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A new approach for fabrication of titanium-titanium boride periodic composite via additive manufacturing and pressure-less sintering

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ABSTRACT

This study proposes a new additive manufacturing (AM) based methodology to fabricate periodic metal matrix composite architectures, with a focus on titanium (Ti)-titanium boride whisker (TiB_w) composites. The manufacturing method includes binder jetting AM of the titanium matrix reinforced periodically by the extrusion of a custom-developed highly loaded resin, containing titanium di-boride ($TiB_{2)}$ particles. A low-temperature pressure-less sintering method was then applied to increase the mechanical strength of green samples produced from the additive manufacturing step. The sintering process also fosters the chemical reaction between the matrix and ceramic, resulting in the growth of titanium boride whisker (TiB_w). The ceramic volume fraction and sintering protocol were studied as two main input variables in the design and fabrication steps. Investigating the influence of input parameters on the volume fraction and morphology (whisker formation) of TiB determined that the physical properties of the specimens, such as stiffness, were affected. The data analysis suggested a higher possibility for the formation and growth of TiB_w as the temperature elevated in the sintering step (1400 °C). The ranges of 1.6 ± 0.2 GPa–3.7 ± 0.4 GPa and 83.9 ± 18.7 MPa–165 ± 13.2 MPa for the Young's modulus and Yield stress of the specimens were obtained, respectively. The stiffness of the samples was enhanced significantly by increasing the temperature and volume fraction. In particular, those samples sintered up to 1400 °C displayed 6.4%–15.2% improvement in the stiffness, although only a small fraction of the ceramic material was incorporated into the design: 2% and 4%, respectively. The similar trend of the improvement in density of the porous matrix was observed (i.e., 4.5%–19%). The range of mechanical and structural properties of the periodic composite developed in this study demonstrated the relevance of applying this method to the fabrication of biomedical and other lightweight titanium composite structures.

1. Introduction

Metal matrix composites (MMC) have become relevant on a widely diverse area of applications due to incorporating unique metallic properties into the structures enhanced with reinforcement elements [1,2]. Among the wide range of MMC examples, titanium matrix composites (TMC) have been studied extensively in various industrial applications, some of which include: aerospace, automotive, and biomedical [3]. The popularity of this group of composites is mainly attributed to the Ti properties such as the high strength to weight ratio, thermal and oxidation resistance, and biocompatibility, all in which are fortified through the addition of the filler properties: high thermal resistivity, wear, and fatigue resistance [4].

From several reinforcement materials introduced in Ti matrix, titanium di-boride (TiB_2) has displayed the most promising results with regard to stability in the chemical reaction between Ti and TiB_2, in addition to similarity in their thermal expansion coefficient [3] which increases the mechanical stability. Some of the notable properties of TiB_2 include a high melting point, a high stiffness to density ratio, and hardness [5]. The chemical reaction between Ti and TiB_2 results in the formation of titanium boride (TiB) crystal [6]. The success in the synthesis step is influenced by sintering conditions [6,7], volume fractions of ceramic (TiB_2) [8], as well as the particles size [7]. The impact of each parameter on the growth of TiB has been studied thoroughly, by validating the morphology (whisker, plate, and cluster) and grain size [3]. Each of these parameters controls the properties of TMC such as stiffness, hardness, thermal conductivity, creep performance, etc. [8]. Although the majority of the studies in the literature have focused on Ti-TiB composites as an essential candidate for industrial applications, few studies demonstrated promising results in the biocompatibility of the composite while used in the fabrication and coating of biomedical implants [9–11].

Introducing additive manufacturing (AM), which is based on fashioning 3D objects through a layer-by-layer construction, has created a

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promising path towards the fabrication of structures with complexity in topology and variation in materials. In powder-bed AM, structuring the multi-material objects has been addressed by applying different methods based on the type of process [12–17]. One way is through applying bimodal powders containing matrix and reinforcement powder [18–22]. However, this approach for the fabrication of MMC can only be benefited from the AM ability to structure a complex geometry but from controlling the materials layout point of view, it is similar to that offered by other conventional methods. The binder jetting method (one class of the powder-bed AM) is based on constructing a 3D object throughout spreading the layers of powder followed by injecting a liquid binder to provide the adhesion between the layers and final integrity of the green samples. Most often, the process is followed by a post-processing step such as sintering, particularly in the case of printing the structure out of ceramics or metals materials [23]. To create a multi-material structure in this method, injecting multiple liquid binders [24], in addition to retrofitting a system for selectively encapsulating solid particles [25] of two or more materials, have been suggested.

In the present study, a binder jetting system in conjunction with a material extrusion [26] is employed to propose a new method for manufacturing MMC structures. Due to the minimum requirements in the material selection, each of these methods has demonstrated a great potential in the fabrication of structures from the wide range of the materials (metals, ceramics, polymers and composites) with different thermal properties. Therefore, the combination of these AM approaches can generate a vast possibility in fabricating of objects from multi-material and with controlled distribution while eliminating the necessity of an assembly step. This range of the part fabrication cannot be achievable by other AM methods such as fused deposition modeling (FDM) due to the limitation in the range of materials. This limitation is mainly resulted from the system requirement to low melting temperature in most commercially available systems [27] which creates the need of the multi-step fabrication and assembly [28]. Although the recent progress in the fabrication of metals and composites filaments by Markforged [MA, USA] has been reported and applied in the literature [29], the speed of manufacturing parts from the similar material is much higher in binder jetting compared to FDM or other extrusion systems. Additionally, creating multi-material TMC structures with controlled on the location of the reinforcement has not been reported by FDM to the best knowledge of the authors.

