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# Mechanical properties of TiB/Ti-6Al-4V alloy composites fabricated by spark plasma sintering

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## Article Info

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### Abstract

High specific strength, fatigue strength, ductility, and weldability are only a few of the outstanding features shown by Ti-6Al-4V alloy. Wear resistance and rigidity are two areas where Ti-6Al-4V alloy falls short. Composites with a TiB reinforced Ti-6Al-4V alloy matrix (TiB/Ti-6Al-4V) were created using a spark plasma sintering (SPS) technique to enhance the strength characteristics, stiffness, and wear resistance. With a sintering temperature of 900 °C and a holding period of 30 min, the composites of TiB/Ti-6Al-4V alloys containing 4.2% TiB achieved the maximum tensile strength of 1174 MPa. Composites with a volume fraction of 16.7 vol.% TiB had a Vickers microhardness of 517 HV and a Young's modulus of 159 GPa, both of which grew as the TiB volume percentage did. Composites containing 4.2% TiB had greater tensile strength at 400 °C compared to the mildly annealed Ti-6Al-4V alloy. Composites containing 4.2% TiB had 350 MPa and 200 MPa fatigue limits, after 107 cycles at ambient temperature and 600 °C, respectively. Composites containing 1.4 and 4.2 vol.% TiB showed no increase in creep stresses at room temperature as the creep time increased.

### Keywords:

Topics covered include spark plasma sintering, titanium matrix composites, titanium volume fraction, titanium-6Al-4V alloy, and titanium (TiB). Tensile characteristics, Weariness level, Behavioural changes at room temperature

## 1. Introduction

Titanium alloy Ti-6Al-4V is the most popular choice because it has a wide range of desirable characteristics, including high specific strength, fatigue strength, ductility, weldability, and resistance to stress corrosion cracking. Wear resistance and rigidity are two areas where Ti-6Al-4V alloy falls short. Ceramic reinforcements in Ti-6Al-4V alloy matrix composites (TMCs) have been the subject of extensive research in an effort to enhance the strength properties, stiffness, and wear resistance. Various types of ceramic reinforcements have been studied in this context, including TiB, WC, TiC, TiAl, and SiC. Reinforcements must typically be chemically stable, tougher, and stiffer than the matrix. According to recent research (Ravi Chandran et al., 2004; Moris et al.,

2007), TiB is now considered a very compatible and effective reinforcement for pure titanium matrix composites. Here are the reasons: Firstly, solid-state composite processing involves relatively low temperatures (under 1000 °C), which makes manufacturing easy. Secondly, there is no intermediate phase between Ti and TiB. Thirdly, TiB forms as long, pristine single-crystal whiskers that are thermodynamically and mechanically stable in the Ti matrix. Since TiB's density and thermal expansion coefficient are similar to titanium's, it delivers improvements in stiffness and strength without adding bulk or creating residual stresses. Despite the fact that studies on TiB/Ti-6Al-4V alloy composites have been published, the impact of the TiB volume percentage on

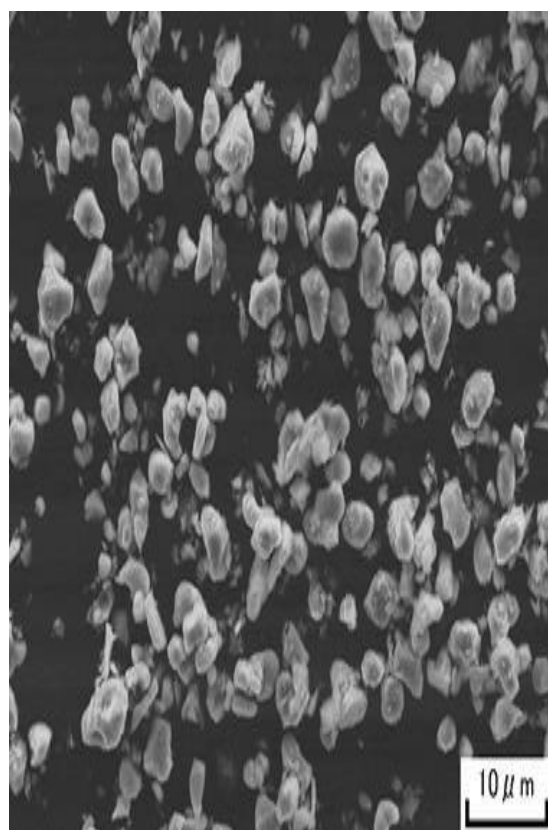
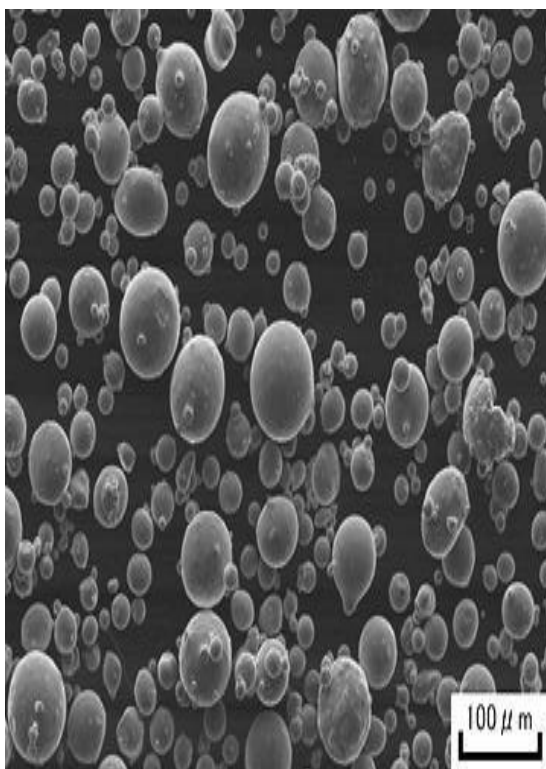
mechanical capabilities and mail:zuihi@aero.cst.nihon-u.ac.jp \*Address: Department of Aerospace Engineering, Nihon University 7-24-1 Narashinodai, Funabashi, Chiba 274-8501, Japan The strength characteristics at high temperatures are not yet understood. The spark plasma sintering (SPS) procedure was used to create TiB/Ti-6Al-4V alloy composites in this study, which are able to withstand the  $\beta$  transus temperature (980 °C) of Ti-6Al-4V alloy. The

microstructures and mechanical characteristics of TiB/Ti-6Al-4V alloy composites, including tensile strength, stiffness, and Vickers microhardness, are examined in this work as a function of the TiB volume %. In addition, the room-temperature creep behaviour, fatigue strengths at room temperature and 600 °C, and tensile strength at temperatures below 700 °C of TiB/Ti-6Al-4V alloy composites were also studied.

