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Microstructure evolution of SiC_p/Al-Ti foils during hot rolling and reaction annealing

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Abstract: SiC_p/Al-Ti foils were prepared by hot-pressing and hot rolling. The diffusion reaction between Ti and SiC_p/Al was investigated and multi-layers of intermetallics formed during reaction annealing at 1 200 °C. The microstructure and the composition in each intermetallic layer were characterized using scanning electron microscope (SEM) equipped with energy dispersive spectroscope (EDS). The products from the reaction between SiC and titanium aluminides were determined by X-ray diffractometer (XRD) and the thermodynamics of the reaction was investigated using differential thermal analysis (DTA). The results show that different reactive layers form in the sample and SiC particles disappear with the formation of intermetallic layers after annealing. Intermetallic multi-layers are determined to be Ti_3Al, TiAl and $TiAl_3$. SiC particles react with titanium aluminide during reaction annealing to form Ti_3AlC, TiC, Ti_5Si_3 and Ti_3SiC_2.

Key words: titanium aluminide; diffusion reaction; SiC; composite

1 Introduction

Titanium aluminide alloys have attracted lots of attentions in aerospace and automobile applications for their low density, high strength and good oxidation resistance at elevated temperatures [1−2]. The specific strength and specific stiffness of titanium aluminide composites are higher compared with titanium aluminide alloys and they can be used at relatively higher temperatures [3−4]. The effect of in-situ reinforcements on mechanical properties at high temperature is much better than that of ex-situ ones for the clean interface between reinforcement and matrix [5]. The processing of intermetallic composites in the form of sheets or foils is of more interest for certain applications [6−7]. But, TiAl alloys are relatively brittle and difficult to shape into sheet materials via conventional hot and cold deformation processing methods. Recently, sheet processing of TiAl alloys and their composites have been accomplished via two different routes: ingot metallurgy (casting followed by hot isostatic pressing) and powder metallurgy [8−9]. Microstructure and mechanical properties of Ti-45Al-8.5N-(W, B) (molar fraction, %) alloys fabricated by elemental powder metallurgy (EPM) have been studied. The compression strength of EPM high Nb-containing TiAl alloy is higher, but its ductility is much worse than that of as-cast alloy [8]. The high cost of such sheets has limited their applications.

In this work, the sheets of titanium aluminide composites were prepared through reaction annealing of rolled SiC_p/Al-Ti foils. The sheets with reinforcements in situ were first prepared by only one step. The microstructure of the sheets was analyzed and the reaction mechanism was determined.

2 Experimental

The SiC_p/Al composite with volume fraction of reinforcement of 5% was prepared by powder metallurgy. It was prepared by the vacuum hot pressing at 600 °C for 1 h under the pressure of 30 MPa. Then, foils of 0.6 mm were obtained by hot extrusion and hot rolling at 400 °C. Titanium (99.8%, mass fraction) foils with thickness of 0.5 mm were selected to bond them.

The foils of Ti and SiC_p/Al were roughened with a steel-wire brush, cleaned ultrasonically in methanol, and then cut into squares of 30 mm×30 mm. Alternating layers of titanium and SiC_p/Al composites foils were stacked one by one to form a total of eleven layers of

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titanium and aluminum composites stacked together with titanium on both ends. These sandwiches were pressed under a pressure of 20 MPa at 500 °C for 60 min in vacuum to achieve sufficient bonding strength, then, were rolled at 400 °C.

Reaction annealing for the rolled SiC_p/Al-Ti foils was performed at 1 200 °C. The microstructure evolution and reactive phase of SiC_p/Al-Ti foils were characterized by SEM with EDS and XRD. The thermodynamics of the reaction during the annealing was investigated by DTA with a sample consisting of SiC particles, Ti and Al powders blended uniformly with a molar ratio of 1:1:1.

3 Results and discussion

The hot-pressed SiC_p/Al-Ti foils exhibit good bonding between the Ti and SiC_p/Al foils. The interfaces between the bonded Ti and Al foils are fairly straight, as seen from Fig. 1(a). Microstructure of the multi-layered SiC_p/Al-Ti foils rolled to 81.7% reduction in thickness is shown in Fig. 1(b). It can be found that good bonding is achieved. It is observed that no reaction occurs between Ti foils and SiC_p/Al foils after hot rolling at 400 °C. Also, it is found that a necking phenomenon takes place in the titanium layers of the foils. This may be due to the low ductility and high deformation hardening ratio of titanium compared with SiC_p/Al.

