Formation of Magnesium Aluminate (Spinel) in Cast SiC Particulate-Reinforced Al(A356) Metal Matrix Composites

NING WANG, ZHIRUI WANG, and GEORGE C. WEATHERLY

Transmission (TEM) and scanning electron microscopy (SEM) are employed to study the SiC/Al-alloy interface in a cast SiC_p/Al(A356) metal matrix composite (MMC). Magnesium aluminate (spinel), $MgAl_2O_4$, was found at the interface as a reaction product after material processing. Comparisons of the crystal structure, structure factor, and interface reaction thermodynamics between $MgAl_2O_4$ and MgO have been carried out. The results from these comparisons confirm the experimental observation; *i.e.*, the favored interface phase is magnesium aluminate (spinel). Based on the thermodynamic analysis, the presence of oxygen in various forms in the system during processing, such as SiO_2, Al_2O_3, and MgO, is believed to be the source which supplies the oxygen for the formation of $MgAl_2O_4$.

I. INTRODUCTION

THE mechanical properties of metal matrix composites (MMCs) are controlled to a large extent by the structures and properties of the reinforcement/metal interface. It is believed that a strong interface permits transfer and distribution of the load from the matrix to the reinforcement, resulting in an increased elastic modulus and strength. From a metallurgical point of view, the desired interfacial region in a composite relies on several factors. For instance, (1) an intimate contact between the reinforcement and the matrix needs to be established through satisfactory wetting of the reinforcement by the matrix to ensure adequate adhesion and (2) the rate of chemical reaction at the interface should be very low and extensive interdiffusion between the component phases should be avoided so that the reinforcement will not be degraded. The choice of the reinforcing and matrix materials for a composite system often cannot satisfy these requirements at the same time. One of the ways of achieving such a desired interface while not having to sacrifice the performance of the composite is to apply a thin coating or coatings on the reinforcement,[1,2] which is chemically compatible with both the reinforcement and the matrix. Selective addition of alloying elements has also proved to be effective in improving wettability and reducing interdiffusion.[3] From a mechanical point of view, on the other hand, the behavior of the interface plays a significant role in controlling the strength of composites. The normal component of interfacial stresses would tend to cause interface debonding, while the shear component could be the cause of splitting of phase boundaries. This effect becomes more pronounced as the difference in the elastic constants between the two constituent phases increases. The following are some important factors in considering the interface properties: (1) a strong interface, as mentioned above, would ensure transfer of load from matrix to the reinforcement, (2) the mechanical compatibility requirement at the interface would result in stress discontinuity or a steep stress gradient at the interface because of the differences in the elastic and/or thermal coefficients between the reinforcement and matrix, and (3) a moderate coating phase between the reinforcement and matrix may decrease the stress gradient at the interface. Therefore, an ideal interface should be a mechanics continuum, involving coherency of the bond at the atomic level.[4] Because of these metallurgical and mechanical considerations, it is important to characterize the interfacial microstructure to understand the mechanical behavior of a composite and the interaction between the reinforcement and matrix during processing and service.[5] Studies of reinforcement/metal interfaces have been directed toward the recognition of general rules that govern bonding and interface behavior, both from a theoretical and experimental viewpoint.[6] This usually includes (1) identification of the interfacial reaction products,[3,4,7] (2) study on the wettability (contact angle) between the reinforcement or coatings on the reinforcements and molten matrix metal alloys,[8,9] (3) theoretical calculation of binding energy of the interface,[10] (4) crystallographic relationships and the atomic structure at the interface,[11-14] and (5) the thermodynamics of interfacial reactions.[6,15]

Two major problems encountered in the development of cast silicon carbide particulate-reinforced aluminum-matrix composites are (1) the reactivity of silicon carbide with molten aluminum at higher processing temperatures, which frequently results in the formation of reaction products at the SiC/Al interface and degrades the reinforcing particulates and (2) the poor wetting characteristics of silicon carbide by liquid aluminum at lower processing temperatures,[42] which usually results in weak bonding between SiC and Al matrix after solidification. In the process of fabricating SiC/Al composite materials, the weak bonding can be improved by advanced casting techniques, such as squeeze casting[16] and semi-solid slurry processing,[17] *etc.* Chemical reactions at the SiC-Al interface depend on several factors, such as temperature, pressure, atmosphere, chemical composition of the aluminum matrix, and surface chemistry of the silicon carbide. Several studies[9,18-22] have shown

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that, in the SiC/Al system, Al_4C_3 will form during processing at the interface according to the reaction

$$4Al + 3SiC = Al_4C_3 + 3Si$$

It has been proposed[22] that the reaction product Al_4C_3 could result in a degradation of the reinforcement strength and the interfacial strength. To overcome these problems, techniques for surface treatment[23,24,25] and fabricating[5,26,27] have been developed to ensure that the reinforcement can be wetted by the molten aluminum alloy and to inhibit any reaction between the reinforcement and the liquid metal. However, the surface treatments may result in the degradation of the interfacial structures, especially on high-temperature exposure for a long period of time, either during fabrication or in service.

