



Microstructure characterization and mechanical properties of TiB₂/TiAl in situ composite by induction skull melting process

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ABSTRACT

Microstructures and mechanical properties of Ti–45Al–2Nb–1.5V–1Mo–0.3Y + 3 vol% TiB₂ in situ composite fabricated by induction skull melting (ISM) process were investigated. Results showed that ISM process is feasible to fabricate high-quality in situ TiB₂/TiAl composite. The composite exhibits much finer lamellar colony size and lamellar spacing, compared with its TiAl base. Hexagonal primary and needlelike second TiB₂ particles uniformly distribute mainly near lamellar colony boundaries with a few inside lamellar colonies. TiB₂ crystals in current TiAl composite exhibit faceted growth mode along [0001] direction. The diverse morphologies of TiB₂ are principally ascribed to intrinsic crystal structure and different growth stages during solidification. This composite exhibits much better tensile strength and ductility than its TiAl matrix, with tensile strength (UTS) 610 MPa and elongation (ϵ) 0.7% at room temperature, and UTS 505 MPa and ϵ 12% at 800 °C, respectively.

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1. Introduction

As potential structural materials for high-temperature applications, γ -TiAl alloys suffer from poor ductility and bad fracture toughness. Thus, considerable efforts have been devoted to improve their mechanical properties over the last decades [1,2]. Among the various attempts made to improve the ductility and properties of TiAl alloys, such as alloying, heat treatment, thermal-mechanical processing, introducing borides offers both excellent grain refinement and composite/dispersion strengthening effects [2–4].

TiB₂ has been identified as the most attractive reinforcement to TiAl matrix composites, mainly due to its comparable thermal expansion coefficient to TiAl alloy and excellent chemical compatibility with TiAl matrix [5], significant attentions have been paid to develop TiB₂/TiAl matrix composites by powder metallurgy (PM) and ingot metallurgy (IM) techniques [6–13], i.e. XDTM [6,7], SHS [8], reactive sintering [9,10], and plasma arc melting [11] and vacuum arc remelting (VAR) [12,13]. Compared with PM process, IM technique offers significant advantages to prepare TiAl matrix composites, such as high compactness and good cleanness, fast processing, large scale production, cost-effective and flexibility. Martin Marietta Laboratory has fabricated TiAl + 6 vol% TiB₂ in situ composite ingots using XD + ISM process [14], TIMET produced 110 kg TiB₂/TiAl composite ingot by VAR method [13]. However, clustering

of reinforcements easily takes place during arc melting and solidification process, which deteriorates the final mechanical properties. Akio Hirose et al. have reported that, using plasma arc melting process, TiAl composite containing 5 vol% TiB₂ has the best tensile strength of 550 MPa, which is 140 MPa greater than that of the Ti–34 mass% Al base, but degradation occurs in the composite containing 7 vol% TiB₂ and in the composites reinforced by coarse TiB₂ powders [11]. Up to now, induction skull melting (ISM) technique is considered as one of the best methods to produce TiAl alloys [15,16]. Many physical phenomena are involved in ISM, such as induction heating, electromagnetic stirring and electromagnetic suspending. Therefore, using ISM process, clean and dense TiB₂/TiAl composites with homogeneous microstructure are expected to be fabricated, simultaneously producing ingot or castings by pouring. In spite of numerous investigations on preparation and mechanical properties of in situ synthesized TiAl composites, by now, little attention has been paid to TiB₂/TiAl in situ composites via ISM process.

The objective of the present work is to investigate the microstructure and mechanical properties of in situ synthesized TiB₂/TiAl composite via ISM process. The growth mechanism and tensile properties of this TiB₂/TiAl composite were discussed in detail.

2. Experimental

The stoichiometric ratio of the current composite was designed as Ti–45Al–2Nb–1.5V–1Mo–0.3Y (at%) + 3 vol% TiB₂. Excessive content of reinforcements is out of significant meaning, due to the

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poor ductility of TiAl alloys. The parent materials used in this study contained Ti sponge (>99.99%), pure Al (>99.99%), master alloy Al–Nb (52.6% Nb), Al–V (58.5% V), Al–Mo (50.7% Mo), Al–Y (80.2% Y) (all in wt.%). To fabricate in situ synthesized TiB₂/TiAl composite, powder mixtures of boron particles (2–5 μm), Ti and Al particles (–325 mesh) were first thoroughly blended in a V-blender with weight ratio of Ti–20%Al–10%B and were subsequently cold compressed into several small compacts. Finally, these powder compacts were placed dispersedly in the core of crucible before melting. The experimental procedure can be briefly described as follows: Firstly the copper crucible is charged with raw materials with placing sequence of Ti sponge, Al blocks, master alloys, mixture compacts, Ti sponge, and Al blocks. After the melting chamber is vacuumized and is backfilled with argon, induction heating starts to melt the loaded raw materials, with power increasing rate of 1–1.5 kW/s. During melting process, the water cooling system assures the formation of skull layer of TiAl composite on the inner surface of crucible, which prevents the contamination to melt from crucible. Generally the skull in upper place of the crucible falls down when the melt temperature is high enough. It is about at heating power over 290 kW that the loaded materials are completely melted, now keep the TiAl composite melt at molten state and electromagnetic stirring condition for 2–5 min. Finally, tilt the crucible to pour the melt into a steel mould preheated at 400 °C. A TiB₂/TiAl composite ingot with dimensions of $\Phi 140 \text{ mm} \times 160 \text{ mm}$ was gained after cooling in the preheated steel mold. To study the microstructure evolution and properties of this TiAl alloy with high boron addition, an ingot of the TiAl matrix with the same dimensions was produced as well. Subsequently, both the TiAl matrix and composite ingots were hot-isostatically pressed (HIPed) at 1250 °C and 175 MPa for 4 h, followed by furnace cooling. Finally homogenizing annealing at 1320 °C for 1 h and 950 °C for 48 h was conducted.

