



Interfacial microstructure characterization and mechanical behavior of NiTi fiber reinforced Al₃Ti composite

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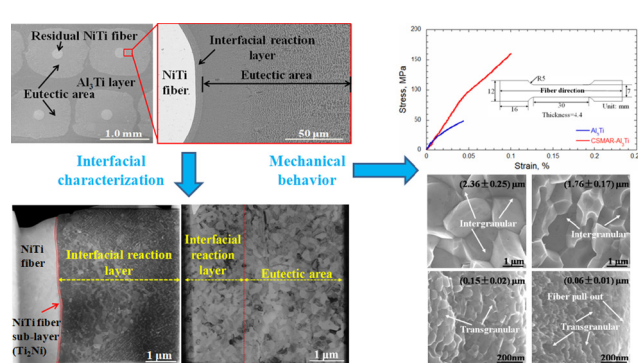
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HIGHLIGHTS

- Continuous shape memory alloy NiTi fiber reinforced Al₃Ti composite (CSMAR-Al₃Ti) is fabricated.
- Interfacial reaction layer of CSMAR-Al₃Ti composite shows a mixed structure of multi-phases.
- Crack propagation can be hampered or blunted by interfacial fine grain strengthening effect of CSMAR-Al₃Ti composite.
- The CSMAR-Al₃Ti composite can effectively improve the ductility of Al₃Ti alloy.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 12 November 2017
Received in revised form 23 January 2018
Accepted 30 January 2018
Available online 01 February 2018

Keywords:

Al₃Ti alloy
NiTi fiber
Interfacial characterization
Mechanical behavior

ABSTRACT

To improve the ductility of Al₃Ti alloy, the continuous shape memory alloy NiTi fiber (CSMAR) was introduced into intermetallic Al₃Ti matrix for fabricating the novel CSMAR-Al₃Ti composite in this work. Microstructure characterizations demonstrated that the CSMAR-Al₃Ti composite mainly consists of Al₃Ti layer, NiTi fiber, eutectic area and interfacial reaction layer. EBSD results indicated that the eutectic area is made up of Al₃Ti and Al₃Ni phases, the Al₃Ti phase shows a strong [001] crystallographic oriented structure, while the Al₃Ni phase has a non-textured structure. TEM results showed that the interfacial reaction layer between NiTi fiber and eutectic area is a multiple phase mixture, including various Ti-Al and Ni-Al intermetallics. Furthermore, TEM and HRTEM analyses revealed a newly formed Ti₂Ni layer between NiTi fiber and interfacial reaction layer. Tensile test results confirmed that the CSMAR-Al₃Ti composite could effectively improve the ductility of the Al₃Ti alloy. Based on the systematic investigations of interfacial microstructure characterization, mechanical behavior and fracture morphology observation, it is found that the toughening mechanism of CSMAR-Al₃Ti composite is related to the interfacial fine grain strengthening effect with the gradual distribution characteristic. In addition, the excellent metallurgical bonding between fiber reinforcement and matrix is also beneficial to the mechanical properties.

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1. Introduction

Among the Ti-Al compound system, intermetallic Al₃Ti alloy exhibits the highest elastic modulus (~216 GPa), lowest density (~3.36 g/cm³), and superior oxidation resistance compared to other intermetallic alloys, TiAl and Ti₃Al [1]. However, due to its stable tetragonal D0₂₂

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crystalline structure, the necessary dislocation slip for plastic deformation is inhibited [2,3]. Therefore, Al_3Ti alloy shows high brittleness and poor plastic deformation capacity at room temperature, which severely limits its engineering applications [3].

In the recent decades, compositing method has been successfully employed to improve the ductility of Al_3Ti alloy via introducing some reinforcements in the brittle Al_3Ti matrix, such as particles [4], fibers [5,6], ductile phase [7] and ductile layers [8,9]. For example, our recent work indicated that the ductility of Al_3Ti alloy could be improved effectively by introducing ductile Al phase in the brittle Al_3Ti matrix [7]. Due to the ductile Al phase uniformly distributed around the Al_3Ti grains, the inherent brittle deformation behavior of Al_3Ti alloy was converted into the mixture of brittle deformation behavior and plastic deformation behavior. Furthermore, some studies demonstrated that the metal-intermetallic laminate (MIL) composite $\text{Ti}/\text{Al}_3\text{Ti}$, a novel laminate structure composite, could effectively improve the ductility of Al_3Ti alloy [8,10]. Owing to its unique laminated structure, the crack bridging, crack blunting and crack deflection occurred during deformation [8,11]. These toughening mechanisms could effectively decrease local stress, reduce nucleate energy of micro-crack and decrease stress concentration [9,11,12], which were beneficial to improve the ductility of Al_3Ti alloy.

Based on the compositing method, owing to its unique shape memory effect and excellent super-elastic deformation capacity, the shape memory alloy (SMA) has been widely used as the reinforcements in the aluminum matrix composite [13,14], magnesium matrix composite [15,16] and metal-intermetallic laminate (MIL) composite [17,18]. Due to the back stress strengthening effect and compressive stress strengthening effect created by the NiTi fiber reinforcement, Furuya et al. [19] indicated that the mechanical properties of Al could be improved effectively by the NiTi fiber reinforced aluminum matrix composite. Furthermore, Wang et al. [18] confirmed that the good metallurgical bonding between NiTi fiber reinforcement and intermetallic matrix in the MIL composite and the super-elastic deformation capacity of NiTi fiber were key factors for improving the mechanical properties of MIL composite $\text{Ti}/\text{Al}_3\text{Ti}$ by NiTi fiber reinforcement.

