Comparison of Porosity Measurement Techniques for Porous Titanium Scaffolds Evaluation

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Abstract. Porous titanium has been used for grafts and implant coatings as it allows the mechanical interlocking of the pores and bone. Evaluation of porous scaffolds for bone regeneration is essential for their manufacture. Porosity, pore size, pore shape and pore homogeneity are parameters that influence strongly the mechanical strength and biological functionality. In this study, porous titanium samples were manufactured by powder metallurgy by using pure titanium powders mixed with a pore former. The quantification of the porosity parameters was assessed in this work by geometric method and gamma-ray transmission, the non-destructive techniques and metallographic images processing, a destructive technique. Qualitative evaluation of pore morphology and surface topography were performed by scanning electron microscopy and optical microscopy. The results obtained and the effectiveness of the techniques used were compared in order to select those most suitable for characterization of porous titanium scaffolds.

Introduction

Characterization of materials is of fundamental importance for the development of new materials, both in terms of the reengineering process of preparation, as for potential applications. In this sense there is an increasing number of studies on materials with suitable properties, which has been implemented through the porous materials, named scaffolds. The area of suitable properties and their correlation with the processes of preparation has been the object of various science fields and medicine in particular.

Scaffolds are archetypes for cell interactions where take place migration, proliferation and vascularization of osseous tissue and new bone formation [1]. 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) [2].

For high integration into surrounding tissue, scaffolds should reproduce bone morphology and function. Parameters such as porosity volume fraction pore size, shape, interconnectivity and distribution must be considered for constructing metallic scaffolds. Pores may be closed type or interconnected type, which allows tissue to infiltrate into the scaffold. In general, there is 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 has been studied by many researchers, being the main factor the size of interconnected pores. In order to optimize the mineralized bone ingrowth, there is an agreement that pore sizes must be in the range of 100–400 μm and the porosity quantity in the range of 40–90% [3,4]. Micropores (< 20 μm) and nanopores (1–10 nm) results in larger surface area that is believed to contribute to higher bone inducing protein adsorption, favoring cellular adhesion and implant osseointegration. In addition, a high pore wall roughness has also shown to promote intimate contact with bone and a better implant fixation [5,6,7].

Porosity evaluation of scaffolds is of great importance for their design and processing. Different kinds of information about porosity can be achieved depending on the analytical method and usually a combination of techniques is required. 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 (GM), gamma-ray transmission, and metallographic images processing, 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. Qualitative evaluation of pore morphology and surface topography were performed by scanning electron microscopy (SEM) and optical microscopy. The results obtained and the effectiveness of the techniques used were compared in order to select those most suitable for characterization of porous titanium scaffolds.

Materials and Methods

Pure Ti powder grade 2 (Micron Metals-EUA) made by HDH (hydrogenation-dehydrogenation) process, with acicular shape, particle size range of 149-177 μm and an organic additive (urea), as pore former, were used to make the samples by a powder metallurgy route. Two cylindrical samples with 9.2 mm/height and 11.4 mm/diameter, composed by 70% wt-Ti/30% wt-urea (210-250 μm particle size) and 100%Ti, were compacted by uniaxial compaction at 300 MPa and 450 MPa, respectively. One cylindrical sample with 5.4 mm/height and 8.4 mm/diameter, composed by with 85% wt-Ti/15% wt-urea (149-177 μm 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 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 (4.5 g/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, size pore distribution and average autocorrelation function C(u) were determined by quantitative metallographic analysis (QMA), using Imago

software, in about 15 random images for each sample. The Imago software is a program with tools to estimate physical parameters using samples images with microstructural information. The images presented noise interferences and illumination gradients; because of this they underwent cuts to choose the region of interest and submitted to filter treatment in order to eliminate the noise interferences.

The gamma-ray transmission technique consists in the attenuation that an incident radiation beam undergoes 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 [8]. 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 table 1 and 2 present the porosity values from the Ti samples, obtained by the three techniques analyzed in this work. SEM and optical micrograph images (Figs. 1, 2) illustrated the porous microstructure of the samples with 70% and 85% Ti, which consisted of closed micropores less than 50 μm and large interconnected macropores in the range of 100–500 μm. The sample with 100% Ti presented only closed micropores less than 100 μm (Figs. 3a, 3b). According to the porosity results (Tables 1, 2) and pore morphology (Figs. 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 [5,7].

