

Porous Structure Characterization in Titanium Coating for Surgical Implants

M.V. Oliveira^{a*}, L.C. Pereira^b and C.A.A. Cairo^c

^a INT/DMCM - Instituto Nacional de Tecnologia
Av. Venezuela, nº 82/607, 20081-310 Rio de Janeiro - RJ, Brazil
^b UFRJ/PEMM/COPPE, Universidade Federal do Rio de Janeiro
CT/Bloco F/sala 210, Ilha do Fundão - Cidade Universitária
^c CTA/IAE/AMR, Centro Técnico Aeroespacial

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Powder metallurgy techniques have been used to produce controlled porous structures, such as the porous coatings applied for dental and orthopedic surgical implants, which allow bony tissue ingrowth within the implant surface improving fixation. This work presents the processing and characterization of titanium porous coatings of different porosity levels, processed through powder metallurgy techniques. Pure titanium sponge powders were used for coating and Ti-6Al-7Nb powder metallurgy rods were used as substrates. Characterization was made through quantitative metallographic image analysis using optical light microscope for coating porosity data and SEM analysis for evaluation of the coating/substrate interface integrity. The results allowed optimization of the processing parameters in order to obtain porous coatings that meet the requirements for use as implants.

Keywords: titanium, coating, implant, powder metallurgy

1. Introduction

This work is a preliminary study of titanium porous coating processing through powder metallurgy techniques for surgical implants purposes. The main objective was to determine processing parameters to produce the porous coatings with adequate microstructure for implant fixation. Different materials were used as substrates with different surface conditions to observe the surface effect on coating adhesion. The results allowed optimization of the process in order to achieve a system with optimal degree of porosity from the substrate to coating in further research.

2. State-of-Art

Porous coatings for implant stabilization have been considered an alternative to bone cement fixation. The latter has been the predominant approach for fixation of orthopedic implants and the major reported cause of implant loosening¹. These coatings present a three-dimensional interconnected array of pores throughout the thickness, which

allows bone tissue ingrowth. The high load bearing requirements over long periods (more than 20 years) in applications such as hip joint and knee replacements suggest the use of a metal powder surface layer to provide the required high strength and toughness^{2,3}. Although bioceramics are known to be more biocompatible, a study comparing titanium porous coating and hidroxiapatite coating, both applied in titanium substrates, demonstrate that the two systems were able to attain the same percentages of bone ingrowth⁴. Porous metallic coatings have been shown a higher bone/metal shear strength than other types of fixation and, as a consequence of a stronger interfacial bond, they present a decreased propensity for implant loosening¹.

Fracture mechanics of highly loaded implants also must be taken into account in the production of porous coatings implants. Cracks and wear debris in the bone interface generated from the irregular porous surface can also lead to loosening of the implant. In addition to, there is a decrease in corrosion resistance due to the larger surface area exposed to the physiological medium, compared with non-

*e-mail: marizeva@int.gov.br, lula@metalmat.ufrj.br, cairo@iae.cta.br

porous biomaterials³. In spite of it, excellent passivation characteristics have been observed in pure titanium porous compacts and there is no indication that these higher corrosion rates give rise to significant alterations in systemic responses *in vivo*⁵. It appears that when the high-surface-area implants are well fixed, there is a lower level of metal ion released³.

Porous metallic coatings can be produced onto solid substrates by powder metallurgy/PM techniques using powders or fibers, thermal-spray techniques and VMC/void metal composite, a cast technique³. Sintered coatings can be produced by placing the powder (or fiber) over the implant surface, mixed with a proper binder or not, followed by sintering. Conventional PM techniques can be used through uniaxial or cold isostatic pressing - CIP⁶.

The performance of a sintered porous coated system is accepted to be greatly dependent on the coating adhesion and on the coating morphology^{7,8}. A specific type of porosity is required in order to promote new bone formation infiltrated in the coating. A minimum pore size of approximately fifty micrometers is necessary for the growth of mineralized bone into a porous structure⁹. This size is needed for blood to penetrate the porous structure to allow the growing of the bone at depth of more than $\approx 100 \mu\text{m}$. The optimum pore size required for implant fixation is considered to be in the range of $50\text{--}400 \mu\text{m}$ ⁹ and pore volume fraction usually in the range of 30 and 40%¹⁰. This condition represents a compromise between maintaining the coating strength and providing adequate pore size for tissue ingrowth.

