

Evolução na Ciência e Engenharia de Materiais

Henrique Ajuz Holzmann
(Organizador)



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APRESENTAÇÃO

A engenharia de materiais, se tornou um dos grandes pilares da revolução técnica industrial, devido a necessidade de desenvolvimento de novos materiais, que apresentem melhores características e propriedades físico-químicas. Grandes empresas e centros de pesquisa investem maciçamente em setores de P&D a fim de tornarem seus produtos e suas tecnologias mais competitivas.

Destaca-se que a área de material compreende três grandes grupos, a dos metais, das cerâmicas e dos polímeros, sendo que cada um deles tem sua importância na geração de tecnologia e no desenvolvimento dos produtos. Aliar os conhecimentos pré-existentes com novas tecnologias é um dos grandes desafios da nova engenharia.

Neste livro são explorados trabalhos teóricos e práticos, relacionados as áreas de materiais, dando um panorama dos assuntos em pesquisa atualmente. Apresenta capítulos relacionados ao desenvolvimento de novos materiais, com aplicações nos mais diversos ramos da ciência, bem como assuntos relacionados a melhoria em processos e produtos já existentes, buscando uma melhoria e a redução dos custos.

De abordagem objetiva, a obra se mostra de grande relevância para graduandos, alunos de pós-graduação, docentes e profissionais, apresentando temáticas e metodologias diversificadas, em situações reais.

Boa leitura!

Henrique Ajuz Holzmann

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IMPROVEMENT OF TITANIUM SURFACE WITH PLASMA NITRIDING TREATMENT

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ABSTRACT: Titanium is a biomaterial used mostly in the production of prostheses with the aim of replacing joints in medical areas and dentistry. Many methods have been used in the treatment of titanium surfaces aiming at better interaction with the biological environment, and should lead to osteointegrations and consequently to successful implantation. Commercially pure grade two titanium discs were submitted to two methods of surface modification: polish and plasma nitriding in planar configuration. Different surfaces were characterized to observe the effect of processing in the structure, roughness, and wettability of the superficial layer. Titanium disk samples were nitrided using a hollow cathode discharge (HCD) configuration of a standard plasma nitriding system in a N_{20%}H_{80%} atmosphere at pressures of 2,5 mbar, and a temperature of 450°C for 1 hour.

KEYWORDS: titanium; plasma nitriding; planar configuration.

1 | INTRODUCTION

Studies in the Implant Dentistry area have rapidly expanded during the past 40 years. The recognition that titanium implants could be used for oral rehabilitation of edentulous patients

has stimulated much research, leading to higher success rates (ALVES e WASSALL, 2009). The titanium biocompatibility is attributed to its surface properties, most notably to an oxide layer that protects the corrosion of the material and also allows a favorable environment for initial cell events responsible for osteointegration (CARNEIRO e FERNANDES, 2010).

Most of the reactions in biology occur on surfaces, not in solutions. Therefore, the modifications made in material target the selective interactions with specific cell types by the recognition of biomolecular events. The surface should be developed for the precise deposition and orientation of the proteins. This way, the body will specifically recognize (CASTNER e RATNER, 2002; TIRREL et al., 2002). The surface properties of the biomaterial could be modified by physical and chemical means, such as plasma spraying, grit blasting, acid etching, and anodization (GUÉHENNEC et al., 2007). Plasma nitriding has been described as a promising method in the modification of titanium surfaces due to the characteristics of the process in improving the performance of numerous superficial properties such as roughness, wettability, and chemical composition. This leads to a favorable environment for cell adhesion while maintaining the material biocompatibility (CZARNOWSKA et al., 2000; NEBE et al., 2007).

In plasma nitriding, metallic surfaces are put in contact with a plasma made by the application of voltage between two electrodes in a sealed reactor and completed by a nitriding gas (usually mixtures of H₂-N₂) at a pressure ranging from 1 to 10 mbar. As the voltage is applied between the electrodes, electrons are accelerated in the direction of the cathode, colliding with atoms and gas molecules, ionizing atoms and exciting other species that form the plasma. The accelerated ions collide with the surface of the titanium, making the superficial characteristics (GUERRA NETO et al., 2009 a). Two settings are obtained depending on the nitriding protocol: planar configuration or cathodic cage. Polished titanium, nitrided in planar configuration and cathodic cage, exhibit different characteristics on the surface, but it is known that this treatment increases the superficial roughness and causes changes in the oxide layer thickness. When comparing the treated with non-treated samples, most likely giving two important properties for this surface: great hydrophilicity, which ensures the preserved state of the adsorbed proteins, and greater resistance to the corrosion process, which avoids the occurrence of oxide/reduction reactions (SÁ et al., 2009). Studies show that the removal torque of the nitriding surfaces is twice that of non-treated surfaces. Additionally, the osteointegrated perimeter is larger than other treated surfaces from this process (GUERRA NETO et al., 2009 b; KLEIN et al., 2010). In the present work, a hollow cathode discharge was used for the nitriding of Ti discs and studied the presence of nitride phases on the surface, surface texture, roughness and wettability.

