Microstructure and dry sliding wear behavior of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ titanium matrix composite at elevated temperatures

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Abstract: The microstructure and dry sliding wear performance of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite at 25 °C, 500 °C and 600 °C were systematically investigated. Results show that the solidification matrix microstructure is the typical Widmanstätten structure. The eutectic TiC particles are uniformly distributed in the matrix in the form of feathery, long rod and strip-like shapes. Meanwhile, the interface between TiC and titanium matrix is clear and without any reaction. The wear rate of $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ at 600 °C and 500 °C is reduced by 95.8% and 79.9%, respectively, compared with that of the value of $51.8 \times 10^{-6} \text{ mm}^3 \cdot \text{mm}^{-1}$ at 25 °C. The friction coefficient of the steady-state period at 25 °C, 500 °C, 600 °C is 0.48, 0.8, 1.2, respectively, and the variation extent of the friction coefficient at elevated temperatures is greater than at 25 °C. The wear mechanism is changed from the mixture of adhesive wear and abrasive wear to mild oxidation wear with rising temperature. It can also be concluded that the composite possesses excellent high temperature wear resistance. The high-wear resistance of the composite is attributed to the Fe_2O_3 and TiO_2 . The presence of Fe_2O_3 and TiO_2 reduces the wear rate, increases the friction coefficient, and also improves the range-ability of the friction coefficient.

Key words: titanium matrix composite; as-cast; microstructure; wear behavior; wear mechanism

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Titanium alloys are usually used in aviation, space, automobile, and biochemical industries due to their superior performance, such as high strength, light weight, and excellent corrosion resistance^[1-3]. At present, the working conditions of aerospace engine components are becoming more and more stringent, which put forward higher requirements for the mechanical properties, high temperature oxidation resistance and wear resistance of titanium alloys. When used as transmission gear, bearing, and brake, where friction and wear resistance are mainly considered, good wear resistance at room temperature and high temperature is crucial. However, traditional titanium alloys have an extremely poor tribological property which is caused by low surface hardness, poor work hardening ability, low anti-plastic deformation ability, and low surface oxides protection^[4-6]. Therefore, it is necessary to systematically investigate the room temperature and high temperature wear properties of titanium alloys in order to find the right way to improve the wear resistance.

The common way to improve the wear property of titanium alloys is to form in-situ synthesis ceramic particles reinforced titanium matrix composites. Traditional choices of reinforcements are Al_2O_3 , SiC , Si_3N_4 , TiB_2 , TiB , TiN and TiC , which can greatly improve the fatigue strength, high temperature stability, and wear and oxidation resistance^[4, 7-9]. Choi et al.^[10] studied the friction and wear behavior of $(\text{TiB}+\text{TiC})$ reinforced pure Ti matrix composites. The B_4C particles with 150 μm or 1,500 μm diameter were added

during preparation. The results show that the reinforcements synthesized with 150 μm B_4C have poorer wear resistance than those using 1,500 μm B_4C . Kim et al.^[11, 12] found that the wear rate of the composites decreased with the increase of the volume fraction of the reinforcement. Yang et al.^[13] investigated the dry sliding wear behavior of 10vol.% (TiB+TiC)/Ti-6Al-4V matrix composites at various test conditions. The results show that low wear loss was achieved at lower sliding speed at higher temperature or higher sliding speed at lower temperature.

Previous literatures mainly reported the influence of reinforcement content, size and its distribution on the room temperature wear properties, and fewer studies focused on the high temperature titanium alloy matrix and test temperatures, especially test temperatures higher than 500 °C. In this study, as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ titanium matrix composite is selected as the research material. Ti-1100 alloy, as a near- α high temperature titanium alloy, can be used at temperatures up to 600 °C^[14-17]. Our previous works found that 0.5wt.% Nb addition could significantly improve the strength and high temperature oxidation resistance of Ti-1100 alloy^[18, 19]. In addition, TiC has prominent compatibilities with the Ti matrix and chemical stability which is caused by their similar density (TiC: 4.91 $\text{g}\cdot\text{cm}^{-3}$, Ti: 4.51 $\text{g}\cdot\text{cm}^{-3}$) and thermal expansion coefficient^[10]. The aim of this work is to explore the effect

of temperature (25 °C, 500 °C, 600 °C) on the dry sliding wear behavior of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ titanium matrix composite. Moreover, the wear mechanism was also clarified.

