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Determinants of the surface quality, density and dimensional correctness in selective laser melting of the Ti-13Zr-13Nb alloy

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Abstract. Selective laser melting is widely used for custom-designed elements. Successful manufacturing depends on laser treatment parameters and material features. This research aimed to determine the effects of laser power, scan time and hatch distance on surface quality, relative density and dimensional precision for cuboids made of the Ti-13Zr-13Nb alloy. The influence of energy density, energy flux and pre-heating was seen to be decisive to different degrees for the quality of the final specimen. The results obtained were used to produce prosthetic crowns and bridges. The thermal stresses that appeared resulted in a deflection of the bridges and consequently in a change in design approach.

Keywords: selective laser melting / titanium alloys / prosthetic elements / 3D printing quality

1 Introduction

3D printing of metallic parts is usually performed by additive manufacturing with the use of a laser beam and is known as selective laser melting (SLM)/selective laser sintering (SLS) [1]. Until now, a variety of elements, machine parts, and goods have been manufactured from titanium and its alloys, especially for medicine. The obtained so far alloys are shown in Table 1 [2–10].

The dental, joint and maxillofacial implants are made either by foundry or by CNC technique. The manufacturing of medical goods by SLM is the cheapest of the above techniques as some details may be obtained at the same time. The primary efforts are focused on the process optimization. The conventional machining route resulted in higher-dimensional accuracy and smooth surface finish of prostheses, and the rapid prototyping involved lesser time and cost, demonstrating rougher prosthesis surface and lower internal porosity [1]. The high accuracy of titanium and Co-Cr scaffolds may be obtained by SLM [12], but the process optimization needs considering the vast number of parameters. The zig-zag laser path strategy for SLM made Ti-6Al-4V robust specimens resulted in better accuracy, hardness and corrosion resistance within all planes [13]. When comparing three manufacturing methods for some dental Co-Cr details, conventional casting, laser sintering and CAD/CAM milled technique, the significant differences in mechanical properties were observed, but all techniques were able to produce the
specimens satisfying the ISO standards for dental alloys [14]. The bone augmentation using pre-formed titanium mesh with bone graft and alloplastic material was made by 3D modeling and design, and manufacturing of models and pre-formed meshes by either fused deposition modeling (FDM) or direct metal laser sintering (DMLS) [15].

The SLM method was sometimes combined with other technologies to optimize the process. For the Ti-6Al-4V alloy, the selective laser melting (SLM) and 5-axis CNC milling were joined to produce aerospace parts from titanium powder with lower wastage [16]. The SLM was also combined with hot isostatic pressing (HIPS) to manufacture the Ti-6Al-4V alloy [17].

The heat treatments of SLM made parts were investigated to improve the mechanical properties. Such improvement was achieved for the Ti-6Al-4V alloy by the precision forging at 950°C [18] and by recrystallization annealing [19].

In medicine, the dental implants, maxillofacial implants, and prosthetic parts were the most often produced by the SLM method. The SLM followed by laser gas nitriding was used to make the dental implants of increased wear resistance [20]. The titanium denture base plates were CAD/CAM designed, and rapid laser formed [21]. The customized brackets were made of 316 stainless steel by SLM, using 316 L stainless steel at process parameters optimized to achieve the best shape accuracy, dimensional precision and proper density of orthodontic goods [22]. The SLM was also applied [23] to fabricate thin titanium alloy frameworks for a complete maxillary denture.

In many reports, the process parameters appropriate for the necessary quality of SLM were looked for titanium and some of its alloys. For the laser sintering of titanium, the Nd:YAG laser of power in the range 10–100 W focused for 10 s on loose Ti powder of 63–315 μm particle size [24]. The Nd:YAG laser of power in the range 10–100 W focused for 10 s on loose Ti powder of 63–315 μm particle size [24]. For the Ti-6Al-4V alloy, the best accuracy was obtained at laser power 95 W, layer thickness 30 μm, scan speed 125 mm/s, hatching space 130 μm and energy density 195 J/mm² [25]. The composite Ti-6Al-7Nb and hydroxyapatite were manufactured with a laser power of 50 W [26].

The texture and mechanical properties of SLM made titanium were dependent on laser power: at 50 W a strong-textured Ti with anisotropic mechanical properties and at 250 W a low-textured material with high strength was obtained [27].

