Influence of Processing on the Mechanical Properties of Ti-6Al-4V-Based Composites Reinforced with 7.5 mass% TiC and 7.5 mass% W

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7.5 mass% TiC and 7.5 mass% W powder blends were densified with Ti-6Al-4V blends by combined cold and hot isostatic pressing (CHIP) method to result in three types of composites with differing processing histories, namely hot isostatic pressing (HIPping), extrusion, and casting, which were compared with their monolithic counterparts. Tensile and microhardness tests, along with compressive testing, were carried out to investigate the mechanical properties of powder metallurgy (P/M) Ti-6Al-4V-7.5%TiC-7.5%W composites at ambient temperature. Dissolution of W powder in the Ti-6Al-4V matrix during consolidation seems to be limited by the presence of TiC particles, which can in turn influence mechanical properties of the composites. Cast Ti-6Al-4V-7.5%TiC-7.5%W composite exhibits superior hardness, yield strength, and tensile strength, with a decrease in ductility, compared to its monolithic counterpart and other composites. [doi:10.2320/matertrans.MER2008049]

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1. Introduction

As a growing number of orthopedic devices are being installed in younger and more active patients, i.e., approximately 500,000 total hip replacement surgeries conducted in the U.S. alone in the year 2000,¹ there is an increasing demand for improved orthopedic materials that can provide adequate mechanical properties.² Among the conventional orthopedic alloys such as stainless steels, cobalt-base alloys, and titanium-base alloys that have been used in the medical device industry,³–⁸ great attention has recently been paid to Ti-6Al-4V alloy, developed originally for use in aerospace applications, due to its comparatively lower modulus, high strength, superior corrosion resistance, and biocompatibility.⁹–¹⁶ In particular, the excellent corrosion resistance and biocompatibility of Ti-6Al-4V is attributed primarily to the formation of a tenacious oxide layer upon contact with air.¹²,¹¹,¹⁵

Despite the increasing popularity of the Ti-6Al-4V alloy in the medical device field, there is a concern about its poor wear resistance, especially for use in the replacement of highly stressed bone implants or wear-prone prostheses. Attempts have been made to improve its wear resistance by reinforcing the alloy with a hard ceramic phase, such as W, TiC, or TiB₂,¹² or with a combination of more than one ceramic phase, as the use of the more abrasive secondary phase has been found to be a highly effective method to enhance the wear resistance of metal matrix composites.¹⁷,¹⁸ Although the concurrent presence of W and TiC particles is expected to increase the strength and hardness of Ti-6Al-4V, as has been proved for Ti-based composites from Ref. 19, excessive additions of reinforcement particles can also degrade ductility severely.

A Ti-6Al-4V-based composite reinforced with 7.5 mass% TiC and 7.5 mass% W particles may be of particular interest, because the hardness, and most likely the wear resistance, of the composite may be maximized with limited sacrifice in ductility, as has been reported from the previous study of Ti-based composites reinforced with various amounts of TiC and W particles.¹⁹ In the present study, we processed Ti-6Al-4V-based composites reinforced with 7.5 mass% TiC and 7.5 mass% W particles through various processing routes, i.e., hot isostatic pressing (HIPping), extrusion, and casting, to examine their mechanical properties and to investigate the influence of the processing route on the microstructure and mechanical properties of the composites at ambient temperature. Microhardness, compressive, and tensile tests were performed and the results were compared with those of their monolithic counterparts.

2. Materials and Experimental Procedure

Three different processing methods, namely HIPping, extrusion, and casting, were applied to produce un-reinforced Ti-6Al-4V alloys (termed “Monolithic 1, 2, and 3”) and Ti-6Al-4V-based composites reinforced with 7.5 mass% TiC and 7.5 mass% W (termed “Composite 1, 2, and 3”), as displayed in Table 1. All alloys and composites were processed such that the combined cold and hot isostatic pressing (CHIP) technology was utilized as follows.²⁰ Ti-6Al-4V (all compositions are given in weight percent) powder blends (here called “Monolithic”) were prepared using Ti powder (<150 μm) and Al-V master alloy powder. For composite materials, W and TiC powders (both <10 μm) were mixed and was compacted in a die of 10 mm diameter. The compacted materials were HIPped at 1100°C for 45 min with a 350 MPa argon gas pressure.

