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Role of powder metallurgical processing and TiB reinforcement on mechanical response of Ti–TiB composites

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ABSTRACT

In this work, titanium–titanium boride (Ti–TiB) composites were synthesized by three different powder metallurgical techniques, namely, spark plasma sintering (SPS), hot iso-static pressing (HIP) and vacuum sintering (VS). The mechanical properties of the composites were determined using the nanoindentation technique. The role of the material processing route and TiB reinforcement employed on the mechanical properties of the composites was investigated. The results revealed that the composites processed by SPS possessed improved mechanical properties relative to those of the composites prepared by the HIP and VS techniques. Furthermore, reinforcement of the composites with TiB enhanced the hardness, elastic modulus and contact stiffness, whereas it reduced the fracture toughness and indentation creep.

1. Introduction

Titanium-based composites are being developed to meet specific requirements for defense, automobile, aerospace, biomedical and allied engineering applications [1,2] because of their good resistance to corrosion and wear [3,4]. Ceramic reinforced titanium composites exhibit good mechanical properties and bonding strength [5,6]. TiB has been identified as one of the reinforcement materials that is most compatible with titanium, exhibiting good thermo-chemical stability and mechanical properties [7,8]. Titanium composites have generally been processed through combustion synthesis [4], solidification [5,69,10], casting [7] and direct laser cladding [8]. However, powder metallurgical techniques have received increasing attention due to their low level of material wastage and the near net shape achieved during processing. Such techniques overcome the drawbacks of conventional methods, such as the pollution of reinforcements, wettability between ceramic particles and their corresponding matrix and intricate processing steps [11–14]. A review of the available literature shows that there is no specific work reported on Ti–TiB composites processed by powder metallurgical techniques such as Hot Iso-static Pressing (HIP), Spark Plasma Sintering (SPS) and Vacuum Sintering (VS).

The mechanical properties estimated by conventional indenters require large force to produce the necessary cracks for analysis [15]. However, nanoindentation is a versatile technique that overcomes the limitations of conventional indentation and offers direct measurement of mechanical properties [16–18]. Limited studies have been reported on the estimation of mechanical properties of Ti–TiB composites by nano indentation technique. The main objectives of this work were to process Ti–TiB composites through SPS, HIP and VS techniques and characterize their mechanical properties through nanoindentation. Additionally, the effects of the processing route and reinforcement adopted on the mechanical properties of the composites were investigated.

2. Materials and methods

Commercially available titanium (Ti-325 mesh and 99.5%), titanium diboride (TiB_2-325 mesh) and ferromolybdenum (β stabilizer) powders were mixed together to prepare the composites. A β stabilizer was added to increase the β phase content of the composites, which is more ductile than the α phase. The two Ti–TiB composites composed of Ti-78%, TiB_2 -6% FeMo-16% and Ti-70%, TiB_2-15%, FeMo-15% were processed through three powder metallurgical techniques, SPS, HIP and VS.

To process composites by SPS, the milled powders were pressed uniaxially at 20 MPa and maintained at approximately 1100 °C. The powders were heated by spark discharge between the particles,

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which facilitates a high heating rate, thereby accelerating the sintering process and reducing the sintering time, which controls the grain structure. To prepare composites by the HIP process, the milled powders were packed in a container and heated to 1200 °C and maintained at 120 MPa to reduce shrinkage. During the process, the environment was maintained at approximately 10^{-6} m bar for a period of 5 h to eliminate internal voids and to improve the homogeneity and bonding strength. To prepare composites by the VS process, the powder mixtures were compressed in a 100 t capacity UTM at a load of 350 kN. Consolidation was carried out in a vacuum furnace maintained approximately at 1200 °C for 5 h. TiB_2 reacted with Ti and transformed into TiB during sintering.