The TMC structures were composed of Ti matrix, which was manufactured through binder jetting, and TiB_2 reinforcement which was encapsulated periodically within the matrix all in a single step processing. To incorporate the ceramic (TiB_2) into the matrix architecture, a highly loaded acrylic based ceramic-resin composite was developed. The resin must demonstrate certain properties such as high volume fraction of ceramic, a proper range of viscosity, UV curability, low shrinkage in the curation step to maintain the dimensional accuracy, etc. [30]. In material extrusion systems, dispensability of the material is another factor which can be controlled by adjusting the viscosity [31].

The heat treatment step was incorporated into Ti-TiB_2 composite processing to promote the solid-state sintering between matrix particles and facilitates the chemical reaction between Ti and TiB_2, yielding to the TiB formation. To fabricate the samples, two different reinforcement volume fractions, through variation of the number of the ceramic droplets periodically distributed within the layout, as well as two sintering protocols [7,32] were selected as input parameters. To this end, six categories of samples were designed and manufactured by the proposed system. Among the different methods of sintering proposed in the literature [3], the pressure-less sintering (known as gravity sintering too) was selected due to the design and limited integrity of the green samples collected from the binder jetting AM approach. The sintering protocols were differed based on the highest sintering temperatures: a maximum of 1200 °C, which was proposed by Patel and et al. [7], and a maximum of 1400 °C based on the Ti sintering protocol previously developed by our group [23]. Several characterization steps were conducted to investigate the influence of the variable input process parameters on the physical properties and microstructure of the composite specimens.

2. Material and methods

2.1. Material and processing

In this study, for binder jetting AM system, titanium powder was selected to form the matrix with the following powder characteristic: spherical powder with the size distribution of 75–90 μm purchased from Phelly Materials [CP Ti, PhellyMaterials, Ber-genfield, NJ]- ASTM (F67-06 Grade 2). To increase the bond between the powder particles and integrity of the green parts, 3 wt% of polyvinyl alcohol (< 63 μm), [Alfa Aesar, Ward Hill, MA], was added to Ti powder and mixed for 4 h. This step was performed by employing a jar mill [US Stoneware, East Palestine, OH]. Next, the binder jet 3D printer beds were filled with this powder mixture, and ZB60 [Zb™ 60, 3D Systems, Burlington, MA] was applied as a liquid binder in the print head [ImTech 610, OR, USA].

To prepare the reinforcement, a ceramic resin was developed from 67 wt% of titanium di-boride (TiB_2) particles [powder, < 10 μm, Sigma-Aldrich, Oakville, Canada] loaded on a UV curable polymeric liquid to facilitate the material extrusion process. The polymeric part of the resin included 19 wt% bisphenol-A ethoxylated diacrylate (BAE) [Ebecryl 150, Cytec, NJ, USA], 2 wt% cellulose acetate butyrate (CAB) [Sigma-Aldrich, Oakville, Canada], 2 wt% Phenylbis (PI) (2,4,6-trimethyl benzoyl) phosphine oxide (Irgacure 819) [Sigma-Aldrich, Oakville, Canada], and acetone. Next, the resin was prepared by mixing all compounds for 30 min. The previous study showed the dramatic rate of decomposition for the proposed acrylic polymer near 400 °C [31]. The low decomposition temperature promised a complete removal of the polymer from final TMC structures subjected to the sintering. The viscosity of the material mixture was measured by employing a high shear viscometer [BROOKFIELD CAP 2000, Brookfield AMETEK, MA, USA] (Fig. 1) through the steps detailed elsewhere [31]. As shown in this figure, the resin demonstrated a shear-thinning behaviour which suggested its suitability for the dispensing process [33].

Prior to the manufacturing of the composite samples, the ceramic resin was loaded on the extrusion system and the droplets were injected on Ti substrates by applying the following system input parameters: 200 μm needle, 200 kPa pressure and 0.1 s for dispensing time (Fig. 2). This step was performed to determine the droplets morphology: average diameter = 0.48 ± 0.07 μm and height = 0.23 ± 0.04 μm. Comparing the data with those in our previous study [31] suggests a layer thickness of 200 μm as an appropriate selection for the binder jetting system.

For additive manufacturing of composite structures, the six groups

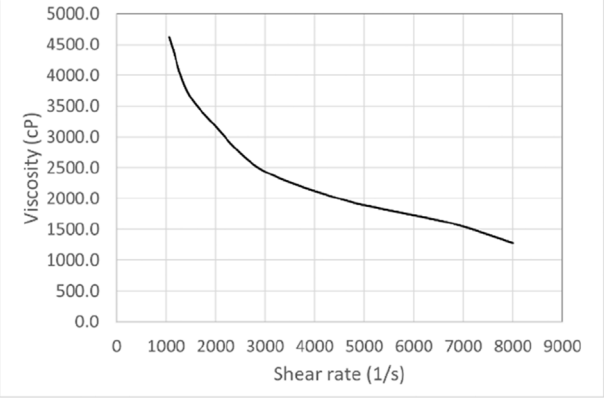

Fig. 1. Viscosity of the highly loaded ceramic resin.