Article ID: 1003 - 6326(2003)02 - 0315 - 05

Microstructure evolution during preparation of in-situ TiB reinforced titanium matrix composites①

SHANG Jun-ling(尚俊玲), LI Bang-sheng(李邦盛), GUO Jing-jie(郭景杰), FU Heng-zhi(傅恒志)
(School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China)

Abstract: By means of DTA, SHS quenching, XRD and SEM, the microstructure evolution during fabricating of TiB reinforced in-situ titanium matrix composite was studied. DTA result shows that there are two exothermic and one endothermic reaction during heating of Ti-B-Al system. The exothermic reactions correspond to the solid-solid diffusion reaction between Ti and Al, and the reaction of TiB formation respectively, and the endothermic reaction corresponds to melting of Al. The sample of SHS quenching presents four regions: unreacted region, heat-affected region, reacting region and product region. The microstructures of these regions reflect the process of microstructure evolution during preparation of in-situ TiB reinforced TMC. Ti, B, Al powders are in simple mechanical contact in the unreacted region; the sharp angles of original metal particles have become round and B diffuses into melted Al in the heat-affected region; TiB whiskers begin to appear and grow in the interior of reacting region. In product region, the TiB whiskers are uniform and fine. The result of XRD indicates that the reinforcements formed in product region are TiB whiskers. And the TiB is a hexagonal prism with a pyramidal head.

Key words: in-situ; titanium matrix composite; microstructure evolution
CLC number: TB 331 **Document code:** A

1 INTRODUCTION

Titanium matrix composites are promising materials which can be used extensively in aerospace and automotive industries because of their excellent mechanical properties at room temperature and elevated temperature[1-4], such as low density, high strength, and corrosion-resistance. Among the in-situ fabrication methods of titanium matrix composites[5-11], self-propagating high-temperature synthesis (SHS) method has unique advantage, that is, the reinforcements are fine and dispersed. In SHS, there is an exothermic reaction accompanied with the release of heat. The heat liberated is adequate to sustain the reaction by the rapid propagation of a combustion front without the further addition of energy[12]. A requirement for the propagation of a reaction in a self-sustaining mode is high enthalpy of reaction. The reactions with relatively low enthalpy changes need to be heated prior to the initiation of the reaction. The advantage of this process is the self-generation of energy and the production of high purity products, because all volatile impurities evaporate at high temperature obtained by exothermic reaction. It is economical and short-cut technology to fabricate high-quality particle reinforced in-situ titanium matrix composites, and it is possible to obtain complex components with casting technology.

In Ref. [13], the titanium matrix composite reinforced by in-situ TiB whiskers was fabricated successfully in Ti-B-Al system. The result showed that addition of Al promoted the formation of TiB whisker reinforcements and the temperature of TiB formation was decreased. But there was a lack of understanding of microstructure evolution during preparation of in-situ TiB reinforcing titanium matrix composite. The aim of this work is to study the microstructure of each intermediate reaction stage by SHS quenching combined with SEM, so as to understand the microstructure evolution during preparation of in-situ TiB reinforced titanium matrix composites. Differential thermal analysis (DTA) experiment is used to find out the sequence of multiple reactions during TiB formation.

2 EXPERIMENTAL

In this work, the Ti-B-Al system was the mixture of Ti, B, and Al powders. Table 1 shows the particle size, impurity and purity of Ti, B and Al elements. Titanium powders, amorphous boron powders and aluminum powders were blended in a mixer; the mass composition of the Ti-B-Al system is Ti 88.3%, B 7.5%, Al 4.2%.

