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In-situ TiB/near α Ti matrix composites manufactured by selective laser melting

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ABSTRACT

TiB reinforced near α Ti-matrix composite was fabricated in this work using selective laser melting from a mixture of CrB_2 and commercially pure Ti powders. The corresponding composites present an almost fully dense structure for suitable laser energy density conditions. The X-ray diffraction and microstructure analysis indicate that the TiB and β-Ti phase appears for parts obtained with a low scanning speed of the laser beam. The parts obtained at high and low scanning speeds show higher hardness and lower wear rate than those obtained for intermediate scanning speed which, on the contrary, show the highest density. The wear behavior of the as-processed parts is compared with that of pure Ti parts also obtained by selective laser melting.

1. Introduction

Thanks to its high strength, low density, high biocompatibility and high corrosion resistance, titanium (i.e. commercially pure Ti) is widely used for a great variety of applications, for example, aerospace, automobile and bio-technology [1,2]. However, the limits of wear resistance and high-temperature mechanical properties of CP-Ti are holding back many potential structural applications. Titanium matrix composites (TMCs) present a high wear resistance while keeping their mechanical properties. Ceramics, such as TiC [3], TiB_x [4], and SiC [5], were proposed as reinforcement in TMCs, due to their high hardness. Particularly, boride compounds enjoy high popularity given their low density, good chemical stability and good bonding between ceramic particles and titanium matrix [6]. However, the bond strength is relative low, because non metallurgical bonding was formed. Solid state reactions have been proposed to fabricate in-situ reinforced TMCs with high interfacial bond strength. In the past decades, ingot metallurgy [7], mechanical alloying [8], spark plasma sintering [4], powder metallurgy [9], and additive manufacturing [10,11] were all considered to produce TMCs. Compared with other methods, selective laser melting (SLM) is attracting increasing interest due to its high manufacturing flexibility and the production of fine microstructures [12]. In this process a complex part can be designed using Computer-Aided Design (CAD) software; after that, the part will be built layer-by-layer with a computer controlled laser beam which will selectively melt each powder layer at a given Laser Scanning Speed (LSS). Compared to conventional casting alloys, SLM processed alloys exhibit different wear [13] and corrosion [14] behaviors, due to the high heating/cooling rate. Besides the classical alloys already used with SLM, such as, Al [15], Ni [16], Fe [17] and Ti [18] base alloys, a number of Metal Matrix Composites have also been processed, such as AlSi10Mg + TiC [19], Fe + SiC [20] and Ti + TiB [4]. Attar et al. investigated systematically the influence of the parameters [21] and powder particle shape [22] on mechanical properties of SLM processed Ti matrix composites. The mechanical properties could be improved by adding ceramic reinforcement.

Owing to its high corrosion and wear resistance, CrB_2 has already been used as particle reinforcement for several common metals, for instance, Al [23] and Fe [24]. However, so far no works seem to have considered chromium boride particles to reinforce titanium and its alloys. Additionally, chromium boride has a good chemical stability, but the possibility of *in-situ* reaction between Ti and chromium boride is evidenced by the negative reaction enthalpy between Ti and CrB_2. This *in-situ* reaction between Ti and CrB_2 would thus ensure a good interfacial bonding between the Ti matrix and the CrB_2 particles. Chromium (Cr) is also a strong β phase stabilizer for Ti, thus beneficial to form the (α + β) two phase

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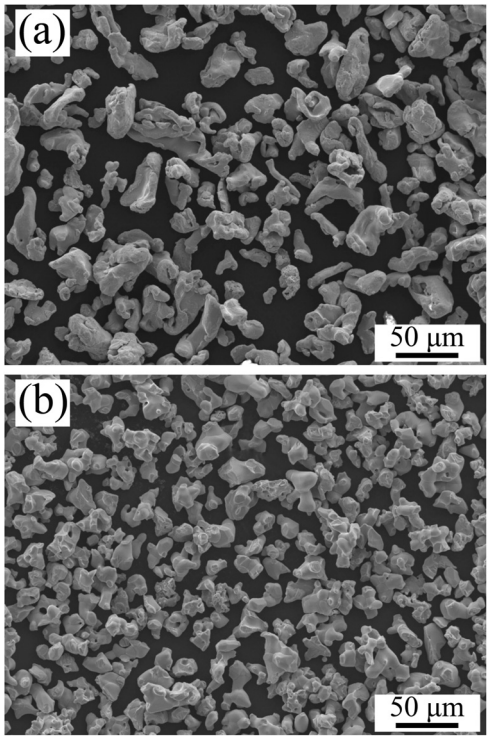

Fig. 1. Morphology of powders (a) Ti and (b) CrB2.

structure, which presents the potential application as biomaterial [25].

In this study, TiB/near α-Ti matrix composites were produced by SLM from a Ti and CrB_2 powders mixture, with several LSSs under an inert argon gas environment. The relationship between densification, microstructure and microhardness was then investigated with a focus on the interfacial region between the CrB_2 particles and the Ti matrix. The ball-on-disk wear test was applied to determine the wear/friction behavior of the obtained TMC.

2. Method

Commercially pure Ti (−81 + 25 μm) (Sulzer Metco Technologies Co. Ltd., Switzerland) and CrB_2 (−38 + 11 μm) (Metachim, Co. Ltd., Russia) powders were used in this study (as seen in Fig.1).

