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| 学 位 論 文 題 目 | A Study on the Fabrication of Porous Ti-6Al-4V Alloys Using Laser Powder Bed Fusion Technology （レーザ粉末床溶融結合方式 （LPBF）による多孔質 Ti-6Al-4V 合金の造形） |
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# 論文内容要約

Porous Ti6Al4V alloys fabricated using the LPBF technique have been widely used for implants, because the introduction of pores effectively reduces the elastic modulus of implants, overcoming the general stress-shielding problem caused by a much larger elastic modulus than human bone. Further, pores exposed on the surface of the implant have been proven to allow cells to grow inside, corresponding to a high biocompatibility and an osseointegration strength. To comprehensively investigate pore formation and resulting performance variations, the study was conducted from the powder spreading process, LPBF-manufactured monolayers and bilayers, and LPBF-manufactured samples.

The study involves 5 chapters in all. Chapter 1 simply introduces commonly used materials and fabrication methods of metallic implants, and their general requirements．Chapter 2 describes the influence of packing density on the quality of LPBF-manufactured samples, and a suitable layer thickness value was recommended to produce porous materials for high-performance implants. By using the recommended layer thickness, Chapter 3 prepare LPBF-manufactured monolayers and bilayers to investigate their surface morphology (closely related to inner pores of a sample) and wettability (a material property related to the biocompatibility), as well as the control methods. The scanning speed was verified to be effectively in controlling surface morphologies of LPBF-manufactured samples, which are expected to form different inner pores after stacking multiple layers. Therefore, samples with different porosities were produced by changing laser scanning speed in Chapter 4. Chapter 4 focuses on the mechanical and biomedical performance of these porous LPBF-manufactured samples. Both mechanical properties and biocompatibility of LPBF-manufactured samples were improved due to the existence of pores when working as bioimplants. Chapter 5 summarizes the whole study.

In chapter 2, simulations and physical samples of a roller-based powder spreading system are used investigate the effects of various parameters on the quality of powder layers with regards to void fraction. Simulations are used to determine the effect of gap height and substrate type on void fraction and powder dynamics at the particulate scale, while physical samples are non-invasively studied to determine the effect of layer thickness. The conclusions are as follows:

1) It is found that a gap height larger than the maximum particle diameter is required to achieve a full bed coverage of the particle layer. Furthermore, the void fraction decreases rapidly when the gap height is greater than (but less than double) the largest particle diameter. Further gap height increase only slightly decreases the void fraction for powder-on-substrate deposition and seems to increase the void fraction for powder-on-powder deposition, which we have attributed to increased weight from the new powder layers and a lack or resistance from the underlying layers due to their mobility.
2) CT scans of physical samples show that void fraction increases slightly as the layer thickness increases from 30 to 60 mm, which agrees well with simulation results for powder-on-powder deposition for gap heights of 100–250 mm because they shared the same relationship between the layer thickness and average particle diameters. A slight correlation between the void fraction of powders and sintered areas demonstrates that, although powder layer quality may influence sintered quality, energy density has an overriding effect.
3) Further simulations of the spreading of particle layers on a separate substate bed and substrate combinations show that the void fraction of particles spread on a particle bed is lower than that spread on a separate solid substrate, but that the void fraction of a particle layer on top of an adjacent solid substrate is the lowest of either. It is concluded that the mobility of particles in the loose particle bed facilitates a "densification," but, at the cost of the particle layer on top of its adjacent solid substrate (which represents the sintered sections of the previous layer). Although the denser powder may better support the part, it may also contribute to increases in the part's porosity.
4) Considering its application in medical field, larger porosity is preferable, but a layer thickness of 60 um is easy to cause molding failure especially for samples with larger height. Thus, the layer thickness of 45 um was adopted for further investigation in controlling porosity of as-built LPBF-manufactured samples.

In chapter 3, monolayer and bilayer LPBF samples were characterized as the surface parameters Sa and Sz, and the surface roughness factor $r_{surf}$ . Obviously, both the laser scanning directions and the number of laser scans affected the morphologies of these LPBF surfaces, but their Cas did not changed a lot. However, the Cas of monolayer surfaces always were higher than that of bilayer surfaces. The detailed conclusions were summarized as follows:

1) The monolayer surface generated by the laser scan in the direction y was rougher than that in the direction x.

The simulated work on the powder spreading quality verified a rougher particle distribution in the direction y, which affected the surface quality of the monolayer surface by the leading difference in laser absorption .

2) The bilayer surface xx was rougher than yx at high laser energy but less rough at low laser energy. More boundary pores on the 1st layer were covered by the melting of the 2nd layer, leading to a better surface of combination yx. As for the sample S400, many pores appeared on the laser tracks and yx become rougher than xx due to the rougher particle distribution in the direction y .

