Materials Characterization 143 (2018) 110–117

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Effect of laser power on oxygen and nitrogen concentration of commercially pure titanium manufactured by selective laser melting

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ARTICLE INFO

Keywords:
Additive manufacturing
Selective laser melting
Titanium
Oxidation
Nitriding

ABSTRACT

This study analyzed the variation of mechanical properties and its causes with increasing the laser power in the fabrication of pure titanium by selective laser melting (SLM). SLM samples were fabricated using commercially pure titanium grade 1 powder when the scan speed was 1000 mm/s and the laser power as 120, 200, 280, 360, and 440 W, respectively. As the laser power increased, the hardness and strength of the samples increased gradually. During the SLM processing, the concentrations of oxygen and nitrogen in the SLM samples were increased, which resulted in the increase of hardness and strength. The SLM equipment used in this study removed oxygen in the chamber by flowing high purity argon gas and fabricates the sample while preserving the oxygen concentration in the atmosphere to 0.2%. Evaluating the possibility of oxidation and nitriding during the SLM process by thermodynamic analysis, it was found that the process occurred under conditions in which temperature and residual oxygen and nitrogen partial pressure led to oxidation and nitriding.

1. Introduction

Metal additive manufacturing (AM) is a process of fabricating products by selectively and repetitively melting metallic powder using a heat source, such as laser or electron beam, unlike the conventional powder metallurgy process. It is difficult to fabricate complex geometrically shaped products by the conventional manufacturing process, which involves machining or cutting. However, AM technology can directly fabricate the final product having a complex internal structure especially manufacturing with the electron beam melting (EBM) and selective laser melting (SLM) of powder bed fusion type [1–3]. Therefore, research about the AM technology is actively being conducted to apply the method to a wide range of industries, including the medical and aerospace fields such as patient-specific artificial orthopedic implants and high-performance heat exchangers [4–6].

Titanium and titanium alloys are widely used in biomedical and aerospace applications due to titanium's high strength-to-weight ratio, good biocompatibility, and excellent corrosion resistance. Previously, Ti-6Al-4V alloy was the most frequently used due to its excellent specific strength. However, Ti-6Al-4V alloy shows lower corrosion resistance than that of commercially pure titanium (CP–Ti) [7] and the Al and V released from Ti-6Al-4V alloy is possibly toxic to the human body [8]. In particular, Al has been suggested to induce Alzheimer's disease [9,10]. Therefore, the research for using CP–Ti has been conducted in the metal AM field.

Generally, the mechanical strength and wear resistance of CP–Ti fabricated by AM technology are improved because the residual stress caused by the thermal shock, through the locally-repeating process of rapid cooling and heating [11,12]. In some conditions, the formation of martensitic microstructure also improves the mechanical strength [13]. Therefore, CP–Ti is expected to have a broad application field by applying AM technology in the medical or chemical-plant industries.

Previous studies on AM with CP–Ti have focused on the mechanical properties and structure variation by the process conditions, such as the laser power, scan speed, hatching distance, and layer thickness [14–16]. However, research regarding the changes in chemical composition of light elements, such as oxygen and nitrogen, corresponding to the process conditions have not been reported. We recently reported that the EBM process could induce the refining effect of oxygen and nitrogen on CP–Ti [17]. The inside of the EBM chamber is maintained in a vacuum state at approximately 10^{-3} Torr for the emitting electron beam. Thus, the oxygen reduction occurred because of the low oxygen partial pressure in the chamber during the process.

In the SLM process, the partial pressure of residual gases may

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https://doi.org/10.1016/j.matchar.2018.03.003
Received 3 January 2018; Received in revised form 22 February 2018; Accepted 4 March 2018
Available online 05 March 2018

increase, depending on the process conditions, since the most of SLM is not produced in a vacuum environment. In the case of titanium, when the concentration of oxygen, nitrogen, and carbon are increased in a specific range, mechanical properties such as hardness and strength are increased [18–20]. The potential for change in the composition of CP–Ti parts with the SLM process conditions needs to be investigated, because changes in light element concentrations can affect the mechanical properties directly.

The SLM process is fundamentally based on the interaction between the laser and metallic powder such as absorption, melting, reflection, convection and conduction etc. The major considerable process parameter in the SLM process could be represented with the laser power and scan speed related to the irradiated laser energy [13]. However, the absorptance of energy which can significantly affect to the interactions, is sensitively influenced by the laser exposure time due to the increase of temperature at the specific region. The risen temperature can decrease the electrical conductivity (Wiedemann-Franz law) then it could also reduce the reflectance of powder (Hagen-Rubens relation). Thus, with the variable scan speed conditions, it is hard to exactly calculate the maximum temperature rise because of the varying absorptance. The value of maximum temperature rise corresponding to the SLM process conditions is important to understand the oxidation and reduction according to Ellingham diagram. In this study, therefore, to clearly understand the effect of laser power on the relationship between the mechanical properties and chemical composition of CP–Ti, only the laser power was controlled, while retaining the other process parameters during the SLM process. Additionally, the potential to increase chemical concentration of light elements, such as oxygen and nitrogen, during the SLM process was analyzed from a thermodynamic perspective.

2. Experimental Procedure

Spherical CP–Ti powder (ASTM F67 Grade 1, supplied by AP&C, Canada) was used as the initial material for manufacturing the CP–Ti part by SLM. The particle size distribution of the powder was determined using a particle size analyzer (Malvern, Mastersizer 3000). Fig. 1 shows particle shape of the CP–Ti powder and particle size distribution, indicating that d_{10}, d_{50}, d_{90} were 21.3 μm, 32.0 μm, and 46.4 μm, respectively.