### 1.1 Materials

Commercial gas-atomized Ti-6Al-4V alloy powder with an average particle size of 37  $\mu\text{m}$  was used as the matrix, as shown in Fig. 1(a). TiB<sub>2</sub> powder with

an average particle size of 2.7  $\mu\text{m}$  was used as the reinforcement, as shown in Fig. 1(b).



(a) Ti-6Al-4V alloy (b) TiB<sub>2</sub>

Fig.1 SEM micrographs of Ti-6Al-4V alloy and TiB<sub>2</sub> powders.

### 1.2 Fabrication, microstructural analysis, and mechanical tests

The Ti-6Al-4V alloy and TiB<sub>2</sub> powders were blended with a planetary ball mill at 200 rpm for 10 min. The blended powder mixture was placed into a general-purpose graphite die and consolidated using a spark plasma sintering machine (Dr. Sinter, SPS-3.20IV,

Sumitomo Coal Mining, Japan). The fabrication conditions are shown in Table 1. The temperature was measured by a K-thermocouple inserted into the graphite die. The vacuum pressure during fabrication .

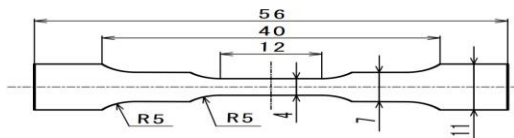
Table 1 Fabrication conditions.

|                |                      |
|----------------|----------------------|
| Heating rate   | 100 °C / min         |
| Temperature    | 800, 900 and 1000 °C |
| Holding time   | 10, 30, and 50 min   |
| Pressure       | 60 MPa               |
| Cooling method | Furnace cooling      |

Using conventional metallographic procedures, the composite specimens were prepared for scanning electron microscopy analysis. The samples were subjected to etching using a mixture of 95% deionized water, 3% nitric acid, and 2% hydrofluoric acid. The scanning electron microscope used for analysis was a Shimadzu SSX-550 from Japan. A Japanese X-ray diffractometer equipment (Rigaku Rint2000) equipped with Cu-K $\alpha$  radiation operating at 20 kV and 10 mA was used to conduct X-ray diffraction studies on the composites.

An Instron testing machine (55R1125) was used to perform the tensile tests on the composites, maintaining a constant speed of 0.5 mm/min for the crosshead. Specimens were tested for stresses using a strain gauge that was fastened to their surfaces. For every volume fraction of TiB, tensile tests were performed three times

minimum. An infrared heat lamp was used to heat the specimens in the air before to the elevated-temperature tensile test. A wire electric discharge machine was used to manufacture these specimens, which have a thickness of 2 mm. A Shimadzu Microhardness Tester HMT-2(T) with a 9.8 N load and a dwell period of 30 s was used to test the Vickers microhardness. For every specimen, the mean hardness of ten indentations was recorded. Using a Shimadzu servo-hydraulic test equipment, the composites were subjected to fatigue life tests at both room temperature and 600 °C. The fatigue life of the composites was tested using a sine wave loading cycle. The testing was carried out using a stress ratio,  $R$  ( $\sigma_{\min}/\sigma_{\max}$ ), of 0.1, a frequency of 10 Hz, and load control. As previously stated, the composites' room-temperature creep stresses were measured using the strain gauge.



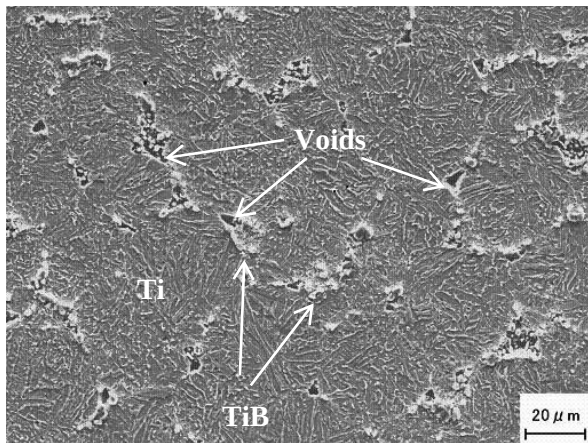
Tensile and fatigue test specimen (b) Creep test specimen Fig. 2 Schematic of tensile, fatigue and creep test specimens.

## Results and discussion

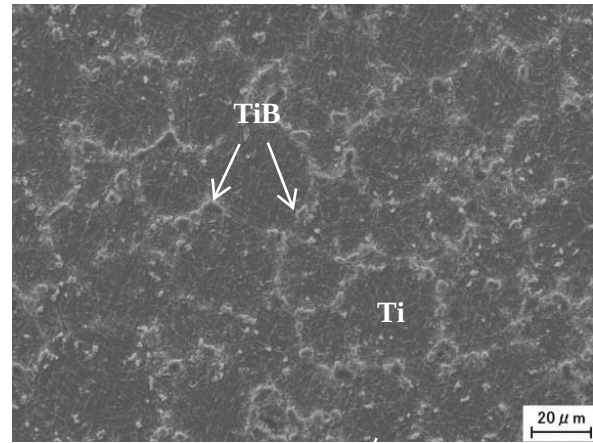
### Determination of optimum sintering temperature and holding time