DTA experiment was conducted in argon gas at linear heating rate of 10 ℃/min up to 1 500 ℃. The mixture was cold pressed into cylinder sam-

① **Foundation item:** Project (G2000067202 - 2) supported by State Key Development Project of Fundamental Research
Received date: 2002 - 04 - 22; **Accepted date:** 2002 - 07 - 30
Correspondence: SHANG Jun-ling, doctor, + 86-451-6413931; E-mail: shangjl77@163.com

Table 1 Particle size, impurity and purity of Ti, B and Al

Element	Average size/ μm	Purity/ %	Impurity/ %
Ti	20	> 98	–
B	1	⩾95	–
Al	29	–	Fe ⩽0.3, Cu ⩽0.17, Si ⩽0.35

ples with diameter 20 mm and height 40 mm by 60 t universal material testing machine for SHS quenching, which made use of the exothermic reaction in SHS. In this process, the sample was ignited first on the upper part, and then the combustion front transmitted to the lower part. Meanwhile, the lower part of the sample was quenched, and therein the transmission of the combustion front was stopped, the microstructures corresponding to the intermediate reaction stages were remained. The quenching media could be liquid argon, chill metal etc; here, the pure copper with high heat-conductivity was used. Fig. 1 is the schematic of SHS quenching device. The SHS quenching device was made of three parts: copper pole, heat-insulator and ignitor. The copper pole with a 130 mm diameter and 160 mm height was used as quenching media, which had a hole with diameter of 20 mm and height of 10 mm in the centre of the top. The ring-shaped heat-insulator was used to prevent the sample from being oxidized during reaction. The heat-insulator with 130 mm diameter and 30 mm thickness had a through central hole with diameter of 20 mm. The ignitor was used to initiate the self-propagating reaction. Because of the fast heat loss due to copper pole, the combustion synthesis could not spread all over the whole sample. The combustion front stopped at where the sample was contacted and quenched with copper pole. The area adjacent to combustion front presented four regions: unreacted region, heat-affected region, reacting region and product region which are denoted by *A*, *B*, *C* and *D* respectively as shown in Fig. 2. The sample was cut along its axis and polished. The microstructure of each region was observed under SEM. XRD was conducted to product region of sample quenched. The DTA device is Perkin-Elmer. The XRD device is Philips Analyzer.

3 RESULTS AND DISCUSSION

3.1 DTA experiment

The DTA curve for the Ti-B-Al system is shown in Fig. 3. It is clear that there are multiple reactions in this system. In the following paragraphs the reactions taken place in the Ti-B-Al system will be discussed.

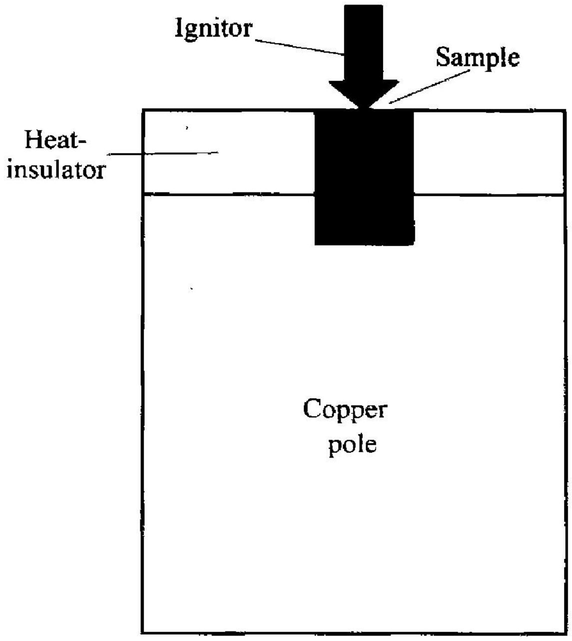

Fig. 1 Schematic of SHS quenching device

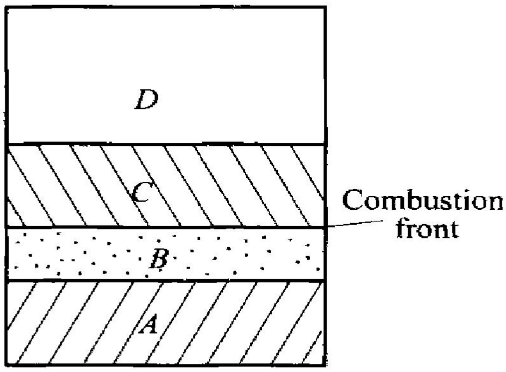

Fig. 2 Four regions schematic of quenched sample

In Fig. 3, there are two exothermic peaks, *a* and *c*, an endothermic peak, *b*. The temperature of endothermic peak is 665 ℃, so *b* corresponds to melting point of Al. Before melting of Al, there is a small exothermic reaction, *a*, which is a solid-solid surface diffusion reaction between Al and Ti. Titanium-aluminum compounds are formed. The equations are:

