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Microstructural Characterization and Analysis of β-Titanium Alloy (Ti-35Nb-7Zr-5Ta) Modified by Laser Beam for use in Implantology

Caracterização Microestrutural Como Análise em Liga β de Titânio (Ti-35Nb-7Zr-5Ta) Modificada por Feixe de Laser para Uso em Implantodontia

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Abstract

The Ti-6Al-4V alloy presents elements harmful to the organism, such as aluminum and vanadium, demonstrating the importance of developing new Ti alloys with the addition of non-toxic elements. The elements were melted and machined for the study; the samples were divided into groups without surface treatment (Group I) and with surface modification by Yb: YVO_4 laser beam irradiation in two conditions: 20Hz (Group II) and 35Hz (Group III). Structural and microstructural characterization was performed by evaluating Young's module, scanning electron microscopy (SEM) with elemental microanalysis by X-ray dispersive energy (EDX), and X-ray diffractometry (XRD). The results report an alloy of predominantly β structure and the formation of a layer of titanium oxides on the surface of the alloy in the three conditions studied, indicating protection of the material from chemical reactions related to its environment exposed to (NaCl 0.9%). The characterizations showed favorable results to laser beam irradiation, noting the formation of TiO_2 compounds that are important and responsible for the attraction of bone tissue cells and bone/implant interaction.

Keywords: Dental Alloys. Titanium. Dental Implants. Surface Modifications.

Resumo

A liga Ti-6Al4V apresenta elementos nocivos ao organismo como alumínio e vanádio, demonstrando a importância do desenvolvimento de novas ligas de Ti, com adição de elementos não tóxicos. Para a realização deste estudo, os elementos foram fundidos a arco voltaico e usinados. As amostras foram divididas em grupos sem tratamento de superfície (Grupo I) e com modificação de superfície por irradiação de feixe de laser Yb:YVO_4 *em duas condições: 20Hz (Grupo II) e 35Hz (Grupo III). A caracterização estrutural e microestrutural foi realizada por avaliação de módulo de Young, microscopia eletrônica de varredura (MEV) com microanálise elementar por energia dispersiva de raios X (EDX) e a Difratometria de raios X (DRX). Os resultados demonstraram uma liga de estrutura predominantemente β e formação de uma camada de óxidos de titânio sobre a superfície da liga nas três condições estudadas, indicando uma proteção do material á reações químicas em relação ao meio ao qual se expõe (NaCl 0,9%). As caracterizações apresentaram resultados favoráveis à irradiação por feixe laser, notando-se a formação de* TiO_2 *que são importantes e responsáveis pela atração de células do tecido ósseo e interação osso/implante.*

Palavras-chave: *Ligas Dentárias. Titânio. Implantes Dentários. Modificações de Superfície.*

