Fabrication of Porous Ti Structures with Nanostructures from Ti Powders by Femtosecond Laser Pulses

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The fabrication of titanium (Ti) porous structures covered with periodic nanostructures from micro Ti powders by femtosecond laser-induced sintering and simultaneous laser-induced periodic surface structures (LIPSS) are demonstrated. Different types of structures are obtained only by controlling the laser power of a femtosecond laser at wavelength 800 nm: sintered structures and sintered/melted structures with periodic nanostructures of period 550~650 nm.
DOI: 10.2961/jlmn.2015.03.0013
Keywords: femtosecond laser, sintering, melting, LIPSS

1. Introduction

A laser additive manufacturing method for metal parts fabrication, such as selective laser melting (SLM), involves the use of a high-power continuous laser beam for selectively heating powdered metallic material to a melting temperature by scanning cross-sections generated from 3D model data on the surface of a powder bed [1]. The laser-induced heat melts and joins the powder particles and thereby consolidates them, forming a solid mass.

Porous structures with an extremely high specific surface area, have received a great deal of attention in many promising applications because of their specific properties. The laser additive manufacturing method was investigated for the fabrication of metal porous structures [2] and has recently drawn attention to the applications. Heat pipes with porous wick structures [3], porous nickel-titanium biomedical inserts [4], and porous titanium scaffolds [5] have been produced by using SLM.

A thermal model for selective laser sintering of titanium powder using a pulsed laser was presented [6]. Compared to a nanosecond laser, the simulation results show that 150 ps pulse duration leads to a high peak skin temperature rise (~3700 K), which would be sufficient to melt the surface layer of powder after one pulse. Pulsed selective sintering of titanium powder with a Nd:YAG laser of pulse duration 0.1~1 μs was then shown [7]. The irradiated energy is absorbed in a narrow layer of individual particles and leads to a high temperature on the surface of the particle. After thermalization, heat flows mainly towards the center of the particle until a local steady state of the temperature within the particle is obtained. Recently, an experimental setup to observe the inter-particle necks in selective laser sintering of titanium powder by a nanosecond pulsed laser was presented [8].

Most previous studies use continuous or long-pulsed (nanosecond) lasers to investigate the powder sintering/melting process. Recently, sintered structures were built by the femtosecond laser pulse irradiation of dispensed tungsten powder (powder size < 1 μm) layers [9]. In addition to particle melting, laser-induced periodic surface structures (LIPSS) on the melted structures were shown. More recently, full melting of high-temperature powder materials (e.g. tungsten, powder size of few micros) by a femtosecond laser pulse to fabricate single-layer and multi-layer sintered structures was presented [10, 11], but LIPSS structures were not shown. In addition, femtosecond laser pulse irradiation of dispensed titanium micro-powder has undergone a few investigations.

In this study, the fabrication of titanium (Ti) porous structures covered with periodic nanostructures from micro Ti powders by femtosecond laser-induced sintering and simultaneous LIPSS is demonstrated. The deposited micro Ti powders is irradiated by a femtosecond laser pulse with linear polarization of different laser powers to form sintered/melted structures. Scanning electron microscopy (SEM) images of top-view and cross-sectional structures are presented.

2. Experiment

To disperse Ti particles by spin-coating, the suspension solution of Ti powders (average particle diameter of 40 μm) in ethanol and ethylene glycol was prepared at room temperature in an ultrasonic bath. The Ti particle suspension solution was then spun on a Ti substrate at 400 rpm and then heated on a hot plate at 100°C for 5 min. The thickness of the deposited powder layer was about 180 μm.

The Ti powder's surface was irradiated in air by a Ti:sapphire femtosecond laser with a wavelength of 800 nm, a repetition rate of 1 kHz, and a pulse duration of ~120 fs. The laser beam was linearly polarized and spatially filtered, resulting in an essentially Gaussian profile. The focus lens had a long working distance 10x objective lens with 0.26 NA. The position of the objective lens could be adjusted vertically (i.e. Z-axis). The laser beam with a defocused spot diameter (~500 μm) was focused on the top surface of the powder layer. The irradiated area was above ten times that of the particle (diameter ~40 μm).