So far, research has shown that the SLM manufacturing is suitable to produce various medical parts. Such goods are much cheaper than those made by other methods, but the accuracy of the obtained details is significantly shaped by some process parameters, individually established for any material and kind of part. This research aimed to determine the most critical process parameters and their optimal values for manufacturing the prosthetic foundations and bridges of the Ti-13Zr-13Nb alloy, nowadays the most appropriate medical titanium alloy for many applications.

### Table 1. The Ti alloys manufactured by selective laser melting.

| Alloy                | Reference |
|---------------------|-----------|
| Ti-6Al-4V-Mo        | 2         |
| Ti-24Nb-4Zr-8Zn     | 3         |
| Ti-Re               | 4         |
| Ti-5.5Al-3.4Sn-3.0Zr-0.7Mo-0.3Si-0.4Nb-0.35Ta | 5 |
| Ti-Nb               | 6         |
| Ti-15Ta-xZr (1.5, 5.5, 10.5 or 15.5Zr) | 7 |
| Ti-15Mo-5Zr-3Al     | 8         |
| Ti-5Al-2.5Sn        | 9         |
| Ti-13Zr-13Nb        | 10        |

The Ti-13Zr-13Nb powder was obtained by electrode induction-melting gas atomization (TLS-Technik, Germany). The powder grain size ranged between 50 and 100 μm. Over 90% of grains were spheroidal in shape. The chemical composition was: Zr 13.0, Nb 13.0, Fe 0.005, C 0.004, N 0.019, O 0.011 wt.% and Ti the remainder.

The project was divided into two stages. In the first stage, using model specimens, cuboids of two dimensions, 5 × 0.05 × 5 mm and 5 × 0.2 × 5 mm, were designed with the Autodesk Inventor Professional 2015 software. At the second stage, the foundations and prosthetic bridges were designed using similar CAD/CAM software based on real models obtained from scans of the mouth of some patients. Afterward, the stl. files were developed for the SLM process.

The specimens were manufactured by the Realizer SLM 100 equipment utilizing the ytterbium one mode fiber laser CW, series YLR-100-SM, at a wavelength of about 1070 nm. At the first stage, the following parameters were used: power laser 80, 90 and 100 W, laser beam distance 30, 40 and 50 μm, the exposure time for a single point 80, 90 and 100 μs. The laser beam size was set at 100 μm and the thickness of a single layer at 30 μm. Tests were carried out for all 27 combinations of the above parameters. At the second stage, based on these tests, selected laser treatment parameters were applied.

At the first stage, the surfaces of melted specimens were examined with the stereoscopic light microscope, and linear dimensions of the upper sides were measured to an accuracy of 0.01 mm. Based on that, the visible surface quality was assessed by Quality Function Deployment. Eleven features were assessed, each in a 5-point range, from one (the worst) to five (the best), by a team of two assessors that pointed out:

- difference between the thickness of the melted specimen at the center and the thickness compared to the specification of the CAD model;
- difference between the average of two upper sides of the melted specimen and the dimension according to the CAD model;
straightness of melt based on an examination of the upper surface;
bulges on the melted part based on an examination of the upper surface;
average sample length should be as close as possible to the CAD model dimensions. The differences between the length of the CAD model and mean of 10 measurements of sample length along its whole height were estimated;
sample length measured along the specimen’s upper edge (last layers of the printed sample);
visibility of layers;
unmelted powder particles on the surface;
local agglomerates of melts;
bold vertical edges;
unevenness – to assess this, features 8, 9 and 10 were taken into account.

Afterward, the density of melted specimens was measured for the best six of twenty-seven combinations of parameters. This was based on tests carried out with micro computer tomography (μCT) using the v|tome| xs 240 system from General Electric. Then two final combinations of parameters, of the highest density, qualitatively best surface and dimensions the closest to the CAD model, were investigated.

### 3 Results

The results of investigations of all combinations of three laser treatment parameters are shown in Table 2. The results are not unequivocal. The QFD analysis (Tab. 3) demonstrates the influence of all parameters on the surface quality.

