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Laser engineered net shaping of quasi-continuous network microstructural TiB reinforced titanium matrix bulk composites: Microstructure and wear performance

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ABSTRACT

Titanium (Ti) and its alloys have been successfully applied to the aeronautical and biomedical industries. However, their poor tribological properties restrict their fields of applications under severe wear conditions. Facing to these challenges, this study investigated TiB reinforced Ti matrix composites (TiB-TMCs), fabricated by in-situ laser engineered net shaping (LENS) process, through analyzing parts quality, microstructure formation mechanisms, microstructure characterizations, and workpiece wear performance. At high B content areas (original B particle locations), reaction between Ti and B particles took place, generating flower-like microstructure. At low B content areas, eutectic TiB nanofibers contacted with each other with the formation of crosslinking microstructure. The crosslinking microstructural TiB aggregated and connected at the boundaries of Ti grains, forming a three-dimensional quasi-continuous network microstructure. The results show that compared with commercially pure Ti bulk parts, the TiB-TMCs exhibited superior wear performance (i.e. indentation wear resistance and friction wear resistance) due to the present of TiB reinforcement and the innovative microstructures formed inside TiB-TMCs. In addition, the qualities of the fabricated parts were improved with fewer interior defects by optimizing laser power, thus rendering better wear performance.

1. Introduction

Owing to their high strength-to-weight ratio and outstanding biocompatibility, titanium (Ti) and its alloys have attracted a great deal of attentions in aeronautical and biomedical industries [1,2]. However, Ti and its alloys exhibit poor wear performance which limits their applications in fabrication of parts (e.g. gears and bearings [3,4], jet engine compressors [5], etc.) those will work under severe wear conditions. In addition, Ti alloys, such as Ti-6Al-4V, releases aluminum and vanadium ions for long time usage, causing health-related problems [6,7].

To improve Ti and its alloys' wear performance (including indentation and friction wear resistances), adding ceramic reinforcements (e.g. Al_2O_3 [8], SiC [9], TiN [10], TiC [11], TiB [12], etc.) to form titanium matrix composites (TMCs) gains its popularity [8–12]. Compared with other ceramic reinforced TMCs, TiB reinforced TMCs (TiB-TMCs) exhibit more specific benefits and have been extensively investigated in recent years [2,3,9,13–17]. The major reasons include: (1) The residual stresses at interfaces of TiB and Ti can be reduced or even eliminated owing to their similar densities and thermal expansion coefficients [14,18]; (2) A relatively small amount of TiB reinforcement can largely increase the composites' modulus and wear performance on account of the long-whisker shape of TiB [18]; and (3) Surface modified pure Ti with TiB shows good cell growth by performing *in vitro* biocompatibility test under static condition [13].

TiB-TMCs can be fabricated by both ex-situ and in-situ processes. Compared with ex-situ ceramic reinforced TMCs, the in-situ ceramic reinforced TMCs demonstrate following advantages: (1) The in-situ process provides a way of uniformly embedding the rigid ceramic reinforcement in the metal or alloy matrix; (2) The ceramic reinforcement and the Ti matrix are thermodynamically stable with the in-situ process, thus rendering less degradation of TMCs at elevated temperature [12,19]; (3) The size of the in-situ formed ceramic reinforcement is much finer; and (4) The heat generated during the in-situ reaction can be utilized. Based on the aforementioned advantages, excellent physical and mechanical properties can be expected with the in-situ process [10,20].

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Traditional ceramics reinforced TMCs manufacturing processes include casting [15] and powder metallurgy [16]. These traditional manufacturing processes have problems of high energy consumption and high cost for additional machining [17,21,22]. In addition, difficulties arise when producing complex-shaped parts by traditional manufacturing processes [23,24]. To overcome the shortcomings associated with traditional manufacturing processes, additive manufacturing (AM) technology has been developed and widely used to produce metallic components, including TMCs [25]. Laser additive manufacturing (LAM) technology, mainly including selective laser sintering/melting (SLS/M) and laser engineered net shaping (LENS), is regarded as one of the most attractive AM technologies for fabrication of metal based materials [26–28]. Based on a layer by layer powder spreading mechanism, SLS can partially melt the powder via sintering [26]. To meet the demand of producing near-full-dense parts with better mechanical properties, SLM has been developed to fully melt the powder [3,29]. Compared with powder-bed based SLS/SLM processes, the beam-deposition based LENS process exhibits more advantages, such as lower labor intensity, higher fabrication efficiency [30,31], and parts remanufacturing capability [32–36]. To improve materials' surface properties, laser coating (aka, laser cladding) is widely used by LENS process [1,9,10,13,17,27,37]. Laser coating's drawbacks of poor coating-substrate adherence [38] and lacking in uniformity [39] restrict its fields of applications.

Facing to these problems, this paper has fabricated bulk TiB-TMCs, on which limited investigations have been conducted, with uniform distribution of TiB reinforcement inside TMCs by in-situ LENS process. The effects of reinforcement (TiB) and energy input (laser power) on the qualities, microstructure characterizations, and wear performance of the fabricated bulk parts are also analyzed.