Microstructures were produced by translating the sample by using an X-Y stage. The analyzed areas were made by scanning in an area of 3× 3 mm^2 in size. The

scanning speed was 0.025 mm/s, and the lateral displacement distance between the two adjacent scanning paths was set at 0.2 mm. The surface morphology of the sintered structures was examined by SEM (JEOL JSM-7001F).

3. Results and Discussion

Figure 1 presents the microscopic image of Ti structures. Area (A) shows the surface as-deposited powders. After the laser processing at Area (B) with a laser power of 200 mW, it can be seen that the laser irradiated surfaces appear opaque. Fig. 2 presents the magnified microscopic image of the Ti powders' surfaces (a) as-deposited powders and (b)-(d) processed with laser powers of 40, 120, and 200 mW. There was no obvious morphology difference between Figs. 2(a) and (b). At high laser powers, as shown in Figs. 2(c)(d), it can be seen that the laser irradiated surfaces appear opaque.

Figure 3 presents top-view SEM images of the Ti structure's surface irradiated by laser powers of 40, 80, 120 and 200 mW, respectively. In Fig. 3(a), sintered necks between two particles were formed and the surface of the sintered powders was not obviously changed. When the power was increased, the morphology of the powder was changed gradually. In Fig. 3 (c), the individual particle surfaces became extremely rough and some structures were formed on the sintered powder surface. The melted layers (molten liquid) of individual Ti particles caused closer binding among particles and resulted in pore size reduction. In Fig. 3(d), the powders were almost molten, and the pores among the particles almost disappeared. The surface appeared flat, and some structures were formed on the surface.

Figure 4 shows the magnified SEM images of Area (A) of Fig. 3. Periodic nanostructures were formed. The orientation was approximately perpendicular to the direction of the polarization (E) of the laser beam. From 2D fast Fourier transform (2D-FFT) analysis, it was found that the mean periods were about 550~650 nm, which were slightly less than for a laser wavelength of 800 nm.

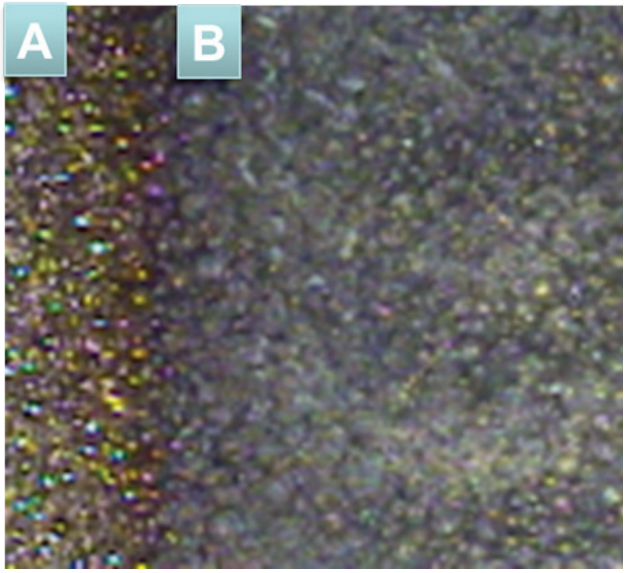

Fig. 1 Microscopic image of Ti structures: (A) before and (B) after the laser processing

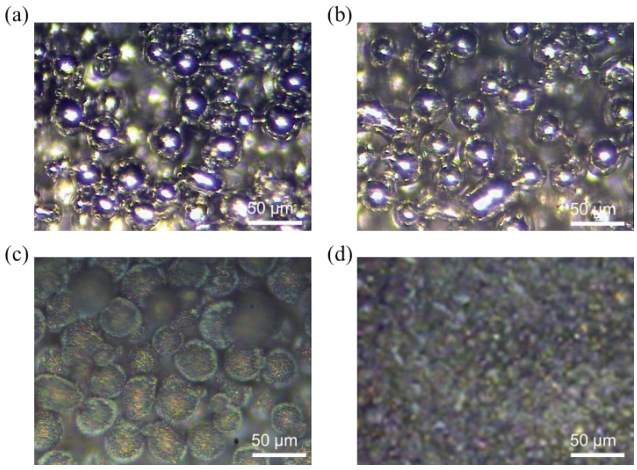

Fig. 2 Magnification of microscopic images of Ti structures (a) before and after the laser processing by (b) 40, (c) 120 and (d) 200 mW