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**Table 2. Dimensions of model specimens at different laser treatment parameters.**

| No. | Laser power [W] | Scan time [μs] | Hatch distance [μm] | Scan rate [m/s] | Energy density [J/mm²] | Thickness [mm] Centre | Edge 1 | Edge 2 | Length [mm] Centre | Upper edge |
|-----|-----------------|----------------|--------------------|-----------------|------------------------|-----------------------|-------|-------|-------------------|------------|
| 1   | 100             | 80             | 30                 | 0.38            | 2.67                   | 0.32                  | 0.34  | 0.34  | 5.22              | 5.23       |
| 2   | 100             | 90             | 30                 | 0.33            | 3.00                   | 0.30                  | 0.36  | 0.39  | 5.21              | 5.22       |
| 3   | 100             | 100            | 30                 | 0.30            | 3.33                   | 0.34                  | 0.43  | 0.42  | 5.14              | 5.22       |
| 4   | 90              | 80             | 30                 | 0.38            | 2.40                   | 0.31                  | 0.38  | 0.35  | 5.21              | 5.27       |
| 5   | 90              | 90             | 30                 | 0.33            | 2.70                   | 0.30                  | 0.38  | 0.35  | 5.21              | 5.23       |
| 6   | 90              | 100            | 30                 | 0.30            | 3.00                   | 0.32                  | 0.36  | 0.37  | 5.17              | 5.19       |
| 7   | 80              | 80             | 30                 | 0.38            | 2.13                   | 0.34                  | 0.43  | 0.43  | 5.20              | 5.24       |
| 8   | 80              | 90             | 30                 | 0.33            | 2.40                   | 0.31                  | 0.38  | 0.36  | 5.22              | 5.25       |
| 9   | 80              | 100            | 30                 | 0.30            | 2.67                   | 0.35                  | 0.37  | 0.36  | 5.13              | 5.18       |
| 10  | 100             | 80             | 40                 | 0.50            | 2.00                   | 0.31                  | 0.37  | 0.41  | 5.20              | 5.23       |
| 11  | 100             | 90             | 40                 | 0.44            | 2.25                   | 0.32                  | 0.39  | 0.42  | 5.20              | 5.26       |
| 12  | 100             | 100            | 40                 | 0.40            | 2.50                   | 0.31                  | 0.38  | 0.39  | 5.18              | 5.17       |
| 13  | 90              | 80             | 40                 | 0.50            | 1.80                   | 0.25                  | 0.35  | 0.37  | 5.25              | 5.26       |
| 14  | 90              | 90             | 40                 | 0.44            | 2.03                   | 0.33                  | 0.38  | 0.39  | 5.22              | 5.25       |
| 15  | 90              | 100            | 40                 | 0.40            | 2.25                   | 0.34                  | 0.40  | 0.36  | 5.15              | 5.20       |
| 16  | 80              | 80             | 40                 | 0.50            | 1.60                   | 0.28                  | 0.33  | 0.36  | 5.19              | 5.26       |
| 17  | 80              | 90             | 40                 | 0.44            | 1.80                   | 0.29                  | 0.38  | 0.39  | 5.21              | 5.26       |
| 18  | 80              | 100            | 40                 | 0.40            | 2.00                   | 0.32                  | 0.35  | 0.41  | 5.16              | 5.16       |
| 19  | 100             | 80             | 50                 | 0.63            | 1.60                   | 0.24                  | 0.34  | 0.36  | 5.24              | 5.26       |
| 20  | 100             | 90             | 50                 | 0.56            | 1.80                   | 0.28                  | 0.36  | 0.39  | 5.20              | 5.26       |
| 21  | 100             | 100            | 50                 | 0.50            | 2.00                   | 0.24                  | 0.29  | 0.26  | 5.14              | 5.15       |
| 22  | 90              | 80             | 50                 | 0.63            | 1.44                   | 0.25                  | 0.36  | 0.38  | 5.24              | 5.25       |
| 23  | 90              | 90             | 50                 | 0.56            | 1.62                   | 0.24                  | 0.32  | 0.29  | 5.20              | 5.23       |
| 24  | 90              | 100            | 50                 | 0.50            | 1.80                   | 0.27                  | 0.31  | 0.33  | 5.17              | 5.21       |
| 25  | 80              | 80             | 50                 | 0.63            | 1.28                   | 0.29                  | 0.34  | 0.35  | 5.23              | 5.23       |
| 26  | 80              | 90             | 50                 | 0.56            | 1.44                   | 0.24                  | 0.33  | 0.35  | 5.25              | 5.27       |
| 27  | 80              | 100            | 50                 | 0.50            | 1.60                   | 0.24                  | 0.27  | 0.32  | 5.17              | 5.19       |
Table 3. Results of QFD analysis at different laser treatment parameters.

