



SURFACE MODIFICATION OF Ti6Al4V ALLOY BY PULSED LASER: INFLUENCE OF PARAMETERS VARIATION

Rogério Góes dos Santos

Polytechnic School of Mechanical Engineering – University of São Paulo, São Paulo – Brazil.

goesrogerio@usp.br

Gleicy de Lima Xavier Ribeiro

Advanced Measurement Laboratory, SENAI Institute for Innovation in Advanced Manufacturing and Microfabrication, 379 Bento Branco de Andrade Filho St., Santo Amaro, SP 04757-000, Brazil.

gleicy.limaxavier@gmail.com

Gilmar Ferreira Batalha

Dept. of Mechatronics and Mechanical System Engineering, Polytechnic School of Engineering – University of São Paulo, São Paulo – Brazil.

gfbatalh@usp.br

Abstract: An ytterbium pulsed laser was used to modify Ti-6Al-4V surfaces. With pulse duration of 150 ns and wavelength of 1,064 nm, the laser variation was based on three parameters: speed, power and number of passes. Was observed the influence of this laser parameters variation on surfaces characteristics. In this sense, this work sought to identify correlations between pulsed laser parameters (nanosecond temporal width) and effects generated in grade 5 titanium (Ti-6Al-4V alloy) in terms of roughness and topography, effects directly associated with osseointegration of prostheses and dental implants. The methodology of planning the experiment and characterizing the surfaces are described, as well as the methods for extracting effects are detailed. It was found that the three parameters used for laser variation (speed, power and number of passes) influenced the roughness values and topography pattern. The increase in speed and the number of steps contributes to the increase in roughness, while the increase in power caused a reduction.

Keywords: pulsed laser parameters, surface modification, surface characterization, surface integrity, dental implants.

1. INTRODUCTION

Laser texturing is a manufacturing process used to modify material surfaces through the incidence of pulsed laser beams. It is a surface structuring process that can be completely digitized, capable of generating textures and engravings (2D/3D) manufactured in a precise and direct manner, aiming to improve and/or change the aesthetic appearance, functionality and performance of product surfaces and components. Thus, among the techniques available for surface modification, laser texturing stands out for its versatility, precision and repeatability, especially in medical, dental, industrial and aerospace applications (GF MACHINING SOLUTIONS, 2017).

Considering the range of materials associated with these different applications and segments, grade 5 titanium (alloy Ti-6Al-4V) is one of those that stands out, both in the interaction with pulsed laser (allowing for sharp, clean and permanent textures), as well as for its high mechanical strength, low density, excellent corrosion resistance and high melting point, properties that, as a biomaterial, favor its use in medical prostheses, dental and orthopedic implants (CAMPBELL, 2006; HUTCHING and SHIPWAY, 2017; SCHAEFFER, 2012).

Using laser texturing for surface modification of Ti-6Al-4V alloy, Melanie *et al.* (2016) investigated the results of implant osseointegration, comparing it with other types of processes and different surface conditions of the same material, citing that laser texturing has been a successful alternative to increase implant fixation, given its ability repeatability of patterns to create pores in the material, generating rough surfaces.

Other studies cite roughness as a relevant effect associated with osseointegration of dental prostheses and implants, such as Grizon *et al.* (2002) who, in their studies to improve the osseointegration of titanium surfaces of dental implants, observed that the increase in roughness values generated more robust bone responses. And Christoph *et al.* (2019) who used pulsed laser, varying parameters, to create titanium surfaces, with increased cell adhesion, highlighting the potential of this process in improving the osseointegration of dental implants, demonstrating that this process presented roughness values lower than the reference value used by the authors.

Uhlmann *et al.* (2018), also consider the effects of roughness in their studies on the effects of laser texturing on Ti-6Al-4V surfaces for dental implants. As well as Á Joób-Fancsaly *et al.* (2002), who in their studies on the structuring of titanium surfaces (for dental implants) using pulsed laser, investigated the influence of the variation of the geometries created on the surfaces of the material, which is associated with roughness; the authors mention that when it comes to osseointegration, smoother surfaces are less favorable than rough surfaces.

