FABRICATION OF NITRIDE CERAMICS COMPOSITE BY REACTIVE INFILTRATION

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Abstract

Reactive infiltration is an innovative manufacturing process of metal matrix composites with low cost and low environmental impacts. In this study, reactive infiltration of a (Ti+TiN) blended powder preform with molten aluminum was examined. Titanium powder as an infiltration aid was mixed with TiN powder by various blending ratios. The preform and aluminum ingot were heated together up to 1273K - 1673K by an induction furnace in N_2 gas atmosphere. Spontaneous infiltration of molten aluminum into the blended powder did not occur when either processing temperature or blending ratio of titanium as infiltration aid was not high enough. Spontaneous infiltration took place when processing temperature was 1673K. But when molar ratio of the TiN/Ti was 19/1 or higher, the preform was not completely infiltrated by molten aluminum even at 1673K. It was confirmed that Al_3Ti, AlN was formed after the infiltration was completed.

1 Introduction

Ceramic/metal composites have been investigated for decades on account of their high modulus, yielding stress and creep resistance [1-3]. Many processing routes have been proposed for the fabrication of this kind of material. Although the liquid-metal infiltration process has been proposed industrially for automotive applications [4,5], the process can be further simplified if the infiltration of powder preforms with molten metals occurs spontaneously. The schematic illustration of reactive infiltration process is shown in **Fig1.** The following aspects are the benefits of the reactive infiltration process.

- *cost-effective process*
- *wide range of materials selection*
- *capability of near net shape production in one step*
- *high volume fraction of reinforcement*

However, good wettability between the solid powder and the liquid metal is required in spontaneous infiltration process. The combination between titanium and molten aluminum shows good wettability. Therefore, by using titanium as infiltration aids, the spontaneous infiltration of a titanium nitride (TiN) and titanium blended powder preform with molten aluminum can be expected. The reactions between titanium and aluminum can be written as

$$Ti + 3\,Al = Al_3Ti + 146kJ \qquad (1)$$

Spontaneous infiltration is promoted by making use of this exothermic reaction. In this experiment, the following reaction is expected and aluminum nitride (AlN) and titanium aluminide (Al_3Ti) are synthesized.

$$Ti + x\,TiN + (4x+3)\,Al \rightarrow (x+1)\,Al_3Ti + x\,AlN \qquad (2)$$

As a result of the reactions, production of Al_3Ti, AlN/Al composite with high volume fraction of nitride ceramics is expected. The aim of this study is to search a possibility of the fabrication of nitride ceramics composite by the reactive infiltration of the blended powder preform of titanium and TiN with molten aluminum. In the presentation, effect of processing parameters (processing temperature, perform powder composition) on the infiltration capability and microstructure are mainly dealt with.

2 Experimental procedures

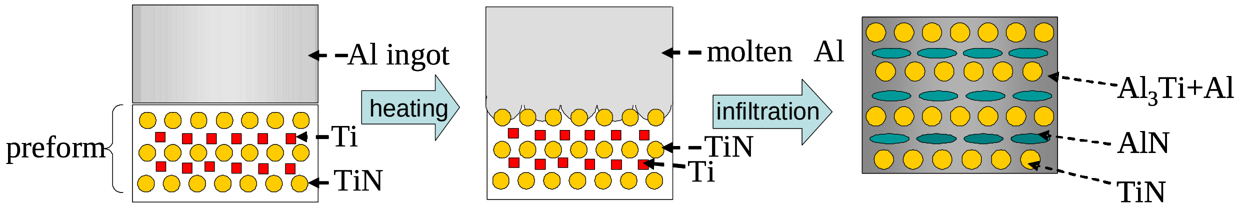

Fig.1.Schematic illustrations of reactive infiltration process

TiN powder (average size: 52μm) and titanium powder (average size: 45μm) were used as starting materials. TiN powder was selected because it releases nitrogen and synthesizes AlN when reacted. However, wettability between the TiN powder and molten aluminum is not good enough to realize spontaneous infiltration. Then, titanium powder was mixed with TiN powder by various blend ratios. In this paper, the blended powder of titanium and TiN is expressed as a [Ti+TiN] blended powder. Then, the [Ti+TiN] blended powder was compacted under a pressure of 200MPa to make a preform. The shape of perform was columnar and its diameter and height were 10mm and 15mm, respectively. The compacted blended powder (4g) was located at a bottom part of an alumina (Al_2O_3) crucible, and an aluminum ingot (6g) was located on the blended powder as shown in **Fig.2**. The specimen was then heated up to 1273K~1673K by an induction furnace in N_2 gas atmosphere. After the heating process, the specimen was cooled and taken out of the crucible, and the vertical cross section was observed. Microstructures of the composites were examined by scanning electron microscope (SEM). A qualitative analysis was carried out by the X-ray diffraction (XRD) method.