$$Ti + 3Al \rightarrow TiAl_3 \quad (1)$$

$$Ti + Al \rightarrow TiAl \quad (2)$$

$$3Ti + Al \rightarrow Ti_3Al \quad (3)$$

The most interesting observation in Fig. 3 is the exothermic reaction *c* at 710 ℃, which follows the melting of Al closely. Since the heat released fast and strongly, it can be assumed that it is an exothermic reaction between Ti and B. the equation is given as

$$Ti + B \rightarrow TiB \quad (4)$$

3.2 SEM microstructure

The microstructure of unreacted region *A* is shown in Fig. 4. It can be seen that the Ti particles and Al particles are of irregular shapes with sharp angles and have relatively big particle size ;

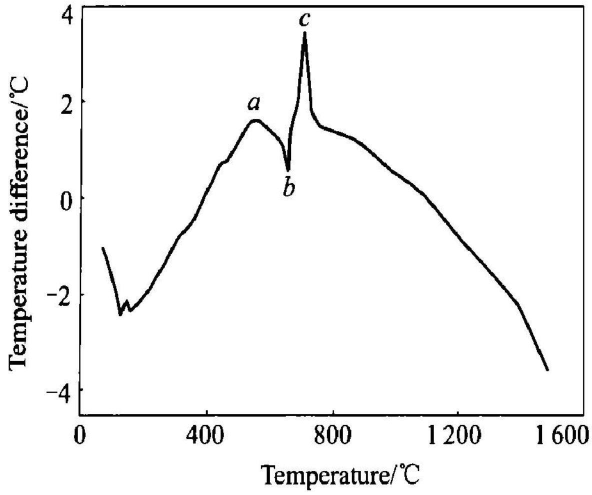

Fig. 3 DTA curve of Ti-B-Al system

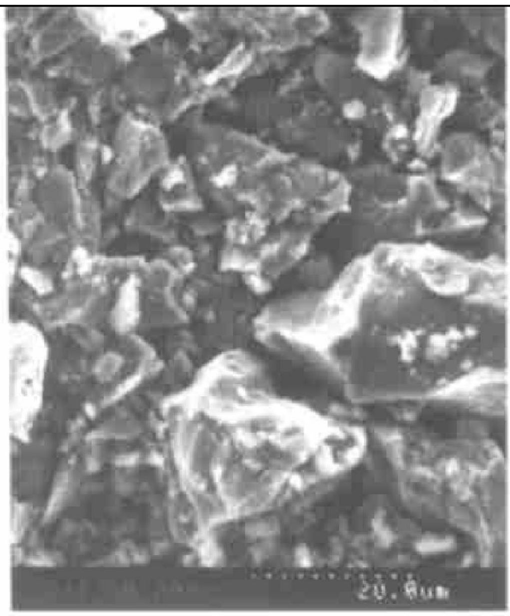

Fig. 4 Microstructure of unreacted region

the particles of B are very fine. These particles are in simple mechanical contact.

The region close to the unreacted region is the heat-affected region. Fig. 5 is the microstructures of heat-affected region *B*. The heat-affected region can be divided into three subregions. The typical morphologies of each subregion are given in Fig. 5 (a), 5(b) and 5(c) respectively. From Fig. 5(a), the microstructure of unreacted region is still remained. The sharp angles of original metal particles have become round. On the basis of DTA curve of Ti-B-Al system in Fig. 3, the Al has melt in the stage of Fig. 5(a). Melted Al is adsorbed on the surface of Ti particles. The sharp angles of Ti particles become round. In Fig. 5(b), the morphology of Ti particles is further changed. According to DTA curve, the reason is that after melting of Al, the Al spreads out completely on Ti particle surface. And before melting of Al, there are titanium-aluminum compounds formed on the contact interface between Ti and Al particles. The reaction helps the sharp angles disappear too. B particles stick to the surface of melted Al, as shown in Fig. 5(b). In Fig. 5(c), B particles have become very little, which results from B diffusing into liquid Al. Melting of Al promotes the diffusion of B into Ti cross liquid Al, and the heat released by formation of titanium-aluminum compounds provides energy to generation of TiB (see Ref. [13]). Ti particles surrounded by Al squeeze together closely.

