DEVELOPMENT OF TITANIUM DENTAL IMPLANTS USING TECHNIQUES OF POWDER METALLURGY

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ABSTRACT

Titanium is an attractive material for dental and biomedical applications, because of high corrosion resistance, excellent biocompatibility and high mechanical strength combined with low density. However, the high reactivity of titanium in the liquid phase make it difficult to produce it by fusion, so a alternative is powder metallurgy (P/M) method. Powder Metallurgy has been used to manufacture porous implants. The presence of a porous surface is desirable because it improves the osteointegration increases the adhesion between the bone tissue and the implant, being favorable for transporting body fluid. This paper proposes to characterize the commercial pure titanium powder obtained by process of hydride-dehydride, obtain samples with adequate porosity by uniaxial pressing and vacuum sintering and evaluate the corrosion behavior of sintered titanium in Hank´s solution. The results showed that the titanium powder of angular shape after uniaxial pressing of 400 MPa and sintered in vacuum at 1150 ° C, allowed obtaining samples with adequate surface porosity of around 17%. In potentiodynamic polarization curves revealed no typical behavior of passive metals but show low current density, that increasing corrosion resistance.

Keywords: titanium implants, powder metallurgy, porosity and electrochemical behavior.

1. INTRODUCTION

For RATNER biomaterial can be defined as a material that is produced to be in contact with biological systems in order to treat, augment and replace any tissue, organ or function of the body[1]. A material for use as an implant or protheses must meet the concepts of biocompatibility and biofunctionality, high corrosion resistance and low density with high mechanical strength [2, 3]. Titanium and its alloys meet these requirements.

Titanium alloys are used in orthopedic prostheses while in manufacturing screws and dental implants are used pure titanium, since the mechanical stresses are not as high in these applications [3]. Some studies indicate that the high modulus of elasticity of 120 GPa for titanium, compared to the bone tissue around the implant of 10-30 GPa

may result in stress shielding, so the difference in modulus of elasticity is a determining factor for implant failure [4].

The presence of pores in the implants may help in decreasing of the modulus elasticity compared to bone tissue and allows the anchorage of the biological tissue around the implant and improves the bonding properties of the interface [4].

One technique to get porosity at the surface of a component is a powder metallurgy. Using this technique it is possible to manufacture implants with interconnected pores, allowing bone growth in these areas, thus favoring to osteointegration. Powder metallurgy is a group of techniques used for producing, characterizing of metalic or ceramic powders and consolidation by compacting and sintering.

The physical characteristics of the powder should be known, because they influence in subsequent steps of compacting and sintering [5]. Uniaxial pressing involves the compaction of powder into a rigid die by applying pressure in axial direction through a rigid punch. Sintering is a thermal process carried out at temperature below the melting point of the metal majority. The driving force for sintering to occur is obtained by reducing the free energy of the system due to the temperature increase, happen the reduction of surface area, the growth of necks between the particles and densification [5]. In the sintering of powders of titanium should use high vacuum to minimize oxidation of the surface and facilitate the sintering [5, 6].

Dental implants are in contact with body fluids of complex composition and the presence of dissolved gases in these fluids may form differential aeration cells that produce electrochemical processes on the surface of the implant [7]. Therefore, evaluation of electrochemical properties is important and can be performed by means of potentiodynamic polarization in a simulated body fluid such as Hank's solution. According to ANTUNES, & COSTA OLIVEIRA [8] in potentiodynamic polarization curves in porous samples is not observed a large and stable passive region, because of the greater surface area exposed in the electrolyte. The values of current density were low in corrosion potential and observed values of potential typical of the oxygen evolution reaction [8].

The objective of this work is to characterize the pure titanium powder, produce samples with adequate porosity by uniaxial pressing techniques and vacuum sintering and evaluate the electrochemical behavior of sintered in Hank's solution.

2. MATERIALS AND METHODS

The chemical composition of titanium powder was determined by X-ray fluorescence dispersion wavelength and is shown in Tab. 1.

Table 1 – Chemical composition of titanium powder determined by X-ray fluorescence.

Composition	Ti	Fe	Cr	Ni	Ca	S	Cu
(%)	99.80	0.08	0.04	0.02	0.02	0.02	0.02

The physical characteristics of the powder such as particle morphology determined by scanning electron microscopy, particle size distribution by CILAS equipment, bulk density, flowability and particle friction between the funnel Hall.