The aim of the present work is to study the interfacial microstructure in a SiC/Al system composite produced by a molten metal mixing route using electron microscopy. Particular attention is focused on the morphology and phase identification of the reaction products at the interface, as they will play an important role in controlling the interfacial microstructure and mechanical properties of the composite. The formation mechanism of these interaction products will also be discussed.

II. EXPERIMENTAL

The composite materials used in this investigation were supplied by Alcan International Ltd. in the as-cast condition. A commercial-grade aluminum alloy (A356) with a nominal composition of 92.7 wt pct Al, 7 wt pct Si, and 0.3 wt pct Mg was employed as the matrix material. This alloy was then reinforced by 15 vol pct SiC partic ulates with an average size of 5 to 10 μm. A molten metal mixing technique[28] was used for preparing the composites. The mixing was carried out with a rotating dispersing impeller immersed in the molten mixture for a time sufficient to wet the particulates and to distribute them throughout the molten metal.[28]

The interface between the SiC reinforcement and the aluminum alloy matrix was examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), together with X-ray energy-dispersive spectrometry (EDX). Specimens for SEM observation were prepared by dissolving away the matrix in a 10 pct aqueous solution of NaOH at room temperature in order to reveal the interaction products on the surface of the silicon carbide. Secondary electron images were obtained using an S-570 scanning electron microscope at an accelerating voltage of 20 keV to show the morphology of the SiC surface. Specimens for TEM analysis were sectioned from as-cast composite material on an ISOMET low-speed saw into 0.4-mm-thick slices. Three-millimeter-diameter disks were taken from these slices using a Precision Coring Tool (model V7110) and lapped to about 100-μm thickness on metallographic papers and further polished on a glass plate to about 80 μm using diamond pastes. Argon ion milling was used for the final thinning of the thin foil for TEM study, operating at 4 kV with an ion current of 80 ~ 90 μA, a limit current of 3 mA, and a sample inclination of 15 deg to the ion beam. The TEM samples were examined in an H-800 transmission electron microscope equipped with a TRACOR-NORTHERN* TN-2000 EDX system operating at 200 kV.

III. RESULTS AND DISCUSSION

A. *Electron Microscopy*

In order to study the SiCl/Al interface, SEM was used to examine the morphology of the interface. Due to the selective etching of aluminum matrix, the SiC interfacial morphology could be readily revealed by dissolving the matrix in a 10 pct aqueous solution of NaOH. Figure 1 is a typical SEM micrograph showing the morphology of SiC surface obtained by this chemical dissolution method. As can be seen, in addition to the eutectic Si particles (the larger particles on SiC surface) which nucleated on the SiC particulates, many small particles are also observed on the SiC surface, indicating that chemical reactions occurred between particles and the matrix alloy. Figure 2 shows the SiC surface morphology (seen at a higher magnification) where the metal matrix has been chemically dissolved. It should be noted that the reaction products present on the SiC surface are in the form of discrete small particles. The interaction at the interface between SiC and matrix probably occurred during fabrication of the composite. X-ray energy-dispersive spectrometry analysis indicated that the small particles seen in Figure 2 were magnesium-rich phases (Figure 3). Kohyama *et al.*[29] also observed microsegregation of magnesium at the interface in a study of the SiC/Al(A5052) system. They proposed that the microsegregation of magnesium is due to the formation of MgO at the interface. In fact, there are a number of potential chemical reactions that may occur at the interface in the processing of the composite used in the present study. Based on the available thermodynamic data,[30-33,43] the possible reactions are listed in Table I. Reactions [1], [2], or [3] should be considered if chemical reactions between the reinforcement and molten aluminum alloy occur because of the silica coating at the surface of silicon carbide particulates formed during the surface treatment process. These reactions will form MgO, Al_2O_3, $MgAl_2O_4$, and dissolved Si. When the SiO_2 coatings are consumed by Reactions [1] through [3], the molten matrix alloy will directly contact silicon carbide, and Reactions [10] or [11] may take place. As a result, the Al_4C_3 forms as a stable compound in the melt and the solidified composite, with the excess Si becoming alloyed with the matrix. The presence of Al_4C_3 in the melt complicates processing, and the carbide may serve as sites for corrosion in the solidified composite.[22] There is also some evidence that the presence of Al_4C_3 at the SiC/Al interface interferes with the effective transfer of load between matrix and reinforcement, leading to a degradation in the mechanical properties. Therefore, this reaction must be controlled in order to maintain the composite integrity and maximize the composite properties.[34] Lloyd *et al.*[22] also reported that, in an A6061 matrix, Al_4C_3 was often associated with the Mg_2Si intermetallic, which is possibly formed by Reaction [11].

