Research Article

Fabrication of Bioactive Porous Ti Metal with Structure Similar to Human Cancellous Bone by Selective Laser Melting

D. K. Pattanayak, T. Matsushita, H. Takadama, A. Fukuda, M. Takemoto, S. Fujibayashi, K. Sasaki, N. Nishida, T. Nakamura, and T. Kokubo

Address correspondence to D. K. Pattanayak, deepak@isc.chubu.ac.jp

Received 11 November 2010; Accepted 2 December 2010

Abstract Porous titanium (Ti) metal with a structure similar to that of human cancellous bone was fabricated by selective laser melting (SLM) process. SEM observation showed that the core part of the walls of the porous body was completely melted by the laser beam and weakly bonded with small Ti particles on its surface. These Ti particles were joined with the core part by heating above 1000 °C, with remaining micro cavities on their surfaces. Tensile strength of the as-prepared solid rod was 530 MPa and gradually decreased with increasing temperatures to 400 MPa at 1300 °C, whereas its ductility increased with increasing temperatures. NaOH treatment formed fine network structure of sodium hydrogen titanate (SHT) on the walls of the porous Ti metal. The SHT was transformed into hydrogen titanates by HCl treatment and finally anatase and rutile by the heat treatment. Thus treated porous Ti metal formed apatite on its surface in simulated body fluid (SBF) within 3 days.

Keywords selective laser melting; porous Ti metal; NaOH-HCl treatment; bioactivity; simulated body fluid

1 Introduction

Selective laser melting (SLM) is a convenient process for fabricating metallic components of complicated internal structure with high accuracy and minimum defects in one step [2,3]. In this process, laser beam driven by computer is scanned on a sheet of metallic powders to melt a part of powders. Fresh powders were spread over the solidified layer and again laser beam melt the second layer. As a result, 3D structure of the metal with a structure analogous to the 3D image in computer is formed.

In the present study, SLM technique was used to fabricate porous Ti metal with structure similar to human cancellous bone. The effect of heat treatment temperatures on morphology and mechanical properties were evaluated.

The effect of chemical treatment on its apatite-forming ability in SBF was evaluated.

2 Materials and methods

A porous Ti metal with a structure similar to that of human cancellous bone was prepared from Ti metal powders (grade 2) below 45 μm in size by SLM process. They were subjected to heat treatments at different temperatures from 700 °C to 1300 °C in argon atmosphere for 1 h. Their surface morphologies were observed under a field emission scanning electron microscope (FE-SEM; Hitachi S-4300, Japan). As a reference, solid rod 5 mm in diameter was prepared by the same SLM process, and the effects of heat treatment on its mechanical properties were investigated.

The cancellous Ti metal which was fabricated by SLM process and heat-treated at 1300 °C for 1 h in argon atmosphere was soaked in 5 M NaOH solution at 60 °C for 24 h and in 0.5 mM HCl solution at 40 °C for 3 h. It was heat-treated at 600 °C for 1 h in an Fe-Cr-Al electric furnace in air atmosphere. Surface morphology of such treated Ti metal was observed under FE-SEM, and its crystalline phases were identified by Raman spectroscopy (FT-Raman; LabRAM HR-800, Horiba Jobin Yvon, France). Thus, the treated Ti metal was soaked in SBF [1] for 3 days and the apatite-forming ability was observed under FE-SEM.

3 Results and discussion

Figure 1(a) is a schematic representation of an SLM process. After examining effects of laser power and scanning speed on microstructure and density of the product, they were selected 117 W and 227.5 mm/s respectively in the following experiments. Figure 1(b) is an example of porous Timetal with a structure similar to that of human cancellous bone produced by SLM process.

¹Department of Biomedical Sciences, College of Life and Health Sciences, Chubu University, Aichi 487-8501, Japan

²Department of Orthopaedic Surgery, Graduate School of Medicine, Kyoto University, Kyoto 606-8507, Japan

³Sagawa Printing Co., Kyoto 617-8581, Japan

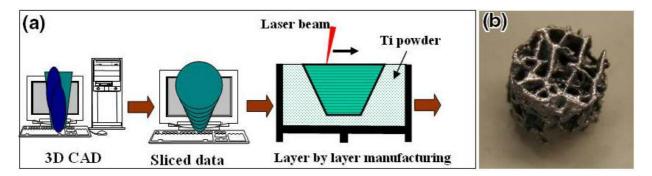


Figure 1: (a) Schematics of selective laser melting processing and (b) prepared porous Ti with a structure similar to that of human cancellous bone.

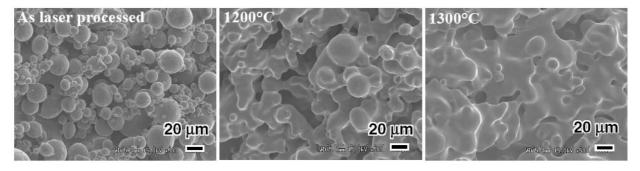


Figure 2: FE-SEM photographs of surfaces of porous Ti metal as fabricated by SLM process and those heat-treated at 1200 °C and 1300 °C in argon atmosphere.

