

## SiC/Aluminum Composites Fabricated by *in situ* Processing using Reactive Infiltration Aid

Makoto Kobashi<sup>1</sup>, Masaru Hirako<sup>2</sup>, Naoyuki Kanetake<sup>1</sup>

<sup>1</sup>Department of Materials Science and Engineering, Nagoya University, 1 Furo-cho, Chikusa-ku, Nagoya  
464-8603, Japan

<sup>2</sup>Graduate Student, Graduate School of Engineering, Nagoya University, 1 Furo-cho, Chikusa-ku, Nagoya  
464-8603, Japan

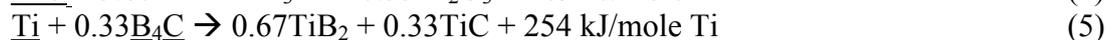
Pressureless infiltration, as one of the fabrication methods of metal or intermetallics matrix composites, is an attractive process on account of its cost-effectiveness and capability of producing near-net-shape and high ceramic fraction composites. However, good wettability between a solid powder preform and a liquid metal is required to realize the spontaneous infiltration. In this paper, the effect of blending the reactive infiltration aid in the SiC powder preform on the spontaneous infiltration of molten aluminum was investigated. As the reactive infiltration aid, titanium, nickel, titanium oxide (TiO<sub>2</sub>), nickel oxide (NiO) and boron carbide (B<sub>4</sub>C) powders were selected. The infiltration aid was blended with SiC powder by various volume fractions (0~40vol%), and then compacted to make a preform. The compacted powder preform and an aluminum ingot were set in a crucible, and heated in argon gas atmosphere. Molten aluminum successfully infiltrated into the powder preform by adding the infiltration aids except for NiO and TiO<sub>2</sub>. After infiltration, aluminum matrix composites with dispersions of SiC and *in situ* formed particles were obtained. Both processing temperature and time were important parameters to reduce the residual voids. The SiC powder tended to decompose vigorously when nickel powder was used as the infiltration aid, whereas such decomposition was not observed when titanium or (Ti+B<sub>4</sub>C) powder was selected.

**Keywords:** Pressureless infiltration, silicon carbide, heat of reaction, combustion reaction, thermite reaction.

### 1. Introduction

A demand for producing a low-density and high-stiffness material is increasing. For example, high specific stiffness, good tribological performance and high wear resistance are simultaneously required for a brake disk of high speed vehicles. High thermal conductivity and precisely-controlled thermal expansion are required in the field of semiconductor related materials such as heat sinks and heat spreaders. However, it is difficult to satisfy such demands by a monolithic material, and therefore manufacturing technology for ceramic/metal composites with high ceramic fraction becomes important. One of the prominent processing methods of producing metal matrix composites is a squeeze casting. This process can be much more simplified if the infiltration takes place without applying pressures on a molten metal. The reactive infiltration aims at realizing the spontaneous infiltration with a help of both good wettability and high heat of reaction [1-5]. Fig. 1 shows a schematic illustration of the reactive infiltration process of [SiC + infiltration aid] powder preform with molten aluminum. Silicon carbide powder is blended with an infiltration aid powder and compacted to make a preform. An aluminum ingot is located on the preform and the whole sample is heated. After aluminum melts, an exothermic reaction between aluminum and the infiltration aid occurs and molten aluminum infiltrates into the preform. As the infiltration aid, titanium, nickel, titanium oxide (TiO<sub>2</sub>), nickel oxide (NiO), boron carbide (B<sub>4</sub>C) powders were used. The following reactions were expected by using the infiltration aid (underlined elements are the infiltration aids).





The roles of the infiltration aid powder are considered as shown below

- (i) Good wettability between the infiltration aid and molted aluminum assists the infiltration (for titanium and nickel only).
- (ii) Heat of reaction between the infiltration aid and molten aluminum raises the local temperature at the infiltration front and makes the wettability between SiC and molten aluminum better (for all infiltration aids).

