Microstructure and Mechanical Properties of In-situ Synthesized Al$_2$O$_3$/TiAl Composites

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Abstract

Al$_2$O$_3$ particle-reinforced TiAl composites are successfully reaction-synthesized from the powder mixture of Ti, Al, TiO$_2$, and Nb$_2$O$_5$, using the hot pressing reaction synthesis technique. The microstructure and mechanical properties of the as-sintered products are investigated. It is found that in the as-sintered products consisting of $\gamma$-TiAl, $\alpha_2$-Ti$_3$Al, Al$_2$O$_3$, and NbAl$_3$ phases, the fine Al$_2$O$_3$ particles tend to disperse on the grain boundaries. With the Nb$_2$O$_5$ content increasing, the grains are remarkably refined and the Al$_2$O$_3$ particles are dispersing more uniformly in the TiAl matrix, forming a partial lamellar structure containing $\gamma$ and lamellar phases. The hardness of the in-situ composites increases gradually, and the bending strength and the fracture toughness of the as-sintered products reach the maximum value of 398.5 MPa and 6.99 MPa m$^{1/2}$, respectively, as the Nb$_2$O$_5$ content increases to 6 wt%.

Keywords: TiAl; composites; microstructure; mechanical properties; hot pressing

1 Introduction

Intermetallic compounds have long been considered to be ideal materials destined for high-temperature applications in aircraft and space shuttle turbine engines$^{[1-2]}$. Particular attention has been paid to the aluminides, especially $\gamma$-TiAl$^{[3]}$, thanks to its low density (3.8 g/cm$^3$), high melting point (about 1 773 K), excellent resistance to corrosion and oxidation.

However, its adoption is limited by its poor ductility and fracture toughness at room temperature and poor strength at elevated temperature. To improve these properties, of late, a second phase such as boride, carbide or nitride has been introduced to prepare composites. As a result, a number of composites including TiB$_2$/TiAl$^{[4]}$, SiC/TiAl$^{[5]}$, TiC/TiAl$^{[6]}$, and Ti$_2$AlC/TiAl$^{[7]}$ have been developed to associate the high strength and stiffness inherent in these ceramics with the low density of TiAl alloy. The existence of second-phase particles can prevent the movement of the grain boundaries, restrain the growth of grains, and improve the heat-resistance stability$^{[8-9]}$.

Al$_2$O$_3$ has been chosen as a ceramic reinforcement because of its advantageous thermo-mechanical behavior, inclusive of wear resistance, environmental stability, high temperature strength, and so on. Therefore, there arises a requirement to investigate the TiAl intermetallic matrix composites (IMCs) reinforced by Al$_2$O$_3$ particles.

In the previous study$^{[10-12]}$, the hot pressing process was used to produce IMCs based on particle-reinforced TiAl. It was found that dense in-situ IMCs were easily produced from pure titanium, pure aluminum, and oxide powders, using the process that offered the Al$_2$O$_3$-reinforced TiAl IMCs’

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superior properties. In this article, the hot pressing method is used to fabricate in-situ Al$_2$O$_3$/TiAl composites based on the Ti-Al-TiO$_2$-Nb$_2$O$_5$ system with clean, contaminant-free interfaces. Furthermore, the microstructure and mechanical properties of the as-sintered composites are studied in detail.

2 Experimental

Powders of Ti (280 mesh, 99.3% purity), Al (200 mesh, 99.5% purity), TiO$_2$ (0.5 µm, 99% purity), and Nb$_2$O$_5$ (500 mesh, 99.5% purity) were used as raw materials. The powder mixture of 57.46Ti-36.78Al-5.76TiO$_2$ was ball milled in alcohol for 2 h separately together with 0, 2 wt%, 6 wt%, 10 wt%, and 22 wt% Nb$_2$O$_5$. The samples were marked by A, B, C, D, and E, respectively. Further processes included drying at 80 °C for 5 h and sieving with a 200 mesh. Then the as-milled powders were pre-formed in a hot pressing setup under a vacuum of less than 10$^{-2}$ Pa, as illustrated in Fig.1. The sintering was carried out in two steps: pre-sintering at 600 °C for 1 h and final sintering at 1200 °C for 2 h, at a fixed pressure of 35 MPa. Then, the samples were held for 1 h at 1200 °C followed by cooling down to room temperature.

![Experimental setup for synthesizing Al$_2$O$_3$/TiAl.](image)

The fracture toughness was measured on the universal testing machine by using the single edge pre-cracked beam (SEPB) method with a notch depth of 0.4W (W is the width of specimen), at a cross-head speed of 0.5 mm/min, with a loading span of 30 mm. The fracture toughness, $K_{IC}$, is calculated by the following equation:

$$K_{IC} = Y \times 3PL a^{1/2}/(2BW^2)$$

where $P$ is the breaking load of the specimen, and $L$, $b$, and $h$ denote the span, width, and height, respectively.

