

COMPRESSIVE PROPERTY COMPARISONS BETWEEN LASER ENGINEERED NET SHAPING OF IN SITU TiB_w-TMCs AND CP-Ti MATERIALS

Yingbin Hu^a, Hui Wang^a, Fuda Ning^a, Weilong Cong^{a,*}, Yuzhou Li^b

^aDepartment of Industrial Engineering, Texas Tech University, Lubbock, TX, 79409, USA

^bSchool of Electro-mechanical Engineering, Guangdong University of Technology, Guangzhou, Guangdong, 510006, China

*Corresponding author at: Department of Industrial Engineering, Texas Tech University, Lubbock, TX 79409-3061, USA. Phone: 8067423543. Fax: 8067423411

Email address: weilong.cong@ttu.edu

Abstract

Titanium (Ti) and its alloys are widely used in chemical, aeronautical, and biomedical industries. However, their poor load endurance properties affect their fields of applications especially under severe loading conditions. To enhance these properties, TiB_w reinforcement was synthesized by in situ chemical reaction between elemental Ti and boron. Strong interfacial bonding between TiB_w reinforcement and Ti matrix was obtained due to the in situ chemical reaction. Owing to its capability of producing difficult-to-machine bulk composites with uniform properties, laser engineered net shaping (LENS) technique was utilized to fabricate TiB_w reinforced Ti matrix bulk composites. Few researches have been reported on these three-dimensional metal based bulk composites by using LENS. In this work, effects of TiB_w reinforcement and laser power on compressive properties were investigated. The microstructures of the fabricated parts were observed and analyzed by using scanning electron microscopy.

Keywords

Laser engineered net shaping (LENS); TiB_w reinforcement; Titanium matrix composites (TMCs); Compressive properties

1 Introduction

As excellent corrosion resistance metallic materials, titanium (Ti) and its alloys are recognized for their high strength-to-density ratio and outstanding biocompatibility, thus rendering wide applications in chemical, aeronautical, and biomedical industries. [1,2]. However, for severe working conditions where high bearing capacity property is necessary (e.g. tooth's root implants [3], actuator strut in the engine thrust direction control system [4], etc.), ceramic reinforcements are required to improve compressive properties of Ti and its alloys. Compared with other ceramic reinforced Ti matrix composites (TMCs), titanium boride whisker (TiB_w) reinforced TMCs (TiB_w-TMCs), exhibiting more benefits, are of particular interest. (1) TiB_w reinforcement is chemically compatible with TMCs since there are no intermediate phases between TiB and Ti phases [5]. (2) The residual stresses and stress concentration effects induced by TiB_w reinforcement can be weakened or eliminated as a result of comparable thermal expansion coefficients and densities between TiB and Ti [6]. (3) Strong interfacial bonding

between TiB and Ti can be formed as a result of excellent thermodynamic and chemical stability between these two phases [7,8].

In recent years, in situ TMCs have gained their popularity due to the benefits exhibited by in situ TMCs over ex situ TMCs [9]. Strong interfacial bonding between TiB and Ti as well as better mechanical properties can be obtained with the formation of finer TiBw and the uniform distribution of rigid ceramic TiBw reinforcement in the Ti matrix [9-11]. In addition, the energy generated during chemical reaction between elemental Ti and boron (B) can be utilized to facilitate fabrication process.

Conventional fabrication processes (e.g. casting, powder metallurgy, etc.) of ceramic reinforced TMCs are usually time consuming and labor intensive [8,12,13]. In addition, post-processing, which requires new tooling and production line, is needed to assist fabricating complex-shape parts. To overcome these problems, laser additive manufacturing (LAM), the concept of which is free-to-design, is proposed to fabricate ceramic reinforced TMCs [8,10]. Among all the LAM techniques, laser engineered net shaping (LENS) has specific advantages of small heat affected zone, easy-to-control cooling, small substrate deformation, and changeable dilution [14]. In addition, the unique features of LENS make it an irreplaceable technique in part repair of worn components [15]. Therefore, LENS has been used to fabricate bulk parts with mixed Ti and B powders in this paper.