In this paper, we investigated the capability of spontaneous infiltration and the microstructure of the infiltrated composites to understand the efficiency of the infiltration aids.

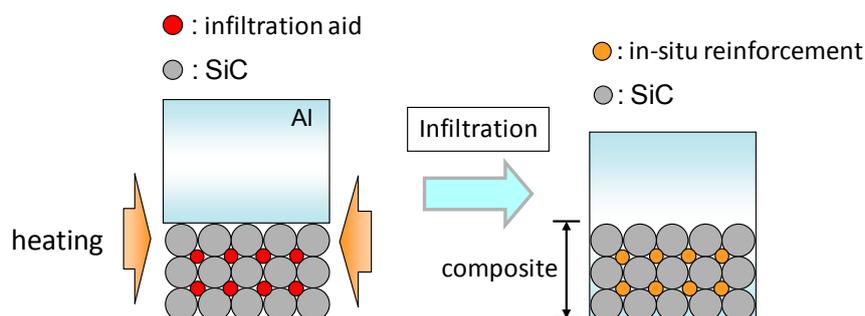


Fig. 1 Reactive infiltration process proposed in this research.

## 2. Experimental procedure

As infiltration aids, titanium (ave. 10 $\mu\text{m}$ , 99.9%up), nickel (4-7 $\mu\text{m}$ , 99.8%), TiO<sub>2</sub> (<45 $\mu\text{m}$ , 99.9%), NiO (<45 $\mu\text{m}$ , 99%), B<sub>4</sub>C (ave. 10 $\mu\text{m}$ , 99%up) powders were used in this experiment. B<sub>4</sub>C powder was used with titanium powder as indicated in Eq. 5. The SiC powder (150 $\mu\text{m}$ ) and the infiltration aid were blended by various blending ratios (volume ratio of the infiltration aid in the preform: 0~40vol%). The blended powder was compacted at room temperature by 200MPa to make a preform (relative density of the preform: 0.65-0.70). The preform was inserted in a crucible as shown in Fig. 2, and an aluminum ingot was located on the preform. The specimen was then heated up to a processing temperature (700, 1000°C) by an induction furnace in Ar gas atmosphere. The specimen was cooled in the furnace after being held at the processing temperature for 0~7200s. The vertical cross-section of the specimen was observed by scanning electron microscope (SEM). The quantitative analysis of the specimen was carried out by energy dispersive X-ray analysis (EDX) and X-ray diffraction analysis (XRD).

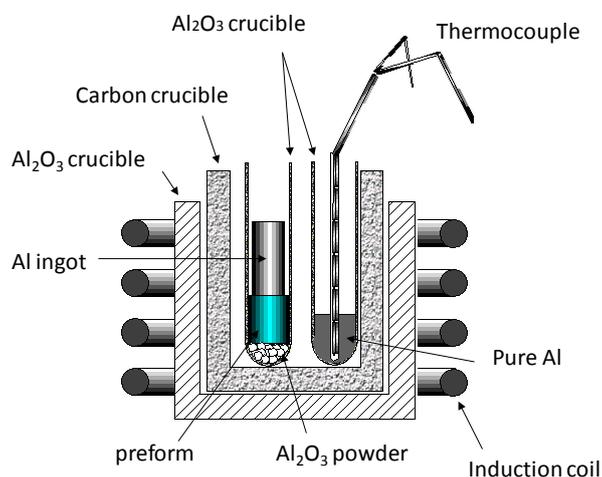


Fig. 2 Schematic illustration of the experimental apparatus.