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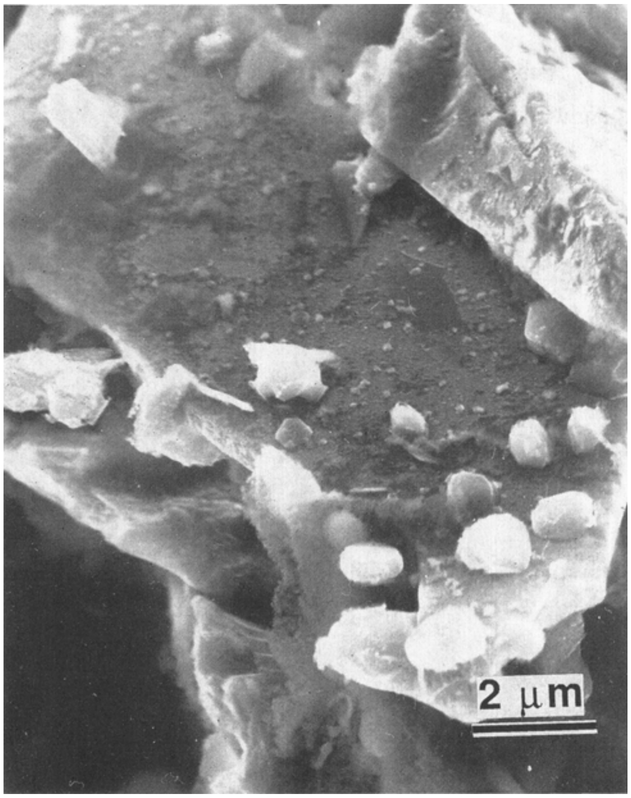

Fig. 1—SEM of SiC surfaces (the Al matrix has been dissolved in a 10 pct solution of NaOH), showing reaction products and eutectic Si (larger particles) on the surface of SiC particles.

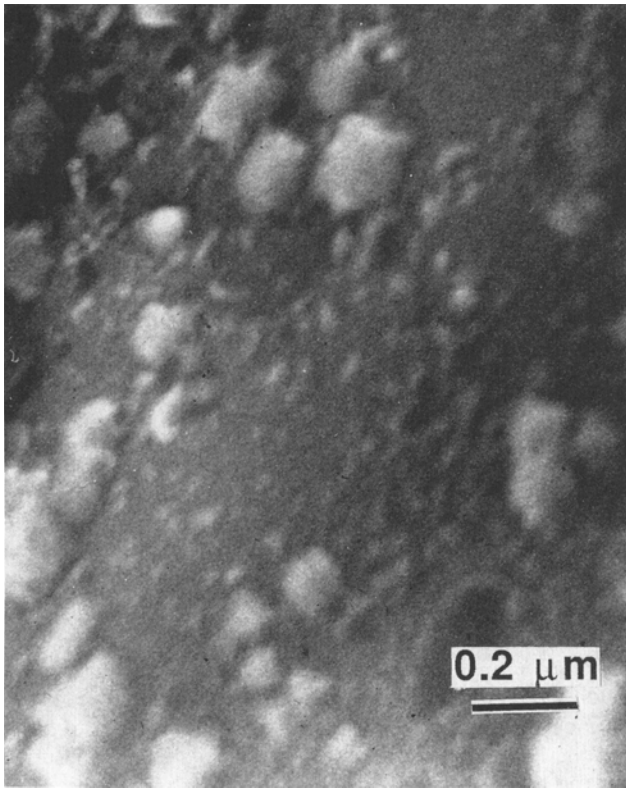

Fig. 2—The surface morphology of SiC surfaces at higher magnification, showing interfacial reaction products present on the SiC surface in the form of discrete particles.