This paper is organized as follows: Following with this introduction, Section 2 describes the experiments set-up and measurements procedures. Then, in Section 3, phase identification and microstructure analysis are discussed to provide insight into effects of TiB reinforcement and laser power on wear performance of the fabricated bulk parts. Conclusions are drawn in Section 4.

2. Experiments and measurements procedures

2.1. Powder materials and powder treatment

In this paper, commercially pure Ti (CP-Ti) powders (Atlantic Equipment Engineers Inc., Upper Saddle River, NJ, USA) with the purity of 99.7% and the average particle size of 150 μm were used. The purity of B powders (Chemsavers, Inc., Bluefield, WV, USA) was higher than 96% and the average particle size was 2 μm.

According to the binary Ti-B phase diagram, when the B content was 1.6 wt.%, eutectic solidification process would occur with the generation of ultrafine eutectic TiB, which was beneficial to fracture toughness and bending strength [3,40,41]. Therefore, the weight ratio of 98.4:1.6 between Ti and B powders was adopted in this paper. The powders were premixed by planetary ball mill machine (ND2L, Torrey Hills Technologies LLC., San Diego, CA, USA) prior to LENS process. To well distribute B powders into Ti powders without significant size reduction and reaction between Ti and B, ball milling parameters were optimized, including the ball-to-powder weight ratio of 5:1, the fixed rotation speed of 200 rpm, and the milling time of four hours.

2.2. Experimental set-up and experimental conditions

2.2.1. Experimental set-up

The fabrication experiments were performed on a LENS machine (450, Optomec Inc., Albuquerque, NM, USA) which consisted of laser system (with 400 W IPG fiber laser), chamber system, powder and inert gas delivery system (with coaxial four-nozzle), and control system (with integrated computer), as illustrated in Fig. 1.

To prevent Ti from being oxidized at high temperature, the sealed chamber was purged with the inert argon gas to keep a low oxygen level (<200 ppm) before the fabrication process. Then, the powder and inert gas stream (generated by the powder and inert gas delivery system) as well as the laser beam (generated by the laser system) were simultaneously ejected to the targeted substrate workpiece through deposition head. The laser beam melted the certain area of substrate, forming a molten pool. Powders were caught and melted by the molten pool, resulting in an increased molten pool volume. Then, the deposited powders solidified due to heat dissipation once the laser beam left. Based on the designed structures, the laser beam and the powder stream would follow the trajectory generated by the control system to deposit the first layer. Afterwards, the deposition head ascended one layer thickness to a new set position for the next layer deposition. Treated as the new "substrate", the first layer would be selectively melted to catch powders for forming the second layer. Similar process was repeated many times until the designed 3D structure was built layer by layer.

2.2.2. Experimental conditions

In this investigation, four parts were fabricated on the Ti substrate under each combination of input variables. The dimensions of the parts were 8 mm × 8 mm × 20 layers. Based on preliminary results, the LENS parameters were fixed at optimal values, including the deposition head scanning speed of 11 mm/s, the hatch distance of 381 μm, the layer thickness of 420 μm, and the powder feeding rate of 1.65 g/min. A zigzag scanning pattern with 90° orientation changing for each layer was adopted to reduce the effect of scanning orientation.

2.3. Phase analysis and microstructure characterization

To observe and analyze the microstructure of the fabricated parts, the cutted parts were ground and polished using a MetaServ 250 single grinder-polisher machine (49-10055, Buehler, Lake Bluff, IL, USA), and then etched by Kroll's Reagent (HF: 3%; HNO_3: 6%; water: balance) (Etchant Store, Suite N Glendora, CA, USA) for ten seconds. To have a better observation of the TiB structure, 20% HCl was used to etch some of the polished surfaces for ten hours. The Scanning Electron Microscope (SEM) (Crossbeam 540, Carl Zeiss AG, Oberkochen, Germany), equipped with Energy-Dispersive X-ray spectroscopy (EDX) system and Electron BackScatter Diffraction (EBSD) system, was used to observe the microstructures of the polished and etched cross-sections of the parts, as well as the morphologies and shapes of the powders. Phase composition analysis (EDX spectrums for phases and phase fractions) was conducted with Oxford EDX system. As required by EBSD pattern acquisition, the stage was tilted to the 70° orientation with 20 kV acceleration voltage to study the structures and phases of the materials in the SEM. The basic processes for indexing an EBSD pattern included detecting the diffraction bands, measuring the angles between these bands, and matching these bands to the database for the given material [42]. Such EBSD indexing processes were implemented using an integrated AZtec software.

2.4. Mechanical properties testing

Wear performance (including indentation wear resistance and friction wear resistance) was tested on the polished surfaces (perpendicular to the deposition direction) of fabricated parts. The microhardness value, which was measured using a Vickers

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