Blended powder formulations containing 4.2% TiB were subjected to several sintering temperatures and holding durations in order to ascertain the optimal combinations. Figures 3 and 4 show scanning electron micrographs of the composites that were manufactured. The micrographs clearly reveal that the Ti-6Al-4V matrix powders are encircled by reinforcing particles and tiny whiskers. At the interface of the matrix particles were areas that were rich in TiB and those that were deficient in TiB. Composites made at 800 °C for 10 minutes had a lot of empty space between the matrix particles; however, when sintered at 900 and 1000 °C, all of the empty space was filled in. Figures 3(c) and 4(c) demonstrate that the size and quantity of particles decreased as the sintering temperature or holding time rose, and that the majority of the particles converted into fine whiskers. During sintering, TiB<sub>2</sub> interacted with Ti to generate either TiB particles or needle-shaped TiB whiskers (Fan, Z., et al, 1996). The results showed that at higher sintering temperatures and longer sintering times, a large amount of TiB<sub>2</sub> particles could be converted into TiB whiskers. In Figure 5, we can see the X-ray diffraction patterns of the composites made with 4.2 vol.% TiB/Ti-6Al-4V

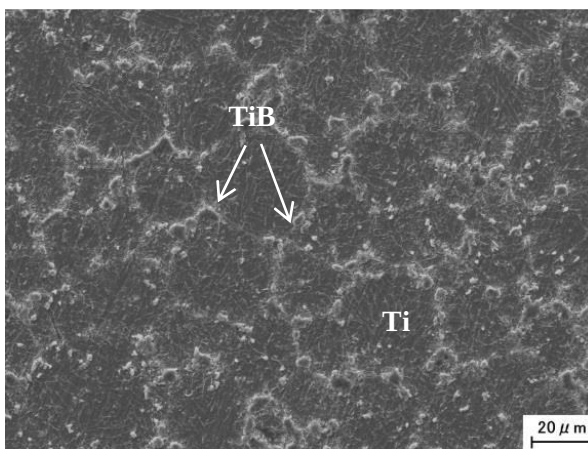
alloy using various sintering temperatures and holding durations. The creation of TiB is temperature and holding time dependent because when sintering temperature and time rose, the TiB peaks likewise increased while the TiB<sub>2</sub> peaks declined. A Ti-6Al-4V alloy compact and TiB/Ti-6Al-4V alloy composites were made with varying sintering temperatures and holding durations, and their tensile strengths are shown in Figure 6. The tensile strengths of the TiB/Ti-6Al-4V alloy composites reached their peak at 900 °C, as shown in Figure 6(a). The tensile strength of the Ti-6Al-4V compacts, however, showed a little increase when the sintering temperature was raised. At a sintering temperature of 900 °C, the TiB/Ti-6Al-4V alloy composite and the Ti-6Al-4V alloy compact display the maximum tensile strength, as indicated in Figure 6 (b). Composites made at 1000 °C for 10 minutes and 900 °C for 50 minutes showed a decline in their tensile strengths. This might be because tiny whiskers with a high TiB volume percentage emerge at the border as a result of the matrix particles' grain expansion. Thus, 900 °C is the ideal sintering temperature and 30 minutes is the ideal holding period.



