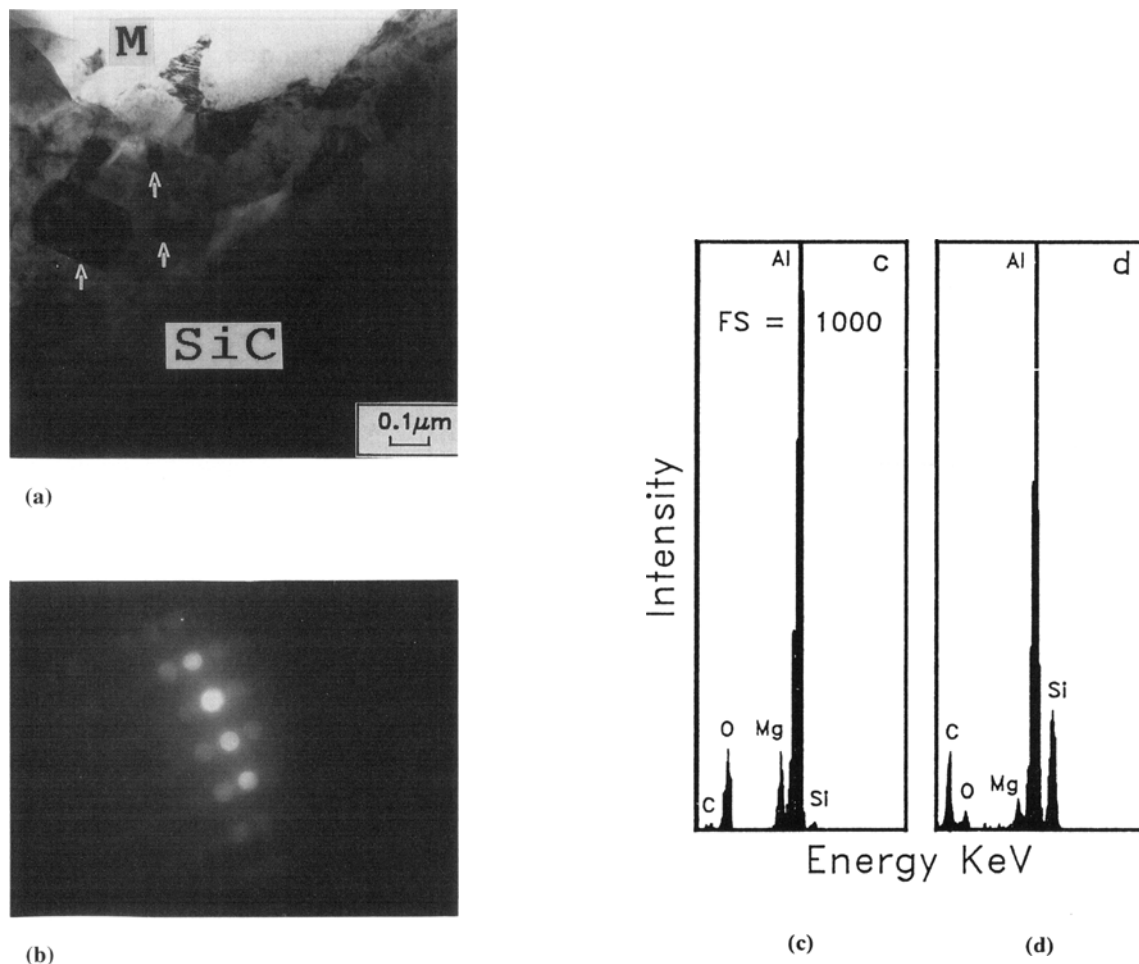


Fig. 9 (a) TEM photograph of the interface region showing discrete crystals (arrows) in the remelted composite reinforced with uncoated SiC particles. (b) Microdiffraction pattern of the largest crystal (arrow in a). It is indexed as fcc MgAl_2O_4 with $a_0 = 0.803$ nm. Zone axis, $B = [112]$. (c) X-ray spectrum acquired from the same crystal shown in (b). The intensity ratio of O/Mg is 1.1, which is near that of a spinel standard. (d) Typical X-ray spectrum from the interface region in (a) showing a carbon peak larger than that from the matrix.

ticles do not span the entire thickness of the foil so that the relative contributions from Al and MgO vary, but if Mg was entirely bound with O, then the O/Mg ratio should be constant.