3 Results and Discussion

3.1 Microstructural characterization

Fig.2 presents the XRD patterns of the in-situ composite samples prepared by hot pressing at 1200 °C for 60 min. According to XRD analysis, the as-sintered samples, which mainly consist of $\gamma$-TiAl, $\alpha_2$-Ti$_3$Al, and Al$_2$O$_3$ as three major phases together with a significantly smaller amount of NbAl$_3$ phase, have no peaks indicative of unreacted Ti, Al, TiO$_2$ or Nb$_2$O$_5$. Although it is hard to quantify the individual phases in the products, it is clear that the $\alpha_2$-Ti$_3$Al/$\gamma$-TiAl ratio and the intensity of the Al$_2$O$_3$ diffraction peaks increase with the Nb$_2$O$_5$ content increasing. The presence of the above-mentioned phases in the composite samples confirms the feasibility of the following in-situ reactions:

$$3\text{TiO}_2 + 4\text{Al} = 2\text{Al}_2\text{O}_3 + 3\text{Ti} \quad (1)$$
3Nb$_2$O$_5$ + 28Al = 5Al$_2$O$_3$ + 6NbAl$_3$  \( (2) \)

4Ti + 2Al = TiAl + Ti$_3$Al  \( (3) \)

Fig. 2  XRD patterns of in-situ Al$_2$O$_3$/TiAl composites with various Nb$_2$O$_5$ contents.

Fig. 3 shows the typical microstructures of Al$_2$O$_3$/TiAl composites with various Nb$_2$O$_5$ contents synthesized via hot pressing. With the Nb$_2$O$_5$ content increasing, the structure of the as-sintered composites becomes finer with remarkably refined grains of the composites with Al$_2$O$_3$ particles remaining on the grain boundaries. Also, the increase in Nb$_2$O$_5$ content renders more uniform distribution and dispersion of Al$_2$O$_3$ particles. The brighter areas change from an agglomerating state into an inter-penetrating network structure. From a comprehensive observation of the microstructures, there are pores that existed, which quantitatively decrease with the Nb$_2$O$_5$ content increasing.

Fig. 4 illustrates the optical micrographs (OM) of the as-sintered composites. The microstructure of the samples consists of single phase regions of $\gamma$-TiAl and lamellar regions of $\gamma$-TiAl+$\alpha_2$-Ti$_3$Al with a dispersion of randomly oriented Al$_2$O$_3$ particles. As shown in the optical micrographs, Al$_2$O$_3$ reinforcements, uniformly distribute in the matrix, and comprise of rod-like particles in the size of 0.4-2.5 $\mu$m. The grain size and the thickness of the lamellar batten are measured to be 9-35 $\mu$m and 0.6-5.0 $\mu$m, respectively.
EDS analysis shows that the thicker bright laminae are of nearly equiaxed $\gamma$-TiAl, whereas, the darker laminae are too thin to be identified by quantitative EDS analysis. With the Nb$_2$O$_5$ content increasing, the number of such fine Al$_2$O$_3$ particles dispersed in the matrix increases. This can be attributed to the ever-increasing amount of reinforcements (Al$_2$O$_3$) in the matrix as a result of more favorable kinetics of in-situ reactions at higher temperatures. These observations are again found to be in agreement with the conclusions drawn from the XRD study (see Fig.2).

**3.2 Mechanical properties**

The relative density measured by the Archimedes method increases steadily with the Nb$_2$O$_5$ content increasing, as illustrated in Fig.5. As shown in Fig.5, the density rises gradually from 96.5% to 97.5% as the Nb$_2$O$_5$ content increases from 2 wt% to 22 wt%.

Table 1 lists the mechanical properties of the composites at room temperature. A closer observation of the Rockwell hardness data reveals that the hardness of the as-sintered composites increases from 70.9 HRA to 75.5 HRA with the Nb$_2$O$_5$ content increasing from 0 to 22 wt%. The increase in hardness can be correlated with the amount of in-situ Al$_2$O$_3$ reinforcements. The amount of Al$_2$O$_3$ particles increases with the Nb$_2$O$_5$ content increasing. In the meantime, the NbAl$_3$ phase having a higher melting point and hardness than $\gamma$ or $\alpha_2$ phase, fills up a part of the pores, thereby significantly increasing the relative density and the hardness of the composites.

