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Tailoring ultrafine grained and dispersion-strengthened Ti_2AlC/TiAl composite via a new fabrication route

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Abstract. ***In situ*** **Ti_2AlC/TiAl composite was fabricated by hot-pressing method via the reaction system of Ti_3AlC_2 and Ti-Al pre-alloyed powders at low temperature of 1150°C. The composite mainly consisted of TiAl, Ti_3Al and Ti_2AlC phases. Fine Ti_2AlC particles were homogeneously distributed and dispersed in the matrix. Ti_2AlC played the significant role of deflecting and blunting the cracks and contributed to improve the mechanical properties of the composite. The Vickers hardness, flexural strength and fracture toughness were 6.2 GPa, 993.9 ± 50.8 MPa and 6.7 ± 0.3 MPa $m^{1/2}$, respectively.**

Keywords. **Metal–matrix composites; mechanical properties; microstructures; sintering.**

1. Introduction

γ-TiAl-based intermetallic alloys have been extensively investigated as potential high-temperature materials in the gas turbine industry because of their attractive properties, such as low density, excellent high-temperature strength and good oxidation resistance [1–3]. However, their applications are limited by their low room temperature ductility and insufficient strength at ambient temperature or elevated temperature above 800°C [4,5]. Many studies indicate that the composite technology is an effective approach to improve the properties of γ-TiAl by introducing suitable reinforcements [3–6].

Owing to the excellent physical and mechanical properties of Ti_2AlC, such as high Young's modulus, high strength, excellent machinability, high electrical and thermal conductivities, especially, almost same thermal expansion coefficient of Ti_2AlC (8.8×10^{-6} K^{-1}) and TiAl (12×10^{-6} K^{-1}) [7], it has been considered as the most attractive reinforcement for γ-TiAl alloys. For example, Ti_2AlC/TiAl composites were fabricated by combustion synthesis and hot-pressing consolidation in the Ti–Al–Nb–C system [8]. Yang *et al* [9] produced Ti_2AlC/TiAl composite by spark plasma sintering (SPS) using pre-alloyed Ti–50 at% Al powders and 10 vol% carbon nanotubes as raw materials. Although Ti_2AlC could enhance the strength and ductility of the TiAl intermetallic, Ti_2AlC reinforcements were mainly generated by *in-situ* synthesis reaction via Ti/Al/C or Ti/Al/CNTs systems [8,9].

In our study, ultrafine grained and dispersion-strengthened Ti_2AlC/TiAl composite was first fabricated by hot-pressing method via the reaction system of Ti_3AlC_2 and Ti–Al pre-alloyed powders. The reinforcing and toughening mechanisms of the composite were investigated.

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2. Experimental

Commercial powders of Ti (99.8% purity, content of oxygen and nitrogen is 0.04 and 0.02 wt%, respectively, ~35 μm), Al (99.6% purity, content of oxygen and nitrogen is 0.11 and 0.04 wt%, respectively, ~55 μm) and TiC (99.9% purity, ~20 μm) were used to prepare high purity Ti_3AlC_2 powders via 2TiC–Ti–1.2Al system [10] and Ti–Al pre-alloyed powders via Ti–48Al (at%) system at 900°C for 30 min. Then, 15 wt% Ti_3AlC_2 and 85 wt% Ti–Al pre-alloyed powders were precisely weighed and milled sufficiently for 2 h using alcohol as an agent to avoid oxidation. Then, the mixed powders were put into a graphite mould pre-sprayed with boron nitride (BN) coatings and then hot-pressed at 1150°C for 2 h with a rate of 10°C min^{-1} under a pressure of 30 MPa in vacuum (0.01 Pa). Subsequently, the product was cooled down to room temperature.

The phase and microstructures of the composite were examined by X-ray diffraction (XRD) and scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS), respectively. The flexural strength and fracture toughness were measured by electromechanical universal testing equipment using the three point bending mode with a span of 20 mm. The dimensions of samples were $3 \times 4 \times 25$ mm^3 and $3 \times 4 \times 30$ mm^3 with a notch of 1 mm depth and 0.1 mm width, respectively. The crosshead speeds were 0.5 and 0.05 mm min^{-1}, respectively. Vickers hardness was tested by the hardness testers at the load of 9.8 N for 15 s.

