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## Fabrication of in situ  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites with controlled quasi-network architecture using reactive infiltration

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#### **ABSTRACT**

Fully dense Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites were successfully in situ synthesized by reactive infiltration using Ti powders and Al–Si alloys as raw materials. Fine  $Ti<sub>5</sub>Si<sub>3</sub>$  particles exhibit a unique quasi-network distribution, remarkably hindering the coarsening of  $\alpha_2/\gamma$  lamellar colonies. Compared to the as-cast samples, the mean size of lamellar colonies is much finer and thus the resulting  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites show the enhanced room temperature elongation, up to 2.5%, while retaining the ultimate tensile strength of 414 MPa. Moreover, Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites display superior high temperature tensile properties. Consequently, the novel Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites have potential for high temperature applications in aeronautics and aerospace.

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#### 1. Introduction

Compared to TiAl intermetallic compound, TiAl matrix composites are more attractive for aerospace applications due to outstanding creep resistance and oxidation resistance [\[1\]](#page--1-0). Recently,  $Ti<sub>5</sub>Si<sub>3</sub>$  particles are introduced into TiAl matrix, thus enhancing many mechanical properties  $[2,3]$  $[2,3]$ . Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites are commonly fabricated by ingot metallurgy (IM) [\[2](#page--1-0)–[5\]](#page--1-0) and powder metallurgy (PM) [\[6,7\].](#page--1-0) However, regardless of IM or PM, additional processes including homogenization treatment [\[2\]](#page--1-0) and/or hot isostatic pressing [\[6\]](#page--1-0) must be utilized to homogenize and densify the materials, largely increasing the production cost and coarsening the microstructure. Furthermore, the room temperature elongation ( $\leq$  1%) of Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites is far from the desired level and high temperature performances also need to be further improved [\[3\]](#page--1-0). Therefore, it is of much significance to exploit an alternative method for producing TiAl matrix composites.

In the present work,  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites were prepared using reactive infiltration including two stages: (i) infiltration of molten Al–Si alloys into porous titanium performs to obtain Ti–Al bimetal composites; (ii) reaction annealing of Ti–Al bimetal composites. As reported in our previous work [\[8\],](#page--1-0) Ti–Al bimetal composites showed excellent deformability. Thus complex

<http://dx.doi.org/10.1016/j.matlet.2016.09.015> 0167-577X/@ 2016 Elsevier B.V. All rights reserved. components of  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites could be directly attained by the preforming of Ti–Al bimetal composites followed by reaction annealing. This could provide a feasible near-net-shape method for solving the forming of brittle TiAl matrix composites. In this paper, fabrication parameters were investigated. The emphasis was on microstructure evolution and the reaction mechanism between Ti and Al–Si alloys. Finally, tensile properties at different temperatures were evaluated.

#### 2. Materials and methods

68 wt% Ti powders and 32 wt% Al–6Si alloys were used to synthesize Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites by reactive infiltration. Firstly, spherical Ti powders (100  $\mu$ m) were pressed into porous titanium preforms with porosity of 45 vol%. Secondly, the infiltration of molten Al–6Si alloys into porous titanium preforms was carried out at 630 °C under a pressure of 20 MPa in vacuum furnace, thus achieving Ti–Al bimetal composites. Thirdly, reaction annealing included two stages: (i) initial annealing of Ti–Al bimetal composites was conducted at 1250 °C for 1 h with no pressure in order to consume Al completely. The pressure was subsequently raised up to 40 MPa and kept for 2 h to synthesize  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites; (ii) secondary annealing was performed at 1370  $\degree$ C for 15 min to obtain fully lamellar TiAl composites.

Microstructure evolution of Ti–Al bimetal composites during reactive infiltration and morphologies of  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites were characterized using optical microscope (OM, Olympus





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PMG-3), scanning electron microscope (SEM, Quanta 200FEG) and transmission electron microscope (TEM, FEI Tecnai G2 F30). Various phases were identified by X-ray diffraction (XRD, Empyrean), SEM/EDX and TEM. The composite density was determined by Archimedes' principle. Tensile tests were conducted at 25–850 °C on an Instron-5500 universal testing machine with an initial rate of 0.5 mm/min.

