Mechanical Properties of Particulate Reinforced Titanium-based Metal Matrix Composites Produced by the Blended Elemental P/M Route

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The titanium-based metal matrix composites (MMC) reinforced with relatively large-sized ceramic particulates have been produced by the blended elemental (BE) P/M route. The extra low chlorine titanium powder, Al_3Ti master alloy powder, and FeB and Cr_3C_2 ceramic powders were used as starting materials. The dispersion of either TiB or TiC particulate reinforcements in the matrix were therefore made *in situ* during the sintering process. The distribution of particulate reinforcements in the matrix was reasonably random. Tensile tests were done at temperatures up to 923 K. It has been found that the tensile strength of these composites are superior to those of the unreinforced matrix alloys in the whole temperature range examined. The elastic modulus was also increased by the presence of these ceramic reinforcements. No ductility was found for both composites at room temperature, but some ductility was observed at temperatures above 623 K. These observations were interpreted in terms of the failure mechanisms observed using scanning electron microscopy.

KEY WORDS: metal matrix composites, titanium alloy, powder metallurgy, ceramic particulates, tensile strength.

1. Introduction

Titanium alloy are ideally suited for airframe and gas turbine engine components because of their unique combination of mechanical properties. One of drawbacks of titanium alloys, is that the service temperature is limited to 873 K due to a degradation of creep strength, metallurgical stability and environmental resistance.¹⁾

Titanium aluminides are receiving increased attention for application as high performance materials but these alloys are intrinsically brittle, and success in improving low temperature ductility while maintaining high temperature mechanical properties has been limited.

However, for future aircraft design, there is still a demand for high temperature titanium alloys having superior combinations of tensile strength, creep resistance, stiffness, corrosion resistance and thermal stability compared to conventional alloys in the temperature range between 873 and 1273 K.

Fabrication of titanium-based metal matrix composites (MMC) creates considerable potential for improvement in various mechanical properties, particularly in stiffness and strength. There are two basic types of MMC, *i.e.*, long fiber reinforced and particulate reinforced. Of these, development activities are currently centered

around SCS-6 fiber reinforced MMC³⁻⁶⁾ but these are highly anisotropic in properties and fabrication is complex and expensive. Interest has increased in ceramic particulate reinforced MMC in recent years because these have isotropic characteristics and can be processed more cheaply using conventional technologies such as ingot metallurgy,⁷⁾ casting⁸⁻⁹⁾ and powder metallurgy.¹⁰⁻¹⁴⁾

Blended elemental (BE) powder metallurgy (P/M) has been considered to be one of the most attractive net shape technologies¹⁵⁾ for producing various types of titanium-based MMC with tailored microstructures. The pioneering work in this field was carried out by Abkowitz of Dynamet Technology Inc., who first developed a Ti-6Al-4V matrix composite reinforced with TiC particulates.¹²⁾ However, mechanical property data available for the P/M processed MMC are still lacking. Particularly, high temperature tensile data and comparison with the unreinforced matrix alloy have received relatively little attention.

In the present study, titanium alloy-based MMC containing relatively large-sized ceramic particulates have been produced by the proprietary BE P/M route. The details of the fabrication and the results of the microstructural characterization and the high temperature tensile tests done on the composites and matrix alloy

Fig. 1.

Processing steps for the fabrication of BE P/M composite.

will be described in this paper.

2. Experimental Procedure

The composites were made up using the processes of powder blending, cold isostatic pressing (CIP), vacuum sintering and hot isostatic pressing (HIP). As can be seen in Fig. 1, our proprietary technique was incorporated in this BE P/M method, in which matrix alloy-forming powders were blended with either boron-containing ceramic powder such as FeB or carbon-containing ceramic powder such as Cr_3C_2 . During the sintering process, these ceramic powders are dissolved in the matrix and the dissolved and diffused B or C atoms react chemically with titanium to form titanium boride or titanium carbide in the matrix.