Figure 1 shows the illustration of LENS process. The powder stream generated from the powder feeder was melted by the laser beam ejected from the laser source, forming a high-temperature molten pool. The molten pool would catch more powders to increase its volume. When the laser beam moved away, the molten pool solidified due to heat dissipation. Following the trajectory designed by the CAD file, one layer was built with the moving of deposit head. Afterwards, the deposit head ascended, leaving space for the next layer fabrication. In the end, the final bulk part was built by repeating these procedures layer by layer.

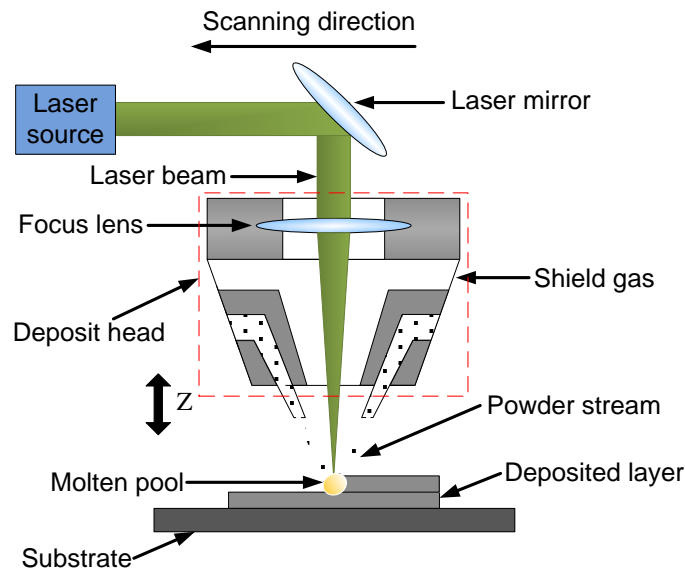


Figure 1. Illustration of LENS process

The remainder of this paper is organized as follows: Section 2 presents descriptions of experiments and measurements procedures. Effects of TiBw reinforcement and laser power on the compressive properties of the fabricated bulk parts will be analyzed and discussed in Section 3. Section 4 will describe the conclusions drawn from this paper.

2 Experiments and measurements procedures

2.1. Powder treatment

Commercially pure Ti (CP-Ti) powders (Atlantic Equipment Engineers Inc., Upper Saddle River, NJ, USA) (average size of 150 μm , purity of 99.7%) and B powders (Chemsavers, Inc., Bluefield, WV, USA) (average size of less than 5 μm , purity of more than 96%) were used in this paper. According to the Ti-B phase diagram, eutectic solidification occurred, when the weight percent of B was around 1.6%, with the formation of nano-size TiB whiskers, thus rendering refinement of Ti grains and better mechanical properties [10,16]. It was reported by Wei et al. that high fracture toughness and bending strength were achieved at this weight ratio for TiBw-TMCs [17]. In this paper, the CP-Ti powders were premixed with 1.6 wt.% B powders by planetary ball mill machine (ND2L, Torrey Hills Technologies LLC., San Diego, CA, USA). Based on preliminary results, the ball mill parameters were set at optimized level: including rotation speed of 200 rpm, ball-to-powder weight ratio of 5:1, and milling time of four hours. After ball mill treatment, flowability and compactability of the powders were improved. In addition, the B powders were well distributed in the Ti powders.

2.2. Experimental conditions

Table 1 shows process parameters adopted in this paper. The input fabrication variables, such as scanning speed (11 mm/s), powder feed rate (1.65 g/min), hatch distance (381 μm), etc., were optimized based on preliminary results. The scanning direction, initiating from 45°, rotated 90° for each layer to weaken the effects of scanning orientation on the fabricated parts. In order to prevent oxygen from contaminating fabricated parts, the oxygen level was kept below 500 ppm. The dimensions of the fabricated cylindrical parts were $\phi 6 \text{ mm} \times 15 \text{ layers}$.