1 Experimental procedure

1.1 Material preparation

In situ synthesized 8vol.% $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite was obtained by a vacuum induction melting (VIM) furnace. To synthesize this composite, sponge titanium (99.80wt.%), sponge zirconium (99.40wt.%), high purity aluminum (99.99wt.%), Al-50wt.% Nb master alloy, tin (99.90wt.%), silicon (99.50wt.%), molybdenum (99.95wt.%) and carbon powder (99.90wt.%) were chosen as raw materials. The above materials were compressed into blocks to reduce the element evaporation and improve the melting efficiency. The melting process was carried out in a controlled vacuum atmosphere of dry high purity argon maintained at 1,000 Pa. After the raw materials were completely melted, the melt was held at 1,700 °C for 2 min. Then the composite mixture liquid was poured into a graphite mold, which was preheated to 400 °C, to form a plate casting with a wall thickness of 12 mm. The chemical composition of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite was tested by a fluorescent spectrometer, and the result is shown in Table 1.

Table 1: Chemical composition of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite (wt.%)

Compositions	Al	Si	Zr	Mo	Sn	Nb	C	Ti
$\text{TiC}_p/\text{Ti-1100-0.5Nb}$	6.361	0.493	3.811	0.312	2.589	0.487	1.430	Bal.

For microstructure observation and wear testing, the specimens were cut from the ingots using an electro-discharge machine (EDM). The wear specimens were then machined to cylindrical pins with the dimension of $\Phi 6\text{ mm}\times 12\text{ mm}$. Commercially pure GCr15 steel was chosen as counter-face disk ($\Phi 70\text{ mm}\times 10\text{ mm}$) and its nominal chemical composition was 0.95–1.05 C, 0.15–0.35 Si, 0.20–0.40 Mn, 1.30–1.65 Cr and balance of Fe (wt.%). The GCr15 disk was austenitized at 860 °C, oil quenched, and then tempered at 160 °C for 3 h to obtain an average hardness of 58 HRC.

1.2 Dry sliding wear test

The dry sliding wear experiments were carried out on a pin-on-disk high-temperature abrasion tester (MG-2000 type), as shown in Fig. 1. The GCr15 counter-face disk was installed on a rotating lower shaft, the test specimen was fixed in a bracket attached on a stationary upper shaft by screw, and then pressed against the counter-face disk under a vertical weight by the dead load. The wear-test machine is carried with a 2 kW resistance heater which can reach a maximum temperature of 600 °C. Meanwhile, a dynamic sensor is installed on the top of the upper spindle to measure the friction torque. The friction coefficient is calculated by measuring the friction torque and loading vertical load. The coefficient of friction μ can be obtained by the following equation:

$$\mu = F/N = M/0.03N \quad (1)$$

where M is the friction torque (N·m); N is the load (N).

The wear experiments were carried out under air atmosphere at the temperatures of 25 °C, 500 °C and 600 °C, respectively. The normal load was chosen as 100 N, and the sliding velocity was 0.75 $\text{m}\cdot\text{s}^{-1}$ for the sliding distance of 900 m. The surface roughness (Ra) value of the disks and the contact surfaces of the test pins was about 0.38 μm by grinding against an 800-grit SiC sandpaper before each test, and then ultrasonically cleaned in acetone.

Fig. 1: Schematic diagram of pin-on-disk type sliding wear tester

The wear rate was obtained by calculating the wear amount of the test specimen using an electronic analytical balance with an accuracy of 0.1 mg. Before each measurement, the pin specimens needed ultrasonic cleaning in acetone and drying. The data represents the mean value of three wear test results. The weight losses of the composites were then calculated to volume using the density of $4.586 \text{ g}\cdot\text{cm}^{-3}$. Finally, the wear rate was obtained from the volume loss divided by the sliding distance. The wear rate W_r can be obtained by the following equation [2]:

$$W_r = m / (\rho L) \quad (2)$$

where m is the weight loss; ρ is the density; L is the sliding distance.

1.3 Characterization

Phase analysis of the wear debris and the worn surface was recorded by X-ray diffraction (XRD) with a D8 FOCUS type X-ray diffractometer using Cu-K α radiation and carried out at 40 kV and 300 mA. The microstructures and compositions were performed by Nikon Eclipse MA100 type optical microscope (OM), Quanta 450 FEG type, S-4800 type scanning electron microscope (SEM), FEI Tecnai G² F30 type transmission electron microscope (TEM) and energy dispersive X-ray spectrometer (EDS). Specimens for OM and SEM observation were prepared through a conventional mechanical polishing followed by etching with Kroll's reagent (6vol.% HNO₃, 3vol.% HF, 91vol.% H₂O). To maintain the accuracy of the test, the cross-sectional analysis specimens were mounted in epoxy resin, and subjected to gold spray treatment. Specimens for TEM observations were thinned to 50 μm by grinding with SiC sandpaper and were punched to 3 mm discs. The final stage of thinning was performed by argon ion milling with an incident angle of 10° until perforation occurred.