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1 Introduction

Pure titanium (Ti-cp) and titanium-based alloys have been considered ideal metallic materials for biomedical applications for some years. The Ti-6Al-4V alloy has for a long time been the primary metal alloy used in biomedical devices due to its excellent mechanical, chemical, and biocompatibility properties. The composition of the alloy offers superior characteristics to Ti-cp, attributing this excellence to the elements of the alloy, which can be α-stabilizers and β-stabilizers (LOPES; DONATO; RAMGI, 2016; NICHOLSON, 2020)there are several problems concerning the corrosion of implants, for example, the high concentration of fluoride ions, which make an acid medium. Considering that titanium has excellent biocompatibility and some resistance to corrosion, one way to enhance this property is alloying Ti with other metals. The most used alloy is Ti-6Al-4V, in spite of its toxicity. Hence, there is a need to make new alloys which are resistant to corrosion and less toxic. One that stands out is Ti-15Mo. The objective of this review is to compare these two alloys in terms of corrosion behaviour and possible treatments to improve their corrosion resistance. RESUMO Atualmente existe uma necessidade crescente de materiais biocompatíveis devido à toxicidade dos metais usados. Nos implantes dentários existem vários problemas relacionados com a corrosão dos implantes, como a elevada concentração de iões fluoreto, tornando o meio ácido. Considerando que o titânio tem uma excelente biocompatibilidade e alguma resistência à corrosão, uma forma de melhorar esta propriedade é formando ligas de Ti com outros metais. A liga Ti-6Al-4V é a mais usada, apesar da sua toxicidade. Consequentemente, há necessidade de fazer novas ligas que sejam resistentes à corrosão mas menos tóxicas. Uma que se destaca é a Ti-15Mo. O objetivo desta revisão é comparar estas duas ligas em termos do comporta-mento à corrosão e possíveis tratamentos para melhorar a resistência à corrosão. Palavras-chave: Ligas de Titânio, Corrosão, Implantes Dentários, Osseointegração, Tratamentos de Superrcie P05","author":[{"dropping-particle":"","family":"Lopes","given":"Cátia S.","non-dropping-particle":"","parse-names":false,"suffix":""},{"dropping-particle":"","family":"Donato","given":"Mariana","non-dropping-particle":"","parse-names":false,"suffix":""},{"dropping-particle":"","family":"Ramgi","given":"P.","non-dropping-particle":"","parse-names":false,"suffix":"-"}],"container-title":"Corrosão e Protecção de Materiais","id":"ITEM-1","issue":"2","issued":{"date-parts":[["2016"]]},"page":"05-14","title":"Comparative corrosion behavior of titanium alloys (ti-15mo and ti-6al-4v. However, it has a toxicity content associated with aluminum and neurotoxicity or Alzheimer's disease (KAWAHARA *et al.*, 2020)aluminum is not essential for life. On the contrary, aluminum is a widely recognized neurotoxin that inhibits more than 200 biologically important functions and causes various adverse effects in plants, animals, and humans. The relationship between aluminum exposure and neurodegenerative diseases, including dialysis encephalopathy, amyotrophic lateral sclerosis and Parkinsonism dementia in the Kii Peninsula and Guam, and Alzheimer's disease (AD, in addition to microcytic anemia and osteomalacia (SCHIFMAN; LUEVANO, 2018). Therefore, other non-toxic elements are being proposed to replace aluminum (Al) and vanadium (V), such as niobium (Nb), zirconium (Zr), tantalum (Ta), molybdenum (Mo). These elements provide the alloy with good biocompatibility, elastic modulus, less than the Ti-6Al-4V alloy, and good corrosion resistance (AFONSO *et al.*, 2010).

Studies by Abu-Amer *et al.* stress "loosening" as a common situation in surgical-loaded implants, such as orthopedic and dental implants. They evaluate the elastic modulus of the biomaterial as a factor widely observed by researchers since, with an incompatibility in the elastic modulus, the bone, and the implant can lead to not obtaining the tensions necessary for bone remodeling, leading to an opposite result (bone resorption), due to stress shielding and thus leading to loosening and loss of the implant. (ABU-AMER; DARWECH; CLOHISY, 2007) The β titanium alloys with elements such as niobium, zirconium, and tantalum, have an excellent property of elastic modulus, which is around 55 GPa, being closer to the elastic modulus of the bone, which is 10-40 GPa (KURODA *et al.*, 2020; LONG, M; RACK, 1998), which reduces osteoclastic activity, favoring osseointegration. (ZHANG *et al.*, 2017)

Among the titanium alloys, in recent years, the quaternary alloy Ti-Nb-Zr-Ta (TNZT) has been highlighted in studies to present a system with excellent properties, less elastic modulus, good resistance to corrosion, and absence of toxic elements. (MIOTTO *et al.*, 2016; MIYAMOTO; SUDA, 2003)

Many studies emphasize the characteristics of the surface; physical (roughness and topography), physical-chemical (wettability), and chemical (corrosion resistance), as fundamental requirements in the cell-surface interaction, knowing that such properties are elementary in the control of the cellular response (adhesion, proliferation, and differentiation) (MIOTTO *et al.*, 2016; MIYAMOTO, T; SUDA, 2003; QAZI *et al.*, 2005). Surface treatments in titanium alloys have shown advantages for improving cellular response and favoring mechanical and chemical properties (ZHANG *et al.*, 2017). Laser treatment is a technology for surface modification that is gaining prominence in many kinds of research because it provides a clean process (free of residues and impurities), fast, and is repeatable (BRAGA *et al*, 2007; CUNHA *et al*, 2015; FAEDA *et al*, 2009). Researchers proved that surfaces modified by laser beam present better and more stable fixation to the bone to the machined ones and attributes this characteristic to the surface oxidation process, resulting in improved wettability and an increase in free surface energy (DONAGHY *et al.*, 2019; HALLGREN *et al.*, 2003). Many other studies show the surface modification by laser irradiation to obtain an antibacterial surface in metallic materials for biomedical use, such as Gallardo-Moreno *et al.*, showed a reduction in the adhesion rate of Staphylococcus aureus and Staphylococcus epidermidis cells (GALLARDO-MORENO *et al.*, 2009).

