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Selective laser melting of in situ titanium–titanium boride composites: Processing, microstructure and mechanical properties

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Abstract

This study presents results of selective laser melting (SLM) processing of in situ Ti–TiB composites from optimally milled Ti–TiB_2 powder. Optimized tuning of the SLM manufacturing parameters was applied to obtain almost fully dense (>99.5%) Ti–TiB composites. X-ray diffraction and electron diffraction patterns as well as microstructural investigations indicate a chemical reaction during SLM in which irregular-shape titanium diboride (TiB_2) particles react with pure Ti to form needle-shape titanium monoboride (TiB) particles. Transmission electron microscopy investigations reveal that Ti grains are refined significantly due to the existence of B. The microhardness, yield stress and compressive strength of the SLM-produced Ti–TiB composites increase to 402 Hv, 1103 MPa and 1421 MPa, respectively, compared to 261 Hv, 560 MPa and 1136 MPa, respectively, for the SLM-produced commercially pure Ti. These improvements are mainly due to strengthening and hardening effects induced by TiB particles and refinement of Ti grains. Fractography analyses show that a mixture of splitting/shearing and smooth/rough zones covers the fracture surfaces of failed composite samples after compression testing.

Keywords: Selective laser melting; Ti–TiB composite; Microstructure; Mechanical properties; Fractography

1. Introduction

Titanium and its alloys are widely used in the chemical, aeronautical and biomedical industries due to their excellent corrosion resistance, low density, appropriate mechanical properties and high biocompatibility [1,2]. However, Ti-based alloys exhibit relatively poor wear resistance and low hardness, which restricts their fields of application. The addition of ceramic reinforcements can effectively improve wear resistance, specific strength and high-temperature durability of pure Ti [3–5]. Several reinforcements such as SiC [6], WC [7], TiC [8] and TiB_2 [9] are used to develop Ti-based matrix composites. Nevertheless, more beneficial properties can be achieved by incorporating titanium monoboride (TiB) particles to produce Ti–TiB composites. Firstly, boron is biocompatible [10], thus rendering Ti–TiB composites potential candidates for medical applications [4,11]. Secondly, there is only a small density difference between Ti (4.51 g cm^{-3}) and TiB (4.54 g cm^{-3}) and, in addition, already a relatively small amount of TiB reinforcement is sufficient to increase both stiffness and strength of the composites compared to other Ti compounds [12]. Thirdly, TiB exhibits good chemical stability at high temperatures as well as good thermodynamic stability, providing non-reactive matrix/

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reinforcement interfaces. The similar thermal expansion coefficients (CTEs) of TiB and the Ti matrix (7.2×10^{-6} °C^{-1} for TiB and 8.2×10^{-6} °C^{-1} for Ti matrix [13]) reduce deleterious incompatibilities that can promote residual stresses in brittle reaction zones [14].

TiB particles can be produced by an in situ reaction of Ti with titanium diboride (TiB_2), which leads to the formation of good interfacial bonding between the Ti matrix and the TiB reinforcements [15]. The great advantages of in situ reactions for producing in situ metal matrix composites compared to ex situ methods have been reported in Ref. [16]. Various common technologies such as casting [17], powder metallurgy [18] and combustion synthesis [19] have been applied to manufacture Ti-based matrix composites. Generally, traditional manufacturing techniques for producing Ti-based materials involve highly time-, energy- and material-consuming processing steps. Furthermore, Ti is strongly reactive, making the manufacturing processes tedious. Therefore, emerging advanced technologies such as selective laser melting (SLM), which allows the generation of complex-shape parts in a highly efficient way without any additional steps, possess great potential to simplify and speed up the manufacturing process.

