

# INTERFACE REACTION OF $\text{TiC}_p$ -REINFORCED Ti-MATRIX COMPOSITE<sup>①</sup>

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**ABSTRACT** Interface reaction of  $\text{TiC}_p$ -reinforced Ti-matrix composites has been studied. Degradation reaction of TiC and C atoms diffusing into matrix results in nonstoichiometric interface reaction layer forming around  $\text{TiC}_p$ . Electron diffraction patterns prove that the interface layers are clean without other reaction products. The layers possess reciprocal character, and the interface width varies with process conditions.

**Key words** Ti-matrix composites TiC interface reaction

## 1 INTRODUCTION

Titanium is a highly active element which reacts with almost all reinforcers, resulting in properties of Ti matrix composite in contrast with the composite rule.

For a long time, interface reaction has been the main challenge in developing Ti-matrix composites. The Ti-matrix composites includes two categories: fiber-reinforced and particle-reinforced. Comparatively speaking, the development of the particle-reinforced Ti-matrix composites makes slower progress, especially the investigation of melt-casting particle-reinforced Ti-matrix composites stagnates for a long time, which is mainly due to strong interface reaction. Powder metallurgy was used in particle-reinforced Ti-matrix composites at early stage, but clean powder was difficult to obtain. Alcoa Lab and University of Birmingham co-worked on Ti-6Al-4V+TiC and Ti-24Al-11Nb+TiC of particle-reinforced Ti-matrix composites by powder metallurgy as well as the interface reaction in the composites<sup>[1-3]</sup>, and found nonstoichiometric annuli around  $\text{TiC}_p$ , which were considered as  $\text{Ti}_3\text{Al}(\text{NbAl})\text{C}^{[2]}$  or  $\text{Ti}_2\text{C}^{[3]}$ . In 1990s, there have been a few reports about Ti-matrix composites by melt-casting<sup>[4]</sup>. After XD<sup>TM</sup> process was developed by Martin Marietta, the interface re-

action of reinforcements with Ti-matrix can be inhibited and clean interface can be obtained<sup>[5]</sup>, which promotes the development of melt-casting composites. Recently, a process of in situ formation of three dimensional  $\text{TiC}_p$  was reported by Lin *et al* in Auburn University and from then on the melt-casting particle-reinforced Ti-matrix composites have been recognized<sup>[6]</sup>. Following are investigations on interface reaction of the TiC particle-reinforced Ti-matrix composites by PTMP (pre-treatment melting process).

## 2 EXPERIMENTAL PROCEDURE

The composite of Ti-15S+10%  $\text{TiC}_p$  was prepared by PTMP. A  $\phi 90\text{ mm} \times 110\text{ mm}$  (about 3.5 kg) ingot produced by electrode consumption vacuum arc furnace was conventionally forged into  $\phi 12\text{ mm}$ . Then metallographic samples were cut from the forged bar, and the microstructures and interfaces observation and analysis for the as-forged and as-annealed at  $800 \sim 1050^\circ\text{C}$  for 1 h were carried out by JSM-35C SEM and JEOL JEM-200 CX TEM, respectively.

The compositions of the particle, the interface and the matrix were analyzed by PHI600 Multifunction Scanning Auger Electron Microprobe. The samples were sputtered with Ar ion for 5 min. TEM samples were made as follows: cut

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the bar to 0.5 mm by spark erosion, polished down to about 0.05 mm by using sand paper and then thinned by twin-jet process.

### 3 RESULTS AND DISCUSSION

Fig. 1 show the SEM and TEM photos of the composite, which indicate that TiC<sub>p</sub> with original irregular edge becomes smoother and has the tendency to spheroidize. There is obvious contrast between TiC<sub>p</sub> center and edge for both as-forged and as-annealed. The particles were surrounded by bright annuli, which are the interface layers of TiC<sub>p</sub> and Ti-matrix.

Table 1 shows the Auger analysis results of the chemical composition of interface reaction layer. The result of the center of TiC<sub>p</sub> (point 1) corresponds to stoichiometric composition of TiC. In the annuli near the particle (point 2) C concentration reduces slightly, and the result accords with the stoichiometric composition in principle. At the center of annuli (point 3) C concentration is only half of that in the TiC center, but much higher than that in the matrix. At the connection of the annuli and the matrix (point 4) C concentration reduces continuously. The result of adjacent matrix (point 5) shows that the C concentration is higher than that in the matrix.

These data indicate that C concentration decreases progressively from TiC<sub>p</sub> center to adjacent matrix. The degradation reaction occurs on TiC,

and C atoms diffuses into the matrix. Nb and Mo are found in TiC<sub>p</sub>, which means Nb and Mo diffuse into the matrix retrogressively. The high O content is related to the surface contamination and insufficient ion sputtering time. The degradation reaction of TiC<sub>p</sub> which forms non-stoichiometric interface layers around the particles is identical with those of Refs. [3] and [4]. Electron diffraction patterns of TiC and the interface are shown in Fig. 2; their diffraction patterns are the same.  $\alpha$ -Ti diffraction pattern is shown in Fig. 2(b). No new reaction products are found in the interface. In view of this only degradation reaction of TiC occurs in interface reaction between TiC<sub>p</sub> and Ti-matrix, resulting in nonstoichiometric interface layers around TiC<sub>p</sub>.

