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Corrosion behavior and antimicrobial activity of titanium and Ti-30Ta alloy

Atividade antimicrobiana e corrosão do titânio e da liga Ti-30Ta

Comportamiento a la corrosión y actividad antimicrobiana del titanio y la aleación Ti-30Ta

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Abstract

The global market of dental implants is expecting to make USD 13.01 billion circulate by 2023 from USD 9.50 billion in 2018. The study of the materials to fabrication the implants has increased with the objective of extending the useful life of the material avoiding having to replace. Therefore, in this study we investigated the electrochemical behavior, wettability characterization and antimicrobial activity of titanium and the Ti-30Ta binary alloy. The titanium was cut into discs of 10 mm in diameter. The Ti-30Ta alloy was obtained from titanium and tantalum pure metals in a high purity argon atmosphere, homogenized in a vacuum at 1000ºC for 24 hours, cold -worked by a rotary swaging process and solubilized at 950 ºC for 2 hours followed by water cooling. Then, the bars were cut into discs. Corrosion resistance tests evaluated the electrochemical behavior, the wettability of the substrate surfaces was investigated using a sessile drop method and the biofilm formation was investigated by of S epidermidis. This study aims to investigate the corrosion resistance of Ti cp and the Ti-30Ta alloy at electrolyte solution NaCl 0,15M + NaF 0,03M and tested biofilm formation. From the results obtained, we concluded that the electrochemical behavior of both surfaces shows good resistance to corrosion solution and hydrophilic (< 90 °) behavior. However, the Ti-30Ta alloy decreases the adhesion of *S epidermidis* bacteria.

Keywords: Titanium; Ti-30Ta alloy; Corrosion behavior.

Resumo

O mercado global de implantes dentários estima o valor de US $ 13,01 bilhões até 2023 contra US $ 9,50 bilhões em 2018. O estudo dos materiais para a fabricação dos implantes tem aumentado com o objetivo de estender a vida útil do material evitando sua substituição. Assim, neste estudo investigamos o comportamento eletroquímico de corrosão, molhabilidade e atividade antimicrobiana do titânio e da liga binária Ti-30Ta. O titânio foi cortado em discos de 10 mm de diâmetro. A liga Ti-30Ta foi obtida a partir de titânio e tântalo em forno a arco voltaico, homogeneizada em vácuo a 1000ºC por 24 horas, trabalhada a frio e solubilizada a 950 ºC por 2 horas seguido de resfriamento em água . Em seguida, as barras foram cortadas em discos. Os testes de resistência à corrosão avaliaram o comportamento eletroquímico, a molhabilidade das superfícies do substrato foi investigada usando o método da gota séssil e a formação de biofilme foi investigada por de *S epidermidis.* Este estudo tem como objetivo investigar a resistência à corrosão do Ti cp e da liga Ti-30Ta em solução eletrolítica NaCl 0,15M + NaF 0,03M e formação de biofilme testado. Pelos resultados obtidos concluímos que o comportamento eletroquímico de ambas as superfícies apresenta boa resistência à corrosão e comportamento hidrofílico (< 90 °). No entanto, a liga Ti-30Ta diminui a adesão da bactéria *S epidermidis.*

Palavras-chave: Titânio; Liga Ti-30Ta; Comportamento de corrosão; Atividade antimicrobiana.

Resumen

El mercado global de implantes dentales espera hacer circular USD 13.01 mil millones para 2023 desde USD 9.50 mil millones en 2018. El estudio de los materiales para la fabricación de los implantes se ha incrementado con el objetivo de extender la vida útil del material evitando tener que reemplazarlo. Entonces, en este estudio investigamos el comportamiento electroquímico, la caracterización de la humectabilidad y la actividad antimicrobiana del titanio y la aleación binaria Ti-30Ta. El titanio se cortó en discos de 10 mm de diámetro. La aleación Ti-30Ta se obtuvo a partir de metales puros de titanio y tántalo en una atmósfera de argón de alta pureza, homogeneizado al vacío a 1000ºC durante 24 horas, trabajado en frío mediante un proceso de estampación rotatoria y solubilizado a 950 ºC durante 2 horas seguido de enfriamiento con agua Luego, las barras se cortaron en discos. Las pruebas de resistencia a la corrosión evaluaron el comportamiento electroquímico, se investigó la humectabilidad de

las superficies del sustrato utilizando un método de gota sésil y la formación de biopelículas fue investigada por *S epidermidis*. Este estudio tiene como objetivo investigar la resistencia a la corrosión de Ti cp y la aleación Ti-30Ta en solución electrolítica NaCl 0,15M + NaF 0,03M y la formación de biopelículas probadas. De los resultados obtenidos se concluye que el comportamiento electroquímico de ambas superficies muestra buena resistencia a la corrosión por solución y comportamiento hidrofílico (<90 °). Sin embargo, la aleación Ti-30Ta disminuye la adhesión de la bacteria *S epidermidis*.