The fixation efficacy of coatings is strongly dependent on the geometric characteristics of the coating layer as well as on the coating mechanical integrity and its bonding to the underlying substrate. Volume fraction porosity is related to the particle interconnectivity and to the particle size. The pore size distribution throughout the coating thickness and particle interconnectivity, i.e. the inter-particle neck size (for sintered powders, wires or fibers), also affect the coating strength and the adherence of coating to the substrate interface¹¹.

Titanium and titanium alloys have been successfully used in dental^{4,11} and orthopedic^{6,7} applications, because they combine excellent corrosion resistance and high resistance/weight ratio with biocompatibility. Powder metallurgy (PM) is a favorable fabrication technique for titanium porous coating systems, as they have the advantage to produce superior mechanical properties and stronger adhesion to the substrate. The coating porosity level can be graded from a highly porous surface layer to denser ones in contact with the substrate, providing appropriate strength to sustain loading⁷.

3. Experimental Procedure

The materials used as substrates and coatings are shown

in Table 1. Pure titanium (ASTM Grade 1) wrought (extruded) cylindrical rod with 6.9 mm diameter and a Ti-6Al-7Nb/PM cylindrical rod with 5.3 mm diameter, according to previous research¹², were used as substrates. These substrates were used in different surface conditions, as reported by the roughness level (Table 1). For the coatings, the powder used was a pure titanium sponge powder in the $44\text{--}840 \mu\text{m}$ size range. This was separated into two particle sizes ranges, $44\text{--}297 \mu\text{m}$, and $420\text{--}590 \mu\text{m}$, in order to obtain different porosity levels and also to evaluate the influence of particle size on the coating porosity morphology.

Coating Processing

For porous coating processing, titanium powders were compacted over the substrate surface by cold isostatic pressing technique (CIP), followed by sintering treatment. Cylindrical rubber CIP moulds were used, sealed with rubber plugs bottom and top. Mould filling with powder was made with the substrate positioned in the center. The opening gap between the substrate and the mould was completely filled with the tapered titanium powders. Compaction pressures were in the range of 200–400 MPa. After compaction, the rubber plugs and the mould were carefully removed to avoid damage of the porous coating. The compacted coatings were then sintered in a high vacuum furnace with graphite heating element and a titanium crucible. The sintering temperature was $1400 \text{ }^\circ\text{C}$, according to previous research¹³, with one hour step and vacuum of 10^{-6} Torr.

Substrate and Coating Characterization

The surface condition of type 1, 2 and 3 substrates (Table 1) was as received and cleaned. Type 2 substrate was cleaned and ground with a silicon carbide paper grit (#120).

Table 1. Materials used to produce the substrates and the corresponding porous coating.

Substrate Material	Coating	
	Roughness (μm)	Powder size range ^d (μm)
1. Pure titanium wrought rod ^a	1.3	44-297
2. Pure titanium wrought rod ^b	2.3	44-297
3. Ti-6Al-7Nb P/M rod ^c	3.2	44-297
4. Ti-6Al-7Nb P/M rod ^c	3.2	420-590

a: Produced by CAMACAN/Brazil. Surface in the as received condition;

b: Produced by CAMACAN/Brazil. Surface ground with a silicon carbide paper grit (#120);

c: Produced by CTA/Brazil. Surface in the as received condition;

d: Pure titanium sponge powder produced by CTA/Brazil.

The substrate surface roughness was measured with help of a profilometer. The coating porous surface of sintered specimens was analyzed in SEM topographic views. The specimens were transversally cut and prepared by standard metallographic methods. Quantitative metallographic analysis by automatic image technique was used to determine the percentual porosity and pore size range^{14,15}.

