MECHANICAL PROPERTIES OF PURE TITANIUM MODELS PROCESSED BY SELECTIVE LASER MELTING

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Abstract

The influence of laser processing parameters on mechanical properties and microstructure of pure titanium models made by selective laser melting is investigated. The density of the models can reach higher than 95% under proper conditions. Although the tensile strength test shows results comparable to the wrought material, the impact and torsional fatigue strengths are low because of porosity and oxygen pick-up suggested by increasing of hardness. Hot isostatic pressing allows almost full densification and greatly improves mechanical properties.

1. Introduction

Rapid prototyping (RP) techniques are layer wise additive manufacturing processes which transform 3D CAD models into physical parts. Recently, a lot of effort has been made to realise rapid tooling (fabrication of moulds and dies for injection moulding or casting respectively) and rapid manufacturing (direct fabrication of components). Selective laser melting (SLM), selective laser sintering (SLS) and laser engineered net shaping (LENS) are some of the processes used today for direct fabrication of metal components [1-3].

The direct fabrication of metal components by rapid prototyping seems to be suitable for small or single lot production and small-sized parts of complicated geometry. The fabrication of prostheses and implants are good candidates. Titanium and its alloys are considered to be very good materials for implant because of their known biocompatibility and high strength to weight ratio [4].

In selective laser melting, a single component powder is melted and solidified by scanning of a CO_2 or an Nd:YAG laser onto a powder bed, which is different from SLS that uses metal powder encapsulated with a polymer or a combination of different metal powders of low and high melting points. In direct fabrication using single component metal powders, a balling phenomenon occurs, resulting in porosity which has bad influence on the accuracy and mechanical properties of the final product [5-6]. A second process for full densification is usually necessary.

The aim of this work is to investigate the influence of the processing parameters on the mechanical properties of models built by SLM using pure titanium powder grade 1. A pulsed Nd:YAG laser with maximum average power of 50W and maximum peak power of 3 kW was used to process titanium powders. In the present work, the density, hardness, Charpy impact energy and torsional fatigue strength are measured and optical and electronic microscopes are used for microstructure analysis.

2. Experimental Methods

Commercial pure titanium powder grade 1 (TILOP 45 supplied by Sumitomo Sitix) is used. The powder is made by IAP (Induction Melting Gas Atomising Process), which leads to spherical particles and a very low amount of interstitial impurities (O = 0.12%, H = 0.005%, N = 0.009%, C = 0.008% and Fe = 0.032% in mass). The particle diameter distribution is under 45 µm, and the average particle size is 25 µm. The apparent density of the powder is around 64% of the real density.

Figure 1 shows the selective laser melting system. A pulsed Nd:YAG laser LUXSTAR with maximum average power of 50 W is used. The maximum peak power of the pulsed laser is 3 kW, the pulse width is in the order of milliseconds and the laser beam diameter is 0.8mm at the focal point. The laser head attached to an x-y table is scanned onto the powder bed, building each layer. A piston is used to move the powder bed 0.1 mm downwards after each layer is scanned. The process is carried out in a closed chamber continuously filled with argon because of the high reactivity of titanium to interstitial elements such as oxygen, nitrogen, carbon and hydrogen.

Figure 2 shows the hatching method. One hatching cycle consists of: outline scanning; outline and x direction scanning; outline scanning; outline and y direction scanning. This pattern is repeated until the component is built completely.