3. Results and discussion

Figure 1 displays the XRD patterns of the as-synthesized Ti_3AlC_2 powders, Ti–Al pre-alloyed powders sintered at 900°C for 30 min and Ti_2AlC/TiAl composite sintered at 1150°C for 2 h via the reaction system of Ti_3AlC_2 and

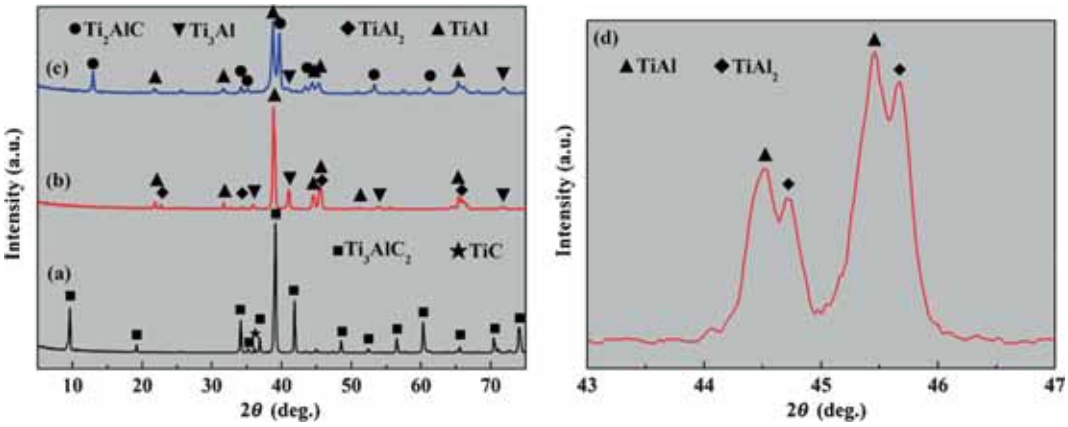

Figure 1. XRD patterns of the (**a**) as-synthesized Ti_3AlC_2 powders, (**b**) Ti–Al pre-alloyed powders sintered at 900°C for 30 min, (**c**) Ti_2AlC/TiAl composite sintered at 1150°C for 2 h via the reaction system of Ti_3AlC_2 and Ti–Al pre-alloyed powders and (**d**) Ti–Al pre-alloyed powders sintered at 900°C for 30 min (the diffraction angle is between 43 and 47°).

Ti–Al pre-alloyed powders. As shown in figure 1a, the as-synthesized Ti_3AlC_2 powders are composed of Ti_3AlC_2 and a small amount of TiC phases. The Ti_3AlC_2 and TiC content in the as-synthesized powders can be quantitatively estimated from the integrated XRD peak intensities according to the following equations [11]:

$$\omega_{TC} = 1.084/(I_{TAC}/I_{TC} + 1.084), \quad (1)$$

$$\omega_{TAC} = 1 - \omega_{TC}, \quad (2)$$

where ω_{TC} and ω_{TAC} are the mass percentages of TiC and Ti_3AlC_2, respectively. I_{TAC} and I_{TC} are the integrated diffraction intensities of Ti_3AlC_2 (104) peak and TiC (111) peak, respectively. The calculations show that the content of Ti_3AlC_2 is about 99.4 wt%. Moreover, it is found that the Ti-Al pre-alloyed powders after sintering at 900°C for 30 min mainly consist of TiAl, Ti_3Al and $TiAl_2$ phases (figure 1b). Especially, as clearly seen in figure 1d, the diffraction peaks of $TiAl_2$ phase appear obviously. XRD results of Ti_2AlC/TiAl composite presented in figure 1c accurately confirm that it mainly consists of γ-TiAl, α_2-Ti_3Al and Ti_2AlC phases, then constitutes $\alpha_2 + \gamma$ dual phase alloy. Actually, the dual phase alloys have exhibited room temperature and high temperature strengths equivalent to that of super alloys [12]. Moreover, the interstitial content such as oxygen and nitrogen cannot be detected from XRD due to the high vacuum (0.01 Pa) and low content. However, the Ti_3AlC_2 and $TiAl_2$ phases are not detected by XRD analysis. It is indicated that Ti_3AlC_2 and $TiAl_2$ had reacted completely corresponding to the reactions as follows:

