Effect of Forging Temperature on Microstructure and Mechanical Properties of In situ (TiB+TiC)/Ti Composites

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In this paper, 5 vol% (TiB+TiC)/Ti-1100(Ti–6Al–2.75Sn–4Zr–0.4Mo–0.45Si) composites were fabricated using in situ technologies between Ti and B4C powders. Phase identification was carried out by X-ray diffraction. β transus temperature of the composite was measured by metallographic techniques. The composite after ingot breakdown was forged in various temperature ranges. Microstructure of the composite after forging at various temperatures was studied by optical microscopy (OM). Mechanical properties of the composite after forging at various temperatures were evaluated by tensile tests at 873 K. It was found that the β transus temperature of the composites was around 100 K higher than that of monolithic Ti-1100 alloy. Different microstructures were obtained after forging at different temperatures. The composite with different microstructures offered different mechanical properties, which was shown in the tensile tests. [doi:10.2320/matertrans.47.1750]

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1. Introduction

With the rapid development of technology in aerospace and atomic energy, the requirement for materials for such applications is increasing. Titanium matrix composites (TMCs), reinforced with ceramic particles, have considerable potential for improving properties and service temperature and can be extensively applied in areas such as aerospace, advanced weapon systems and the automotive industry, because of their high specific strength, good specific modulus and resistance to elevated temperatures.1,2) TMCs with better properties can be prepared by the in situ technique. Compared to powder metallurgy method, the preparation process by in situ techniques is simple and equipments used in traditional casting also can be used in this method. Furthermore, this method overcomes the shortcomings of traditional techniques, such as the problems of pollution of reinforcements and wettability between ceramic particles and matrix encountered in the casting technique. Therefore in situ synthesized TMCs have been widely studied.3–5) However, the machining property of these composites is inferior because of the distribution of the reinforcement with high hardness in the soft matrix. It is difficult to machine a workpiece with complex configuration, which restricts the application and development of the composites. In order to solve these problems, hot forging of materials can be used, which saves raw materials and reduces cost. On the other hand, Ti-1100(Ti–6Al–2.75Sn–4Zr–0.4Mo–0.45Si) is a near α Ti alloy. Because of their superior microstructure stability, toughness and excellent specific strength, near α Ti alloys and its composites (TMCs) are used very popular for structural applications at high temperature. Near α Ti alloys offer considerable scope for obtaining a variety of microstructures by thermomechanical processing6) (TMP). Alloy specific studies have demonstrated significant benefits of using thermomechanical processing to alter microstructure and mechanical properties of near α Ti alloys.7–10)

In this paper, 5 vol% (TiB+TiC)/Ti-1100 composites were fabricated using in situ reactions between Ti and B4C powders. β transus temperature of the composite was measured by metallographic techniques. The composite after ingot breakdown was forged at various temperatures. Microstructure of the composite after forging was studied by optical microscopy (OM). Mechanical properties of the specimens were evaluated by tensile tests at 873 K.

2. Experimental Procedure

For preparing 5 vol% (TiB+TiC)/Ti-1100 TMCs, the raw materials were grade I sponge titanium (99.95 mass%, average particle size: 1–5 mm), B4C powders (99.8 mass%, average particle size: 5–7 μm), aluminum thread (98 mass%), silicon (99.5 mass%, average particle size: 10–20 μm), sponge zirconium (98.8 mass%, average particle size: 1–5 mm) and master alloys of other alloying elements such as Ti–Sn and Al–Mo. The nominal alloy composition of Ti-1100 was Ti–6Al–2.75Sn–4Zr–0.4Mo–0.45Si and the amount of B4C powders added in the present material was 1.86 mass%. Firstly, the stoichiometric amounts of sponge titanium, B4C powders, Al thread, sponge Zr, and master alloys of Ti–Sn and Al–Mo were blended thoroughly, and then they were compacted into pellets with a mold using a hydraulic pressure machine (the maximum pressure is 2 × 107 N). The size of the pellets is 60 mm in diameter and 450 mm in length. The pellets were joined with the electrode of a consumable vacuum electrode arc melting (VAR) furnace. Then, the pellets were melted homogeneously in the furnace. In order to ensure the chemical homogeneity of the composites, the ingots were melted at least three times. The reinforcements were synthesized utilized the reaction between Ti and B4C as following reaction:

\[ \text{Ti} + \text{B}_4\text{C} \rightarrow 4\text{TiB} + \text{TiC} \]  (1)