2 Results and discussion

2.1 Microstructure

Figure 2 shows the matrix microstructure and the distribution of TiC in the as-cast TiC_p/Ti-1100-0.5Nb composite. The

TiC particles are evenly distributed in the composite, as shown in Fig. 2(a), and two kinds of shapes of TiC can be seen in Fig. 2(b), i.e., feathery shape and long rod or strip shape. The solidification precipitation path of TiC is related to the aluminum content in the titanium alloy. When the Al content varies from 40at.% to 48at.% to form TiAl alloys, the phase transition of TiC/TiAl variation from Al and C content can be summarized as follows: melt Ti+Al $\rightarrow$ TiAl₃, TiAl₃+Ti $\rightarrow$ TiAl+Ti₃Al, TiAl₃+C $\rightarrow$ TiC [20, 21]. When the Al content is relatively low, TiC can be formed by L $\rightarrow$ β -Ti+TiC eutectic reaction [22]. In this work, Al content is 6.361wt.% (Equivalent to 10.216at.%), and the feathery and long rod or strip shape TiC belong to eutectic TiC. The volume fraction of TiC is approximately 8% by the quantitative analysis, which is consistent with the designed value. The matrix exhibits the Widmanstätten structure with alternate lamellae of α and β along a specific orientation within individual colonies.

Figure 3(a) displays the bright field TEM images of TiC at longitudinal cross-section. It can be clearly observed that the interface between TiC and Ti matrix is flat and clean without any interfacial reaction. Figure 3(b) reveals the selected area electron diffraction (SAED) pattern of TiC marked in Fig. 3(a). The TiC possesses the cubic structure originating from the [110] zone axis with the lattice constant $a=0.43274 \text{ nm}$.

2.2 Wear rates

Figure 4 displays the wear rates of the as-cast TiC_p/Ti-1100-0.5Nb composite under different test temperatures. It can be concluded that the wear rates of the as-cast TiC_p/Ti-1100-0.5Nb composite significantly decreased with increasing testing temperature. The wear rate at 600 °C was reduced by 95.8% and 79% compared with that at 25 °C and 500 °C, respectively. Meanwhile, compared with 25 °C, the wear rate was reduced by 79.9% at 500 °C. The wear rate at 600 °C was an order of magnitude lower than that at 25 °C. The above results show that the wear behavior changes with the testing conditions. The composite possesses poor room temperature tribological resistance compared with high temperature conditions, which shows good agreement with the reported results of titanium alloys [23-26].

Fig. 2: Microstructure of as-cast TiC_p/Ti-1100-0.5Nb composite: (a) OM; (b) SEM

Fig. 3: TEM images of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite: (a) bright field image; (b) SAED pattern taken from the Region A in (a)

Fig. 4: Wear rates of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under different test temperatures

Fig. 5: Friction coefficient of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under different test temperatures

2.3 Friction coefficient

The friction coefficient of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under different test temperatures is shown in Fig. 5. The process of dry sliding friction consists of two parts: one is the break-in time and the other is the steady-state time. The break-in period length increases with the increase of testing temperature, and the time increases from 100 s at 25 °C to 400 s at 600 °C. The average friction coefficient of the steady-state period at 25 °C, 500 °C, 600 °C is 0.48, 0.8, 1.2, respectively. The average value and variation extent of the friction coefficient also increase with the rising testing temperature. The friction coefficient of the composite at 600 °C is improved 150% compared with that of at 25 °C.