4. Results And Discussion

Figure 1 shows the appearance of the as sintered porous systems (substrate plus coating). From the SEM images of the specimens produced with greater porosity and, compacted with the lowest pressure, 200 MPa, the coatings exhibit open porosity (Figs. 2a - 2d). Interparticle bonds in Fig. 2c indicate that sintering was in an advanced stage. The pore size



Figure 1. As sintered type 3 porous system, compacted at 200 MPa.

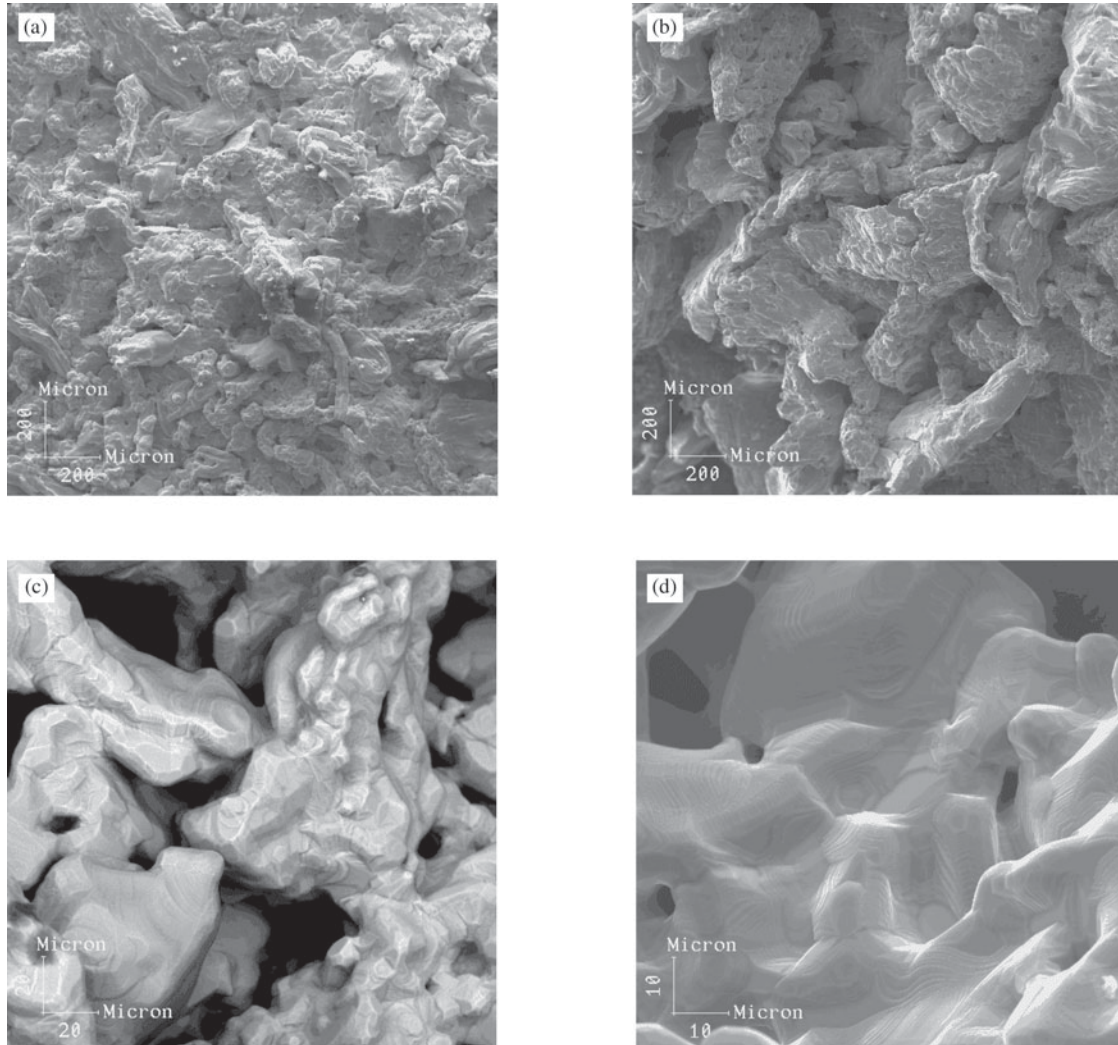


Figure 2. SEM surface topographical images of the specimens compacted at 200 MPa: type 2 in (a) (c); type 4 in (b), type 1 in (d).