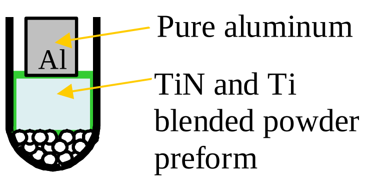

Fig.2. Schematic illustration of the experimental set-up

3 Results and discussion

3.1 Effect of the processing temperature

Fig.3. shows vertical cross sections of specimens fabricated at various temperatures and molar blending ratios of TiN/Ti. spontaneous infiltration of molten aluminum into the [Ti+TiN] blended powders did not occur when processing temperature was 1273K or 1473K and molar blending ratio of TiN/Ti was 8 or 9. However, the infiltration was realized on condition that the processing temperature was 1673K and molar blending ratio of TiN/Ti was 8 or 9. The XRD data of the specimen (TiN/Ti=9, processing temperature: 1673K) were shown in **Fig.4.** It was confirmed form the XRD results that AlN and Al_3Ti were formed, although residual TiN still existed. Microstructure of the specimen (TiN/Ti=9, processing temperature: 1673K) is shown in **Fig.5**. The light gray part in the figure was confirmed as Al_3Ti, the black particles were AlN and the white particles were confirmed as residual TiN by an X-ray micro-analyzer. From Fig.4, the conversion of TiN and titanium to Al_3Ti and AlN was confirmed.

3.2 Effect of TiN/Ti powder blending ratio

Fig.6. is vertical cross section of several specimens with various molar blending ratios of TiN/Ti. The infiltration was realized on condition that the processing temperature was 1673K and molar blending ratio of TiN/Ti was below 17. But when molar blending ratio was over 19 and the processing temperature was 1673K, spontaneous infiltration didn't occur or stopped before completion. When molar blending ratio was low, there were many pores in the specimen. This is because the large amount of titanium addition resulted in increasing the exothermic heat of reaction [equations (1)] and raised the specimen temperature became more than an adequate level. When molar blending ratio of TiN/Ti was high, spontaneous infiltration of molten aluminum into the [Ti+TiN] blended preform went well.

3.3 Effect of the holding time

When molar blending ratio of TiN/Ti was 19 or 20 and processing temperature was 1673K without holding time, spontaneous infiltration stopped

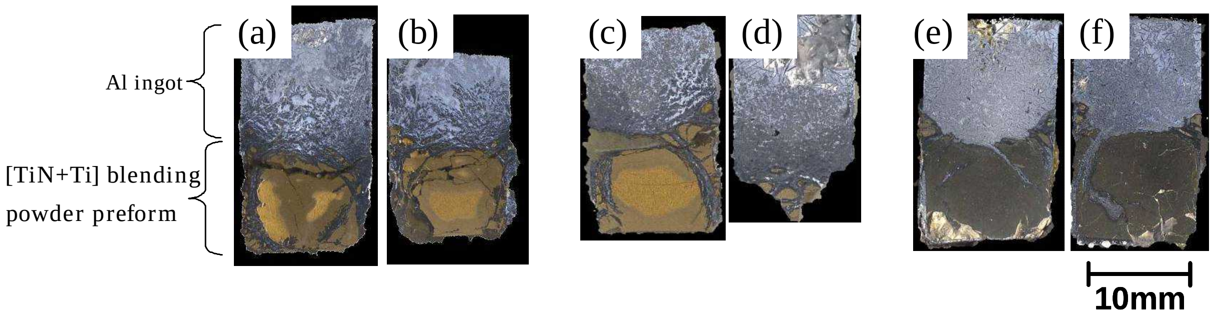

Fig.3.Vertical cross section of several specimens in several process temperatures. (a)1273K, TiN/Ti=8 (b)1273K, TiN/Ti=9 (c)1473K, TiN/Ti=9 (e)1673K, TiN/Ti=8 (f)1673K, TiN/Ti=9

before completion. Then, holding time (1 hour) was given to these specimens. Infiltration of molten aluminum into the [TiN+Ti] blended perform was confirmed. Vertical cross section of these specimens with 1 hour holding is shown in **Fig.7.** And microstructures of these specimens are shown in **Fig.8**.

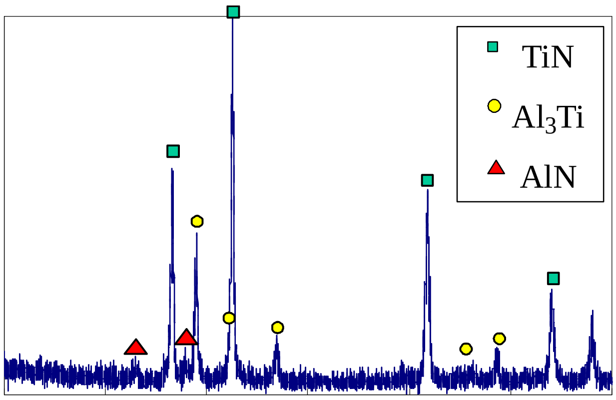

Fig.4.XRD data of the specimen in 1673K (TiN/Ti=9)