The microstructures were characterized by scanning electron microscope (SEM), X-ray diffraction (XRD) and transmission electron microscope (TEM) techniques. XRD was carried out using Cu K α radiation ($\lambda = 0.154157 \text{ nm}$) and 2θ from 20° to 100°. The polished samples for SEM observation were deep etched by Kroll's solution. Thin TEM foils were prepared using standard procedures by argon ion beam thinner.

Flat tensile samples of 5 mm $\times$ 2 mm cross-section and 20 mm gage length for room temperature (RT) tests and 6 mm $\times$ 2 mm cross-section and 18 mm gage length for 800 °C tests were prepared, respectively. The whole tensile test process was conducted on Instron universal test machine, driven at crosshead speed of 0.5 mm min^{–1}.

3. Results and discussion

3.1. Microstructures of TiB₂/TiAl in situ composite

Fig. 1 shows the XRD patterns of the as-synthesized TiB₂/TiAl in situ composite and its matrix. The XRD results suggest that current TiB₂/TiAl composite mainly consists of γ , α_2 and TiB₂ phases, and TiB₂ phase has been synthesized completely during induction melting process. No other metastable borides were detected. By contrast, its TiAl matrix is composed of dominant γ and α_2 phases, as well as a little B2 phase.

Fig. 2 shows the microstructure of as-synthesized TiB₂/TiAl composite and its matrix. As shown in Fig. 2(a), the microstructure of composite matrix is characterized by randomly orientated fully lamellar colonies with mean grain size of approximately 90 μm and lamellar spacing about 250 nm. Meanwhile, ultra fine TiB₂ particles are observed uniformly distributing mainly near grain boundaries and a few inside lamellar colonies. Nearly no casting shrinkage

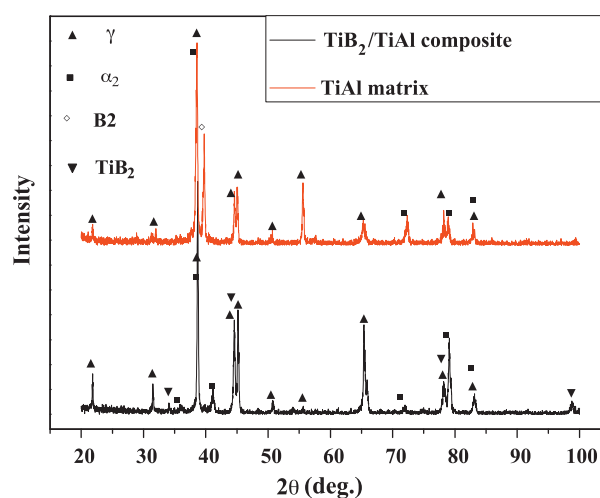


Fig. 1. XRD patterns of as-synthesized TiB₂/TiAl composite and matrix.

holes or severe clustering can be found, which is mainly ascribed to the strong electromagnetic stirring during induction heating and heat preservation process. There is no doubt that, electromagnetic stirring facilitates uniform dispersion of alloy elements and synthesized TiB₂ particles (primary TiB₂) in the melt. Compared to conventional XD + VAR or plasma arc melting route to fabricate in situ TiAl/TiB₂ composite ingot, ISM is a special XD process. The same is that boron fully dissolves into the melt and precipitates out as boride during solidification. What differentiates ISM most from the others is the separation of solidification from melting. In XD + VAR or plasma arc melting route both the melting and solidification occur in the same pool/crucible in a continuous manner, while in ISM the two steps can be controlled separately. That is, the melting proceeds in water cooled copper crucible, and the subsequent whole solidification process takes place in steel or ceramic mold after pouring. Therefore, ISM process exhibits excellent technical advantages over PM and arc melting methods in compactness, homogeneity and cost economy, to prepare TiAl in situ composites. Moreover, comparing to the coarse lamellar microstructure of the TiAl base shown in Fig. 2(b), both the lamellar colony size and lamellar spacing of the present TiB₂/TiAl composite are much finer and more uniform. As well known, coarse cast TiAl alloys can be refined effectively by boron addition [7,17,18]. With boron addition increasing, both the lamellar colonies and mean lamellar spacing become finer during solidification and are effectively stabilized during hot processing of TiAl alloys or composites. And the refining mechanism of boron is generally considered as heterogeneous nucleation and boundary pinning of primary β or α phase during solidification [17,18]. As to current TiAl + 3 vol% TiB₂ composite, primary TiB₂ precipitated first from melt during melting and solidification prior to β -Ti phase, followed by the eutectic reaction of β + second TiB₂ during solidification in the steel mold. Accordingly, the matrix microstructure was refined by boron addition through hindering β phase growing up and then restricting the coarsening of subsequent α phase.

3.2. Morphologies and growth mechanism of TiB₂ particles

Fig. 3(a) is a general view of morphologies of the synthesized TiB₂ crystals. Overall, the TiB₂ particles feature blocky, plate and dominant needle morphologies. The blocky particles reveal much larger diameter (1–4 μm) and shorter length than the needle. We can clearly see that the needle TiB₂ particles generally exhibit large aspect ratio up to 5, with diameter no more than 1.5 μm and length ranging from 5 μm to 8 μm . Comparing to the Ti–47Al–4(Cr, V,

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