distribution along the coating surface of all specimens was very broad, small pores along large pores appear, as illustrated in Figs. 2c and 2d. The type 2 porous system (Figs. 2a and 2c) contains open pores with dimensions of approximately 20-50 μm while the coating made with larger particle size powder, type 4, has larger surface pores, of 100-300 μm (Fig. 2b). These results were expected, because it is known that larger particles sizes and inferior compaction pressures will produce larger pore sizes. The substitution of the compacting mould material for another one that allows lower pressures without damaging the surface of the compacted coating would probably produce more porous structures.

Table 2 shows the quantitative metallographic analysis for pore size range and percentual porosity. Higher poros-

ity fraction was achieved for the type 4 system, because it was produced with larger particle size powders and narrower size distribution. Maximum porosity fraction (% porosity) was obtained for the systems compacted at 200 MPa. Type 3 system shows inferior values of porosity for the same compaction pressures, compared with the other systems. This is probably because of the differences caused during manual mould filling technique. Homogeneity may be improved by changing filling mode from the manual to a tap instrument powder filling. Quantitative analyses of the porous size distribution along the coatings thickness and the porous percentage along the substrate/coating interface are in course, to give indication respectively of the coating adhesion and the porosity quantity that meet the requirements

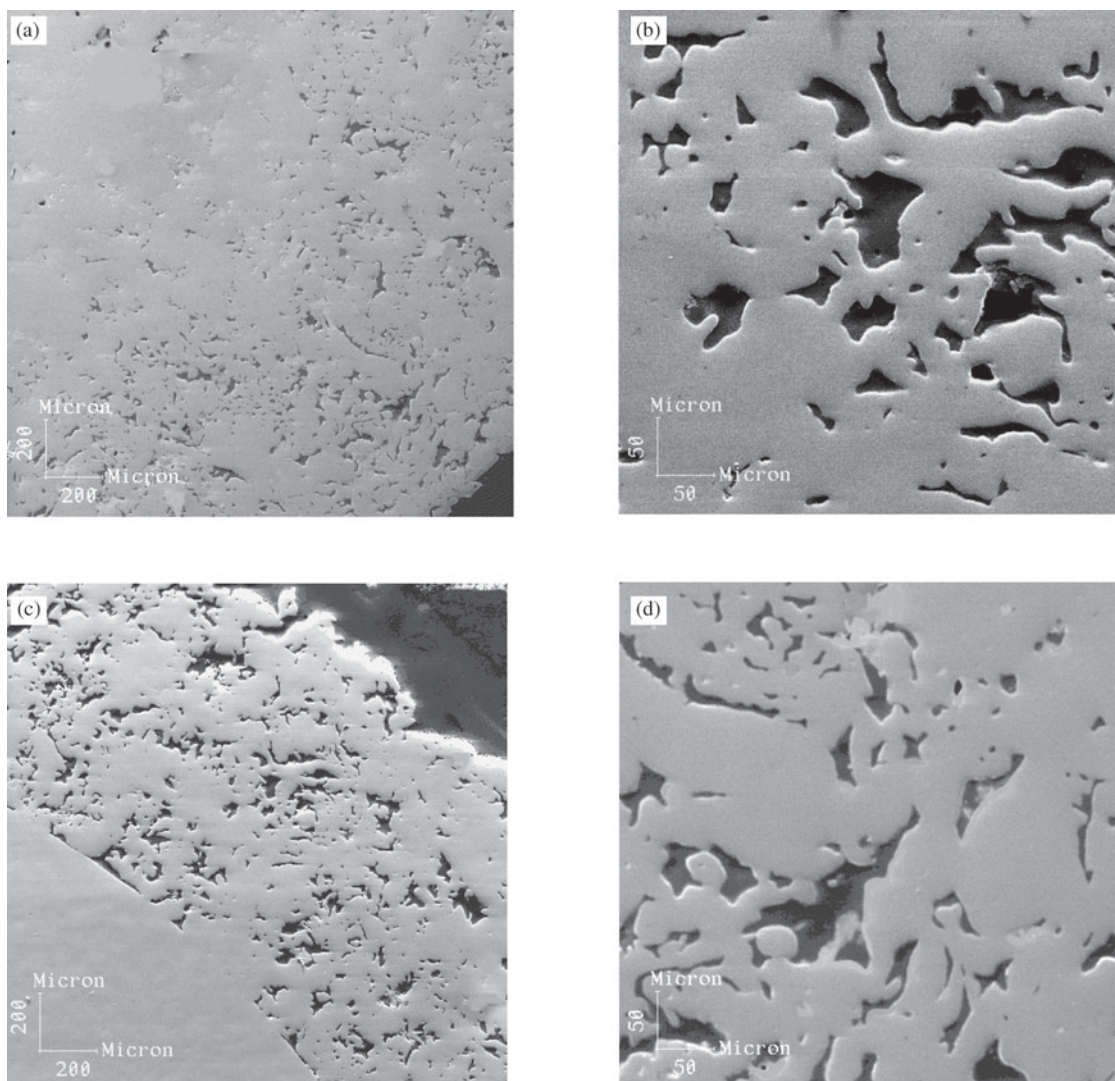


Figure 3. SEM photomicrographs showing the coatings of polished specimens compacted at 200 MPa: type 1 in (a) (b) and type 3 compacted at 200 MPa in (c) (d).

Table 2. Isostatic pressures used and coating properties for the different substrate types.

Substrate Type	Isostatic Pressure (MPa)	Coating Aspects	
		Pore size* (μm)	Porosity (%)
1	200	3 - 91	23
	450	3 - 38	7
2	200	3 - 80	21
	300	3 - 65	11
	400	3 - 40	7
3	200	3 - 56	11
	300	3 - 70	8
	400	3 - 43	8
4	200	3 - 119	24

* The size minimum "3 μm " was chosen to define the range (the majority of the pores were greater than this size in pixels).

for use as implants.