As the additive quantity is higher, the pore sizes are bigger (Figs. 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 [9]. 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 (GRT) have shown excellent agreement with the values measured by the geometric method (GM-Table 1). On the other hand, the values measured by quantitative metallographic analysis (QMA) are substantially higher than those obtained from GRT and geometric method. Also the standard deviation values of the QMA measurements are much higher (3.00 to 6.10) than those obtained by the other three techniques (gamma-ray/0.96 to 2.70; GM/1.41 to 2.80).

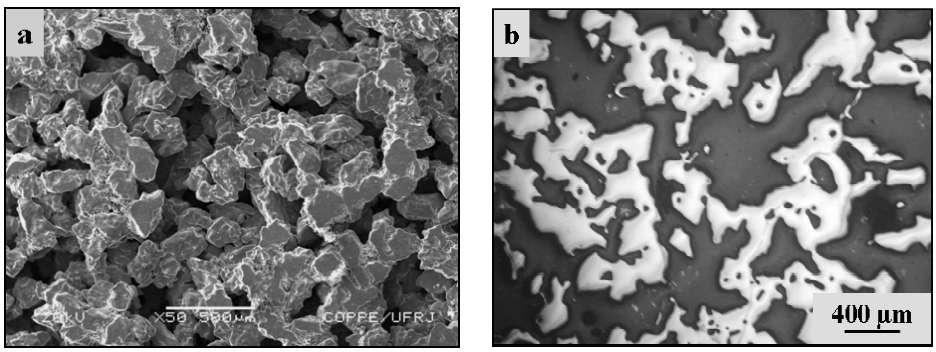

Fig. 1 – SEM topographic view (a); optical micrograph of the 70% Ti/30% urea sample (b).

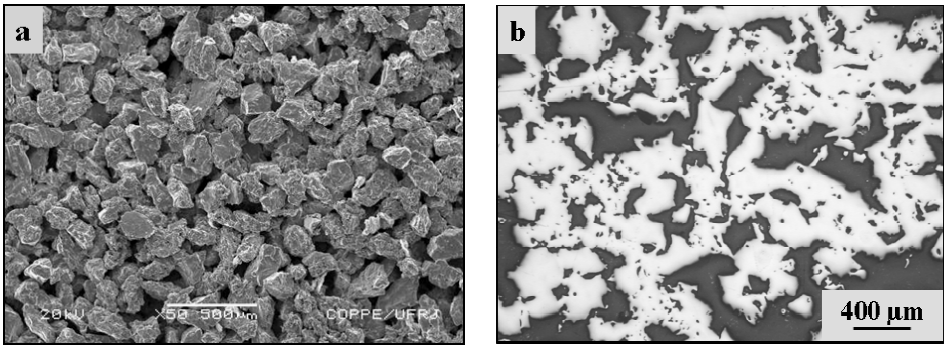

Fig. 2 – SEM topographic view (a); optical micrograph of the 85% Ti/15% urea sample (b).

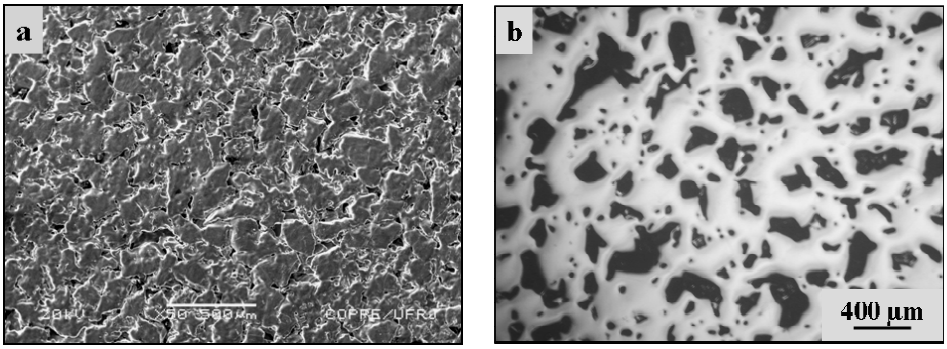

Fig. 3 – SEM topographic view (a); optical micrograph of the 100% Ti sample (b).

