

Reaction Products of Al/TiC Composites Fabricated by the Pressureless Infiltration Technique

K.B. LEE, H.S. SIM, and H. KWON

The interfacial reaction products of the Al-Mg/TiC_p composite fabricated by the pressureless infiltration method were analyzed using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and transmission electron microscopy (TEM). During the fabrication of composites, reaction products with various morphologies and sizes were formed in the Al matrix as well as in the vicinity of the TiC particles by the interfacial reaction between the Al alloy and the TiC particles. From the EDS and selected-area diffraction pattern (SADP) analysis, Al₄C₃, Al₁₈Ti₂Mg₃, Ti₂AlC, Al₃Ti, and TiAl could be identified to form as interfacial reaction products. Both the size and the amount of the reaction products were increased with increasing fabrication temperature as well as fabrication time. Coarse Al₃Ti was barely observed in water-quenched composites, while it was observed at all fabrication temperatures (700 °C to 1000 °C) in furnace-cooled conditions.

I. INTRODUCTION

MANY types of ceramic phases have been added to Al matrix composites as reinforcement: particles, whiskers, and fibers of the oxides (Al₂O₃, MgO, TiO₂, etc.), carbides (SiC, TiC, B₄C, etc.), nitrides (AlN, BN, Si₃N₄, etc.), and borides (TiB₂, etc.). Recently, the Al-Ti-C system has become of great interest in the grain refining industry, because it may be a viable alternative to the Al-Ti-B system.^[1,2,3] Therefore, the Al-Ti-C system has been studied by a number of researchers. More recently there has been a growing interest in the development of technologies for Al composites reinforced with TiC particles because of good wettability with Al, high hardness, high temperature stability, and low weight. Accordingly, there were many reports about Al/TiC composites fabricated by various methods including XD process, powder metallurgy (P/M) and casting route, etc.^[4-30]

Because the interfacial reaction in Al composites reinforced with ceramic phases depends on several fabrication parameters (e.g., temperature, holding time, atmosphere, and chemical composition of both the Al matrix and the reinforcements), the characteristics of composites fabricated will be changed, depending on the fabricating procedure and composite system.^[31] While there are several binary (Al₃Ti, TiAl, and Al₄C₃) and ternary (Ti₂AlC and Ti₃AlC) carbides that have been reported in Al-Ti-C systems, their results do not agree with each other. Thus, the disagreement in phases reported could possibly be related to the difference in fabrication methods. Prasad *et al.*^[28] and Pandey *et al.*^[25] have observed Al₃Ti and Al₄C₃ phases as reaction products in Al/TiC composite, which was fabricated by the conventional P/M route, whereas Mitra *et al.*^[6] have reported Al₃Ti, Al₄C₃, Ti₂AlC, and TiAl phases in Al/TiC composite, which was fabricated by the XD process.

The interfacial reaction between Al matrix and reinforcement in Al composites is very important, because it can

affect the way that the load is transferred at the interface and then can affect the mechanical properties of the composites.^[29] In addition, the interfacial reaction can change the composition of the matrix and reinforcement. Although limited interfacial reactions may enhance the load bearing capabilities of composites, extensive interfacial reaction will form to the detriment of composites. Therefore, in order to obtain optimum mechanical properties, it is important to achieve good bonding between the Al matrix and the ceramic reinforcements via appropriate interfacial reaction. In the case of the Al/TiC composites, however, the interfacial reaction between Al matrix and TiC reinforcement is not clearly established, as mentioned previously. In addition, no data are available on the Al/TiC_p composite fabricated by a pressureless infiltration technique under a nitrogen atmosphere. Therefore, in this study, the Al-Mg/TiC_p composite was fabricated by the pressureless infiltration method and the microstructures were investigated.

II. EXPERIMENTAL PROCEDURE

The average size of Al, Mg, and TiC particles used in this study was about 50, 13, and 7 μm, respectively. After these powders (Al-2.5 wt pct Mg-10 vol pct TiC) were blended by roll mixing in an alumina jar, the powder mixture was put into a crucible. A 5052 Al ingot was placed on the loose powder bed in a crucible. This assembly was heated to a required temperature in the range of 700 °C to 1000 °C and held for 1 and 5 hours under a flowing nitrogen atmosphere (1000 cc/min) in a tube furnace. Then, the assembly was cooled to ambient temperature under the nitrogen atmosphere. In order to investigate the effect of cooling rate on interfacial reaction, the assembly was heated to the same condition under a flowing nitrogen atmosphere in a tube furnace and then quenched into water. Figure 1 shows the heating and cooling curves in the furnace-cooled condition. The average heating and cooling rates are about 15 °C/min and 9 °C/min, respectively. The schematic arrangement employed for the composite fabrication in this study is given elsewhere.^[32,33,34]

The resulting microstructures and reaction products were investigated by optical microscopy, X-ray diffractometry (XRD), scanning electron microscopy (SEM), and transmission

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electron microscopy (TEM). For SEM observation, specimens were prepared by dissolving away the Al matrix in a solution of methanol bromine and examined using a JEOL* 1210LV scanning electron microscope equipped with

*JEOL is a trademark of Japan Electron Optics Ltd., Tokyo.

an energy-dispersive X-ray spectroscopy (EDS) operated at 10 to 20 kV. Thin foils for TEM analysis were ground mechanically to a thickness of about 60 μm and then punched to 3-mm-diameter discs. Finally, the discs were thinned by dimpling and ion milling (5 kV, at a tilt angle of 4 to 6 deg). All samples were examined by a JEM 1210 transmission electron microscope coupled with an EDS system operated at 120 kV.

III. RESULTS

Because the spontaneous infiltration of molten metal occurred at all required temperatures under a nitrogen atmosphere, it was possible to fabricate Al-Mg matrix composites

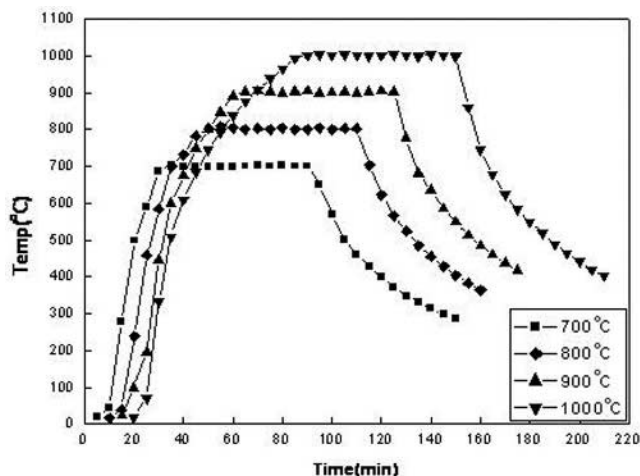


Fig. 1—Heating and cooling curves in furnace-cooled condition.

reinforced with TiC particles. The spontaneous infiltration behavior of molten metal was explained in detail in previous articles.^[32,33,34] Figure 2(a) shows the relatively uniform distribution of the TiC particles in the water-quenched composite. Even though there is no agglomeration and segregation of the TiC particles, it can be seen that reaction products of various morphology and size are observed in the Al matrix, as well as in the vicinity of the TiC particles according to the fabrication conditions ((b) through (e)). As the fabrication temperature increases from 700 °C to 1000 °C, both the size and the amount of the reaction products increase. However, such coarse reaction products were barely observed in water-quenched composites (Figure 2(a)). In addition, reaction products increased with increasing fabrication time (Figure 3).