Table 1. Process parameters

Parameters	Values or range	Units
Scanning speed	11	mm/s
Powder feed rate	1.65	g/min
Hatch distance	381	μm
Number of layers	15	/
Scanning orientation	45, 135, 225, and 315	°
Oxygen level	<500	ppm

2.3. Measurement procedures

The fabricated parts were cut through cross-section, then ground and polished by a grinder-polisher machine (MetaServ 250, 49-10055, Buehler, Lake Bluff, IL, USA). Afterwards, these parts were etched with Kroll's reagent (HF: 3%; HNO₃: 6%; water: balance) for ten

seconds. To observe and analyze the microstructural characterizations of the parts, the scanning electron microscope (SEM) (S-4300, Hitachi, Tokyo, Japan) was used.

Before compressive test, both planar sides of each part were ground and polished by the grinder-polisher machine. Along the deposit head moving direction (Z), as is shown in Figure 1, compressive test was performed on the fabricated cylindrical parts by using a tensile tester (AGS-50kNXD, Shimadzu, Kyoto, Kyoto Prefecture, Japan), at a constant cross-head speed of 0.005 mm/s. For each condition, the tests were conducted at least three times at room temperature to have better statistics.

3 Experiment result and discussion

Figures 2(a) and 2(b) show effects of laser power on the defects (e.g. micro-cracks and micro-pores) formation of fabricated CP-Ti parts. It can be seen in Figure 2(a) that not only micro-pores but also micro-cracks presented. In Figure 2(b), micro-pores were dominant, whereas micro-cracks could hardly be seen. The reason lies in the fact that micro-cracks were prone to form at the interfaces of neighboring fabricated layers as a result of insufficient laser power (125 W) [14]. By increasing the laser power to 200 W, the top part of the former solidified layer could be remelted, resulting in a better interfacial bonding between neighboring layers with less cracks or pores. As is reported, the defects, from where dislocations easily initiated with applied force, were stress concentrators, resulting in weakened mechanical properties [18]. Figure 2(c) shows the cross-section characterizations of TiBw-TMCs processed with 200 W laser power. In LENS process, in situ exothermic chemical reaction between Ti and B occurred, forming prismatic TiBw reinforcement. As a kind of rigid ceramic reinforcement, TiBw contributed to the strengthening effect on the TiBw-TMCs [19]. In addition, the exhibition of TiBw reinforcement could lead to Ti grain refinement, which was also beneficial to the mechanical properties [6,10].

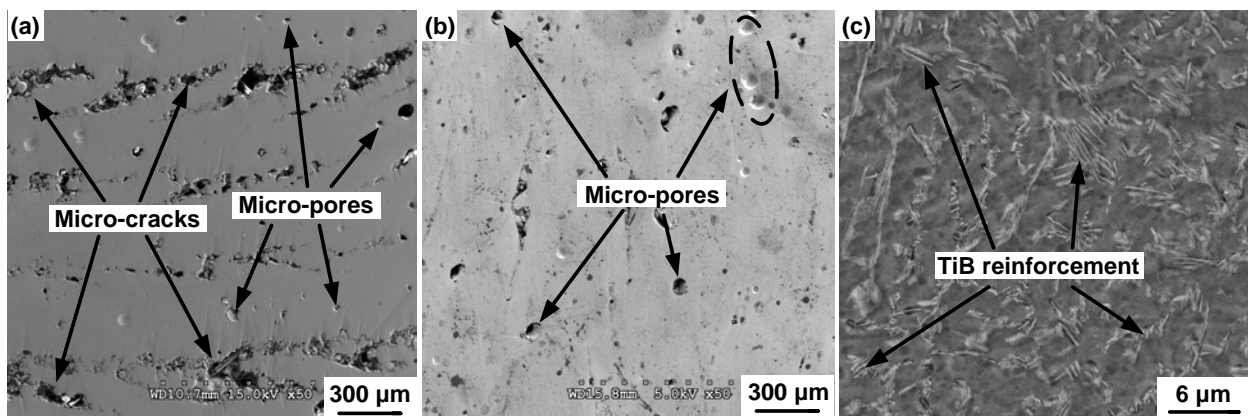


Figure 2. SEM observations of cross-section characterizations of (a) CP-Ti at 125 W; (b) CP-Ti at 200 W; (c) TiBw-TMCs at 200 W.