2 | MATERIAL AND METHODS

Pure titanium grade II samples were used, measuring 15mm of diameter and 1,5mm

thickness, prepared at the Laboratory of Plasma Processing Materials (LabPlasma) and divided into three groups: experimental group 1, n=18 discs submitted to plasma nitriding treatment at planar configuration and group 2 at cathodic cage. Group 3, n=18 discs just polished. The discs were polished with grit silicon carbide sandpaper in running water and polished (AROTEC) in colloidal silica (SiO₂) suspended with 0.1- μ m particles. Until a final finishing left the surface rinsed in an ultrasound bath with enzymatic detergent, distilled water and acetone for 10 minutes each to remove contaminants, dried at room temperature, and appropriately conditioned. The samples placed in a sealed stainless steel chamber (reactor). Two techniques used in a plasma reactor to compare the effects on the sample surfaces: planar and cathodic cage nitriding. Alves et al, 1995, developed the nitriding protocol used.

The analysis by electron microscopy scanning for texture characterization and X-ray diffraction (XRD) for crystallographic structure and chemical composition conducted at the Northeast Center of Strategic Technologies – CETENE. Three samples of each group analyzed by the atomic force technique (AFM) for roughness. The wettability analyzed by determination of the static contact angle or sessile drop technique. The topographic characterization and superficial roughness realized at three samples of each group through the electron microscopy scanner –SEM– (PHILIPS). Each sample individually evaluated for the achievement of a surface image and therefore characterization, giving the possibility of comparing a treated surface with a non-treated surface. For X-ray diffraction, four samples of each group used. Surface phases studied using Shimadzu X-ray diffraction equipment. For X-ray incidence, two configurations were used: Bragg-Brentano and grazing with a 5° beam. Both used CuK α radiation and grazing angles between 30° and 80°, steps of 0,02 and 0,6 seconds for step at a 2/min speed. To analyze chemical composition, Energy Dispersive Spectrometry (EDS) with the SEM, which used to observe the surface texture, submitted three samples of each group to analysis. To analyze wettability, eight samples of each group used. An adjustable-volume digital micropipette positioned perpendicularly and very close to a flat surface used to deposit 0.25 mL of a saline solution onto the surface of the sample. To standardize the test and because of the very small drop size, angle changes monitored at 1, 30 and 60 seconds.

3 | RESULTS

3.1 WETTABILITY

Wettability of 8 samples from each group was obtained by the determination of the static contact angle for each sample measured by the program SurfTens 3.0.

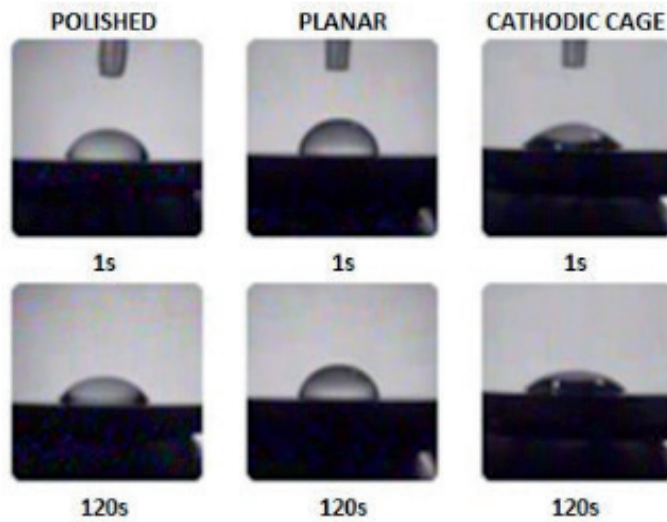
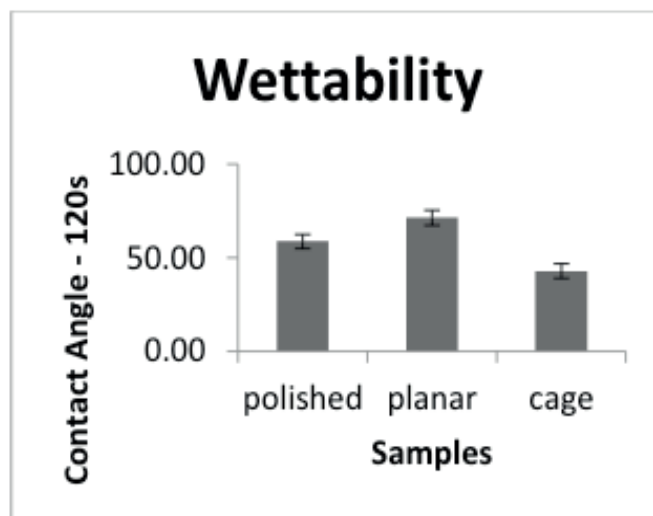