As previously mentioned, several binary (Al_3Ti , TiAl , and Al_4C_3) and ternary (Ti_2AlC , Ti_3AlC) phases have been reported as reaction products in Al-Ti-C systems. However, because the relative XRD peak location of each phase is similar, it is difficult to clearly identify the types of the reaction products by XRD analysis. Therefore, the clear identification of the reaction products was performed by SEM and TEM observation.

Figure 4(a) shows SEM micrographs and EDS spectra of the reaction products obtained in a furnace-cooled composite, after dissolving away the Al alloy matrix with a solution of methanol bromine, and Figure 4(b) is the SEM backscattered image. Various reaction products were observed in the Al matrix as well as in the vicinity of TiC particles. According to the analytical results obtained from EDS analysis, reaction products were identified as TiAl , Al_3Ti , and Ti_3Al , respectively.

Figure 5 shows SEM micrographs and EDS spectra of the reaction products obtained in a solution-treated composite. It is evident that another reaction product was formed on the surface of Al_3Ti with a rectangular shape. This reaction product was not observed in composites without solution treatment and identified as $\text{Al}_{18}\text{Ti}_2\text{Mg}_3$.

Other reaction products are shown in Figure 6. As a result of the EDS analysis, those phases coincided with the

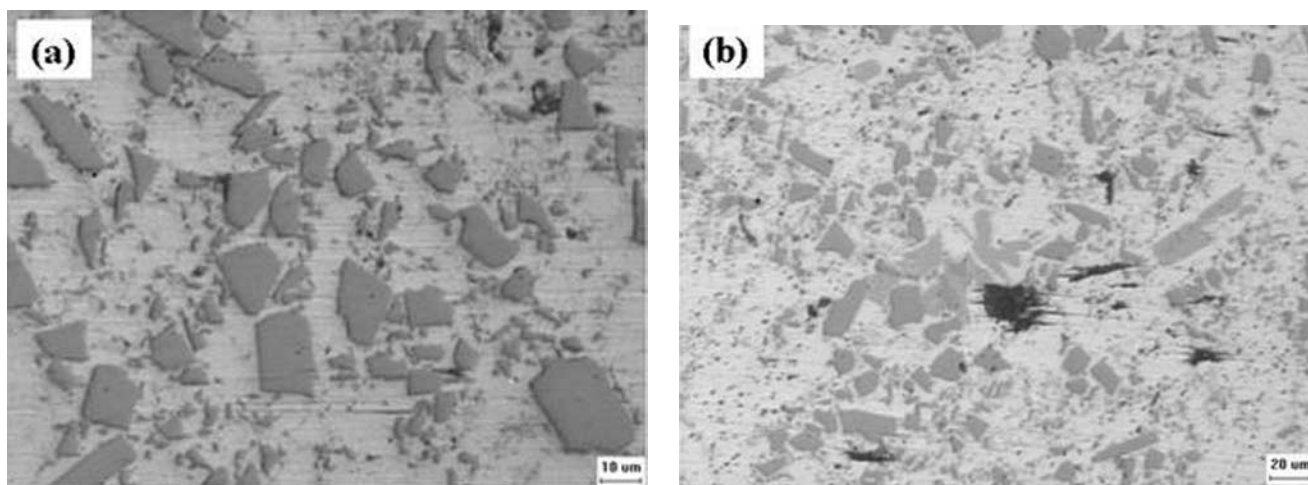


Fig. 2—Optical micrographs showing the distribution of reinforcement and reaction products in Al-Mg/TiC composites: (a) water quenched at 800 °C; and (b) through (e) furnace-cooled condition at 700 °C, 800 °C, 900 °C, and 1000 °C, respectively.

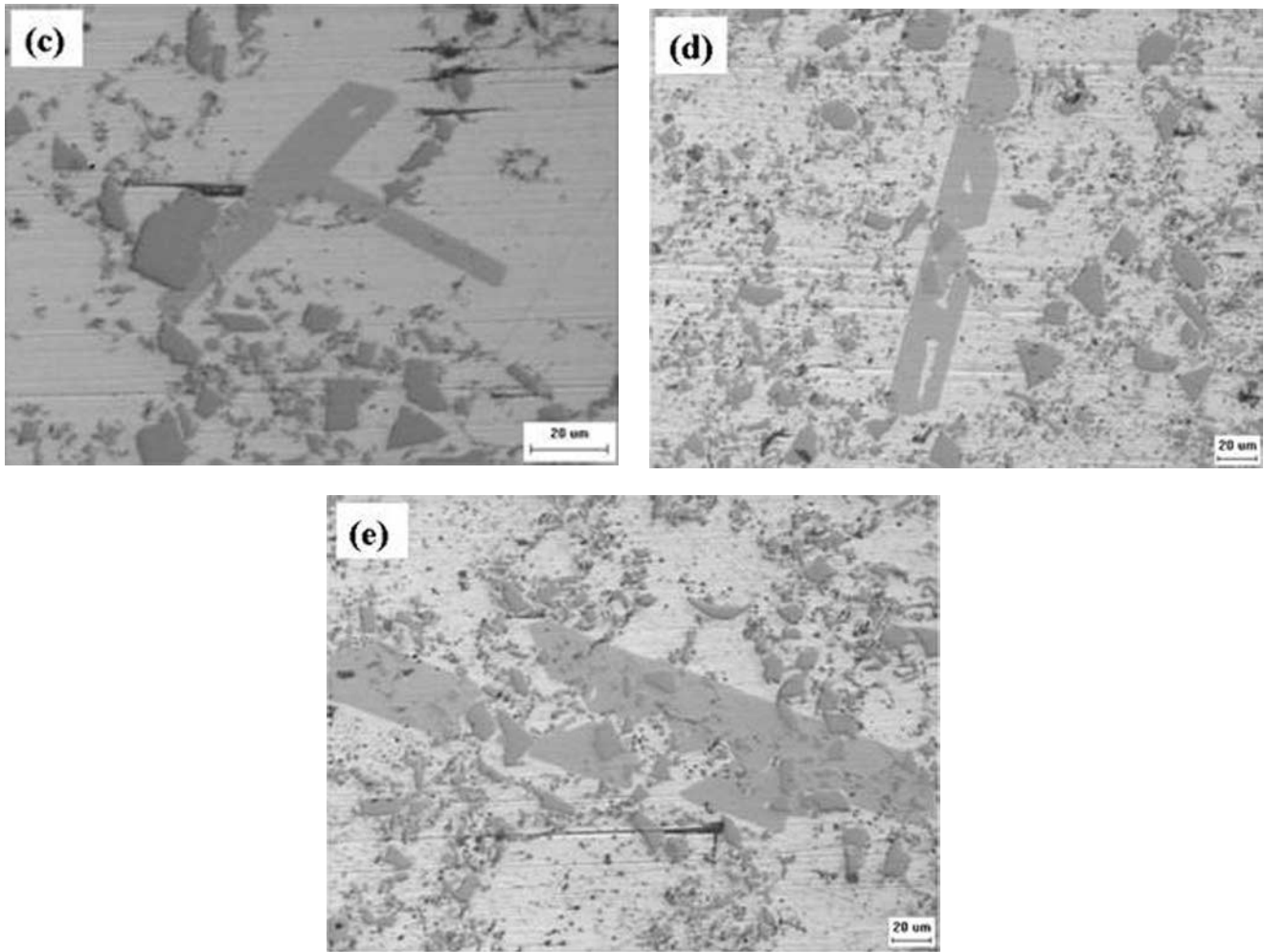


Fig. 2—(Continued). Optical micrographs showing the distribution of reinforcement and reaction products in Al-Mg/TiC composites: (a) water quenched at 800 °C; and (b) through (e) furnace-cooled condition at 700 °C, 800 °C, 900 °C, and 1000 °C, respectively.