Roughness can be observed as the surface irregularities inherent in the interaction between the manufacturing process and material and, in addition to being associated with the topography and integrity of modified surfaces, it is directly influenced by the parameters of the aforementioned manufacturing processes. Thus, the correlation between roughness and process parameters can contribute to manufacturing strategies and optimizations, as well as to the observation of aspects associated with performance and safety in functional applications of products and components (SANTOS and SALES, 2007; JESUS, 2013; MIRHOSSEINI *et al.*, 2007; DENKENA *et al.*, 2018; TAYLOR ROBSON, 2003).

The present study sought to verify the influence that the variation of parameters of the pulsed ytterbium laser can generate on the modification of Ti-6Al-4V alloy surfaces, considering the dental implants context.

2. METODOLOGY

A factorial experiment with two levels and three factors was structured. Each one of the sixteen tests conducted was on sixteen individual samples. There are two surfaces samples for each one of the eight parameters combinations (A, B and C): V35P50N1; V35P50N4; V35P100N1; V35P100N4; V140P50N1; V140P50N4; V140P100N1 and V140P100N4. The tests were replicated in order to verify the process repeatability and variations in the mean roughness values Sa, Sz, Ra and Rz; where Sa and Sz represent area roughness (3D) and Ra and Rz represent profile roughness (2D); with emphasis on Sa and Ra that, according to Manera *et al.* (2017), are roughness parameters recommended in the area of dental implants.

For surface texturing, a GF P1000U laser texturing machine was used, with: a Ytterbium fiber laser source, wavelength of 1,064 nm, máx. power between 45 and 50 Watts, pulse duration of 150 ns and frequency of 2 kHz. The characterization of surface roughness and topography, both before and after laser texturing, was performed using the interferometry technique (optical profiling), by Taylor Robson interferometer; a ZEISS Axio CSM 700 confocal microscopy; and, a Hitachi TM3000 SEM (Scanning Electron Microscope).

3. RESULTS

Topographies for each textured sample are showed in Figure 1, organized in such a way that it is possible to view the two samples, side by side, for each laser parameter combination. There is a similarity in the topographies of the textured surfaces pairs, demonstrating the repeatability of the process, as stated by GF Machining Solutions (2017).

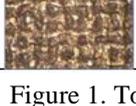
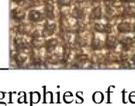
Textured surfaces			
	Surfaces topographies		Pulsed laser parameters
1		10 	Samples: 1 and 10. Parameters: V=35 mm/s / P=50% / N=1
4		11 	Samples: 4 and 11. Parameters: V=35 mm/s / P=50% / N=4
8		12 	Samples: 8 and 12. Parameters: V=35 mm/s / P=100% / N=1
7		15 	Samples: 7 and 15. Parameters: V=35 mm/s / P=100% / N=4
13		16 	Samples: 13 and 16. Parameters: V=140 mm/s / P=50% / N=1
5		14 	Samples: 5 and 14. Parameters: V=140 mm/s / P=50% / N=4
2		3 	Samples: 2 and 3. Parameters: V=140 mm/s / P=100% / N=1
6		9 	Samples: 6 and 9. Parameters: V=140 mm/s / P=100% / N=4

Figure 1. Topographies of textured surfaces.

It is verified that, for all sixteen samples, there was an increase in the values of the four roughness parameters (Sa, Sz, Ra and Rz), the results obtained represent the difference between the roughness values after and before the laser. Therefore, untreated surfaces had the lowest roughness values, a condition also found and described by Mirhosseini *et al.* (2007). The results of the variations in roughness values are shown in Table 1 and Figure 2, considering (Δ) the percentage between the roughness of the samples after and before laser texturing.

Table 1. Correlation between laser parameters and roughness increase (in percentagem, %).