Palabras clave: Titanio; Aleación Ti-30Ta; Comportamiento de corrosión.

1. Introducion

Dental implants are metal posts or frames acts as a replacement for the root of missing teeth. Artificial tooth root has a function to support a replacement tooth or bridge. Nowadays, it is the closest option to mimic a natural tooth without affecting the nearby teeth (Jiang et al., 2020). In 1942, Per-Ingvar Brånemark developed the dental implants as we currently know them and named the process of connecting the metal and jawbone as osseointegration (Brånemark, Öhrnell, Nilsson, & Thomsen, 1997; Brinemark, Engstrand, Ohmell, & Grondahl, 1999) .

The global market of dental implants is expecting to make USD 13.01 billion circulate by 2023 from USD 9.50 billion in 2018. This field of biomedical application grew due to the increased number of the geriatric population and corresponding age-related dental diseases and willingness to adopt dental cosmetic surgery. Also, the accessibility because of the number of dentists working in this area(“Dental Implants Market Size & Share - Global Forecast to 2023 | Growing at a CAGR of 6.5% | MarketsandMarkets,” n.d.).

Titanium cp and alloys are the most used biomaterial to manufacture dental implants due to the combination of their excellent chemical and mechanical properties. Nevertheless, there are some disadvantages, such as the elastic modulus(Geetha, Singh, Asokamani, & Gogia, 2009; Kashi & Saha, 2010; Pandey, Awasthi, & Saxena, 2020; Verma, 2020).

Mismatching elastic modulus between titanium implants and the human jawbone is a general problem for implantology. According to Hooke's law, when loading of stress, the implant and bone will share a similar amount of deformation if they share a similar amount of modulus. Dental titanium implants have high mechanical strength and rigidity. However, the elastic modulus (E-105 GPa) is higher than the human bones (E=17-25 GPa). It led to stress shielding and long-term stress stimulation at the bone/implant interface. Wolff's law says that

a healthy bone will remodel itself to the stress load. Consequently, the result will be bone resorption and atrophy in the bone tissues around the implant. This situation could lead to implant loosening and failure (Kulkarni et al., 2015; Niinomi, 1998; Zhou & Niinomi, 2008).

On this context, research has added elements to Ti alloy to alter the long-term useful life. Tantalum shows excellent biocompatibility (Capellato, Smith, Popat, & Claro, 2012; Capellato, Escada, Popat, & Claro, 2014; Capellato et al., 2013; Capellato et al., 2020) and the elastic modulus is closer to the bone (E = 69 GPa) than Ti cp (E = 105 GPa) and others alloys (Zhou, Niinomi, & Akahori, 2004).

