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PROPERTIES OF PURE Ti IMPLANTS FABRICATED BY ADDITIVE MANUFACTURING

This study was carried out to evaluate the aspect of microstructure and mechanical property development on additive manufactured pure Ti at elevated heat-input. For this work, pure Ti powder (commercial purity, grade 1) was selected, and selective laser melting was conducted from 0.5 to 1.4 J/mm. As a result, increase in heat-input led to the significant grain growth form 4 μm to 12 μm, accompanying with the change of grain shape, correctly widmanstätten structured grains. In addition, Vickers microhardness was notably increased from 228 Hv to 358 Hv in accordance with elevated heat-input, which was attributed to the increased concentration of oxygen and nitrogen mainly occurred during selected laser melting process.

Keywords: Pure Ti, Selected laser melting, Heat-input, Microstructure, Mechanical property

1. Introduction

Additive manufacturing (AM), which is referred to as 3D printing, has been deeply getting the limelight as a powerful fabrication technology for biomaterials, tissues, and organs due to the freeform nature in design and manufacturing [1]. In addition, AM makes it possible to develop medical devices with complex shapes and multiple materials that cannot be easily manufactured by conventional techniques such as casting and plastic deformation. Lately, selective laser melting (SLM) and electron beam melting (EBM) as AM process for biomaterials and medical devices are receiving a lot of attention due to the use of metallic based materials [1,2]. In particular, the nonferrous metals with high melting point (≥1500°C) such as pure Ti, Ti alloys and Co-Cr alloys are requested SLM or EBM for obtaining the fully melted state. However, SLM on pure Ti for orthopedic implants has been rarely reported so far.

Titanium and its alloys are widely used in aerospace, chemical and power plants, and biomaterials due to high specific strength and excellent corrosion resistance [3,4]. In particular, pure Ti is not including the toxic elements such as Ni, Al and V for human body so that it is attractive to apply for the biomaterials and medical devices [5,6]. Moreover, its mechanical properties are suitable for the in-vivo materials like the orthopedic implants. However, the researches on pure Ti applying SLM have been rarely reported. Therefore, this work was carried out to evaluate the microstructure and mechanical property of pure Ti implants fabricated by SLM process.

2. Experimental

Spherical commercial purity (CP) Ti powder (grade 1) as an initial material was selected, and selected laser melting (SLM) method as an additive manufacturing process was introduced. In order to investigate the effect of elevated heat-input during SLM process, cube-shaped samples of $10 \times 10 \times 10$ mm^3 were fabricated under the conditions of 0.5, 0.9 and 1.4 J/mm by Farsoon Technologies' FS271M model, respectively. Also, to obtain the high purity specimens, melting chamber was sustained with the high purity argon gas. Hereafter, in order to analyze the composition of the initial powder and the SLM specimens, the concentration of oxygen and nitrogen were analyzed using an O/N analyzer (LECO, 763 series) and the concentration of carbon and sulfur were analyzed using a C/S analyzer (LECO, 744 series).

In order to evaluate grain boundary characteristic distribution (GBCD) such as grain shape, size and misorientation angle, electron back-scattering diffraction (EBSD) analysis, a Hitachi 4300SE field-emission-gun scanning-electron microscope (FEG-SEM) equipped with a TSL-OIM^{TM}, was introduced. For this work, specimens were machined by 5 mm × 5 mm in size, mechanically ground and then electro polished under 20 V and –40°C conditions on surface by the solution comprised with 100 ml perchloric acid and 900 ml methanol. Sample surfaces were then analyzed by orientation image mapping (OIM) system incorporated with SEM. For the evaluation of mechanical property, Vickers microhardness was employed and carried out on the parallel section of the building direction with a load of 1.96 N and a dwell time of 10 s.

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3. Results and discussion

Shape and size distribution on CP-Ti powder used in this work is shown in Fig. 1. Spherical CP-Ti powder ranging between 10 and 60 μm was introduced as an initial material for manufacturing the cube shaped CP-Ti specimens by the SLM, as shown in Fig. 1(a). The particle size distribution of the powder was determined using particle size analyzer (Malvern, Mastersizer 3000). Particle size distribution is indicating that d_{10}, d_{50}, d_{90} were 21.3 μm, 32.0 μm, and 46.4 μm, respectively, as shown in Fig. 1(b).