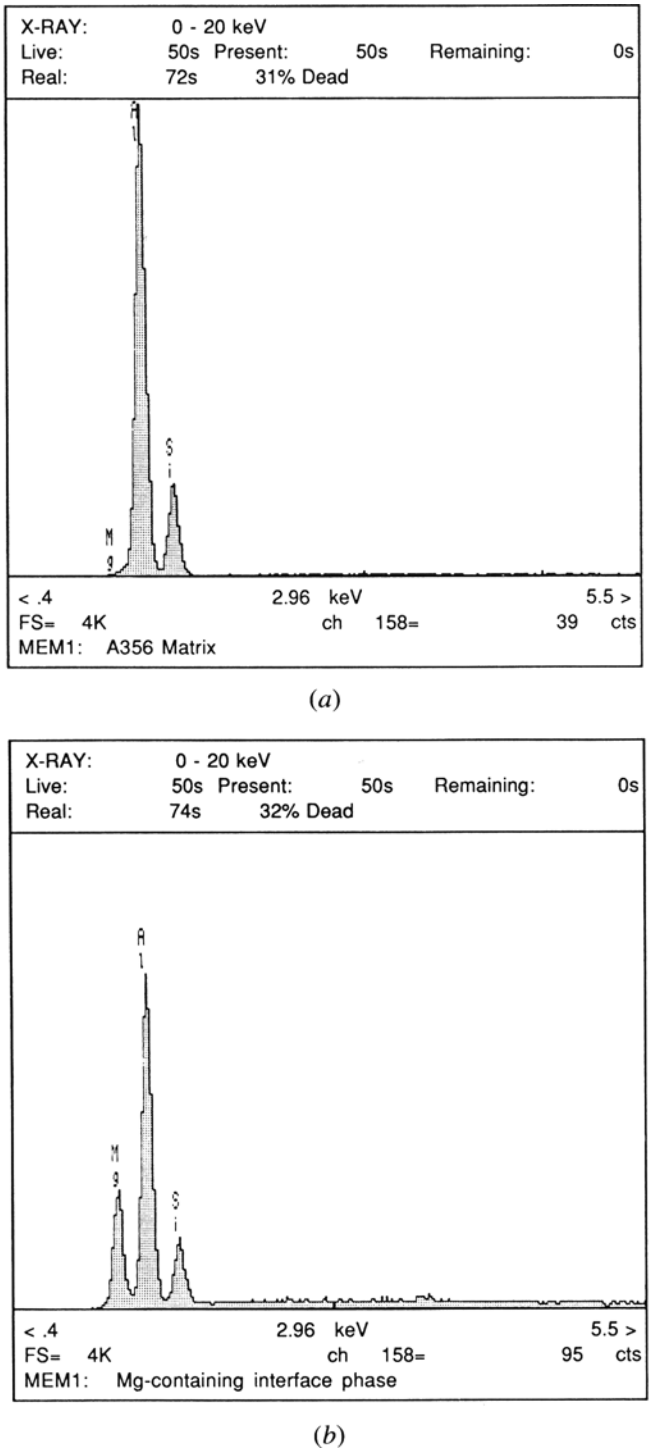

Fig. 3—X-ray energy dispersive spectrum from (*a*) A356 matrix and (*b*) interface reaction product at the SiC/Al interface in Fig. 2, showing that the interface phase contains a significant amount of magnesium.

The other reactions involve the formation of $MgAl_2O_4$ and MgO, although Eq. [8] can probably be ignored because the kinetics of this reaction will be very slow, as both reactants and products are solid.

Figure 4 is a typical TEM micrograph, showing the presence of reaction products at the SiC/Al interface, growing from the SiC surface into the aluminum matrix with a characteristic angular shape. The EDX analysis in the STEM mode showed that these angular-shaped

Table I. Possible Chemical Reactions Which May Occur During the Processing of the Composites

Number	Possible Chemical Reactions
1	$[SiO_2] + 2\{Mg\} = 2[MgO] + \{Si\}$
2	$[SiO_2] + 4/3\{Al\} = 2/3[Al_2O_3] + \{Si\}$
3	$[SiO_2] + 1/2\{Mg\} + \{Al\} = 1/2[MgAl_2O_4] + \{Si\}$
4	$\{Mg\} + 1/3[Al_2O_3] = [MgO] + 2/3\{Al\}$
5	$\{Mg\} + 4/3[Al_2O_3] = [MgAl_2O_4] + 2/3\{Al\}$
6	$\{Mg\} + [Al_2O_3] + \{O\} = [MgAl_2O_4]$
7	$\{Mg\} + 2\{Al\} + 4\{O\} = [MgAl_2O_4]$
8	$[MgO] + [Al_2O_3] = [MgAl_2O_4]$
9	$[MgO] + 2\{Al\} + 3\{O\} = [MgAl_2O_4]$
10	$4\{Al\} + 3[SiC] = [Al_4C_3] + 3\{Si\}$
11	$2\{Mg\} + [SiC] = [Mg_2Si] + [C]$

Note: The different types of brackets are employed to represent solid [ ] and liquid { }, respectively.

phases contain a significant amount of magnesium.[35] Therefore, there is some uncertainty as to whether this interfacial product is $MgAl_2O_4$ or MgO.[13,29,36] To answer this question, a direct and convincing method is to carry out a comparison of the structure factors of the two possible Mg-containing phases, MgO and $MgAl_2O_4$.

Magnesium oxide, MgO, has a cubic crystal structure (type B1) with a lattice constant $a = 4.213$ Å. The coordinates of the eight atoms in the unit cell are:

four magnesium atoms at 000; $\frac{1}{2}\,\frac{1}{2}\,0$; $\frac{1}{2}\,0\,\frac{1}{2}$; $0\,\frac{1}{2}\,\frac{1}{2}$;
four oxygen atoms at $\frac{1}{2}\,\frac{1}{2}\,\frac{1}{2}$; $\frac{1}{2}\,00$; $0\,\frac{1}{2}\,0$; $00\,\frac{1}{2}$.