The densities of the composites were determined by the water immersion method. It is an average value of three measurements taken for each sample. The microstructures were studied using X-ray diffractometry (XRD), scanning electron microscopy (SEM) and electron probe microanalysis (EPMA). The mechanical properties (hardness, elastic modulus and contact stiffness, fracture toughness and indentation creep) of the composites were measured through the nanoindentation technique. Among these properties, elastic modulus and hardness hardness of Ti–TiB composites are of great attention due to their higher modulus of TiB relative to titanium [15–18]. Two samples were tested for each composition. Four indentations were done on each sample and adequately spaced such that their behavior was not affected by nearby indentations. The values obtained in the observations were within ±2% deviations.

3. Results and discussion

Table 1 depicts the volume fraction of the TiB phases in the composites measured by an image analyzer. The XRD results (Fig. 1(a) and (b)) confirm the presence of both β and α Ti phases without intermediate phases (Ti_3B_4). The spectra clearly indicate that the composites sintered through the SPS process feature more dominant TiB and Ti phases than the composites processed through the other two techniques (HIP and vacuum sintering).The presence of few TiB_2 peaks in the VS and HIP composites indicate the slow diffusion of boron atoms in TiB, which is similar to the result reported in an earlier reported study [9]. Moreover, there is evidence of FeMo, with a small (310) peak in the VS composite containing 17.6 vol% TiB. The EPMA results (Fig. 2) confirm the presence of Ti (as lighter, gray

Table 1
Mechanical properties of Ti–TiB composites.

Powder (in weight percentage)			TiB reinforcement (in volume percentage)		Nomenclature	Mechanical properties						
Ti	TiB_2	Fe Mo	Target	Estimated		Density (g/cc)	Contact depth (nm)	Hardness (HV)	Elastic modulus (GPa)	Fracture toughness	Indentation creep (%) (MPa $m^{1/2}$)	Contact stiffness (N/m)
78	6	16	20	24	SPS	4.9	832.39	837	162.64	14.53	1.41	0.812
				20.6	HIP	4.92	897.09	819.29	169.51	14.22	1.54	0.801
				17.6	VS	4.2	928.42	600	141.76	12.91	2.03	0.809
70	15	15	40	38.5	SPS	4.75	819.27	992.73	190.46	12.26	1.04	0.819
				38.3	HIP	4.90	874.06	913.27	179.04	11.81	1.28	0.826
				37.9	VS	4.685	866.26	772.64	167.04	10.72	1.38	0.818