Table 1 – Porosity measurements by gamma-ray transmission technique (GRT), geometric method (GM) and quantitative metallographic analysis (QMA).

Sample	Porosity (%)		
	GRT	GM	QMA
70%Ti/30%urea	49.07 ± 1.13	49.69 ± 1.41	60.10 ± 6.10
85%Ti/15%urea	43.50 ± 0.24	43.86 ± 2.80	47.80 ± 3.00
100% Ti	15.75 ± 0.19	15.67 ± 1.76	25.80 ± 5.80

Figure 4 presents the frequency of pores as a function of pore size range for 2D images (Figs. 1b, 2b and 3b), which indicates that approximately 65.3%, 61.4% and 65.1% of the material porous phase refers to pores with radius varying from 42.00 to 144.00 µm, from 15.36 to 66.56 µm and from 16.50 to 54.00 µm for 70%Ti/30%urea, 85%Ti/15%urea and 100% Ti samples, respectively.

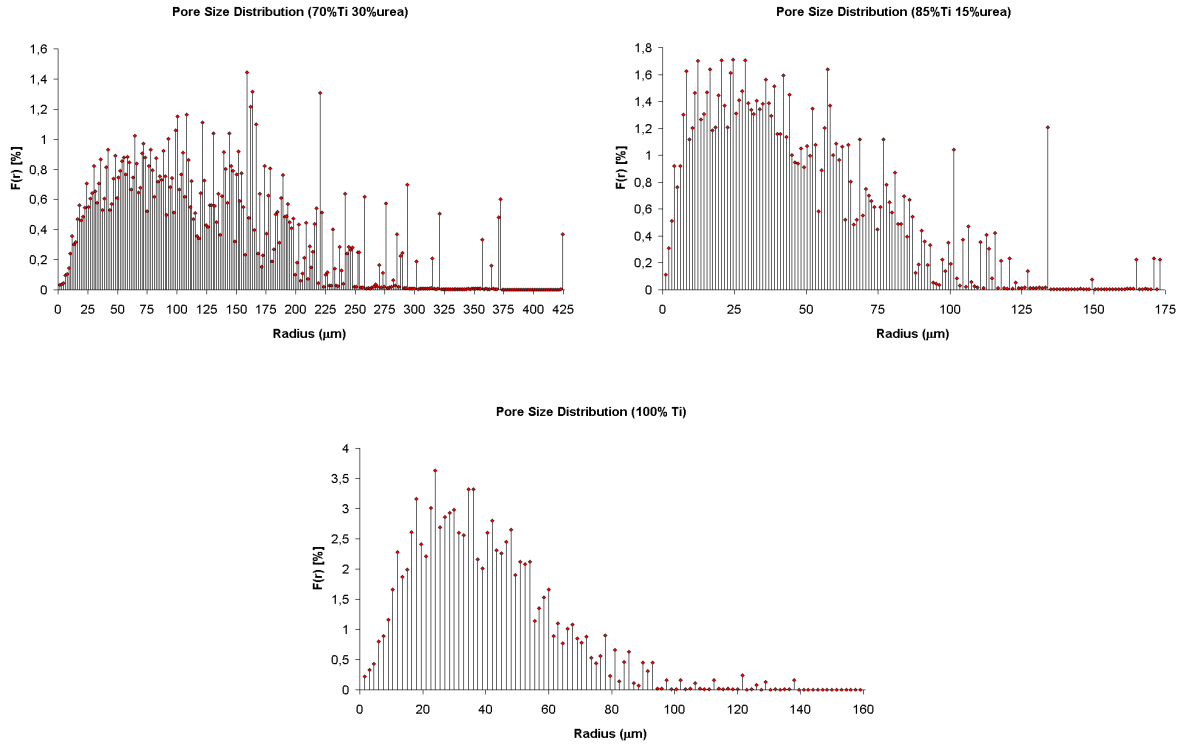

Fig. 4 – Pore size distribution: frequency of pores x pore radius

While the porosity is related to the probability of an arbitrary pixel of the image to belong to the pore phase, the average autocorrelation function, C(u), relates to the probability of finding two pixels separated by u and belonging to the pore phase. These relations constitute the first and second order statistics (or moments) of the image. The average autocorrelation function C(u) was determined with goal to generate 3D models of the samples in order to determine the porosity and the pore size distribution based on 3D volume. However, only 85%Ti 15%urea sample presented agreement between 2D and 3D average autocorrelation functions, indicating that the generated model can be represent the sample. Fig. 5a shows the 2D binary section generated by model of the 85%Ti/15%urea sample. The 3D model was created by truncated Gaussian method [10,11,12], which constructed a cube with 250^3 voxels, spatial resolution of 4.10 μm and magnifying factor of 4 (Fig. 5b) with estimated value of porosity of 48.2 %. Fig. 6 presents the frequency of pores as a function of pore size range for 3D model, which indicates that approximately 57.6% of the material porous phase refers to pores with radius varying from 20.48 to 57.34 μm. The pore size distribution curve of the 3D reconstructed model does not reproduce pore sizes bigger than 65.5 μm, which were measured in 2D images. The 3D volume with 250^3 voxels is not sufficient to generate big pores. To generate a 3D volume bigger than 250^3 voxels it is necessary to use computers with high image processing performance.

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 has many advantages over conventional structural characterization methodologies as it is faster, non-destructive and also does not require sample preparation. By this technique the total porosity of samples can be determined, including closed and open pores, being more representative to report the porosity quantity and homogeneity of Ti samples.

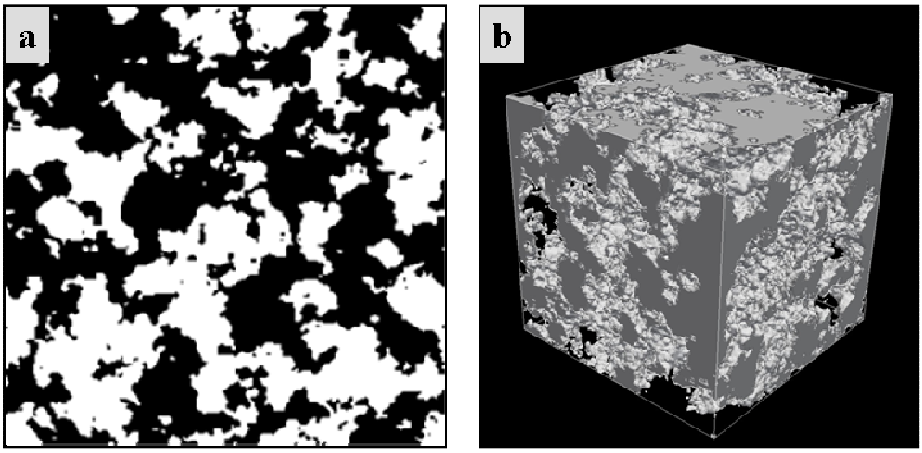

Fig. 5 - 85%Ti/15%urea sample: 2D binary section generated by the model (a) and 3D model generated by truncated Gaussian method (b).

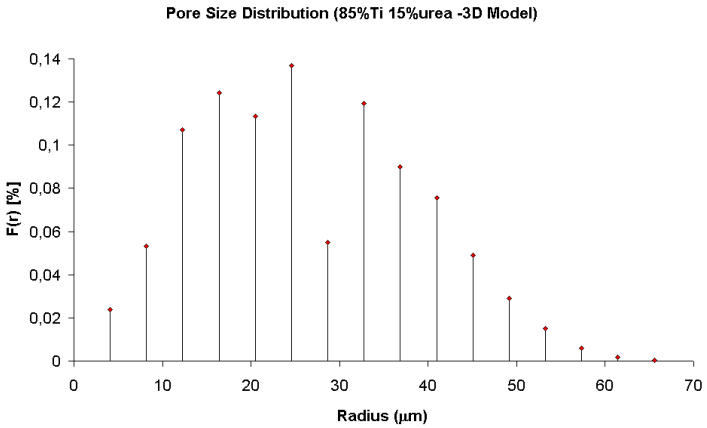

Fig. 6 – Pore size distribution of 3D model of the 85%Ti/15%urea sample.

Conclusions

The porous samples processed by powder metallurgy with pore former additive presented the porosity morphology requisites of scaffolds for surgical use. The characterization of a porous media by image analysis is directly influenced by the software operator intervention. The choice of regions of interest and the noise artifacts presented by the images, besides the illumination gradients, can be responsible for the differences among the porosity values presented by the QMA over the GRT and GM techniques.

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