Orientation image maps of SLM specimens fabricated by elevated heat-input are shown in Fig. 2. Specimen with heat-input of 0.5 J/mm was composed of grains ranging between 2 and 16 μm with an average size of 7.6 μm, comparatively equiaxed grains, as shown in Fig. 2(a). Increase in heat-input led to the increased grain size and the change of grain shape. As a result, specimens with heat-input of 0.9 and 1.4 J/mm showed more coarsened grains with an average size of 10.1 and 14.2 μm relative to the 0.5 J/mm, as shown in Figs. 2(b) and 2(c), respectively. In particular, the widmanstätten structured grains like a lath shape were significantly increased in accordance

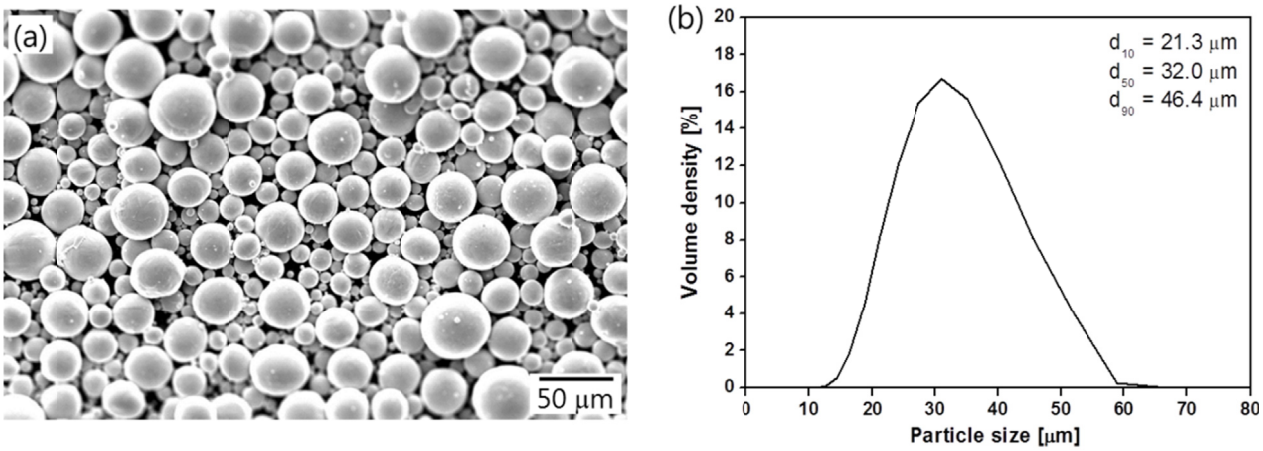

Fig. 1. (a) Particle shape of the CP-Ti powder and (b) its particle size distribution