After remelting at 700 °C for 1 h, a number of discrete crystals were observed at the interface in addition to the small MgO particles observed in the as-cast condition. These crystals were identified using microdiffraction and EDS. One of the microdiffraction patterns acquired from the crystal (arrows in Fig. 9a) was indexed as the [112] zone axis of MgAl_2O_4 with a lattice parameter of $a = 0.803$ nm (Fig. 9b). This zone axis unambiguously distinguishes MgAl_2O_4 and MgO [20]. In addition, the X-ray spectrum shown in Fig. 9(c), acquired from the same crystal, has an O/Mg intensity ratio equal to 1.1, which is near the value obtained from a spinel standard (≈ 1.3) and much higher than that from a MgO standard (≈ 0.16).

MgAl_2O_4 can form as a result of a number of reactions involving MgO, Al, O, and SiO_2 .^[11,21] Initially, as discussed above for the as-cast composite, MgO is favored because of the extensive Mg segregation at the interface. During remelting at

700 °C, homogenization of the Mg concentration in the liquid occurs, thus favoring the formation of MgAl_2O_4 .

Finally, a large carbon peak was often observed in X-ray spectra of the interface region of the remelted composites (Fig. 9d), indicating that degradation of SiC occurred during remelting according to Reaction 1. The amount of SiC consumed, however, was estimated from the increase in the microhardness of the composite with uncoated particles remelted for 60 min (Fig. 6) to be ≈ 3 to 5% of the total amount present. This is considerably lower than the data of Lloyd et al.,^[22] for which about 25% of the total amount of SiC was consumed in a 6061-20 vol% SiC composite after holding at 700 °C for 1 h, as measured from changes in the liquidus temperature.

4.2 Composites with TiO_2 -Coated SiC Particles

Figures 10(a) and (b) are TEM micrographs of the composites with TiO_2 -coated SiC in the as-cast and remelted (1 h at 700 °C) conditions, respectively. In the as-cast condition,