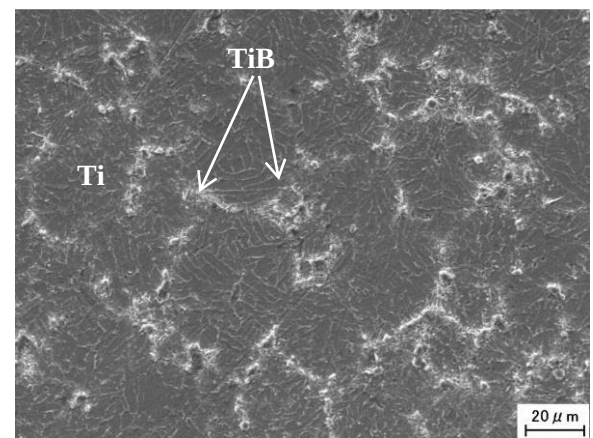
(a) 800°C



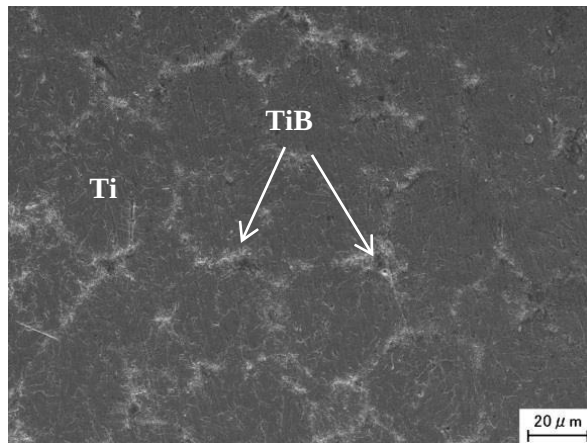
(a) 10 min



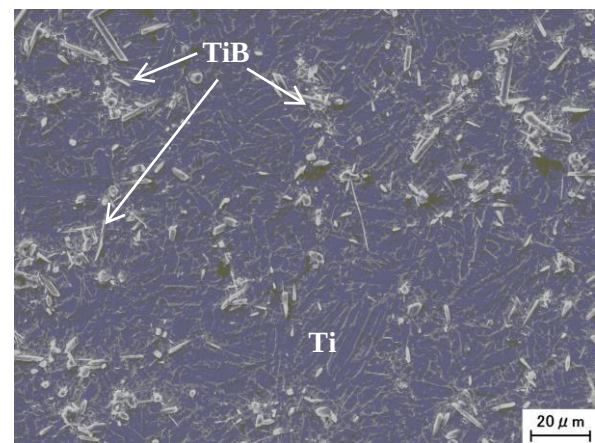
(b) 900°C



(b) 30 min



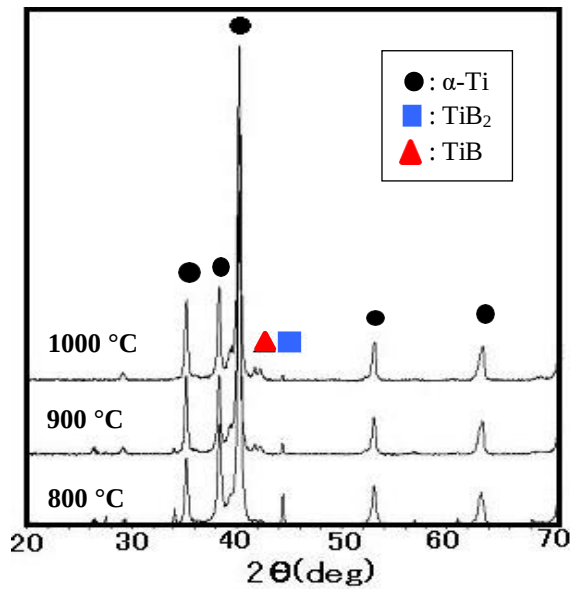
(c) 1000°C



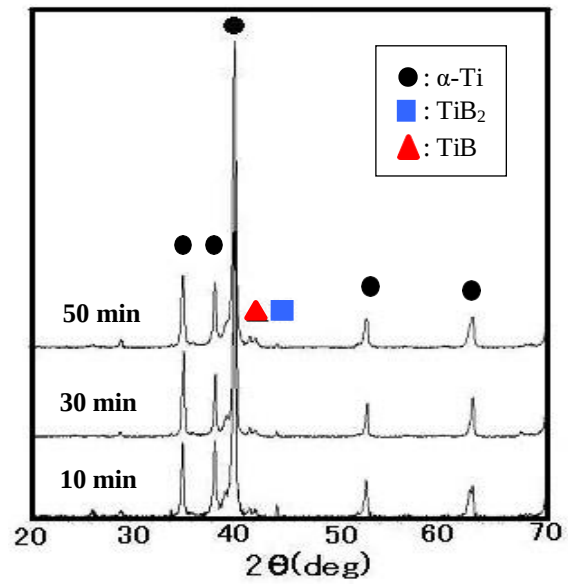
(c) 50 min

Fig. 3 Microstructures of 4.2 vol.% TiB/Ti-6Al-4V alloy composites fabricated for different sintering temperatures and for 10 min.

Fig. 4 Microstructures of 4.2 vol.% TiB/Ti-6Al-4V alloy composites fabricated for different holding times and at 900 °C.

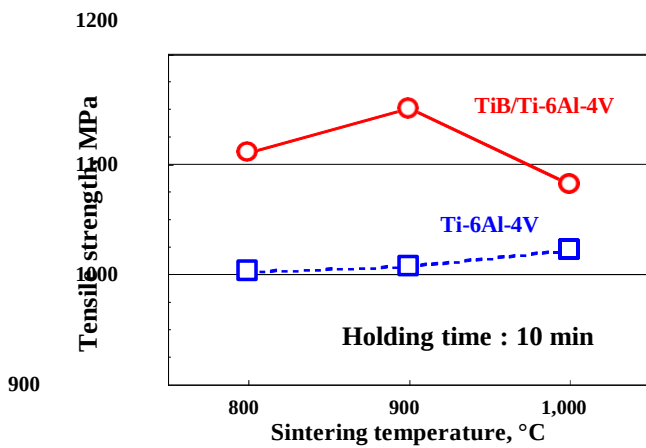


(a) Holding time: 10 min

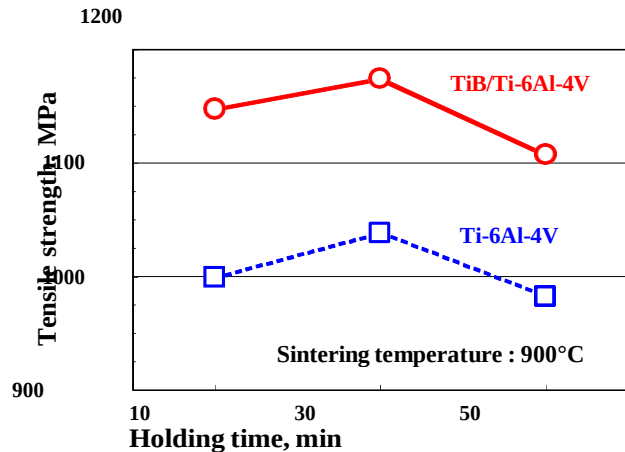


(b) Sintering temperature: 900 °C

Fig. 5 X-ray diffraction patterns of the TiB/Ti-6Al-4V alloy composites with 4.2 vol.% TiB fabricated by different sintering temperatures and holding times.



(a) Holding time: 10 min



(b) Sintering temperature: 900 °C

°C Fig. 6 Tensile strengths of Ti-6Al-4V compacts and TiB/Ti-6Al-4V alloy composites versus sintering

temperature and holding time and crack propagation tests.

### 1.3 Mechanical properties of the TiB/Ti-6Al-4V alloy composites

#### 3.2.1 Tensile properties at room temperature and Vickers microhardness

Figure 7 presents the variation of the tensile strength and the elongation of TiB/Ti-6Al-4V composites with different TiB volume fractions. Tensile and 0.2% yield strengths of a mild annealed Ti-6Al-4V alloy are shown in Fig. 8 (William. H., et. al., 1980). As shown in Fig. 7, the tensile strength of the TiB/Ti-6Al-4V composites increased with increasing TiB volume fraction from 0 vol.% to 4.2 vol.%, and then decreased at higher TiB volume fractions. The composite with 4.2 vol.% TiB have the highest tensile strength of 1174 MPa. The dashed line shows the tensile strength of a mild annealed Ti-6Al-4V alloy obtained from Fig. 8.