**Table 1 Chemical composition of interface reaction layer (mole fraction, %)**

Position	Ti	Mo	Nb	C	O
Point 1	44.0	1.2	2.25	0.6	2.0
Point 2	46.1	1.4	2.0	47.6	3.0
Point 3	64.6	1.6	3.52	5.2	5.1
Point 4	77.2	1.9	4.11	0.6	6.3
Point 5	77.3	2.0	4.3	8.3	7.6

It is found that certain reaction of reinforcements and matrix in composites are necessary, which ensures favourable connection between particles and matrix. However, the extent of reaction must meet the requirement of composite



**Fig. 1 SEM(a) and TEM(b) photos of Ti-15S+ TiC<sub>p</sub>**

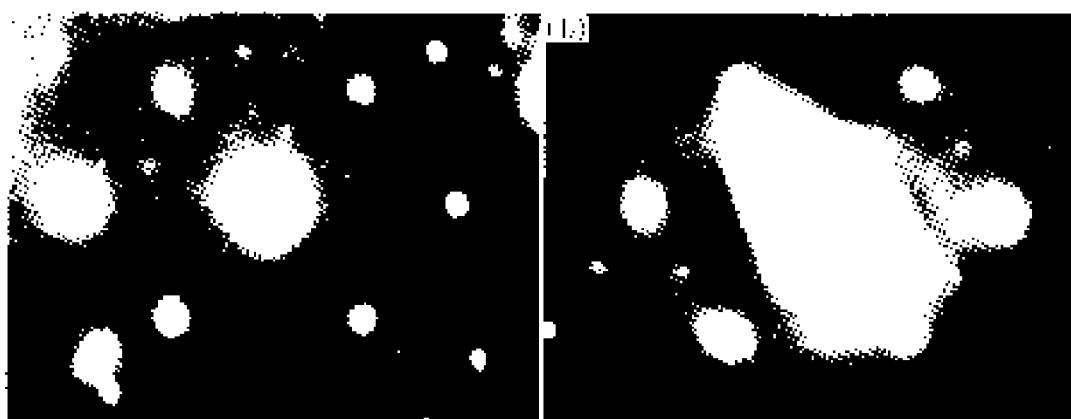


Fig. 2 Electron diffraction patterns of  $\text{TiC}_p$  (a) and interface (b)