| No. | Laser power [W] | Scan time [μs] | Hatch distance [μm] | QFD value |
|-----|----------------|----------------|---------------------|-----------|
| 1   | 100            | 80             | 30                  | 37        |
| 2   | 100            | 90             | 30                  | 34        |
| 3   | 100            | 100            | 30                  | 36        |
| 4   | 90             | 80             | 30                  | 28        |
| 5   | 90             | 90             | 30                  | 28        |
| 6   | 90             | 100            | 30                  | 34        |
| 7   | 80             | 80             | 30                  | 25        |
| 8   | 80             | 90             | 30                  | 27        |
| 9   | 80             | 100            | 30                  | 34        |
| 10  | 100            | 80             | 40                  | 34        |
| 11  | 100            | 90             | 40                  | 33        |
| 12  | 100            | 100            | 40                  | 36        |
| 13  | 90             | 80             | 40                  | 30        |
| 14  | 90             | 90             | 40                  | 29        |
| 15  | 90             | 100            | 40                  | 32        |
| 16  | 80             | 80             | 40                  | 31        |
| 17  | 80             | 90             | 40                  | 30        |
| 18  | 80             | 100            | 40                  | 37        |
| 19  | 100            | 80             | 50                  | 32        |
| 20  | 100            | 90             | 50                  | 28        |
| 21  | 100            | 100            | 50                  | 43        |
| 22  | 90             | 80             | 50                  | 33        |
| 23  | 90             | 90             | 50                  | 39        |
| 24  | 90             | 100            | 50                  | 41        |
| 25  | 80             | 80             | 50                  | 30        |
| 26  | 80             | 90             | 50                  | 31        |
| 27  | 80             | 100            | 50                  | 43        |

Table 4. Densities of selected model specimens expressed as the ratio of real density to theoretical density.

| No. | Relative density [%] |
|-----|----------------------|
| 3   | 96.92                |
| 12  | 96.74                |
| 15  | 97.95                |
| 18  | 98.24                |
| 21  | 97.62                |
| 24  | 97.67                |

For six selected combinations, the density of model specimens examined with the μCT is shown in Table 4. Pore size ranged between $3.5 \times 10^{-6}$ and $5 \times 10^{-8}$ mm$^3$.

Figures 1 and 2 demonstrate the differences between real and CAD model dimensions obtained using the μCT v|tome| xs system from GE and GOM Inspect software form GOM GmbH.

For the next step of the research, two sets of parameters were chosen and applied to two selected specimens, No. 18 and 18a, which had the highest relative density, and No. 21 and 21a, which had the lowest dimensional difference between the CAD model and printed out specimens and the highest surface quality. Cuboids of given dimensions were used: for specimens 18 and 21 $5 \times 0.2 \times 5$ mm, and for specimens 18a and 21a $5 \times 1 \times 5$ mm. Table 5 shows the values obtained at different laser treatment parameters and Figure 3 - the μCT image for specimen No. 21a.

Based on the results, the prosthetic elements (crowns and bridges) were made of the investigated Ti alloy at the parameters given above. The examples are shown in Figures 4–6. The dimensional quality was very good for crowns, but the bridges were substantially de

4 Discussion

The research showed that for the Ti-13Zr-13Nb alloy the best results could be obtained by taking into account some specific material features of the Ti alloy as the low thermal conductivity and distinct differences between the melting temperature of three elements that make up this alloy, i.e., Ti, Zr, and Nb.

As a rule, the highest densities of laser melted parts are obtained at the highest energy density. High energy density may be achieved at the highest laser power and scan rate values (one of the fundamental laser work parameters). This research did not confirm this supposition for the Ti alloy. The highest density 98.24% was found at the energy density 2 J/mm$^2$ while at the highest energy density 3.33 J/mm$^2$ the density was relatively low – 96.92%.

Presumably, at higher energy density the smaller powder grains (in a fraction comprising of grains of different size) may be evaporated in a shorter time resulting in a little smaller density of the melted layer. The better surface quality of the printed out specimens was also observed at lower energy density values. The best surface quality was achieved with laser treatment at laser power 80 W, scan time 100 μs and hatch distance 40 μm, with high dimensional quality and straightness of melting. The highest density and the best dimensional accordance of CAD and melted specimens (an accuracy between the target pattern and printed sample) can be obtained at laser power 100 W, scan time 100 μs and hatch distance 50 μm.

The laser energy during the SLM process is used to heat the powder above melting temperature. Between laser energy application associated with heating and laser beam
Fig. 1. Differences between the CAD model (dark yellow bar in the middle part of the sample) and printed out specimen No. 3 Top view.

