REACTIVE SYNTHESIS OF TiC/Al COMPOSITE AND ITS MECHANICAL PROPERTIES

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ABSTRACT The synthesis of titanium carbide (TiC) in molten aluminum was attempted using a reaction between titanium and carbon. A powder blend consisting of titanium, carbon and aluminum was located in the bottom of a crucible, and an aluminum ingot was placed above the powder blend. During the starting materials were heated by an infrared furnace, the powder blend was infiltrated with molten aluminum; and the reaction between titanium and carbon provided the TiC/Al composite. The time required for the complete conversion of titanium and carbon to TiC was significantly shortened by using the compacted powder blend. Vickers hardness of the TiC/Al composite was higher than that of monolithic aluminum.

Keywords: *metal matrix composite, reactive infiltration, combustion reaction, titanium carbide, aluminum*

1. INTRODUCTION

The infiltration of a ceramic preform with molten metals has been used as one of the processing routes for aluminum matrix composites [1,2]. Generally, non-reactive metal-ceramic systems have been used in this infiltration technique in order to prevent the degradation of the ceramic phase. Materials obtained by this process basically consist of a continuous metal matrix and discontinuously dispersed ceramic particles. Reactive systems have begun to be introduced into the infiltration process recently [3,4]. Since the ceramics are synthesized *in situ*, this process can provide a continuous ceramic matrix [5]. This is one of the outstanding features of this technique in that a bulk ceramic can be synthesized at relatively lower temperatures in a short time. Another advantage is that the highly reactive combination of a ceramic with a molten metal is in many cases wettable [6,7], which enables a spontaneous infiltration.

In this work, titanium, carbon and aluminum powders were used as the starting materials, and the spontaneous infiltration of the powder blend with molten aluminum was attempted. As a result of the reaction between titanium and carbon shown below;

$$Ti + C \rightarrow TiC \qquad (1)$$

titanium carbide (TiC) is synthesized in molten aluminum.

The aim of this work is to synthesize TiC/Al composites via the infiltration route, and to this end the reactive infiltration of a compacted [Ti + C + Al] powder blend with molten aluminum is dealt with in this paper. We particularly focus on (i) observation of the composite microstructure and (ii) investigation of the effect of the aluminum addition on the reaction kinetics.

2. EXPERIMENTAL PROCEDURE

2.1 Infiltration process

The starting materials used in this work were titanium powder (99.8%Ti, with a particle size under 44μm), carbon powder (99.7%C, with a particle size of 5μm) and aluminum powder (99.8%Al, with a particle size under 45μm). The schematic illustration of the experimental setup for the spontaneous infiltration is shown in **Fig.1**. For the preparation of the starting material, the titanium and carbon powders were mixed with aluminum powder (Ti : C : Al molar ratio = 1:1:0-4.0). As is shown in Fig.1, the powder blend was placed at the bottom of the alumina (Al_2O_3) crucible (inner diameter: 13mm). An aluminum ingot was then placed on the powder blend. The chamber was evacuated using a rotary pump and backfilled with nitrogen gas (99.9% pure). The specimen was then heated up to the processing temperature in an induction furnace, and held at this temperature for 0-7200s. After the holding period, the specimen was cooled in the furnace and removed from the crucible. The vertical cross-section was observed and analized by scanning electron microscopy (SEM), an electron probe X-ray microanalysis (EPMA) and X-ray diffraction (XRD) techniques.

2.2 Differential thermal analysis

Titanium, carbon and aluminum powders were mixed with the aluminum powder at molar ratios of 1:1 (Ti:C) and 1:1:1 (Ti:C:Al). The powder blends were consolidated by applying a pressure of 1000MPa and a piece of the compact was probed using differential thermal analysis (DTA). The analysis was conducted at a heating rate of 10K min^{-1}.

3. RESULTS

3.1 Advantage of using reactive system

As is described in the introduction, the chemical reaction was used to synthesize TiC as a reinforcement for aluminum. To confirm the advantage of using the reactive infiltration technique, the infiltration behaviors of molten aluminum into TiC powders and the [Ti + C (Ti:C=1:1)] powder blend was compared. The result is shown in **Table 1**. The spontaneous infiltration of the [Ti + C] powder blend with molten aluminum occurred during the 3600s holding at 1473K; however, the TiC powder was not infiltrated by molten aluminum under the same experimental condition. **Figure 2** shows the temperature profile of the molten aluminum measured by a thermocouple inserted in the crucible during the heating process. The dotted line in the figure, which indicates

the temperature profile of the specimen with the [Ti + C] powder blend, shows a relatively steeper increase in the temperature between points A and B. This indicates that an exothermic reaction took place during this period and made the temperature of the specimen higher. The reasons for using the [Ti + C] powder blend enables the spontaneous infiltration are considered to be twofold:

1. The exothermic reaction raises the temperature of the whole system, which makes the wettability between aluminum and carbon better.
2. Titanium powder becomes a path for molten aluminum's infiltration.