#### 3. Results and discussion

#### 3.1. Microstructure evolution during reactive infiltration

Microstructure evolution of Ti–Al bimetal composites during reactive infiltration are indicated in Fig. 1. After the infiltration at 630 °C, spherical Ti particles are uniformly distributed in Al–6Si alloys, obtaining Ti–Al bimetal composites. As shown in the inset of Fig. 1(a), thin reaction layers are found around Ti particles and identified as Ti(Al, Si)<sub>3</sub> phases with the compositions of 21.89 at% Ti–66.75 at% Al–11.36 at% Si based on SEM/EDX. This is in consistence with XRD results (see in Fig. 1(b)).

As shown in Fig.  $1(c)$  including the inset, after initial annealing of Ti–Al bimetal composites at 1250 °C, obviously, the microstructure contains numerous network cells composing of three different phases: light grey spherical phases, dark grey phases and white particles. Based on SEM/EDX, the respective compositions are 71.89 at% Ti–25.85 at% Al–2.26 at% Si, 47.02 at% Ti–46.75 at% Al– 6.23 at% Si and 60.19 at% Ti–8.68 at% Al–31.13 at% Si, corresponding to  $\alpha_2$ -Ti<sub>3</sub>Al, γ-TiAl and Ti<sub>5</sub>Si<sub>3</sub>, shown in the inset of Fig. 1(c). XRD shows the same results (see Fig. 1(b)). Spherical  $\alpha_2$  phases in the center of network cells are surrounded by  $\gamma$  phases. The walls of network cells are consisted of Ti<sub>5</sub>Si<sub>3</sub> particles. Besides, some of Ti<sub>5</sub>Si<sub>3</sub> particles are uniformly dispersed within  $\gamma$  phases. The mean size of network cells is  $100 \mu m$ , close to the size of original Ti particles.

After secondary annealing at 1370 °C, lamellar microstructure is achieved as shown in Fig.  $1(d)$ . The selected area electron diffraction (SAED) patterns shown in Fig.  $1(e)$  confirm that the fully lamellar structure is composed of  $α_2$ -Ti<sub>3</sub>Al and γ-TiAl, and the dispersed particles are identified as  $Ti<sub>5</sub>Si<sub>3</sub>$ , which agrees well with XRD results shown in Fig. 1(b). The majority of  $Ti<sub>5</sub>Si<sub>3</sub>$  particles are precipitated at the boundaries of lamellar colonies, forming a quasi-network structure, while a small quantity of  $Ti<sub>5</sub>Si<sub>3</sub>$  particles are dispersed within lamellar colonies. The lamellar colony size is in the range of  $30-60 \mu m$ , much smaller than that of as-cast samples (several hundred micrometers) [\[2,3,5\].](#page--1-0) The average lamellar spacing is approximately 200 nm (see Fig.  $1(e)$ ). This means that in situ  $Ti<sub>5</sub>Si<sub>3</sub>$  particles restrict the coarsening of lamellar colonies significantly. In addition, the composites show a low density of 3.89  $g/cm<sup>3</sup>$  and a high relative density of 99.6%.

#### 3.2. Formation mechanism of quasi-work Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites

Formation mechanism of quasi-network  $Ti<sub>5</sub>Si<sub>3</sub>/TiAl$  composites is illustrated in [Fig. 2](#page--1-0) During infiltration, molten Al–Si alloys continuously infiltrate into porous titanium performs, meanwhile Ti



Fig. 1. Microstructure evolution of Ti-Al bimetal composites during reactive infiltration (a) optical microstructure of Ti-Al bimetal composites. The inset is an enlarged SEM image of (a), showing Ti(Al,Si)<sub>3</sub> interfacial layers; (b) XRD patterns for the infiltration at 630 °C, initial annealing at 1250 °C and secondary annealing at 1370 °C; (c) SEM morphologies of spherical Ti<sub>3</sub>Al/TiAl composites with quasi-network Ti<sub>5</sub>Si<sub>3</sub> particles after initial annealing. The inset is a magnified SEM image of a network cell; (d) representative SEM image of resulting quasi-network Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites after secondary annealing; (e) TEM images of resulting Ti<sub>5</sub>Si<sub>3</sub>/TiAl composites and SAED patterns of Ti<sub>5</sub>Si<sub>3</sub>, TiAl and Ti<sub>3</sub>Al phases.

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