This study to evaluate the structural and microstructural characterization of the Ti-35Nb-7Zr-5Ta quaternary metal alloy, modified by a laser beam at the frequencies of 20Hz and 35Hz. The elastic modulus was analyzed using scanning electron microscopy (SEM) with elemental microanalysis by X-ray dispersive energy (EDX) and X-ray diffraction (XDR), aiming at exploratory data precedes the study of electrochemical corrosion tests and mechanical properties.

2 Material and Methods

The alloy elements (Ti, Nb, Zr, and Ta) were acquired in plates considering a degree of purity greater than or equal to 99.0%, and then cut into strips and pickled in an acid solution composed of H_2O, HF, HNO_3, H_2SO_4. They were melted in an arc fusion furnace (experimental) with an argon atmosphere, in which ingots of approximately 10 cm in length and 1 cm in thickness and weight 40 g were obtained and subjected to heat treatment at 1000 ° C for 8 hours, slow air cooling. Then, they were machined and cut to obtain specimens in the dimensions of 8.0 mm in diameter and 2.0 mm in thickness, which was separated into groups, with three specimens analyzed per group. Group I (specimen without surface modification), Group II (specimen with surface irradiated by laser at 20 Hz), Group III (specimen with surface irradiated by laser at 35 Hz).

The specimen was mechanically polished with 320 to 2000 mesh sandpaper and submitted to a cleaning process with isopropyl alcohol and distilled water, in ultrasound, for 30 minutes each. Polishing was carried out in an electric polisher (Arotec, Aropol 2V), with a cloth soaked in 0.5 µm granulation alumina, and again subjected to the cleaning process. To reveal the structure of Group I, the specimens was immersed for 10 seconds in a Kroll's reagent, composed of 50 ml of distilled water, 10 ml of HF, and 20 ml of HNO_3. Groups II and III specimens were separated and sent for Yb: YAG laser beam irradiation in the parameters mentioned in Table 1.

Table 1 - Parameters used for laser irradiation in the Ti-35Nb-7Zr-5Ta alloy

Laser Property	Parameters
Beam power (W)	maximum
Scan Speed (mm/s)	100
Space between scans (mm)	0.01
Pulse frequency (Hz)	20 -35
Average exposure area (mm)	0.8

Watts, mm/s - millimeters per second, mm - millimeters, Hz - Hertz frequency

Source: Research data

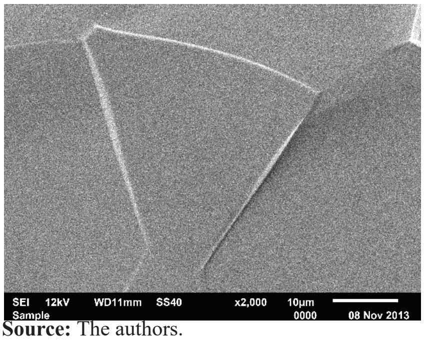
After cleaning, all specimens were analyzed by SEM with a JEOL-JSM microscope, model T-330 A. Coupled with a dispersive energy spectroscopic analysis system - EDX allows performing a semi-quantitative microanalysis of the alloy elements. An X-ray diffractometer (Rigaku, model system D / MAX - 2100 / PC was used with copper Ka radiation (1.5405 A) and a speed of 2 θ/min accelerated copper K source with a potential of 40 kV and a current of 20 mA and angular scan between 10 and 100°.

The elastic modulus analysis was performed using three samples on a disk of 8.0 mm in diameter X and 2.0 mm in thickness, in a planar module by ATCP Sonelastic equipment, connected to a computer with software dedicated to signal processing.

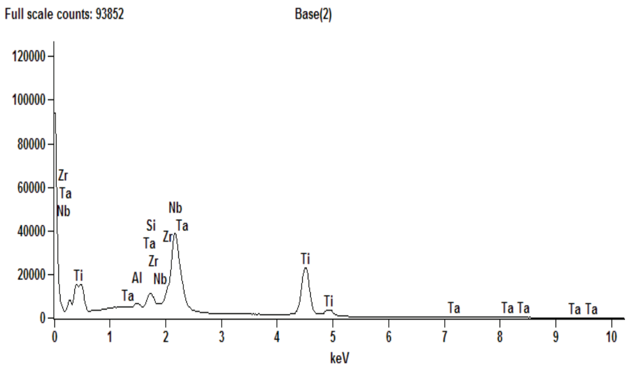
3 Results and Discussion

Figure 1, which represents the group of samples without surface treatment, mainly presented the β phase. The homogeneity of the structure and the alloy density can be attributed to the presence of regular grains, evidenced by Kroll's solution, in which the largest grains of size involving smaller grains are noted. Such results corroborate with data from the literature (LI *et al*., 2019) where the authors evaluate the microstructure and obtained similar results.