3) The Cas of all monolayer and bilayer surfaces decreased as time went by, and no obvious difference between monolayer surfaces y and x, and bilayer surfaces yx and xx. But the Cas of monolayer surfaces were larger than that of bilayer surfaces on the same day due to the anti-penetration of the dense base plate.

4) The number of laser scans significantly affected the morphology of LPBF samples, especially the pore distribution. Adding another laser scan allowed materials to be sufficiently melted and expand in the height due to the surface tension, resulting in a larger surface roughness factor $r_{surf.}$ It also allowed materials connection, which was beneficial in forming a better surface .

5) As time went by from day 1 to day 31, the decrease in Cas of 1 scan surfaces larger than that of 2 and 4 scans surfaces, indicating that the increasing laser scan accelerated surface stabilization. The final Cas of monolayer surface can be ordered as 1 scan, 2 scans, and 4 scans from large to small, but the Cas of bilayer surfaces at 4 scans were slightly larger than that of 2 scans.

6) Overall, the surface roughness factor r increased as the laser scanning speed, which will be used as a parameter to fabricate LPBF-manufactured samples with different porosities to evaluate their mechanical and biomedical performance.

In Chapter 4， pores derived from gaseous atmosphere and condensation in the LPBF process were utilized to generate different porous structures in the study. Uniform porous LPBF samples were prepared by changing the laser scanning speed, whereas gradient samples were composed of sections sintered at different laser speeds. Based on the porosity results from CT images, compression test, 30° bending and fatigue tests, and in vitro experiment, the following conclusions could be drawn:

1) LPBF samples with porosity between 8.59% and 31.9% were fabricated by changing the laser scanning speed, which provided a design reference for the gradient structure . Combined with the surface morphologies in Chapter 3, it does verify that a rougher surface of monolayer or bilayer corresponds to a more porous sample.

2) The maximum compressive force decreased from 1.5 Gpa to 0.8 Gpa, and the elastic modulus changed from 20.1 Gpa to 14.6 Gpa when the porosity increased, agreed well with the rule that a higher porosity leads to a lower elastic modulus.

3) The maximum bending force of LPBF samples was negatively related to the porosity and significantly lower than that of bulk commercial Ti6Al4V, indicating that pores reduce the material strength.

4) Four loads were applied to conduct 30° fatigue tests. The number of cycles to failure decreased as the load increased for the LPBF samples. Further, the fatigue performance exhibited a negative correlation with porosity under low cyclic loading .

5) LPBF surfaces with varied porosities of 0.1254, 0.1661 and 0.2255, and average pore radii of 26.9953 μm, 28.2702 μm and 34.4856 μm. Cells cultured on these LPBF surfaces increased as time went by. On the 3rd day, the number of cells on these LPBF samples fluctuated but didn't exceed that on the control surface. Cells on S430 exceeded that on the control surface on the 7th day, indicating that pores facilitated biocompatibility .

6) Although pores in the study did not significantly promote the cell proliferation in the study, the cell invasion into pores was confirmed and was expected exhibit high osseointegrative strength as an implant . Further, the slight changes in cell proliferation coincide with the changes in contact angle under changing laser scanning speeds.

In sum, the study innovatively compares the void fraction of particles, powders, and sintered parts to reveal their relationships, both numerically and experimentally. The layer thickness was verified to affect the powder spreading quality and then influence the sintered quality. Considering the requirements of the biomedical application, the layer thickness of 45 μm was recommended to produce different porous samples for improving the performance. Next, LPBF-manufactured monolayers and bilayers reveal the influence of laser scanning strategies on the pore formation process. Not only do laser direction and the number of laser scans contribute to the pore distribution, but also the laser scanning speed shows a more significant and regular effect on surface morphology, telling by the surface roughness factor rsurf. According to the laser stacking process, a rougher surface tends to form samples with more inner pores. Therefore, the laser scanning speed was used to generate samples with changing pore distribution for testing their performance. At last, LPBF-manufactured Ti6Al4V samples with changing porosities were proved to have stratified elastic moduli and biocompatibility as bioimplants. But fatigue strength deserves further strengthening for a longer reliable time.

The whole study is focused on the mechanical properties and biocompatibility of LPBF-manufactured porous materials. When it comes to the actual application, samples are always with complex geometries, which may bring difficulty in the LPBF technique, and the enlarged dimension may affect the performance distribution of the entire product. Further, the corrosion resistance and the antibacterial also deserve further verification for improving the performance of these LPBF-manufactured samples.