Compressive tests were performed to investigate effects of TiBw reinforcement and laser power on compressive properties. As was reported, a compressive test was not as susceptible as a tensile test to minor defects. The minor defects inside the solid parts could cause premature failure and then affect property analysis [10]. Figure 3 shows effects of TiBw reinforcement and

laser power on ultimate compressive strength (UCS) of the fabricated parts. The central tendency was represented by the average value of the measured data under each condition. The standard errors, which were calculated by dividing the standard deviation by the square root of the number of measurements, were used to represent error bars' positive and negative values. It can be seen that the UCS was significantly increased with the increase of laser power from 125 W to 200 W for both CP-Ti parts and TiBw-TMCs. Higher densification degree with no obvious micro-cracks of the parts fabricated with 200 W laser power, as aforementioned, could be one possible reason. Another possible reason could be larger residual stress induced by 200 W laser power, leading to the enhancement of UCS. Owing to the present of TiBw reinforcement, TiBw-TMCs exhibited superior UCS, as compared with that of CP-Ti parts at corresponding laser power level. It can be concluded that a dramatic increment of 38.2% (from 760.9 MPa to 1051.4 MPa) could be achieved by importing TiBw reinforcement into the Ti matrix as well as choosing high laser power (200 W). Similarly, yield strength increased with the increase of laser power for both CP-Ti parts and TiBw-TMCs, as is shown in Figure 4. At all levels of laser power, yield strengths of TiBw-TMCs were higher than those of CP-Ti parts.

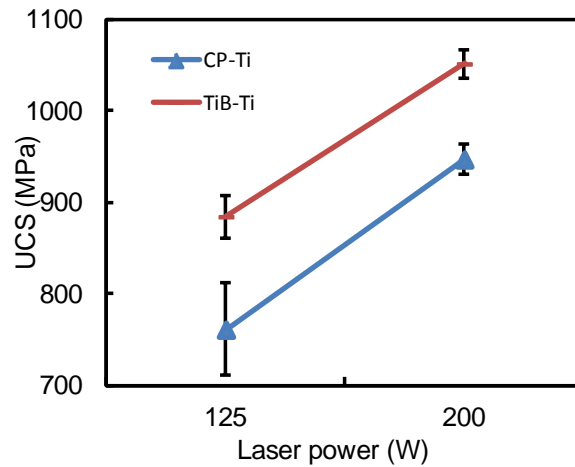


Figure 3. Effects of TiBw reinforcement and laser power on ultimate compressive strength (UCS).

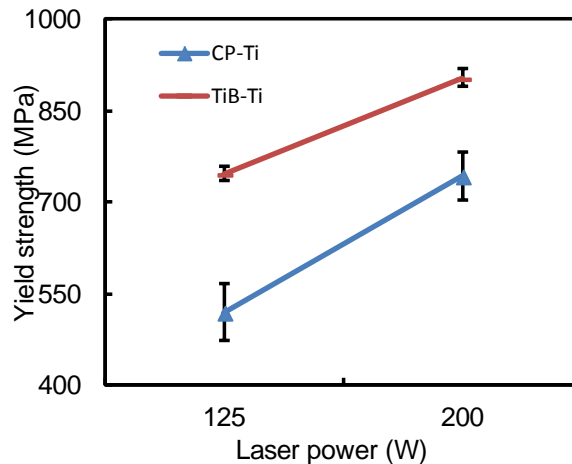


Figure 4. Effects of TiBw reinforcement and laser power on yield strength.