Figure 1 - Microstructural analysis in SEM / EDX group I (without treatment)



Source: The authors.



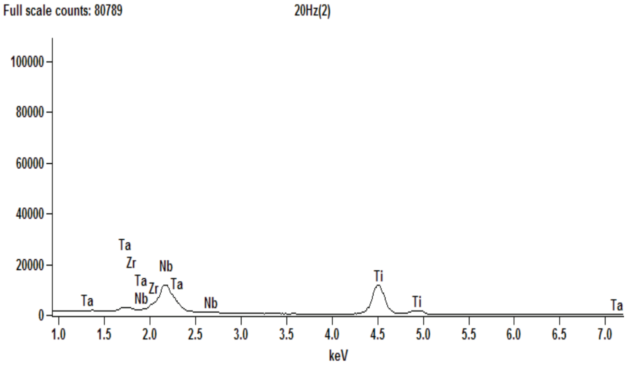
Source: Research data.

The oxide film is formed naturally in untreated samples and is only a few nanometers thick. In addition to being responsible for essential properties such as corrosion resistance and reduction in bacterial adhesion, data presented in studies by Quinn *et al.* (2020), where the claim the thickness of the film directly influences the number of load carriers on the surface of the oxide film. These load carriers are responsible for the surface/bacteria interaction, the thicker the oxide layer on the surface, the fewer the number of load carriers (JEYACHANDRAN; NARAYANDASS, 2010; QUINN *et al*., 2020). The studies by Donaghy *et al*., where they state the elemental composition of the quaternary alloy of the TNZT system in conjunction with surface treatment can improve osseointegration by cell dissemination (adhesion, proliferation, and differentiation) and reduction in bacterial adhesion, in addition to noting the presence of TiN in the oxide film formed on the surface, playing an essential role in improving the biocompatibility and antibacterial action of the biomaterial. (DONAGHY *et al.,* 2019)

In Figure 2, there is a change in the surface caused by laser beam irradiation, which can be called the ablation process. This process led to the rupture of the macroscopic topography of the substrate surface, providing an increase in roughness and surface area. An important feature for osseointegration, favoring the cellular response.

Figure 2 - Microstructural analysis in SEM / EDX group II (20 Hz laser)

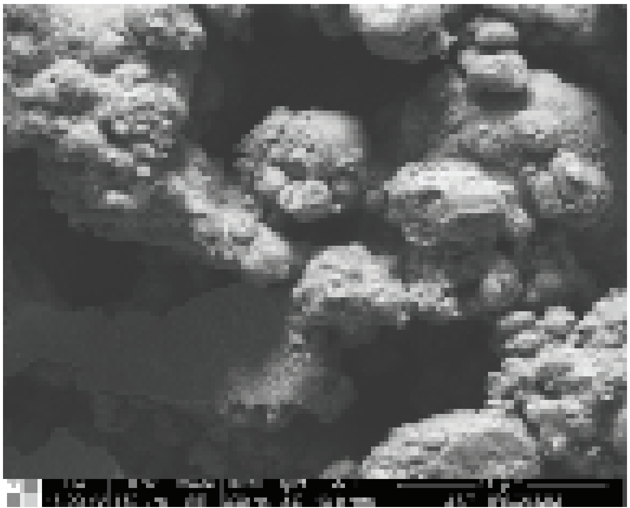
Source: The authors.



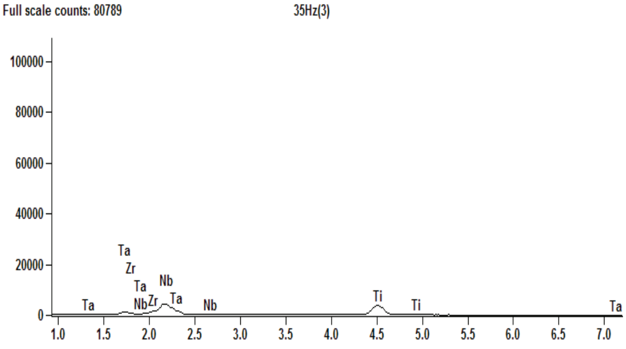
Source: Research data

Figure 3 shows the micrograph of the alloy surface after irradiation by laser beam at 35 Hz. An increase in roughness and surface area is observed, as well as globular structures formed on the surface due to the process of melting and rapid solidification of the metal.