Two types of titanium-based MMC were fabricated using this proprietary technique. One type of composites, Ti-5wt%Al-2.5wt%Fe containing 15 wt% titanium boride, was made by blending extra low chlorine (ELCL) titanium powder (Cl < 10 ppm, O ~ 1 000 ppm, -100 mesh), Al_3Ti master alloy powder (-150 mesh) and FeB ceramic powder (-325 mesh). The unreinforced Ti-5Al-2.5Fe matrix alloy, which belongs to high strength α - β type alloy and was developed for medical applications,¹⁶⁾ was also made¹⁷⁾ and tested to show differences from the composites. The other type of composites, Ti-5wt%Al-13wt%Cr containing 10 wt% titanium carbide, were prepared using ELCL titanium powder, Al_3Ti master alloy powder and Cr_3C_2 ceramic powder (-325 mesh). As will be described later, these titanium boride and titanium carbide reinforcements were identified to be TiB and TiC, respectively. The Al_3Ti and FeB ceramic powders were made in our laboratory by crushing the arc-melted button ingots. The titanium, Al_3Ti master alloy powder and ceramic powder were blended in a V-shaped blender. The CIP'ing was done at 400 MPa and vacuum sintering were performed for 14.4 ks at 1573 K. Sintered composites were finally HIP'ed at 1203 K and 200 MPa to produce full dense composites. The oxygen content of the matrix alloys after HIP treatment was 1 450 ppm.

The high temperature tensile tests were performed in the temperature range up to 923 K in a vacuum with 3.5 mm diameter by 16 mm gauge length specimens at a cross head speed of 0.1 mm/min.

3. Results

3.1. Microstructure

The microstructures of an unreinforced Ti-5Al-2.5Fe

Fig. 2. Microstructure of unreinforced BE P/M Ti-5Al-2.5Fe in the HIP'ed condition.

and two types of the ceramic particulate reinforced titanium-based MMC are shown in Figs. 2, 3 and 4, respectively. The corresponding X-ray diffraction patterns are also shown to indicate the phases present in the composites.

The microstructure of as-HIP'ed Ti-5Al-2.5Fe consists of coarse α - β two phase structure with a colony size of ~ 150 μm and massive α phase at the grain boundaries. The Ti-5Al-2.5Fe-based composite results in microstructure consisting of α - β two phase matrix with both needle-shaped and globular-shaped titanium borides. These two types of titanium borides were identified to be TiB by X-ray diffraction analysis, as shown in Fig. 3. The reason why two different types of TiB were formed is not clear at this moment. It can be seen that the TiB reinforcements are distributed reasonably randomly in the matrix. There was no cluster of the TiB reinforcements seen throughout the specimen. The morphology of each alpha lath in the matrix differs significantly between the unreinforced alloy and composite, with the aspect ratio of the laths being markedly smaller in the composite. It seems that the length of lath is limited by the particulate spacing. Both the microstructure of unreinforced matrix alloy and the matrix microstructure of composite are in a state of thermally equilibrium in the as-HIP'ed condition and these will remain unchanged during the high temperature tests up to 923 K. The optical microscopy observation and X-ray analysis of the Ti-5Al-13Cr-based composite in the as-HIP'ed condition revealed a matrix microstructure of primarily β phase and randomly distributed globular-shaped TiC reinforcements, as has been shown in Fig. 4. Small peaks which correspond to the diffraction

Fig. 3. Microstructure of BE P/M Ti-5Al-2.5Fe-15TiB composite in the HIP'ed condition and the corresponding X-ray diffraction scan.

Fig. 4. Microstructure of BE P/M Ti-5Al-13Cr-10TiC composite in the HIP'ed condition and the corresponding X-ray diffraction scan.

Table 1. Summary of mechanical properties of composites and matrix alloys produced by the blended elemental P/M route.

	Test temp. (K/°C)	E (GPa)	0.2 YS (MPa)	UTS (MPa)	EL (%)	RA (%)	Density (g/cm ³)
Ti-5Al-2.5Fe-15TiB	293/ 20*	151	—	1092	0.0	0.7	4.485
	623/350		883	950	1.3	1.7	
	773/500		683	803	1.8	3.5	
	848/575		641	702	2.0	2.8	
	923/650		368	399	6.6	13.8	
Ti-5Al-13Cr-10TiC	293/ 20**	125	1111	1111	0.2	0.7	4.608
	623/350**		979	979	0.2	0.9	
	773/500		835	868	0.9	3.0	
	848/575		709	726	2.1	4.0	
	923/650		300	380	16.5	23.8	
Ti-5Al-2.5Fe	293/ 20		850	947	14.3	35.0	4.462
	623/350		583	725	13.9	41.5	
	773/500		455	584	19.9	52.7	
	923/650		131	147	35.9	70.6	

* Specimen failed before 0.2 % yield strength was obtained.

** Specimen failed before ultimate tensile strength was obtained.

Fig. 5. The temperature dependence of the ultimate tensile strength of BE P/M Ti-5Al-2.5Fe-15TiB composite and BE P/M Ti-5Al-2.5Fe matrix alloy.