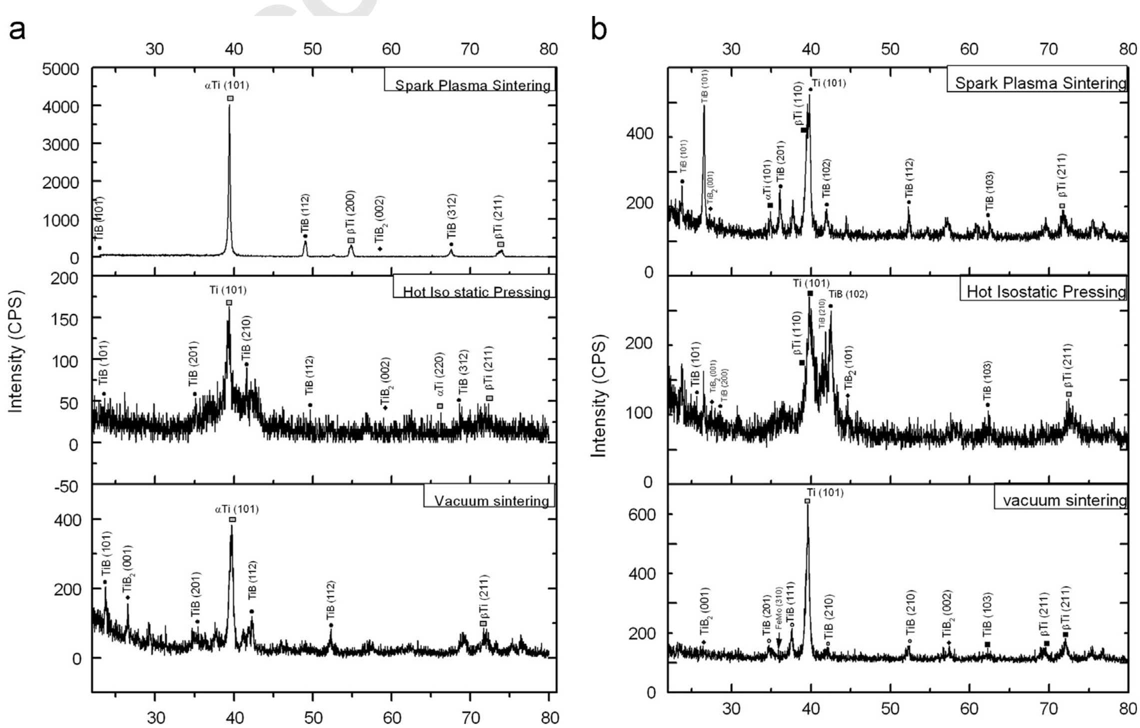

Fig. 1. XRD Patterns.(a) SPS (Ti–24% TiB), HIP (Ti–20.6% TiB), and VS (Ti–17.6% TiB) and (b) SPS (Ti–38.5% TiB), HIP (Ti–38.3% TiB), and VS (Ti–37.9% TiB).

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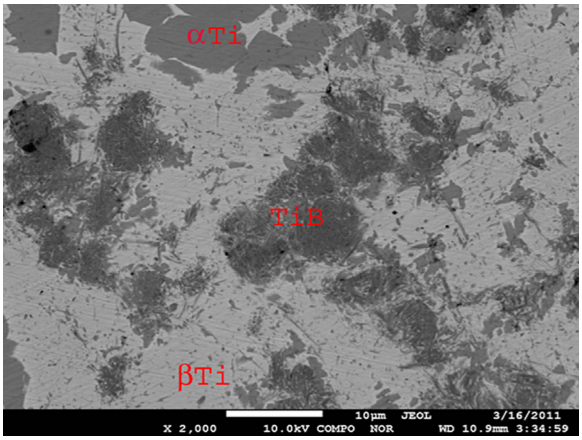

Fig. 2. EPMA image of SPS (Ti–38.5% TiB).

regions) and TiB (as dark regions) in the SPS composite, with a TiB content of 38.5%. The microstructure of Ti–TiB composites (with 38.5 and 24 vol% TiB) processed through SPS depicts fine TiB needles and whiskers as shown in Fig. 3(a) and (b). Similarly, spherical, hard TiB particles were observed in the composites (with 38.3 and 20.6 vol% TiB) processed through HIP as illustrated in Fig. 3(c) and (d). Three types of TiB morphologies such as plate-like, needle-shaped and short, agglomerated whiskers, were observed in the composites (with 37.9 and 17.6 vol% TiB) processed through VS (Fig. 3e and f).

The mechanical properties of all of the processed composites are summarized in Table 1. The standard deviations of all the measurements are within 3.2%. The hardness increased significantly with the content of the TiB reinforcement. The SPS composites (Ti–38.5 vol% TiB) exhibited a high hardness value of 992.73 HV due to its short consolidation period, forming fine TiB needles as shown in Fig. 3(a). Similarly, hard TiB particles increased the hardness of the composites processed through HIP, as confirmed in Fig. 3(b). A low hardness