Figure 3 - Microstructural analysis in SEM / EDX group III (35 Hz laser)



Source: The authors.



Source: Research data

On both surfaces modified by laser beam, it was found the ablation process did not cause loss of elementary stoichiometry, data are confirmed by the EDX analysis. Although there is a high concentration of Ti, it was possible to observe the evident presence of Nb and Ta (β-stabilizing elements). Since Zirconium is a neutral element, it acquired the β-stabilizer function, and is also observed in the oxide layer.

Table 2 shows the levels of interstitial gases (oxygen) with relevant concentrations for the specimens irradiated by laser. Therefore, all specimens showed slight variation when the experimental values were compared. The chemical composition of the alloy without surface modification was homogeneous, with significant differences between the volume and the surface in relation to the specimen with surfaces irradiated by laser, caused by the formation of the thick oxide layer.

Such results show the crystalline structure of the titanium-based alloy with the addition of β-stabilizing elements, such as Nb, Zr, and Ta, is sensitive to the concentration of such elements characterizing homogeneous distribution. Conserving the proposed stoichiometry, which corroborates with data from the literature as a study by Quadros *et al.*, where the structural and microstructural characteristics of a titanium-based alloy with the addition of β-stabilizing elements such as tantalum and zirconium were evaluated. (QUADROS *et al.*, 2019)

Table 2 - Elemental composition of the Ti-35Nb-7Zr-5Ta alloy by the EDX technique, under the three conditions analyzed in relation to the ideal mass composition

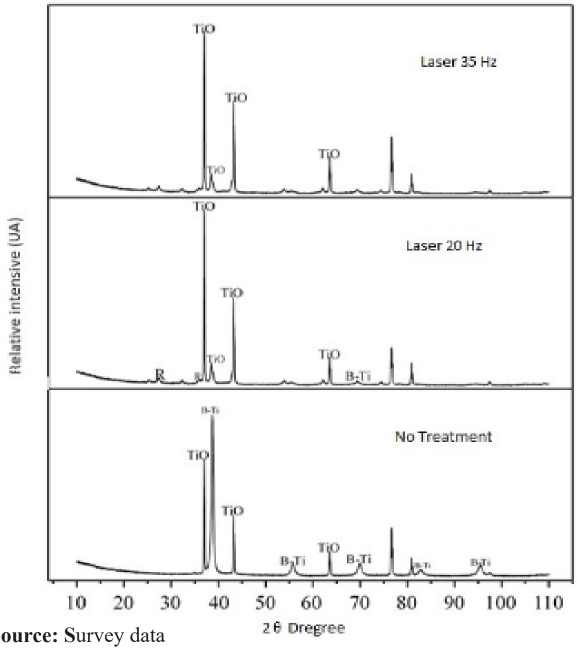
Elements	Composition (%)			
	Ti-35Nb-7Zr-5Ta (ingot approximate percentage)	Ti-35Nb-7Zr-5Ta (No surface modification)	Ti-35Nb-7Zr-5Ta Laser 20 Hz	Ti-35Nb-7Zr-5Ta Laser 35 Hz
Titanium (Ti)	53	48	44	42
Niobium (Nb)	35	35	19	21
Zirconium (Zr)	7	8	5	5
Tantalum (Ta)	5	8	2	3
Aluminum (Al)	_____	0.90	_____	_____
Silicon (Si)	_____	0.09	_____	_____
Oxygen (O)	_____	_____	30	29

Hz - Hertz frequency

Source: Research data

Figure 4 shows an X-ray diffraction analysis of the Ti-35Nb-7Zr-5Ta alloy in which the presence of the oxide layer on the surface is observed. The results obtained are per the SEM and EDX analyses (TiO: titanium oxide, JCPDS No. 65-2900, β-Ti: Beta Titanium, JCPDS No. 44-1288), in addition to indicating the predominance of the β phase, (R: TiO_2 Rutile, JCPDS No. 86-147), on surfaces irradiated by laser at both 20 Hz and 35 Hz, marks the presence of the rutile phase, which is a mineral formed from titanium dioxide, considered the more stable form of the oxide. Tantalum and niobium are β-stabilizing elements. The presence of these elements causes zirconium, a neutral element, to act on the β phase stability, giving verity to previous tests. Similar results can be observed in the literature (QUADROS *et al*., 2019)