Fig.1 – Contact angle measurement in 1second and 120 seconds at titanium different surfaces.

The contact angle measured at 1 and 120 seconds after the contact drop with the titanium surface. There was a reduction of the contact angle in the course of the time at the same surface, and a higher wettability was observed at the cathodic cage when the 3 groups were compared. These results can also be observed in table I.



Graphic I – Average of the contact angle, in degrees, after 120 seconds at different titanium surfaces.

WETTABILITY RESULTS

SAMPLES	Polished		Planar		Cathodic Cage		CONTACT ANGLE
	1s	120s	1s	120s	1s	120s	
	1	65.1	58.32	82.19	77.77	53.12	
2	65.58	63.88	79.94	76.49	46.23	43.87	
3	64.18	57.72	79.46	72.14	48.28	38.14	
4	60.6	57.6	78.19	70.74	47.22	44.25	
5	63.72	56.73	78.96	74.45	48.56	45.12	
6	65.89	54.65	79.83	69.79	50.8	43.18	
7	59.2	55.45	68.96	64.89	46.37	39.98	
8	68.02	65.21	81.86	76.67	47.55	39.02	
mean	6.403.625	58.695	7.867.375	7.144.667	4.851.625	4.298.875	
dp	2.881.943	3.824.227	415.327	4.074.745	2.357.813	3.954.168	

Table I – Contact angle values in degrees.

3.2 NANOTOPOGRAPHY

The AFM analysis conducted to obtain information about the surface topography (texture and roughness). Table II shows the roughness parameters at nm.

AFM RESULTS AT NANOMETERS

TREATMENT		Ra	Rz	Rq	Rp	Rv
Polished	1	0.636	13.190	0.804	5.434	7.756
Polished	2	0.429	9.734	0.588	6.681	3.053
Polished	3	0.810	9.223	1.171	6.356	2.867
	mean	0.625	10.716	0.854	6.157	4.559
	dp	0.191	2.158	0.295	0.647	2.771
Planar	1	2.902	42.598	3.964	13.097	29.501
Planar	2	3.833	51.711	5.281	17.481	34.320
Planar	3	3.770	54.302	5.704	21.039	33.263
	mean	3.502	49.537	4.983	17.206	32.361
	dp	0.520	6.147	0.907	3.978	2.533
Cage	1	3.772	42.113	4.716	26.019	16.095
Cage	2	3.026	43.730	4.220	26.119	17.045
Cage	3	3.089	42.890	4.879	27.078	16.028
	mean	3.296	42.911	4.605	26.405	16.389
	dp	0.414	0.809	0.343	0.585	0.569

Table II – Roughness parameters obtained with AFM in micrometers.

ROUGHNESS PROFILE (Rp/Rz)

Polished	0.575
Planar	0.347
Cage	0.615

Table III – Roughness profile obtained by the division Rp/

The figures 2,3 and 4 represent the surface topography at 2D ,3D and the roughness profile of the polished sample, one submitted to glow discharge at cathodic cage and one at planar configuration, respectively.

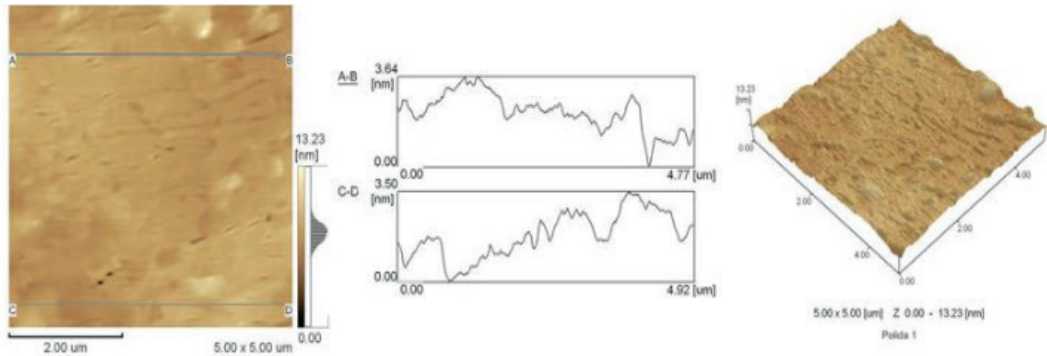


Fig.2 - Surface topography of a 5 μm area at polished sample. 2D on the left, roughness profile at the center, 3D topography on the right.