SLM is a developed additive manufacturing (AM) technique which has attracted increasing interest throughout the last five 5 years as an AM process. This advanced technique is able to produce near net-shape structures by selectively melting/consolidating successive layers of powders under a protective atmosphere, using a computer-controlled laser beam [20]. In the SLM process, a high intensity laser beam selectively scans across a powder bed, thereby selectively melting the irradiated particles, which solidify to form a solid layer. A new layer of powder is deposited on top of the previously formed solid layer and the cycle continues until the part is complete [21,22].

Only a limited number of investigations on SLM processing of Ti-based composites have been carried out so far [23,24], in which Ti–TiC composites with relative densities up to 98.5% were processed. Strengthening through reinforcements, stimulation of nucleation, refinement of the microstructure and processing almost fully dense parts are very important factors for the improvement of mechanical properties. Therefore, it is essential to investigate the starting powders, their optimal preparation, the SLM manufacturing parameters and the metallurgical reactions involved in order to produce ceramic-reinforced Ti materials of high relative density (> 99.5%).

It has been recently reported that an optimum combination of fracture toughness and bending strength in Ti–TiB composites was achieved when only 5 wt.% of TiB_2 powders was used and further increase of the weight per cent over 5% showed negative effects [13]. In this work, SLM-processing of in situ Ti–TiB composites starting from Ti–5 wt.% TiB_2 powders was studied for the first time. At first, in order to reach the highest possible density, preparation of the starting powders and SLM manufacturing parameters were optimized. Afterwards, the obtained microstructures, phase constitutions, microhardness and compressive strengths were investigated as the objectives of this work. This study reports that SLM is able to produce almost fully dense (>99.5%) in situ Ti-based composites, containing a uniform distribution of reinforcing TiB particles within the Ti matrix.

2. Experimental

2.1. Starting powders and SLM processing

Commercially pure Ti (CP-Ti) powder (99.7% purity, $d_{50} = 48.69$ μm) and TiB_2 powder (98.9% purity, $d_{50} = 3.5$–6 μm) were used in this study. The powders were mixed under protective argon atmosphere to obtain a Ti–5 wt.% TiB_2 mixture and poured into ball-mill vials. C15 carbon steel balls with a diameter of 10 mm were used and the ball-to-powder weight ratio was set to 5:1. Afterwards, the powder mixture was ball-milled using a Retsch planetary ball mill for 1–4 h at a fixed rotation speed of 200 rpm. A MTT SLM 250 HL machine containing a 400 W Yb:YAG fiber laser with an 80 μm spot size was used to produce several cylindrical parts under protective high-purity argon atmosphere. The samples were manufactured with various laser powers of 165–185 W, laser scanning speeds of 118–154 mm s^{-1} and fixed layer thickness and constant hatching distance of 100 μm. The layers were scanned using a continuous laser mode according to a zig-zag pattern, which was alternated 90° between each layer.

2.2. Phase identification and microstructural characterization

Phase characterization of the starting Ti–5 wt.% TiB_2 powder and SLM-processed samples was carried out with X-ray diffraction (XRD) using an X'Pert PRO MPD device with Co K_α radiation ($\lambda = 0.1790$ nm) at 40 kV and 40 mA, using a continuous scan mode over a wide range of $2\theta = 30$–$100°$. The morphology of the starting powders and the Ti–5 wt.% TiB_2 powder milled for different times was studied using a Zeiss 1555 scanning electron microscope (SEM). The density of the SLM-processed samples was measured by the Archimedes method. Moreover, X-ray computed tomography (CT) was applied using a GE nanotom device to ensure that almost fully dense samples were free of any major defects. To study the microstructure, the SLM-processed samples were sectioned both in transverse (*X–Y*) and in longitudinal (*X–Z*) directions, then ground and polished, using standard metallographic procedures. Afterwards, the samples were etched using a solution containing 10% HF, 5% HNO_3 and 85% distilled water (volume fractions) and investigated using an Olympus PMG 3 optical microscope (OM), a Zeiss 1555 SEM and a Tecnai T20 transmission electron microscope (TEM) operated at 200 kV.