Table 1 Composition and processing history of Ti-6Al-4V and Ti-6Al-4V-7.5TiC-7.5W composites examined.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Composition (mass%)</th>
<th>Processing history</th>
</tr>
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<tbody>
<tr>
<td>Monolithic 1</td>
<td>Ti-6Al-4V</td>
<td>HIPped</td>
</tr>
<tr>
<td>Monolithic 2</td>
<td>Ti-6Al-4V</td>
<td>Extruded</td>
</tr>
<tr>
<td>Monolithic 3</td>
<td>Ti-6Al-4V</td>
<td>Cast</td>
</tr>
<tr>
<td>Composite 1</td>
<td>Ti-6Al-4V-7.5%TiC-7.5%W</td>
<td>HIPped</td>
</tr>
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<td>Extruded</td>
</tr>
<tr>
<td>Composite 3</td>
<td>Ti-6Al-4V-7.5%TiC-7.5%W</td>
<td>Cast</td>
</tr>
</tbody>
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were added and blended into Monolithic blends before compaction. The powder blends were densified by the combined cold and hot isostatic pressing method (CHIP method\textsuperscript{20}), as described in the following steps. First, green billets of Monolithic and Composite blends were produced by cold isostatic pressing (CIP). These billets were then vacuum-sintered and densified by HIP, followed by slow cooling within the press (forming Monolithic 1 and Composite 1). Monolithic 2 and Composite 2 were produced by subsequent extrusion, followed by vacuum annealing and furnace-cooling. On the other hand, for cast alloy and cast composite (Monolithic 3 and Composite 3), the Vertical Direct Chill (VDC) process\textsuperscript{21} was applied, followed by HIPping and annealing.

The microstructure of the products was evaluated by optical microscopic observation of cross-sections produced by grinding on SiC paper and polishing with 0.05 μm colloidal alumina, followed by etching with a modified Kroll’s reagent (5% nitric acid, 10% HF, and 85% water) for several to hundreds of seconds. Matrix microhardness was measured with a Vickers indenter using a 200 g load and an indent time of 10 s on epoxy-mounted cross-sections in matrix regions apart (\( \sim 1 \) mm) from W particles. Tensile tests were performed at ambient temperature on specimens machined to ASTM E-8 proportional standards with 36 mm gauge length and 6.4 mm gauge diameter.\textsuperscript{22} The crosshead speed was 12.7 mm/min, corresponding to an initial strain rate of \( 6.2 \times 10^{-3} \text{ s}^{-1} \). The strain was measured with a typical extensometer with 25.4 mm gauge length. The compression specimen was of circular cylindrical shape with a diameter of \( \sim 7 \) mm and an aspect ratio of 3 : 1, according to ASTM standards E-9.\textsuperscript{23} The compression test was carried out to strains of over 10%, well past its yield point, maintaining a low strain rate of \( 1 \times 10^{-4} \text{ s}^{-1} \) using a computer-controlled servohydraulic test system.

3. Results and Discussion

3.1 Microstructure

Figures 1(a)–(f) show micrographs of etched cross-sections for three monolithic Ti-6Al-4V alloys (Monolithic 1–3) and three Ti-6Al-4V-based composites reinforced with 7.5 mass% TiC and 7.5 mass% W (Composite 1–3) processed by HIPping, extrusion, and casting, respectively. The characteristic features include an etched acicular matrix (Figs. 1(a), 1(c), and 1(e)), white and round W particles and their surrounding dark-etched shell (Fig. 1(b)), and TiC particles with the size ranging from \( \sim 2 \) to \( \sim 20 \) μm (Figs. 1(b), 1(d), and 1(f)). The etched acicular matrix was identified previously\textsuperscript{6,7} as the Widmanstätten \( \alpha/\beta \) matrix structure with most of the W segregated in the \( \beta \) phase and the dark-etched shell as a matrix diffusion zone with high W content from the formation of non-equilibrium phases. As for the Widmanstätten \( \alpha/\beta \) structure, the \( \alpha \) phase began to appear in the form of platelets (Figs. 1(a), 1(c), and 1(e)) as the alloy slowly cooled in the press from near the transus temperature\textsuperscript{24} with some \( \beta \) phase (thin dark regions in Figs. 1(a), 1(c), and 1(e)) retained between \( \alpha \) platelets (Fig. 2\textsuperscript{25}); the majority of matrix in Monolithic 1–3 consisted, however, of primary \( \alpha \) platelets, which is in good agreement with the report by Jovanović et al.\textsuperscript{26} where approximately 95 vol% \( \alpha \) and only 5 vol% \( \beta \) phase was observed for furnace-cooled Ti-6Al-4V. Additionally, there was no martensitic structure, designated as \( \alpha' \) phase and usually formed upon the quenching process,\textsuperscript{27,28} observed in the matrix of Monolithic 1–3, as expected for the slow cooling process.\textsuperscript{26}