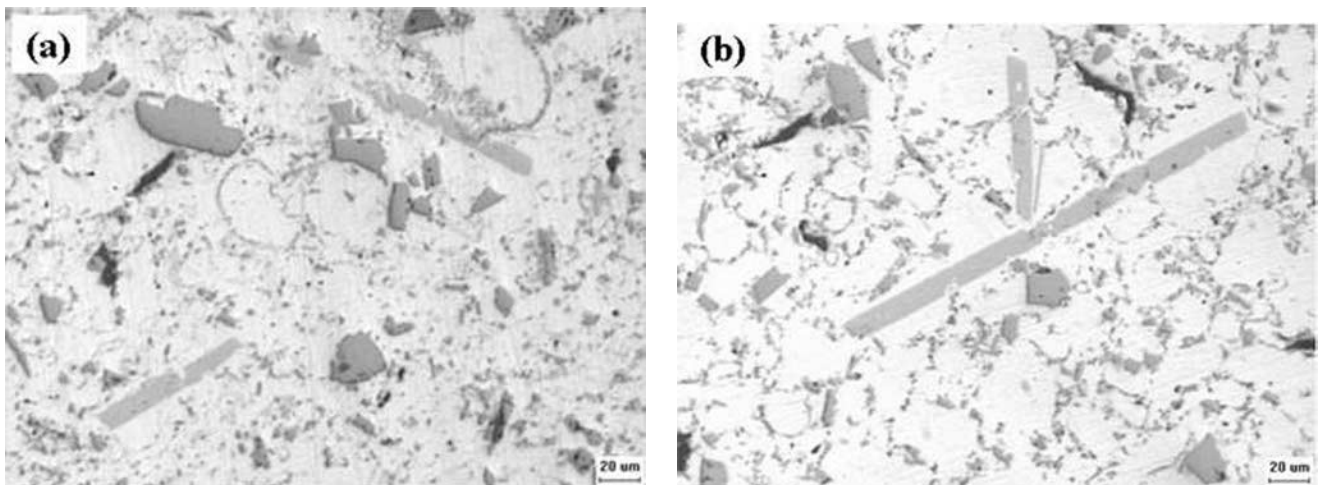


Fig. 3—Optical micrographs showing the reaction products in furnace-cooled composites at 800 °C: (a) 1 h and (b) 5 h.

stoichiometry of AlN and $MgAl_2O_4$, respectively. Both phases have been commonly observed in all the composites fabricated, using the present pressureless infiltration technique in the presence of Mg and nitrogen.^[32,33,34]

Figure 7 shows bright-field and dark-field images and selected-area diffraction patterns (SADPs) of the reaction product observed by TEM in the furnace-cooled composite. This reaction product was identified as Al_3Ti with a

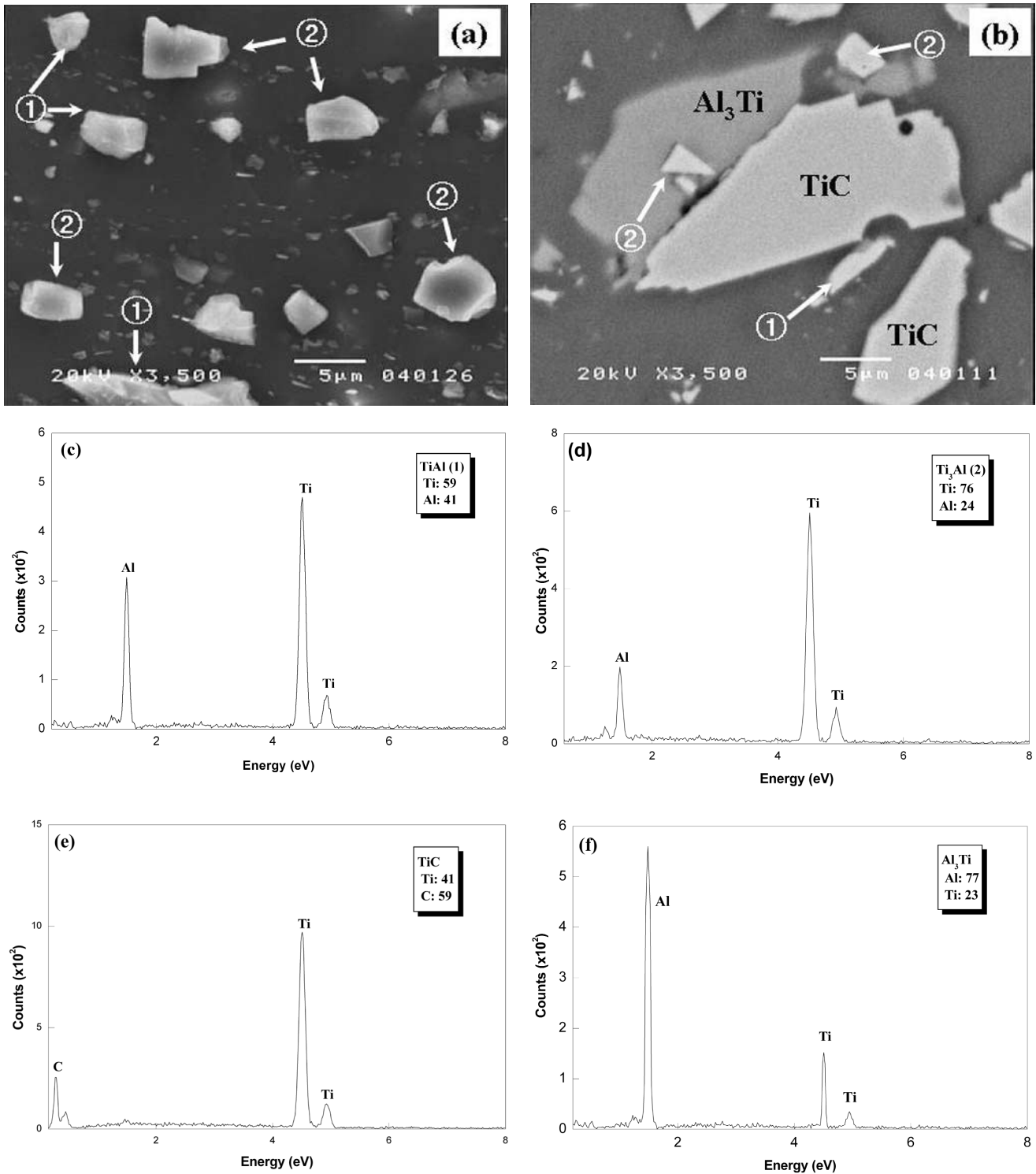


Fig. 4—SEM micrographs and EDS spectra of the reaction products obtained in a furnace-cooled composite, after dissolving away the Al alloy matrix with a solution of methanol bromine: (a) secondary electron image, (b) backscattered image, (c) TiAl , (d) Ti_3Al , (e) TiC , and (f) Al_3Ti .