Effects of TiBw reinforcement and laser power on maximum strain, which could be utilized to represent ductility, are shown in Figure 5. The present of TiBw reinforcement led to smaller average maximum strain than that of CP-Ti parts. In another word, the TiBw reinforcement was detrimental to the ductile properties of TiBw-TMCs. Huang et al. reported that ceramic reinforced TMCs exhibited low room-temperature damage tolerance (e.g. ductility and fracture toughness) due to the lack of ductile phase which could inhibit crack propagation [20]. For CP-Ti parts, the increase of laser power led to the decrease of the average maximum strain from 64.9% to 54.3%. The high maximum strain value could possibly be ascribed to the high compatibility induced by the low densities of the parts fabricated with 125 W laser power. For TiBw-TMCs, a slight increment of maximum strain from 24.8% to 26.2% was achieved by increasing laser power from 125 W to 200 W. Figure 6 shows effects of TiBw reinforcement and laser power on toughness. It can be seen that the present of TiBw reinforcement had negative impacts on toughness. For both CP-Ti parts and TiBw-TMCs, toughness increased with the increase of laser power.

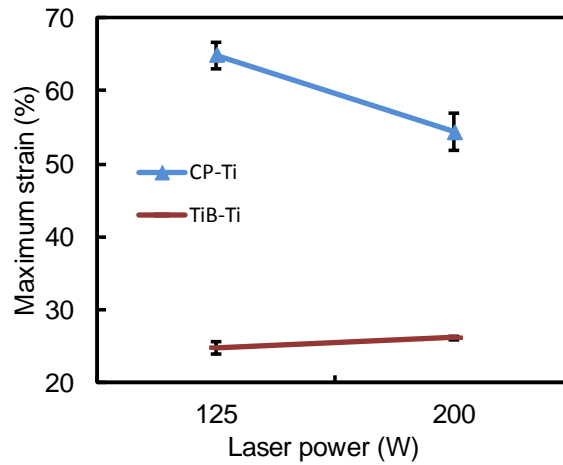


Figure 5. Effects of TiBw reinforcement and laser power on maximum strain.

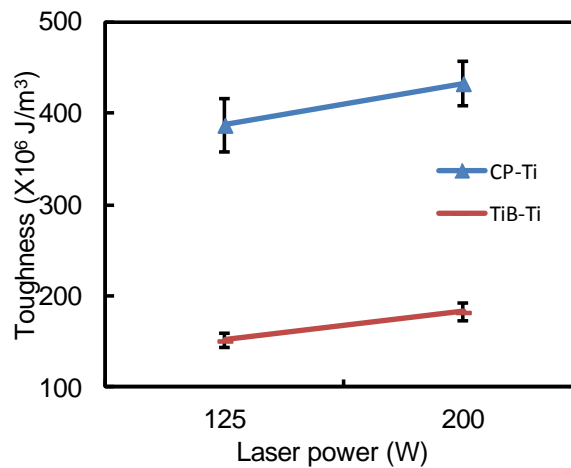


Figure 6. Effects of TiBw reinforcement and laser power on toughness.

4 Conclusions

In this investigation, bulk CP-Ti parts and TiBw-TMCs parts were fabricated and effects of TiBw reinforcement and laser power on compressive properties were investigated. The main conclusions are drawn as follows:

(1) By increasing laser power from 125 W to 200 W, the densification degree of the parts fabricated could be improved with fewer defects inside. Due to this reason, the UCS and the yield strength increased with the increase of laser power. The increased laser power adversely affected maximum strain, whereas it positively affected toughness.

(2) Prismatic TiBw was formed as a result of the chemical reaction between elemental Ti and B in LENS process. Due to the grain refinement and strengthening effects induced by TiBw reinforcement, both the UCS and the yield strength were enhanced. The lacking in ductile phase of TiBw-TMCs led to weakened maximum strain, thus rendering worse toughness.