$$2TiAl_2 + Ti_3Al + TiAl = 6TiAl, \quad (3)$$

$$Ti_3AlC_2 = Ti_2AlC + TiC, \quad (4)$$

$$TiC + TiAl = Ti_2AlC. \quad (5)$$

Figure 2 shows SEM images of the as-obtained Ti_2AlC/TiAl composite after polishing and etching, and the fracture surface of the as-obtained Ti_2AlC/TiAl composite and the TiAl alloy prepared at 1150°C for 2 h. We can observe in figure 2a that the *in situ* Ti_2AlC particles with an average dimension of 3 μm are homogeneously distributed and dispersed in the matrix. The fracture surface of the as-obtained Ti_2AlC/TiAl composite indicates that ultrafine γ-TiAl grains and dense structure can be obtained. Furthermore, it is found that some of Ti_2AlC particles are excluding during the breaking process, as seen clearly in figure 2b. Particularly, typical α_2/γ lamellar colonies can be obtained. Compared to the TiAl alloy prepared at 1150°C for 2 h from Ti–Al pre-alloyed powders (figure 2c), the dimension of the ultrafine γ-TiAl grains in the as-obtained Ti_2AlC/TiAl composite are 5–10 μm, which is far lower than that of the grains in the TiAl alloy with the dimension of 15–25 μm, which contribute to improve the mechanical properties of the composite.

Table 1 lists the mechanical properties of some typical materials. Vickers hardness of the as-obtained Ti_2AlC/TiAl composite is 6.2 GPa, about 1.32 times of Ti_2AlC (4.7 ± 0.2 GPa [13]) and 2.14 times of TiAl alloy (2.9 GPa [14]). The flexural strength of the product reaches the outstanding value of 993.9 ± 50.8 MPa, about 1.64 times of Ti_2AlC (606 ± 20 MPa [13]), 2.16 times of TiAl alloy (460 MPa [14]), 2.49 times of Al_2O_3/TiAl composite (398.5 MPa [15]), 2.05 times [14] and 1.52 times of Ti_2AlC/TiAl composites [16] and 3.15 times of Ti_3AlC_2–Ti_2AlC/TiAl composite (316 MPa [17]), respectively. The fracture toughness value measured by the single-edge notch beam (SENB) method is close to that of Ti_2AlC, Al_2O_3/TiAl, Ti_2AlC/TiAl and Ti_3AlC_2–Ti_2AlC/TiAl composites, as 6.7 ± 0.3 MPa $m^{1/2}$. But we found that the fracture toughness of the composite is slightly lower than that of TiAl alloy sintered at 1300°C [14]. Higher sintering temperature is beneficial to the grain growth and densifying, thereby improves the fracture toughness. Furthermore, the interstitial impurities of oxygen and nitrogen can influence the ductility and toughness. It indicates that Ti_2AlC/TiAl composite fabricated by hot-pressing

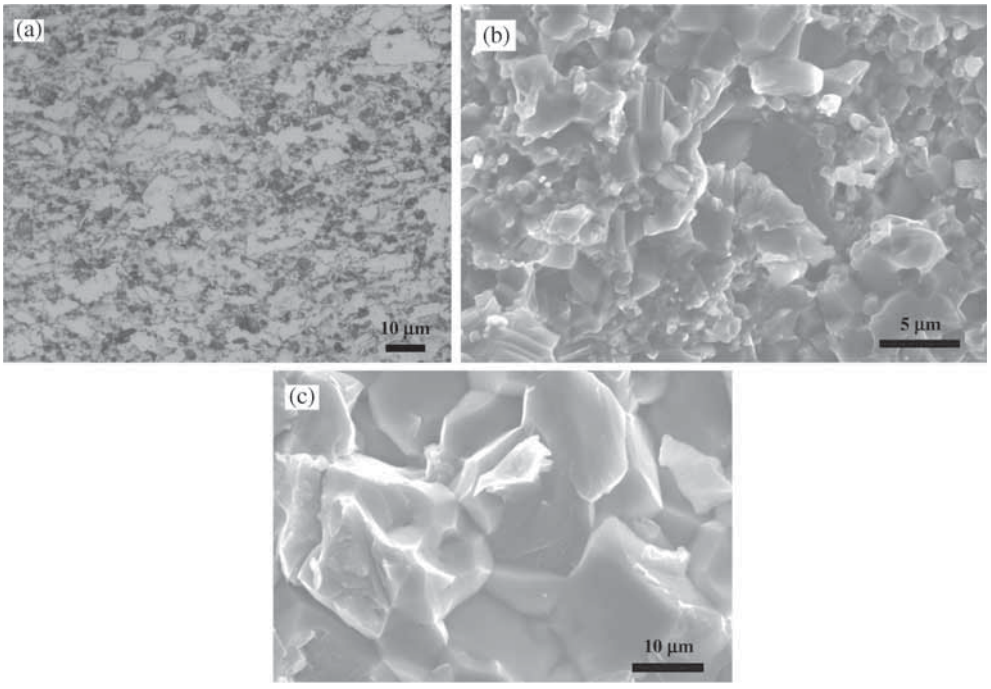

Figure 2. SEM images of the (**a**) as-obtained $Ti_2AlC/TiAl$ composite after polishing and etching, (**b**) the fracture surface of the as-obtained $Ti_2AlC/TiAl$ composite and (**c**) TiAl alloy.

