



Selective laser melting of TiB₂/316L stainless steel composites: The roles of powder preparation and hot isostatic pressing post-treatment



Bandar AlMangour^{a,*}, Dariusz Grzesiak^b, Jenn-Ming Yang^a

^a Department of Materials Science and Engineering, University of California Los Angeles, Los Angeles, CA 90095, USA

^b Department of Mechanical Engineering and Mechatronics, West Pomeranian University of Technology, Szczecin, Aleja Piastów 17, Poland

ARTICLE INFO

Article history:

Received 24 July 2016

Received in revised form 12 December 2016

Accepted 26 December 2016

Available online 29 December 2016

Keywords:

Stainless steel matrix composites

Mechanical alloying

Selective laser melting

Hot isostatic pressing

Wear

ABSTRACT

Selective laser melting (SLM), a promising additive manufacturing technology, was used to fabricate TiB₂/316L stainless steel composites. An important factor in manufacturing composites via SLM is feedstock powder preparation. In this work, the powders were prepared by either direct mixing or ball milling. The evolution of constituent phases, microstructural features, and size distribution with milling time was investigated. This research determined that powder particles were both coarsened and refined during the early stages of milling (0–6 h), depending on the milling time. After 8 h of milling, the powders exhibited a wide size distribution and stable spherical morphology, while the average crystallite size of the stainless steel matrix phase significantly decreased to 11.11 nm due to severe plastic deformation. The powders obtained after 8 h of mixing or milling were processed via SLM, and the microstructures exhibited a continuous ring-like structure of uniformly dispersed TiB₂ particles. The hardness of the nanocomposite sample prepared from the ball milled powder exceeded that for samples made with the directly mixed powder, but only for those with the highest content of 15 vol.% TiB₂ reinforcement particles, due to finer particle sizes and enhancement of the wetting behavior in the molten pool. To increase the final density of these SLM-fabricated components with high hardnesses, a hot isostatic pressing (HIP) post-treatment was applied. The microstructural characteristics of the HIPed samples evolved with HIP holding time from equiaxed grains to segregated regions of coalesced reinforcement particles. In addition, significant drops in hardness and wear resistance values were observed after HIP treatment due to the high-temperature annealing effect.

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1. Introduction

Stainless steel is an alloy of steel with a high concentration of chromium, which forms a passive coating that protects the material from corrosive oxidation [1,2]. This characteristic allows the use of stainless steel in applications that would normally exclude carbon steels. Because stainless steel exhibits limited hardness and wear resistance, the incorporation of an additional hardening phase into the stainless steel matrix can significantly enhance its performance [3,4] by forming a new composite material with improved physical and mechanical properties [5].

The increasing emphasis on the design and processing of stainless steel matrix composites (SMCs) stems from their exceptional properties, including high specific moduli, thermal stability, strength, and wear resistance [6,7]. The ability of SMCs to be enhanced by the addition of particle reinforcements can expand their applications to the

aerospace, biomedical, and other high-tech industries. Common SMC reinforcements include phase carbides (SiC, TiC, WC), nitrides (TaN, TiN), borides (TiB, TiB₂, WB), metal oxides (Al₂O₃), and carbon fibers [7–10]. In particular, TiB₂ is one of the best and most compatible ceramic reinforcements for improving mechanical properties of the steel matrix [11]. Generally, increasing the amount of the ceramic reinforcement material corresponds to an increase in hardness, wear resistance, and elastic modulus of a composite (assuming that no defects are present) [12,13].

The main technologies to SMC manufacturing are high-pressure diffusion bonding, casting, and powder metallurgy [5,12,14]. However, these techniques are expensive and usually require lengthy post-processing steps, such as machining. In addition, one of the primary challenges associated with fabricating composites containing nanosized reinforcement particles is reinforcement agglomeration, which results in microstructural inhomogeneity and diminished mechanical properties [15–17]. For this reason, selective laser melting (SLM) has been recently used for processing SMC components [13,18,19]. It is an additive manufacturing (AM) technique based on a layer-by-layer incremental manufacturing concept, which enables rapid fabrication of three-dimensional (3D) shapes with complex geometries [20–22]. During SLM, parts are built via selective fusing and consolidating thin layers

Abbreviations: AM, additive manufacturing; CAD, computer-aided design; COF, coefficient of friction; HIP, hot isostatic pressing; MA, mechanical alloying; SEM, scanning electron microscopy; SLM, selective laser melting; SMC, stainless steel matrix composite; TEM, transmission electron microscopy; XRD, X-ray diffraction.

* Corresponding author.

E-mail address: balmangour@gmail.com (B. AlMangour).

of metal powders in a layer-by-layer manner, using a high-energy laser beam that follows 3D computer-aided design (CAD) models. Owing to the high SLM solidification rates, the obtained melt undergoes a unique non-equilibrium metallurgical state, which leads to a distinctly fine microstructure and superior mechanical properties (in contrast to the traditional SMC manufacturing methods) [20]. As compared to traditional manufacturing routes, the SLM technique has many benefits, including high levels of process flexibility and net shape manufacturing achieved without using dies or molds [21]. Therefore, we believe that AM of composite components has the potential to fulfill future demands for novel materials with unique properties and thus revolutionize SMC applications.