tetragonal structure and lattice parameters $a = 3.7037$ nm and $c = 8.4265$ (theoretical value $a = 3.8537$ nm and $c = 8.5839$ nm, space group: $I4/mmm$).^[35]

Figure 8 shows bright-field and dark-field images and SADPs of the reaction product observed by TEM in the 5052Al/TiC composite. This reaction product was identified

as $\text{Al}_{18}\text{Ti}_2\text{Mg}_3$ with a cubic structure and lattice parameters $a = 14.7540$ nm (theoretical value $a = 14.7875$ nm, space group: $Fd3m$).^[35]

Figure 9 shows bright-field and dark-field images and SADPs of the reaction product observed by TEM in the 5052Al/TiC composite. This reaction product was identified

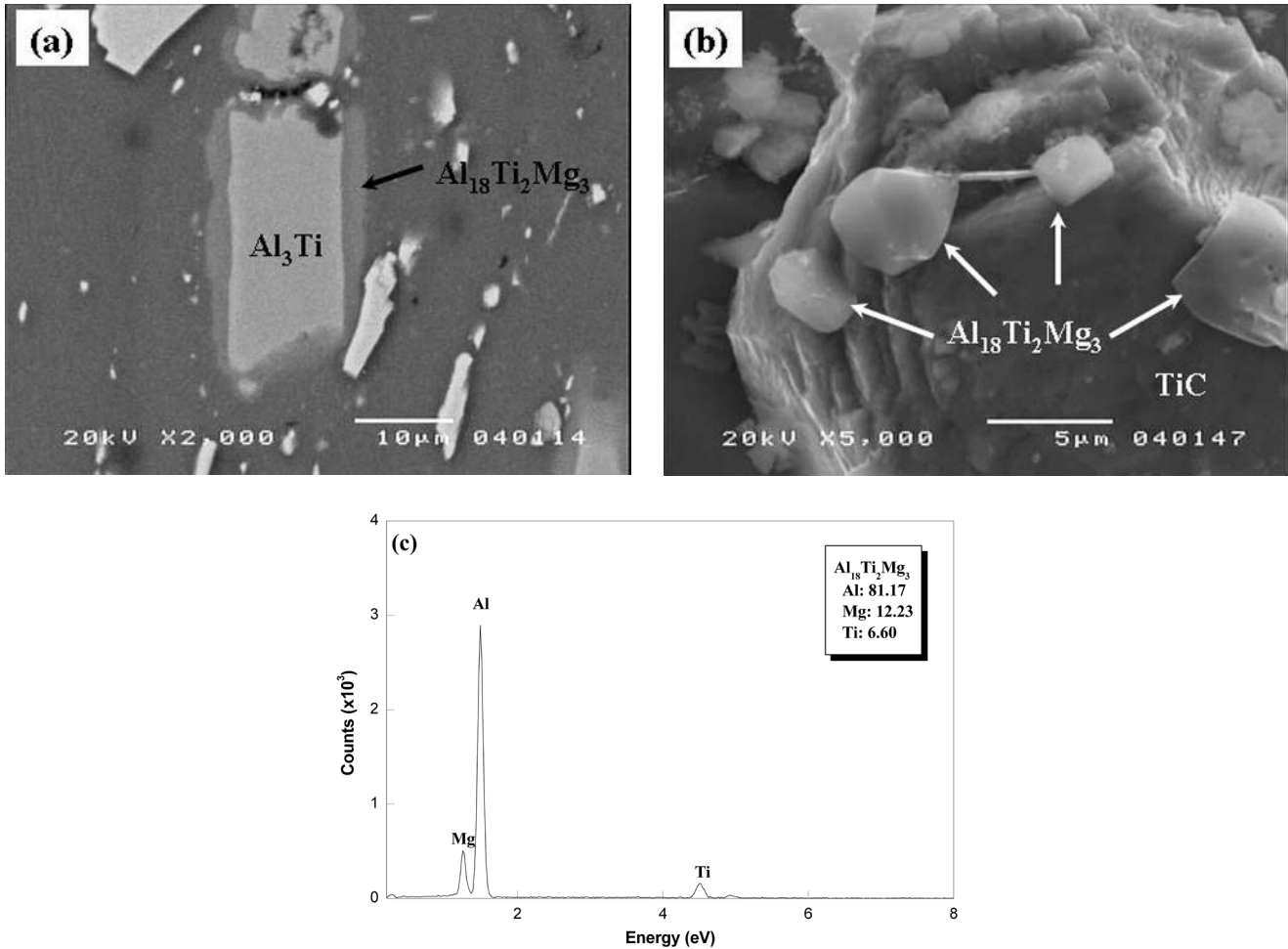


Fig. 5—SEM micrographs and EDS analysis showing the interfacial reaction products in the Al-Mg/TiC composite ($\text{Al}_{18}\text{Ti}_2\text{Mg}_3$).

as Ti_2AlC with a hexagonal structure and lattice parameters $a = 3.044 \text{ nm}$ and $c = 13.6178 \text{ nm}$ (theoretical value $a = 3.04 \text{ nm}$ and $c = 13.60 \text{ nm}$, space group: $P63/mmc$).^[35]

Figure 10 shows a secondary electron image and elemental dot-mapping images observed by SEM in the 5052Al/TiC composite. It can be seen that fine particles were formed between the Al matrix and TiC particle. Both Al and C elements were detected by dot mapping. Thus, it is believed that this reaction product is Al_4C_3 .

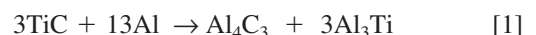
IV. DISCUSSION

The interfacial reaction between the metal matrix and reinforcement in metal matrix composites (MMCs) is very important because strong interfacial bonding permits the transfer and distribution of the load from the matrix to the reinforcement. Therefore, the nature of the interface is one of the most important factors to consider when designing a MMC.^[29] In order to evaluate its effects, first, the reaction product must be closely examine according to each fabrication condition. However, because the extent of the interfacial reaction and the type of reaction products formed are dependent on the various processing variables including temperature,

time, pressure, atmosphere, matrix composition, surface chemistry of reinforcements, *etc.*,^[31] it is difficult to determine clearly the effect of interfacial reaction on the properties of MMCs.

Similar to other Al composite systems, it was known that various reaction products were formed as a result of interfacial reaction in Al composites reinforced with TiC phases. Actually, many researchers have reported thermodynamic interpretations of phase formation (*i.e.*, Al_4C_3 , Ti_3AlC , Al_3Ti , *etc.*) in the Al-Ti-C system. There is, however, a difference in the reaction products reported in the Al/TiC composite. The disagreement in phases reported could possibly be related to the difference in fabrication routes (Table I).