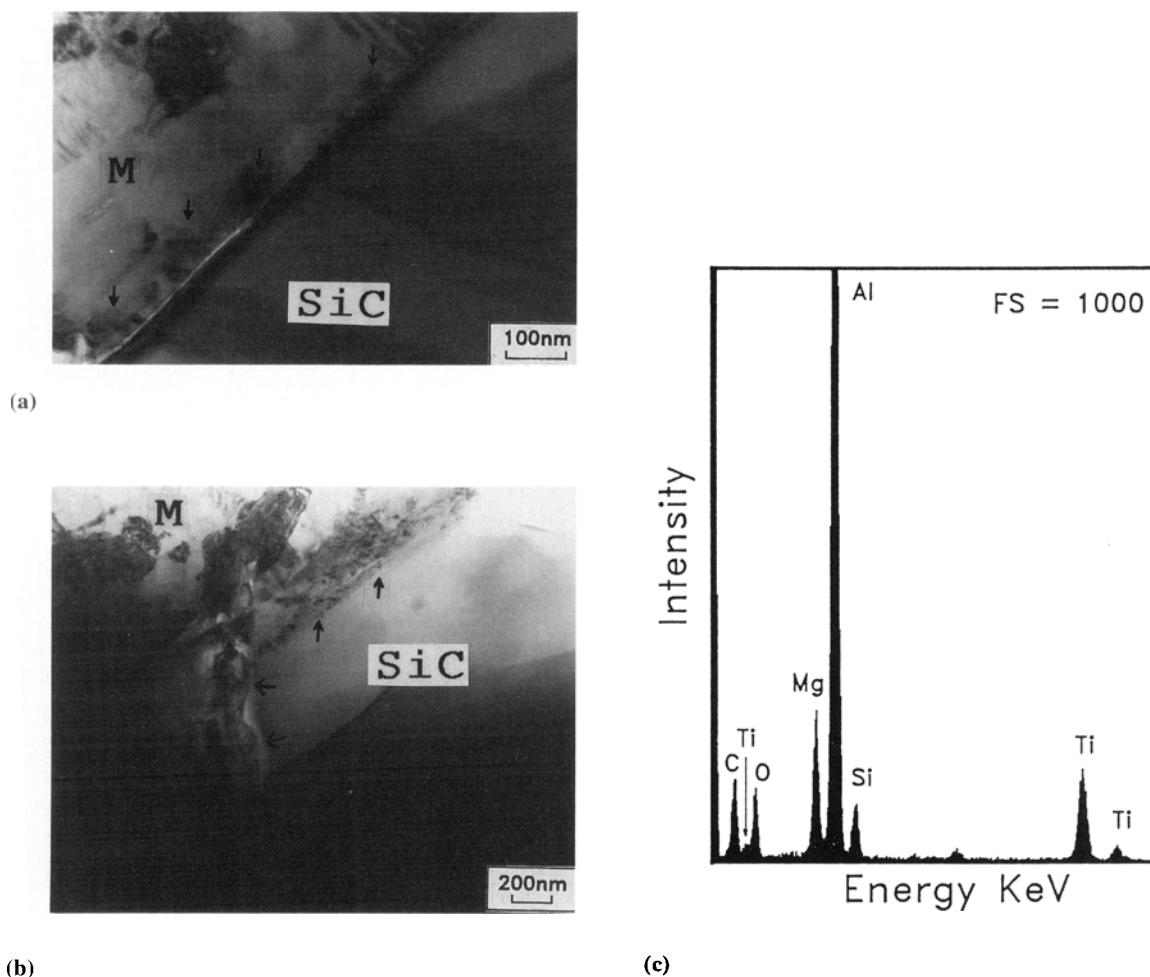


Fig. 10 TEM micrographs of composite with TiO_2 -coated SiC particles in the as-cast (a) and remelted (b) conditions. The arrows in (a) indicate clusters consisting of fine TiO_2 and MgO particles (c). The small arrows in (b) indicate small MgO particles.

clusters of very fine particles are seen along the interface (arrows in Fig. 10a). The average size of the clusters (≈ 50 to 100 nm) observed in Fig. 10(a) is on the same order of magnitude as that of the clusters observed on the surface of as-delivered TiO_2 -coated SiC (Fig. 4). Figure 10(c) shows a typical X-ray spectrum obtained from one of the clusters. The oxygen, magnesium, and titanium peaks in the spectrum are consistent with the presence of TiO_2 particles, stable during processing, and of MgO formed as a result of the extensive Mg and O segregation measured at the interface (Reactions 2 and 3).

In the composite remelted for 1 h at 700°C , the TiO_2 particles were still intact and apparently more MgO particles were observed, indicating that Reaction 2 continued to proceed during the remelting treatment at 700°C .

After 2 h of remelting at 700°C , however, significant degradation of SiC particles occurred, as indicated by the very irregular contour of the SiC particles and the formation of Mg_2Si particles as a result of the liberation of Si and C (Fig. 11). Thus, Fig. 11 shows the jagged contour of SiC, Mg_2Si particles, and some MgAl_2O_4 crystals. Moreover, TiO_2 particles were no longer observed along the interface, and Ti was not detected

near the interface, indicating that the initial TiO_2 particles were not stable during remelting at 700°C for 2 h.

