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Configuration design and fabrication of laminated titanium matrix composites



Hongqiang Duan, Yuanfei Han *, Weijie Lu *, Liqiang Wang, Jianwei Mao, Di Zhang

State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200240, China

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ABSTRACT

The introduction of particulate reinforcements into titanium alloys can improve the strength and stiffness, but it generally results in unsatisfactory plasticity and ductility. In this study, in situ synthesized multilayer titanium matrix composites were designed and fabricated by learning from the microstructure of nature biological materials with attractive mechanical properties. Laminated Ti-(TiB + La₂O₃)/Ti composites with different reinforcement volume fraction were fabricated through powder metallurgy, followed by hot rolling process. The results show that pore defects, TiB whisker and large La₂O₃ particle agglomerates can be observed in the composites after vacuum sintering at 1573 K, further the pores are disappeared and the grains are refined significantly after hot rolling direction. The tensile testing results indicate that the Ti-10 vol.% (TiB + La₂O₃)/Ti composite shows promising mechanical properties due to the configuration design of laminated structure, which results in an improvement of 30% in elongation with <4% decline in tensile strength compared with 5vol.% (TiB + La₂O₃)/Ti composites. Moreover, further increase in reinforcement volume fraction leads to slight increase in tensile strength and significant decrease in elongation due to the increasing of large La₂O₃ agglomerates.

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1. Introduction

Titanium matrix composites (TMCs) have drawn considerable interest in aerospace, automotive, chemical, biochemical and other advanced structural applications due to excellent mechanical properties, such as the lightweight, high strength, high stiffness, excellent wear resistance and high temperature durability [1,2]. The in situ synthesis method is considered as a cost-saving fabricating process to obtain TMCs with isotropic properties and good interfacial integrity [3,4]. Powder metallurgy (PM) [5,6,7], mechanical alloying (MA) [8,9], self-propagating high temperature synthesis (SHS) [10] and rapid solid-ification processing [11] have been used to in situ fabricate TMCs. Previous studies have found that the in situ introduced reinforcements can significantly improve the tensile strength, but the poor ductility and toughness was unavoidable [12,13,14], which seriously limits their applications.

Inspired from nacre structure in nature, Clegg [15] firstly proposed a method of preparing tough ceramics by coating silicon carbide sheets with graphite, so as to introduce weak interfaces and thus reduce brittleness of ceramics, which soon becomes a hot topic in ceramic. For metals, grain refinement is one of the best methods to improve the strength without decreasing ductility, but it loses validity when the grain sizes fall below ~1 μ m [16]. Lu [17] proposed that multiscale hierarchical structures provide a possible route to optimizing overall properties on report published in Science. Huang [18] summarized the

* Corresponding authors. E-mail addresses: hyuf1@hotmail.com (Y. Han), luweijie@sjtu.edu.cn (W. Lu). classification of inhomogeneous phases and composites with tailored inhomogeneous microstructures, the previous research of TMCs with 3D continuous reinforcement-rich network and laminated structure showed promising mechanical properties compared with uniformly reinforced composites. Pandey [19] described an architectural approach for toughening discontinuously reinforced aluminum (DRA) alloys. The laminated composite consisted of alternate layers of a 7093/SiC/ 15p DRA and an unreinforced aluminum-manganese alloy. Fracture toughness showed an improvement of 79% in an under-aged condition and an improvement of 53% in the peak-aged condition compared to the monolithic DRA. Jiang [20] developed a flake powder metallurgy method to fabricate biomimetic nano-laminated CNT/Al composites, which performed well-balanced strength and ductility (435 MPa, 6%) compared with materials fabricated by conventional methods. However, a much simpler method should be developed to design and fabricate titanium because of its sensitivity to oxygen and the demand of cost saving. Liu [21,22] fabricated the laminated Ti-TiB_w/Ti composites composed of Ti layers and TiB whisker reinforced Ti composite layers by reaction hot pressing and diffusion welding. Both the two laminated composites showed an improvement in fracture ductility. Ti-Al₃Ti metallicintermetallic laminate (MIL) composites were fabricated through reactive sintering using Ti and Al foils [23,24]. Crack bridging and crack deflections by Ti layers were found to be primarily responsible for enhancing fracture toughness. Smith [25] prepared a kind of laminated composite composed of monolithic titanium alloy IMI 834 and Ti alloy/ Ti-SiC_f by diffusion bonding. The effective Young's moduli of the