The samples were compressed into cylindrical die and had a diameter of 8.5 mm. Following determined the density of the green compact on the basis of mass / volume of samples. The samples were sintered in vacuum at a pressure of $1x10^{-6}$ torr with heating rate 10°C /min until the temperature reaches 1150 °C. The samples remained at this level for one hour during cooling were vacuum until it reaches room temperature.

The characterization of the sintered samples was performed by determining the geometric density and continued with the aid of optical microscopy and X-ray diffraction. Samples were cut in cross section prepared by metallographic techniques. The cleaning of the samples was performed at the ultrasound for 5 minutes in acetone in order to remove water adsorbed in the pores. The etching performed with a solution of 5 ml HNO_3, and 10 ml HF, 85 ml H_20. In X-ray diffraction was used wavelength of 1.154 $x10^{-10}$ generated by Cu K_α tube at intervals of 30 ° to 100 ° to evaluate phases present.

The evaluation of properties was performed by electrochemical technique of polarization in Hank's solution (37 ± 2) °C with chemical composition showed Tab.2, it was soon used a thermostatic bath and a thermometer for temperature control.

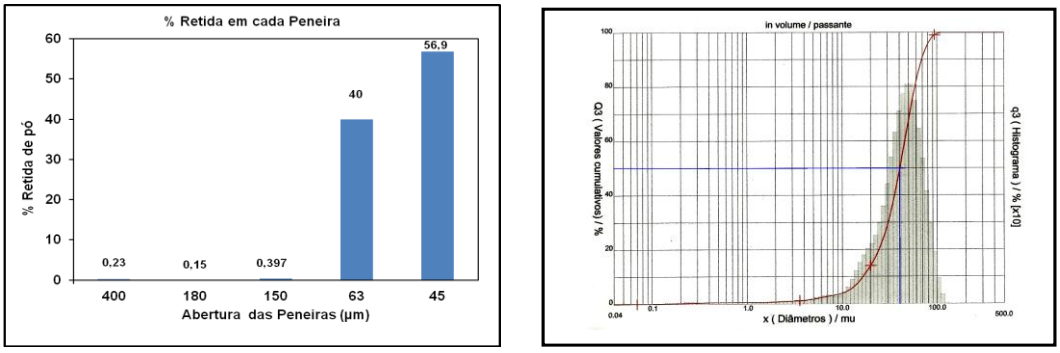
Table 2- Chemical Composition of Hank´s solution
Source: Investigation of the Corrosion Behavior of Porous Titanium in Hank`s Solution [8].

Components	NaCl	KCl	$MgSO_4.7H_2O$	$CaCl_2.2H_2O$	$Na_2HPO_4.2H_2O$	KH_2PO_4	$C_6H_{12}O_6H_2O$
Concentração (Mol/L)	0.1369	0.0054	0.0008	0.0013	0.0003	0.0004	0.0050

The polarization assay was initiated after 5 minutes. The measurements started of -800 mV at scan rate of 1 mV/s until reaching the final potential of 3000 mV.

3. RESULTS AND DISCUSSION

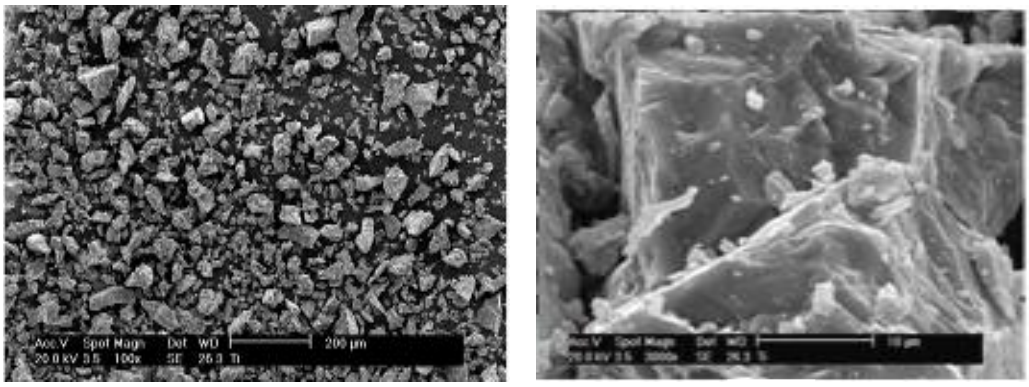
The Fig.1a is indicated particle size distribution by sieving. The sieve with opening of 325 mesh had a greater amount of mass retained and the average particle size was estimated to be 50 μm. When performing the assay with equipment CILAS there was an average particle size of 45 μm as Fig.1b.