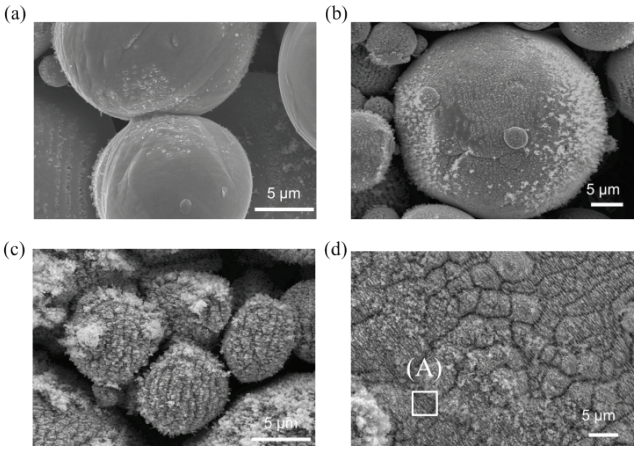

Fig. 3 Top-view SEM images of Ti structures irradiated at laser powers of (a) 40, (b) 80, (c) 120 and (d) 200 mW

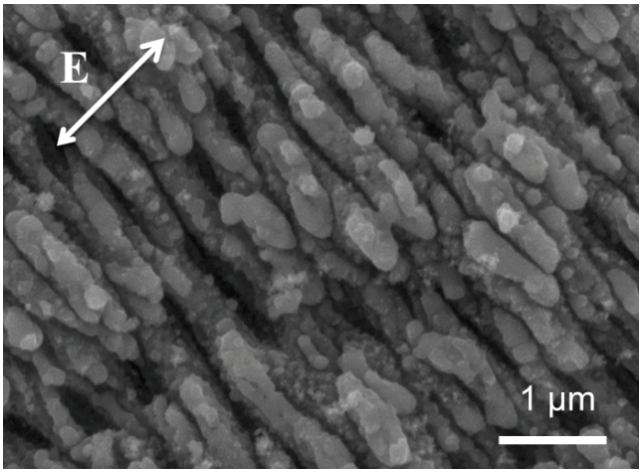

Fig. 4 Magnified SEM images of Area (A) of Fig. 3

Recently, the surface modification of Ti plates using femtosecond laser pulse was presented, such as Ti:sapphire (790 nm) [12] and Yb:KYW (1030 nm) [13]. Periodic surface structures with ~600 nm and ~820 nm periods perpendicular to the laser polarization were formed for irradiated

laser wavelengths of 790 nm and 1030 nm, respectively. The formation of periodic nanostructures on metals may be attributed to the optical interference of incident laser irradiation and the excited surface plasmon wave. When metals are irradiated by a normal incident linearly polarized laser beam, the period Λ of the nanostructures is given by [14]:

$$\Lambda = \frac{\lambda}{\eta} \quad (1)$$

where λ is the laser wavelength (in this study λ =800 nm), $\eta = \mathrm{Re}[\varepsilon/(\varepsilon+1)]^{1/2}$, is the real part of the refractive index of the air-metal interface for surface plasmons, and ε is the dielectric parameter of the metal. At room temperature, η is calculated to be ~1 at λ =800 nm for titanium [15]. From Eq. (1), Λ is calculated to be approximately the laser wavelength of 800 nm, and ~1.3 times higher than the experimental period 550~650 nm, since η may be increased when the metal is heated by a high-intensity ultrafast pulsed laser [14]. The ε of the metal can change due to the decreasing electron relaxation time $\tau_e \propto 1/{T_e}^2$, resulting from the rapid increase in electron temperature T_e by an ultrafast pulsed laser [16].