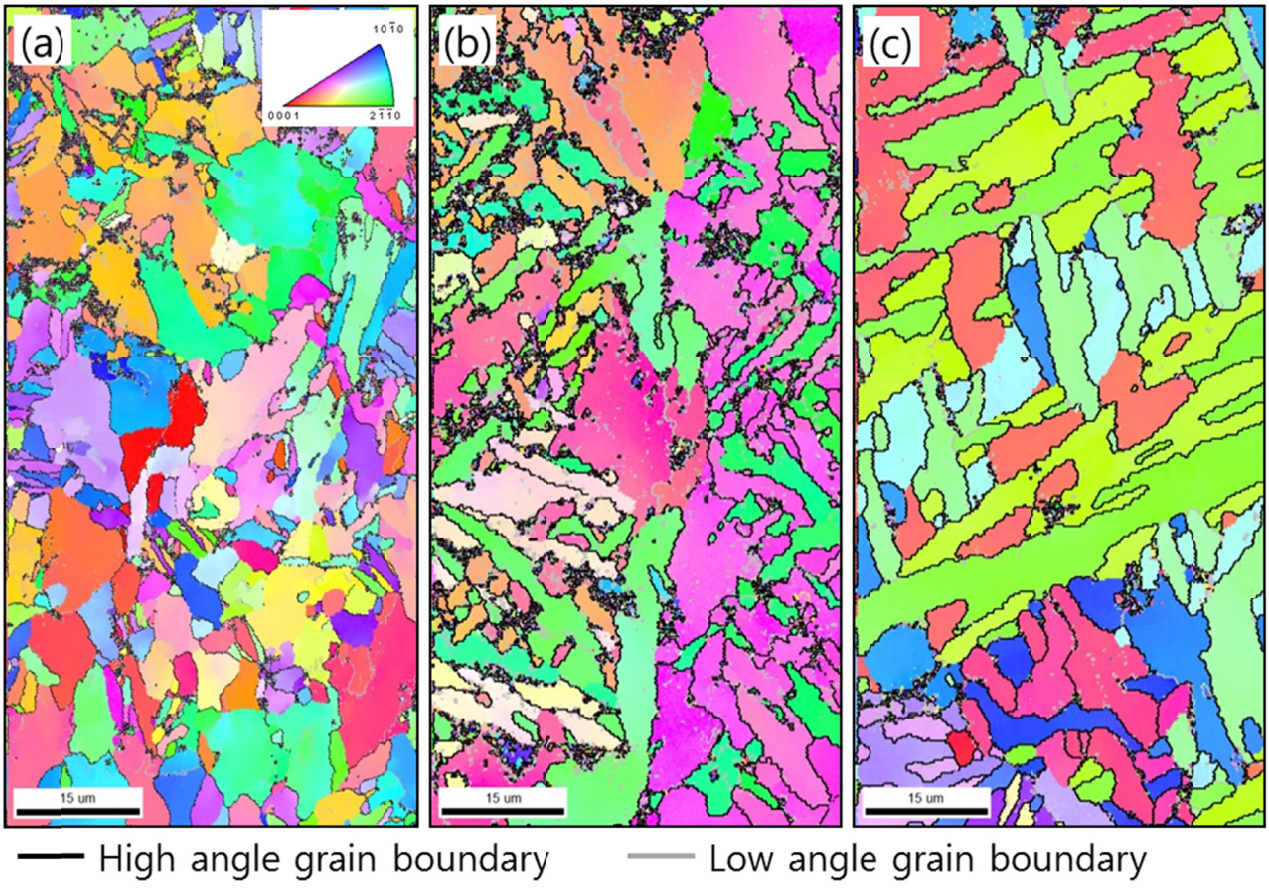

Fig. 2. Orientation image maps of SLM processed pure Ti at elevated heat-input of (a) 0.5 J/mm, (b) 0.9 J/mm and (c) 1.4 J/mm

with an elevated heat-input, as shown in Figs. 2(a), 2(b) and 2(c), respectively.

Misorientation angle distribution, applicable to evaluate the sound solidification and crystallization, on SLM specimens observed at Fig. 2 is shown in Fig. 3. At the specimen with heat-input of 0.5 J/mm, the high angle grain boundaries among whole grain boundaries were occupied approximately 78%, as shown in Fig. 3(a). Increase in heat-input was contributed to the significant increase in 60° distribution among the high angle grain boundaries, which was identified by widmanstätten structured grains. Consequently, its fraction was increased to 19% at 0.9 J/mm and 47% at 1.4 J/mm, as shown in Figs. 3(b) and 3(c), respectively. Meanwhile, low angle grain boundaries were decreased from 22% at 0.5 J/mm to 15% at 1.4 J/mm, respectively. Change in average grain size by elevated heat-input is shown in Fig. 4(d), as mentioned above, its size was notably increased in accordance with elevated heat-input.