CP–Ti SLM samples were fabricated by Farsoon Technologies' FS271M model with a 500 W Yb-fiber laser and a dynamic focusing galvanometer scanning system. A schematic illustration of the SLM equipment is depicted in Fig. 2.

For the experimental design, the SLM conditions used in this study led to the full melting of CP–Ti without side effects like swelling, collapse, or balling phenomena. The range of full melting was investigated using the microstructure map using the process parameters with power of 80–440 W and scan speed of 500–2000 mm/s as shown in Fig. 3. The laser spot size, hatching space, and layer thickness were fixed to 120 μm, 120 μm, and 30 μm, respectively. Since the various process parameters and hatching strategy can affect the properties of the part, the same conditions for whole specimens were applied, except the laser power. According to the microstructure map, to investigate the effect of the laser power on CP–Ti parts fabricated by the SLM, cube-shaped samples of 10 × 10 × 10 mm and rectangular-shaped samples 20 × 20 × 100 (height) mm were fabricated with a fixed scan speed of 1000 mm/s and a laser power of 120, 200, 280, 360, and 440 W, respectively. The cube-shaped samples were used for hardness, microstructure, and chemical composition analyses and the rectangular-shaped samples were used for tensile test. Hereafter, the samples fabricated with a laser power of 120, 200, 280, 360, and 440 W will be designated as LP120, LP200, LP280, LP360, and LP440, respectively.

For the SLM process, firstly, high-purity argon gas (99.999%) was introduced into the SLM chamber to remove oxygen. The oxygen concentration in the chamber was measured using a galvanic cell-type

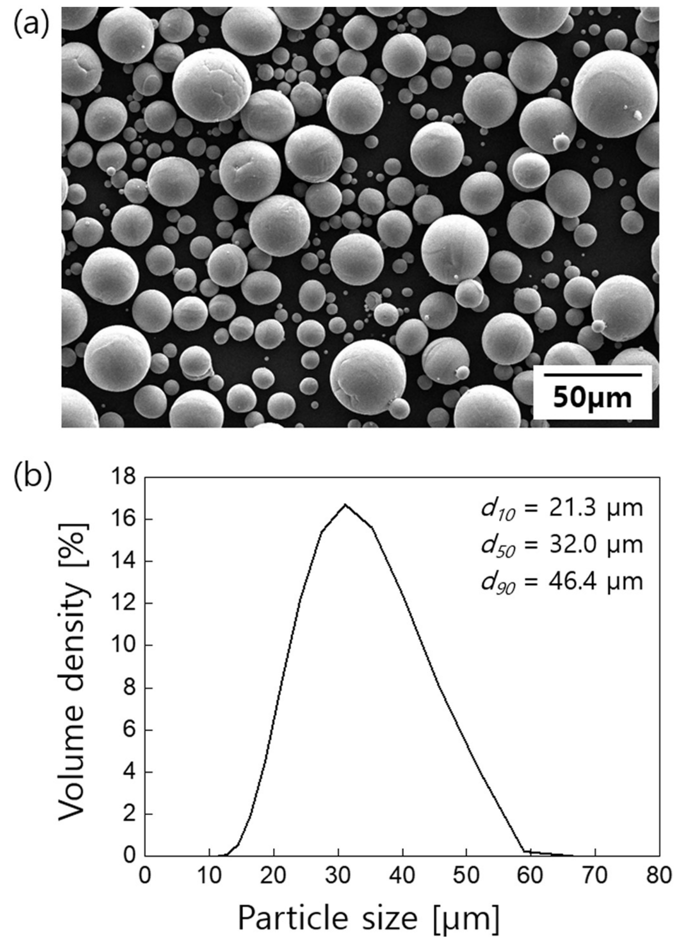

Fig. 1. (a) Particle shape of the CP–Ti powder and (b) its particle size distribution. The powder had a spherical shape and d_{10}, d_{50}, d_{90} were 21.3 μm, 32.0 μm, and 46.4 μm, respectively.

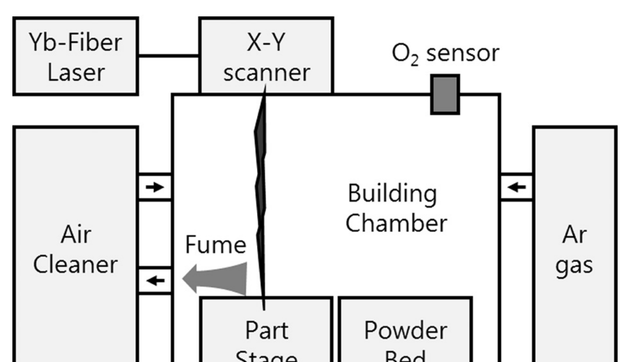

Fig. 2. The diagram of the SLM equipment that used for fabricating the SLM samples.

oxygen sensor (GS Yuasa, KE-25), which was placed on the upper side in the SLM chamber (Fig. 2). The oxygen sensor can measure the oxygen concentration in the atmosphere in the range from 0 to 100% with a ± 1% accuracy of the measured value and its response time is 15 s. The SLM process was begun after reaching 0.2% of the oxygen sensor value. In order to maintain the atmosphere in the SLM chamber during the process, argon gas was injected continuously and its flow rate was automatically controlled to maintain the oxygen concentration in the atmosphere at 0.2%.

For the analysis, the cube-shaped SLM samples were cut in half parallel to the building direction. After grinding and polishing, the