The next region is the reacting region *C*. Fig. 6 is the microstructure of reacting region. From Fig. 6 (a), there are TiB whiskers growing in the interior of sample. In Fig. 6 (b), there are TiB whiskers on the whole matrix.

Fig. 7 is the microstructure of product region *D*, which shows the morphologies of TiB grown up freely in shrinkage porosity zone of sample. TiB can be identified by XRD of product region, as shown in Fig. 8. It can be seen that there are TiB reinforcements and Ti matrix, and no titanium-aluminum compounds, which illuminates that reaction between Ti and B is complete. Because the sample was fabricated by SHS quenching , the time of reaction is

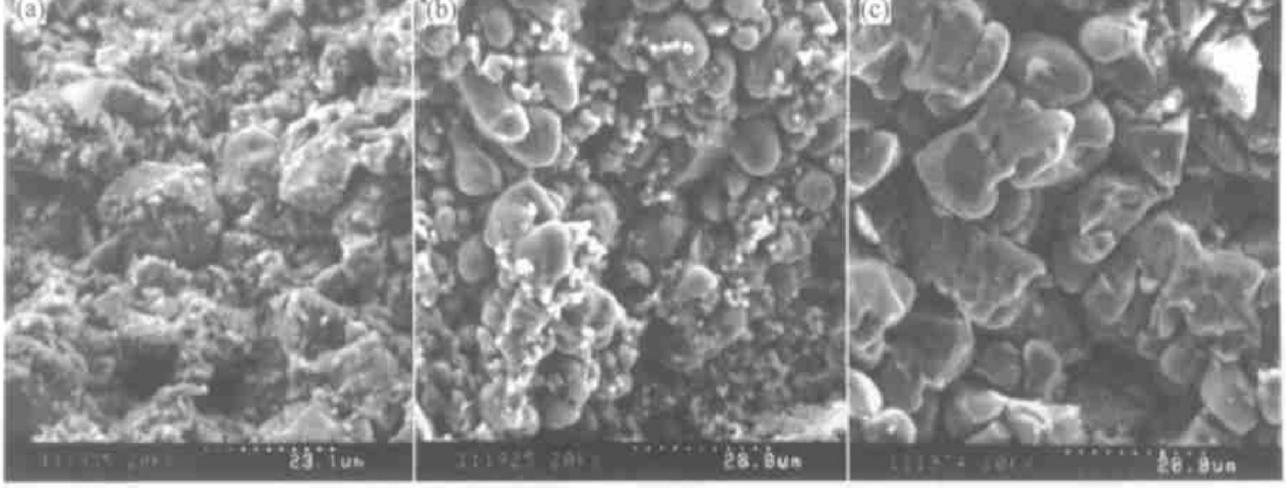

