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Fabrication and properties of continuous unidirectional Mo fiber reinforced TiAl composites by slurry casting and vacuum hot pressing

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ABSTRACT

TiAl matrix composites reinforced by continuous fibers are potential structural materials for application in aerospace industry. However, the complicated fabrication procedures and high cost limit their applications. In this paper, slurry casting was proposed to simplify the fabrication procedures, and metallic Mo fibers instead of brittle ceramic fibers were applied to improve the room temperature brittleness and high temperature strength of the TiAl matrix, due to molybdenum's low price, good toughness and excellent high temperature mechanical properties. Firstly, slurries were prepared by mixing Ti and Al elemental powders with organic binder (polymethyl methacrylate) and solvent (acetone); and then a monolayer of preform was fabricated by coating a layer of aligned Mo fibers with a certain thickness of slurry; after reaching the expected number of layers the preform were cut and finally degassed and consolidated by vacuum hot pressing to produce composites. 20 vol.% Mo_f/TiAl composites with Mo fibers distributing uniformly in the matrix without bending were obtained. The matrix and fibers are well bonded through a reaction zone which consists of a thin δ-(Mo, Ti)$_3$Al inner layer and a thick β'-(Mo, Al)Ti outer layer. The composite consolidated at 1100 °C exhibits a good balance of room temperature and high temperature (800 °C) strength, 721 MPa and 731 MPa, respectively. The Mo fibers' plastic deformation, necking and debonding contribute to the toughening of the composites. And the composites are greatly strengthened at high temperature due to the good properties of Mo fibers.

1. Introduction

TiAl intermetallic compound alloy is a potential structural material for application in aerospace industry because of its high melting point, low density, good resistance to oxidation and creeping at elevated temperatures. However, TiAl suffers from low strength at elevated temperatures and poor toughness at room temperature [1]. A promising way to overcome these deficiencies is to reinforce TiAl by means of continuous ceramic fibers [2–10]. For many years intensive investigations have been carried out and mainly focused on fabrication methods, in order to balance the high cost and high quality production. Because of strong reaction between fiber and matrix at the melting point (~1465 °C) of TiAl, the fabrication route using infiltration of liquid metal has been avoided in order to protect the fiber from severe damage [11]. As a result, solid-state consolidation is finally the only viable manufacturing technique which allows minimal fiber damage. Foil–fiber–foil (FFF) route is extensively used for matrices with good ductility, but suffers from an inherent difficulty in fabricating the required TiAl matrix foils [12,13]. The common fabricating routes for TiAl matrix composites today include powder cloth (PC) [14,15], continuous binder-powder coating (CBPC) [13,16–19], vacuum plasma spraying (VPS) [20], physical vapor deposition (PVD) [21] such as magnetron sputtering (MS) [22,23] and electron-beam physical vapor deposition (EB-PVD) [24]. VPS and PVD allow better control of the coating thickness and fiber homogeneity, but the production rate is relatively slow and thus costly. VPS also has the problem of gas contamination (oxygen and nitrogen) and superficial fiber damage, which results from the impact of molten droplets. PC is a relatively low cost technique, but the incomplete elimination of binder and poor fiber distribution are still challenges. CBPC has been developed at University of London by Guo and Beeley [13,16,17] based on PC. It is an effective technique and has flexibility for non-planar components. Similarly, the key point of CBPC is to select a proper binder which is able to stick the matrix particles and fiber reinforcement together and can be decomposed at a low temperature without leaving any residuals. Polypropylene carbonate (PPC) [13], polystyrene (PS), polyoxyethylene (POE), polydimethylsiloxane (PDMS) [18] and polymethyl methacrylate (PMMA) [19,25] have been considered as candidate binders. PMMA is regarded as a more suitable one because it is easy to be obtained and it has a low decomposing

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temperature (under 380 °C), very low content of residuals (lower than 0.02%, mass fraction [25]) and no pollution to environment.

In respect of reinforcement selection, ceramic fibers, such as SiC [2–4,6,11] and Al_2O_3 [5,7], are considered as excellent reinforcement candidates to produce TiAl matrix composites, due to their excellent thermal stability, high specific modulus and strength both at room and at elevated temperatures (up to 1000 °C), regardless of their high price. However, despite the great potential to improve the matrix strength, brittle ceramic fibers damage easily during consolidation process and also have a limitation in enhancing the toughness of brittle matrix at room temperature. Therefore, attempts to apply other good fiber reinforcements require to be carried out.

Slurry casting using PMMA as binder, another alternative route based on CBPC, has been proposed in this work. Metallic molybdenum fibers were firstly used to reinforce TiAl with the purpose to improve the room temperature toughness and high temperature strength, because it has good toughness, high melting point, good microstructure stability and high strength at elevated temperature. Detailed fabricating route, including preparing preforms by slurry casting and subsequent consolidation by vacuum hot pressing, was described. Microstructure and mechanical strength of the obtained composites were also investigated.

