Tensile behavior of TiB-reinforced Ti matrix composites with different titanium powders

Hiroshi IZUI*, Akinori OOTA**, Konomi MATSUURA** and Shoji KAMEGAWA**

* Department of Aerospace Engineering, Nihon University
7-24-1 Narashinodai, Funabashi, Chiba 274-8501, Japan
E-mail:izuhi@aero cst.niho n-u.ac.jp
**Graduate School of Science and Technology, Nihon University
7-24-1 Narashinodai, Funabashi, Chiba 274-8501, Japan

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Abstract
Although titanium alloys are known to possess low density, high specific strength, and excellent corrosion resistance, their low specific stiffness and wear resistance have restricted their widespread application. Cost-effective discontinuously reinforced titanium and its alloys containing titanium boride (TiB) are emerging as possible candidates for overcoming these limitations. The mechanical properties of titanium matrix composites (TMCs) are mainly dependent on the matrix composition, and on the volume fraction, and distribution of reinforcements. The distribution of reinforcements in the matrix depends on the particle shape and size of the Ti matrix powder. The purpose of this study was to investigate the effect of Ti powders produced by different manufacturing processes on the tensile behavior of titanium compacts and TiB reinforced Ti matrix composites (TiB/Ti). The Ti powders were produced by the hydride-dehydride (HDH) or the gas atomization (GA) process with particle sizes of <45 μm and <150 μm. The TiB/Ti composites were produced by a spark plasma sintering process. The Ti compact using Ti particle sizes of <45 μm, with higher oxygen content, possessed high tensile strength. This is because of the influence of oxygen as an interstitial strengthening element in the titanium alloy, which is well known. The TiB/Ti composites using HDH Ti powder with a particle size of <45 μm had the highest Young’s modulus, tensile strength, and Vickers microhardness. For the HDH Ti powder with a particle size of <45 μm, small TiB clusters connected like a network were uniformly distributed around the Ti matrix particles. Cracks in the composites initiated at the TiB clusters when a tensile load was applied to the composites. The presence of small TiB clusters inhibited the formation of cracks.

Key words: Titanium matrix composites, Hydride-dehydride Ti powder, Gas-atomized Ti powder, TiB whiskers, TiB clusters, Spark plasma sintering, TiB volume fraction, Tensile properties

1. Introduction
Titanium and its alloys possess high specific and fatigue strengths, excellent corrosion resistance, and low density. However, their widespread application has been restricted because of their low specific stiffness and wear resistance. In order to overcome this issue, titanium matrix composites (TMCs) using reinforcements such as TiB, TiC, or B₄C have been developed (Panda, et al., 2003, Gorsse, et al., 2003, Melendez, et al., 2011, Kumar, et al., 2012, Liu, et al., 2009, Abderrazak, et al., 2011, Zadra, et al., 2014, Hulbert, et al., 2009, and Ni, et al., 2006). TiB as a reinforcement for Ti has several advantages compared with other reinforcements (Ravi Chandran, et al., 2002, and Tamirisakandala, et al., 2006). TiB is currently recognized as one of the most compatible and effective reinforcement for Ti (Moris, et al., 2007). Since the density and thermal expansion coefficient of TiB are comparable to those of titanium, TiB offers increases in strength and stiffness without increasing density or generating residual stresses. Although TiB can be formed in titanium by a solid-state reaction of Ti and B source powders during consolidation, intermediate phases between Ti and TiB are not formed. Furthermore, in the crystallographic relationship between Ti and TiB, the interfaces
between the Ti matrix and TiB are quite smooth and free from any interfacial phase. Solid-state fabrication techniques based on powder metallurgy and sintering can be applied to manufacture TiB/Ti composites.

The mechanical properties of TMCs depend on the matrix composition, as well as the volume fraction and distribution of reinforcements. Theoretically, the tensile strength and stiffness of TMCs increase with increasing reinforcement volume fraction. A harmonic-structured Ti-48 at%Al/Ti composite with a network region of a Ti-Al alloy and dispersed region of pure Ti had higher strength and ductility than Ti-48 at% Al compact (Fujiwara, et al., 2013). In practice, the increase of the reinforcement volume fraction causes the agglomeration of reinforcements between the matrix particles. The fracture mechanism of the TMCs involves the fracturing of reinforcement particles (da Silva et al., 2006). In other words, the initial cracks occur in the agglomerations of reinforcement particles under tensile load. There are many pores and unsintered reinforcement particles in the agglomerations. Therefore, the decreases in tensile strength and wear resistance of TMCs are related to the agglomeration of reinforcements caused by the high volume fraction of reinforcements. Although the degree of TiB agglomeration is strongly sensitive to the particle size and shape of the Ti powder, there have been very few studies on the effects of the agglomeration of reinforcements on the tensile properties of TMCs (Patel et al., 2009, Chicosha et al., 2014).

