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Surface Finishes for Ti-6Al-4V Alloy Produced by Direct Metal Laser Sintering

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The implant's surface is responsible for direct interaction with the human body. For cases where osseointegration must be favored and the risk of bacteria proliferation is lower, rough surfaces are more suitable, while for implants where the risks are higher, a reduced surface roughness is required. This study aimed to analyze and produce different surface finishes on samples of Ti-6Al-4V alloy produced by additive manufacturing technique of Direct Metal Laser Sintering (DMLS). Surfaces of the samples were analyzed in the as-built condition, after blasting, after chemical etching, after electropolishing and two different combinations of these methods. The surfaces were studied using the technique of scanning electron microscopy, and surface roughness and mass measurements. The lower roughness value was obtained after a combination of blasting and chemical etching. Blasting results in a surface with uniform roughness while chemical etching cleans the surface and reduces its roughness.

Keywords: *surface finishes, Ti-6Al-4V alloy, additive manufacturing, direct metal laser sintering (DMLS)*

1. Introduction

The increase of life expectancy in recent decades has increased the population and, consequently, increased the surgeries number of partial or total replacement of some part of the human body[1,2]. Currently, additive manufacturing allows production of custom prosthetic implants, fitting them directly to patient needs. It can be used in many medical specialties including neurosurgery, maxillofacial surgery, craniofacial and plastic surgery, oncology, dentistry and orthopedics[3-8].

The main metallic materials used in orthopedic implants are stainless steel alloys, cobalt-chromium alloys and titanium alloys[1,9,10]. The Ti-6Al-4V alloy is considered biocompatible to the human body. Biocompatibility is the ability of a material to fulfill its function in a patient for a specific application[9], and biomaterials are natural or synthetic substances that are tolerated transiently or permanently in the human body[11]. One of the main desirable properties of biomaterials for orthopedic implants is the low modulus of elasticity due to bone reabsorption, besides biocompatibility, corrosion resistance (degradation), mechanical strength, fatigue resistance and wear resistance, which is especially the case of metallic materials.

The surface of an implant has a direct influence on its anchorage in bone[9,12-14]. It is responsible for direct contact with patient tissues and is a key factor for osseointegration, protein adsorption, interaction with cells, the creation of the interface between the implant and the body and as well as for the development of tissues. Topography, chemical characteristics, charge and wettability are the most important properties relating the surface with osseointegration[13,15]. Osseointegration is the anchoring of an implant to the living bone, obtained through contact between them, where bone cells migrates to the implant surface, reaching stability and a durable anchoring, allowing the transmission and distribution of loads to surrounding tissues[12,13,16]. Osseointegration helps in the healing process and in stability and durability of the implant[13]. Rough surfaces, porous coatings and surfaces with osteoconductivity and osteoinductivity in body fluids are shown to be good surfaces for osseointegration[15].

Titanium rough surfaces are effective to further a mechanical attachment with the tissues, increasing the stress distribution at the interface and causing osseointegration[15]. On the other hand, surfaces with high roughness can enhance the development of bacteria in areas with low blood flow[17]. Furthermore, the increased roughness causes an increase in surface area and, consequently, an increase in the quantity of ions released by the implant surface[18].

This study aimed to analyze the surface finishes of blasting, chemical etching and electropolishing in Ti-6Al-4V alloy produced by direct metal laser sintering (DMLS) process.

2. Experimental Procedures

Cylindrical samples with dimensions of 10 mm x 5 mm (diameter x height) were produced from commercial powder of Ti-6Al-4V (Figure 1), donated by the National Institute of

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Biofabrication (INCT - BIOFABRIS), by direct metal laser sintering (DMLS) process using the additive manufacturing equipment EOSINT M270 from EOS GmbH Electro Optical Systems (Figure 2). DMLS is a layer by layer process which uses a laser beam that is directly exposed to the metal powder, fusing and consolidating thin layers. A DMLS process scheme is shown in Figure 3[19].

