Characterization of Porous Titanium for Bioengineering Applications

Marize Varella de Oliveira, Laboratório de Tecnologia de Pós, Instituto Nacional de Tecnologia, INT, e-mail: marizeva@grante.ufsc.br
Antonio Marcos Clemente de Moraes, Departamento de Física, Universidade Estadual de Londrina, UEL, e-mail: toninho_bmx@hotmail.com
Anderson Camargo Moreira, Laboratório de Meios Porosos e Propriedades Termofísicas, Universidade Federal de Santa Catarina, UFSC, e-mail: anderson@lmpt.ufsc.br
Carlos Roberto Appolôni, Departamento de Física, Universidade Estadual de Londrina, UEL, e-mail: appoloni@uel.br
Luiz Carlos Pereira, Programa de Engenharia Metalúrgica e de Materiais, Universidade Federal do Rio de Janeiro, UFRJ/COPPE, e-mail: lula@metalmat.ufrj.br

Introduction

Porous structures, named scaffolds, are archetypes for cell interactions where take place migration, proliferation and vascularization of osseous tissue and new bone formation [Dunand, D.C., 2004]. These scaffolds provide a better mechanical stability at the implant-bone interface than denser structures. Porous titanium (Ti) has been used as coatings for fixation dental and orthopaedic implants and as synthetic grafts, as it allows the mechanical interlocking of the pores and bone (bone ingrowth) [Li, H., 2007].
Scaffolds should reproduce bone morphology and function for high integration into surrounding tissue. Pore size, volume fraction, shape and distribution are critical features for bioengineering applications. Pores may be closed or interconnected, in general, porous structures have a mixture of both types. The porosity of most implants may have a suitable combination of mechanical properties and pore morphology. The pore size required for implant fixation must be in the range of 100–500µm and the porosity quantity of 40–90% [Ryan, G., 2006]. Micropores (<20µm) and nanopores (1–10nm) favor cellular adhesion and implant osseointegration [Karageorgiou, V., 2005; Simmons, C.A., 2002].
Porosity evaluation of scaffolds is of great importance for their design and processing. In this study, porous titanium samples were manufactured by powder metallurgy, which has low processing temperature, suitable for metals with high contamination susceptibility, like Ti. The porosity quantification was assessed by non-destructive methods: geometric method and gamma-ray transmission and quantitative metallographic analysis, a destructive method, in order to compare their efficacy for porosity evaluation. Pore morphology and surface topography were evaluated via scanning electron microscopy and optical microscopy.
.

Methodology

Pure Ti powder grade 2 (Micron Metals-EUA) made by HDH (hydrogenation-dehydrogenation) process, with acicular shape, particle size range of 149-177µm and an organic additive (urea), as pore former, were used to make the samples by a powder metallurgy route. Two cylindrical samples with 9.2mm/height and 11.4mm/diameter, composed by 70%wt-Ti/30%wt-urea (210-250µm particle size) and 100%Ti, were compacted by uniaxial compaction at 300 MPa and 450 MPa, respectively. One cylindrical sample with 5.4mm/height and 8.4mm/diameter, composed by with 85%wt-Ti/15%wt-urea (149-177µm particle size) was compacted by cold isostatic compaction at 300 MPa. All samples were treated at 200ºC/2h to eliminate the organic additive and sintered at 1200ºC/2h in vacuum furnace (~10^{-6} Torr).
The total porosity (P), obtained by the geometric method (GM) is given by P = 100 – RD, being RD the relative density, which is determined dividing the geometric density (mass/volume) by the absolute Ti density (4.5g/cm^3). About 8 measurements for each sample were performed in the GM method.
Sample transverse sections were prepared for optical microscopy using the standard methodology. Porosity volume fraction measurements were performed by quantitative metallographic analysis (QMA), using the Image ProPlus version 4.0 software, in about 20 random images for each sample.
The gamma-ray transmission technique consists in the attenuation that an incident radiation beam suffers when go across this material. The experimental setup is constituted by a micrometer automated table for the sample positioning, Am-241 radioactive source (59.53 keV, 100 mCi), 2 mm diameter Pb collimators, NaI(Tl) detector and appropriate nuclear electronics [Appoloni, C.R., 2004]. The transmission measurements were accomplished taken 4 different positions in a random order along the longitudinal axis, with 9 measurements for position at 300s.

Results and Discussion

Figures 1, 2 and 3 show the SEM topographic views and optical micrographs of the Ti samples and tables 1 and 2 present the porosity values from the Ti samples, obtained by the three techniques analyzed in this work.

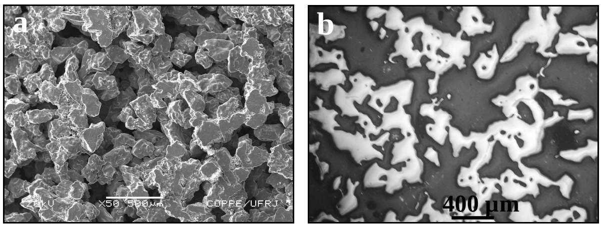

Figure 1: SEM topographic view (a) and optical micrograph (b) of the 70% Ti/30% urea sample.