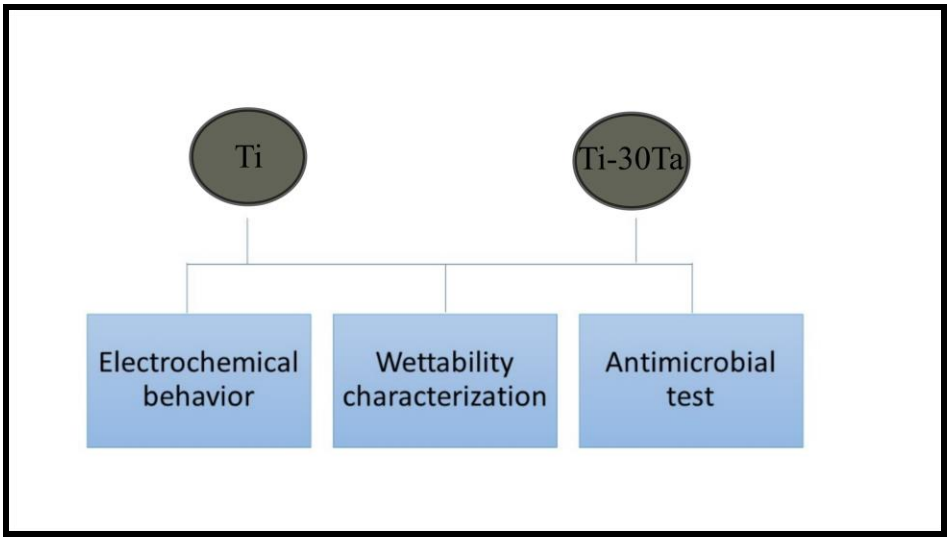
The high corrosion resistance is one of the requirements in biomedical material applications. The corrosion resistance of a material refers to its ability to withstand corrosion in a particular medium. The definition of corrosion resistance refers to how metal can withstand damage caused by chemical reactions. Other studies investigated the corrosion resistance of Ti-Ta alloys, however, there are still questions about the comparison between Ti-30Ta and Ti cp (Abdeen, El Hachach, Koc, & Atieh, 2019; Kaur & Singh, 2019; Manam et al., 2017; Mareci, Chelariu, Gordin, Ungureanu, & Gloriant, 2009; Mendis et al., 2020).

In addition, the antimicrobial effect of the surface can be the difference between implant success and failure.

Thus, in this study investigates the corrosion resistance of Ti cp and the Ti-30Ta alloy at electrolyte solution NaCl 0,15M + NaF 0,03M and tested biofilm formation.

2. Metodology

Figure 1. Shows graphically the methodology applied to this work.



Source: The authors.

2.1 Samples preparation

The bars of commercially pure titanium grade 1 were cut into discs of 10 mm in diameter and 3 mm in thickness The Ti-30Ta alloy was obtained from titanium (Ti) and tantalum (Ta) pure metals in a high purity argon atmosphere. The ingot was homogenized in a vacuum at 1000ºC for 24 hours. Then, the alloy was cold -worked by a rotary swaging process and solubilized at 950 ºC for 2 hours followed by water cooling. The bars were cut into discs of 10 mm in diameter and 3 mm in thickness (Capellato, Smith, Popat, & Alves Claro, 2015). The samples were separated into group 1- Ti cp and group 2- Ti-30Ta.

2.2 Electrochemical behavior

Corrosion resistance tests evaluated the electrochemical behavior of Group 1- Ti cp and group 2- Ti-30Ta. The characterization was performed by a three electrodes cell. The counter-electrodes of graphite, reference electrode of saturated calomel electrode, and the working electrode, with 1 cm² exposed of groups 1 and 2. The electrolyte solution contained 0,15M de NaCl + 0,03M de NaF, pH= 6. The scanned region from -0.8 V until values close 3.5 V with a speed of 0.166 mV/s. The potentiostat was employed to obtain the potentiodynamic polarization curves.

2.3 Wettability characterization

The wettability of the substrate surfaces was investigated using a sessile drop method (2 ml) with a contact angle goniometer (Kruss DSA 10), equipped with video capture. The resulting images at the water-substrate interface were fit using the circle fitting profile.

2.4 Antimicrobial test

The biofilm formation of *S epidermidis* was investigated by the reference strains [American Type Culture Collection (ATCC)], *S epidermidis* (ATCC 6538). Agar brain heart infusion (BHI) seed the strains and incubated at 37ºC for 24 hours. Then, colonies of microorganisms were suspended in sterile physiological solution [0.9 % sodium chloride (NaCl)] and adjusted to a 0.5-turbidity on the MacFarland scale (1.5 × 108 UFC/mL).

All the samples were placed in 24 well plates with 2 mL of BHI broth supplemented with 5 % sucrose and inoculated with 0,1 mL of the bacterial suspension, incubated at 37 ºC for 48 hours, and the media was changed after 24 hours. It was washed aseptically with 2 mL of sterile physiological solution and sonicated for 30 seconds. Aliquots of 0.1 mL were seeded on BHI agar plates and incubated for 24 hours at 37ºC. The number of colonies was counted, calculated in CFU/mL and transformed in log10.