Figure 3 (a) and (b) shows cross-sections of the specimens made from the [Ti + C] powder blend with different holding times at 1473K ((a):3600s hold, (b):7200s hold). It is seen in the Fig.4 (a) that Al_3Ti is formed in the aluminum matrix, indicating the reaction shown in Eq.1 had not been completed after a 3600s hold. However, with a 7200s hold, the conversion of titanium into TiC had been completed and a homogeneous dispersion of the TiC particles are clearly visible (Fig.4 (b)).

3.2 Synthesis from compacted powder blends

In the previous section, the advantage of using the [Ti + C] powder blend was discussed. The disadvantage of this process is that it requires more than a 3600s hold at 1473K, which could not be considered as a low enough temperature nor short enough processing time for the fabrication of the aluminum-based material. A compacted powder, therefore, was used instead of the loose powder blend in order to induce a combustion reaction. **Figure 4** shows a cross-section of the specimen made from a compacted powder blend (Ti:C:Al molar ratio = 1:1:1). During fabrication process, this specimen was heated only up to the melting point of aluminum (933K) and was not held at this temperature. In Fig.4, three-dimensionally continuous TiC formed in the aluminum matrix is visible. This indicates that using a compacted powder blend enables the synthesis of TiC phase only by heating the system to the melting pint of aluminum. The synthesis of TiC was also tried from a [Ti + C] compacted powder (Ti:C molar ratio = 1: 1) under the same experimental condition (processing temperature: 933K, holding time: 0s). The observation of the cross-section revealed that the formation of TiC had not occurred but, instead, Al_3Ti and carbon were observed.

In order to confirm the difference between the compacted [Ti + C + Al] and [Ti + C] powder blends, differential thermal analysis was carried out on these powder blends; and the results are shown in **Fig.5**. According to Fig.5, the [Ti + C + Al] powder blends shows an endothermic peaks(around 933K) followed by a sharp exothermic peak (TiC formation) whereas no clear peaks were found from the [Ti + C] powder blends. Considering the results denoted in the above, it is understood that the TiC formation from titanium and carbon can not be achieved at 933K without blending the aluminum powder.

Aiming at investigating the effect of the aluminum addition and its optimum additional ratio on the possibility of combustion reaction, six powder blends with different additional ratios of aluminum were prepared (Ti:C:Al molar ratio = 1:1:0.5-4.0). The compacted powder blends (Diameter: 10mm, Thickness: 10mm) were located on a heater and heated to induce the combustion

reaction. **Figure 6** shows the temperature profile of the six powder blends. In this experiment, temperatures of the specimens were measured by a thermocouple embedded in each compacted powder. A large amount of heat that could not be measured by the thermocouple was released in cases where Ti:C:Al molar ratios were 1:1:0.8 and 1:1:1. In these specimens, the formation of TiC was observed after the combustion reaction; however, only relatively smaller increases in temperatures were detected when molar fraction of aluminum was 0.5, 0.7, 2.0 and 4.0. Thus, the optimum range of aluminum addition is roughly confirmed to be between 0.7 and 2.0.

3.3 Vickers hardness

Vickers hardness of the specimens made from the loose [Ti + C (Ti:C = 1:1)] powder blend and the compacted [Ti + C + Al (Ti:C:Al = 1:1:1)] powder blend was measured. The Vickers hardness of each specimen is shown in **Fig. 7**. The hardness of the specimen fabricated from the compacted [Ti + C + Al] powder blend shows a higher value in comparison with the others. The higher volume fraction of TiC and the 3-dimensionally-continuous morphology of TiC are considered to contribute to the higher hardness.

SUMMARY

The spontaneous infiltration of the powder blend consisting of titanium and carbon with molten aluminum occurred; therefore, the TiC/Al composite was successfully fabricated. Due to the combustion reaction initiating at the melting point of aluminum, the rapid synthesis of TiC became possible by mixing the aluminum powder with the compacted [Ti + C] powder blends. The Vickers hardness of the specimen made from the compacted powder showed the higher value in comparison with the one made from the loose powder blends.

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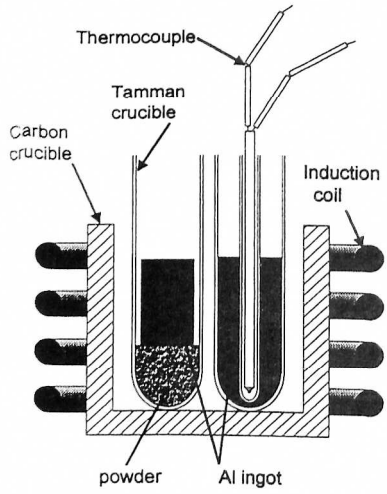

Fig.1 Schematic illustration of the experimental setup.

Table 1 Possibility of spontaneous infiltration.

Starting Powders	
TiC powder	[Ti+C] powder
Non-infiltrated	Infiltrated

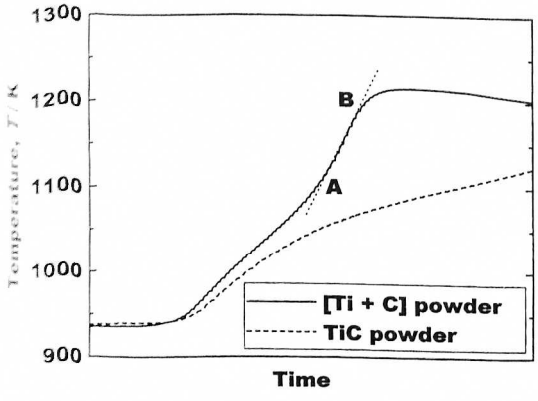

Fig.2 Temperature profile of molten aluminum during the heating process.