Fig. 5 Microstructures of heat affected region

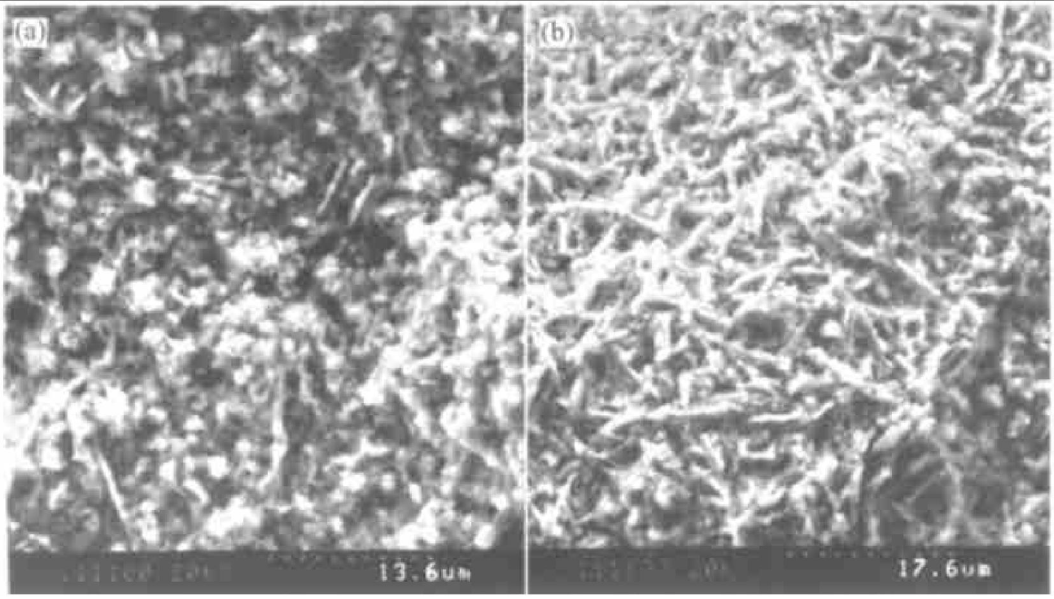

Fig. 6 Microstructures of reacting region

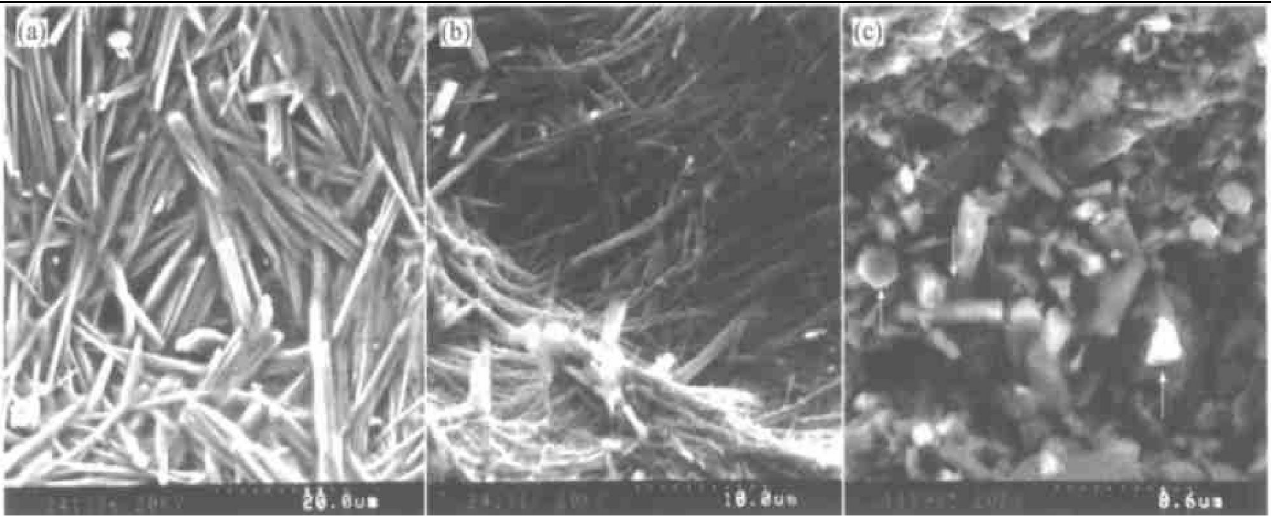

Fig. 7 Microstructures of product region

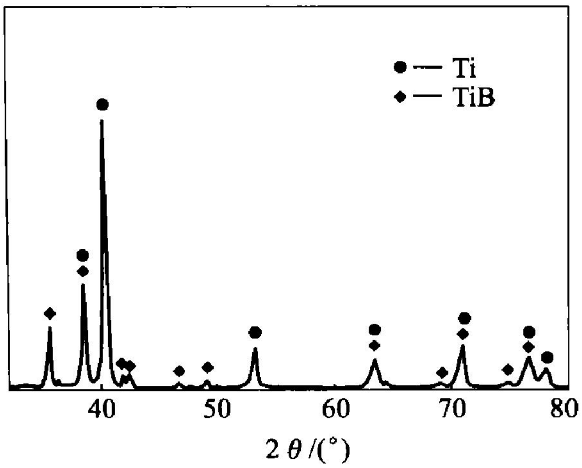

Fig. 8 XRD of product region

short, and the extent of reaction and the growth of whisker are limited unavoidably. But in Fig. 7(a), it can be seen that the whiskers are uniform, fine and staggered. The Fig. 7 (b) is the typical morphology of completely reacted region. In Fig. 7(c), it is obvious that the shape of TiB whisker is a hexagonal prism with a pyramidal head. Three arrows from left to right indicate the shape of the end, profile and head respectively.