A backscattered electron image of the cross section of SiC_p/Al-Ti foils after reaction annealing at 1 200 °C is shown in Fig. 2(a). It is observed that different reactive layers are formed in the sample. The EDS equipped on SEM was used to analyze the composition of each

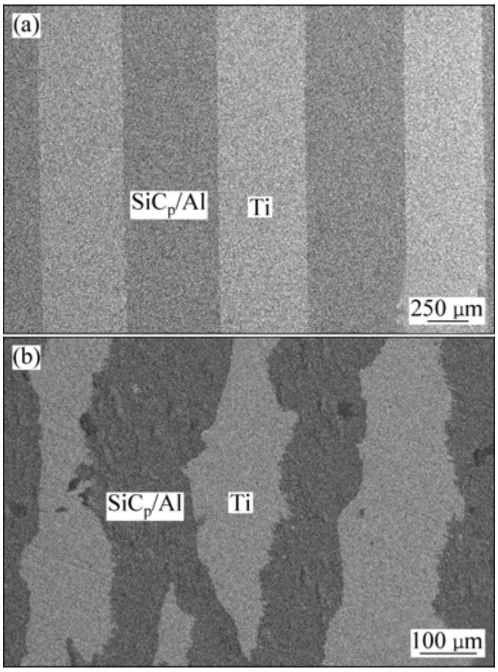

Fig. 1 Cross section microstructures of SiC_p/Al-Ti foils: (a) After hot-press; (b) After hot rolling

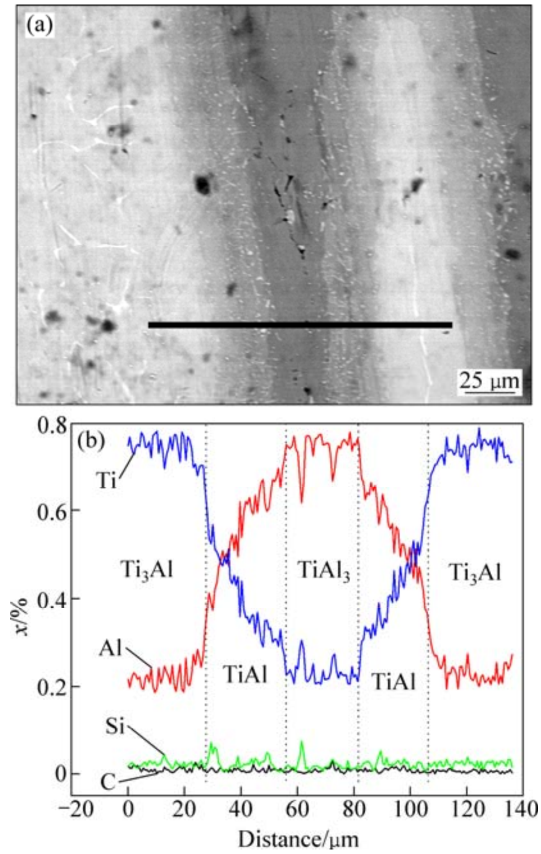

Fig. 2 Microstructure and composition of reactive layers of SiC_p/Al-Ti foils after annealing at 1 200 °C: (a) Backscattered electron image; (b) EDS profile

reactive layer. From Fig. 2, it can be seen that the reaction area of the sample is separated into three kinds of layers which are Ti_3Al, TiAl and $TiAl_3$. This phenomenon is in good agreement with previous research work [10−11]. Previous researchers have found that the route involves two steps: 1) formation of $TiAl_3$ phase at the interfaces between SiC_p/Al and Ti foils; 2) gradual transformation of $TiAl_3$ phase into TiAl phase, and formation of Ti_3Al due to the diffusion of Ti element into TiAl phase. Therefore, the following reaction can be concluded:

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

$$2Ti+ TiAl_3 \rightarrow 3TiAl \quad (2)$$

$$2Ti+ TiAl \rightarrow Ti_3Al \quad (3)$$

From Fig. 2, it can be seen that SiC particles disappear with the formation of intermetallic layers after annealing. This reveals that there exists a chemical reaction between SiC particles and TiAl during annealing. Figure 3 shows the DTA curves of the SiC_p-Al-Ti mixed powders upon heating to 1 200 °C at a rate of 20 °C/min. An exothermic peak occurs near 640 °C (indicated by *A* in Fig. 3). Previous research work suggests that $TiAl_3$ is formed from the reaction between Ti and Al at about

640 °C [12]. At approximately 665 °C, a sharp endothermic peak is attributed to the melting of Al (indicated by *B*), which is followed by a second large exothermic peak (indicated by *C*) associated with the formation of TiAl by the reaction between the previously formed $TiAl_3$ and Ti, and Ti_3Al is also synthetized during this reaction. DTA curve in Fig. 3 also exhibits an endothermic peak at 900 °C (indicated by *D*) due to the phase transformation of α-Ti to β-Ti. At about 1 050 °C, a new exothermic peak (indicated by *E*) is observed and can be attributed to the reaction of SiC with the titanium aluminide, which is in agreement with previous research work [13].