The tensile strengths of the composites containing lower than 8.4 vol.% TiB are higher than those of the mild annealed Ti-6Al-4V alloy. As the TiB volume fraction increased, the tensile elongation of the composites decreased sharply, as shown in Fig. 7. Figure 9 shows the fracture surfaces of the composites with 4.2 and 16.7 vol.% TiB. Intergranular fracture modes were observed in both composites. Figure 10 presents the microstructures of the composites with 16.7 vol.% TiB. For the composites with a high TiB volume fraction, the microstructure consists of a TiB-rich network boundary around the matrix particles,

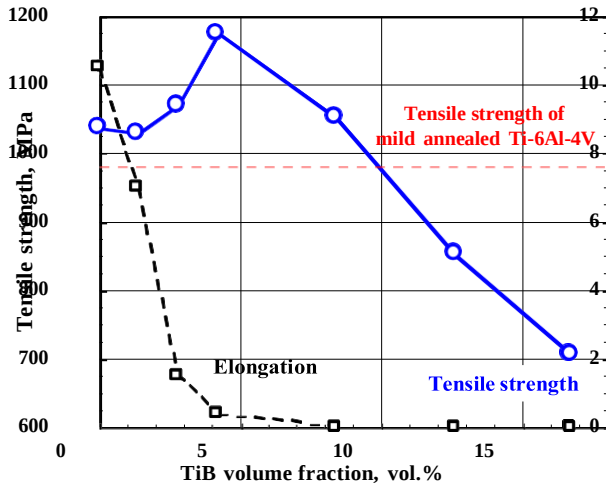


Fig. 7 Tensile strengths of TiB/Ti-6Al-4V alloy composites with different TiB volume fractions.

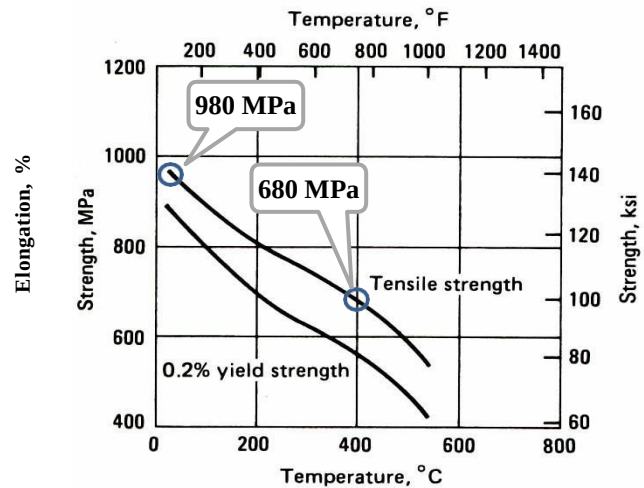
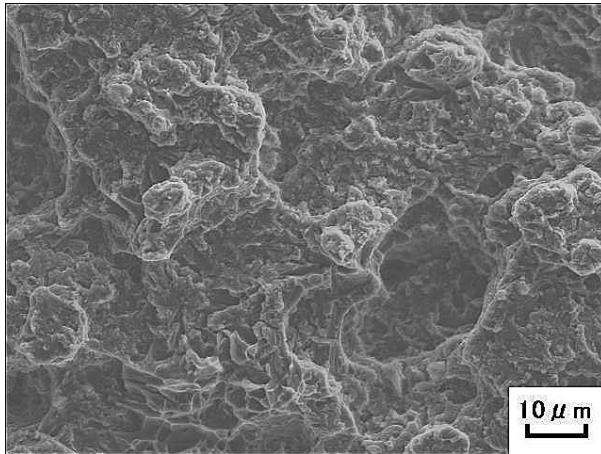
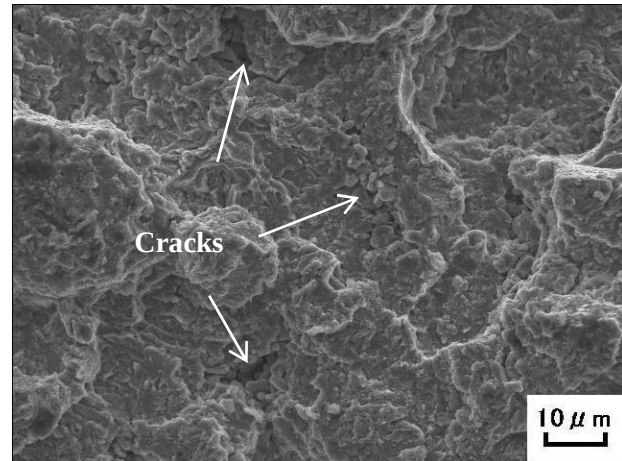


Fig. 8 Tensile strength and 0.2% yield strength of mild annealed Ti-6Al-4V alloy.



(a) 4.2 vol.% TiB

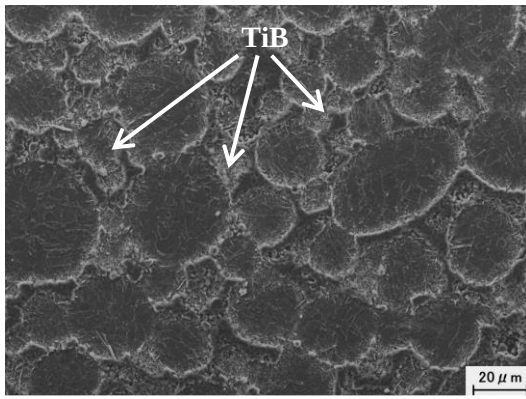


(b) 16.7 vol.% TiB

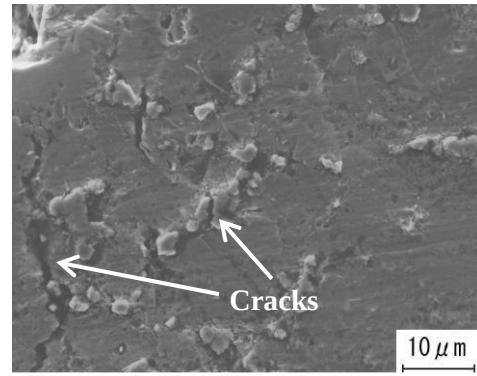
Fig. 9 SEM images of fracture surfaces of TiB/Ti-6Al-4V alloy composites with 4.2 vol.% and 16.7 vol.% TiB.

