Effect of B$_4$C Size on Tensile Property of (TiB+TiC) Particulate Reinforced Titanium Matrix Composites by Investment Casting

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The aim of this research is to evaluate the microstructure and tensile property of in-situ particulate (TiB+TiC) reinforced titanium matrix composites (TMCs) synthesized by investment casting process. Different size of B$_4$C (1500, 150 and 0.5 μm) were added to the titanium matrix during vacuum induction melting which can provide the in-situ reaction of $5\text{Ti} + \text{B}_4\text{C} = 4\text{TiB} + \text{TiC}$. The tensile property of TMCs was investigated in accordance with the reinforcement distribution by B$_4$C size. The size and morphology of in-situ synthesized reinforcements by fine B$_4$C were very minute and distributed homogeneously than coarse B$_4$C. Moreover, the improvement of the tensile strength and ductility of TMCs was caused by not only load transfer strengthening but also the matrix grain refinement and homogeneous distribution of the reinforcements.

Keywords: titanium, composites, in-situ, tensile, microstructure

(Received March 18, 2011; Accepted July 5, 2011; Published August 24, 2011)

1. Introduction

Titanium matrix composites (TMCs) have several attractive characteristics, such as elastic modulus, high temperature property, wear and oxidation resistance. However, it is adopted in limited area, such as aerospace and automobile, because of its high manufacturing cost and extreme affinity in molten state. The particulate reinforced TMCs were more desirable compared to the continuous reinforced TMCs owing to low cost process and isotropic property. There are several candidates for the reinforcements as TMCs, but TiB and TiC have an outstanding compatibility with the titanium matrix, because of the similar density, analogous coefficient of thermal expansion.

Despite extensive work on the manufacturing of the particulate reinforced TMCs, previous researches were mostly based on the powder metallurgy and some are casting process. However, most of manufacturing processes have a common problem with the matrix/reinforcement interface reaction and agglomerations which cause the deterioration of mechanical property. To overcome these drawbacks, in-situ synthesis method was developed. This technique, which known as the reaction process between the matrix and specific adding element, can assure the clean interface between the matrix and reinforcement and homogeneous distribution of the reinforcements.

In this research, therefore, we adopt to the investment casting process for the economical considerations. In addition, in-situ synthesis method also developed to ensure not only homogeneous distribution but also controlled interfacial reaction between the matrix and reinforcements. Boron carbide (B$_4$C) was added to the titanium melts using vacuum induction melting which can provide the in-situ reaction of $5\text{Ti} + \text{B}_4\text{C} = 4\text{TiB} + \text{TiC}$.

2. Experimental Procedure

The mold for TMCs was prepared by the investment casting process, so called lost-wax method. Prepared mold was mounted in the vacuum induction melting furnace. After that, B$_4$C (99% purity, 1500 μm, 150 μm and 0.5 μm) was added to pure Ti (99% purity, Grade 2), following which the synthesis of the TMCs was carried out using vacuum induction melting. The pressure of the furnace atmosphere was held at $1.33 \times 10^{-1} \text{ Pa}$ and charged with inert argon gas at a pressure of $4.9 \times 10^{2} \text{ Pa}$. The oxide mold was superheated sufficiently to overcome the problem of low viscosity of TMCs, and then 1.88 mass% B$_4$C was added to the pure Ti in the graphite crucible in order to form the 10 vol% of (TiB+TiC) reinforcement. The melts were poured into the mold by centrifugal force with 10G.

The melts was maintained 70 seconds after beginning of the in-situ synthesis, and the pouring temperature was 2023 K. The in-situ synthesis of the reinforcement in the Ti matrix was investigated using electron probe micro-analyzer (Shimazu EPMA 1600) and scanning electron microscope (HITACHI S-3000H). EBSD (Electron Back-scattered Diffraction) was performed on sectioned samples to evaluate the grain size of pure Ti and TMCs.

The tensile tests of TMCs were conducted in a MTS 810 universal testing machine. Each tensile specimen was machined according to the ASTM E8 subsize which was given a gage length of 25 mm and gage width with 6 mm. Specimens were machined with grip regions 17 mm wide by 35 mm long at each end of the sample. The TMCs specimens of this investigation were subjected to mechanical testing by B$_4$C size (1500, 150 and 0.5 μm) under the initial strain rate of 0.001 s$^{-1}$ at room temperature, and the values shown are the average of at least 10 measurements.

3. Results and Discussion

3.1 Microstructure observation of in-situ particulate (TiB+TiC) reinforced TMCs

In previous research, the reinforcements of in-situ synthe-
sized TMCs by investment casting process were composed of the needle like TiB and spherical TiC. The microstructure characteristics in TMCs examined in this research are the reinforcement size according to the different B₄C size. As seen in Fig. 1, there is a dramatic change in the reinforcements. In-situ synthesized reinforcements by 1500μm B₄C were very fine and distributed homogeneously than 1500μm B₄C. However, several problems were occurred in 0.5μm B₄C. Reinforcements were not fully synthesized since the nano-sized B₄C particles were agglomerated in Ti melts as shown in Fig. 1(d), and the reinforcements were distributed inhomogeneously. In EPMA element mapping images as shown Fig. 2 and 3, the morphology and size of needle like TiB and spherical TiC were clearly distinguished. In the case of needle like TiB, the size was radically reduced, however, spherical TiC was unable to discriminate between 1500 and 150μm B₄C.
150 \mu m B_4C. Meanwhile, in Fig. 4, it can be seen that the agglomerated B_4C were under *in-situ* reaction with Ti matrix. Thus, the size and morphology of *in-situ* reinforcements were quite different according to the B_4C size of 1500, 150 and 0.5 \mu m. In the case of the conventional manufacturing process of MMCs, the size and morphology of reinforcements was maintained except for the interfacial reaction between the matrix and reinforcement since the reinforcements were added directly to the matrix. However, the reinforcements were synthesized by the chemical reaction between the matrix and specific additive. For this reason, the size, morphology and distribution of the reinforcements by *in-situ* reaction were considerably influenced by the process parameters such as the reaction time, superheating and cooling rate. Especially, despite of maintaining the same process parameters above-mentioned, the size, morphology and distribution of the *in-situ* reinforcements by 150 \mu m B_4C shows a clear distinction compared with 1500 \mu m B_4C. From these results, it is believed that the fine B_4C particles promote the increase of the nucleation site of the reinforcements by *in-situ* reaction.