Processing parameters are shown on Table 1. The blasting was performed on the upper surface of the sample, using grit with an average particle size of 200 µm. The chemical etching step was carried out using a solution containing 2% of hydrofluoric acid and 20% of nitric acid. The samples were completely immersed in the solution at room temperature for 25 minutes, using a rod for agitation. The electrochemical polishing on the samples was performed using the Struers LectroPol-5 equipment. A solution containing 5% of perchloric acid (60%) in acetic acid was used as electrode, and a 0.5 cm^2 mask for polishing of the top surface of the sample was selected. The samples were then polished at room temperature of 25 °C for 5 minutes using a voltage of 55 V under a current of 0.3 A.

Table 1. Processing parameters used for building the Ti-6Al-4V alloy samples.

Processing parameters	
Power (W)	**170**
Scanning speed (mm/s)	1250
Hatch spacing (mm)	0.1
Layer thickness (mm)	0.03
Strategy	Zigzag - 45º between layers

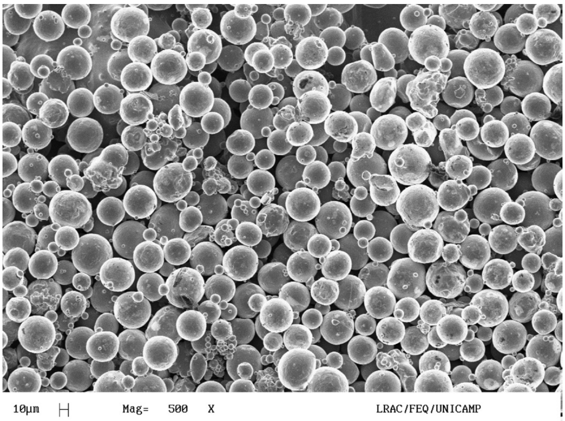


Figure 1. Ti-6Al-4V alloy commercial powder used for DMLS.

Figure 2. Additive manufacturing equipment EOSINT M270.

The surfaces of the samples were analyzed in Zeiss – EVOMA15 scanning electron microscope with Smart SEM software using the secondary electron emission. Mass measurements were performed using a Shimadzu – AX200 scale at each step of surface finishing. Also, roughness measurements for each step of surface finishing of the samples were made using a Mitutoyo SJ-201 rugosimeter. Five measurements were made for each sample, turning them 30º at each measurement. The values shown are the mean values and their standard deviations.

3. Results and Discussion

Figures 4a and 4b show the mean values for surface roughness average (Ra) and mean roughness depth (Rz) of samples in conditions as-built (AB), after blasting (B), after chemical etching (CHE), after electropolishing (EP) and after different combinations of methods. The B – CHE combination gave the lowest surface roughness value. While blasting is responsible for reducing surface roughness and making it more uniform, chemical etching reduces even more the roughness. The electropolishing finish presented high values of roughness, even when made after B - CHE treatment, showing concordance with the work of Pyka et al.[20].

Table 2 shows the results for mass analysis, where the reduction is the percentage mass reduction relative to the as-built condition, and the relative reduction is the percentage mass reduction relative to the previous step. The mass measurements are plotted in Figure 4c. The CHE treatment showed the highest mass reduction. This happened due to two factors: the CHE process works by removal of material by surface oxidation, causing ionization of atoms that come off the matrix; the sample was completely immersed in the reagent, causing all the surfaces of the sample to be attacked. For EP and B the mass reduction occurs on a smaller scale. However, it is noteworthy that only the top