### 3. Results and discussion

#### 3.1 Spontaneous infiltration of molten aluminum

First of all, SiC powder preform without an infiltration aid addition was supplied to the infiltration experiment. As a result, molten aluminum did not infiltrate at all after being held at 1000°C for 3600s. Then, effects of infiltration aids on the capability of spontaneous infiltration were evaluated by adding 20vol% titanium, nickel, TiO<sub>2</sub>, NiO and (Ti+B<sub>4</sub>C) in the SiC preform. Fig. 3 shows the macroscopic vertical cross-sections of the infiltrated specimens. The spontaneous infiltration did not occur at all when NiO was added. The infiltration of TiO<sub>2</sub> added specimen was not completed as shown in Fig. 3. Titanium, nickel, (Ti+B<sub>4</sub>C) powders turned out to be effective to assist the infiltration, even though the heats of thermite reactions (Eqs. 3 and 4) are much higher than those of the reactions between elemental powders (Eqs. 1 and 2). Therefore, it can be concluded that both good wettability and high heat of reaction are essential to realize the spontaneous infiltration.

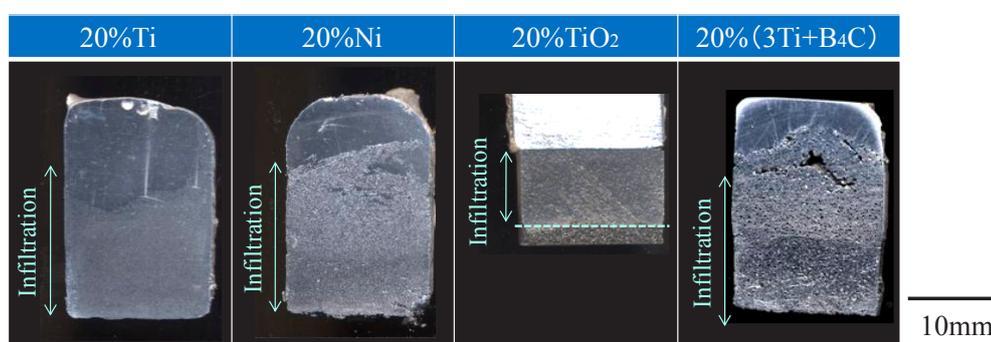


Fig. 3 Vertical cross-sections of the infiltrated specimens with various infiltration aids.

The amount of infiltration aid in the preform was varied for Ti and Ni added specimens. The cross-sections are shown in Fig. 4. With regard to the Ti added specimens, the residual pores are apparently observed by adding 10vol%. This might be caused by the insufficient improvement of wettability. As for the 40vol%Ti specimen, an explosive reaction occurred and the preform was swollen as shown in Fig. 4. It is known that the Al-Ti intermetallics expand when they are fabricated by the combustion reaction [6]. Therefore, by increasing the titanium amount in the preform, the influence of the expansion of the Al-Ti intermetallics became prominent. When nickel powder was used, the expansion of the preform did not occur even by adding 40vol%. However, non-infiltrated part shown by a dotted circle in Fig. 4 appeared. The reason for this infiltration defect will be discussed in the following section.

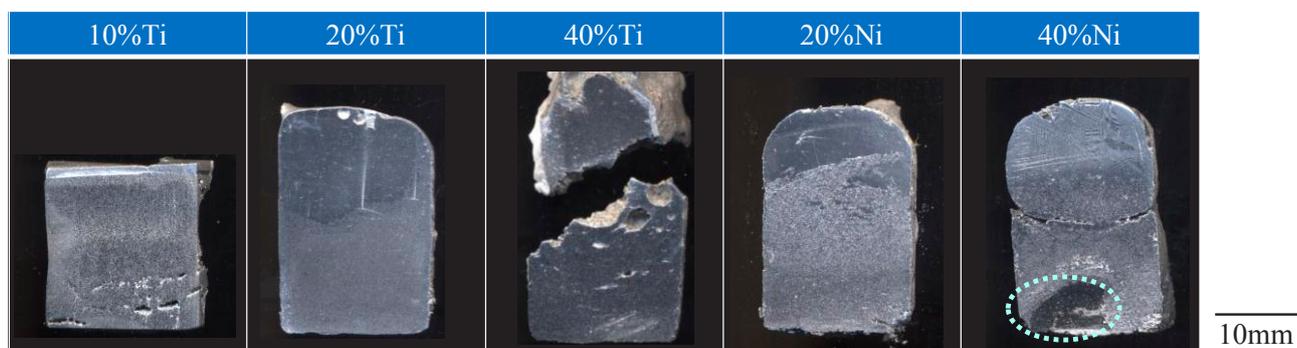


Fig. 4 Cross-sections of the specimens with various amounts of titanium and nickel powder additions.