Table 1. The mechanical properties of some typical materials.

Materials	Vickers hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa $m^{1/2}$)	Ref.
Ti_2AlC	4.7 ± 0.2	606 ± 20	6.9 ± 0.2	[13]
TiAl	2.9	460 ± 21	7.19 ± 0.09	[14]
15wt%$Ti_2AlC/TiAl$	–	486 ± 16	7.78 ± 0.13	[14]
$Al_2O_3/TiAl$	–	398.5	6.99	[15]
$Ti_2AlC/TiAl$	–	653.2 ± 76.8	6.6 ± 0.5	[16]
Ti_3AlC_2–$Ti_2AlC/TiAl$	–	316 ± 11	7.3 ± 0.4	[17]
$Ti_2AlC/TiAl$	6.2	993.9 ± 50.8	6.7 ± 0.3	This work

method via the reaction system of Ti_3AlC_2 and Ti–Al pre-alloyed powders does not reduce the toughness of the product. Conversely, the strength is improved dramatically.

During the breaking process of the materials, crack propagation is the nature of reflecting the toughness of composites [18]. When cracks expand along the grain boundaries, the reinforcing agent with obvious pinning effect is capable of deflecting the crack growth path and absorbing the crack propagating energy. As a result, the toughness of the composite can be improved dramatically, and it has already been confirmed in table 1. Figure 3 presents the typical crack propagation of the TiAl alloy and $Ti_2AlC/TiAl$ composite prepared at 1150°C for 2 h. As seen in figure 3a, the TiAl alloy prepared at 1150°C for 2 h from Ti–Al pre-alloyed powders exhibits intergranular fracture, and such a fracture is a typical brittle fracture. As seen in figure 3b, for the $Ti_2AlC/TiAl$ composite, the crack deflection, Ti_2AlC particulates pulling-out and transgranular fracture are the main energy dispersive modes, contributing to the increase in fracture toughness. Therefore, the *in situ* formed Ti_2AlC particles play the significant role of deflecting and blunting the cracks, indicating the positive influence on the mechanical properties. Moreover, as seen in figure 1c, γ-TiAl + α_2-Ti_3Al dual phases can be obtained, which constitute a lamellar structure. The lamellar structure yields large plastic strains near the crack tip and increases resistance to crack propagation with crack length [19]. Actually, due to the α_2/γ dual phases and ultrafine grains, the composite exhibits best ductility and strength at room temperature. At the moment, the Hall–Petch relationship controls the microstructure–property relationship of the composite. Moreover, the dispersed Ti_2AlC particulates may also be responsible for the significantly modifying effects for the composite.

Above all, the crack grows in a manner of large deflection, increasing the surface area and changing the stress field distribution, and there exist many fine *in situ* formed Ti_2AlC particles simultaneously, then improving the fracture toughness. The dense structure, ultrafine grains and dispersed