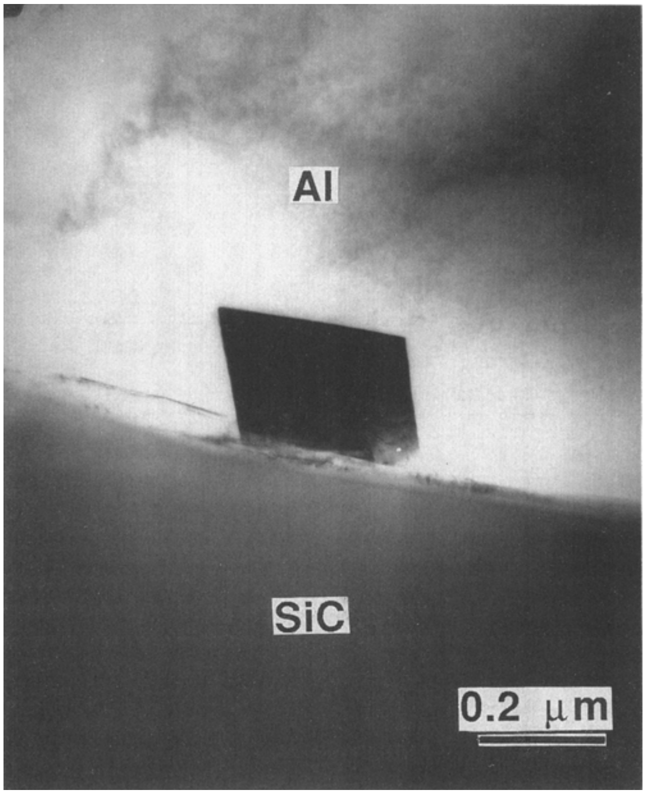

Fig. 4—Transmission electron micrograph showing an angular-shaped reaction product crystal at the SiC/Al interface.

Magnesium spinel,[37] $MgAl_2O_4$, is also a cubic crystalline (type $H1_1$) phase with $a = 8.0800$ Å. The coordinates of the 56 atoms in the spinel unit cell are:

eight magnesium atoms at $[000; \frac{1}{2}\,\frac{1}{2}\,0; \frac{1}{2}\,0\,\frac{1}{2}; 0\,\frac{1}{2}\,\frac{1}{2}] +$ $000; \frac{1}{4}\,\frac{1}{4}\,\frac{1}{4}$;
sixteen aluminum atoms at $[000; \frac{1}{2}\,\frac{1}{2}\,0; \frac{1}{2}\,0\,\frac{1}{2}; 0\,\frac{1}{2}\,\frac{1}{2}] +$ $\frac{5}{8}\,\frac{5}{8}\,\frac{5}{8}; \frac{5}{8}\,\frac{7}{8}\,\frac{7}{8}; \frac{7}{8}\,\frac{5}{8}\,\frac{7}{8}; \frac{7}{8}\,\frac{7}{8}\,\frac{5}{8}$;
thirty-two oxygen atoms at $[000; \frac{1}{2}\,\frac{1}{2}\,0; \frac{1}{2}\,0\,\frac{1}{2}; 0\,\frac{1}{2}\,\frac{1}{2}] +$ $xxx; x\bar{x}\bar{x}; \bar{x}x\bar{x}; \bar{x}\bar{x}x; (\frac{1}{4} - x)\,(\frac{1}{4} - x)\,(\frac{1}{4} - x); (\frac{1}{4} - x)\,(\frac{1}{4} + x)\,(\frac{1}{4} + x); (\frac{1}{4} + x)\,(\frac{1}{4} - x)\,(\frac{1}{4} + x); (\frac{1}{4} + x)\,(\frac{1}{4} + x)\,(\frac{1}{4} - x)$.

with $x \approx -1/8$. The kinematic structure factor for a reflection *hkl*, *i.e.*, the amplitude of the reflection from the plates (*hkl*), is given by

$$\mathbf{F}_{hkl} = \sum_j f_j(\theta) \exp\,[2\pi i(hx_j + ky_j + lz_j)] \qquad [1]$$

where x_j, y_j, and z_j are the fractional atomic coordinators, $f_j(\theta)$ the atomic scattering amplitude for atom *j*, and *h*, *k*, *l* the Miller indices of the reflections $\mathbf{g}_{hkl}$. Using this expression and the atomic coordinates listed above, the structure factors of $MgAl_2O_4$ and MgO are calculated in Table II. Magnesium Oxide or $MgAl_2O_4$ can be distinguished by comparing the predicted intensities in the electron diffraction patterns. For example, (200), (240), *etc.* are forbidden reflections for $MgAl_2O_4$, while they are allowed reflections for MgO. The predicted diffraction patterns for $MgAl_2O_4$ are displayed in Figure 5. The spots marked by (*) are double diffraction ones. In the present work, a number of single-crystal diffraction patterns of the angular Mg-containing phase were obtained (Figures 6(a) through (d)). The {200} reflections in the [001]* diffraction pattern (Figure 6(a)) are missing, which agrees with the structure factor rule for $MgAl_2O_4$. The appearance of diffraction spots {200} in [011]* and [013]*, {420} in [112]*, *etc.* (Figures 6(b) through (d)) is considered to arise from double diffraction. In addition, all of the diffraction patterns can be indexed in agreement with the $MgAl_2O_4$ structure. This analysis indicates that the reaction products having an angular shape are magnesium spinel, $MgAl_2O_4$.