seen in Figure 10 (a). The fractures started at the TiB-rich network border and spread along it, as seen in Figure 10 (b). Hence, the network distribution of TiB is responsible for the reduction in tensile strength. Figure 11 shows that there was a clear relationship between the volume % of TiB and the composites' Young's modulus. In comparison to the compact Ti-6Al-4V alloy, the composite containing 16.7 vol.% TiB has a Young's modulus of 159 GPa, or almost 1.4 times higher. In comparison to TiB's 371 GPa Young's modulus, the compact Ti-6Al-4V alloy has a Young's modulus of 113 GPa. For matrix particles with a larger TiB volume %, the

current experimental findings are in excellent accord with the theoretical predictions based on the law of mixing. The fissures in Ti matrix composites reinforced with TiB began at those reinforcements. Figure 12 shows the Vickers microhardness variation of the composites as a function of the volume % of TiB. From 356 HV to 517 HV, the microhardness rose as the TiB volume percent increased. At less than 1800 HV, TiB exhibits a Vickers microhardness. Hence, the microhardness increase was thought to be caused by the dispersion of TiB.



(a) TiB/Ti-6Al-4V composite



(b) Tensile test specimen

Fig. 10 Microstructures of TiB/Ti-6Al-4V alloy composites with 16.7 vol.% TiB and its tensile test specimen.

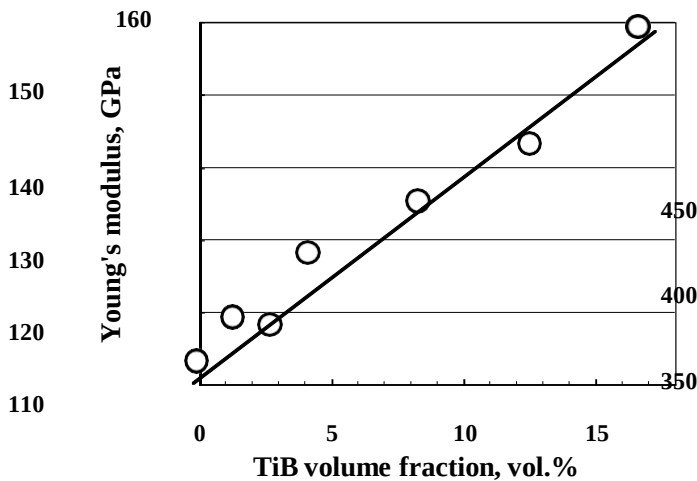


Fig. 11 Variation of Young's modulus of TiB/Ti-6Al-4V alloy composite with TiB

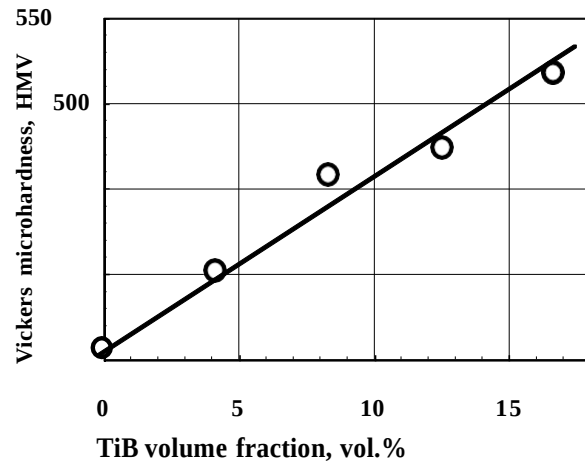


Fig. 12 Variation of Vickers microhardness of TiB/Ti-6Al-4V alloy composite with TiB volume fraction.

### 3.2.2 Tensile strengths of Ti-6Al-4V alloy compacts and the composites with 4.2 vol.% TiB under 700 °C

Figures 13 shows the tensile strengths of the Ti-6Al-4V alloy compacts and the TiB/Ti-6Al-4V alloy composites with 4.2 vol. % TiB under 700 °C. At

400 °C, the tensile strength of the composite is higher than that of the compact and the mild annealed Ti-6Al-4V alloy obtained from Fig. 8. At 600 °C and 700 °C, the tensile strengths of the composites are the same as those of the compacts.

### 3.2.3 Fatigue strengths of Ti-6Al-4V compacts and composites with 4.2 vol.% TiB at room temperature and 600 °C

Ti-6Al-4V alloy compacts and composites with 4.2 vol.% TiB are shown in Figure 14 together with the connection between maximum applied stress and fatigue life (S-N data). When the maximum applied stress was reduced, the fatigue lifetimes of the composites and compacts at both temperatures rose. Even after more than 107 cycles, the composites showed no signs of failure at tensile stress values of 200 MPa at 600 °C and 350 MPa at ambient temperature. Compared to the annealed Ti-6Al-4V alloy, the composite has a lower room-temperature fatigue limit. The Ti-6Al-4V alloy compact had a significantly lower fatigue strength than the composite at room temperature. In terms

of fatigue life, the composites and compacts differed somewhat. Figure 15 shows the fatigue fracture surfaces of the composites that include 4.2% TiB. Figure 15 (a) shows that under the room temperature scenario, the composite was broken at the border of the TiB network. In contrast, the composite displayed a ductile transgranular fracture mode in the 600 °C example. At higher temperatures, the matrix particles may have thermally softened, making it simpler for fractures to spread within them. This could explain the observed phenomenon.