2.4 Morphologies of worn surfaces

Figure 6 displays the worn surfaces morphology of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under different test temperatures. Plastic deformation zone and tearing traces were easily recognized on the worn surfaces at 25 °C [Fig. 6(a)]. It is clearly observed that the wear failure was caused by shearing and cracking of the adhesive connections between rough tips on the contacting surface due to the high local contact pressure during sliding. Moreover, grooves along the sliding direction

arose from the worn surface, which were generated due to the continuous crushing and plowing by the wear debris or hard counter-face disk. Clearly, the features mentioned above belong to typical adhesive and abrasive wear. When the test temperature reaches 500 °C and 600 °C, oxides could be produced according to the EDS (Table 2) analysis. In that case, the worn surface lost its metallic luster and was substituted by a smooth oxide layer. During sliding, the oxide layers would partly fragment [Figs. 6(b) and (c)] because of their brittleness. Hence, at 500 °C and 600 °C, the $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite exhibited the typical characteristic of mild oxidative wear. At 25 °C, the test specimen directly contacts with the counter-face, due to its poor resistance to plastic deformation and work hardening capacity^[27, 28], there would be furrows on the wear surface under the frictional shear force. With further increase of test temperature, more tribo-oxides were produced on the wear surface under the influence of temperature and friction heat, and then an oxide layer with lots of tribo-oxides was formed. The existence of an oxide layer hinders direct contact between the friction pairs and reduces adhesive wear. Meanwhile, the oxide in the worn surface plays a role of solid lubrication. Therefore, the composite possesses superior high temperature wear resistance compared to at 25 °C.

Fig. 6: Morphology of worn surfaces of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under different test temperatures: (a) 25 °C; (b) 500 °C; (c) 600 °C

Table 2: EDS analysis of worn surfaces marked in Fig. 6 (wt.%)

	C	O	Al	Si	Zr	Nb	Sn	Mo	Fe	Ti
A	5.86	3.43	6.94	0.87	3.88	0.91	3.12	0.89	1.70	Bal.
B	4.30	18.48	6.40	0.89	2.80	0.80	2.66	0.58	13.41	Bal.
C	4.92	22.45	6.56	0.80	2.85	0.90	3.05	0.88	13.22	Bal.

2.5 XRD analysis of worn surfaces

XRD spectra of the worn surfaces under different test temperatures are illustrated in Fig. 7. At 25 °C, only Ti appeared on the worn surface, and the tribo-oxides were not detected due to the low oxygen content (Table 2) and the sensitivity of XRD. At 500 °C and 600 °C, the XRD peaks of TiO_2 and Fe_2O_3 appeared, which meant that the interdiffusion existed among Fe, O, and Ti atoms and some TiO_2 and Fe_2O_3 particles were produced on the worn surfaces at 500 °C and 600 °C. Obviously, more tribo-oxides appeared at 600 °C than at 500 °C, as shown in Fig. 6. It can be observed that the wear rates changed to lower values once oxides presented and significantly decreased with the increase of oxides (Figs. 4 and 5). However, the friction coefficient shows an opposite trend. The amount of tribo-oxides seemed to determine the wear resistance of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite, especially the amount of Fe_2O_3 . The fine oxide particles will form a continuous tribo-oxide layer on the wear surface due to its fast sintering rate and excellent bearing capacity, so the tribo-oxide layers containing Fe_2O_3 and TiO_2 will significantly decrease the wear rate of the composite. However, the existence of Fe_2O_3 and TiO_2 increases the chemical interaction

between the worn surface and the counter-face as well as increasing the friction between the wear surface and the counter-face, thus increasing the friction coefficient. The similar role played by oxides during the friction process was also confirmed by Refs. [29] and [30].

Fig. 7: XRD patterns of the worn surfaces under the different test temperatures

2.6 Wear debris

Figure 8 shows the morphologies of the wear debris of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite. At 25 °C, most of the debris existed in the form of flakes, and fewer existed in helix-shape [Fig. 8(a)]. The room temperature wear mechanism of adhesive wear and abrasive wear has been recognized in Fig. 6(a). This result is in good agreement with that reported by Gao [31], which showed that the flaky debris and helix-shape debris were the typical characteristics of adhesive and abrasive wear. At the test temperature of 500 °C, the debris size substantially decreased, and the thickness

increased. Meanwhile, it also generated the spherical debris, as shown in Fig. 8(b). The spherical debris is the typical characteristic of contact fatigue wear, which indicates that the wear mechanism at 500 °C is given priority to oxidation wear, and is accompanied by contact fatigue wear. The EDS analysis results of the wear debris are showed in Fig. 9 and Table 3. It can be concluded that both the helix-shape and spherical debris confirmed that the Fe element came from the GCr15. It is also indicated that fatigue wear and oxidation wear easily occur in GCr15 steel at high temperature. At 600 °C, little debris is collected due to the lower wear rate.

