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# Enhanced fracture toughness of TiB<sub>w</sub>/Ti<sub>3</sub>Al composites with a layered reinforcement distribution



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#### ABSTRACT

The prospect of combining laminated structure design and tailored reinforcement distribution to toughen brittle materials is investigated.  $TiB_w/Ti_3Al-Ti_3Al$  laminated composites consisting of alternating  $TiB_w/Ti_3Al$  and  $Ti_3Al$  layers were fabricated by reaction annealing of stacked, alternating foils of  $TiB_w/Ti$  and  $Ti_3Al$  layers were fabricated by reaction annealing of stacked, alternating foils of  $TiB_w/Ti$  and  $Ti_3Al$  layers were fabricated by reaction annealing of stacked, alternating foils of  $TiB_w/Ti$  and  $Ti_3Al$  layers were fabricated by reaction annealing of stacked, alternating foils of  $TiB_w/Ti$  and  $TiB_w/TiB_w/Ti$  and  $TiB_w/TiB_w/Ti$  and  $TiB_w/TiB_w$ 

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#### 1. Introduction

Titanium aluminides ( $\alpha_2$ -Ti<sub>3</sub>Al) and their composites are of engineering significance due to the potential for high temperature services [1,2]. However, the addition of reinforcements generally leads to poor ductility and fracture toughness of the composites [3]; in other words, high strength and high toughness are usually mutually exclusives in many engineering materials [4]. Great endeavors have been made to pursue the strength-toughness synergy [5-7]. Recently, Lu et al. [8,9] proposed that the overall properties (especially for toughness) of the composites can be enhanced by tailoring the distribution of reinforcements in a controlled way to form novel multi-scale hierarchical structures, compared with a conventionally homogeneous composite structure. This material design strategy has been successfully applied into metal matrix composites, where simultaneous improvements in the strength and toughness were found [10,11]. However, no particular attention is placed on the matrix of brittle Ti<sub>3</sub>Al intermetallic compounds, and the effect of the reinforcement distribution on the fracture toughness of brittle materials is still unclear. Because the mechanical properties depend strongly on the

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microstructure, we undertook to investigate the hypothesis that it is feasible to adjust the reinforcement distribution to achieve more optimal combinations of strength and toughness, and that one can reinforce a brittle matrix with a brittle phase to enhance toughness.

Among these multi-scale inhomogeneous reinforcement distributions, laminated structure, i.e., in form of layered reinforcement distribution, stands out because of the relatively simple preparation process and highly controlled architecture [12,13]. Easy fabrication of laminated composites can be processed by reaction bonding of metal foils, an approach that permits variations in the layer thickness and phase volume fractions through the selection of initial foil thickness [11,14].

The aim of this work is to explore the possibility of tailoring the reinforcement distribution to toughen brittle materials. The matrix is selected as brittle Ti<sub>3</sub>Al intermetallic compounds, and TiB whisker (TiB<sub>w</sub>) with a high length/diameter ratio is the reinforcement. The layered distribution of TiB<sub>w</sub> is achieved by reaction annealing of pure Al foils and TiB<sub>w</sub>/Ti foils, and the reaction process and synthesis mechanism of the composites are described in details. Three-point bending tests are performed to measure the fracture toughness of as-fabricated composites. The contribution of layered TiB<sub>w</sub> distribution on the fracture toughness is also discussed.

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#### 2. Experimental procedures

#### 2.1. Sample preparation

Commercial pure Al foils (99.7% purity,  $30 \times 50 \times 0.1 \text{ mm}^3$ ) and 3 vol% TiB<sub>w</sub>/Ti composite foils  $(30 \times 50 \times 0.4 \text{ mm}^3)$  were employed to prepare TiB<sub>w</sub>/Ti<sub>3</sub>Al-Ti<sub>3</sub>Al laminated composites. These foils were chemically etched in 10 wt% NaOH and 10 vol% HF solutions, respectively, water flushed, alcohol cleaned, and dried. Subsequently, the stacked laminates of alternating TiBw/Ti and Al foils were hot-pressed at 515 °C for 1.5 h under 40 MPa to obtain TiB<sub>w</sub>/ Ti-Al composites with well-bonded interfaces, and then annealed in a vacuum furnace as follows: (i) an initial annealing was carried out at 700 °C for 1 h to convert all elemental Al into TiAl<sub>3</sub>; (ii) in order to eliminate the Kirkendall voids formed in the initial annealing stage, a necessary densification process was conducted under 40 MPa at 1200 °C for 3 h, and finally, (iii) the composite was annealed at 1250 °C for 2 h to further reaction-diffusion and obtain the desired microstructure composed of alternating Ti<sub>3</sub>Al and TiB<sub>w</sub>/Ti<sub>3</sub>Al layers. The spatial coordinate system of the samples is represented by rolling (RD), normal (ND), and transverse (TD) directions.