Phase identification was carried out via X-ray diffraction using D-max IVA automatic X-ray diffractometer. Metallographic techniques were used to measure the β transus temperature of the composite. The specimens for β transus temperature tests were cubes with a gauge size of 10 mm. The
specimens were heated in a box furnace with a temperature accuracy ±2 K, after hold for 30 minutes then quenched into water. The forging of the composite was performed using a hydraulic pressure machine (the maximum pressure is $3 \times 10^6$ N). The molds were heated to 1073 K before forging, and the temperature of specimens during forging was measured using infrared ray techniques. The amount of deformation was calculated as the total reduction in cross section (The total reduction in cross section is $A_0/A_i$, where $A_0$ is the cross sectional area before hot forging and $A_i$ is the cross sectional area after hot forging). The specimens after forging were quenched into water. For optical microscopy (OM) were cut from hot-forging rods. Then the specimens were prepared using conventional techniques of grinding and mechanical polishing. The specimens were etched in Kroll’s reagent (composition: 1–3 mL HF, 2–6 mL HNO$_3$, 100 mL water). The microstructure of specimens was characterized using a LECO2000 optical microscopy. The hardness values of the composite were measured by an HR-150A hardness test. The size of specimen plate for tensile tests was 1.5 mm in thickness and 40 mm in length. They were machined from hot-forging rods with the specimen axis parallel to the hot-forging direction. Three specimens for each hot-forging treatment were tested using a SHIMADZU AG-100KNA test machine. The average strain rate was $2.0 \times 10^{-3}$ s$^{-1}$.

3. Results and Discussion

3.1 Phase identification and reinforcement observation

Figure 1 shows XRD patterns of the specimen after VAR. It confirms that Ti has reacted with B$_4$C and TiB and TiC have formed in composite during preparation process. The optical microscopies of reinforcements of composites are presented in Fig. 2. The results of energy dispersion X-ray spectroscopic (EDS) study show that the gray needles are TiB, while the near-exiquaxed reinforcements are TiC. The different shapes of the reinforcements are related to their crystal structures and growth mechanisms during solidification process.$^{11}$

3.2 The β transus temperature and various forgings of 5 vol% (TiB+TiC)/Ti-1100 composite

It is well known that β transus temperature is a very important parameter for the thermomechanical processing of Ti alloys and its composite. Metallographic techniques were used to measure the β transus temperature of the composite, and the results were presented in Fig. 3. The β transus temperature of the present composite was identified as

![Fig. 1 X-ray diffraction patterns of 5 vol% (TiB+TiC)/Ti-1100 composite after VAR.](image1)

![Fig. 2 Optical micrographs of reinforcement in the composite are presented in (a), and (b), respectively (unetched).](image2)

![Fig. 3 Optical micrographs of 5 vol% (TiB+TiC)/Ti-1100 composite after quenched from (a) 1393 K and (b) 1403 K.](image3)
The $\beta$ transus temperature of the composites was around 100 K higher than that of monolithic Ti-1100 alloy (the $\beta$ transus temperature is around 1288 K).