Fig. 1. Comparaison entre le modèle CAO (barre jaune dans la partie centrale de l’échantillon) et le modèle produit n° 3. Vue de dessus.

Fig. 2. Differences between the CAD model (navy blue bar in the middle part of the sample) and printed out specimen No. 21. Top view.

Fig. 2. Comparaison entre le modèle CAO (barre bleue dans la partie centrale de l’échantillon) et le spécimen imprimé n° 21. Vue de dessus.
removal followed by cooling, different phenomena occur such as the transformation of solid particles of an alloy into liquid droplets, diffusion of the element in the formed liquid and crystallization of all elements. The power density (laser power on a unit area) and the exposure time in a single area (time means in which the scanning system focuses the laser beam at a single point) within the build layer (area) must be appropriately adjusted to melt the alloy; if one of these process parameters is too low, the melting cannot likely be completed, and the alloy structure after SLM would be inhomogeneous. The high temperature of the liquid causes a significant mixing of elements in a liquid state; the higher energy and exposure time, the better mixing. However, too high energy density and exposure time may result in excessive evaporation of lighter elements, the turbulent behavior of the liquid and an accumulation of thermal energy. During cooling period, the classical crystallization process occurs via formation of embryos and nuclei and growth of crystals. If the cooling rate, determined by a

| No. | Laser power [W] | Scan time [μs] | Hatch distance [μm] | Scan rate [m/s] | Width [mm] | Thickness [mm] | Relative density [%] |
|-----|----------------|---------------|--------------------|----------------|------------|----------------|---------------------|
| 18  | 80             | 100           | 40                 | 0.40           | 5.08       | 0.33           | 97.73               |
| 18a | 80             | 100           | 40                 | 0.40           | 5.14       | 1.11           | 98.86               |
| 21  | 100            | 100           | 50                 | 0.50           | 5.15       | 0.32           | 98.42               |
| 21a | 100            | 100           | 50                 | 0.50           | 5.42       | 1.10           | 98.81               |

Fig. 3. 3D image of the intrinsic structure of specimen No. 21a, obtained by µCT.

Fig. 3. *Image 3D obtenue par µCT de la structure intrinsèque de l’échantillon n° 21a.*
difference between melted part temperature and titanium table temperature, and an environment thermal conductivity (here argon) is too low, the dendritic structure may be formed; if it is high, the desired amorphous, nano-crystalline and small grain structure appears, but simultaneously the cracks may occur. Different elements have different crystallization temperature, which additionally affects the appearing microstructure.

On the other hand, a perfect mixture of elements in a liquid state and fast cooling "frozen" distribution of atoms not allowing for significant diffusion in solid state and the alloy microstructure is far from equilibrium. In this research, no difference in the chemical composition has been noticed so that the diffusion in solid may be assumed as negligible. However, at such fast cooling, the martensitic transformation may take place. Taking into account all above considerations resulting from well-known models of laser melting [1, 28-29], the proper adjustment of these parameters may be said to be extremely important as it affects the homogeneity and surface quality, and the combination of the process parameters depends on the chemical and phase composition. Such an optimal combination was achieved in the present research. These differences are essential for the design of titanium parts and originate from the complex effects of energy density on melting rate and an approached temperature, and the difference between highest obtained temperature and that of the base plate influence the incubation and crystallization rates of the alloy components during the cooling period.

The importance of energy flux was observed in the last tests. Pre-heating the base plate to the proper temperature resulted in better heat dissipation. Energy density and energy flux may have a different influence on the quality of melted parts. When these values are too low, some powder grains may remain unmelted. When they are too high, high thermal stresses may be observed in printed-out objects, leading to greater dimension differences. Such a possibility must be taken into account at the design stage.

5 Conclusions

The energy density, scan rate, and energy flux are essential laser treatment parameters for sintering Ti-13Zr-13Nb alloy by the SLM/DMLS method. The best surface quality is achieved at a lower energy density, and the best density and dimensional accordance at a higher energy density.

The determination of process parameters for selective laser treatment of ternary Ti-13Zr-13Nb alloy is difficult because of the very different melting temperature of all alloying elements, low heat dissipation, and martensitic transformation $\beta \rightarrow \alpha'$ during cooling. The different properties of alloying elements and their different contents cause that the optimal SLM parameters are to be determined individually for each Ti alloy.

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