References

- [1] Donachie, M. J. (2000). Titanium: a technical guide. ASM international.
- [2] Liu, X., Chu, P. K., & Ding, C. (2004). Surface modification of titanium, titanium alloys, and related materials for biomedical applications. *Materials Science and Engineering: R: Reports*, 47(3), 49-121.
- [3] Laoui, T., Santos, E., Osakada, K., Shiomi, M., Morita, M., Shaik, S. K., ... & Takahashi, M. (2006). Properties of titanium dental implant models made by laser processing. *Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science*, 220(6), 857-863.
- [4] Spowart, J. E., & Clyne, T. W. (1999). The axial compressive failure of titanium reinforced with silicon carbide monofilaments. *Acta materialia*, 47(2), 671-687.
- [5] Huang, L. J., Geng, L., & Peng, H. X. (2015). Microstructurally inhomogeneous composites: Is a homogeneous reinforcement distribution optimal?. *Progress in Materials Science*, 71, 93-168.
- [6] Tamirisakandala, S., Miracle, D. B., Srinivasan, R., & Gunasekera, J. S. (2006). Titanium alloyed with boron. *Advanced Materials and Processes*, 164(12), 41.
- [7] Gorsse, S., Le Petitcorps, Y., Matar, S., & Rebillat, F. (2003). Investigation of the Young's modulus of TiB needles in situ produced in titanium matrix composite. *Materials Science and Engineering: A*, 340(1), 80-87.
- [8] Attar, H., Prashanth, K. G., Zhang, L. C., Calin, M., Okulov, I. V., Scudino, S., ... & Eckert, J. (2015). Effect of Powder Particle Shape on the Properties of In Situ Ti–TiB Composite Materials Produced by Selective Laser Melting. *Journal of Materials Science & Technology*, 31(10), 1001-1005.
- [9] Tjong, S. C., & Ma, Z. Y. (2000). Microstructural and mechanical characteristics of in situ metal matrix composites. *Materials Science and Engineering: R: Reports*, 29(3), 49-113.
- [10] Attar, H., Bönisch, M., Calin, M., Zhang, L. C., Scudino, S., & Eckert, J. (2014). Selective laser melting of in situ titanium–titanium boride composites: processing, microstructure and mechanical properties. *Acta Materialia*, 76, 13-22.

- [11] Emamian, A., Corbin, S. F., & Khajepour, A. In-Situ Formation of TiC Using Laser Cladding. Eustathopoulos, N., Nicholas, M. G., & Drevet, B. (Eds.). (1999). Wettability at high temperatures (Vol. 3). Elsevier.
- [12] Zhang, E., Jin, Y., Wang, H., & Zeng, S. (2002). Microstructure and hardness of as-cast in situ TiB short fibre reinforced Ti-6Al matrix composites. *Journal of materials science*, 37(9), 1861-1867.
- [13] Yolton, C. F. (2004). The pre-alloyed powder metallurgy of titanium with boron and carbon additions. *JOM*, 56(5), 56-59.
- [14] Gu, D. D., Meiners, W., Wissenbach, K., & Poprawe, R. (2012). Laser additive manufacturing of metallic components: materials, processes and mechanisms. *International materials reviews*, 57(3), 133-164.
- [15] Atwood, C. J., Smugeresky, J. E., & Gill, D. D. (2006). Laser engineered net shaping (LENS) for the repair and modification of NWC metal components (No. SAND2006-6551). Sandia National Laboratories.
- [16] Murray, J. L., Liao, P. K., & Spear, K. E. (1986). The B– Ti (Boron-Titanium) system. *Bulletin of alloy phase diagrams*, 7(6), 550-555.
- [17] Wei, S., Zhang, Z. H., Wang, F. C., Shen, X. B., Cai, H. N., Lee, S. K., & Wang, L. (2013). Effect of Ti content and sintering temperature on the microstructures and mechanical properties of TiB reinforced titanium composites synthesized by SPS process. *Materials Science and Engineering: A*, 560, 249-255.
- [18] German, R. M. (1984). *Powder metallurgy science*. Metal Powder Industries Federation, 105 College Rd. E, Princeton, N. J. 08540, U. S. A, 1984. 279.
- [19] Meng, Q., Feng, H., Chen, G., Yu, R., Jia, D., & Zhou, Y. (2009). Defects formation of the in situ reaction synthesized TiB whiskers. *Journal of Crystal Growth*, 311(6), 1612-1615.
- [20] Huang, L. J., Geng, L., & Peng, H. X. (2015). Microstructurally inhomogeneous composites: Is a homogeneous reinforcement distribution optimal?. *Progress in Materials Science*, 71, 93-168.