It is well known that the interface between NiTi fiber reinforcement and metal matrix usually plays a critical role on the mechanical behavior of the shape memory alloy (SMA) composites [20]. Therefore, the interfacial microstructure characterization is very important for understanding or controlling the mechanical behavior of the SMA composites. Liu et al. [21] investigated the interface between NiTi fiber reinforcement and 6061Al matrix of the NiTi/6061Al composite. The TEM experimental results indicated that three layers formed at the interface between NiTi fiber and Al matrix, including Al_3Ti layer, Al_9FeNi layer and Mg-O layer. Al_3Ti layer showed some ductility, while the Mg-O layer weakened the interfacial bonding between NiTi fiber and Al matrix because of some discontinuous voids in the Mg-O layer. Coughlin et al. [22] investigated the interface reaction and mechanical behavior of the NiTi fiber reinforced Sn matrix composite. Experimental results indicated that two layers (the layer of the solid solution of Sn-Ni-Ti ternary and the layer of the Sn_3Ti_2 intermetallics) formed between NiTi fiber and Sn matrix. Modulus and hardness of the Sn_3Ti_2 phase were both higher than that of the Sn-Ti-Ni phase. Dong et al. [23,24] fabricated the NiTi/2024Al composite by spark plasma sintering (SPS) method. TEM results indicated that the interface between NiTi fiber and 2024Al matrix consists of a bi-layer structure (layer I and layer II). The layer I was a multiple phase mixture, including Al_3Ti , Al_3Ni , TiO_2 , B_{19}NiTi and $\text{B}_{2\text{NiTi}}$ phases. The layer II also exhibited a multiple phase mixture, including Al_3Ti and Al_3Ni phases. Furthermore, HRTEM results indicated that the TiO_2 was primarily coherent with the Al_3Ti and semi-coherent with the Al_3Ni , and the discontinuous TiO_2 phase could change the fracture crack direction in the interfacial layer, which was beneficial to the mechanical property improvement of the NiTi/2024Al composite. However, to the best of our knowledge, owing to the difficulty of the TEM foil preparation, very few of TEM works have been done to investigate

the interfacial microstructure of the NiTi fiber reinforced brittle inter-metallic matrix composite so far.

The aim of this work is not only to seek a new toughening method for improving the ductility of the Al_3Ti alloy via introducing continuous shape memory alloy NiTi fiber in the Al_3Ti matrix, but also to investigate the interfacial microstructure of the CSMAR- Al_3Ti composite by TEM and HRTEM experiments. To this end, the CSMAR- Al_3Ti composite was fabricated using vacuum hot pressing method. The microstructure observation was performed by scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). The interfacial microstructure characterization between NiTi fiber and eutectic area was conducted using transmission electron microscope (TEM) and high resolution transmission electron microscope (HRTEM). The mechanical behaviors of CSMAR- Al_3Ti composite were carried out by tensile test and nano-indentation test. Finally, the toughening mechanism of CSMAR- Al_3Ti composite was also discussed in this work.

2. Experimental procedure

2.1. Materials fabrication

The CSMAR- Al_3Ti composite was fabricated via metal foil metallurgical technique using vacuum hot pressing furnace (ZRY-10-40). Commercial pure aluminum foils (100 mm × 100 mm × 1.2 mm), titanium alloy (Ti-6Al-4V) foils (100 mm × 100 mm × 0.3 mm and 100 mm × 100 mm × 0.1 mm, respectively) and continuous NiTi fibers ($\Phi = 1.0$ mm) were used as starting materials. Due to the precipitate strengthening effect caused by the vanadium element of Ti-6Al-4V foils in Al_3Ti alloy in our previous work [7], the titanium alloy (Ti-6Al-4V) foil is used in this work as Ti element origination rather than pure titanium foil, and Ti foil refers to titanium alloy (Ti-6Al-4V) foil throughout this paper.

All the starting materials were polished by 180 grit silicon carbide paper and then cleaned using scouring pad under flowing water and followed by ultrasonic cleaning. After drying, the starting foils and fibers were stacked together in the order “Ti-Al-NiTi-Al-Ti” with Ti foils covered on the top and bottom, which was defined as one “unit”, and two “units” were orderly placed together (as shown in Fig. 1a). Next, the stack was used to fabricate the CSMAR- Al_3Ti composite in the vacuum hot pressing furnace (as shown in Fig. 1b). Detailed processing parameters can be seen in Fig. 1c. For comparison, the Al_3Ti alloy was also fabricated using the similar fabrication process, detailed parameters can be seen in our previous work [7].

2.2. Microstructure characterization

Microstructure observation of CSMAR- Al_3Ti composite was conducted on a Hitachi S-3400 scanning electron microscopy (SEM) with electron back-scattered diffraction (EBSD) apparatus. Interfacial microstructure characterization was performed by TEM and HRTEM using FEI Talos F100× field emission transmission electron microscopy (FE-TEM) coupled with energy dispersive spectroscopy (EDS) at an acceleration voltage of 200 kV. TEM foils were prepared using FEI Helios 600i focused ion beam/scanning electron microscopy (FIB/SEM) system and the lift-out technique.

2.3. Mechanical testing

Tensile tests of CSMAR- Al_3Ti composite and Al_3Ti alloy were carried out on an Instron 5500R servo-hydraulic load frame at the strain rate of 0.001/s under room temperature. The tensile strain was recorded by an extensometer and data acquisition system, and the tensile sample information was provided in Fig. 9a. Nano-indentation test of CSMAR- Al_3Ti composite was carried out on an Agilent Nano Indenter G200 with the depth limit of 500 nm at room temperature. The modules and hardness were achieved from the average value at the range of 350 nm–400 nm

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