**Table 1 Mechanical properties of Al$_2$O$_3$/TiAl composites at room temperature**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hardness/HRA</th>
<th>Bending strength/MPa</th>
<th>$K_{IC}$(MPa·m$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>70.9</td>
<td>186.5</td>
<td>3.77</td>
</tr>
<tr>
<td>B</td>
<td>72.0</td>
<td>208.4</td>
<td>4.28</td>
</tr>
<tr>
<td>C</td>
<td>72.4</td>
<td>398.5</td>
<td>6.99</td>
</tr>
<tr>
<td>D</td>
<td>74.6</td>
<td>273.7</td>
<td>5.53</td>
</tr>
<tr>
<td>E</td>
<td>75.5</td>
<td>226.3</td>
<td>4.49</td>
</tr>
</tbody>
</table>

As can be seen clearly in Table 1, the bending strength of the as-sintered composites increases till the Nb$_2$O$_5$ content amounts to 6 wt% and then decreases as the Nb$_2$O$_5$ content continues to rise. The IMCs containing 6 wt% Nb$_2$O$_5$ exhibit the highest bending strength of 398.5 MPa. The strengthening effects of Nb$_2$O$_5$ are credited to the combination of two kinds of strengthening: strengthening by fine dispersed Al$_2$O$_3$ precipitates and by refined colony grain size. Of them, the former plays the chief role. The strengthening effect by the second-phase of Al$_2$O$_3$ can be expressed by

$$\sigma_c = \sigma_f V_f + \sigma_m V_m$$

(4)

where $\sigma_c$ is the strength of the composites, $\sigma_f$ the strength of the second phase particles, $V_f$ the volume fraction of the second phase particles, $\sigma_m$ the strength of the matrix, and $V_m$ the volume fraction of the matrix. With the Nb$_2$O$_5$ content increasing, $V_m$ increases gradually, so it levels with $\sigma_c$.

With regard to the reason why the bending strength of IMCs containing Nb$_2$O$_5$ of more than 6 wt% shows a dwindling tendency, it is found that
the Nb$_2$O$_5$ constituent of more than 6 wt% makes the Al$_2$O$_3$ reinforcements congregate easily on the grain boundaries, as illustrated in Fig.4. The region crowded with Al$_2$O$_3$ particles, especially found in the IMCs with larger Al$_2$O$_3$ contents, will have detrimental effects on the mechanical properties of the IMCs. Therefore, the agglomeration of Al$_2$O$_3$ must be responsible for the decrease in the mechanical properties of the IMCs containing more than 6 wt% Nb$_2$O$_5$.

Table 1 shows the toughness ($K_{IC}$) of the as-sintered composites. The as-sintered product containing 6 wt% Nb$_2$O$_5$ yields a toughness of 6.99 MPa·m$^{1/2}$, which is 85.4% higher than that of the as-sintered sample without Nb$_2$O$_5$. As the Nb$_2$O$_5$ content exceeds 6 wt%, the fracture toughness of the composites decreases gradually. This suggests that it is the dispersion of the hard particles that enables the introduction of a small amount of Al$_2$O$_3$, to improve the fracture toughness. However, further increase in Al$_2$O$_3$ content tends to form a network structure of the brittle Al$_2$O$_3$ phase resulting in the deterioration of toughness. Both the lamellar structure of TiAl and the right content of Al$_2$O$_3$ with felicitous dispersion are requisite for improving toughness.

Mechanical tests have indicated that the 6 wt% Nb$_2$O$_5$ content of IMCs yields the best mechanical properties in other contents. The mechanism that causes the changes in the mechanical properties of IMCs has been discussed by S. L. Kampe, et al[16], who have suggested that the increase in strength of Al$_2$O$_3$-reinforced IMCs comes from both indirect and direct sources[3]: the former refers to the microstructural changes in the matrix of the IMCs, for example, reinforcement-derived grain refinement and interstitial solid solution strengthening; the latter refers to the interaction of dislocations among the reinforced particles. These conclusions also run parallel to the course, along with those of Y. L. Yue, et al[6].

4 Conclusions

Al$_2$O$_3$/TiAl composites can be produced from the powder mixture of Ti, Al, TiO$_2$, and Nb$_2$O$_5$ by way of hot pressing. The as-sintered products consist of γ-TiAl, α$_2$-Ti$_3$Al, Al$_2$O$_3$, and NbAl$_3$ phases. Al$_2$O$_3$ particles tend to disperse on the grain boundaries forming a lamellar γ + α$_2$ structure and the composites consist of an interpenetrating network of fine Al$_2$O$_3$ particles. With the Nb$_2$O$_5$ content increasing, the grains of the composites are being remarkably refined and the dispersion of Al$_2$O$_3$ particles becomes more uniform. The relative density of the composites increases with the Nb$_2$O$_5$ content increasing. The Rockwell hardness increases steadily from 70.9 HRA to 75.5 HRA as the Nb$_2$O$_5$ content increases from 0 to 22 wt%. The bending strength and fracture toughness of the as-sintered composites reach the maximal values of 398.5 MPa and 6.99 MPa·m$^{1/2}$, respectively, when the Nb$_2$O$_5$ content is at 6 wt%, but then decrease when it exceeds 6 wt%.

References


[8] Yang J M, Kse W, Jeng S M. Development of TiC particle-rein-


**Biography:**

**Ai Taotao**  Born in 1981, he received B.S. from Shaanxi University of Science and Technology in 2007, and then became a teacher there. His main research interest lies in metal matrix composites.

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