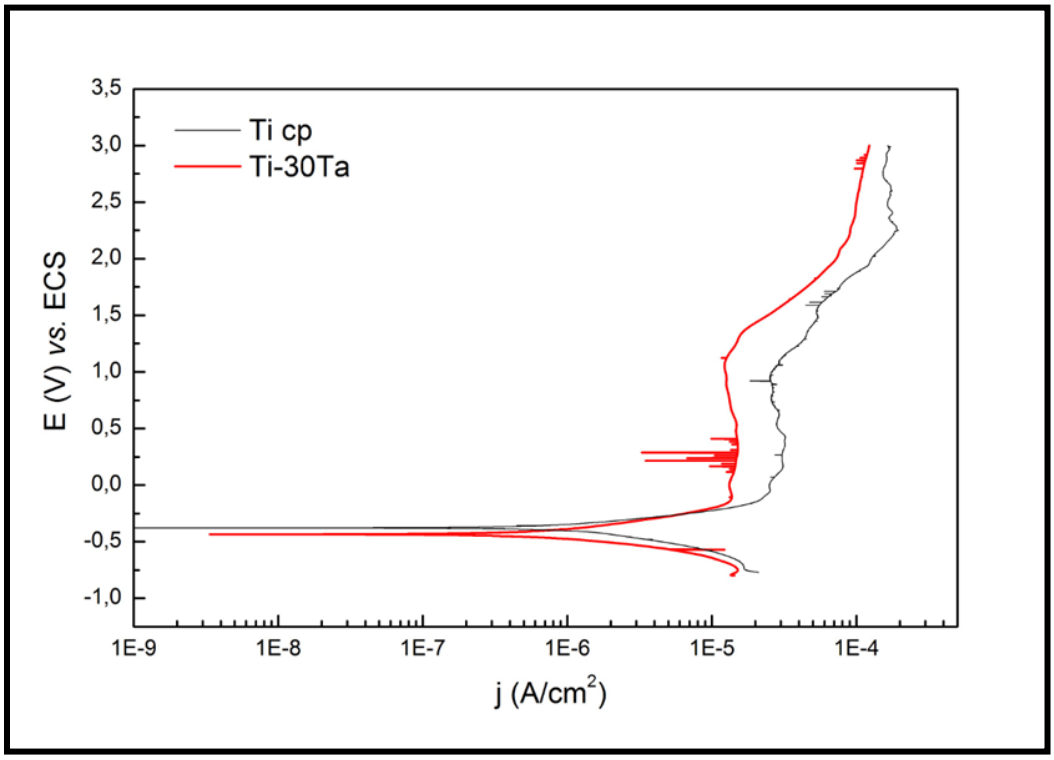
Each experiment was reconfirmed on at least three different substrates (nmin = 3). All the quantitative results were analyzed using an analysis of variance (ANOVA). Statistical significance was considered at $p < 0.05$. During the analysis, variances among each group were not assumed equal and a two-sample t-test approach was conducted using the Microsoft Office Excel data analysis software.

3. Results and Discussion

The mechanical properties are an important criterion to choose biomaterial for use in biomedical implants. Therefore, in this study we investigated the experimental alloy Ti-30Ta with elastic modulus close to the bone (E=69GPa), comparison to Ti Cp (E=105GPa).

Polarization methods such as potentiodynamic polarization are is used for corrosion testing. This technique provides significantly useful information regarding the corrosion mechanisms, corrosion rate and susceptibility of specific materials to corrosion in designated environments. Polarization methods involve changing the potential of the working electrode and monitoring the current, which is produced as a function of time or potential. The potentiodynamic polarization resistance curves are shown in Figure 2. The polarization measurement evaluates the stability and intensity during passive oxide film formation. Group 1- Ti cp and group 2- Ti-30 Ta exhibited typical spontaneous passivation characteristics.

Figure 2. Polarization curves in 0.15M of NaCl + 0,03M of NaF solution at 25°C and -0.8 V - 3.5 V scan rate and speed 0.166 mV/s.



Source: The authors.