Changes in Vickers microhardness and concentration of oxygen and nitrogen during SLM are shown in Fig. 4. Vickers microhardness was notably increased with elevated heat-input

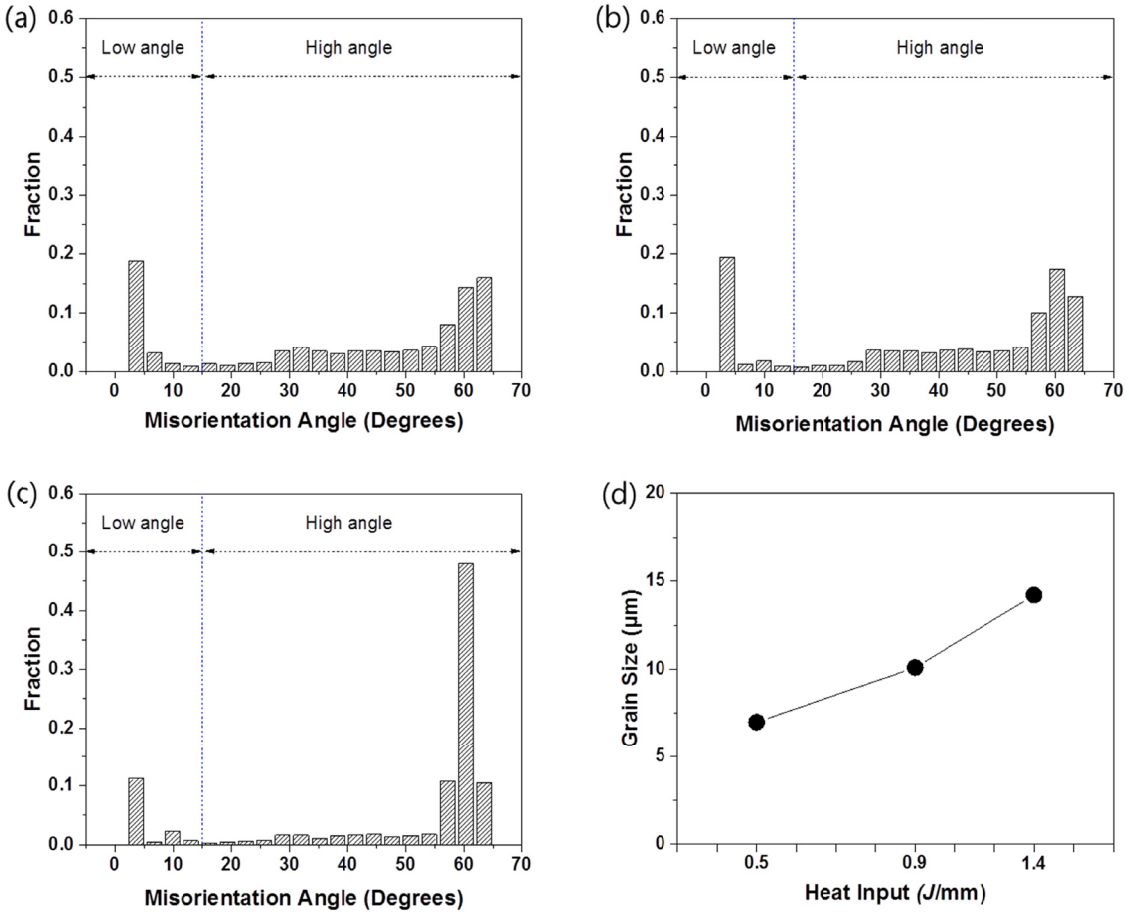

Fig. 3. Change in (a-c) misorientation angle distributions and (d) average grain size of SLM processed pure Ti; Misorientation angle distributions at elevated heat-input of (a) 0.5 J/mm, (b) 0.9 J/mm and (c) 1.4 J/mm