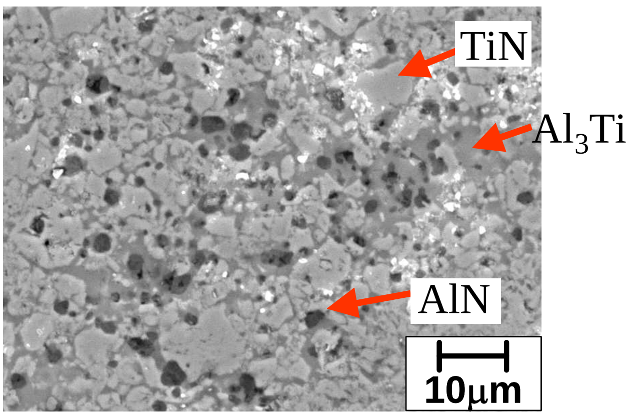

Fig.5. Microstructure of the specimen in 1673K (TiN/Ti=9**)**

4. Conclusions

Blended powders of [Ti+TiN] were infiltrated with molten aluminum at various temperatures and various molar blended ratios of the TiN/Ti powder to produce nitride ceramic composites. Spontaneous infiltration of molten aluminum into the blended powders did not occur when either processing temperature or blending ratio of titanium as infiltration aid was not high enough. Spontaneous infiltration took place when processing temperature was 1673K. But when molar ratio of the TiN/Ti was 19/1 or higher, the preform was not completely infiltrated by molten aluminum even at 1673K. It was confirmed that AlN, Al_3Ti and residual TiN was formed after the infiltration. As a result, nitride

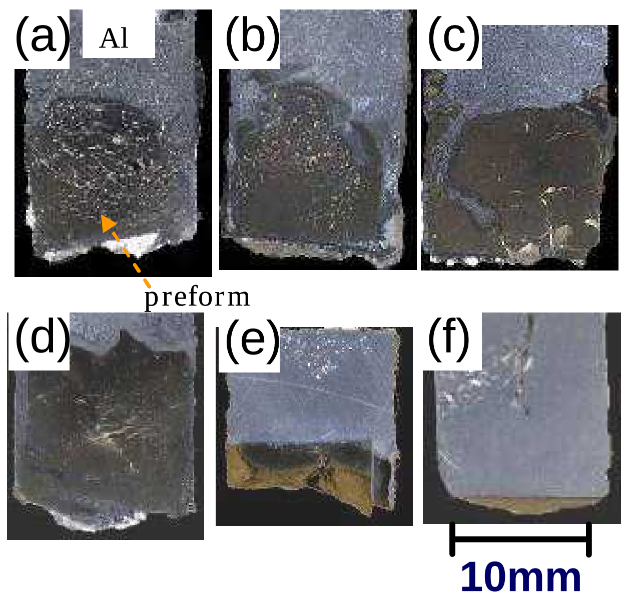

Fig.6. Vertical cross sections of the various molar blended ratios of the TiN/Ti in 1673K for 0 hour. (a) TiN/Ti=0.93, (b) TiN/Ti=5, (c) TiN/Ti=9, (d) TiN/Ti=17, (e) TiN/Ti=19, (f) TiN/Ti=20

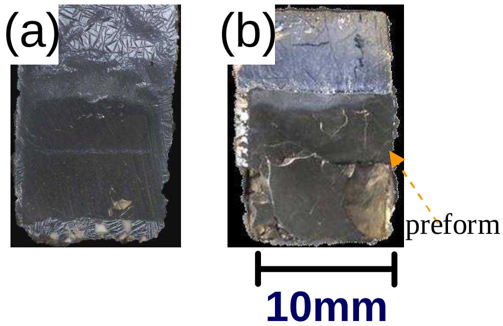

Fig.7. Vertical cross sections of the composite fabricated at 1673K with 1 hour holding. (a) TiN/Ti=19, (b) TiN/Ti=20

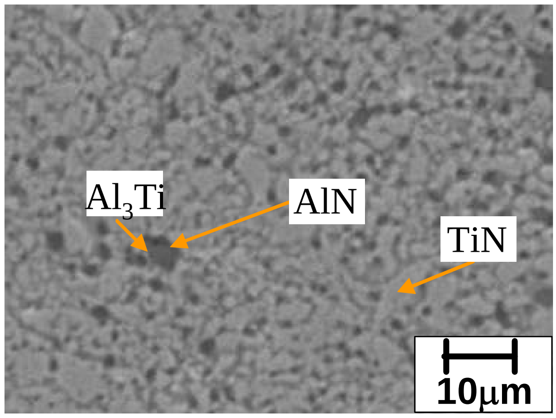

Fig.8. Microstructure of the composite fabricated at 1673K with 1 hour holding. (TiN/Ti=19)

ceramics composite could be fabricated by reactive infiltration.

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