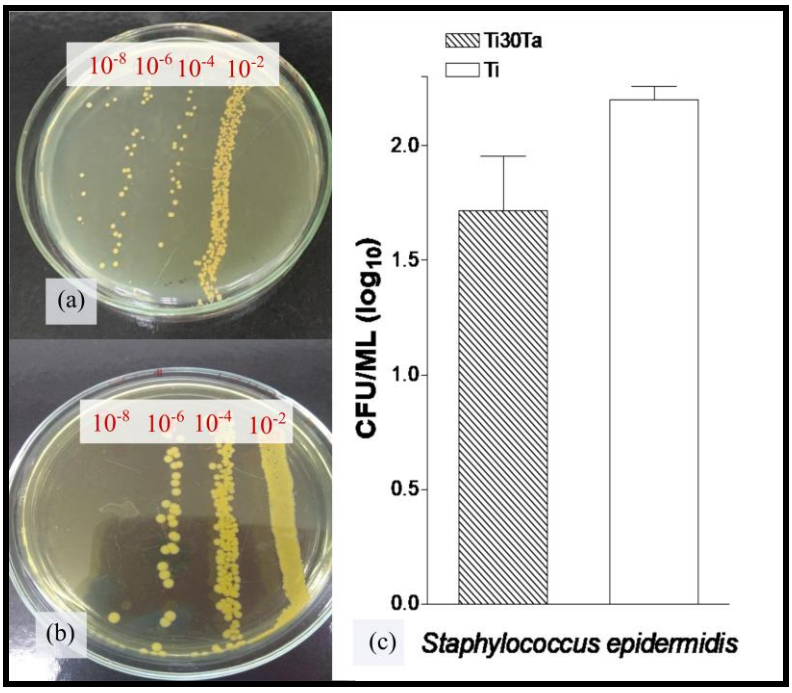
Zhou et al. (2005) evaluated the corrosion resistance of Ti cp, Ti-Ta and Ti6Al4V and they found that the presence of Ta_2O_5 passive films the TiO_2 passive. So, they concluded that Ti-Ta shows a better corrosion resistance than Ti cp and Ti6Al4V (Zhou, Niinomi, Akahori, Fukui, & Toda, 2005). Souza and Robin (2003) studied the corrosion resistance of Ti-Ta at a sulfuric acid solution. The authors found Ti-Ta alloys, with a higher percentage of tantalum, show a corrosion resistance behavior similar to pure tantalum and better than titanium (Souza & Robin, 2003). The corrosion resistance of the Ti-30Ta alloy was through two types of processing: A study by Gill et al. (2011) investigated the powder metallurgy and melting in an arc furnace with results showing that the powder metallurgy alloy has greater resistance to localized corrosion.

The wettability of the surface is an important parameter to evaluated the ideal biomaterial. A metallic biomaterial to biomedical application demands a hydrophilic surface due to cell behavior. The result shows all surfaces as hydrophilic (< 90°). The results show a hydrophilic surface for both groups: group1-Ti cp 57.63+-2,39 and group 2- Ti-30Ta alloy 65.89+-1,81.

The colonies seeded in Petri dishes in serially diluted were the source of colony

counting, in order to be able to determine CFU/ml. Figure 3 illustrates the Petri Dish by a photography. Figure 3 also shows the results of antimicrobial activity study with the adhesion of S epidermidis bacteria lower on Ti-30Ta alloy than Ti. The data are presented as mean ± SEM and the statistical significance was analyzed by analyses of variance (ANOVA), followed by Newman-Keuls post hoc analysis. Results with $P<0.05$ were considered statistically significant.

Figure 3. The figure presents photographs of colonies counting in background, (a) Ti-30Ta Alloy and (b) Ti commercially pure, (c) presents a graphic results of the number of colony forming units per milliliter (CFU/mL) of *S epidermidis*. The UFC/mL of the Ti cp surface is higher than Ti-30Ta.



Source: The authors.

General microbiological pathogen presents associations with implant fail. An alloy able to reduce this problem could generate economy, once it avoids implant replacement, and safety to the patient. (Malhotra et al., 2019). The biofilm test present data that enforce the alloy a higher effectiveness against *S. epidermidis* than titanium. *Staphylococci* are the most common related bacteria group responsible for implant-associated infections. Alloys that inhibit those pathogens can improve the quality of implant procedures (Zimmerli & Sendi, 2011).

4. Conclusion

The dental implant field has been searching for Ti alloys with elastic modulus close to the bone. Therefore, in this study we investigated the corrosion behavior of T-30 Ta alloy and Ti cp. From the results obtained, we concluded that the electrochemical behavior of both surfaces shows good resistance to corrosion solution and hydrophilic (< 90°) behavior. Soever, the Ti-30Ta alloy decreases the adhesion of *S epidermidis* bacteria. All tests were performed *in vitro*, they represent an initial test to base future *in vivo* assays, however they may differ due biological activity. In addition, future works may content other *Staphylococci* yields to compare the alloy's effectiveness.

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