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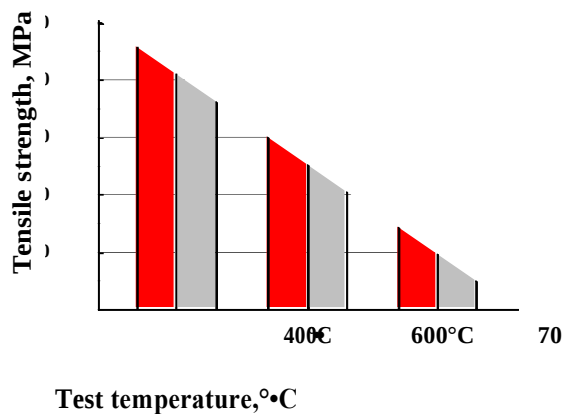
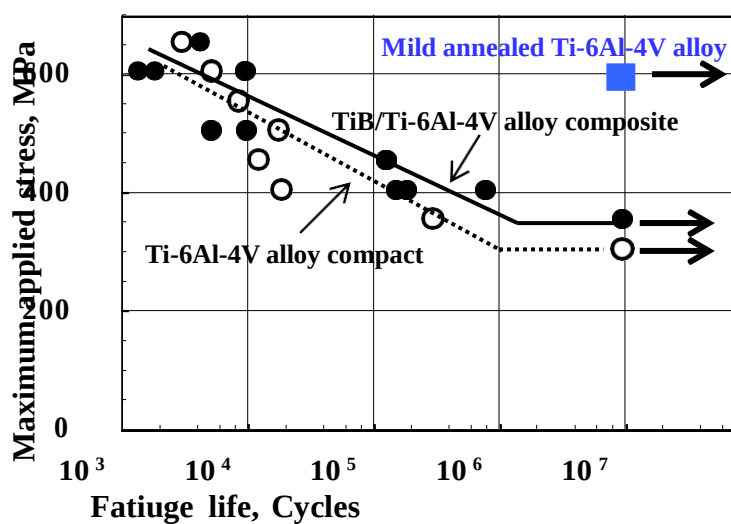
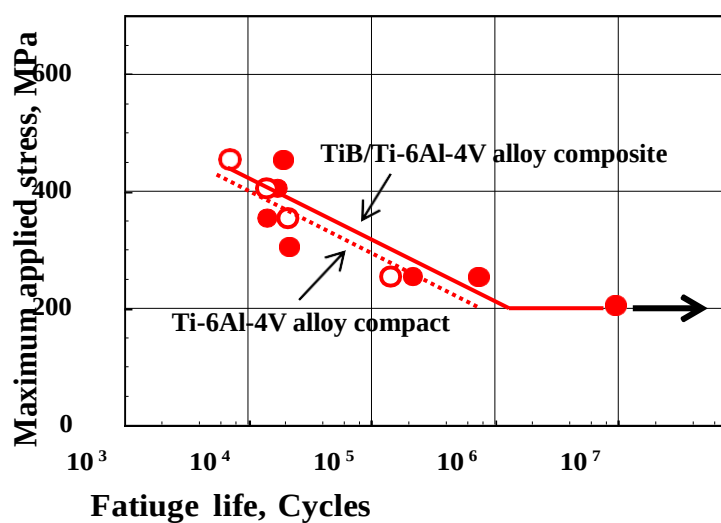


Fig. 13 Tensile strengths of Ti-6Al-4V alloy compact and the TiB/Ti-6Al-4V alloy composite with 4.2 vol.% TiB

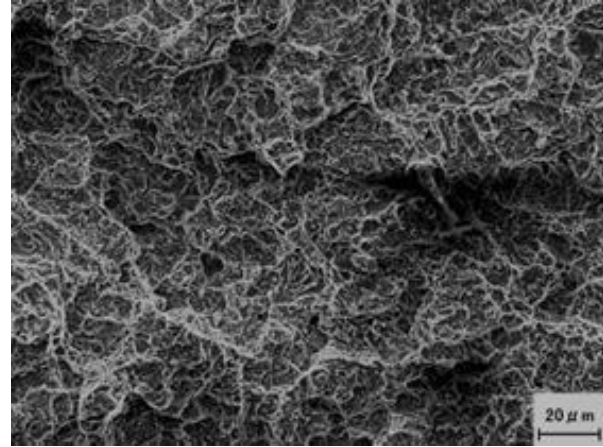
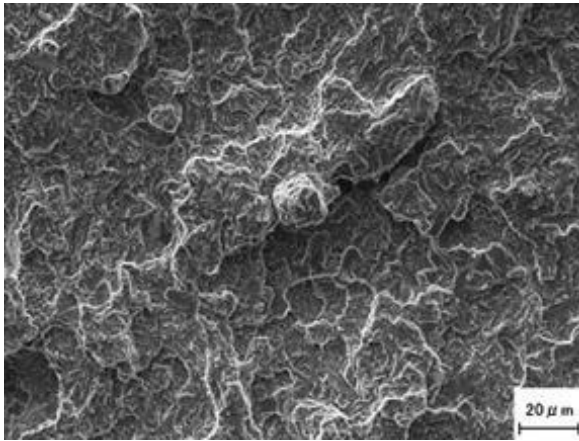


(a) Room temperature



(b) 600°C

Fig. 14 S/N fatigue curves for Ti-6Al-4V alloy compacts and TiB/Ti-6Al-4V alloy composites with 4.2 vol.%TiB at room temperature and 600°C.



(a) 350 MPa and room temperature

(b) 200 MPa and

600°C Fig. 15 Fatigue fracture surfaces of TiB/Ti-6Al-4V alloy composites with 4.2 vol.% TiB.

### 3.2.4 Room-temperature creep behavior of the composites

It is well recognised that titanium alloys, even those in the  $\alpha$ -phase, display creep strain at room temperature. The creep strain occurs because the  $\alpha$ -Ti phase, which is hexagonally organised, experiences dislocation creep (Hasija, et al., 2003; Sato, et al., 2005). Figure 16 shows the creep curves of the Ti-6Al-4V alloy compact and composites containing 1.4% and 4.2% TiB, respectively. Thirty percent of each 0.2 percent

proof strength ( $0.7\sigma_{0.2}$ ) is applied as stresses. As the creep period after loading rose, the creep strain for the Ti-6Al-4V compact also increased. However, none of the two composites showed a rise in creep stresses as creep time increased. It follows that the presence of whiskers and TiB particles, which limit dislocation-related motion in the matrix, is responsible for this outcome.