Those two powders with weight proportion CrB_2: Ti = 1:49 were blended in a tumbling mixer for 60 min and dried at 80 °C for 4 h before being used. Samples were fabricated using an MCP-realizer SLM 250 (MCP-HEK Tooling GmbH, Germany) equipped with a Nd-YAG fiber laser with a spot size of 40 μm and maximum power of 400 W. Small cubes (5 × 5 × 5 mm) were manufactured using 175 W laser power, a 50 μm layer thickness and LSSs ranging from 1000 mm s^{-1} to 2000 mm s^{-1} with Zigzag scanning mode. The operation was performed under a high-purity argon atmosphere containing less than 200 ppm oxygen (as shown in Fig. 2).

The density of the as-fabricated samples was determined using the Archimedes principle with 5 measurements made at ambient conditions. The microstructure of the specimens was observed using optical microscopy (OM) (Nikon, Japan) and scanning electron microscopy (SEM) (JEOL JSM 7800F, Japan equipped with X-ray energy dispersive spectroscopy EDS). X-ray Diffraction (XRD) was performed with a Cobalt anticathode (λ = 1.78897 = Å) operated at 35 kV and 40 mA. The polished samples were etched by a solution consisting of 10 ml HF, 5 ml HNO_3 and 85 ml distilled water for 5 s. Microhardness was measured on polished samples with a Vickers pyramid and a Leitz-Wetzlar (Germany) instrument. The

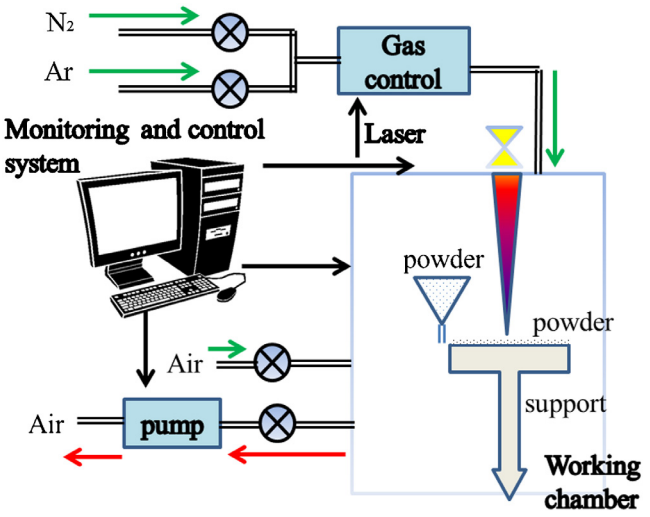

Fig. 2. Schematics of the selective laser melting apparatus.

2.94 N load was applied with a dwell time of 25 s. The values given in the following section correspond to the mean value of a set of 10 measurements. Ball-on-disk wear tests were operated with a CSM-TRIBOMETER instrument (Switzerland) under ambient environment (temperature: 21 °C, humidity: 51%) on surfaces with roughness Ra below 0.02 μm under unlubricated conditions. The counterpart was a 3 mm diameter alumina ball with a mirror finished surface. The rotation diameter, normal load and sliding velocity were 3 mm, 2 N and 15 mm s^{-1}, respectively. The cross-sectional area of the worn tracks was obtained with an Altisurf 500 profilometer. The average values were calculated from 8 random measurements and the morphology and composition of the worn traces were inspected by SEM and EDS respectively.

3. Results and discussions

Fig. 3 shows the XRD spectra of the Ti/CrB_2 composites fabricated by SLM for 1000 mm s^{-1}, 1500 mm s^{-1} and 2000 mm s^{-1} LSS. The patterns for the parts obtained at intermediate and high LSS reveal an identical α/α' Ti (hexagonal close-packed, *hcp*) structure. Probably due to the low CrB_2 content, no obvious peak related to Cr and/or B compounds were detected by XRD. Attar et al. [26] reported similar results in the case of SLM processed CP-Ti. However, as the LSS decreases to 1000 mm s^{-1}, the major β-Ti (body-centered cubic, *bbc*) phase peak appears with a small intensity; this peak disappears when the part is heat treated at 650 °C for 6 h, thus confirming its identification. This seems to be evidence of the diffusion of some Cr in the Ti matrix, Cr being, as said before, a strong β stabilizer. Given the elemental proportions, the reaction between pure Ti and CrB_2 can be considered as two steps: (1) $CrB_2 + Ti \rightarrow TiB_2 + Cr$ with an enthalpy of −153 kJ mol^{-1}, (2) $TiB_2 + Ti \rightarrow 2$ TiB with an enthalpy of −85 kJ mol^{-1}, as reported by Attar et al. [4]. For the high LSS, the time and/or temperature given to the interaction appear not sufficient to achieve a significant rate of reaction, but while the LSS decreases, the laser energy density seems high enough to promote this reaction, leading to the appearance of β-Ti.

A more detailed examination of the small 2θ angles sector reveals that all the as-processed parts present an α'-Ti microstructure, which could be attributed to the high heating/cooling rate of the melted pool. Thus an important residual strain can be observed [27]. The standard major 2θ peak of α-Ti (indicated by the vertical line in the figure) is 47.01°. But looking more closely to this peak, one can observe a change of its location when changing