Fig. 6. The temperature dependence of the ultimate tensile strength of BE P/M Ti-5Al-13Cr-10TiC composite, and BE P/M Ti-6Al-4V and IMI 829 alloys.

from Ti_3AlC were also observed in the X-ray diffraction scan.

No obvious interaction zone is visible in both composites at the resolution available in optical and scanning microscopy. However, in the case of BE P/M Ti-6Al-4V/TiC composite, the formation of the annulus of Ti_2C around the TiC reinforcement has been observed when using transmission electron microscopy.¹¹⁾

3.2. Mechanical Properties

The mechanical properties of the two composites, *i.e.*, Ti-5Al-2.5Fe-15TiB and Ti-5Al-13Cr-10TiC are given in Table 1, and are presented graphically in Figs. 5 and 6, respectively. The baseline data for unreinforced Ti-5Al-2.5Fe matrix alloy and property data for other high temperature alloys produced by the conventional

BE P/M route¹⁷⁾ are also included in these Table and Figures.

The Ti-5Al-2.5Fe-15TiB composite can achieve remarkably higher elastic modulus at room temperature, 151 GPa, 32 % higher than that of unreinforced Ti-6Al-4V, 114 GPa.¹⁸⁾ Although the Ti-5Al-13Cr-10TiC composite also showed a higher elastic modulus, 125 GPa, than the unreinforced Ti-6Al-4V, this value is considerably lower than the Ti-5Al-2.5Fe-15TiB composite. This result will partly be attributed to the fact that the Ti-5Al-13Cr matrix is almost completely β type alloy, as has been revealed by X-ray analysis, and it is known that the β type alloys generally exhibit lower elastic modulus¹⁷⁾ compared to those for α - β type alloys such as Ti-6Al-4V and Ti-5Al-2.5Fe.

The yield and tensile strength for the Ti-5Al-2.5Fe-15TiB composite were superior to those for the unreinforced matrix alloy in the whole temperature range

Fig. 7. Scanning electron micrographs at low and high magnification of the fracture surfaces taken from BE P/M Ti-5Al-13Cr-10TiC composite tested at room and at higher temperatures.

examined. Although high temperature tensile data for Ti-5Al-13Cr baseline alloy are not taken yet, comparison with those for other high temperature titanium alloys such as Ti-6Al-4V and IMI829 (Ti-5.5Al-3.5Sn-3Zr-0.3Mo-1Nb) reveals that the Ti-5Al-13Cr-10TiC composite also have higher tensile strength.

These results demonstrate that the uniform distribution of relatively large-sized ceramic particulates which have superior mechanical properties at high temperatures relative to the matrix alloy is an effective method for improving the elastic modulus and the tensile strength of conventional titanium alloy. However, the ductility of composites was significantly lower than that of the matrix alloy. At room temperature the composites failed in a brittle manner before ultimate tensile strength was obtained. At the highest temperature, 923 K, measurable ductility was obtained for both composites, as shown in Table 1.

3.3. Fractography

Scanning electron micrographs taken from fracture surfaces of the Ti-5Al-13Cr-10TiC composite are shown

in Fig. 7. Irrespective of the test temperatures, the fracture surface consists of the small portions of cleavage fracture and the main regions of dimpled fracture. Optical micrographs of the cross section of tensile test specimens in the vicinity of the fracture area reveal the cracking within the TiC particulates, as shown in Fig. 8. These observations indicate that the cleavage fracture seen on the fracture surface corresponds to the fracture of the TiC particulate. In addition, it seems most importantly that the fracture within the TiC particulate occurred prior to the dimpled ductile fracture of the surrounding matrix alloy. This may be indicative of sufficient bonding at the interface between the particulate and the matrix alloy.

Figure 9 shows the fracture behavior of the Ti-5Al-2.5Fe-15TiB both at room and at elevated temperatures. As is the case for the Ti-5Al-13Cr-10TiC composite, the fracture initiated owing to cracking within the globular-shaped TiB in preference to the dimpled ductile fracture of the matrix alloy.

Fig. 8. Optical micrographs of a cross sectional area both through (left-hand side micrographs) and away from the fracture surface (right-hand side micrographs) taken from BE P/M Ti-5Al-13Cr-10TiC composite tested at room and higher temperatures, showing the fracture of the TiC particulates (indicated by arrows) prior of failure of the surrounding matrix.