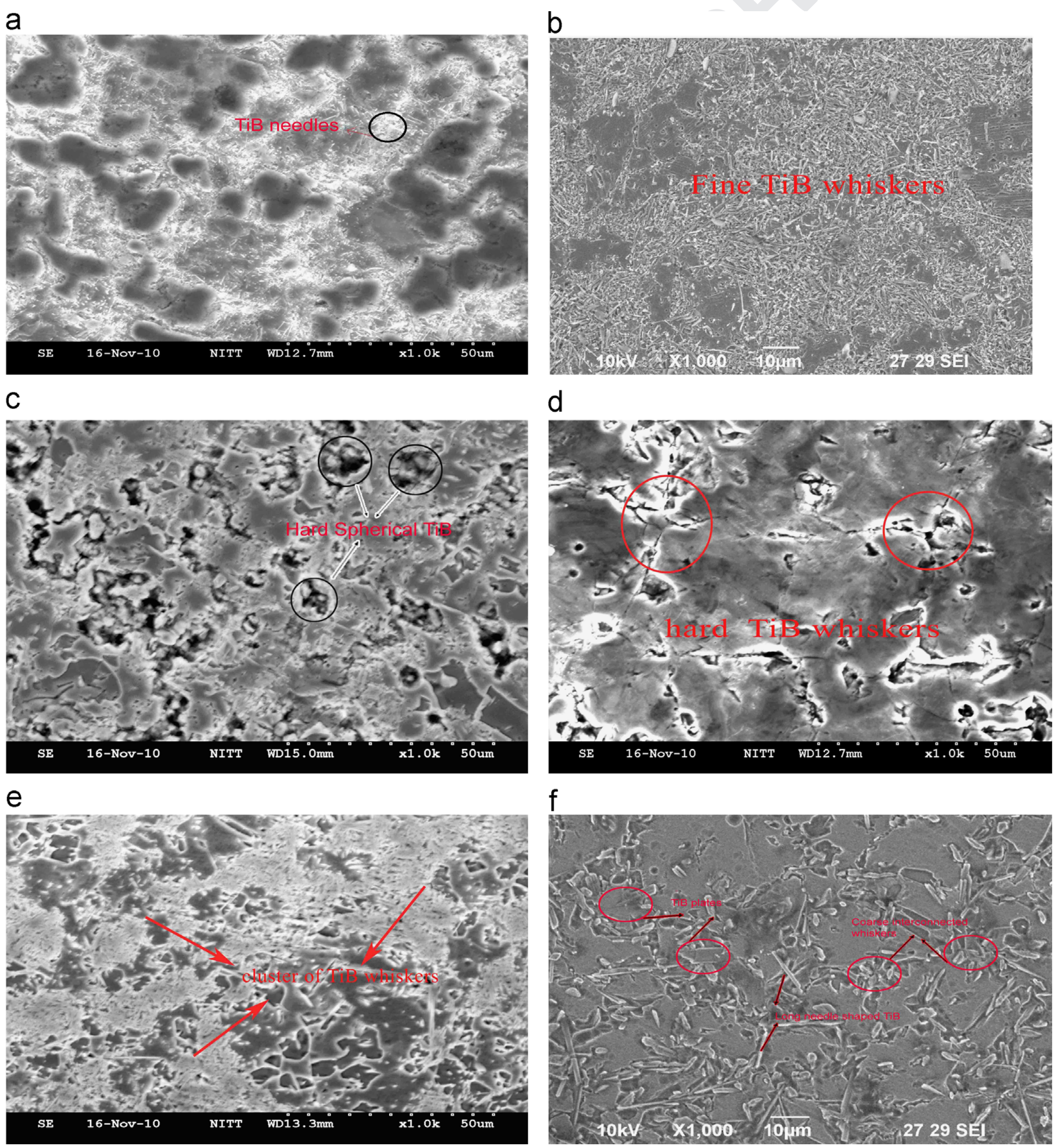

Fig. 3. SEM images (a)SPS- (Ti–38.5% TiB) (b) SPS- (Ti–24% TiB) (c)HIP (Ti–38.3% TiB) (d)HIP -(Ti–20.6% TiB) (e)VS -(Ti–37.9% TiB) and (f) VS (Ti–17.6% TiB).

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value of 600 HV was observed for the VS composites (Ti-37.9 vol% TiB) due to its porosity and the presence of interconnected, needle-shaped whiskers in the matrix, as shown in Fig. 3(c). It was also ascertained that the hardness increased with the decrease in the contact depth of the indenter due to the composite's poor deformation.