Pulsed laser parameters	Samples	Roughness variation (Δ) percentage (%)			
		(Δ +) Sa	(Δ +) Sz	(Δ +) Ra	(Δ +) Rz
V35P50N1	1 and 10	1.135% (11,35 times)	1.143% (11,43 times)	1.160% (11,6 times)	977% (9,77 times)
V35P50N4	4 and 11	1.035% (10,35 times)	545% (5,45 times)	869% (8,69 times)	624% (6,24 times)
V35P100N1	8 and 12	1.597% (15,97 times)	962% (9,62 times)	1.530% (15,3 times)	1.066% (10,66 times)
V35P100N4	7 and 15	2.312% (23,12 times)	1.226% (12,26 times)	2.581% (25,81 times)	1.797% (17,97 times)
V140P50N1	13 and 16	2.595% (25,95 times)	2.478% (24,78 times)	2.515% (25,15 times)	3.314% (33,14 times)
V140P50N4	5 and 14	3.605% (36,05 times)	3.595% (35,95 times)	3.855% (38,55 times)	3.779% (37,79 times)
V140P100N1	2 and 3	1.720% (17,2 times)	1.258% (12,58 times)	1.591% (15,91 times)	1.055% (10,55 times)
V140P100N4	6 and 9	2.620% (26,2 times)	2.167% (21,67 times)	2.947% (29,47 times)	2.740% (27,40 times)

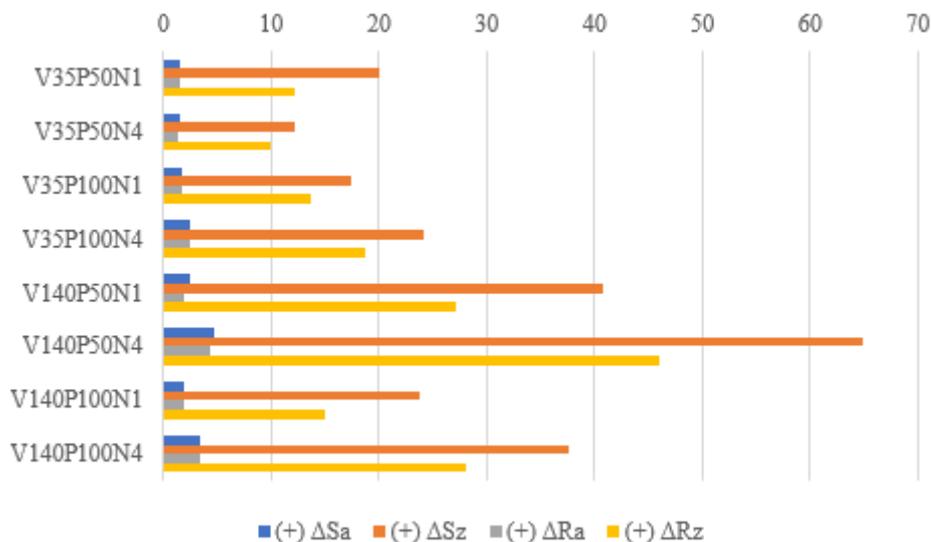


Figure 2. Graphic correlating laser parameters and roughness.

All processed samples showed an increase in their roughness values, for which the speed of 35 mm/s contributed to the lowest roughness values, compared to the speed of 140 mm/s. With a speed of 35 mm/s and a nominal power of 50% (~ 25 W) the variation in the number of passes (1 or 4) resulted in similar roughness values. With a speed of 140 mm/s, for either 1 or 4 passes, the nominal power of 100% (~ 50 W) contributed to an increase in roughness, in relation to the nominal power of 50%. The highest roughness values were observed with a speed of 140 mm/s, a nominal power of 50% (~ 25 W) and 4 passes; by reducing the number of passes to 1, a reduction in roughness values was observed, a reduction that was accentuated when the power was increased to 100% (~ 50 W).

Based on the results described in Table 1, Table 2 was drawn up, in which the largest and smallest variations in the mean roughness values of the sixteen samples are highlighted.