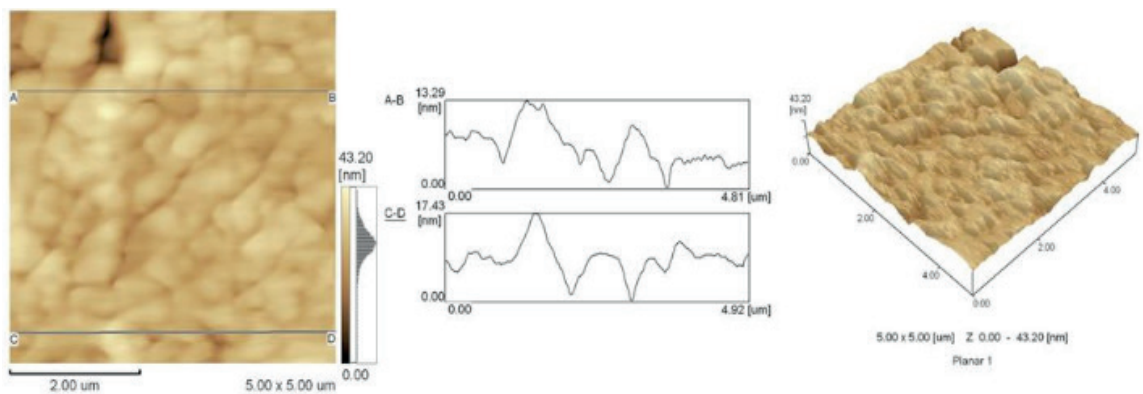


Fig.3 - Surface topography of a 5 μm area at planar configuration. 2D on the left, roughness profile at the center, 3D topography on the right.

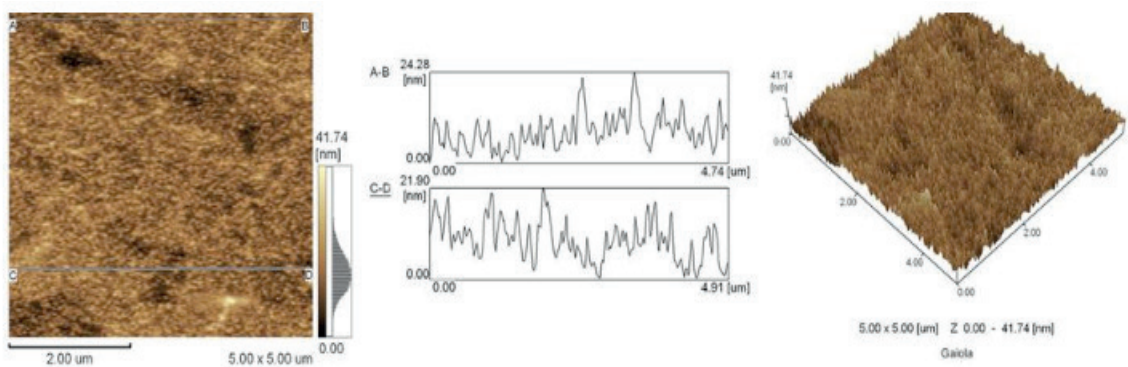


Fig.4 - Surface topography of a 5 μm area at cathodic cage. 2D on the left, roughness profile at the center, 3D topography on the right.

3.3 Rx DIFFRACTION

The Bragg-Bretano geometry and the Grazing angle used to analyze the titanium phases. With Bragg-Bretano the interstice is shown more evidently and with the Grazing angle we can obtain more information about the surface. The pictures 5 and 6 show the diffractogram. The titanium card used to identify the peaks was Ti 44-1294.