The elastic moduli of all of the processed composites increased remarkably with the content of the TiB reinforcement. The elastic modulus of TiB whiskers varies between 324 GPa and 440 GPa [6]. The presence of TiB reinforcements contributed to an increase in elastic modulus that varied from 140 GPa to 190 GPa. The SPS composites (Ti–38.5 vol% TiB) exhibited a higher elastic modulus (190.46 GPa) than the moduli of the other investigated composites due to the presence of fine TiB needles and long TiB whiskers in the matrix, as confirmed in Fig. 3(a). However, the elastic modulus of the VS composites (Ti–17.6 vol% TiB) was relatively lower than the moduli of the SPS and HIP composites due to the presence of small, agglomerated and fine TiB plates in the matrix (Fig. 3(c)).

Furthermore, the fracture toughness of the composites decreased with the content of the TiB reinforcement. The maximum fracture toughness of 14.5 MPa $m^{1/2}$ was observed for the SPS composites (Ti–24 vol% TiB) due to the presence of a higher volume percentage of titanium in the composite. Moreover, the fine TiB needles (Fig. 3(a)) may have decelerated the propagation of cracks from the corners of the specimens by absorbing more energy, leading to an increase in fracture toughness. In the case of the composites processed through HIP (Ti–20.6%TiB), the titanium content of 79.4 vol% led to an increase in fracture toughness, but the resulting value was lower than that of the SPS composites. The density of materials has a distinct effect on fracture toughness [11]. The composites processed by SPS and HIP possessed similar densities and exhibited the same range of fracture toughness value. However, the fracture toughness of the VS composites (Ti–17.6 vol% TiB) was 10.72 MPa $m^{1/2}$, relatively lower than the values measured for the SPS and HIP composites due to the presence of pores and fine TiB plates in the VS composites.

The indentation creep decreased with the increase in the content of the TiB reinforcement. The composites processed by SPS and HIP exhibited the same range of indentation creep. Indentation creep is associated with creep displacement (maximum depth of indentation) and indentation hardness [15]. As hardness increases, indentation creep decreases due to the latter's linear relationship with indentation depth, which was observed to decrease with the increase in the content of the TiB reinforcement. The fine TiB needles in the SPS composites and hard TiB particles in the HIP composites resisted plastic deformation, which reduced the creep values of the composites. The maximum and minimum indentation depths were observed to be 928.42 nm and 819.27 nm for the VS and SPS composites, respectively. An increase in the contact depth of the VS composites caused an increase in the creep value.

The contact stiffness was observed to increase with the volume percentage of TiB. The contact stiffness values of all of the composites were between 0.801 N/m and 0.829 N/m. However, only a small difference in contact stiffness was observed between VS composites containing two different volume percentages of TiB due to the existence of coarse, interconnected whiskers in the matrix, as shown in Fig. 3(c). It has been demonstrated that contact depth affects stiffness values [15]. In this study, the stiffness of the composites increased with a decrease in contact depth, which restricted the deformation of TiB, as clearly indicated by the creep results.

4. Conclusions

The effects of the processing route and TiB reinforcement adopted on Ti–TiB composites were investigated, and the following conclusions were drawn.

- SPS was identified to be the best technique for processing Ti–TiB due to its short consolidation time. The formation of fine grains yielded composites with higher hardness, elastic modulus, fracture toughness, and contact stiffness and lower indentation creep compared to those of the HIP- and VS-processed composites.
- As the content of the TiB reinforcement increased, the hardness, elastic modulus and contact stiffness increased whereas the fracture toughness and indentation creep decreased.
- The mechanical properties showed a close relationship with the contact depth of the indenter. The hardness, elastic modulus and stiffness values were observed to decrease whereas the fracture toughness and indentation creep were observed to increase with the increase in the contact depth of the indenter.

Uncited reference

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