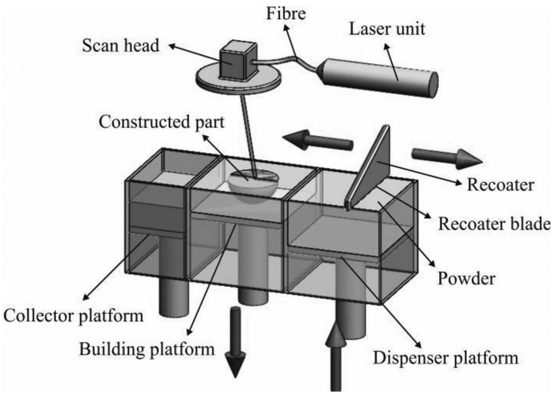


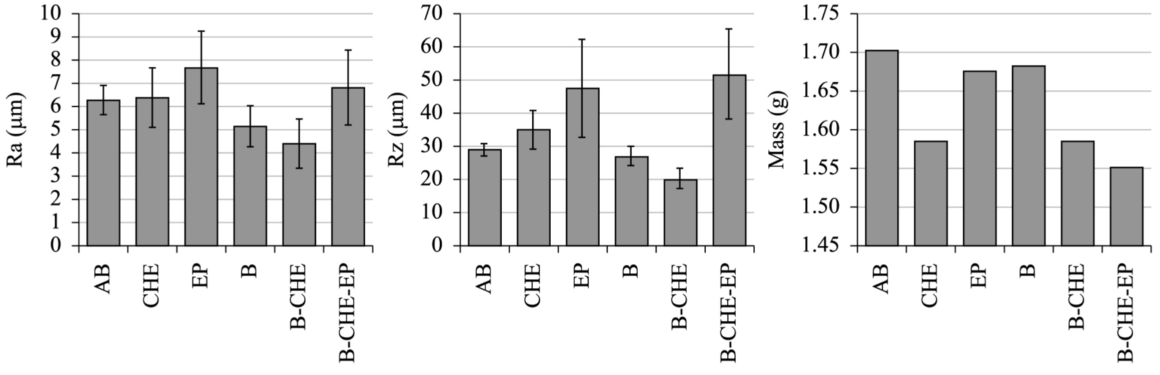
Figure 3. Schematic diagram of the DMLS system.

surface of the sample (25% in area of total surfaces) was treated by reducing the effective area of material removal. The blasting process removes material due to the shape of the grit, which has cutting edges that remove material on colliding with the surface. Electrochemical polishing also removes materials by ionizing surface atoms.

Table 2. Values for mass measurements.

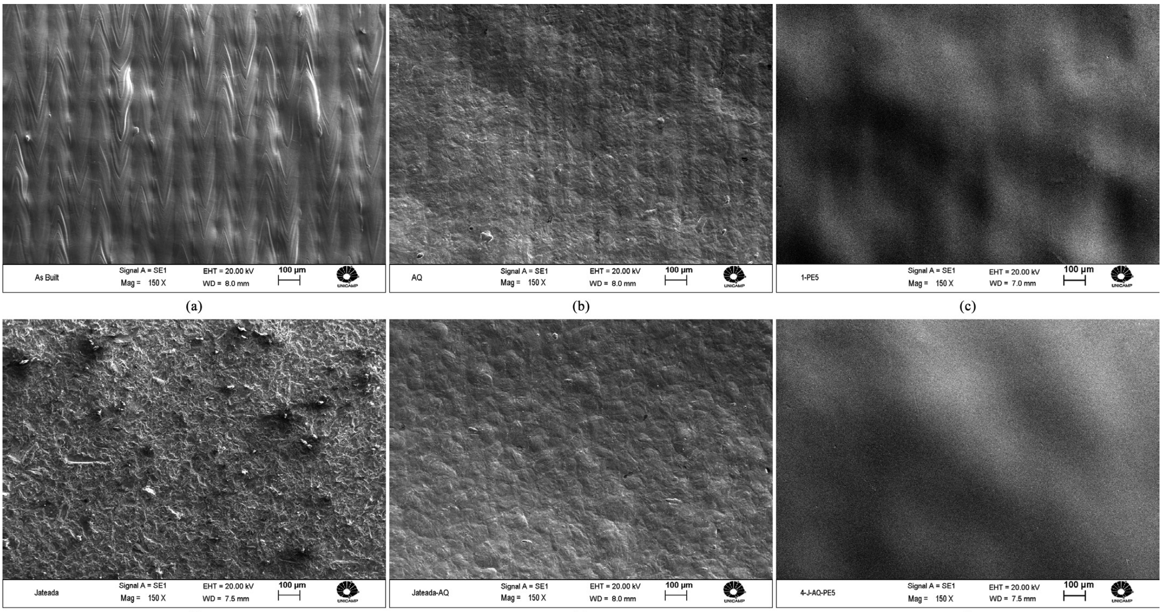
Condition	Mass (g)	Reduction (%)	Relative Reduction(%)
AB	1.708	-	-
EP	1.588	7.03	-
CHE	1.681	1.58	-
B	1.688	1.17	-
B – CHE	1.589	6.97	5.86
B – CHE – EP	1.555	8.96	2.14