Table 2. Average roughness values variation (Δ).

Pulsed laser parameters	Δ = Average variation between the roughness of the two pairs of samples produced with the same combinations of laser parameters			
	ΔSa (μm)	ΔSz (μm)	ΔRa (μm)	ΔRz (μm)
V35P50N4	$1.521 \pm 0,017$	$12.165 \pm 0,355$	$1.409 \pm 0,053$	$9.840 \pm 0,700$
V35P50N1	1.605	19.945	1.43605	12.17
V35P100N1	1.7565	17.365	1.63	13.585
V140P100N1	1.9385	23.705	1.9025	14.955
V35P100N4	2.4725	24.13	2.386	18.666
V140P100N4	3.318	37.64	3.3065	28.0135
140P50N1	2.4925	40.865	1.883	27.1545
V140P50N4	$4.742 \pm 0,567$	$64.845 \pm 8,865$	$4.350 \pm 0,776$	$45.965 \pm 9,315$

It is noted that, for both the smallest and the highest average roughness values, the results were obtained from pairs of samples produced with the same sets of parameters, samples 4 and 11 (V35P50N4), and 5 and 14 (V140P50V4). The variation of the speed values impact on the roughness, since the samples from tests 4 and 11 were textured with a speed of 35 mm/s, while the samples from tests 5 and 14 with a speed of 140 mm/s, in both cases were kept fixed for the other parameters (power and number of passes). Therefore, the increase in speed contributed to the increase in roughness values. Roughness profiles (2D) are showed in Figures 3, 4, 5 e 6.

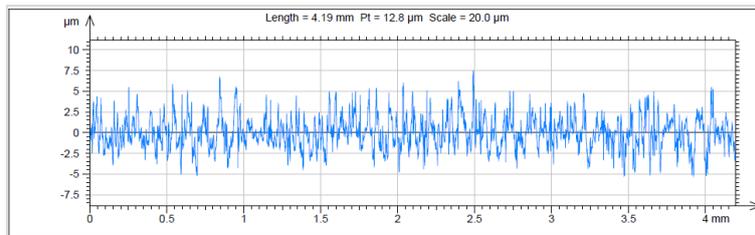


Figure 3. Sample 4 (2D) roughness graphic. Profile at 50%.

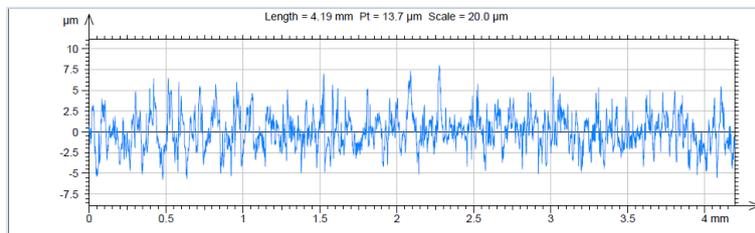


Figure 4. Sample 11 (2D) roughness graphic. Profile at 50%.

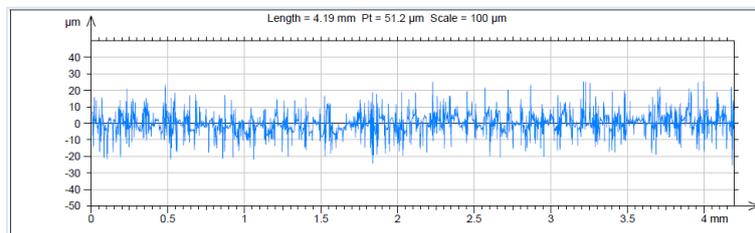


Figure 5. Sample 5 (2D) roughness graphic. Profile at 50%.