Visual examination of all samples found no macroporosity, particularly at the grain boundaries or interfaces. Additionally, there was no perceptibly unbonded or delaminated interface detected between the matrix and reinforcement particles, indicating that both W and TiC can be a good choice for reinforcement of Ti-based alloys.\textsuperscript{29} It is of interest to note that the average thickness of \( \alpha \) platelet (Table 2) for both Monolithic 1 and 3 (12–16 μm), with an exception of Monolithic 2 (1.7 μm) processed by extrusion, was comparatively larger than the reported values of \( \sim 6–7 \) μm\textsuperscript{26} after annealing at similar temperatures (800–900°C), due probably to the significant difference in sample size. On the other hand, it is in approximate agreement with another measured value (\( \sim 17 \) μm) reported by Yapici et al.\textsuperscript{30} It was evident that the presence of W and TiC particles had effects on the microstructural formation of Ti-6Al-4V matrix in Composite 1–3. In particular, the average width of \( \alpha \) platelet was smaller (\( \sim 2 \) μm, Table 2) and its colony size was also finer (\( \sim 45 \) μm) in Composite 2, as compared to those of Composite 1 and 3. Similar microstructural refinements due to the presence of TiC or TiB particles in Ti alloys were also reported elsewhere.\textsuperscript{31,32} The microstructure of Monolithic 2, formed by the application of extrusion process, also showed unique characteristics as compared to Monolithic 1 and 3. Monolithic 2 exhibited much finer thickness of \( \alpha \) platelet (\( \sim 2 \) μm) and severely deformed and elongated primary \( \alpha \) platelets with a high aspect ratio of \( \sim 10 \). Both the extruded alloy and the composite (Figs. 1(c) and 1(d)) showed no evidence of non-uniform flow or macroscopic cracking, as is often observed in materials processed by this method. In addition, the presence of \( \alpha \) platelets was not easily distinguishable from \( \beta \) phase primarily due either to the relatively large volume percent of \( \beta \) phase completely mixed in the matrix or to the fact that the extrusion deformation was accompanied by recrystallization and growth processes.\textsuperscript{30} The extrusion process in this study was performed at temperatures slightly above the \( \beta \) transus temperature (\( \sim 1000°C \)) in which case the volume fraction of \( \beta \) phase present in the matrix was reported to be up to 38% under standard heat treatment conditions,\textsuperscript{33} though the actual microstructure can vary, depending on the exact extrusion and subsequent annealing temperatures.\textsuperscript{24}

According to previous investigations,\textsuperscript{6,8} the fine W particle sizes (\( \sim 10 \) μm), as was used in this study, resulted in a larger amount of W dissolution in both Ti and Ti-6Al-4V matrices, as there indeed exists a theoretically complete solid solubility of W in Ti at elevated temperatures (Fig. 2), with W stabilizing \( \beta \) phase in Ti. To the contrary here, the effect of small W particle size on the degree of dissolution into the matrix was negligible in Composite 1–3 under the current processes, as revealed by the matrix hardness values displayed in Fig. 3, probably due to the concurrent presence of TiC particles which may have affected W dissolution into the matrix.\textsuperscript{19}
3.2 Mechanical properties at ambient temperature

In order to estimate the effect of processing history on local matrix strengthening due to W addition, hardness measurement was performed on the matrix of each material as indicated in Fig. 3. The hardness values of both Monolithic 3 and Composite 3, processed by the casting method, exceed slightly those of Monolithic 1 and 2 and Composite 1 and 2, with both Monolithic 1 and Composite 1 showing the lowest. The hardness values measured for Monolithic 1–3 range from \( \approx 332 \) to \( 390 \) HV, in general agreement with reported literature data (\( \approx 320–380 \) HV\(^{26,30,34} \)). Additionally, the hardness value of Monolithic 2 (\( \approx 368 \) HV) matches those of other extruded Ti-6Al-4V (\( \approx 350–380 \) HV) alloys with the similar heat treatment\(^{30} \) and that of Monolithic 3 (\( \approx 390 \) HV) is also in good agreement with a literature value reported for other cast Ti-6Al-4V alloy (\( 414 \pm 15 \) HV\(^{35} \)).

Shown in Fig. 4 are the tensile stress-strain curves for Monolithic alloys 1–3 and Composite 1–3 at ambient temperature. As expected, the yield strength of Monolithic 1–3 scaled roughly with their hardness results, with Monolithic 3 displaying the highest yield strength (979 MPa) and Monolithic 1 the lowest (902 MPa), although the complete tensile behavior of Monolithic 3 could not be obtained because it fractured prematurely near its yield point, which is seen magnified in the inset in Fig. 4. As opposed to Monolithic 1–3, tensile results of Composite 1–3 showed completely different behavior. They either fractured before reaching their ultimate tensile stresses (Composite 1 and 3) or yielded at a lower stress level (883 MPa) with limited ductility (Composite 2). One likely reason for this is that damage accumulation occurred in the form of cavitation during tension, thus reducing the effective cross-sectional area of the composites and inducing stress concentration.\(^{5} \)