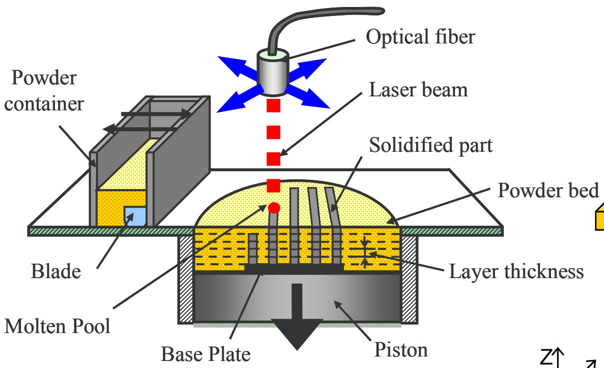


Figure 1 – Sketch of the selective laser melting machine

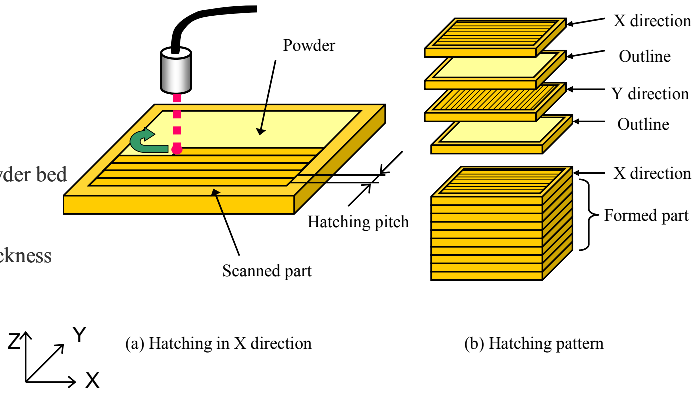


Figure 2 – Hatching method

Cubic samples were used for density and hardness measurements and were examined by optical and scanning electron microscopes, as well as by X-ray diffraction and energy dispersive spectroscopy (EDS). In order to study the influence of the laser processing parameters, peak powers of 1 kW, 0.75 kW and 0. 5 kW were used to build the specimens, while frequency (f) and average power (P_{av}) were kept at 50 Hz and 50 W respectively in all conditions. The scan speed was 6 mm/s. Density was measured by the Archimedes Principle. A micro indentation Vickers tester (Shimadzu HMV-200) was used for hardness measurement.

Specimens for impact testing were prepared following the E23-94b standard [7]. A fully reversed torsional fatigue testing was carried out at a low stress range of 70 MPa to study the influence of different surface conditions. Some samples were also heat treated (stress relieving and hot isostatic pressing) and density, hardness and fatigue strength were measured. The fatigue testing of annealed and hot isostatic pressed specimens was carried out with different stress levels.

3. Experimental Results

3.1 - Density and Hardness

Cubic specimens with lengths of 10 mm were used for density and hardness measurements. Figures 3 and 4 show the influence of peak power on the density and on the hardness of the test specimens respectively. The density is higher than 92% for all conditions. The density and hardness increase as the peak power increases. Microanalysis by EDS showed local high carbon content (around 2% in mass) probably due to debris from the plastic blade used for making the powder bed flat during recoating. The increase of the hardness suggests also oxygen pick-up. The hardness is strongly dependent on the impurities (solid solution) and precipitates in titanium.

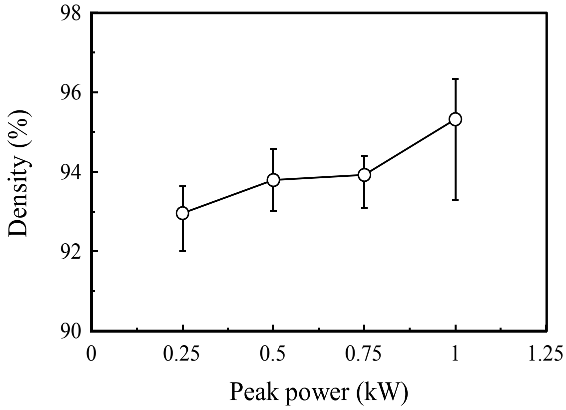


Figure 3 – Influence of the peak power and scan speed on the density; f = 50 Hz and P_{av} = 50 W

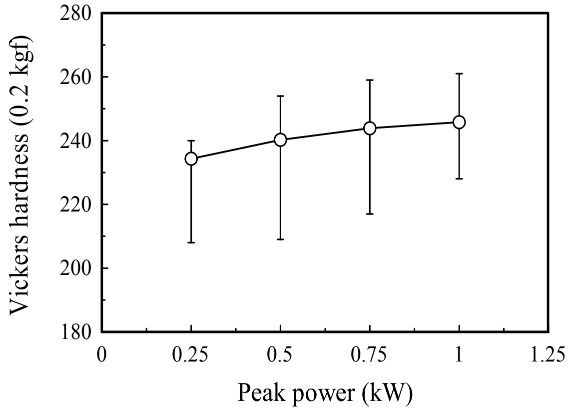


Figure 4 – Influence of the peak power on the hardness; f = 50 Hz and P_{av} = 50 W

3.2 - Impact Strength

The specimens fabricated by Selective Laser Melting initially had dimensions of 54 x 11 x 11 mm and were machined to 53 x 10 x 10 mm for impact testing. The notches were made by milling. Fifteen specimens were fabricated using different peak powers: 1 kW, 0.75 kW and 0.5 kW. It is possible to see the influence of the peak power on the impact energy in figure 5. Figure 6 shows the optical micrographs of the specimens.