(a) (b)

Figure 1 - a) % weight retained on each sieve; (b) Particle size distribution obtained by CILAS.

In Fig. 2a observe morphological aspects, while in Fig. 2b there is the particle surface porosity and roughness due hydrogenation-dehydrogenation process.



(a) (b)

Figure 2- MEV image of titanium powder. (a) particle shape; (b) porous surface e roughness particle.

In Tab. 3 indicates values of bulk density, angle of repose and green density. The value angle of repose is high due of irregular morphology of particles, so particles have high friction between one that hinders the powder flowability. According PRESTA reports that powder with an angle greater than 40 degrees have difficulty filling the cylindrical die [10].

From the result of the compressibility curve it was selected of axial pressure of 400 MPa. The density of sintered obtained was 82.3 % of the theoretical density, while the porosity was 17, 7 %. The values obtained in the characterization of samples of titanium are shown in Tab.3.

Table 3- Values of: Bulk Density, angle repose, green and sintered density.

Bulk Density [g/cm^3]	Angle of Repose [º]	Green Density [g/cm^3]	Sintered Density [g/cm^3]	Porosity [%]
1.31±0.07	42.6±0.89	2.73±0.16	3.72±0,15	17.7

According to Fig. 3 there are different sizes of pores, which ranged from 1 to 30 µm. These measures include 5 random region of the sample.

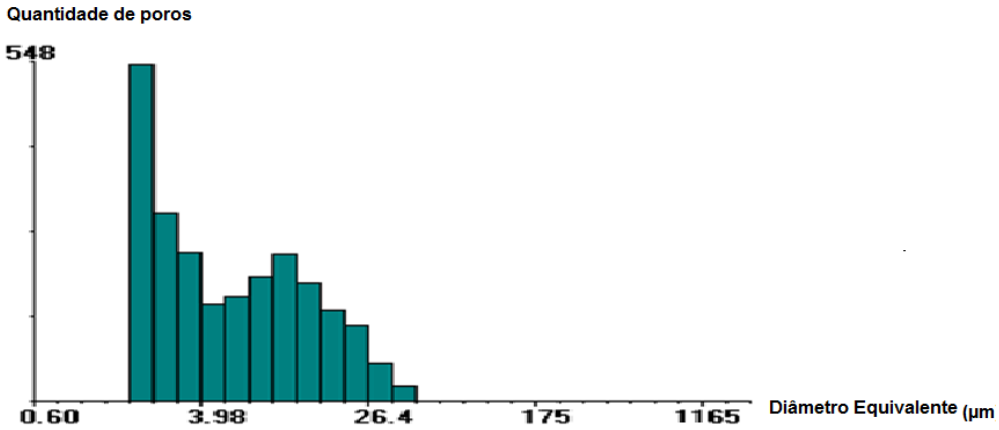
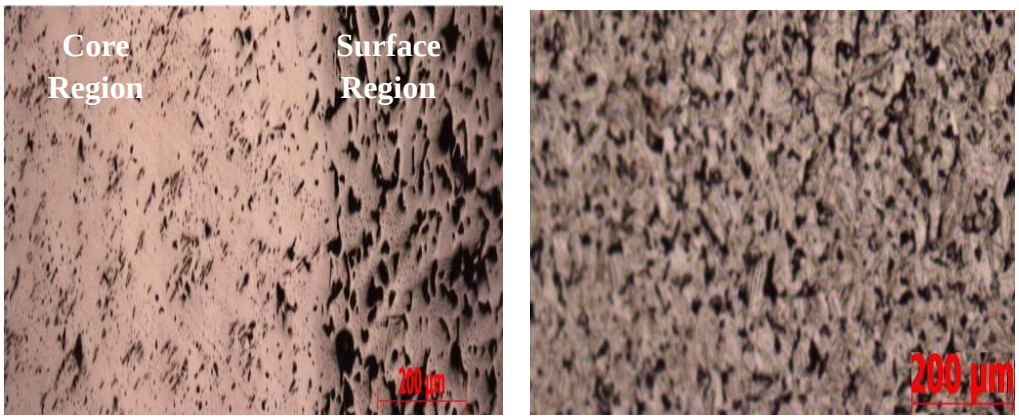


Figure 3- Graph quantity of porous versus equivalent diameter (µm).