The measured thickness of the coatings produced was of approximately 1000 μm . SEM micrographs of a coating section for the systems 1 and 3, compacted at 200 MPa, are shown in Figs. 3a, 3b, 3c and 3d. It can be observed that the porosity is not interconnected, although in some regions (Fig. 3b) pore networks appears together with regions of closed porosity. The specimens compacted at pressures higher than 200 MPa presented only isolated pores. With respect to coating/substrate adhesion, type 1 and 2 specimens (Figs. 3a and 3b) presents smaller coating areas (or contact points) bonded with the substrate than the types 3 and 4 (Figs. 3c and 3d). This is possibly related to the substrate surface roughness and the fabrication process, as higher surface roughness implies better adhesion. The wrought substrates, types 1 and 2 exhibit lower roughness (Table 1), compared with the PM substrates, types 3 and 4, which are more porous on the surface.

While many researches^{4,6,9} make use of cast or wrought substrates, this work employs a wrought substrate and a substrate produced through powder metallurgy. The PM substrate presented the better coating adhesion, from a microstructure point of view, because of its surface porosity and higher roughness. With the continuity of this study, these results can be the basis to produce a functionally graded type PM system, with a degree of low porosity in the substrate to a highly porous surface in the coating.

5. Conclusion

The processing techniques used in this work proved to be suitable for producing titanium porous coatings and good adhesion with the substrate, from a microstructure analysis

point of view. Changes in mould filling approach for isostatic pressing is seen as necessary to optimize homogeneity of the porous coatings. As the pore size distribution along the coating was heterogeneous, the pore size distribution measurements are in course to give a more accurate statistical treatment. The porous structure requirements for surgical implant purposes were not met, indicating that it is necessary to reduce the sintering temperature up to a level where interparticle necks have just been formed. Also the substitution of the compacting mould material for another one that allows lower pressures would probably produce structures with interconnected porosity and higher pore size and porosity.

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