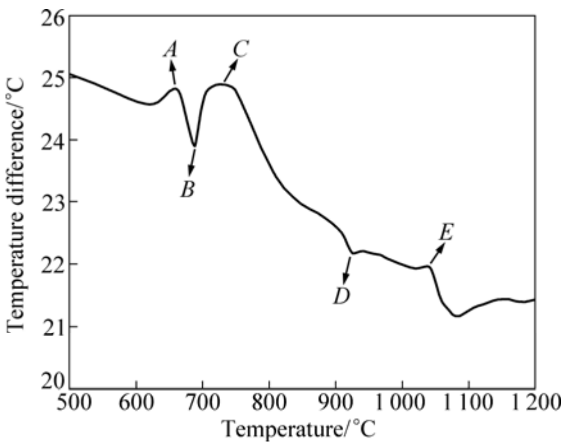

Fig. 3 DTA curve of SiC_p-Al-Ti mixed powders at heating rate of 20 °C/min (up to 1 200 °C)

The reaction products of SiC/Al-Ti foils after reaction annealing were analyzed using XRD, as shown in Fig. 4. It can be seen that the peaks of Ti_3Al, TiAl, $TiAl_3$ appear and peaks of both Ti and Al disappear. This result is in good agreement with the EDS profile shown in Fig. 2. In a word, Ti and Al matrix diffuse to form titanium aluminides. At the same time, SiC peaks also disappear and TiC and Ti_3AlC phases are produced. No

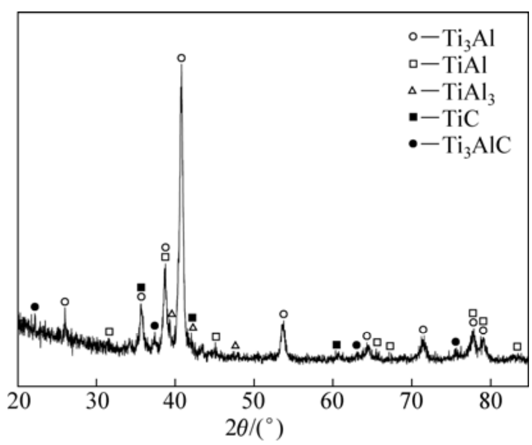

Fig. 4 XRD pattern of SiC_p/Al-Ti after reaction annealing at 1 200 °C

peaks of compounds containing the element Si appear. This is resulted from the negligible amount of these phases which are not enough to be detected by XRD. Many previous works have also proved that Ti_5Si_3 and Ti_3SiC_2 can be produced from the reaction between Ti/TiAl and SiC [14−17]. The in situ fine reinforcement particles which are synthetized and distributed in the reaction layers can be seen in Fig. 3. There may be the following reaction equations between SiC and titanium aluminides during reaction annealing according to the above analysis:

$$8(Ti)+3SiC \rightarrow Ti_5Si_3+3TiC \quad (4)$$

$$14(Ti)+6SiC \rightarrow 3Ti_3SiC_2+Ti_5Si_3 \quad (5)$$

$$Ti_3Al+3SiC+7(Ti) \rightarrow Ti_5Si_3+Ti_3AlC+2TiC \quad (6)$$

4 Conclusions

1) Three kinds of reactive layers, i.e, Ti_3Al, TiAl and $TiAl_3$, occur during the reaction annealing of Ti and SiC_p/Al foils at 1 200 °C. The reaction route involves two steps: formation of $TiAl_3$ phase at the interfaces of SiC_p/Al-Ti laminates; gradual transformation of $TiAl_3$ phase into TiAl phase, and the formation of Ti_3Al formed by the diffusion of Ti element into TiAl phase.

2) The titanium aluminide composite sheets are prepared through reaction annealing. And reinforced particles including TiC, Ti_3AlC, Ti_5Si_3 and Ti_3SiC_2 are produced by in situ reaction between SiC and titanium aluminides.

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SiC_p/Al-Ti 多层复合板材在轧制与反应退火过程的组织演化

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摘 要：通过真空热压及热轧制制备 SiC_p/Al-Ti 复合箔材。研究在 1 200 °C 反应退火时 Ti、Al 及 SiC 颗粒的扩散反应机理与组织演化过程。通过扫描电子显微镜及能谱对多层金属间化合物的微观组织与成分进行观察与分析，通过 X 射线衍射确定 SiC 与 TiAl 化合物之间的反应产物，并采用差热分析法对反应热力学进行分析。结果表明：试样中出现分层现象并且 SiC 颗粒消失；多层金属间化合物的成分确定为 Ti_3Al、TiAl 和 $TiAl_3$；退火过程中 SiC 与 TiAl 化合物发生反应，生成 Ti_3AlC、TiC、Ti_5Si_3 及 Ti_3SiC_2。

关键词：钛铝；扩散反应；SiC；复合材料

(Edited by YANG Bing)