In Fig. 4a shows there is an increased pore diameter of the core to the surface, so greater surface porosity. This aspect is positive because higher porosity improves osteointegration properties and conductivity of organic fluids is favorable [7]. In fig. 4b is observed microstructure composed mainly of α phase, there are also small regions that indicate β phase, however β phase was not detected by X-ray diffraction.



(a) (b)

Figure 4- Images obtained by optical microscopy. (a) Porosity – higher fraction of porosity as well as larger at the surface region; (b) Microstructure of the sintered sample with α and β phases and pores.

A potentiodynamic polarization curve for porous titanium is shown in Fig. 5. The curve doesn't present well defined passive region, because of the larger surface exposed to the electrolyte in the case of porous specimens. Observes that current densities obtained 1200 mV are very low and typical of passive metals and in 1200-1300 mV range is increased current density due of the oxygen evolution reaction.

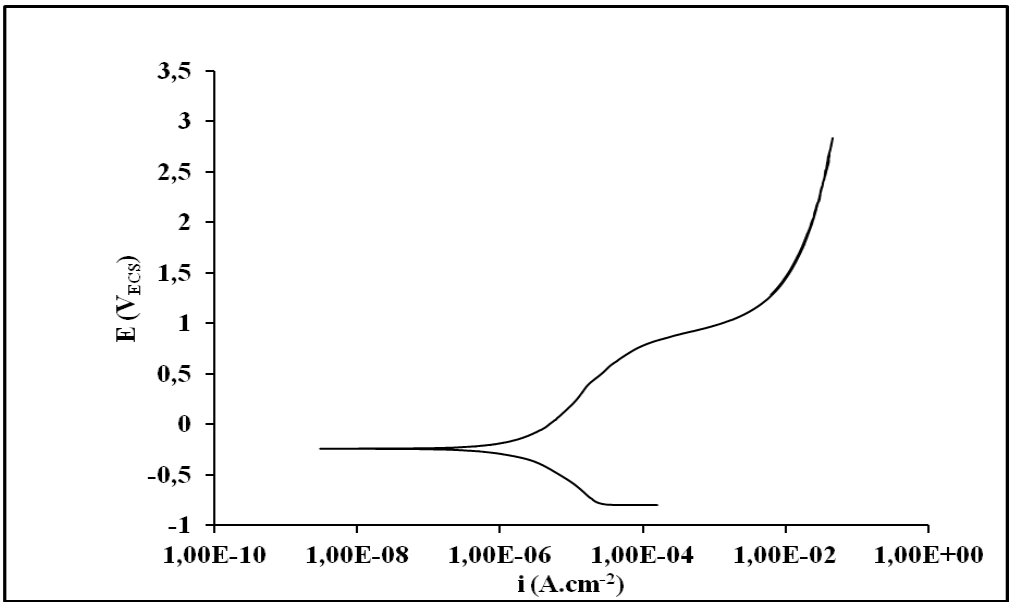


Figure 5- Potenciodynamic polarization curve for porous titatnium tested for 5 minutes at (37±2) ºC.

4. CONCLUSIONS

The titanium powder had particles with an average size of 50 μm by sieving and 45 μm by CILAS and angular shape. The angular shape is a disadvantage because it reduces the flowability difficult to fill the matrix. However favorable uniaxial pressing, which was obtained in 60.5% of the theoretical density of titanium.

In vacuum sintering at 1150 °C was obtained average density of (3.72 ± 0.15) g/cm^3, so the sample shrank 33.05 %. The porosity obtained was 17. 70 %.

The sintered sample showed typical behavior metal passive with a low value of current density in Hank's solution.

The techniques of powder metallurgy can be efficient and viable alternatives for the production of dental implants, due to the porosity to improve anchoring between the implant and bone, which should promote better functional and structural bond.

5. REFRENCES

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