Figure 5(a) presents the low magnification of the top-view SEM images of Ti structures irradiated by a laser power of 200 mW. It can be found that the powders were almost melted and a flat surface was formed. However, as shown in Area (B), some partially melted particles were retained. Figs. 5(b)(c) present the magnified images of Areas (B) and (C), shown in Figs. 5(a)(b), respectively. In Fig. 5(b), the partially melted particle surface is covered with distorted structures. The surrounding area of the partially melted particles was composed of ring-like microstructures, with a period of around 2~3 μm. In addition, the orientations of the periodic microstructures were approximately perpendicular to the radial direction of the particle. The periodic microstructures around the partially melted areas are also shown in Fig. 5(a). It was postulated that during the laser irradiation, enhanced electric intensity (i.e. irradiated fluence) occurred in the surrounding area of the partially melted particles by multiple reflections of the particles surface. In [17], periodic microstructures with periods 1.5~2.4 μm on the Ti plate were formed using femtosecond laser pulses in a vacuum environment. Their experimental results indicate that the required laser fluence for periodic microstructures fabrication was higher than the periodic nanostructures.

In Fig. 5(c), the periodic nanostructures (similar to those observed in Fig. 4) were formed on top of the ring-like micro-structures and melted surface. The periodic nanostructures on top of the melted surface are speculated to be caused by the Gaussian intensity distributed laser beam spot. The nanostructures were formed right after the formation of the melting powder but not simultaneously. Since the central intensity of a Gaussian beam is higher than the outer part, the peak power intensity can be much higher than the melting threshold of Ti particles and melted microstructures are formed after irradiation. When the laser focus spot moves forward, the outer area with lower intensity irradiates on the microstructures and nanostructures are formed on top of them.

Figure 6 shows the cross-sectional SEM images of Ti structures irradiated by laser powers of 80 and 160 mW, respectively. The size of the necks increased with laser power. This can be attributed to the ultrafast melting and re-solidification of Ti particles induced by the femtosecond laser pulse. Previous studies show that the non-equilibrium heat transfer in the electrons and lattice of metals can be described by a two-temperature model, and the locations of the solid-liquid interface can be clearly determined [18].