While various reaction products were also observed in this study, the results were slightly different from those reported by the previous researches. It is believed that this is caused by the difference in the fabrication method, especially fabrication time (contacting time between molten metal and reinforcement) and cooling rate. Mitra *et al.*^[6] have suggested the following chemical reactions between Al and TiC:



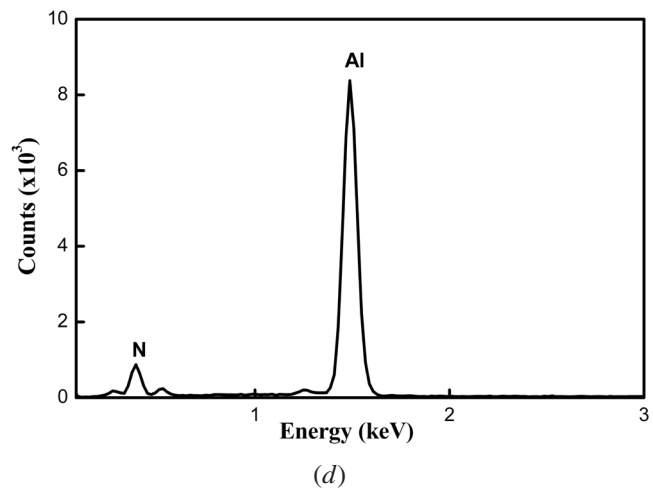
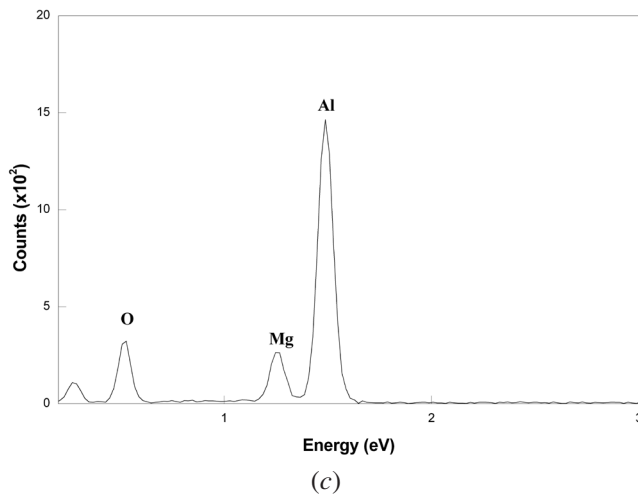
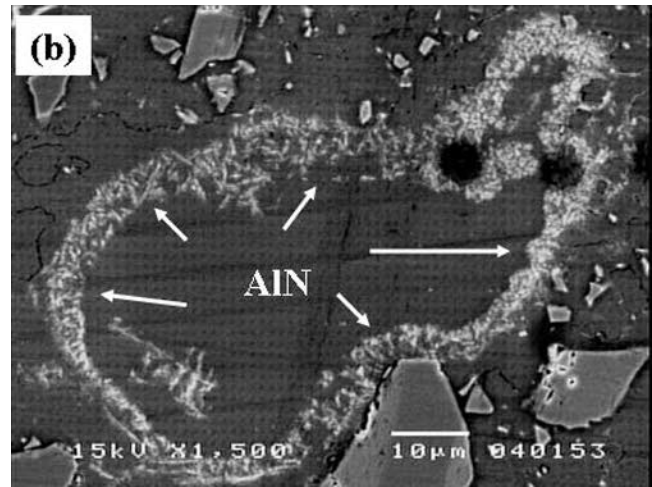
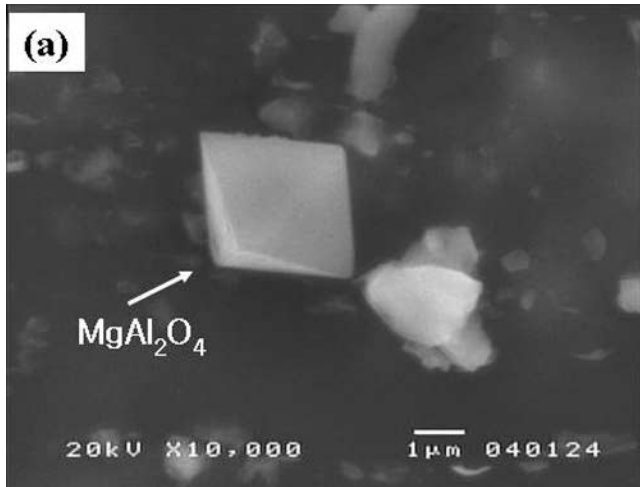
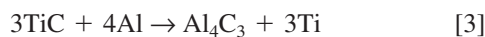
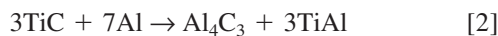


Fig. 6—SEM micrographs and EDS analysis showing the interfacial reaction products in the Al-Mg/TiC composite: (a) and (c) $MgAl_2O_4$ and (b) and (d) AlN .



They have also shown that Reaction [1] will occur only below 760 °C, because the free-energy change for this reaction is positive above 760 °C. However, for Reactions [2] and [3], the free-energy change is always positive for all temperatures. Thermodynamic calculations by Frage *et al.*^[36] indicate that a four-phase equilibrium between Al, TiC, Al_3Ti , and Al_4C_3 occurs at 693 °C and that only by heat treating below this temperature can both Al_4C_3 and Al_3Ti be produced.

As shown in Table I, while both Al_4C_3 and Al_3Ti were observed as common reaction products in Al/TiC composites, the results reported on Al_3Ti formation were not in agreement with each other. The Al_3Ti phase was not formed in the fabrication condition in which the fabrication time was too short, irrespective of temperature. However, the Al_3Ti phase was formed by heat treatment even in that case. While the cooling rate after composite fabrication was not clearly known in the literature, it seems that it has an important effect on the formation of Al_3Ti . Kennedy *et al.*^[7] reported that various reaction products were formed in the Al/TiC composite

fabricated by the stir casting method. There are no signs of reaction between the particles and the matrix in the as-fabricated composite that has only been subjected to 10 minutes at 800 °C, followed by reasonable rapid cooling during casting. However, the heat-treatment reaction between Al and TiC in the Al-10 wt pct TiC composite occurred at 600 °C in the solid state, and was still observed at 800 °C in the liquid state. Formation of the Al_3Ti was no longer observed at 900 °C for 48 hours. A maximum in the quantity of the reaction products formed, and hence the highest reaction rate, was observed at 700 °C. However, in their experiments, the samples were removed from the furnace and allowed to cool to ambient temperature after heat treatment. In addition, cooling to the solid phase (about 10 g in mass) took no longer than 1 minute. It is believed that such cooling rate is comparable to that of the water-quenched condition in this study.

In this study, coarse Al_3Ti was barely observed in water-quenched composites while it was observed at all fabrication temperatures (700 °C to 1000 °C) in the furnace-cooled condition. Albitier *et al.*^[22] reported that the slow cooling rate conditions after infiltration lead to the formation of intermetallic phases such as $CuAl_2$, $TiAl_3$, Ti_3AlC , and $AlTi_3$.