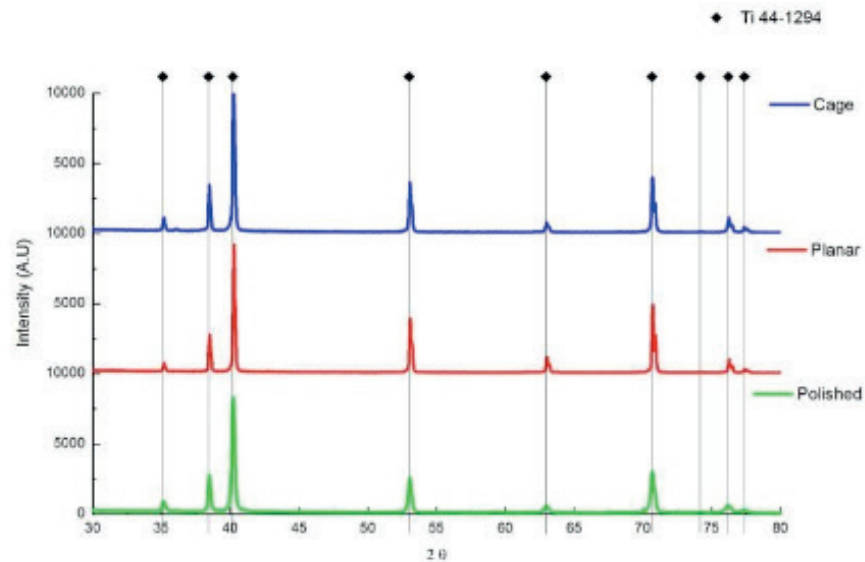


Fig. 5 – X-ray spectrum realized at Theta-2-theta.

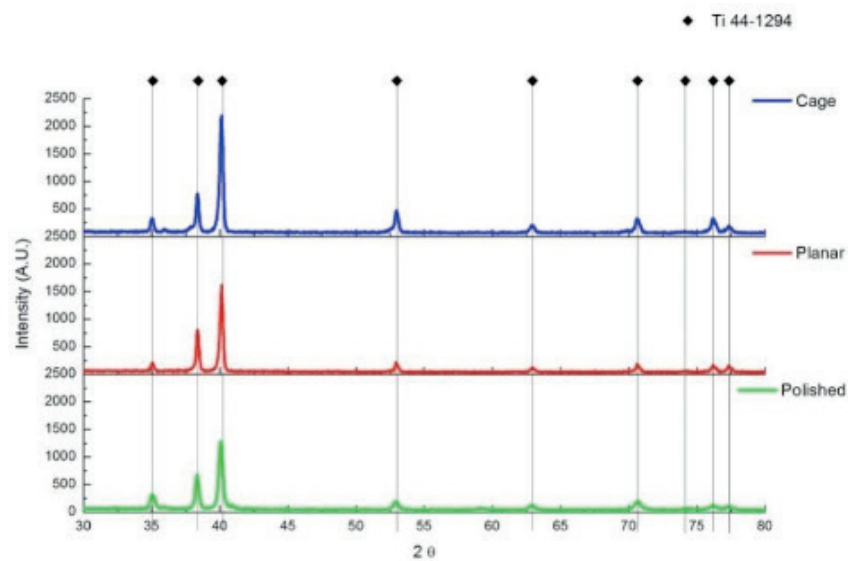


Fig. 6 – X-ray spectrum realized at grazing angle.

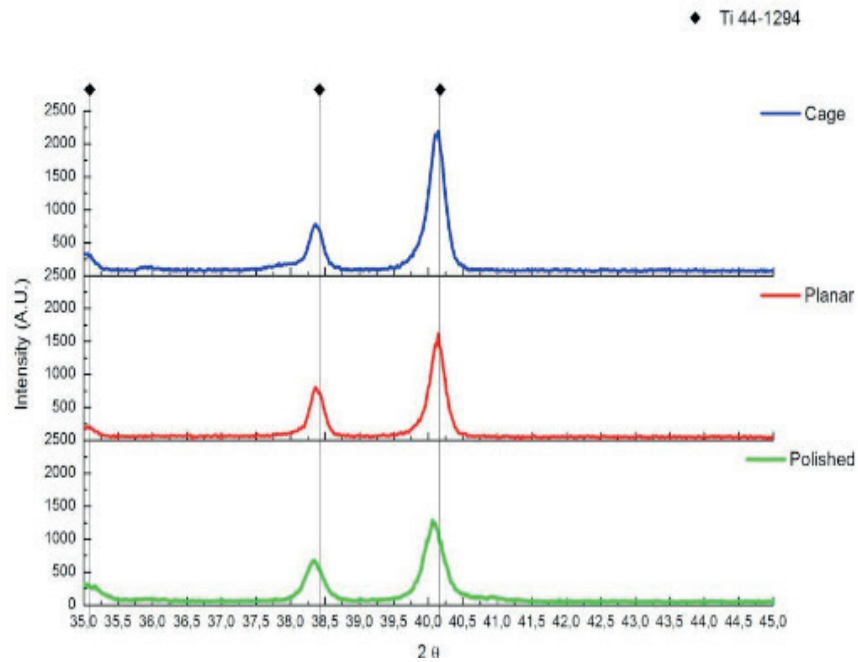


Fig. 7 – More detailed view where the displacement of the peaks can be observed.