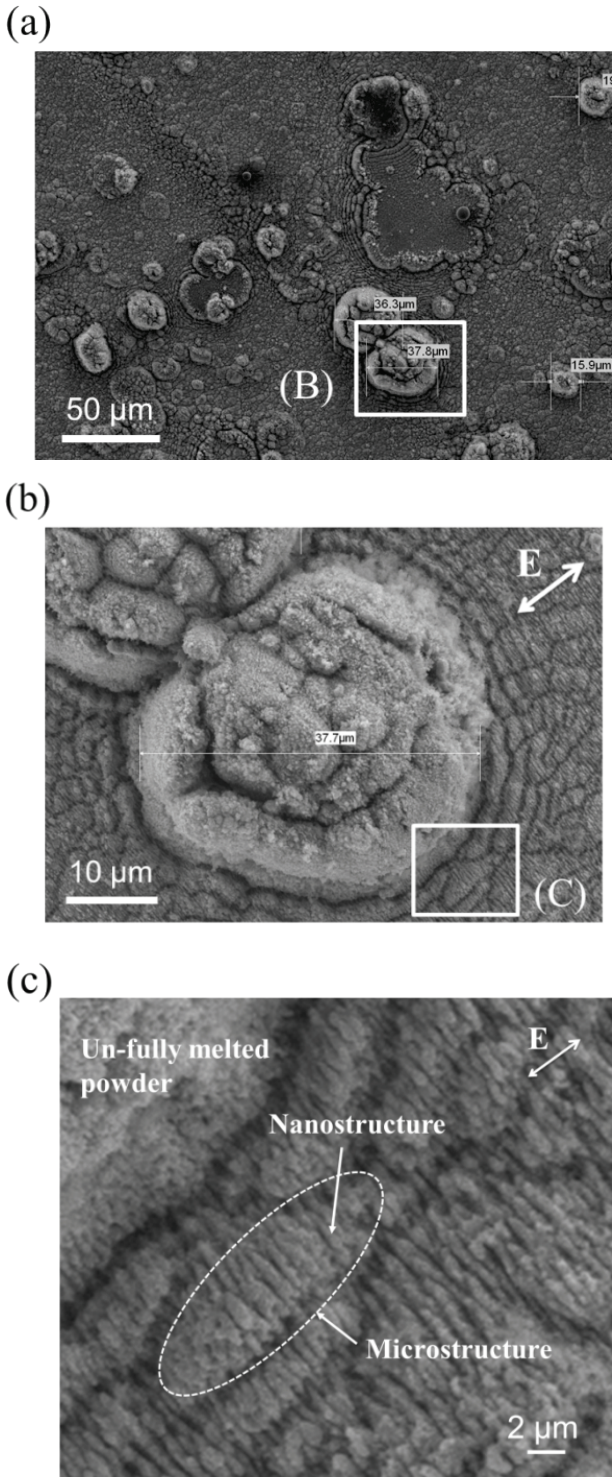

Fig. 5 (a) Top-view SEM images of Ti structures at the laser power 200 mW, (b) magnified images of Area (B), (c) magnified images of Area (C)

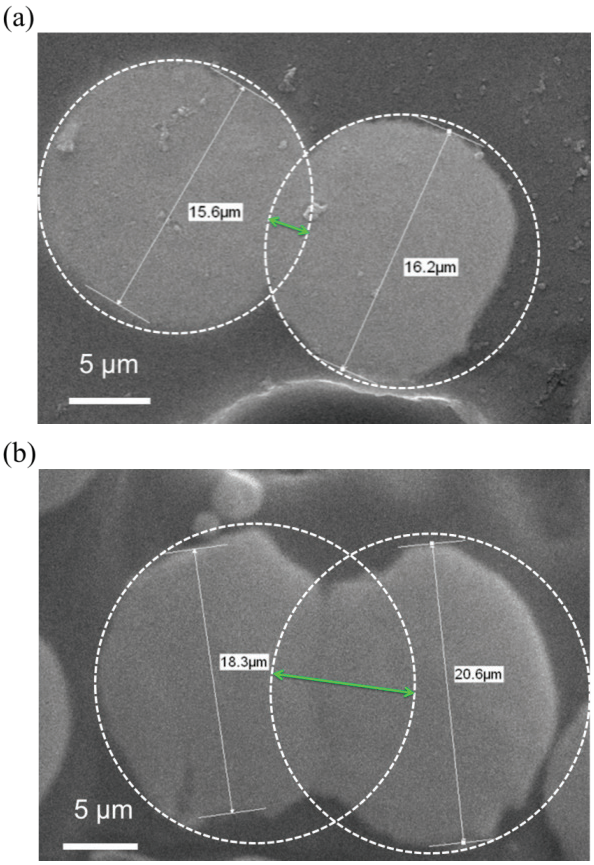

Fig. 6 Cross-sectional SEM images of Ti structures at the laser power (a) 80 and (b) 160 mW

4. Conclusions

This study reports a method of fabricating a titanium porous structure covered with periodic nanostructures from micro Ti powders using femtosecond laser-induced sintering and simultaneous laser-induced periodic surface structures (LIPSS). The experimental results have demonstrated that by controlling the laser power of a femtosecond laser at a wavelength of 800 nm, sintered structures and sintered/melted structures with periodic nanostructures of period 550~650 nm were formed.

Acknowledgements

The authors would like to thank the MOST 103-2218-E-009-025-MY2 and the Ministry of the Economic Affairs (MOEA), Taiwan, R.O.C. for supporting this research

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(Received: May 19, 2015, Accepted: November 26, 2015)