### 3.2 Effect of B_4C size on the tensile property of *in-situ* particulate (TiB+TiC) reinforced TMCs at room temperature

Figure 5 indicates the tensile property of TMCs according to the reinforcement size. The TMCs produced by fine B_4C significantly improves the tensile elongation, and the strength by a small margin. Especially, the tensile elongation of TMCs by 150 \mu m B_4C were about 2 times higher than 1500 \mu m B_4C. 0.5 \mu m B_4C has lowest values of tensile strength and elongation which were caused by the incomplete *in-situ* reaction owing to agglomeration of B_4C. In the view point of the tensile strength improvement, the conventional strengthening mechanisms of MMCs were divided mainly three parts such as the shear-lag strengthening, dislocation strengthening and orowan strengthening. However, among these mechanisms, orowan strengthening were effective not micron scale but nano scale. In additions, in the case of the dislocation strengthening, it is difficult to quantify the dislocation density between the reinforcements, and it is expected that the increase of dislocation density by the coefficient of thermal expansion were insignificant since the coefficient of matrix and reinforcements were similar value (Ti: 4.51, TiB: 4.5 and TiC: 4.95 g/cm^3). Therefore, in this research, we focused on the shear-lag strengthening which were based on the load transfer from the matrix to reinforcements. According to the Cox and Nardone, the tensile strength was improved by the load transfer phenomena. To predict the tensile strength by load transfer strengthening, the mean size and aspect ratio of the reinforcements of TMCs by 1500 and 150 \mu m B_4C were measured using IMAGE PRO PLUS software. The TiB/TiC reinforcements of TMCs by 1500 \mu m have the mean diameter of 6.90 \mu m/7.79 \mu m, with aspect ratio of 2.48/1.95, respectively. In the case of 150 \mu m, the mean diameter is 4.76 \mu m/5.56 \mu m, with aspect ratio of 2.08/1.75. Meanwhile, since the reinforcements of TMCs by 0.5 \mu m were not fully synthesized, it is hard to analyse the typical mean size and aspect ratio of reinforcement. In addition, for the shear-lag calculation, 448 MPa was used as the yield strength of matrix. Figure 6 indicates the results of the calculated and experimental tensile strength of the TMCs by 1500 and 150 \mu m. Comparing the calculated tensile strength of the TMCs by different B_4C size, there are little difference between 1500 and 150 \mu m B_4C. Besides, experimental tensile strength was much higher than calculated value by the load transfer strengthening mechanism. It is hard to explain the effect of the reinforcement size on the tensile strength and elongation. Therefore, we focused on the grain refine effect on the tensile property as well as load transfer strengthening. From the well known Hall-Petch relation, the yield stress is inversely proportional to the grain size which was based on the concept that grain boundary acts as barrier to dislocation motion. Since it is very difficult to distinguish the matrix grain size using general metallographic method, EBSD analysis was adopted to measure the matrix...
grain size. This method can obtain exact information of the specific phase based on the crystal orientation by diffraction pattern. Figure 7 shows a typical EBSD mapping images of the pure Ti and TMCs. In the case of microstructure (Fig. 1), the influence of fine B$_4$C on microstructure (especially reinforcement) was ascertained clearly. Additionally, EBSD mapping images provide the evidence of the refinement of the alpha-Ti matrix grain. The measured average of grain size of the alpha-Ti matrix of the TMCs by 1500 and 150 μm are, as shown Fig. 8, about 17.0 and 12.6 μm respectively, while pure Ti is about 104.3 μm. Therefore, we conclude that the improvement of tensile property is obtained by the matrix grain refinement and homogeneous distribution as well as load transfer strengthening.

4. Summary

Particulate reinforced TMCs can be *in-situ* synthesized by investment casting process, and the reinforcements were homogeneously distributed and there is no interfacial reaction between the matrix and reinforcements. The *in-situ* synthesized reinforcements by 150μm B$_4$C was finer than 1500μm B$_4$C. Especially, the TMCs by 0.5μm B$_4$C was not fully synthesized since the nano-sized B$_4$C particles were

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**Fig. 6** Calculated and experimental yield strength of titanium matrix composites at room temperature. (YS: yield strength).

**Fig. 7** Inverse pole figure maps of (a) pure Ti, and titanium matrix composites with (b) 1500μm B$_4$C, (c) 150μm B$_4$C.

**Fig. 8** Measured alpha-Ti matrix grain size of (a) pure Ti, and titanium matrix composites with (b) 1500μm B$_4$C, (c) 150μm B$_4$C.
agglomerated in Ti melts. It is believed that these results were caused by the fine B\textsubscript{4}C particles promote the increase of the nucleation site of the reinforcements. The in-situ particulate (TiB+TiC) reinforced TMCs by fine B\textsubscript{4}C shows higher tensile property than by coarser B\textsubscript{4}C. Therefore, improvement of tensile property is obtained by the matrix grain refinement and homogeneous distribution as well as load transfer strengthening.

REFERENCES