#### 2.2. Microstructure characterization

Polished cross-sections were prepared after every annealing process and examined by scanning electron microscopy (SEM, FEI Quanta 200F) and X-ray diffraction (XRD, Philips X'Pert) for microstructure observation and phase identification, respectively. Three-point bending tests (sample dimensions:  $2\times4\times20~\text{mm}^3$  according to ASTM E1820 standard, with a notched depth of 1 mm parallel to the loading direction) were employed to study the crack propagation behavior at room temperature (RT). The fracture profile was also examined by optical microscopy (OM) using a sample etched by 5% HF + 5% HNO $_3$  + 90% H $_2$ O (vol.%) solution for 30 s.

#### 2.3. Finite element simulation

Two models of monolithic Ti<sub>3</sub>Al and TiB<sub>w</sub>/Ti<sub>3</sub>Al-Ti<sub>3</sub>Al laminated composites were developed to qualitatively reveal the effects of laminated structures on the stress distribution under flexural loading. Model geometries are set up similar to the experimental standard specifications of the ASTM E1820 [15], and performed only within the linear regime. The model, specimen geometric configurations, and material parameters were shown in

Supplementary materials. This 3D model consisted of several rectangular prisms stacked together to represent the different layers/components, and the thicknesses of both components refer to real imaging of the final microstructure. 3D solid elements were used in meshing and were arranged in an assembly that matches the material system's layout definition. Generally, cohesive elements were used to simulate damage behavior, however in our work, particular emphasis was placed on the elastic response. Therefore tie constraint, which is simply a constraint that keeps the model intact forever (not like cohesive elements that can split into two), is used. The boundary and loading conditions of the model were shown in Supplementary Fig. S3. The translation in z axis (axis coordinates shown in Supplementary Fig. S1) was fixed as  $T_3=0$ . The indenter pins were restrained to move only in Y direction. Frictionless contact condition was applied between the three indenter pins and the specimen body.

The aim of finite element simulation is to reveal the elastic response and stress state of monolithic Ti<sub>3</sub>Al and TiB<sub>w</sub>/Ti<sub>3</sub>Al-Ti<sub>3</sub>Al laminated composites under flexural loading, and we pay more attention on the *xx* normal stress, not von Mises stress, so that quantitative statistic of stress intensity is not strongly recommended. Also, tie constraint was chosen only for elastic deformation. In the future, combining quantitative stress/strain analysis, performing nonlinear simulation, and the choice of cohesive elements is suggested to provide a more comprehensive understanding in dynamic stress/strain evolution process.

#### 3. Results

#### 3.1. Microstructure evolution

Fig. 1 shows the microstructure evolution of TiB<sub>w</sub>/Ti–Al composites during reaction annealing. In the initial stage, the annealing was performed at 700 °C to melt the aluminum (melting point: 660 °C) and reduce the annealing period due to the higher diffusion rate of liquid Al compared with that of solid Al. After 1 h, the microstructure consisted of newly formed TiAl<sub>3</sub> layers (dark grey region, Fig. 1a) and residual TiB<sub>w</sub>/Ti layers (white region, Fig. 1(a)). The porous morphology and growth kinetics of TiAl<sub>3</sub> can be found in our previous work [16], and not given here. After this initial annealing, all the Al was transformed to TiAl<sub>3</sub>, and numerous voids with radius of a few microns were left in TiAl<sub>3</sub> layers owing to the Kirkendall effect [17,18]. These pores were eliminated by the subsequent densification process under 40 MPa at 1200 °C for 2 h, and then the laminate was composed of alternating layers of TiAl<sub>2</sub>

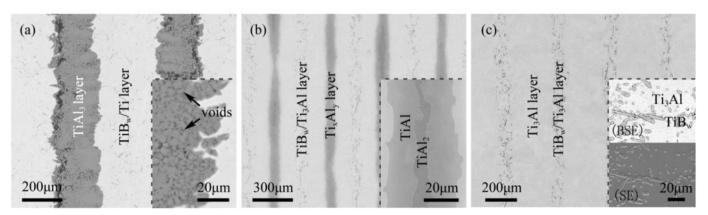


Fig. 1. Scanning electron microscopy (SEM) images showing typical microstructures of  $TiB_w/Ti$ -Al composites treated by (a) an initial annealing at 700 °C for 1 h; (b) a densification process under 40 MPa at 1200 °C for 3 h after (a); (c) a further reaction-diffusion annealing at 1250 °C for 2 h after (b). Inset indicates the morphology of  $TiAl_3$  (a), microstructure evolution (b), and the interface between  $TiB_w$  inclusions and  $Ti_3Al$  matrix (c). All images were captured by backscattered electrons (BSE), except for the bottom right one operated on secondary electron (SE) mode.

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