Figure 2 shows the FE-SEM photographs of surfaces of the porous Ti metal as-prepared and those subjected to heat treatments. It can be seen from Figure 2 that large numbers of partly melted particles were adhered to the surface of the porous body. These particles were sintered and bonded to the body forming cavities on the walls by heat treatment above 1200 °C.

Figure 3 shows the mechanical properties of rod specimen prepared by SLM and those subjected to heat treatments at different temperatures. It can be seen from Figure 3 that, tensile strength of the as-prepared rod was 530 MPa and gradually decreased with increasing temperatures of the heat treatment to 400 MPa at 1300 °C, while elongation of asprepared specimen increased with increasing heat treatment temperatures up to 1300 °C from 15% to 30%. The optical photograph showed that the as-prepared specimen consisted of fine grains and they gradually increased in size with heat treatment temperatures as shown in Figure 4. The changes in mechanical properties of the rod with the heat treatment are attributed to the change in the grain size. The tensile strength and elongation of the Ti rod which was prepared by SLM process and heat-treated at 1300 °C are higher than those of as-cast Ti metal (259 MPa, and 12%) [5].

Figure 5(a) shows FE-SEM photographs of porous Ti metal which was heat-treated at $1300\,^{\circ}\text{C}$ and subjected to NaOH, HCl and heat treatments. A fine network structure

was formed on the surface of Ti metal by the NaOH, HCl and heat treatments.

Figure 6 shows Raman spectra of porous Ti metal which was heat-treated at 1300 °C and subjected to NaOH, HCl and heat treatments. A sodium hydrogen titanate was formed on the walls of the porous Ti metal. This phase was transformed in to hydrogen titanate by the HCl treatment and converted to anatase and rutile by the heat treatment.

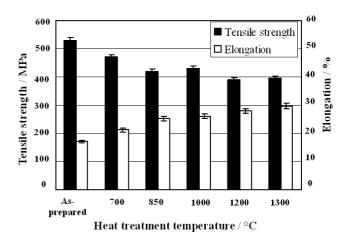
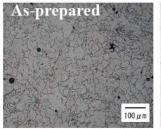


Figure 3: Variation of mechanical properties of solid rod prepared by SLM process with heat treatment temperatures.



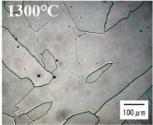


Figure 4: Optical photographs of Ti metal as-prepared by SLM process and that heat-treated at 1300 °C for 1 h in argon atmosphere.

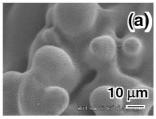




Figure 5: FE-SEM photographs of porous Ti metal which was heat-treated at 1300 °C and subjected to (a) NaOH, HCl and heat treatments, and (b) that subsequently soaked in SBF for 3 days.

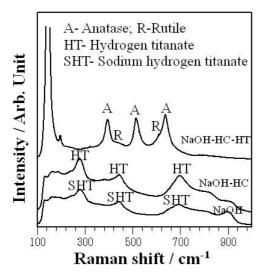


Figure 6: Raman spectra of cancellous Ti metal subjected to NaOH, HCl and heat treatments.

Figure 5(b) shows FE-SEM photographs of porous Ti metal soaked in SBF for 3 days after the NaOH, HCl and heat treatments. It can be seen from Figure 5(b) that walls of the porous body were covered with apatite within 3 days soaking in SBF. This indicates that even porous Ti produced by SLM process also gives osteoconductivity and osteoinductivity when it was subjected to NaOH, HCl and heat treatments [4].

4 Conclusions

Porous Ti metal with a structure similar to that of human cancellous bone was prepared by SLM and subsequent heat treatment. Bioactivity was given to those porous Ti metal by NaOH, HCl and heat treatments. Such a bioactive porous Ti metal is expected to be an artificial bone substitute.

References

- [1] T. Kokubo and H. Takadama, *How useful is SBF in predicting in vivo bone bioactivity?*, Biomaterials, 27 (2006), pp. 2907–2915.
- [2] C. Y. Lin, T. Wirtz, F. LaMarca, and S. J. Hollister, Structural and mechanical evaluations of a topology optimized titanium interbody fusion cage fabricated by selective laser melting process, J Biomed Mater Res A, 83A (2007), pp. 272–279.
- [3] G. Ryan, A. Pandit, and D. P. Apatsidis, Fabrication methods of porous metals for use in orthopaedic applications, Biomaterials, 27 (2006), pp. 2651–2670.
- [4] M. Takemoto, S. Fujibayashi, M. Neo, J. Suzuki, T. Matsushita, T. Kokubo, et al., Osteoinductive porous titanium implants: effect of sodium removal by dilute HCl treatment, Biomaterials, 27 (2006), pp. 2682–2691.
- [5] C. Y. Yen, Comparision of characteristics between hot rolled plate and wire made of titanium, Journal of the Japanese Society for Dental Materials and Devices, 21 (2002), pp. 139–145.