Fig. 8: Morphologies of wear debris of as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite at 25 °C (a) and 500 °C (b)

Fig. 9: EDS analysis of wear debris presented in Fig. 8: (a) Spot A; (b) Spot B; (c) Spot C

Table 3: Compositions of Spots A, B and C marked in Fig. 8

Spot	Element (wt.%)								
	C	O	Al	Si	Zr	Mo	Sn	Ti	Fe
A	9.21	5.75	8.15	1.32	4.42	1.63	3.98	64.28	1.26
B	9.48	4.36	1.06	0.55	0.22	0.17	0.79	0.56	82.81
C	6.23	32.11	1.55	0.58	0.48	0.54	1.48	0.72	56.31

The XRD spectra of the debris under the test temperature of 25 °C and 500 °C are shown in Fig. 10. It can be seen that the phase constitution of the debris formed at 25 °C is quite different from that formed at 500 °C. The debris formed at 25 °C is mainly titanium particles and contains a small amount of Fe, indicating that the wear process at 25 °C is mainly titanium wear accompanied by a small amount of iron wear. At 500 °C, the number of XRD diffraction peaks and intensity of Fe and Fe₂O₃ peaks are increased, while the peaks of Ti are significantly weakened. These results show that the wear process at high temperature is mainly the wear of counter-face, which indirectly indicates that the composite possesses good wear resistance at elevated temperatures.

2.7 Cross-sectional analysis of worn surfaces

To further investigate the interfacial characteristics between

the worn surfaces and the matrix, the cross-sectional morphologies and the elemental distribution of the as-cast TiC_p/Ti-1100-0.5Nb composite under different test temperatures were detected by line scanning, as shown in Fig. 11. It can be observed that a thin and discontinuous tribo-oxide layer with a thickness of about 10.8 μm appeared in Fig. 11(a). The tribo-layer is easily peeled off from the matrix to form debris, thus the composite has poor wear resistance at 25 °C because of its low protection. Meanwhile, the adjacent plastic deformation zone indicates that the tribo-layer at 25 °C has poor bearing capacity. As the test temperature reached 500 °C and 600 °C, the tribo-oxide layer thickness increased to 12.9 μm and 33.8 μm, respectively, and both of the tribo-oxide layers became very compact and continuously distributed [(Figs. 11(c) and (e)]. It is worthwhile to note that almost no plastic deformation layer appears in this case, which demonstrates that the tribo-layer at high temperature possesses great bearing capacity. Therefore, the composite has good wear resistance at a higher temperature.

It can also be observed that the distribution of elements changes with the increase of test temperatures from the worn surface to the matrix, especially for Fe, O and Ti elements. In the tribo-layer, the peak intensity of Ti, Fe and O increases compared with the substrate under various testing temperatures, which indicates that the tribo-layer is not formed by the accumulation of multiple debris, but by the diffusion of elements during the wear process. The content of Fe and O element in the friction layer increases with the increase of test temperatures, which also reveals that the tribo-oxide layer mainly contains titanium oxide and iron oxide.

Fig. 10: XRD patterns of debris under different test temperatures

Fig. 11: Morphologies and elemental distribution curves of worn subsurfaces of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite under various temperatures: (a), (b) 25 °C; (c), (d) 500 °C; (e), (f) 600 °C

3 Conclusions

(1) The solidification matrix microstructure in the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite was found to be the typical Widmanstätten structure. The eutectic TiC is uniformly distributed in the Ti matrix in the form of feathery shape and long rod or strip shape. The interface between TiC and Ti matrix is flat and clean and without any interfacial reaction.

(2) The wear rate of $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ at 600 °C and 500 °C is reduced by 95.8% and 79.9%, respectively, compared with that of the value of $51.8 \times 10^{-6} \text{ mm}^3 \cdot \text{mm}^{-1}$ at 25 °C. The friction coefficients of the composite increase with the increase of temperature, the friction coefficients of the steady-state period at 25 °C, 500 °C, 600 °C are 0.48, 0.8, 1.2, respectively, and the variation extent of the friction coefficient at elevated temperature is greater than that at 25 °C.

(3) The wear mechanism of the as-cast $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite changes from adhesive and abrasive wears at 25 °C to mild oxidative wear at 500 °C and 600 °C.

(4) The high-wear resistance of the $\text{TiC}_p/\text{Ti-1100-0.5Nb}$ composite may be attributed to the appearance of Fe_2O_3 and TiO_2 . At 25 °C, thin and discontinuous tribo-oxide layer does not show the effect of reducing wear. More and continuous tribo-oxide layers are produced at elevated temperatures, which would substantially improve the wear properties.

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