3.4 EDS AND SEM

To analyze chemical composition the samples were submitted to analysis by Energy Dispersive Spectrometry (EDS) with the scanning electron microscopy (SEM) aiming the study of the chemical composition of the surface and the observation of the surface morphology.

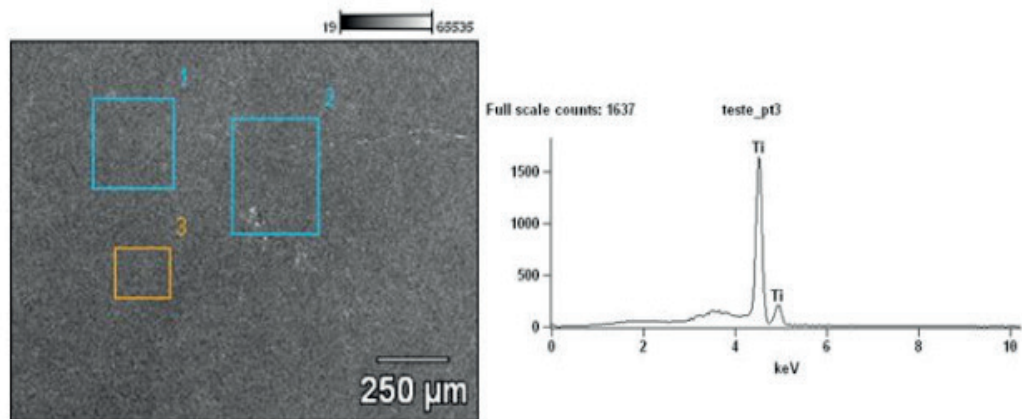


Fig.8 – SEM (original magnification 80x) and EDS of a Polished disc showing only Titanium at surface.

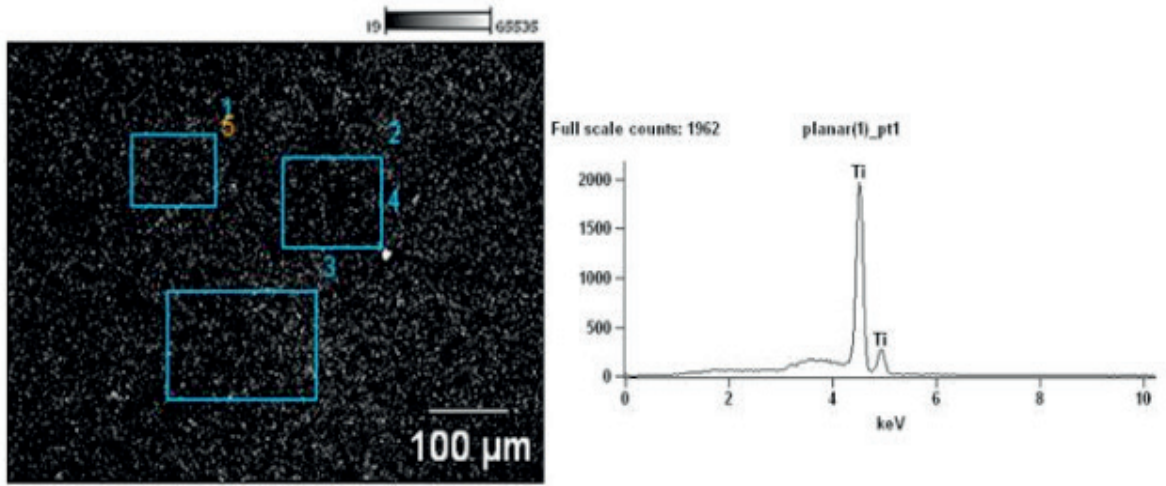


Fig. 9 – SEM (original magnification 200x) and EDS of a disc submitted to a nitriding process at planar configuration. .showing only Titanium at surface.