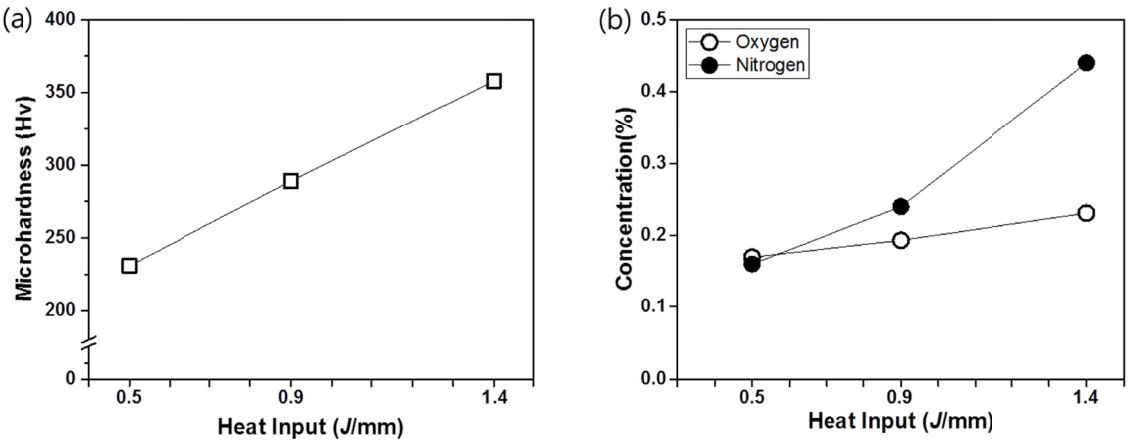

Fig. 4. Change in (a) Vickers microhardness and (b) concentration of oxygen and nitrogen by increased heat-input

in spite of increased grain size, consequently, its values showed 228 Hv (0.5 J/mm), 285 Hv (0.9 J/mm) and 358 Hv (1.4 J/mm), as shown in Fig. 4(a). In case of concentration of oxygen and nitrogen, increased heat-input was contributed to the significant concentration increase during SLM, as shown in Fig. 4(b). Consequently, oxygen concentration was increased from 0.17 at 0.5 J/mm to 0.22 at 1.4 J/mm, respectively. In particular, nitrogen concentration showed notably increased values from 0.16 at 0.5 J/mm to 0.44 at 1.4 J/mm, approximately 2.7 times higher value, respectively.

Grain size was significantly increased with elevated heat-input accompanying with the change of grain shape during SLM process, consequently, the widmanstätten structured grains with an average size of 14.2 μm was fully distributed at the higher heat-inputted specimen (1.4 J/mm). These widmanstätten structured grains were mainly formed during the cooling stage from the melting [2,3,7]. Moreover, fully melted pure Ti with higher heat-input could be formed the coarsened grains due to the increase in holding time at high temperature. This study clearly showed the densely distributed widmanstätten structured grains with grown size at higher heat-input, as shown in Fig. 2.

Concentration of oxygen and nitrogen was notably increased in accordance with elevated heat-input. Generally, SLM typed AM for pure titanium is accompanied with significant higher heat-input (≥2000°C) relative to the melting point (1668°C), which results in the increase in holding time at high temperature during SLM process [2,4]. In addition, the higher heat-input makes a broader heat-affected-zone relative to the lower heat-input, which could promote the accumulation of oxygen and nitrogen concentration due to the increased activation of interstitial atoms. This study directly showed the increased concentration according to the elevated heat-input, which affected the significant increase in mechanical property in spite of the grain coarsening, correctly solid solution strengthening. Consequently, Vickers microhardness indicates 50% higher values at higher heat-input (1.4 J/mm) when compared to the lower heat-input (0.5 J/mm), as shown in Fig. 4. Therefore, with the increase in oxygen and nitrogen concentration in titanium, the hardness increased rapidly by solid solution strengthening.

4. Conclusions

SLM on pure Ti was successfully carried out, consequently, the elevated heat-input led to the increase in grain size from 7.6 μm to 14.2 μm, accompanying with the change of grain shape. Accordingly, widmanstätten structured grains were gradually increased in accordance with elevated heat-input. In addition, Vickers microhardness was significantly increased from 218 Hv to 358 Hv with elevated heat-input, which was attributed to the increase in oxygen and nitrogen concentration during SLM. In other words, the outstanding increase in interstitial atoms on pure Ti is dominantly influenced at increase in microhardness. Therefore, at least, a SLM process of an AM on pure Ti must consider the effect of oxygen and nitrogen concentration which can directly affect in mechanical properties.

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