The possibility of spinel formation as an interfacial reaction product in metals reinforced with oxide fibers has already been noted.[38] Because the relatively open anion (oxygen) structure of spinel permits changes in its bivalent and trivalent cation (metal) structure, the physical, chemical, and mechanical properties may be varied accordingly. Thus, spinel phase may be used to promote interfacial bonding, since they potentially can form strong chemical bonds with both metals and ceramics. Several studies of the Al_2O_3/Al alloy system indicated that the $MgAl_2O_4$ spinel may also be formed at the reinforcement/matrix interface.[4,39] Levi *et al.*[4] reported that the incorporation, wetting, and bonding of the reinforcement could be achieved by alloying aluminum with an element which can interact chemically with the reinforcement to produce a new phase at the interface which is readily wetted. In the case of an Al-Mg alloy, they concluded that the bonding was achieved through the formation of an $MgAl_2O_4$ (spinel) layer by reaction between the reinforcement and the magnesium in the liquid aluminum.

Table II. Structure Factor Rule of $MgAl_2O_4$ and MgO

Phases	F^2_{hkl}	Indices of Planes
$MgAl_2O_4$	0	h,k,l mixed (odd or even) integers
	$32(f_{Mg} + \sqrt{2}f_{Al})^2$	h,k,l odd and $h + k + l = 8n + 1$ $(n = 1, 2, \ldots)$
	$32(f_{Mg} - \sqrt{2}f_{Al})^2$	h,k,l odd and $h + k + l = 8n - 5$
	$32(f^2_{Mg} + 2f^2_{Al})$	h,k,l odd and $h + k + l = 8n - 3$ or $8n - 1$
	$256(f_{Al} - 2f_O)^2$	{222}, {226}, {266}, {666}, . . .
	$64(f_{Mg} + 2f_{Al} + 4f_O)^2$	$h,k,l = 4n$ and $h + k + l = 8n$
	$64(f_{Mg} - 2f_{Al} - 4f_O)^2$	$h,k,l = 4n$ and $h + k + l = 4(2n + 1)$
	$64f^2_{Mg}$	$h,k,l \neq 4n$ and $h + k + l = 4n$
	0	others
MgO	0	h,k,l mixed (odd or even) integers
	$16(f_{Mg} - f_O)^2$	h,k,l odd
	$16(f_{Mg} + f_O)^2$	h,k,l even

McLeod[15] recently studied the kinetics of the growth of spinel $MgAl_2O_4$ in alumina-reinforced Al-Mg alloy matrix composites. His thermodynamic analysis indicated that spinal can form at Mg levels as low as 0.03 wt pct. For both AA-6061 and AA-2014 matrix alloys, the $MgAl_2O_4$ was found to be the stable interfacial phase. Although the properties of many spinels are not fully known, they generally possess high melting points, good mechanical strength, and are chemically stable. Therefore, it might be possible to tailor a strong, stable spinel interface to a whisker-MMC.[38,40,41]

It should be noted that, in the SiC/Al(356) composite system, there is a second interface associated with the addition of SiC, *i.e.*, the interface between SiC and eutectic Si, which should be also taken into account because it may play an important role in controlling the final mechanical properties of the composite, too.

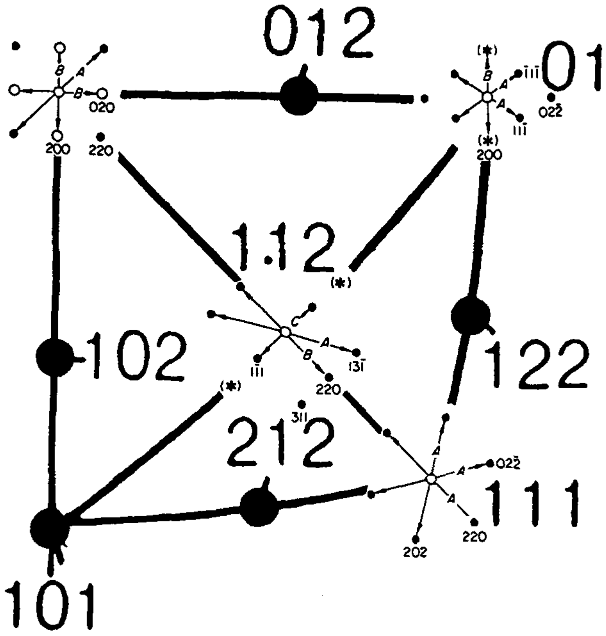

Fig. 5—The predicted electron diffraction patterns for $MgAl_2O_4$. The spots marked by (*) are double diffraction ones.