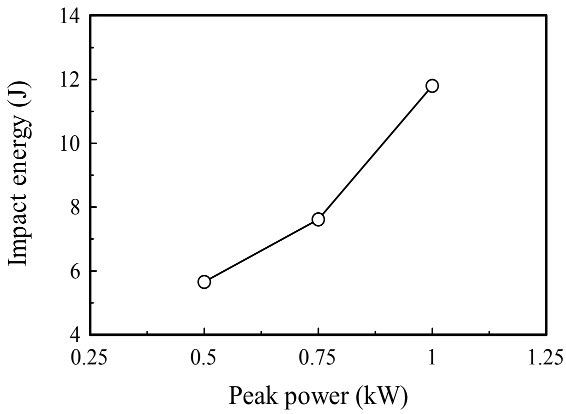


Figure 5 – Influence of peak power on impact energy; f= 50 Hz and P_{av} = 50 W

As the peak power increases, Charpy energy increases. The shape of the pores is changed by the peak power: sharp or crack like pores are common when the peak power is below 1 kW. This is probably the reason for the lower impact energy when peak power is below 1 kW.

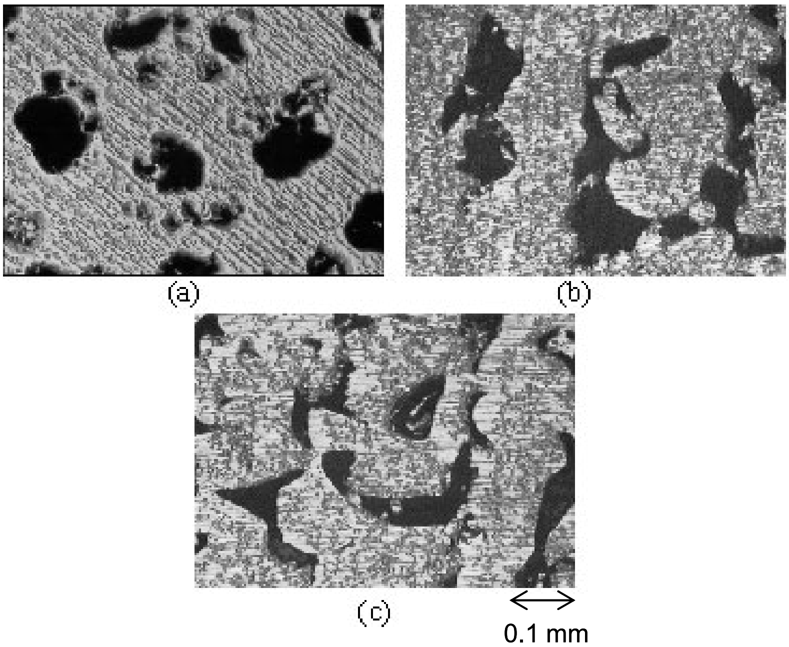


Figure 6 – Optical micrographs of specimens; f = 50 Hz and P_{av} = 50 W
(a) Peak power = 1 kW (b) Peak power = 0.75 kW (c) Peak power = 0.5 kW

3.3 - Torsional Fatigue Strength

In torsional fatigue test of square bars, the maximum shear stress occurs in the middle of the sides and is zero at the corners of the bar. The maximum shear stress can be calculated by the following expression [8]:

$$\tau_{max} = 4.81\frac{T}{a^3}$$

Where a is the length of each side of the square bar and T is the torque applied by the machine. Fully reversed torsional fatigue testing (R = τ_{max} / τ_{min} = -1) was carried out on bar specimens of 50 mm in length and square cross sections of 6 and 7 mm. The frequency of the machine is 33 Hz. The specimens were built using a peak power of 1 kW, a pulse width of 1 msec, average power of 50 kW and a scan speed of 6 mm/s.

Three different surface conditions were investigated: as-formed specimens (just after SLM processing), remelted specimens (the surface of the as-formed specimens was scanned by the laser with same forming conditions) and machined specimens. As a reference, specimens from wrought titanium grade 1 (a = 5 mm) were also tested. Figure 7 shows the SEM micrographs of the surface of each specimen. Some partially fused and sintered powder stick to the surface of the as-formed specimen. After another scanning by the laser, the sintered powder is melted and roughness decreases but some pores are left on the surface during solidification.