Table 3 shows the residual porosity of the specimens fabricated by various conditions. Titanium addition was the most effective for reducing residual porosity. However, as mentioned in the previous section, the additive amount of the infiltration aid must be moderate. Increasing the holding time was an effective method to reduce the porosity.

Table 3 Residual porosity of the specimens with various infiltration aids.

		Mixing ratio			
		20Ti-80SiC	20Ni-80SiC	20(3Ti+B <sub>4</sub> C)-80SiC	20TiO <sub>2</sub> -20Al-60SiC
Holding time	0min	1.0%	12.4%	3.6%	×
	30min	0.9%	2.1%	1.7%	7.2%
	60min	0.3%	1.1%	2.0%	6.2%
	120min	0.7%	1.2%	1.1%	4.8%

### 3.2 Microstructure of infiltrated composites

The microstructure of the composites (infiltration aid: titanium and nickel) are shown in Fig. 5. It is obvious that aluminum penetrated into the gap between SiC particles, and reaction products were dispersed in aluminum matrix. Large intermetallics (Al<sub>3</sub>Ni) were seen only in the Ni added specimen. Such large intermetallics were not seen in the Ti added specimens. Fig. 6 shows the microstructure change of Ni added specimens with holding time (holding temperature: 1000°C). Significant degradation of SiC can be seen even without a holding period. The degradation became more remarkable by holding the specimen for 3600s at 1000°C. EDX and XRD analysis revealed that the reaction product in this specimen was Ni<sub>2</sub>Si. The melting point of Ni<sub>2</sub>Si is 980°C and, therefore, melted during the holding period at 1000°C. Hence, SiC decomposed rapidly in molten aluminum at 1000°C. As mentioned in the previous section, the infiltration did not occur at the bottom part of the specimen when Ni was used. Around the non-infiltrated zone, a large quantity of Ni<sub>2</sub>Si was observed. This means that the decomposition of SiC occurred at the same time as the infiltration, and the concentration of nickel and silicon became very high at the bottom part of the specimen. This concentrated nickel and silicon might prevent the reaction between aluminum and nickel. In contrast to Ni added specimens, a stable reaction product (TiC, melting point: 3170°C) was formed at the interface between SiC and matrix of Ti added specimens (Fig. 7). This reaction product at the interface was not concentrated at the bottom due to its high melting point, and acted as a protective layer for SiC particle, and, therefore the decomposition of SiC was not prominent in Ti added specimens. The reaction product of TiO<sub>2</sub> and (Ti+B<sub>4</sub>C) added specimens were (i) Al<sub>2</sub>O<sub>3</sub> and Al<sub>3</sub>Ti and (ii) TiC and TiB<sub>2</sub>, respectively. Similar to the Ti added specimen, the degradation of SiC was not observed in these specimens.

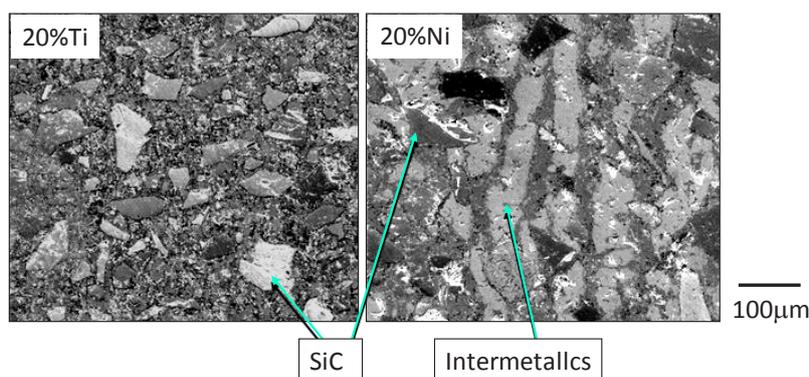


Fig. 5 Microstructure of Ti and Ni added specimens.