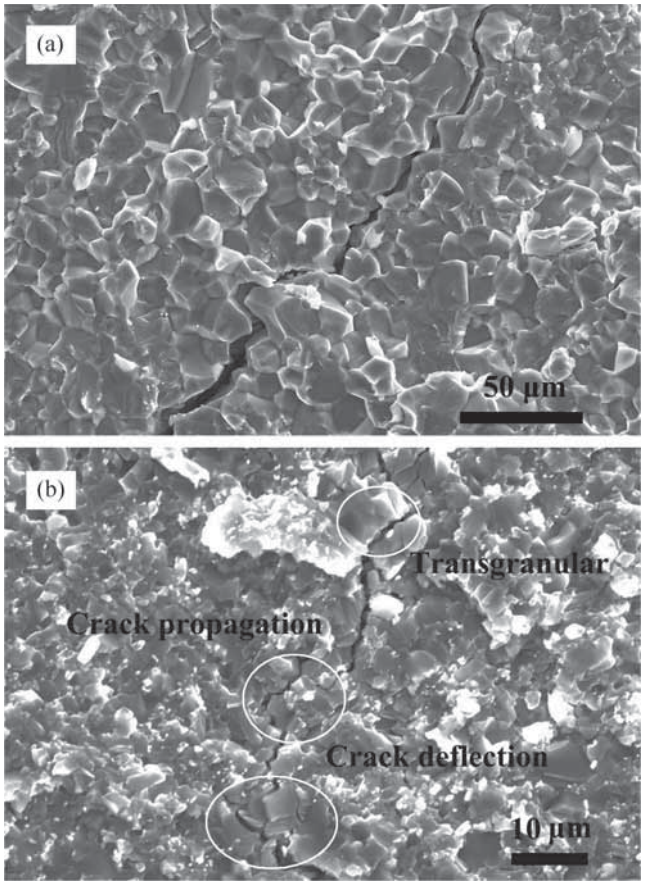

Figure 3. Typical crack propagation of the (**a**) TiAl alloy and (**b**) Ti_2AlC/TiAl composite.

Ti_2AlC particles are also helpful to improve the hardness and strength of the composite.

4. Conclusions

In summary, ultrafine grained and dispersion-strengthened Ti_2AlC/TiAl composite was first successfully fabricated by hot-pressing method via the reaction system of Ti_3AlC_2 and Ti–Al pre-alloyed powders. The product mainly consisted of TiAl, Ti_3Al and Ti_2AlC phases. *In situ* Ti_2AlC particles with an average dimension of 3 μm were homogeneously distributed and dispersed in the matrix, and exhibited significant strengthening and toughening effects to the TiAl matrix composite. Vickers hardness, flexural strength and fracture toughness of the product were 6.2 GPa, 993.9 ± 50.8 MPa and 6.7 ± 0.3 MPa $m^{1/2}$, respectively.

Acknowledgements

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References

[1] Froes F H, Suryanarayana C and Eliezer D 1992 *J. Mater. Sci.* **27** 5113

[2] Kothari K, Radhakrishnan R and Wereley N M 2012 *Prog. Aerosp. Sci.* **55** 1

[3] Ding R G, Li H Y, Hu D W, Martin N, Dixon M and Bowen P 2015 *Intermetallics* **58** 36

[4] Wang J, Zhao N Q, Nash P, Liu E Z, He C N, Shi C S and Li J J 2013 *J. Alloys Compd.* **578** 481

[5] Mutoh Y, Zhu S J, Hansson T, Kurai S and Mizuhara Y 2002 *Mater. Sci. Eng. A* **323** 62

[6] Shu S L, Xing B, Qiu F, Jin S B and Jiang Q C 2013 *Mater. Sci. Eng. A* **560** 596

[7] Barsoum M W, Ali M and El-Raghy T 2000 *Metall. Mater. Trans. A* **31** 1857

[8] Shu S, Qiu F, Jin S, Lu J and Jiang Q 2011 *Mater. Des.* **32** 5061

[9] Yang F, Kong F T, Chen Y Y and Xiao S L 2010 *J. Alloys Compd.* **496** 462

[10] Ai T, Wang F, Zhang Y, Jiang P and Yuan X 2013 *Adv. Appl. Ceram.* **112** 424

[11] Xu X W, Fu C K, Li Y X, Zhu J Q and Mei B C 2006 *Trans. Nonferrous Met. Soc. China* **16** s490

[12] Draper L S, Das G, Locci I, Whittenberger J D, Lerch B A and Kestler H 2003 In Y W Kim, H Clemens and H H Rosenberger (eds) *Gamma titanium aluminides* p 207

[13] Bai Y, He X, Wang R, Sun Y, Zhu C, Wang S and Chen G 2013 *J. Eur. Ceram. Soc.* **33** 2435

[14] Yang C H, Wang F, Ai T T and Zhu J F 2014 *Ceram. Int.* **40** 8165

[15] Ai T T 2008 *Chinese J. Aeronaut.* **21** 559

[16] Ai T T, Liu F, Yu Q, Feng X M, Zhang Y T and Li W H 2014 *Int. J. Mater. Res.* **105** 1118

[17] Ai T T, Wang F, Feng X M and Ruan M M 2014 *Ceram. Int.* **40** 9947

[18] Zhang Z and Chen D L 2006 *Scripta Mater.* **54** 1321

[19] Kim Y W 1998 *Intermetallics* **6** 623