In addition, they reported that Al_3Ti had large, blocky morphology at 700 °C while its shape was changed to needle-

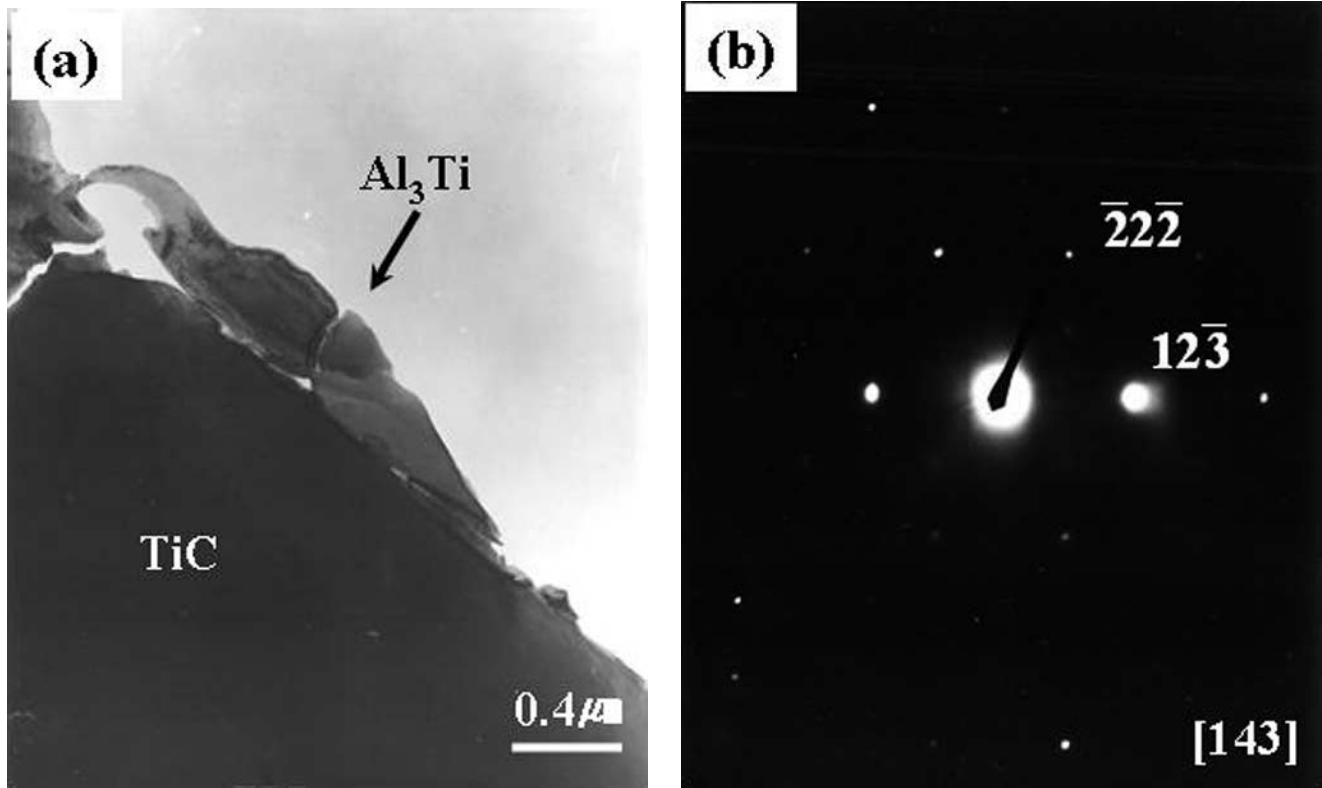


Fig. 7—TEM micrographs showing reaction products, Al_3Ti : (a) BF and (b) SADP.

like at 800 °C. They suggested that because the solubility of Ti in Al increases to 0.5 wt pct at 800 °C, large Al_3Ti blocks did not grow in the melt. In this study, however, reaction products (Al_3Ti) were formed at all temperatures ranging from 700 °C to 1000 °C and Al_3Ti of large, blocky morphology was not observed. On the other hand, while both the size and the amounts of Al_3Ti were increased with increasing fabrication temperature and time, its morphology was barely changed.

In the Al-Ti binary phase diagram, there is a peritectic reaction, $\text{Liquid} + \text{Al}_3\text{Ti} = \text{Al (solid solution)}$, at 665 °C, in which the liquid contains 0.12 to 0.15 pct Ti and the Al contains 1.2 to 1.4 pct Ti. Solid solubility decreases to 0.2 to 0.3 pct Ti at 527 °C, and liquid solubility increases regularly to 1 pct at 927 °C. By quenching from the liquid, up to 5 pct Ti can be held in solution.^[37] In this study, therefore, coarse Al_3Ti was not formed in quenched composites because of the formation of supersaturated solid solution. In addition, no significant morphology change was observed with the increase in both the temperature and time. It is believed that this result is concerned with a difference in fabrication condition. Also, the formation of other aluminides such as TiAl , Ti_3Al , and carbide (Ti_2AlC) can contribute to maintaining a lower Ti composition in the matrix, which is capable of inhibiting or retarding the formation of coarse Al_3Ti . Albiter *et al.* also reported that CuAl_2 , Al_3Ti , Ti_3AlC , and AlTi_3 were formed at an as-fabricated condition, while Al_3Ti , Ti_3AlC was formed at aging conditions.^[22]

Yang *et al.* reported the effect of the Al_3Ti phase on tensile properties in *in-situ* TiC/Al and TiC/Al-20Si-5Fe-3Cu-1Mg composites prepared by spray deposition.^[23,24] According to their results, Al_3Ti was observed to be in a

needle or rectangular structure with a mean size over 20 μm . Even though the 3 pct TiC/Al composite exhibits higher strength than the value of unreinforced aluminum, the tensile elongation was only 4 pct, much lower than the value for unreinforced aluminum, 20 pct. They found that the presence of needlelike Al_3Ti compounds can be eliminated entirely from the final product by using a proper Ti:C molar ratio of 1:1.3 in the Ti-C-Al preform. The tensile elongation of a composite without the Al_3Ti phase was 10 pct, much higher than the value of the composite with Al_3Ti (4 pct). Moreover, improvement in tensile elongation of the composite was accompanied by an increase in the ultimate tensile strength. On the other hand, Pandey *et al.* reported the effect of isothermal heat treatment at 623 °C for various lengths of time from 24 to 171 hours on an Al-20 vol pct TiC_p composite produced by the conventional P/M route.^[25] In the heat-treatment specimens, two additional phases (Al_3Ti and Al_4C_3) were observed, unlike in the as-extruded specimen. They suggested that the formation of these phases seems to be responsible for the increase in the creep strength of the composite on heat treatment. However, these reaction products were finer than the TiC particles (1.4 μm).

In this study, microvickers hardness testing was conducted in the composite after final polishing. As shown in Figure 11, the size of indentation is different in both the Al matrix and the reaction products. Namely, the hardness of the reaction products is higher than that of the Al matrix; then, those reaction products may affect mechanical properties of the composite. Therefore, it is desirable to prevent the formation of Al_3Ti or to control the size of Al_3Ti .