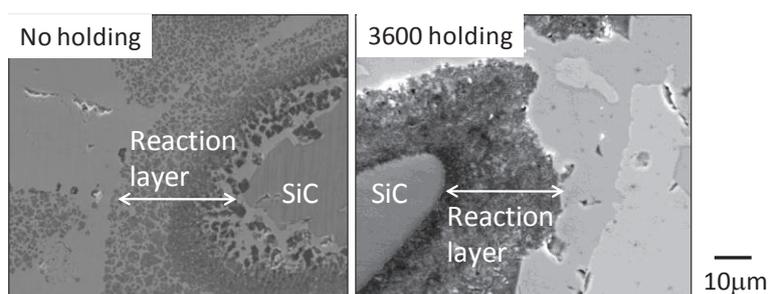


Fig. 6 Microstructure of Ni added specimens (20vol%Ni), showing degradation of SiC particle.

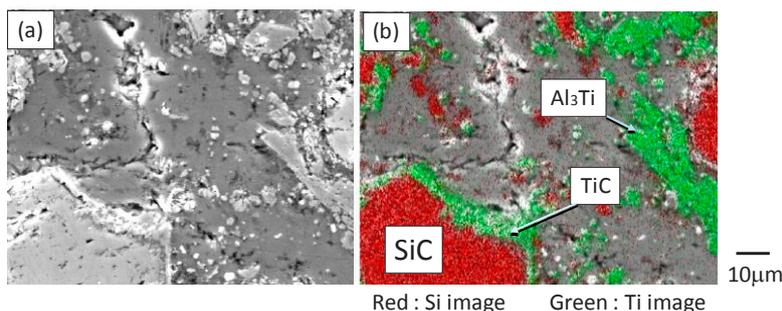


Fig. 7 Microstructure of Ti added specimens (20vol%Ti), showing TiC formation around SiC.

#### 4. Conclusion

The capability of several kinds of infiltration aids for producing SiC/Al composites was investigated. The residual porosity and microstructure of the infiltrated composites were studied and the following results were obtained.

- (1) Titanium, nickel, (Ti+B<sub>4</sub>C) powders turned out to be effective as the infiltration aid. Infiltration did not occur at all by NiO addition, nor completed by TiO<sub>2</sub> addition. Both good wettability and high heat of reaction were desirable to realize spontaneous infiltration.
- (2) With regard to Ti added specimens, residual pores were observed remarkably by adding 10vol%. An explosive reaction occurred and the preform swelled by adding 40vol%. With regard to the Ni added specimens, infiltration did not occur by adding 10vol%. By increasing the Ni addition to 40vol%, non-infiltrated sections appeared. A moderate amount of titanium and nickel addition to realize spontaneous infiltration was 20vol%. Titanium addition was the most effective for obtaining low-porosity specimens.
- (3) Increasing the holding time was an effective method to reduce the porosity in the specimen.
- (4) With regard to Ni added specimens, significant degradation of SiC was observed.

#### References

- [1] T. Choh, T. Mohri and M. Kobashi: J. Mater. Proc. Tech. 63 (1997) 379-383.
- [2] M. Kobashi and T. Choh: J. Mater. Sci. 32 (1997) 6279-6282.
- [3] N.Omura, M.Kobashi and N.Kanetake: J. Japan Inst. Metals 68 (2004) 211-215.
- [4] N.Omura, M.Kobashi and N.Kanetake: J. Jap. Inst. Metals 68 (2004) 367-371.
- [5] D. C. Dunand, J. L. Sommer and A. Mortensen: Metal. Trans. 24A (1993) 2161-2170.
- [6] C.L. Yeh and Y.G. Shen: J. Alloy. Compd. 470 (2009) 424-428.