Fig. 1. Schematic of laminated Ti-(TiB + La₂O₃)/Ti composites.

laminated composites were found to be 1.2–1.8 times greater than pure IMI 834. Lots of studies had been carried out in the mechanical properties of laminated composites fabricated by sintering or diffusion bonding, but it would always result in unsatisfactory performance due to the large grains or weak interface bonding. Thus, further processing should be introduced to enhance the strength and ductility of laminated TMCs.

In this study, vacuum sintering was carried out to prepare the laminated Ti-(TiB + La₂O₃)/Ti composite, followed by hot rolling to optimize the microstructure, which included the layer thickness, relative density, grain size and reinforcements distribution. Lanthanum was introduced with the original purpose of reducing the oxygen content by forming La₂O₃ reinforcements. The microstructure and mechanical behaviors of the laminated Ti-(TiB + La₂O₃)/Ti composites were investigated and compared with homogenous (TiB + La₂O₃)/Ti composite. Moreover, the fracture mechanism and enhancing effect of laminated configuration design were analyzed in details.

2. Materials and methods

Fig. 1 shows the schematic of laminated Ti-(TiB + La₂O₃)/Ti composites. Pure Ti powder (maximum particle size 150 μ m) and LaB₆ powder (maximum particle size 75 μ m) were used in the experiment. The mixing powder of pure Ti and LaB₆ in various proportions was prepared by low energy ball-milling with a speed of 150 rpm for 15 min according to the following reaction equation (Eq. (1)):

$$12Ti + 2LaB_6 + 3[0] = 12TiB + La_2O_3.$$
(1)

The slow ball-milling speed and time were selected to mix powder uniformly and avoid being oxidized, which were important to control the impurity level and obtain homogenous laminated composites.

Four steps were designed in the experiment: (1) Laying powder. The as-prepared mixing powder and pure Ti powder were stacked alternately in a special steel mold with a certain thickness based on theoretical calculation. The powder weight of each layer and smooth level between different layers should be controlled accurately to introduce homogenous laminated structure. Different layer thickness and thickness ratios can be designed freely to obtain various mechanical properties. (2) Cold pressing. The multilayer laminated preform $(\Phi 32.1 \times 30 \text{ mm})$ with relative density of 85% was prepared by cold pressing under 700 MPa pressure for two minutes. High relative density of the laminated preform contributes to the subsequent densification behavior during vacuum sintering. (3) Vacuum sintering. High temperature sintering was carried out in a vacuum molybdenum-wound furnace (ZT-25-16) at 1573 K for 1 h, the heating rate was 10 K/min and the vacuum degree was 4×10^{-3} Pa. The consolidated laminated $Ti-(TiB + La_2O_3)/Ti$ composites were taken out of the furnace, air cooled to room temperature. Further optimal process to reduce the layer thickness, grain size and pore defects was still required to improve the mechanical properties although the relative density of the as sintered laminated composite was over 95%. (4) Hot rolling. The as sintered composites were preheated in a heat treatment furnace at 1323 K for 30 min in order to implement the α phase to β phase transformation because the β phase with bcc structure could be rolled easier at high temperature. The rolling process with a total deformation of 80% parallel to the layer direction was carried out to refine the grain size and reduce the pore defects. Subsequently, the deformed composites were annealed at 873 K for 30 min to relieve residual stress. The relative density of the laminated composites were successfully improved to 100% after hot rolling.

The samples cut from the sintered and rolled laminated composites were characterized respectively by optical microscopy (OM), field emission scanning electron microscopy (FEI Quanta-250 FESEM) and energy dispersive spectrometer (EDS). The tensile test samples were machined from the rolled laminated composites with the tensile axis parallel to the rolling direction. The gauge length of the samples was 10 mm and



Fig. 2. Metallographic photos of the as-sintered composites at low magnification, (a) (TiB + La₂O₃)/Ti composite, (b) Ti-10 vol.% (TiB + La₂O₃)/Ti composite, (c) Ti-15 vol.% (TiB + La₂O₃)/Ti composite.

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