In the present work, Ti compacts and TiB reinforced titanium matrixes (TiB/Ti) were prepared by a spark plasma sintering process using four types of Ti powders having particle sizes of <45 μm and <150 μm, with angular and spherical shapes. This paper focuses on the influence of titanium powders produced by different manufacturing processes on the distribution of TiB clusters, and the mechanical properties, such as tensile strength, stiffness and Vickers microhardness of TiB/Ti composites.

2. Experimental procedures

2.1 Materials

Commercial pure Ti powders were used as the matrix, as shown in Fig. 1 (a)-(d). The powders were produced by the hydride-dehydride (HDH) process and the gas atomization (GA) process with particle sizes of <45 μm and <150 μm. The HDH and GA powders were angular and spherical in shape, respectively. TiB₂ powder with an average particle size of 1.81 μm was used for the reinforcement in Fig. 1 (e).

![Fig. 1 SEM micrographs of pure Ti (HDH and GA) and TiB₂ powders.](image)

2.2 Fabrication, microstructural analysis, and mechanical tests

The Ti and TiB₂ powders were mixed with a planetary ball-mill for 10 min at 200 rpm. The mixed powders were sintered using a spark plasma sintering machine (Dr. Sinter, SPS-3.20IV, Sumitomo Coal Mining, Japan). The sintering conditions included heating at 20 °C/min up to 900 °C, isothermal soaking for 10 min, and furnace cooling. The temperature was measured by a K-thermocouple inserted into a graphite die. A compressive pressure of 70 MPa was applied during heating and cooling. The vacuum pressure during sintering was under 5 Pa. The TiB phase was formed during the SPS process, because it is thermodynamically more stable than the raw TiB₂ phase under excess Ti according to the following reaction:

\[ \text{Ti} + \text{TiB}_2 \rightarrow 2\text{TiB} \] (1)

The sintered specimens for SEM observation were prepared by standard metallographic methods. Specimens were etched using a solution consisting of 95% deionized water, 3% nitric acid, and 2% hydrofluoric acid and were investigated using a scanning electron microscope (Shimadzu SSX-550, Japan).

The tensile tests of the Ti compacts and TiB/Ti composites were carried out using an Instron testing machine (55R1125) at a constant crosshead speed of 0.5 mm/min. Their strains were measured with a strain gage attached to

the surface of the specimen. All tensile tests were performed at least three times at each TiB volume fraction. The observation of the crack propagation was performed by using a servo-hydraulic uniaxial fatigue test machine with a scanning electron microscope (SEM Servopulser, Shimadzu, Japan). Specimens prepared for tensile tests and crack propagation analysis are shown in Fig. 2. These specimens, with a thickness of 1 or 2 mm, were prepared using a wire electric discharge machine. Vickers microhardness was measured using a Shimadzu Microhardness Tester HMV-2(T) with 9.8 N load and a dwell time of 30 s. An average of 10 indents was taken for each specimen.

3. Results and discussion

3.1 Microstructure

A high-magnification SEM micrograph of the composite with 20 vol.% TiB using HDH Ti powder with a particle size of <45 μm is shown in Fig. 3. This was taken in SEI mode using SEM after deep etching the sample to reveal the distribution of TiB clusters and whiskers. TiB clusters and randomly oriented TiB whiskers (TiB$_w$) were formed in the Ti particles and between Ti particles, respectively.

Figure 4 shows the microstructures of the TiB/Ti composites using different Ti powders. These microstructures were taken in the backscattered electron imaging (BEI) mode. As shown in Fig. 4 (a), (c), (e), and (g), for the composites with 10 vol.% TiB, the TiB clusters (dark regions) were discontinuously dispersed between the Ti-TiB$_w$ phases. On the other hand, for the composites with 20 vol.% TiB, it is evident that TiB clusters, connected like a network, were distributed around the Ti-TiB$_w$ phases, as can be seen in Fig. 4 (b), (d), (f), and (h). That is, it can be observed from Fig. 4 (e) and (f) that large TiB clusters were partly formed in the composites using HDH Ti powders with particle sizes of <150 μm. Furthermore, with the GA Ti powders having particle sizes of <150 μm, the large TiB clusters were distributed around the Ti-TiB$_w$ phases as shown in Fig.4 (g) and (h). From Fig. 4 (a - d), for the particle size of <45 μm, the TiB clusters formed in the composites with GA powder were larger than those with HDH Ti powders. In other words, for the composites with the HDH powders, the TiB clusters were distributed more uniformly in the Ti-TiB$_w$ phases.