In Figure 5, it is possible to observe the surfaces of the samples in the AB condition (a), after CHE (b), after EP (c), after B (d), after B and CHE (e) and after combining the three treatments (f). In the AB condition the top surface lines that characterize the laser building strategy of zigzag and the hatch spacing of 100 µm can be seen. In the CHE condition, it can be seen that material has been removed and the grains of the microstructure were lightly revealed, due to the reaction of the reagent with the alloy material. However, the traces of the scanning lines of the laser are still visible. Moreover, after EP, the laser scanning lines are no longer visible. Furthermore, it is possible to see that, although the surface looks essentially flawless, there is an undulation on the surface, as shown by shading in some regions. After B finishing the laser scanning lines also disappear completely. The blasting process works by deforming and removing material from the surface, obtaining as a result a very rugged surface. However, some grit grains attached on the surface of



(a) (b) (c)

Figure 4. Surface roughness average (a), mean roughness depth (b) and mass analysis (c) graphs.



(d) (e) (f)

Figure 5. Micrographs obtained by SEM for each condition. As-built (a), after chemical etching (b), after electropolishing (c), after blasting (d), after blasting and chemical etching (e) and after combining the three treatments (f).

the sample. The B – CHE combination showed the smallest surface roughness value. The CHE finishing cleans the previous step attached grits on the sample surface. Also, there is no trace of scanning lines, and the microstructure grains were revealed. Finally, the B – CHE – EP finishing showed the same appearance as for the EP treatment, indicating that the process is independent of the two preceding treatments.

4. Conclusions

The lower surface roughness value was obtained after combining blasting and chemical etching. Blasting is responsible for leaving a surface with uniform roughness, while the chemical etching is responsible for cleaning the surface and reduce its roughness. Electropolishing showed a mirrored surface finish, but with a high roughness value, showing ineffectiveness in lowering the surface roughness of the material. The mass analysis showed a reduction in weight of the samples after all the treatments. This occurs because all treatments have as principle material removal.