B. *Thermodynamic Consideration*

As described above, $MgAl_2O_4$ was found at the interface between the SiC particulates and aluminum. All of the chemical reactions involving $MgAl_2O_4$ and MgO listed in Table I are thermodynamically possible up to 1000 K. In the reactions listed in Table I, $MgAl_2O_4$ is formed during the processing of the composite by chemical reaction of Mg or MgO with other oxides and/or oxygen in the system. This indicates that the presence of oxygen in the system will play a key role in the formation of spinel. Naturally, one question that arises in the context of possible interfacial reactions during the fabrication of the composite concerns the supply of the oxygen. There are several sources that must be considered. First, as mentioned above, one of the possible supplies of oxygen is SiO_2, since the silicon carbide particulates are roasted at high temperature (800 °C to 1300 °C) before being introduced into the molten aluminum alloy.[28] Actually, thin SiO_2 film (<100 Å) would always be found on the surface of SiC particulates even if they are not intentionally roasted in an oxygen-containing atmosphere.[23] Reactions [1] through [3] refer to this source of oxygen. The liquid alloy reacts first with this silica film during casting and then attacks the silicon carbide.[24] The attack of silicon carbide can be restricted if thicker SiO_2 layers are formed on the SiC particulates during the roasting process. Other sources of oxygen should also be taken into account. As a molten aluminum alloy has an extraordinarily high affinity for oxygen (*e.g.*, only 2.56×10^{-21} atm of oxygen partial pressure is needed to oxidize aluminum at 1000 K), a thin aluminum oxide layer would be continuously formed at the liquid surface and introduced to the SiC/Al interface by stirring. Reactions [4] through [6] are based on this consideration. Levi *et al.*[4] investigated the interface interactions during fabrication of aluminum alloy-alumina fiber composites and found that interaction between the Al_2O_3 fibers and the matrix resulted in the formation of a Mg-rich region around the fibers which was retained during the subsequent heat treatment. Electron and X-ray diffraction studies showed that the interaction zone consisted of $MgAl_2O_4$, MgO, and fine polycrystalline α-Al_2O_3. It was suggested that the $MgAl_2O_4$ spinel formed by the reaction between Mg, which was in solution, and both the Al_2O_3 fiber and fine α-Al_2O_3, which resulted from oxidation of the melt.