An important factor in manufacturing composites via SLM is feedstock powder preparation. The characteristics of the metal powders (such as particle morphology, size, and dispersion) play a significant role in determining the final microstructure and mechanical properties of the SLM-processed composites [18,23]. Two methods are commonly used for the preparation of metal powder mixtures: direct mixing and mechanical alloying (MA) through ball milling. MA is a non-equilibrium, solid-state processing technique, which can be used for the synthesis of advanced metal-ceramic composites at room temperature. The repeated deformation, fracturing, and cold-welding which occur during high-energy ball milling result in a change in morphology, size, and microstructure of metal powders [24–26]. Since ball milling introduces significantly more energy into the powder mixture (as compared to direct mixing), it may substantially affect the properties of the composite material after laser processing [23,27,28], which is why significant research efforts are required to determine the powder morphology, size and particle size distribution characteristics of a powder feedstock. Furthermore, ball milling increases capital costs and possibly lead times, and its complete elimination would reduce costs and improve manufacturing throughput of SMC components.

In addition to the powder preparation methods that affect the performance of SLM-processed components, their level of densification must be also taken into account. One of the major challenges of SLM is the production of parts that are free from pores and cracks, because both of these defects can impact its mechanical properties. Hot isostatic pressing (HIP), which involves the simultaneous application of high temperature and pressure under inert gas atmosphere, is a commonly used industrial technique for material densification and crack healing. It significantly improves the mechanical properties of metallic alloy specimens produced by AM [29–31].

In this work, TiB₂/316L composite powders were prepared by either direct mechanical mixing or ball milling. The TiB₂ content was varied from 2.5 vol.% to 15 vol.%. The resulting phases, microstructural evolution, and physical properties of the milled nanocomposite powders were investigated. Afterwards, various specimens were fabricated by

SLM from the obtained composite powders, and their characteristics, such as phase composition, densification level, microstructure, hardness, and wear properties were evaluated. The results of this research provide insight into the role of initial powder characteristics in the formation of microstructures during SLM processing. In addition, the HIP-post treatment was applied to composites processed using ball milled 15 vol.% TiB₂/ 316L powder in order to examine its influence on composite densification, microstructural evolution, and mechanical properties.

2. Experimental procedure

2.1. Powder preparation

Two starting powder materials were used: gas-atomized 316L stainless steel powder with an average particle diameter of 45 μm and a spherical morphology (Fig. 1a) and TiB₂ powder with a nearly hexagonal prismatic shape and particle sizes of 2–12 μm (Fig. 1b). The 316L matrix was either mixed or ball milled with either 2.5 vol.% or 15 vol.% of TiB₂ powder. Both processes utilized identical equipment and procedures. Mixing was conducted using only the raw powders, while the ball milling also used stainless steel balls. Stainless steel ball milling was conducted at a ball-to-powder weight ratio of 5:1 inside a high-energy planetary mill (Pulverisette 4, Fritsch GmbH) for a period of 2, 4, 6, or 8 h. After 1 h of mixing or milling, the rotation was stopped for 15 min in order to avoid excessive temperature rise inside the grinding bowl. A constant disc rotation speed was maintained for both methods (200 rpm), and both mixing and milling procedures were conducted under protective Ar atmosphere.

2.2. SLM and HIP post-treatment

The feedstock powders obtained after mixing or milling for 8 h were used to fabricate cylindrical composite SLM specimens with diameters of 8 mm and heights of 6 mm. The SLM system consisted of a fiber optic laser, an automatic powder-layering apparatus, an inert Ar gas source, and a computer-based control setup. The main SLM parameters, highlighted in Table 1. An “alternate-hatching” scan pattern was used for specimen fabrication.

A standard HIP post-treatment procedure was subsequently applied to the TiB₂-containing ball milled nanocomposite containing 15% vol. TiB₂ using a commercial service provided by Quintus Technologies, USA. Two HIP-treatment cycles were utilized:

- 1- Heating for 2 h at a temperature of 1150 °C and a pressure of 2070 bar (produced by an Ar gas flow) followed by rapid cooling to 200 °C at a rate of 100 °C/min.

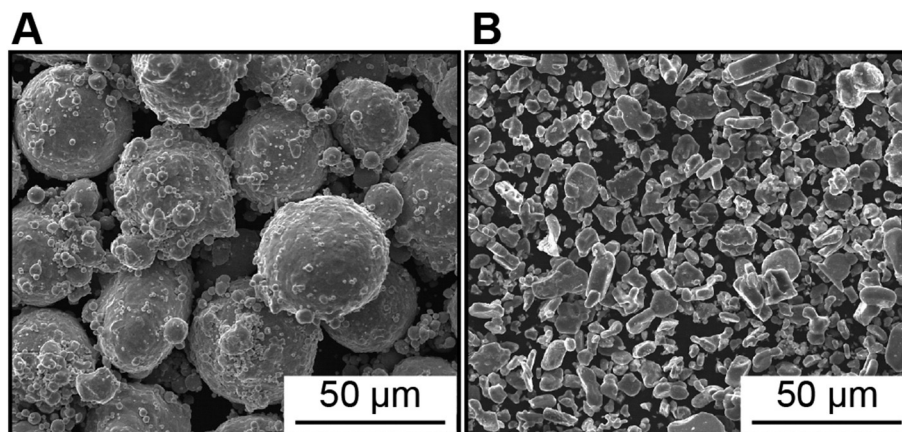


Fig. 1. SEM images showing the microstructures of the starting materials: (a) 316L stainless steel and (b) TiB₂ powders.

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