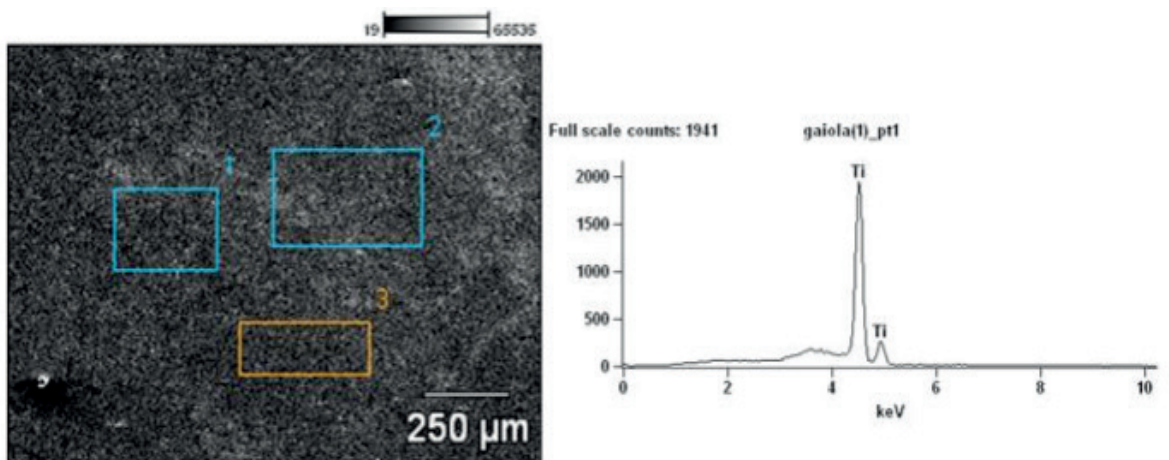


Fig. 10 – SEM (original magnification 85x) and EDS of a disc submitted to a nitriding process at cathodic cage showing only Titanium at surface.

3.5 STATISTICAL ANALYSIS

Tabela x. teste de Kruskal-Wallis

Variáveis	Agrupamento	n	Mediana	Q25 – Q75
Ângulo de contato	Polished a	8	57,66*	55,77 – 62,49
	Planar b	8	73,29*	70,02 – 76,62
	Cathodic Cage c	8	43,52*	39,26 – 44,90
Rugosidade Média	Polished a	3	0,64	0,53 – 0,72
	Planar a	3	3,77	3,33 – 3,80
	Cathodic Cage a	3	3,08	3,05 – 3,43

*valor de $p < 0,05$ entre os grupos.

_{a,b,c} pós-testes entre os grupos com Mann-Whitney e penalizações de Bonferroni.

4 | DISCUSSION

The organism response to the implant is influenced by the implant characteristics as design, biocompatibility, mechanical properties, surface properties and as well characteristics of the individual, like the osseo quality of the surgical bed. Once the implant is placed at the surgical bed, the fluids get in contact with it. The surface of the implant will get in contact with proteins presents in blood. The adsorption of the proteins (their conformation) is crucial for the cell behavior with the implant, which will lead to an osteointegration or to the formation of a fibrous capsule that means the implant failure. Between the parameters that influence the osseointegration, the surface tension is one of the most important (SILVA et al., 2011) , because allows a better spreading of the liquids over metallic surfaces. The blood has 99% of water, so the wettability is so important to the cell behavior, and consequently, to osseointegration (SCHAKENRAAD, 1999; KASEMO, 2002). The chemical composition, the topography and the roughness, with the energy of surface appears as relevant factors to the success of the implant (DONACHIE, 1982; HAZLETT, 1992; GILJEANA et al., 2010).

The three groups formed a distinct texture on the surface. The treated samples showed a texturized surface with amplitude variations on all surfaces, while the polished samples did not. The untreated discs presented a plain surface, with a maximum peak value of 13.23 nm, with evidence of groove and crest direction due to sanding and polishing. In addition, from the roughness profile, peaks and valleys with low intensity could be observed, and small values of the roughness parameters (R_a , R_z , R_q , R_p and R_v) indicate a smooth surface. In the cathodic cage group, the maximum peak value was 41.74 nm . Peaks and valleys emerged with higher intensity and presented themselves evenly. This is consistent with the kind of treatment which produces a regular film without edge effect (PONSONNET, 2003). The roughness parameter values are higher than the polished, thus the surface texture is much rougher. At planar configuration the roughness parameters also presented a surface with higher values compared to the polished, with a maximum peak value of 43.20 nm, Comparing the two treatments, the planar profile was more delicate, with peaks and valleys more rounded, compared with the cathodic cage profile which was more intense, with peaks and valleys more close to each other. Thus, the surface profile of roughness at planar configuration is not as acute as the one at cathodic cage.