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References

1. Jardini AL, Larosa MA, Bernardes LF, Zavaglia CAC and Maciel R Fo. Application of direct metal laser sintering in titanium alloy for cranioplasty. In: *Proceedings of the 6th Brazilian Conference on Manufacturing Engineering*; 2011; Caxias do Sul. Caxias do Sul: ABCM; 2011. p. 1-7.
2. Cardoso FF. *Análise de parâmetros de influência na microestrutura e propriedades de ligas Ti-Mo-Zr aplicadas em próteses ortopédicas*. [Dissertation]. Campinas: Universidade Estadual de Campinas; 2008.
3. Ciocca L, Fantini M, De Crescenzio F, Corinaldesi G and Scotti R. Direct metal laser sintering (DMLS) of a customized titanium mesh for prosthetically guided bone regeneration of atrophic maxillary arches. *Medical & Biological Engineering & Computing*. 2011; 49(11):1347-1352. http://dx.doi.org/10.1007/s11517-011-0813-4. PMid:21779902.
4. Salmi M, Tuomi J, Paloheimo KS, Björkstrand R, Paloheimo M, Salo F, et al. Patient specific reconstruction with 3D modeling and DMLS additive manufacturing. *Rapid Prototyping Journal*. 2012; 18(3):209-214. http://dx.doi.org/10.1108/13552541211218126.
5. Huotilainen E, Paloheimo M, Salmi M, Paloheimo KS, Björkstrand R, Tuomi J, et al. Imaging requirements for medical applications of additive manufacturing. *Acta Radiologica*. 2014; 55(1):78-85. http://dx.doi.org/10.1177/0284185113494198. PMid:23901144.
6. Philippe B. Custom-made prefabricated titanium miniplates in Le Fort I osteotomies: principles, procedure and clinical insights. *International Journal of Oral and Maxillofacial Surgery*. 2013; 42(8):1001-1006. http://dx.doi.org/10.1016/j.ijom.2012.12.013. PMid:23602483.
7. Stübinger S, Mosch I, Robotti P, Sidler M, Klein K, Ferguson SJ, et al. Histological and biomechanical analysis of porous additive manufactured implants made by direct metal laser sintering: a pilot study in sheep. *Journal of Biomedical Materials Research. Part B Applied Biomaterials*. 2013; 101(7):1154-1163. http://dx.doi.org/10.1002/jbm.b.32925. PMid:23564723.
8. Jardini AL, Larosa MA, Maciel R Fo, Zavaglia CAC, Bernardes LF, Lambert CS, et al. Cranial reconstruction: 3D biomodel and custom-built implant created using additive manufacturing. *Journal of Cranio-Maxillo-Facial Surgery*. 2014; 42(8):1877-1884. http://dx.doi.org/10.1016/j.jcms.2014.07.006. PMid:25175080.
9. Liu X, Chu PK and Ding C. Surface modification of titanium, titanium alloys, and related materials for biomedical applications. *Materials Science and Engineering*. 2004; 47(3-4):49-121. http://dx.doi.org/10.1016/j.mser.2004.11.001.
10. Bertol LS, Kindlein W Jr, Silva FP and Aumund-Kopp C. Medical design: Direct metal laser sintering of Ti–6Al–4V. *Materials & Design*. 2010; 31(8):3982-3988. http://dx.doi.org/10.1016/j.matdes.2010.02.050.
11. Maia SS. *Avaliação mecânica de parafusos de titânio e de Ti-6Al-4V para fixação interna rígida*. [Dissertation]. Campinas: Universidade Estadual de Campinas; 2003.
12. Buser D, Nydegger T, Oxland T, Cochran DL, Schenk RK, Hirt HP, et al. Interface shear strength of titanium implants with a sandblasted and acid-etched surface: a biomechanical study in the maxilla of miniature pigs. *Journal of Biomedical Materials Research*. 1999; 45(2):75-83. http://dx.doi.org/10.1002/(SICI)1097-4636(199905)45:2<75::AID-JBM1>3.0.CO;2-P. PMid:10397960.
13. Guo CY, Matinlinna JP, Tang ATH. Effects of surface charges on dental implants: past, present, and future. *International Journal of Biomaterials*. 2012; 2012:1-5. http://dx.doi.org/10.1155/2012/381535.
14. Lausmaa J. Mechanical, thermal, chemical and electrochemical surface treatment of titanium. In: Brunette DM, Tengvall P, Textor M and Thomsen P, editors. *Titanium in medicine: material science, surface science, engineering, biological responses and medical applications*. Berling: Springer-Verlag; 2001 p. 231-266.
15. Ferguson SJ, Broggini N, Wieland M, de Wild M, Rupp F, Geis-Gerstorfer J, et al. Biomechanical evaluation of the interfacial strength of a chemically modified sandblasted and acid-etched titanium surface. *Journal of Biomedical Materials Research. Part A*. 2006; 78(2):291-297. http://dx.doi.org/10.1002/jbm.a.30678. PMid:16637025.
16. Lima PM. *Caracterização de revestimentos de hidroxiapatita depositados por aspersão térmica a plasma sobre a liga Ti-13Nb-13Zr para aplicação em implantes dentários*. [Dissertation]. Campinas: Universidade Estadual de Campinas; 2004.
17. Drstvensek I, Hren NI, Strojnik T, Brajlih T, Valentan B, Pogacar V, et al. Applications of Rapid Prototyping in Cranio-Maxilofacial Surgery Procedures. *International Journal of Biology Biomedical Engineering*. 2008; 1(2):29-38.
18. Wennerberg A, Ektessabi A, Albrektsson T, Johansson C and Andersson B. A 1-year follow-up of implants of differing surface roughness placed in rabbit bone. *The International Journal of Oral & Maxillofacial Implants*. 1997; 12(4):486-494. PMid:9274077.
19. Bineli ARR, Jardini AL, Peres APG, Bernardes LF and Maciel R Fo. Microchannels fabrication in Direct Metal Laser Sintering

(DMLS). In: Bártolo PJ, Lemos ACS, Tojeira APO, Pereira AMH, Mateus AJ, Mendes ALA et al., editors. *Innovative developments in virtual and physical prototyping*. New York: CRC Press; 2011. p. 613-617. http://dx.doi.org/10.1201/b11341-98.

20. Pyka G, Burakowski A, Kerckhofs G, Moesen M, Van Bael S, Schrooten J, et al. Surface modification of Ti6Al4V open porous structures produced by additive manufacturing. *Advanced Engineering Materials*. 2012; 14(6):363-370. http://dx.doi.org/10.1002/adem.201100344.