From Ti-B and Ti-C binary alloy phase diagrams, it can be known that B is almost immiscible in $\alpha$-Ti matrix. The effect of B element on $\beta$ transus temperature is not obvious. But the solubility carbon in $\alpha$-Ti matrix can reach about 0.3 mass%. So the reaction in eq. (1) during preparation of the composite could not perform completely, the increase of $\beta$ transus temperature due to B$_4$C addition results from the dissolution of carbon in matrix. Carbon is one of the $\alpha$ phase stabilizers for Ti alloys, and the effect of carbon in this experiment can be explained by Ti-Al-C ternary phase diagram. The matrix alloys contain $\alpha$ stabilizers Al, Sn, Zr, and O. The effect of these elements can be represented in terms of an Al equivalent content. According to Rosenberg, the equivalent of Al content can be expressed as $[\text{Al}]_{\text{eq}} = [\text{Al}] + [\text{Sn}]/3 + [\text{Zr}]/6 + 10[\text{O}]$. The O concentration in specimens was ranged from 0.05% to 0.09%. For the present matrix alloy $[\text{Al}]_{\text{eq}}$ falls in the range 8.1–8.6. Thus we can refer to the Ti-8Al-C phase system to discuss the $\beta$ transus temperature of the matrix alloy under study. A vertical section of the Ti-8Al-C phase diagram is shown in Fig. 4, and the present composition point of the composite is marked with A in Fig. 4. According to this vertical section, carbon increases the $\beta$ transus temperature rapidly when the C concentration in matrix alloy is below 0.28 mass%. The $\beta$ transus temperature of the composite identified in this experiment is slightly lower than that presented in the Ti-8Al-C ternary phase diagram, which results from Mo and Si which are $\beta$ stabilizers. From Fig. 4, it can be known that the ratio of the amount of carbon dissolved into matrix to that of carbon precipitated as TiC is about 7:3 for this composite at $\beta$ transus temperature.

According to the results of $\beta$ transus temperature measured in this experiment and Fig. 4, three type forgings were conducted to the composite in this experiment, and the parameters used in this forgings are listed in Table 1, and

![Fig. 4 Vertical Section of Ti-8Al-C equilibrium alloy phase diagram.](image)

<table>
<thead>
<tr>
<th>No</th>
<th>Forging temperature range (K)</th>
<th>Forging pressure</th>
<th>Amount of deformation</th>
</tr>
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<tbody>
<tr>
<td>$A_1$</td>
<td>1453–1403</td>
<td>$4.1 \times 10^5$ N</td>
<td>4.3</td>
</tr>
<tr>
<td>$A_2$</td>
<td>1373–1323</td>
<td>$4.8 \times 10^5$ N</td>
<td>4.1</td>
</tr>
<tr>
<td>$A_3$</td>
<td>1333–1263</td>
<td>$6.5 \times 10^5$ N</td>
<td>4.6</td>
</tr>
</tbody>
</table>

![Fig. 5 Optical micrographs of the composite are presented in (a) in cast, (b) after $A_1$ forging, (c) after $A_2$ forging and (d) after $A_3$ forging.](image)
forging temperature ranges used in this experiment are also presented in Fig. 4. $A_1$, $A_2$ and $A_3$ are usually named forging in the $\beta$ phase field, forging in the $(\alpha + \beta)$ field and forging started in the $(\alpha + \beta)$ field and finished in the $\alpha$ phase field, respectively.

### 3.3 Microstructure of the composite after forging at various temperatures

Optical microscopies of the composite before and after forging are presented in Fig. 5. From Fig. 5(a), it can be seen that $\alpha$ grains of the composite in cast is coarser and the size of $\alpha$ grains is about 30–60 um. As presented in Fig. 5(b), Widmanstatten microstructure (containing $(\alpha + \beta)$ lath grains totally) is obtained after $A_1$ forging. As presented in Fig. 5(c), bimodal microstructure (containing equiaxed $\alpha$ grains and $(\alpha + \beta)$ lath grains) is obtained after $A_2$ forging. Generally, Ti alloys are usually thermomechanical processed in $\beta$ phase field or in $(\alpha + \beta)$ phase field using isothermo-forging. Widmanstatten microstructure is obtained after thermomechanical processing in the $\beta$ phase field, and bimodal microstructure is obtained after thermomechanical processing in the $(\alpha + \beta)$ phase field. But the microstructure (as presented in Fig. 5(d)) of the specimen after $A_3$ forging is quite different from common Widmanstatten microstructure or bimodal microstructure. The microstructure consists of near-equiaxed $\alpha$ grains totally. The forming of the microstructure results from the precipitation and growth of $\alpha$ phase during forging. $A_3$ forging was started at 1333 K (in the $(\alpha + \beta)$ phase field). When the temperature of the specimens fell below the $\alpha + \beta \rightarrow \alpha$ transus temperature during forging, $\beta$ phase transformed to $\alpha$ phase. When the $A_3$ forging started, primary $\alpha$ phase in the microstructure refined by the deformation, and the growth of new precipitated $\alpha$ grains was restricted by primary $\alpha$ phase during forging. So fine $\alpha$ grains were formed and residual $\beta$ phase distributed along the boundary of $\alpha$ grains. The size of $\alpha$ grains after $A_3$ forging is about 10–20 um.