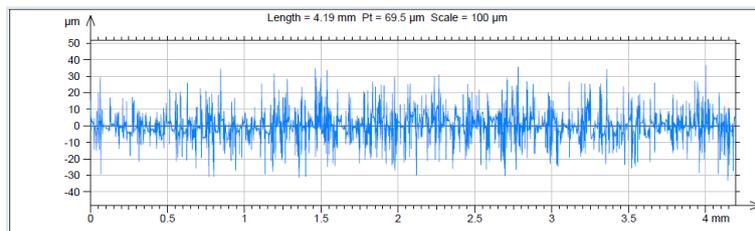


Figure 6. Sample 14 (2D) roughness graphic. Profile at 50%.

Roughness graphics (3D) and SEM images are showed in Figures 7, 8, 9 and 10. Differently from 2D roughness profile (information basically from one line in the middle of the surface), 3D graphics allow the whole surface area observation, consequently, increasing the information quantity and quality, about the surface condition. SEM images contribute showing the surface morphology, as surface's resolidification behaviour and craquelure formation (or not) on oxide surface.

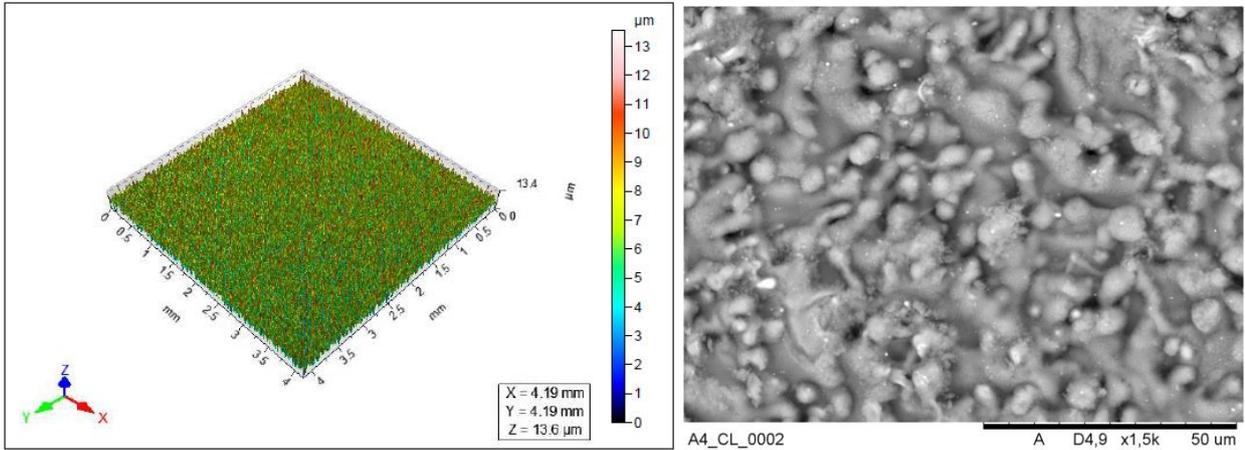


Figure 7. Sample 4: 3D graphics and SEM images, made by:V35P50N4.