Machined specimens (a = 6) were submitted to stress ranges ($\Delta\tau = \tau_{max} - \tau_{min}$) from 70 MPa to 180 MPa. Two specimens were tested under each condition and the relationship between stress and number of cycles can be seen in figure 8. Three of as-formed specimens (a = 7 mm) and three of remelted specimens (a = 7 mm) were tested at a stress range of 70 MPa. The as-formed specimens were fractured after a higher number of cycles than the remelted one, but lower than the machined samples as shown in figure 8. The specimens made by using peak powers of 0.5 kW and 0.75 kW were fractured after only 10^4 cycles at 70 MPa of stress range. The specimens of the wrought material were fractured at stress range of 300 MPa after 10^7 cycles.

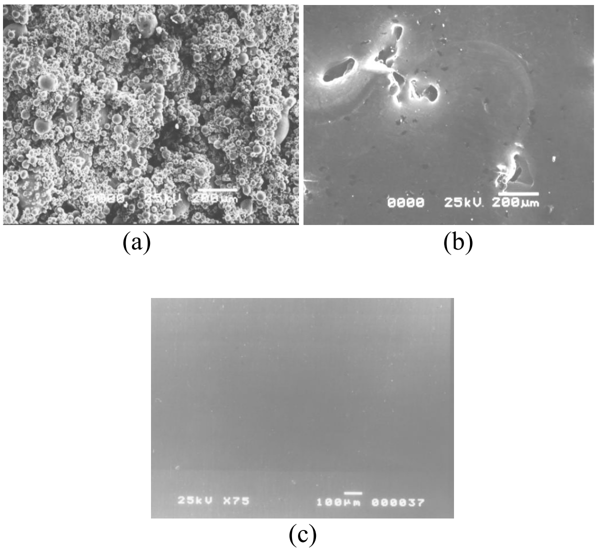


Figure 7 – SEM micrograph of the surface of the specimens (a) as-formed, x70 (b) remelted, x70 (c) machined, x75

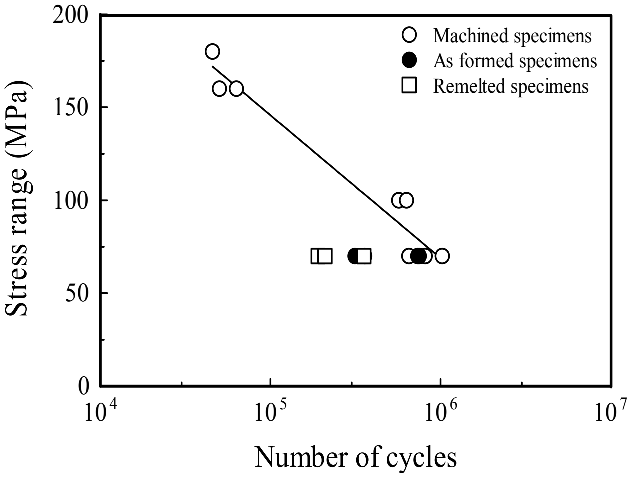


Figure 8 – Torsional fatigue testing

The low fatigue strength of the SLM specimens may be caused by porosity and impurities Residual stresses associated with laser processing may also be a reason for the low fatigue strength. Annealing and hot isostatic pressing (HIP) were used as a second process in an attempt to improve the mechanical properties of the specimens built at a peak power of 1 kW, a pulse width of 1 ms and a frequency of 50 Hz.

In annealing, five specimens were heated (10 °C/min) in a furnace with argon atmosphere and kept at 750 °C for 15 minutes. Two specimens were also hot isostatic pressed in temperature of 850 °C and 100 MPa for 1 hour.

After annealing, torsional fatigue testing was carried out. The specimens did not break up to 10^7 cycles when the stress range was 70 MPa. The same specimens were submitted to a stress range of 100 MPa and fracture occurred to 10^6 cycles. Before heat treatment, the average Vickers hardness was around 245 ±20. The hardness value and its variation on the specimens decreased after annealing. Average hardness after annealing was around 200 (HV 0.2 kgf) with very low dispersion.

The density after hot isostatic pressing was higher than 99% and hardness was the same as after annealing. A test specimen was submitted to stress ranges of 70, 140 and 160 MPa to 10^7 cycles without breaking. The optical micrograph of a hot isostatic pressed specimen and the results of torsional fatigue testing of the heat treated samples can be seen in figures 9 and 10 respectively.