2. Materials and experimental procedures

2.1. Materials

Elemental Ti powders (99.4% purity, <25 μm) and Al powders (99.9% purity, <10 μm) were employed as starting materials for matrix. TiAl matrix was obtained through reaction of the Ti and Al powders during consolidation. The reinforcement Mo fibers, fabricated from Mo(La) alloy by drawing, have an average diameter of 100 μm, a tensile strength of 2.9 GPa and a modulus of 320 GPa. The microstructure of the Mo fiber exhibits a fibrous characteristic along the axial direction and has a starting recrystallization temperature over 1400 °C, showing an excellent thermal stability [26,27]. PMMA and acetone were used as binder and solvent agent of the slurry respectively.

2.2. Fabrication of preforms

The slurry casting method employed to fabricate preforms for composites can be schematically defined by the following steps:

(1) A viscous fluid was produced by dissolving PMMA particles into acetone through magnetic stirring.
(2) A slurry was prepared by mixing the viscous fluid with Ti and Al powders (Ti:Al = 53:47, atom ratio) by magnetic stirring. The stirring time was over 4 h to guarantee the homogeneous dispersion of Ti and Al powders in the slurry.
(3) Preforms were fabricated by fiber winding and slurry casting: first, a layer of fiber with certain fiber spacing was winded on a flat rectangular aluminum plate by winding machine; then the monolayer of the aligned fibers was coated with a certain thickness of slurry; finally a monolayer of preform was formed after the slurry naturally dried and solidified. The fiber winding and slurry casting were performed alternately until it reached the designed number of layers.
(4) According to the graphite mould, preforms were cut off from the plate with a size of $35 \times 35\ mm^2$ by wire cutting and then cleaned with deionized water. After being dried, the preforms were well prepared for the following binder elimination and consolidation process.

The properties of the slurry, such as uniformity, viscosity, electric conductivity and strength after solidification, have strong effect on the quality of the preforms. They are heavily concerned with slurry composition, e.g., the ratio of PMMA, acetone and mixed powders. So slurries prepared with 10 g PMMA and different content of acetone (60, 70 and 80 ml) and powder mixture of Ti and Al (40, 60 and 80 g) were investigated.

As regards the reinforcement fraction, a composite with 20 vol.% fibers was designed. The condensation rate of the preforms during consolidating process was calculated to be 50%. Thus, a fiber spacing of 0.2 mm and a slurry thickness of 0.4 mm were controlled, and the prepared preforms had a fiber fraction of 10 vol.%.

2.3. Processes of degasing and consolidating

The as-received preforms were degassed and consolidated by vacuum hot pressing in a graphite mould. First, the PMMA binder in the preforms was decomposed and eliminated at 380 °C for 1 h, with a preload of 10 MPa and a vacuum of 10^{-2}Pa; then the degassed preforms were hot pressed at 700 °C for 30 min (aiming at promoting the reaction between Ti and Al) and finally consolidated at varied temperatures (1000, 1100, 1200 °C) for 2 h. A constant pressure of 35 MPa was used during this process. After consolidation the samples were cooled slowly inside the furnace. The size of the final composites with 18 layers of Mo fibers was $35 \times 35 \times 4\ mm^3$.

2.4. Microstructure analysis and mechanical properties tests

A transvers and longitudinal cross section of the preforms and composites were prepared using standard metallographic procedures. Microstructure was observed by scanning electron microscopy (SEM). Phase composition of the composites was determined by X-ray diffraction (XRD). Distribution of Ti and Al powders in the preforms and the composition of the reaction zone between the matrix and fibers were analyzed by energy dispersive X-ray spectrometer (EDX) equipped in the SEM. Flexural strengths were measured in air on an Instron-5569 universal testing machine through 3-point bending test with a span of 30 mm, both at room temperature and 800 °C. And a total of five samples were tested for each situation. The samples have a dimension of $3 \times 4 \times 35\ mm^3$ with the fibers parallel to the longitudinal direction. Fracture surface of the composites after bending test was observed by SEM.

3. Results

3.1. Evaluation of preforms

Coupling of the fibers and matrix materials were realized through fabrication of preforms before high temperature consolidation. Quality of the preforms had great influence on the properties of the consolidated composites. A qualified preform should have the following characteristics: no obvious macro-pores, uniform arrangement of Mo fibers, homogeneous distribution of Ti and Al powders, and enough strength to fix the Mo fibers when being cut off from the aluminum plate. The dimension of the materials (powders and fiber) used and the slurry properties had great effect on the compactness of the preforms. Tang et al. [28] have proposed a model which enables the fiber volume fraction of composite to be determined as a function of powder grain size for a given fiber diameter: the larger fiber fraction the preform has, the smaller the grain size is required. According to this model, for the given fiber diameter of 100 μm, a fiber fraction as high as