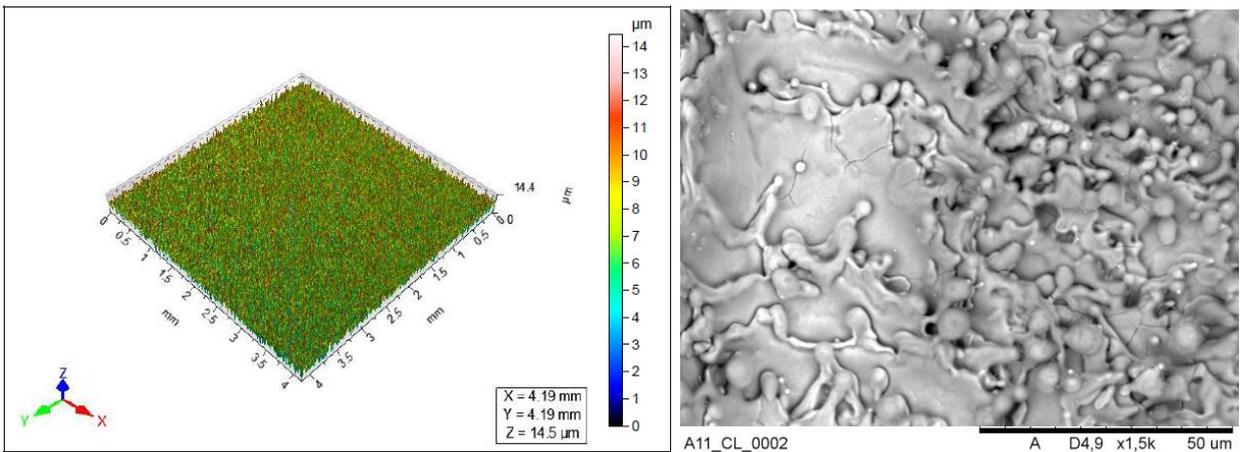


Figure 8. Sample 11: 3D graphics and SEM images, made by:V35P50N4.

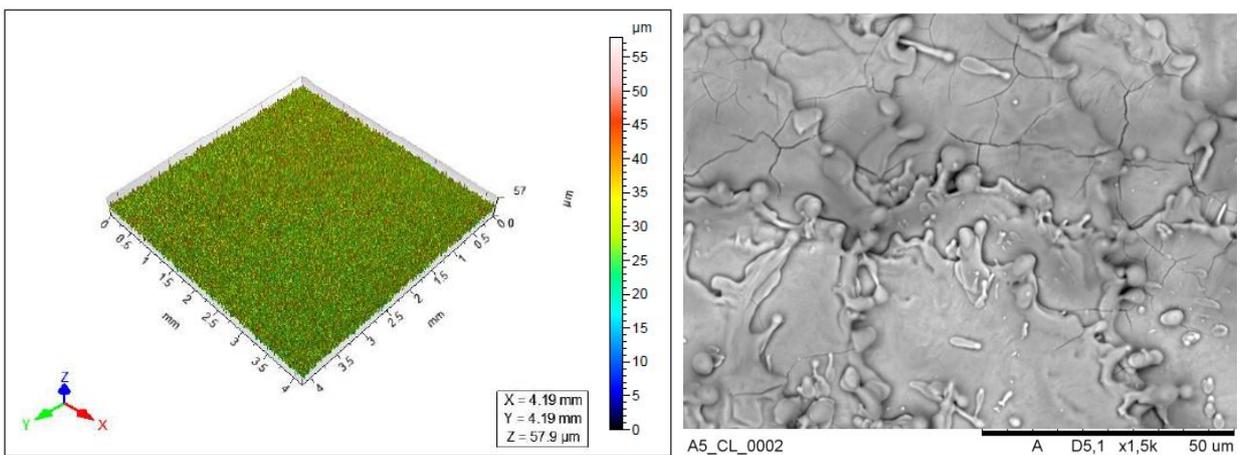


Figure 9. Sample 5: 3D graphics and SEM images, made by:V140P50N4.