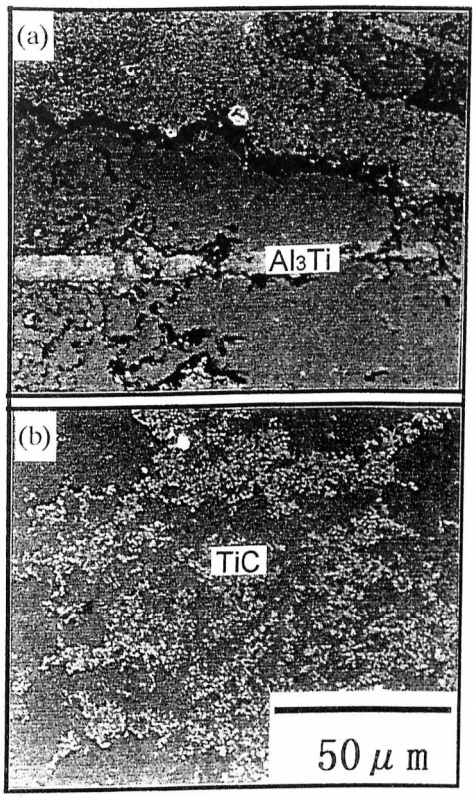

Fig.3 Scanning electron micrographs of the specimens with (a) 3600s hold and (b) 7200s hold at 1473K.

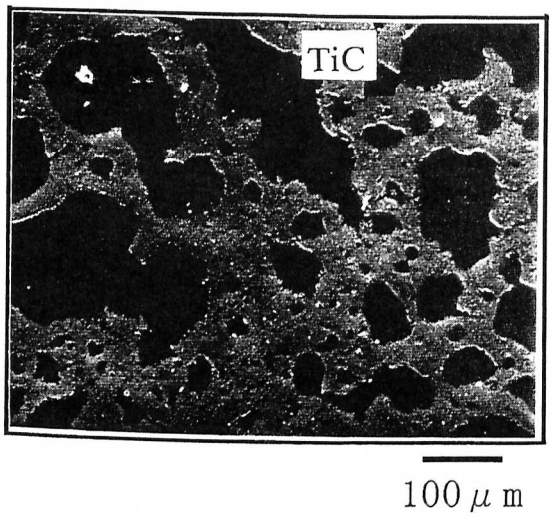

Fig.4 Scanning electron micrograph of the specimen made from a compacted powder.

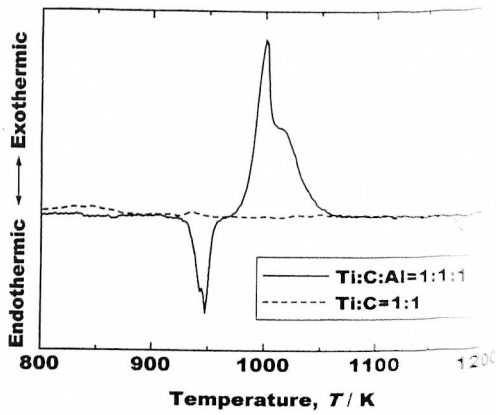

Fig.5 DTA results carried out on the [Ti+C+Al] and [Ti+C] powders.

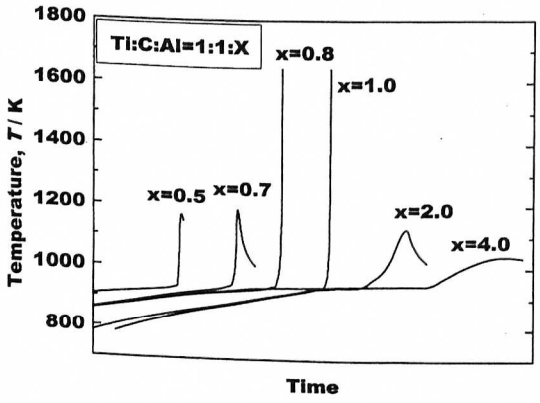

Fig.6 Temperature profiles of the powder blends with various aluminum additional ratios.

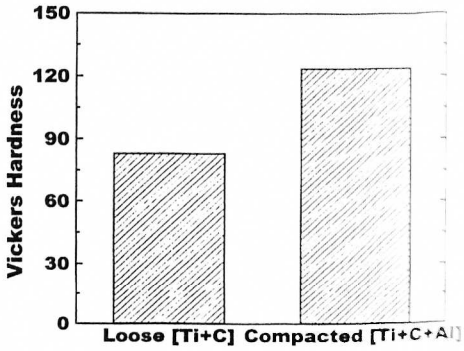

Fig.7 Vickers hardness of the *in situ* composites.