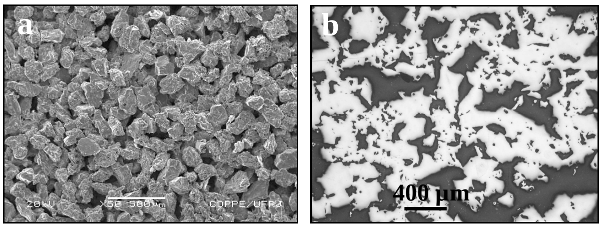

Figure 2: SEM topographic view (a) and optical micrograph (b) of the 85% Ti/15% urea sample.

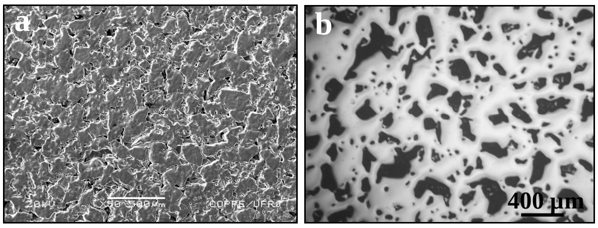

Figure 3: SEM topographic view (a) and optical micrograph (b) of the 100% Ti sample.

Table 1: Samples porosity according to gamma-ray transmission technique.

Sample	Porosity (%)			
	Gamma-Ray Transmission			
	P_1	P_2	P_3	P_4
70% Ti 30% urea	48.70 ± 1.10	50.50 ± 2.70	49.26 ± 0.96	47.80 ± 1.50
85% Ti 15% urea	43.60 ± 1.60	43.30 ± 2.30	43.30 ± 2.10	43.80 ± 2.00
100% Ti	15.70 ± 1.20	15.90 ± 1.10	15.90 ± 1.10	15.50 ± 1.20

Table 2: Samples porosity measured by the geometric method (GM) and the quantitative metallographic analysis (QMA).

Sample	Porosity (%)	
	GM	QMA
70% Ti 30% urea	49.69 ± 1.41	60.53 ± 5.96
85% Ti 15% urea	43.86 ± 2.80	51,06 ± 4,66
100% Ti	15.67 ± 1.76	27,15 ± 4,83

SEM and optical micrograph images (figures 1, 2) illustrated the porous microstructure of the samples with 70% and 85%Ti, which consisted of closed micropores less than 50µm and large interconnected macropores in the range of 100–500µm. The sample with 100%Ti presented only closed micropores less than 100µm (figures 3a, 3b). According to the porosity results (tables 1, 2) and pore morphology (figures 1, 2) of the samples processed with the pore former additive (urea), they presented more adequate porosity for bioengineering applications than the sample processed without pore former [Karageorgiou, V., 2005; Simmons, C.A., 2002].

As the additive quantity is higher, the pore sizes are bigger (figures 1, 2, 3). Also as the additive quantity is higher, the porosity quantity is higher, for all the three methods studied (tables 1, 2). However this difference is not as substantial as expected, according to data of previous research from the authors [Oliveira, M.V., 2006]. Probably, the compaction types used influenced the result, because uniaxial compaction (samples with 70%Ti and 100%Ti) confers less porosity than isostatic compaction (sample with 85% Ti).

The porosity values obtained from gamma-ray transmission (table 1) have shown excellent agreement with the values measured by the geometric method (GM-table 2). On the other hand, the values measured by quantitative metallographic analysis (QMA) are substantially higher than those obtained from gamma-ray transmission and geometric method. Also the standard deviation values of the QMA measurements are much higher (4.83 to 5.96) than those obtained by the other two techniques (gamma-ray/0.96 to 2.70; GM/1.41 to 2.80).

The QMA method is quite dependent on human ability and the analysis were made in only one transverse section of each sample, as sample preparation is time consuming and difficult for soft metals like Ti. Both reasons may induce measurement errors.

In the geometric method, the mass measured is the real one but the method considers the volume sample as a dense piece, without pores, inducing errors in the density value. Also this method is quite dependent on human ability.

The gamma-ray transmission technique is non-destructive and it may analyze several positions, being more representative to report the porosity quantity and homogeneity, compared with the other methods.

Conclusion

The porous samples processed by powder metallurgy with a pore former additive presented the porosity morphology requisites for scaffolds for surgical use. Compared to the other two techniques, the gamma-rays transmission has showed to be a valuable tool for the non-destructive porosity quantification, being more representative to report the porosity quantity and homogeneity of Ti samples.

References

Appoloni, C. R.; Pottker, W. E., Applied Radiation and Isotopes 61, p. 1133-1138, 2004.

Dunand, D. C., Advanced Engineering Materials, v. 6, p. 369-376, 2004.

Karageorgiou, V.; Kaplan, D., Biomaterials, v. 26, p. 5474, 2005.

Li, H.; Oppenheimer, S. M.; Stupp, S. I.; Dunand, D. C.; Brinson, L. C., Jap. Mater. Trans., v. 45-4, p. 1124, 2007.

Oliveira, M. V.; Pereira, L. C.; Reis, L. M., Proceedings of 17º Congresso Brasileiro de Engenharia e Ciências dos Materiais, Foz do Iguaçu/Brasil, 2006.

Ryan, G.; Pandit, A.; Apatsidis, D. P., Biomaterials, v. 27, p. 2651, 2006.

Simmons, C. A.; Meguid, S. A.; Pilliar, R. M., Journal of Orthopedic Research, v. 19, p. 187, 2002.