3.2 Mechanical properties

Figure 5 presents the relationships between the Young’s modulus, tensile strength, elongation, and Vickers microhardness of TiB/Ti and the TiB volume fraction. The Young’s modulus and Vickers microhardness of the TiB/Ti composites increased as the TiB volume fraction increased, as shown in Fig.5 (a) and (d). As shown in Fig.5(a), the green line indicates the theoretical elastic modulus obtained by the rule of mixture (Voigt method) using the elastic modulus value of Ti and TiB of 108 GPa and 371 GPa, respectively (Brandes, et al., 1992, Atri, et al., 1999). The
elastic modulus values of the composites were relatively close to the theoretical value under 15 vol.% TiB. The TiB/Ti composite with the HDH powder having a particle size of <45 μm had the highest Young’s modulus and Vickers microhardness of the Ti powders. The tensile strengths of the composites also increased with increasing TiB volume fraction, and they reached peaks at different volume fractions. The composite with HDH powder having a particle size of <45μm had the highest tensile strength of 955 MPa at a TiB volume fraction of 20 vol.% The tensile strengths of the composites decreased dramatically at 10, 15, or 20 vol.% TiB. The TiB volume fraction at which the peak of the tensile strength appeared was different in composites with different Ti powders. Higher tensile strength of the composites was exhibited at the higher TiB volume fractions. Therefore, the tensile strength of the composites depended on the titanium powders at a high volume fraction of TiB. As shown in Fig. 5 (c), the elongation of the composites decreased with increasing TiB volume fraction. The composites with the HDH Ti powders had lower
The tensile strength of melted Ti increased with oxygen content, because the oxygen content in TiB composites were not dependent on their oxygen contents at a high volume fraction of TiB. This is because the rates of increase of the microhardness with respect to TiB volume fractions were almost the same.

3.3 Crack propagation

Figures 8 and 9 show SEM micrographs taken at tensile stress levels during in situ tensile deformation for the composites with 10 vol.% TiB using GA or HDH Ti powder with particle sizes of <45 μm. The arrows in the figures indicate the loading direction. From Fig. 8 (b, c), the cracks initiated at TiB clusters, and the cracks grew and became
more extensive in the direction perpendicular to the loading direction as the load increased. On the other hand, the composite with HDH Ti powder having particle sizes of $<45\,\mu m$ did not exhibit visible micro-cracks under the applied high stress, as shown in Fig. 9 (b). Figure 10 shows the crack initiation at the fracture areas of the composites with 10 vol.% TiB of HDH or GA Ti powder with particle sizes of $<45\,\mu m$. Compared with the composite with the GA powder, smaller cracks can be observed in the composite with the HDH powder, as shown in Fig. 10 (a). On the discontinuously reinforced titanium composites, the fracture mechanism consists of reinforcement fractures as a result of the load transfer from the matrix to reinforcements (Tjong, et al., 2008). TiB clusters act as crack initiation sites under tensile loading. Namely, the possibility of crack initiation was governed by the size of TiB clusters. Therefore, the composite with the HDH powder having a particle size of $<45\,\mu m$ exhibited excellent tensile strength and Young’s modulus because of the smaller and more uniform distribution of TiB clusters.

Fig. 6 Oxygen contents of Ti compacts and TiB/Ti composites.

Fig. 7 Relationship between oxygen contents and tensile strengths of Ti compacts and TiB/Ti composites with a particle size of $<45\,\mu m$.

Fig. 8 SEM images of tensile specimen with 10 vol.% TiB of GA Ti powder with a particle size of $<45\,\mu m$ taken during application of tensile load.

(a) Stress level: 0 MPa  (b) Stress level: 645 MPa  (c) Stress level: 653 MPa
Conclusions

The influence of titanium powders on the tensile properties of TiB reinforced titanium matrix composites was investigated. The following conclusions can be drawn from this study:

1. The oxygen contents of the Ti powders differed depending on their manufacturing process. The tensile properties and Vickers microhardnesses of the Ti compacts depended strongly on their oxygen contents.

2. The TiB clusters in the composites were attributed to the spaces between the Ti powders. The hydride-dehydride (HDH) powders with a particle size of <45 μm formed smaller TiB clusters in the composites than the other powders.

3. The TiB/Ti composites using HDH Ti powder with a particle size of <45 μm exhibited the highest Young’s modulus, tensile strength, and Vickers microhardness. These mechanical properties of the composites depended on the oxygen content and the distribution of TiB clusters.

4. The TiB volume fraction at which the peak of the tensile strength appeared was different in composites with different Ti powders. Higher tensile strength of the composites was exhibited at the higher TiB volume fractions. Because TiB clusters act as crack initiation sites under tensile loading and the possibility of crack initiation was governed by the size of TiB clusters.

Reference


Fujiwara, H., Kawabata, T., Miyamoto, H., Ameyama, K., Mechanical Properties of Harmonic Structured Composites.
Kumar, M. S., Chandrasekar, P., Chandramohan, P., and Mohanraj, Characterization of titanium-titanium boride composites processed by powder metallurgy, Materials characterization, Vol. 73, No. 7 (2012), pp. 43-51.