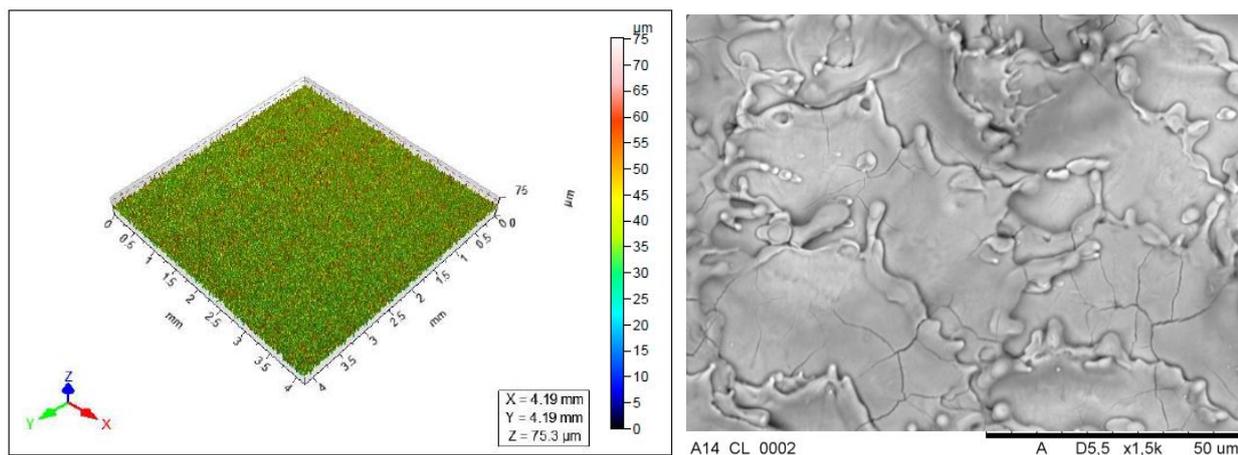


Figure 10. Sample 14: 3D graphics and SEM images, made by:V140P50N4.

In general terms, such behavior is in line with the trends suggested by GF Machining Solutions (2017) which, using laser with the same characteristics as the pulsed ytterbium laser used for the tests in this work, states that the increase in speed results in an increase in roughness. Where, in addition to speed, other parameters are associated (power and number of passes), indicating that the roughness increases as the power values decrease and the number of passes increases, as can be seen in Table 2 and Figure 2.

Roughness increases and oxide craquelures were also observed by Lima e Almeida (2017), but this authors had observed, roughness decrease too. It may be associated with some pulsed laser parameters differences and specificities.

4. CONCLUSION

The present work explored the potential of surface modification through pulsed laser technology, verifying that this process is capable to change (increase) roughness values. It was found that the three parameters (speed, power and number of passes) of the pulsed laser, influenced the roughness parameters (S_a , S_z , R_a and R_z) observed, generating an increase in the roughness values of all tested samples.

Considering (as mentioned by several authors) roughness as one of the relevant factors for cell growth/adhesion in orthopedic and dental implants, it was found that the parameterization and repeatability of laser texturing, through the variation of its parameters, can contribute on the modification of surfaces associated with osseointegration.

5. REFERENCES

- Joób-fancsaly, Á., Divinyi, T., Fazekas, Á., Daroczi, Cs., Karacs, A., and Pető, G., 2002. Smart Mater. Struct. 11819. Disponível em: <<https://iopscience.iop.org/article/10.1088/0964-1726/11/5/330>>. Acesso em: 15 de abr. de 2021.
- Campbell, F. C., 2006. Manufacturing Technology for Aerospace Structural Materials. Great Britain: Elsevier, 2006.
- Coathup, M.J. Blunn, G.W., Mirhosseini, N., Erskine, K., Liu, Z., Garrod, D.R., Li, L., 2016. Controlled laser texturing of titanium results in reliable osteointegration. Journal of Orthopaedic Research. Volume 35. Issue 4, 2016, p. 820-828. Disponível em: < <https://onlinelibrary-wiley.ez67.periodicos.capes.gov.br/doi/full/10.1002/jor.23340>>. Acesso em: 15 de abr. de 2021.
- Denkena, B., Krödel, A., Grove, T., 2019. Influence of pulsed laser ablation on the surface integrity of PCBN cutting tool materials. Int J Adv Manuf Technol 101, 1687–1698. Disponível em: <<https://link.springer.com/article/10.1007/s00170-018-3032-4#article-info>>. Acesso em: 04 de jan. de 2021. DOI: <https://doi.org/10.1007/s00170-018-3032-4>.
- Grizon, F., Aguado, E., Huré, G., Baslé, M.F., Chappard, D., 2002. Enhanced bone integration of implants with increased surface roughness: a long term study in the sheep, Journal of Dentistry, Volume 30, Issues 5–6, 2002, Pages 195-203, ISSN 0300-5712, [https://doi.org/10.1016/S0300-5712\(02\)00018-0](https://doi.org/10.1016/S0300-5712(02)00018-0). (<https://www.sciencedirect.com/science/article/pii/S0300571202000180>).
- Hutchins, I. and Shipway, P., 2017. Friction and Wear of Engineering Materials. Second Edition. United Kingdom: Elsevier, 2017.
- GF Machining Solutions. Laser Process. C7 Tecnologia de funcionamento. Manual do utilizador. 2017.
- Esus, R. E. C. Avaliação dos esforços, temperatura e integridade superficial em torneamento do aço inoxidável SuperDuplex UNS S32760 quando alterado ângulos de posição da ferramenta e os parâmetros de corte. Dissertação (Mestrado) – Universidade Federal de São João Del-Rei. Departamento de Engenharia Mecânica. São João Del Rei, 2013.