These observations indicate that the TiO_2 particles covering SiC were relatively stable during remelting of the composites at 700°C for 1 h and that they protected the SiC from attack by liquid Al. This protection was not effective for longer than 2 h, at which time degradation of SiC occurred with the formation of Al_4C_3 crystals and Si release. The observations also showed that the TiO_2 particles did not form a continuous coating around SiC. Furthermore, the kinetics of the reaction after the incubation time is the same, at a given temperature, for the coated and uncoated particles. These last two observations suggest that dissolution of the TiO_2 particles prior to the SiC contact with the liquid, rather than restricted diffusion or catalysis, is the leading mechanism of protection. This mechanism is supported by a model of wetting on rough surfaces.^[23] Thus, the TiO_2 coating produces asperities on the SiC surface that may prevent contact between the SiC particles and the liquid for wetting angles (Θ) greater than 90° (Fig. 12). No Θ value for the system of interest (liquid Al-1%Mg on solid TiO_2) is available, but other investigations generally reported poor wetting

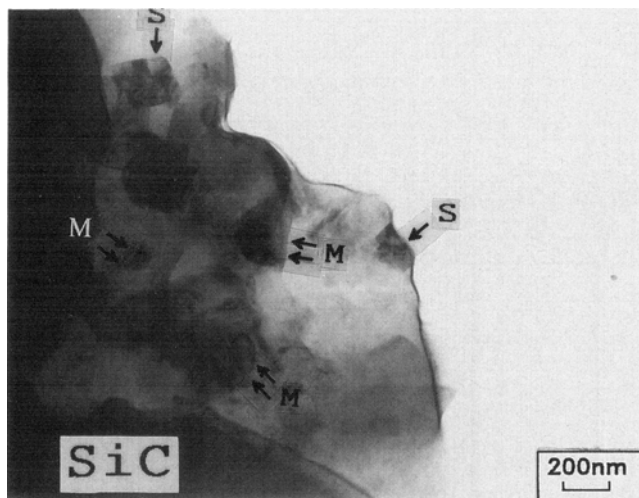


Fig. 11 TEM micrograph of composite with TiO_2 -coated SiC particles after remelting at 700°C for 2 h. Degradation of SiC particles is indicated by their very irregular contours. Mg_2Si particles formed as a result of the liberation of Si ($2\text{Mg} + \text{Si} \rightarrow \text{Mg}_2\text{Si}$) are also observed (M = MgAl_2O_4 ; S = Mg_2Si).

of oxides by liquid metals near their melting temperature.^[24,25] Thus, one could expect a delay in the SiC attack, corresponding to the dissolution time of TiO_2 particles in the molten alloy. However, an intimate contact between the SiC particles and the aluminum alloy was always observed by TEM. This contradictory result may be correlated to the process of solidification of the metal-matrix composite under pressure that changes the wetting parameters. The mechanism of protection is therefore not entirely clear at this stage and will require other TEM studies in samples cooled under normal atmospheric conditions.

5. Conclusion

Several techniques are now available to protect the SiC particles from degradation by liquid aluminum. Oxidation of particles and coating by sol-gel techniques slow the rate of attack, due to restricted diffusion of Al toward SiC and Si from the particle. Noncontinuous coatings obtained by dry mixing of SiC particles with TiO_2 powders appear to proceed in a different manner. A longer incubation time is observed for the reaction, which may be correlated with the dissolution of the powders in the melt. Further experiments are, however, needed to fully elucidate the mechanism of protection.

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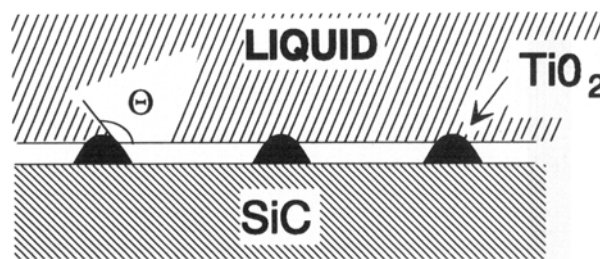


Fig. 12 Composite interface formed on a rough surface, Θ is the contact angle between TiO_2 particles and the liquid aluminum alloy. (From Ref 24.)

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