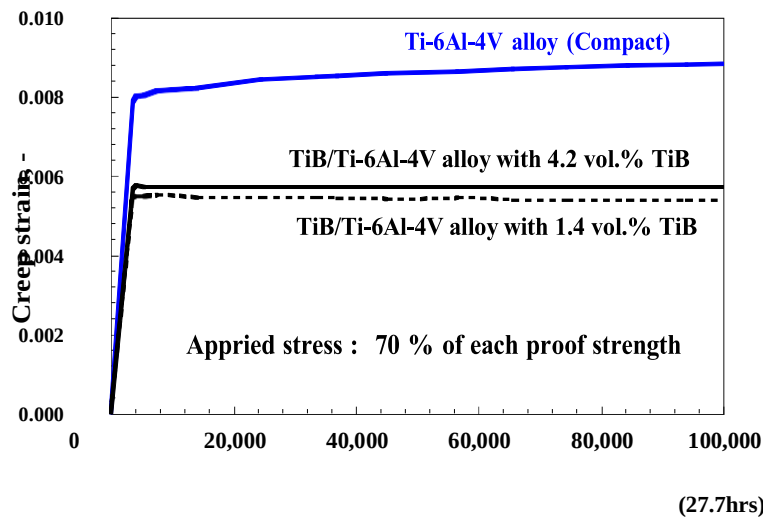


Fig. 16 Room-temperature creep curves of Ti-6Al-4V alloy compact and TiB/Ti6Al-4V alloy composites with 1.4 vol.% and 4.2 vol.% TiB.

### Conclusions

We looked at TiB-reinforced Ti-6Al-4V alloy composites made by spark plasma sintering to see how the mechanical characteristics changed with different volume fractions of TiB. Looking at the data, we may say the following: 1. During the sintering process, the TiB<sub>2</sub> reinforcements were converted into TiB particles or whiskers. A network topology was used to distribute TiB particles and whiskers along the matrix particles. More so, at higher sintering temperatures and for longer holding times, TiB particles were converted into TiB whiskers.

2. Sintering at 900 °C for 30 minutes was the optimal fabrication condition for the TiB/Ti-6Al-4V alloy composites.
3. A tensile strength of 1,174 MPa was recorded by the composites containing 4.2% TiB. The composites outperformed the mild annealed Ti-6Al-4V alloy in terms of tensile strength both at room temperature and 400 °C.
4. As the volume percent of TiB in the composites grew,

the Young's modulus and Vickers microhardness also increased.

5. The composites containing 4.2% TiB had 350 MPa and 200 MPa fatigue limits, after 107 cycles, at room temperature and 600 °C, respectively. Composites containing 1.4% and 4.2% TiB showed much lower room-temperature creep stresses than compacts made of Ti-6Al-4V alloy. 6.

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The kinetic and mechanism of interfacial interaction in sigma fiber-reinforced Ti MMCs was published in Composites Part A, Vol. 28A, No. 2 (1996), pages 131-140 and was authored by Fan, Z., Guo, Z. X., and Cantor, B. The mechanical characteristics of Ti-6Al-4V/TiB composites having TiB reinforcements that are randomly oriented and aligned were discussed in a 2003 article by Gorsse and Miracle in the Acta Materialia, volume 51, issue 9, pages 2427-2442. The mechanical behaviour of Ti-6Al-4V/TiB composites was studied in a micromechanical model using finite element analysis (FEM) by Giannopoulos, G.I., Karagiannis, and Anifantis, N.K. in the journal Computational Materials Science, volume 39, issue 2, 2007, pages 437-445.

The authors of the article "Deformation and creep modelling in polycrystalline Ti-6Al alloys" (Hasiija, V., Ghosh, S., Mills, M.J., & Joseph, D. S. 2003) are listed in the Acta Materialia volume 51, issue 15, pages 4533-4549.

The high temperature tensile characteristics of in situ TiBw/Ti-6Al-4V composites with a new network reinforcing architecture were investigated in a 2012 publication by Huang, Geng, Peng, and Kaveendran (Volume A534, No. 1, pp. 688-692). Koo et al. (2012) found that the mechanical characteristics of TiBw/Ti-6Al-4V composites were affected by the aspect ratios of in situ produced TiB

whiskers. The study was published in Scripta Materialia and can be found on pages 487-490. This review, written by Morsi and published in 2007 in the Journal of Materials Science, examines the processing and characteristics of titanium-titanium boride (TiBw) matrix composites. The article can be found on pages 2037-2047. "Formation of equiaxed alpha and titanium nitride precipitates in spark plasma sintered TiB/Ti-6Al-4V composites" was published in Materials Letters in 2012 and was co-authored by Nandwana, P., Hwang, J.Y., Koo, M.Y., Tiley, J., Hong, S.H., and Banerjee, R. Titanium boride (TiB) composites synthesised using a beta-titanium matrix: the process of TiB creation and the characteristics of the resulting materials, by Panda and Ravi, K. S. C. Materials and metallurgy transactions A, volume 34A, issue 6, pages 1371-1385, 2003. By incorporating boron and using thermo-mechanical processing, Rastegari and Abbasi were able to create a Ti-6Al-4V/TiC composite with enhanced characteristics. Chapters 473-477 of the 2013 edition of Materials Science and Engineering A, Volume A564, Issue 1. Paper: TiBw-reinforced Ti composites: processing, properties, application prospects, and research need (Ravi Chandran, K. S., Panda, K. B., & Sahay, S.S.), volume 56, issue 5 (2004), pages 42-48. The authors of the article "Structure evolutions in a Ti-6Al-4V matrix composite reinforced with TiB, characterised using high energy X-ray diffraction" are Ropars, Gourdet, Delfosse, Gourdet, Tricker, and Aeby-Gautier. Volume 624, Issue 1 (2015), pages 179-188, Journal of Alloys and Compounds Article published in the Journal of the Japan Institute of Light Metal in 2005, pages 604-609, by Sato, E., Yamada, T., Tanaka, H., and Jimbo, I., categorises the creep behaviour of metals and alloys at ambient temperature on their crystalline structures.