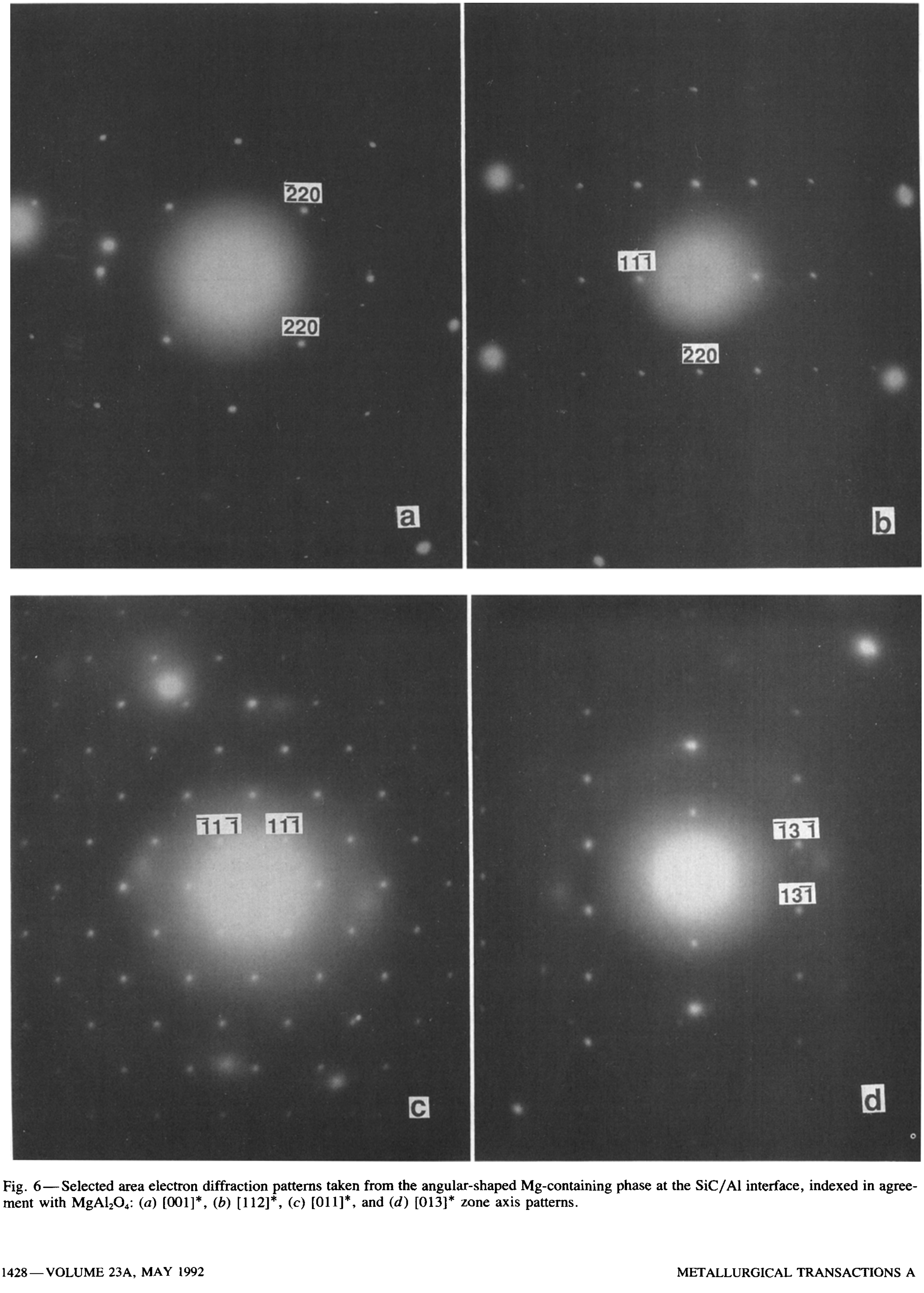

Fig. 6—Selected area electron diffraction patterns taken from the angular-shaped Mg-containing phase at the SiC/Al interface, indexed in agreement with $MgAl_2O_4$: (*a*) [001]*, (*b*) [112]*, (*c*) [011]*, and (*d*) [013]* zone axis patterns.

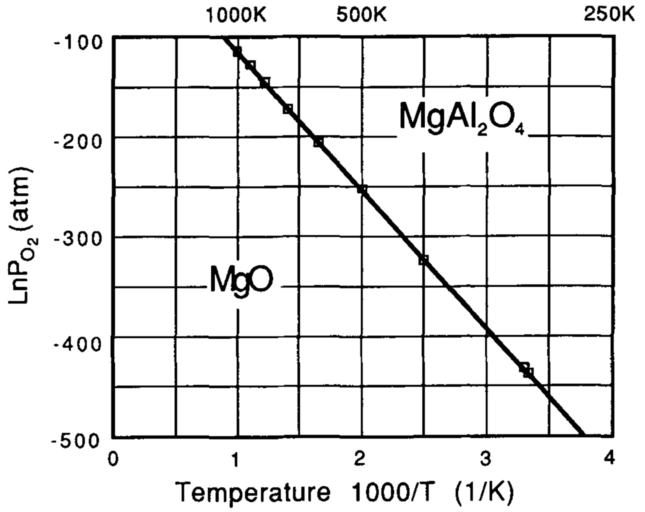

Fig. 7—Temperature/oxygen partial pressure stability regions of MgO and $MgAl_2O_4$ for Reaction [9].

Reaction [9] is of special interest in determining whether $MgAl_2O_4$4 or MgO is formed. Using the thermodynamic data from References 30 and 32, one obtains the simplified equation for the free-energy change of this reaction

$$\Delta G^\circ = 74.393T - 4.0749 \times 10^5 \text{ (cal/mol)} \qquad [2]$$

which has an equilibrium partial pressure of oxygen given by (Figure 7)

$$\ln P_{O_2} \text{ (atm)} = 25.11151 - 136{,}718.67/T \qquad [3]$$

It can be seen that, from Figure 7, the formation of $MgAl_2O_4$ is more thermodynamically favorable in practice, since the reaction can proceed at a very low partial pressure of oxygen. For example, by substituting 1000 K into Eq. [3], it can be shown that a partial pressure of oxygen of only 3.4×10^{-49} atm is needed to establish the thermodynamic equilibrium for Reaction [9]. McLeod[15] carried out a similar calculation for Reactions [4] and [5] and presented the results as stability diagrams (Mg content *vs* temperature). His results show that the stable interfacial phase is $MgAl_2O_4$, which could form at Mg levels as low as 0.03 wt pct. The Mg content in A356 for the present work is 0.3 wt pct, high enough to meet this criterion. The present analysis further demonstrates that spinel formation in Al-based MMCs is an important consideration.

The formation of the magnesium spinel in the MMC system is a very important phenomenon which has to be taken into account in processing the composite material.

IV. SUMMARY

The electron microscopy study presented in this work has again determined that magnesium spinel, $MgAl_2O_4$, is present at the interface between SiC and the aluminum alloy matrix in cast SiC_p/Al(A356) MMCs. The presence of oxygen in various forms, such as SiO_2, Al_2O_3, and MgO, in the system supplies the oxygen for the formation of the $MgAl_2O_4$ phase.

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