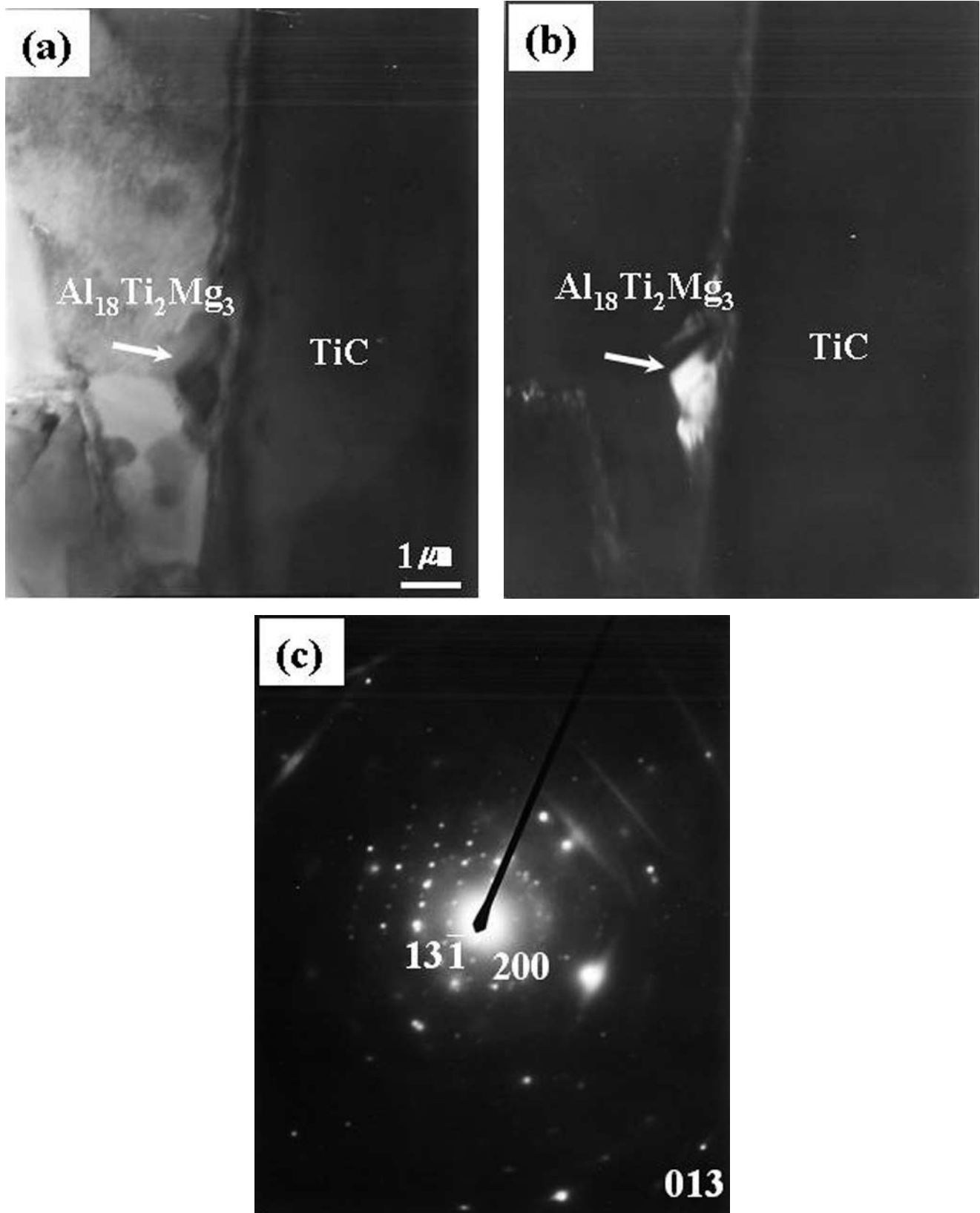


Fig. 8—TEM micrographs showing the reaction product, $\text{Al}_{18}\text{Ti}_2\text{Mg}_3$: (a) BF, (b) DF, and (c) SADP.

Nukami and Flemings^[14] reported that TiAl was not found at any temperature. However, there were many reports that Al_3Ti was formed even above 760°C . In addition, it could

be seen that Al_3Ti was formed by interfacial reaction between the Al alloy and TiC particles even though composites were fabricated at 800°C in this study.

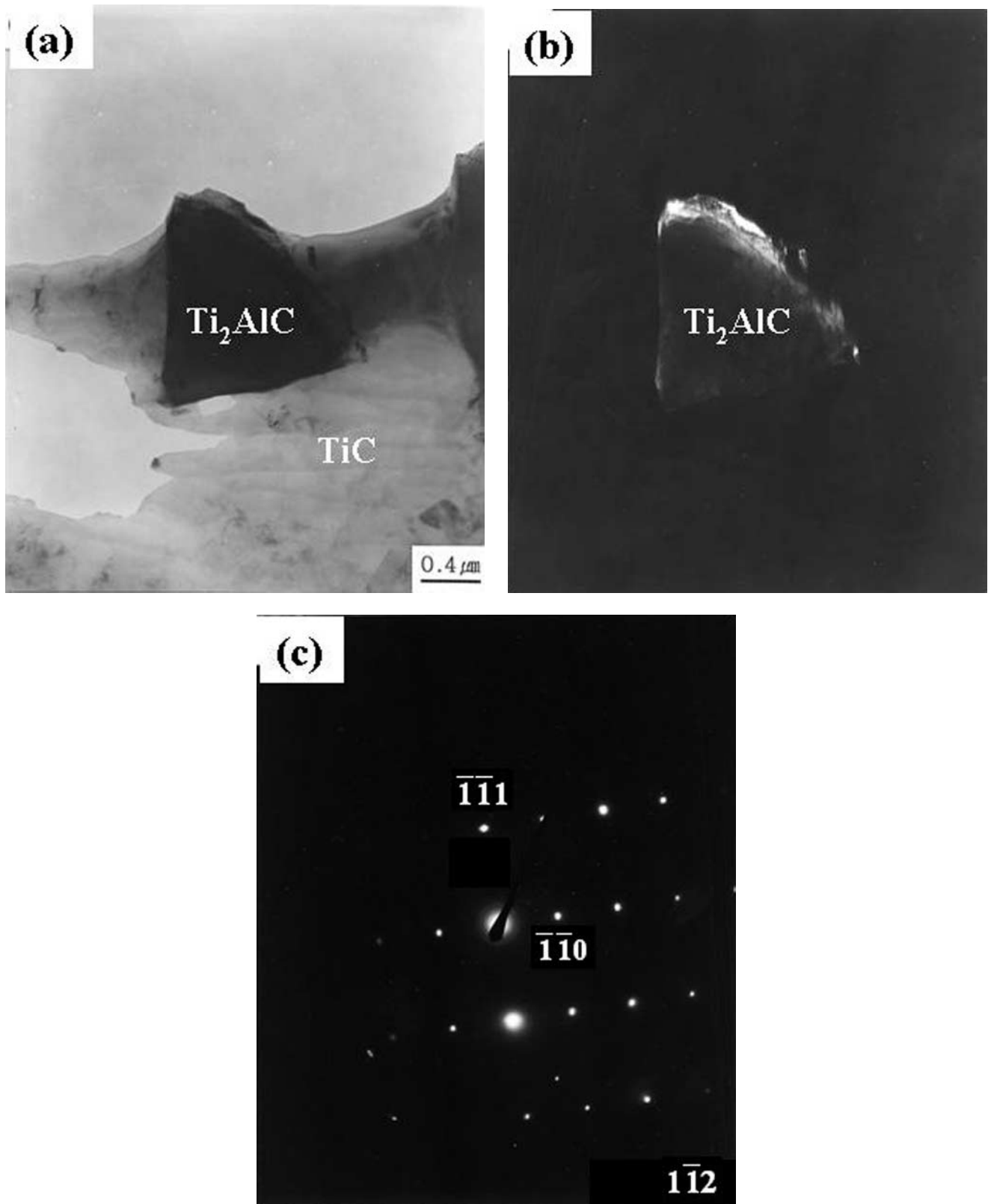


Fig. 9—TEM micrographs showing the reaction products, Ti_2AlC : (a) BF, (b) DF, and (c) SADP.