Figure 4 - X-ray diffraction of the Ti-35Nb-7Zr-5Ta alloy, under conditions: untreated and with surface modification by laser beam irradiation at 20 Hz and 35 Hz



Source: Survey data

The results obtained in the elasticity tests were values around 63 GPa. Table 3 presents the microstructural characteristics of the titanium-based metal alloy, compares the characteristics of the cortical bone, and reports the proximity of the elastic modulus of the alloy in study Ti-35Nb-7Zr-5Ta, with the elastic modulus of bone.

Table 3 - Ti-35Nb-7Zr-5Ta alloy characteristics for use in biomedical devices, in relation to cortical bone

Material Specification	Microstructure	Young's Modulus E(GPa)	References
Ti.cp	**α**	105	Long, Mark and Rack (1998)
Ti-6Al-4V	**α/β**	101-110	Kuroda et al. (1998).
Ti-35Nb-7Zr-5Ta	**β**	55	Kuroda et al. (1998).
		63	Present study
Cortical Bone	Viscoelastic composite (calcium phosphate / collagen)	10-40	Long, Mark and Rack (1998)

Ti cp - Commercially pure titanium, Ti-6Al-4V - Ternary metallic alloy titanium, aluminum, vanadium, Ti-35Nb-7Zr-5Ta - Quaternary metal alloy titanium, niobium, zirconium, tantalum.

Source: Survey data

Because it is a metastable β alloy and has an elastic modulus awfully close to of bone, which differs from Ti-cp (α) and the commercial TAV alloy (α + β) used in the manufacture of implants, the Ti-35Nb-7Zr-5Ta can contribute to a better transfer of load to the adjacent bone. The avoidance of possible bone resorption due to elastic incompatibility and effectiveness promotes bone remodeling (HALLGREN *et al*., 2003; UMMETHALA *et al*., 2020). A recent study (KAUR; SINGH, 2019; MIOTTO *et al*., 2016) discussed it as one of the metal alloys present more favorable and satisfactory results implanted in a cortical bone. (HALLGREN *et al*., 2003; QUINN *et al*., 2020)

Studies carried out by testing the phase transformation of the quaternary metal alloy Ti-35Nb-7Zr-5Ta with the addition of O (0.06 - 0.68) found the elastic modulus can be altered by the element oxygen (O), as it increases the concentration of O and the temperature of aging of the alloy. (QAZI *et al*., 2005)

In biological terms, the lower the elastic modulus of a biomaterial, the better its performance in contact with bone regarding mechanics. This is a determining factor in bone fixation since the mechanical incompatibility between bone/implant is minimized. (KURODA, *et al.*, 2020; LONG, M; RACK, 1998)

According to Brailovski *et al.*, a biomedical device to be used intraosseous must have a porous structure so it is possible to join with live bone and classifies as challenging the mechanism of production of porous material with a "superelasticity," which performs the function bone mechanics and has a biochemical and osteogenic affinity. This study reports Young's modulus of the almost β Ti-Nb-Zr (Ta) alloys, which is between 45-55GPa, approaches the elastic modulus of the cortical bone, which makes it a material of choice for orthopedic implants. While the elastic modulus of the porous samples of this same alloy, which is in the range of 1.5 to 5 GPa, in which the degree of porosity is considered, is closer to the elastic modulus of human trabecular bones. (BRAILOVSKI *et al*., 2011)

4 Conclusion

Surface modification methods favor the protection of the biomaterial and the osseointegration process, positively influencing aspects such as roughness, wettability, and increased surface energy, which can bring improvements in the cellular response. Therefore, we conclude with the results of this study:

- The experimental alloy Ti-35Nb-7Zr-5Ta has microstructural characteristics favor its use in biomedical devices,
- The surface modification by laser beam created a thicker oxide film and greater roughness, maintaining the homogeneity of the alloy elements and improving the topographic characteristics of the biomaterial, an essential factor in favor of the interaction with the bone tissue.

Clinical Implication

The experimental alloy Ti-35Nb-7Zr-5Ta presents a better elastic modulus in relation to the other alloys used in biomedical devices. When subjected to surface treatment, it presents improvements in its structural characteristics, which indicates a protection against aggressive media, minimizing the release of metal ions in the body.

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