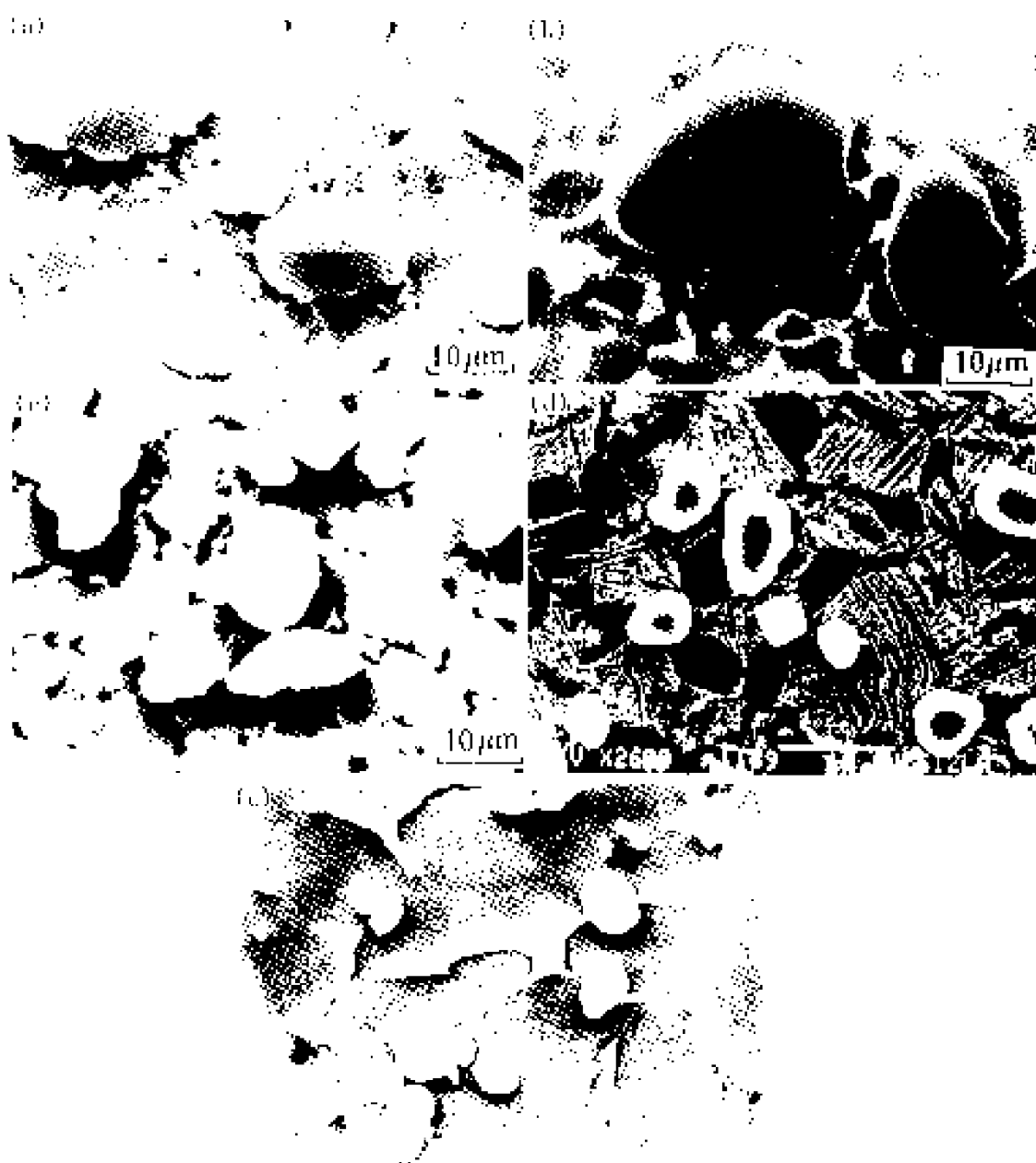


Fig. 3 Interface of  $\text{TiC}_p$  reinforced Ti-matrix composites at different states  
 (a) —Forging state; (b) — $800\text{ }^{\circ}\text{C}$ , 1 h, AC; (c) — $1000\text{ }^{\circ}\text{C}$ , 1 h, AC;  
 (d) — $1050\text{ }^{\circ}\text{C}$ , 1 h, AC; (e) — $1050\text{ }^{\circ}\text{C}$ , 1 h, FC

properties. Fig. 3 shows the extent of interface reaction of Ti-15S+ 10% TiC<sub>p</sub> composites. It is clear that the width of interface reaction layer varies with the state of composites. The width of interface layer for the as-forged is about 0.5~1 μm (Fig. 3(a)). There is not any variation in width of the interface layer for the as-annealed at 800 °C for 1 h (Fig. 3(b)). Interface reaction becomes quicker at above 950 °C and the width grows clearly. The width is 2~4 μm at 1000 °C, 1 h AC (Fig. 3(c)) and 2.5~5 μm at 1050 °C, 1 h AC (Fig. 3(d)). TiC particles in the range of 1~2 μm have almost finished degradation reaction at above 1000 °C, and no particle center with darker contrast is observed. High temperature treatment produces wider interface, which deteriorates the properties of the composites. But high temperature double treatment can improve the microstructure and toughness of the matrix, thus improving the collocation of the reinforcements and the matrix, and making a contribution to integral mechanical properties of the composites. Slow cooling after high temperature treatment can give consideration to the two results. The test shows that slow cooling at high temperature can reduce the interface width evidently. The interface width of the samples treated at 1050 °C, 1 h FC is only about 0.2~0.5 μm, as shown in Fig. 3(e). Compared with the air cooled samples treated at the same temperature, the width of the interface layer has reduced an order of magnitude; it is probably the result of reprecipitation on TiC, which shows that the interface reaction of TiC<sub>p</sub>-reinforced Ti matrix composites has the character of reciprocity. High temperature heating can quicken interface reaction and the interface becomes wider. Slow cooling can make reprecipitation of C atoms, and the

interface becomes narrower. This character of TiC is useful to study composites and it is effective to control the width of interface reaction using the reprecipitation effect.

## 4 CONCLUSIONS

(1) There is a favourable connection between TiC<sub>p</sub> and the matrix in Ti-15S+ 10% TiC<sub>p</sub> composites prepared by PTMP.

(2) The reaction for TiC<sub>p</sub> and the matrix is a degradation reaction of TiC<sub>p</sub>, nonstoichiometric interface layer is formed around TiC<sub>p</sub>. The interface is clean and no other products are found.

(3) The interface reaction between TiC<sub>p</sub> and the matrix is reciprocal. High temperature heating quickens interface reaction and the interface becomes wider, slow cooling can make C atoms reprecipitate on TiC and the interface becomes narrower.

## REFERENCES

- 1 Konitzer D G, Loretto M H. In: Lacombe P ed, Proceedings of the Sixth International Conference on Titanium. France: Physique, 1988: 1057–1062.
- 2 Konitzer D G. Materials Science and Engineering, 1989, A107: 217–223.
- 3 Lortto M H *et al.* Metall Trans A, 1990, 21A(6): 1579–1587.
- 4 Shih D S, Amato R A. Scripta Metall Mater, 1990, 24(11): 2053–2058.
- 5 Bryant J D *et al.* Scripta Metall Mater, 1990, 24(11): 2209–2214.
- 6 Lin Y *et al.* Metall Trans A, 1991, 22A(4): 859–865.
- 7 Zeng Q P *et al.* Rare Metal Materials and Engineering, (in Chinese), 1991, 20(6): 33–38.

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