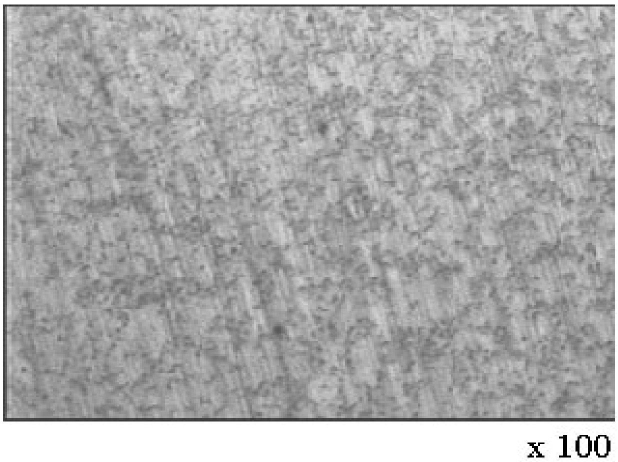


Figure 9 – Optical micrograph after hot isostatic pressing
Peak power = 1 kW

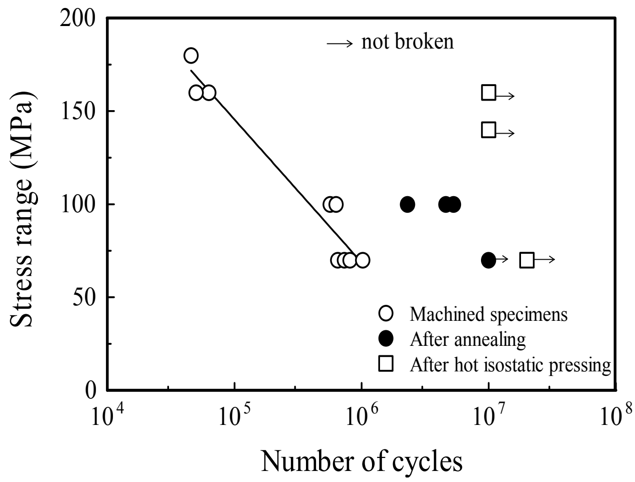
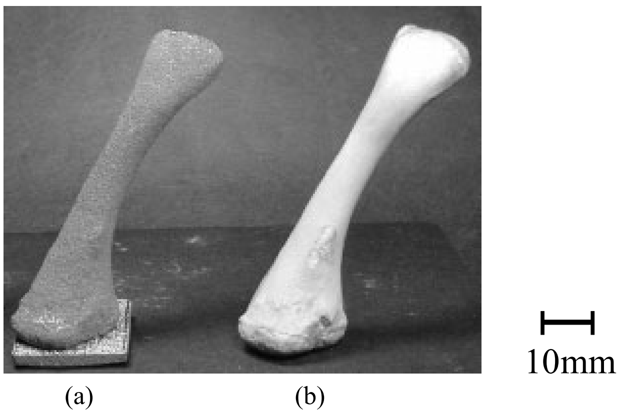


Figure 10 – Torsional fatigue testing results after heat treatment

4. Example

Several techniques can be used for obtaining the images of medical parts. Computerised axial tomography (CAT, usually to non-invasive viewing of bone structures), magnetic resonance imaging (MRI, usually to soft tissues) and reverse engineering are some examples. This data can be imported to STL standard, the extension recognised by RP machines today. Figure 11 is an example of reverse engineering acquisition of data followed by layer manufacturing processing. In order to test the free form capability of the system, a chicken bone was digitalised by a Rolland 3D modelling machine, and the cloud of points were used to generate the STL data. The laser processing parameters were a peak power of 1 kW, a pulse width of 1 ms and a frequency of 50 Hz. The scan speed was 6 mm/s.



(a) (b)

Figure 11 – Example of a part built by selective laser melting process: (a) titanium model (b) real model

5. Summary and Conclusions

(1) Density of the specimens built by SLM is higher than 90% and can reach 95% with a peak power of 1 kW, a pulse width of 1 ms and a frequency of 50 Hz;
(2) Hardness is higher than of the wrought titanium grade 1 because of oxygen pick-up and precipitation of titanium carbide. The hardness increases with peak power;
(3) Charpy impact energy and torsional fatigue strength are low because of porosity and impurities;
(4) After annealing for 15 minutes at 750 °C, hardness decreases and torsional fatigue strength slightly increases;
(5) After hot isostatic pressing, density is higher than 99% and hardness is similar to the annealed specimens. The torsional fatigue strength greatly improves and is expected to be at the level of the wrought material.

Acknowledgements

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