4. Discussion

For the fabrication of the titanium-based MMC using the BE P/M route, there is another technique for dispersing ceramic particulate reinforcements. This technique is simply to blend the intended ceramic powder or particulates such as TiC and TiB₂ with the matrix alloy-forming powders. However, a preliminary experiment done at our laboratory and the report¹⁰⁾ by other researchers revealed that the commercially available very small ceramic particulates ($\sim 1 \mu\text{m}$) tend to agglomerate, and the agglomerated cluster of particulates was found to be the origin of the premature failure during the tensile test even at higher temperatures. To avoid the occurrence of the agglomeration, it is necessary to use relatively larger ceramic particulates. But these are not beneficial for improving composite properties. It is also said¹⁰⁾ that this "simple blending" technique can be limited by interfacial wettability problems, because the ceramic particulates generally have an oxide surface layer which

can affect the adherence of the particulate to the matrix alloy and therefore lead to failure at the interface.¹⁰⁾ Our proprietary "internal reaction" technique can avoid these two intrinsic problems associated with the "simple blending" technique. As seen in Figs. 2 and 3, this technique enables TiB or TiC particulate reinforcements to be dispersed reasonably uniformly in the matrix, which means that there is no macroscopic agglomeration of the FeB or Cr₃C₂ particulates in the powder-mixing stage. In addition, because these reinforcements are formed *in-situ*, contamination at the reinforcement/matrix interface does not occur and hence excellent bonding at the interface can be achieved,¹³⁾ which enables the sufficient load transfer from the matrix to the ceramic reinforcements.

The actual strength of the ceramic particulate is generally very poor primarily due to the existence of stress-concentrating flaws such as voids within the ceramic. Contrary to this, the ceramic particulates which were formed *in-situ* during the sintering process is

Fig. 9. Optical micrographs of a cross sectional area away from the fracture surface taken from BE P/M Ti-5Al-2.5Fe-15TiB composite tested at room and higher temperatures, showing the fracture of the TiB particulates (indicated by arrows) prior to failure of the surrounding matrix.

expected to have almost no macroscopic flaws, since it is reported that the ceramic formed in this manner has been proved to be single crystals.^{9,13)}

Therefore, these three advantages, *i.e.*, reasonably randomly distributed particulate reinforcements, excellent bonding at the reinforcement/matrix interface and the lack of macroscopic flaws within the ceramic reinforcement, would mainly be responsible for the increase in flow stress observed in the present titanium-based MMC.

As shown in Figs. 2 and 3, the microstructure of the matrix has been changed from one with similarly aligned α laths in the unreinforced material to one with relatively randomly oriented low aspect ratio α laths. This microstructural change is known to lead to higher strength by a reduced slip length.^{17,19)} Thus, the refined matrix microstructure compared with that of the unreinforced material would also be responsible for the

increased strength of the composites.¹¹⁾

The tensile ductility of the composites produced in this study showed almost zero at room temperature. Because the fracture initiated within the ceramic particulates and is followed by the ductile failure of the surrounding matrix alloy, it may be reasonable to consider that the size, morphology and distribution of the reinforcement is mainly responsible for the performance of the composite under tensile condition. In fact, it is reported that the tensile strength of the TiC reinforced Ti composites is mostly sensitive too the size of reinforcement particle.⁹⁾ Therefore it will be suggested that further refinement of the ceramic reinforcement through fabrication modifications such as the use of much smaller starting powder materials, may lead to additional improvement in the room temperature tensile ductility of these composites.

5. Conclusion

Two types of the titanium-based metal matrix composites, Ti-5Al-2.5Fe-15TiB and Ti-5Al-13Cr-10TiC, were produced using the proprietary blended elemental P/M route, whereby ceramic powders such as FeB and Cr_3C_2 were used in an attempt to disperse either TiB or TiC particulate reinforcements *in-situ* in the matrix.

The conclusions obtained are summarized as follows:

(1) It seemed likely that there was no agglomerated cluster of FeB or Cr_3C_2 in the powder-mixing stage, and TiB of both needle and globular morphology and TiC of only globular morphology were found to be formed and distributed reasonably randomly in the matrix.

(2) The elastic modulus and tensile strength of these two composites were greatly increased compared to those for unreinforced matrix alloys, demonstrating that the uniform distribution of ceramic particulates in the matrix is an effective method for improving the mechanical properties of conventional titanium alloys.

(3) No ductility was found for both composites at room temperature, but some measurable ductility was obtained at temperatures above 623 K. The tensile fracture originated from the cracking within the ceramic reinforcements, followed by the ductile failure in the surrounding matrix alloy.

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