- Lima, M.S.F. and ALMEIDA, I., 2017. Surface Modification of Ti6Al4V Alloy by Pulsed Lasers: Microstructure and Hydrophobic Behavior. *Mat. Res., São Carlos*, v. 20, supl. 1, p. 8-14, 2017. Disponível em: <http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-14392017000700008&lng=en&nrm=iso>. Acesso em: 04 Jan. 2021. Epub Sep 21, 2017. <https://doi.org/10.1590/1980-5373-mr-2017-0221>.
- Manera, R.D., Rodrigues, A., Matsumoto, H. and Gallego, J., 2017. Surface quality and specific cutting energy in the machining of dental implants.
- Mirhosseini, N., Crouse, P.L., Schmidh, M.J.J., Li, L., Garrod, D., 2007. Laser surface micro-texturing of Ti-6Al-4V substrates for improved cell integration, *Applied Surface Science*, Volume 253, Issue 19, 2007, Pages 7738-7743, ISSN 0169-4332, <https://doi.org/10.1016/j.apsusc.2007.02.168>. Disponível em: <<http://www.sciencedirect.com/science/article/pii/S0169433207003406>>. Acesso em: 18 de jan. de 2021.
- Pou, P, Riveiro, A., Del Val, J., Comesaña, R., Penide, P., Arias-González, F., Soto, R., Lusquiños, F. and Pou, J., 2019. Laser surface texturing of Titanium for bioengineering applications. *Procedia Manufacturing* 13 (2017) 694–701. Disponível em: <<https://www.sciencedirect.com/science/article/pii/S2351978917307370>>. Acesso em: 10 de fev. de 2019.
- Santos, C. S. and Sales, W. F., 2012. *Aspectos tribológicos da usinagem dos materiais*. São Paulo: Artliber, 2007.
- Schaeffer, R. D. *Fundamentals of laser Micromachining*. New York: CRC Press, 2012.
- Taylor Hobson. *Exploring Surface Texture: A fundamental guide to the measurement of surface finish*. 7th edition. England, 2011.
- Uhlmann, E., Schweitzer, L., Kieburg, H., Spielvogel, A. and Huth-Herms, K., 2018. The Effects of Laser Microtexturing of Biomedical Grade 5 Ti-6Al-4V Dental Implants (Abutment) on Biofilm Formation. *Procedia CIRP*, Volume 68, 2018, Pages 184-189, ISSN 2212-8271. Disponível em: <<http://www.sciencedirect.com/science/article/pii/S221282711730985X>>. Acesso em: 04 de jan. de 2021. <https://doi.org/10.1016/j.procir.2017.12.044>.
- Zwahr, C., Welle, A., Weingärtner, T., Heinemann, C., Kruppke, B., Gulow, N., Holthaus, M.G. and Lasagni, A.F., 2019. Ultrashort Pulsed Laser Surface Patterning of Titanium to Improve Osseointegration of Dental Implants. 2019. Disponível em: <<https://doi-org.ez67.periodicos.capes.gov.br/10.1002/adem.201900639>>. Acesso em: 15 de abr. de 2021.

6. RESPONSIBILITY NOTICE

The three authors are the only responsible for the printed material included in this paper.