So, independent of the analyzed roughness parameter, the treated samples showed higher values than the non-treated. These results are in agreement with the literature, the researchers also found that treated samples presented higher values of roughness than the untreated samples (ALVES, 2006; SILVA, 2006). R_p is defined as the maximum height of the profile above the mean line within the assessment length and R_z is defined by. The International ISO system as the difference in height between the average of the five highest peaks and the five lowest valleys along the assessment length of the profile. The reason between R_p and R_z (R_p/R_z) give the roughness

profile, thereby could be observed a more rounded surface if the $R_p/R_z < 0.5$ and a more acute surface if the $R_p/R_z > 0.5$. The sharpest surface is more favorable to wettability (Flower, 1992; WHITEHEAD, 1995). Analyzing the results shown in Table III, the polished samples is the result more close to 0.5, so it's the more flat surface. The planar samples with value 0.347 presents more rounded peaks and the samples treated at cathodic cage obtained a value of 0.615 which indicates a sharp surface. This result is consistent with the wettability, as described below.

The behavior of the proteins on the surface of the titanium implant is dependent on the surface properties, especially adsorption and adhesion. The implant surface wettability (hydrophobic or hydrophilic) influences cell behavior in the initial osseointegration process (WHITEHEAD, 1995; GADELMAWLA, 2002) Wettability was different for each surface. The samples treated at cathodic cage presented the lowest values of the contact angle. Analyzing the roughness, the peaks and vales well distributed around the surface could favor the wettability, whereas the rounded profile with peaks and valleys less acute and more spaced contributed with a higher contact angle, so a less hydrophilicity. The results show that surfaces with higher values of roughness ($R_a = 3,772$ nm) have lower contact angle. Thus, this surface is hydrophilic and polar at cathodic cage. The regular pattern of roughness reduces the possibility of contamination at the microdeformations may contributing to the hydrophilicity. Studies showed that roughness influences directly on wettability, but, so does the cleaning process used on the samples, the chemical heterogeneity of the surface and the surface energy (GADELMAWLA, 2002; LIU, 2004).

Titanium is presented as an element allotropic, that means it exists in more than one crystalline form. At room temperature presents a compact crystalline structure of hexagonal type (HC) that is phase α . This structure becomes a body-centered cubic (CCC), termed phase β , at a temperature of 882.5°C . It is a transitional element of group IVB and can form compounds divalent, trivalent and tetravalent. The valence variable is a characteristic of transition elements because they have incomplete internal electronic orbitals (GADELMAWLA, 2002).

The X-ray diffraction showed that the principal phase founded was Ti- α . This result is in agreement with the literature (SILVA,2006), which states that the Ti-cp presents only the alpha phase until 883°C (GUERRA NETO et al., 2009). At the grazing angle, the amount of information about the surface is larger, and a displacement of the main peaks of the Ti- α can be observed when compared to the titanium without treatment. This indicates a solid solution of interstitial atoms (nitrogen) at titanium (fig. 7). The Bragg law explains this in stating that the angular position is inversely proportional to interplanar distance. In this way, there was a reduction in interplanar distance at a phase promoted by the insertion of nitrogen atoms at titanium network (displacement of the peaks to the right). Despite the absent of TiN on the surface of the treated samples, which is shown at.

To analyze the morphology of the surface, the SEM was applied. The reference

surface (polished disc) showed a smooth morphology without characteristic features. Instead, the morphology of the treated samples highlights the precipitated composites formation on the nitrided sample surface, not detected on the X-Ray Diffraction. Maybe the presence of these precipitates can be an important factor for the wettability evaluation and it's relation with the roughness .

To analyze the chemical composition of the surface, the EDS was realized. It was observed that the main composition of the surface is Titanium at all the three analyzed groups. This is because the EDS machine has difficult to identify elements with atomic number less than 11 ($Z < 11$ – $Z < \text{Na}$) 23. To more refined research, the XPS, which detect all the elements but Hydrogen and Helios, must be applied at these surfaces.

5 | CONCLUSIONS

The results obtained with the methodology used at this work, led to following conclusions:

The nitrided samples with different treatments produced distinct topographies at nanometric level.

The treatment at cathodic cage produced a surface with higher hydrophilicity.

The treatment provoked a displacement of the main peaks of the Ti- ξ , which indicates a solid solution of interstitial atoms (nitrogen) at titanium.

The MEV indicates the precipitated composites formation at the Planar and Cathodic Cage configuration.

The EDS was not effective of showing the formation of nitrides.

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