It is also suggested that the Ti_3AlC phase with the perovskite (*cP5*) structure forms according to the reaction



and is present below 1000 °C as a metastable phase intermediate between Al_3Ti and TiC .^[26]

However, Ti_2AlC , known as H phase, was observed while the perovskite carbide phase was not found in this study.^[30]

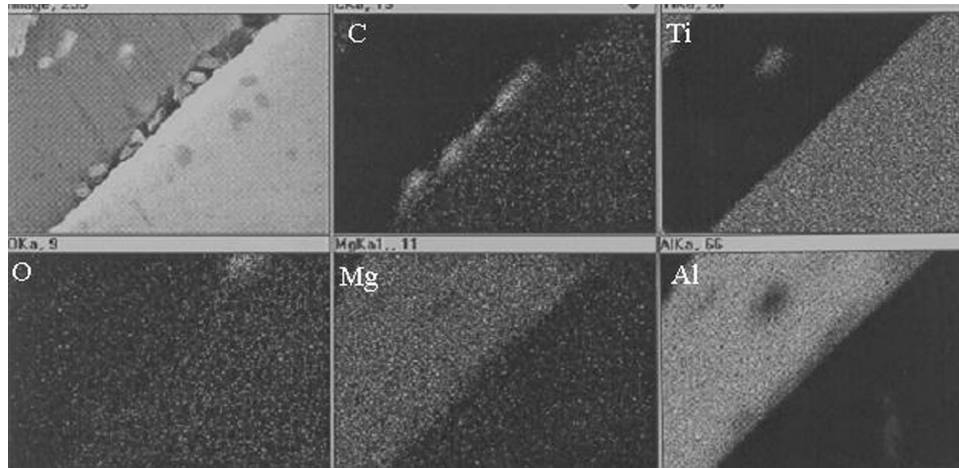


Fig. 10—Secondary electron image and elemental dot-mapping images observed by EDS showing Al_4C_3 .

Table I. Reaction Product Reported in Al/TiC Composites

Fabrication Method	Reaction Products	Reference
TiC (20 pct, 1.4 μm): PM-HR	HR—no reaction product	28
600 °C (24 to 96 h) HT-FC	HT— Al_4C_3 , Al_3Ti	
TiC (15 pct, 0.7, 4.0 μm): XD	AF—no reaction product	6
640 °C (24 to 496 h) HT	HT— Al_4C_3 , Ti_2AlC , Al_3Ti , TiAl	
TiC (20 pct, 1.4 μm): PM-EX (550 °C)	AF—no reaction product	25
623 °C (24, 96, 171 h) HT	HT— Al_4C_3 , Al_3Ti	
TiC (1.2 μm), sessile drop	Al_4C_3 , TiAl_3 (at 800 °C)	29
Al-Mg (1, 4, 8, 20 pct), Ar 750 °C to 900 °C	no TiAl_3 (at 900 °C)	
DERP (900 °C)	MgAl_2O_4 , MgO	
Spray deposition	TiAl_3 , Ti_3AlC_2	17
Al-2024/TiC (1.12 μm , 55 pct)	Al_4C_3 , Al_3Ti	23, 24
Infiltration	AF— TiAl_3 , Ti_3AlC , AlTi_3	22
1200 °C, 2 h (Ar)	HT— TiAl_3 , Ti_3AlC	
CR 12 °C/min to 15 °C/min		
TiC (10 wt pct, 45, 18 μm), stir casting	AF—no reaction product	7
Melt contact for 10 min at 800 °C	700 °C 48 h—blocky Al_3Ti , Al_4C_3	
600 °C to 900 °C 48 h HT	800 °C— Al_4C_3 , Al_3Ti (needle)	
At 700 °C, 1 to 240 h AC	900 °C—no Al_3Ti	

HR: hot rolling, HT: heat treatment, FC: furnace cooling EX: extrusion,
DERP: dripping exothermic reaction process, CR: cooling rate, and AC: air cooling.

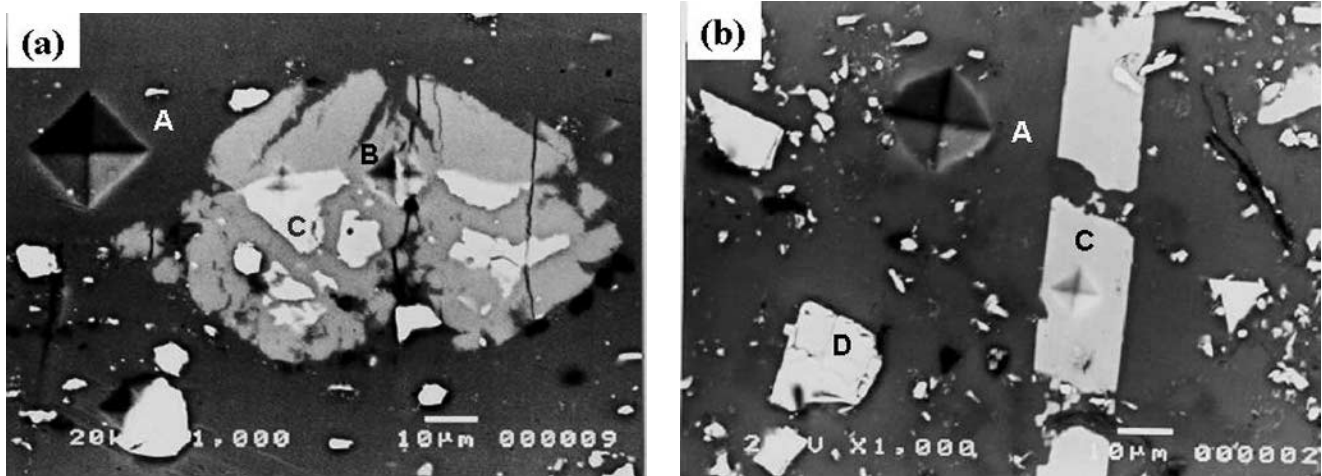


Fig. 11—SEM micrographs showing the Microvickers hardness indentation observed at various reaction products: Al-Mg matrix (A), $\text{Al}_{18}\text{Ti}_2\text{Mg}_3$ (B), Al_3Ti (C), and TiC (D).

Mitra *et al.*^[6] have suggested that Ti₂AlC was formed by the following reaction:



They have proven theoretically that Ti₂AlC is thermodynamically more stable than TiC in the presence of TiAl.^[28] Thus, the presence of TiAl may promote formation of Ti₂AlC. The Ti₂AlC possibly was formed by the preceding reaction, as the TiAl phase was also observed in this study.

V. CONCLUSIONS

The interfacial reaction products of the Al-Mg/TiC_p composite fabricated by the pressureless infiltration method were analyzed using SEM, EDS, and TEM. During the fabrication of composites, reaction products with various morphologies and sizes were formed in the Al matrix as well as in the vicinity of the TiC particles by the interfacial reaction between the Al alloy and the TiC particles. From the EDS and SADP analyses, Al₄C₃, Al₁₈Ti₂Mg₃, Ti₂AlC, Al₃Ti, and TiAl were identified as being formed as interfacial reaction products. Both the size and the amount of the reaction products were increased with increasing fabrication temperature as well as fabrication time. Coarse Al₃Ti was barely observed in water-quenched composites, while it was observed at all fabrication temperatures (700 °C to 1000 °C) in furnace-cooled conditions.

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