The yield strength of both Composite 1 and 3 improved with additions of TiC and W particles, as compared to their monolithic counterparts, as also reported elsewhere,\(^{6–8,36} \).
whereas the extrusion process seemed to degrade the strength of Composite 2, as compared to Monolithic 2, showing the lowest yield (883 MPa) and tensile strengths (966 MPa) with moderate ductility (4.8%). Conversely, the additional extrusion process applied to Monolithic 2 compared to Monolithic 1 (CHIPping without extrusion) increased yield strengths (from 902 to 950 MPa) and ultimate tensile strengths (from 975 to 1036 MPa) with only a slight decrease in elongation (from 14.5 to 12.7%). Some degree of TiC particle clustering was observed in Composite 1–3 (Figs. 1(b), 1(d), and 1(f)), which may have contributed to their low elongation behavior, because the clustered TiC particles can constrain the matrix plastic flow to produce increased stresses in the vicinity of the clustered particles and high triaxiality in the matrix between the particles, as affirmed by other researchers.37–39) In fact, this is a rather common phenomenon observed in Ti-based alloys reinforced with low-volume TiC particles.31,37,40)

It is of particular interest to note that the average thickness of α platelet, known to have a large effect on the mechanical properties of Ti-6Al-4V alloys,24,28,30) was not reflected clearly in the hardness measurement results of Monolithic 1–3 (Fig. 3) nor in their tensile behaviors (Fig. 4) where Monolithic 3 with much greater α thickness (~12 μm) showed conversely a higher hardness value and strength than Monolithic 2 with α thickness of ~1.7 μm. This may be due to the relatively higher density of porosity and the greater content of β phase usually present in extruded alloys than in cast ones. The compressive stress-strain curve for Composite 1 is presented in Fig. 5 in comparison to that of the unreinforced Ti-6Al-4V control alloy.6) Addition of 7.5 mass% W and 7.5 mass% TiC improved the compressive yield strength of Composite 1 by about 20%, from 918 to 1103 MPa, as compared to its monolithic counterpart.

### Table 2

<table>
<thead>
<tr>
<th>Processing routes and materials</th>
<th>Average α plate thickness (μm)</th>
<th>Average colony size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HIPped; Monolithic 1</td>
<td>16</td>
<td>78</td>
</tr>
<tr>
<td>Extruded; Monolithic 2</td>
<td>1.7</td>
<td>45</td>
</tr>
<tr>
<td>Cast; Monolithic 3</td>
<td>12</td>
<td>77</td>
</tr>
</tbody>
</table>

### Fig. 2

Binary phase diagram for the Ti-W system from Ref. 25).

### Fig. 3

The effect of the processing method on matrix hardness.

### Fig. 4

Tensile stress-strain curves for Monolithic 1–3 and Composite 1–3. Shown in the inset is the magnified elastic region particularly for Monolithic 3, which exhibited premature failure during tensile test.
processed via the same route\(^6\) (Fig. 5). The main differences with respect to its tensile curve in Fig. 4 are slightly higher yield stress (1103 MPa for compressive yield strength), higher strain-hardening rates, much higher ultimate stress, and higher fracture strain (>10\% for compression). Tension/compression asymmetry with higher strength and strain hardening rate in compression than in tension is reported to be observed generally in Ti\(^{41}\) and Ti-6Al-4V alloys,\(^{30}\) ascribed possibly to the non-random texture orientation developed during processing or a cavitation mechanism during tension.

4. Conclusions

HIPping, extruding, and casting processes of P/M Ti-6Al-4V with and without TiC and W reinforcements were carried out successfully. It was revealed from the effect of the processing history on the microstructure and mechanical properties of the alloys and composites that:

1) When Ti-6Al-4V alloy was reinforced with TiC and W particles, most of the W and TiC remained undissolved, resulting in a particulate-reinforced “composite”. The microstructure of the Ti-6Al-4V matrices appeared as typical Widmanstätten \(\alpha/\beta\) structures with the lamellar spacing and colony size depending on the processing history.

2) There was a significant improvement in yield strength with some sacrifice in ductility when a Ti-6Al-4V alloy was reinforced with TiC and W particles, whereas the matrix hardness was not influenced by addition of the reinforcements due to the limited dissolution of W into the matrix, apparently affected by the concurrent presence of TiC particles.

3) The strength and hardness of the cast Ti-6Al-4V-7.5\%TiC-7.5\%W composite was superior to those of the composites possessing the same composition but processed by the HIPping or extrusion methods. This observation held also for their monolithic counterparts, with the strength and hardness of the cast Ti-6Al-4V alloy being superior in comparison to those of the HIPped and extruded ones.

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REFERENCES