4 CONCLUTIONS

1) The results of DTA show that there are two exothermic reactions and one endothermic reaction during heating of the Ti-B-Al system. The exothermic reactions correspond to the solid-solid diffusion reaction between Ti and Al and the reaction of TiB formation respectively; and the endothermic reaction involves melting of Al.

2) The sample of SHS quenching presents four regions: unreacted region, heat affected region, reacting region and product region. The microstructures of these regions show clearly the microstructure evolution during preparation of in-situ TiB reinforcing titanium matrix composite.

3) The Ti, B, Al powders are in simple mechanical contact in unreacted region; The sharp angles of original metal particles have become round and B diffuses into melted Al in the heat-affected region; TiB whiskers begin to appear and grow in the interior of reacting region. In product region, the TiB whiskers are uniform, fine and staggered together. And the TiB is a hexagonal prism with a pyramidal head.

REFERENCES

[1] ZHANG Xiao-nong, LV Wei-jie, ZHANG Di, et al. Fabrication and performance of particular reinforcing TMCs [J]. Aerospace Materials & Technology, 1998 (2): 24-26. (in Chinese)

[2] Ranganath S. A review of particulate-reinforced titanium matrix composites [J]. Journal of Material Science, 1997, 32: 1-6.

[3] LV Wei-jie, ZHANG Xiao-nong, ZHANG Di, et al. The progress in research on particular reinforced titanium matrix composite [J]. Materials Review, 1999, 13(6): 15-18. (in Chinese)

[4] Shang J K , Ritchie R O, Monotonic. Cyclic crack growth in a TiC-particulate-rinforced Ti-6Al-4V metal matrix composite [J]. Scr Matell, 1990, 24: 1691-1694.

[5] Koczak M J, Khatri S C, Alisson J E, et al. In: Suresh S, Mortensen A, Needleman A eds. Fundamentals of Metal Matrix Composites [C]. New York: Butterworth Heinemann, 1993.

[6] Sampatqh S, KHATRI S, Shtessel E, et al. Processing and fabrication of advanced materials for high temperature applications II [A]. Ravi V A, Srinvatsan T S eds. TMS, Warrendale, PA, 1993. 223.

[7] Martin Marietta Corp. US 4915908, 1990.

[8] QU Xuan-Hui. HUANG Bo-Yun. Study of preparation and thermostability of TiAl+ TiB_2 composite [J]. Acta Metallurgica Sinica, 1993, 29(5): B236-240. (in Chinese)

[9] Tsang H T, Chao G G, Ma C Y. Effect of volume fraction of reinforcement on tensile and creep properties of in-situ TiB/Ti MMC [J]. Scripta Materialia, 1997, 37 (9): 1359-1365.

[10] Ranganath S, Roy T, Nishra R S. Microstructure and deformation of TiB+ Ti_2C reinforced titanium matrix composites [J]. Materials Science and Technology, 1996, 12(3): 219-226.

[11] CUI Chun-xiang, WU Ren-jie, WANG Hao-wei. Principle and process on the fabrication of in situ MMCs [J]. Development and Application of Materials, 1995, 10(5): 35-39. (in chinese)

[12] Subrahmanyam J. Vijayakumar M. Review self-propagating high-temperature synthesis [J]. Journal of Materials Science, 1992, 27: 6249.

[13] SHANG Jun-ling, LI Bang-sheng, GUO Jing-jie. Effect of Al on TiB generation in in-situ titanium matrix composite [J]. Rare Metal Materials and Engineering. 2003(4): 258-260. (in Chinese)

(Edited by LONG Huai-zhong)