### 3.4 Mechanical properties

The results of hardness and mechanical properties of the composite during tensile tests at 873 K are presented in Table 2. The typical SEM micrographs of the fracture surfaces of the composite are showed in Fig. 6. As Fig. 6(a) presented, a brittle cleavage fracture mode was observed in

<table>
<thead>
<tr>
<th>No</th>
<th>Hardness (HRC)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>In Cast</td>
<td>39</td>
<td>592.4</td>
<td>&lt;3</td>
</tr>
<tr>
<td>$A_1$</td>
<td>47</td>
<td>750.3</td>
<td>8.3</td>
</tr>
<tr>
<td>$A_2$</td>
<td>43</td>
<td>693.6</td>
<td>14.7</td>
</tr>
<tr>
<td>$A_3$</td>
<td>46</td>
<td>722.4</td>
<td>15.6</td>
</tr>
</tbody>
</table>

Table 2 Mechanical properties of the composite after forged in various temperature ranges.

Fig. 6 SEM micrographs of the fracture surface of the composite are presented in (a) in cast, (b) $A_1$ forging, (c) $A_2$ forging and (d) $A_3$ forging.
specimen in cast. But the fracture surface of the specimen after forging (Figs. 6(b), (c) and (d)) consists of the fine dimples. It shows a typical character of ductile fracture. As a result, the composites exhibit a significant improvement in ductility. From Table 2 and Fig. 6, it can be seen that effect of forging on improvement of mechanical properties of the composite is very obvious.

From Table 2, it can be seen that tensile strength of the specimens increase with the increasing in hardness. The specimen after $A_1$ forging offers highest ultimate tensile strength (UTS) and lower elongation, but the specimen after $A_2$ forging offers the higher elongation and lower strength. This result is consistent with earlier reports, i.e. the Widmanstatten microstructure is more creep resistant than the bimodal microstructure. Thiehsen et al. have reported that ductility increased with the amount of equiaxed $\alpha$-phase in the microstructure.

It is notable that the specimen after $A_3$ forging offers higher UTS and highest elongation. The deformation performed until the material was in the $\alpha$ phase field during such treatment, which resulted in work hardening caused by the generation and pilling up of dislocations during deformation. On the other hand, fine $\alpha$ grains also contribute to higher elongation and strength.

4. Conclusions

From this work, some conclusions can be drawn. Compared to the monolithic Ti-1100 alloy the $\beta$ transus temperature of in situ 5 vol% (TiB+TiC)/Ti-1100 composite increase around 100 K, which results from the solute carbon in matrix. Different microstructures are obtained after the composites are forged in the different temperature ranges, i.e. Widmanstatten microstructure is obtained after forging in the $\beta$ phase field, and bimodal microstructure is obtained after forging in the ($\alpha + \beta$) phase field. Microstructure containing fine $\alpha$ grains totally is obtained after forging started in the ($\alpha + \beta$) phase field and finished in the $\alpha$ phase field. During tensile tests at 873 K, the specimen of the composite with Widmanstatten microstructure offers highest UTS and lower elongation, and the specimen with bimodal microstructure offers higher elongation. But the specimen with fine $\alpha$ grains microstructure offers higher UTS and highest elongation.

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