STRUCTURAL CHARACTERIZATION OF TITANIUM POROUS FOAMS BY GAMMA RAYS TRANSMISSION AND X RAY MICROTMOTOMOGRAPHY

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ABSTRACT

The advance in porous media studies and the consequent progresses of their applications in medicine, petroleum industry, metallurgy and others, rises the prominence of the area among scientists. This fact increases the research on different characterization techniques of porous materials. In this work three Titanium foams were analyzed by gamma rays transmission (GRT), and one of these was also analyzed by X-ray microtomography (\(\mu\)-CT). The GRT experimental set consisted by a 2” x 2” NaI(Tl) detector, a 241-Am radioactive source (59,53keV, 100mCi) and a standard gamma spectrometry electronic chain. In the \(\mu\)-CT technique it was used a SKYSCAN 1172 scanner consisting of an X-ray tube (20-100kV e 0-250\(\mu\)A), a CCD detector and a proper mechanic system for sample and detector movement. The system may reach ~0.8\(\mu\)m image resolution. Images of 815 slices of the sample were generated and analyzed by the IMAGO software. It permitted the determination of geometrical parameters like pore size distribution, total porosity and autocorrelation function. The analysis of data, obtained by both techniques, showed that porous media are homogeneous in the reached resolutions (1mm to GRT and 5\(\mu\)m to \(\mu\)-CT). The average total porosities determined by GRT for each sample were \(\phi_1=53,47\pm 0,36\%\), \(\phi_2=55,95\pm 0,23\%\) and \(\phi_3=56,80\pm 0,56\%\), and the determined by \(\mu\)-CT was \(\phi_1=53,47\pm 0,36\%\). The porosity data of the Ti-1 sample shows good agreement from both techniques. The pore size distribution, from \(\mu\)-CT technique of the Ti-1 sample, showed that 57% of porous phase have porous with radius in 20 to 80 \(\mu\)m range.

1. INTRODUCTION

Titanium and titanium alloys are widely used for surgical implants due to their low density, an excellent combination of mechanical properties, high chemical and corrosion resistance and excellent biocompatibility, when compared with other metallic biomaterials. Titanium foams also exhibit lower weight which is desirable to avoid bone resorption and the consequent loosening of the implant [1,2]. Porosity characterization is considered essential for porous surfaced implants because the pore size and pore morphology influence cellular adhesion. An increase in the fraction of open porosity is required because it improves implant to bone fixation by the growing of bone-forming tissues inside de pores [3].
The structural characterization methods available to evaluate samples porosity and pore size distributions are, in most cases, destructives. Nuclear methodologies, like gamma ray transmission and the X-ray microtomography, are alternatives techniques in porous media research.

The most important advantage of gamma ray transmission technique on total porosity determination and others applications is that it is a non-destructive method with high degree of reliability in the acquired data [9].

The µ-CT technique permits the study of internal topology and geometry of samples by image analysis. These images are acquired by sample irradiation using an X-ray source. The image analysis is done with specific software that permits the micro-characterization of samples parameters like porosity, pore size distribution and the two point correlation function [4,5,6].

The µ-CT is an inspection method that provides detailed images by the linear attenuation coefficient mapping of the sample crossed by the x-ray beam. The X-ray attenuation results in projections along the measurement line of the material intern structure are accomplished, transversally of the sample longitudinal axis, originating 2D images of slices of the whole sample that are analyzed with appropriate image analysis software.

This work shows the application of both methodologies on the structural characterization of Titanium foams and the good agreement between them.

2. THEORETICAL

The gamma ray transmission technique consists in the attenuation that an incident radiation beam suffers when go across this material. The Beer's law establishes the relationship between the attenuated radiation intensity by a sample and other parameters of the system. It can be written as:

\[ I = I_0 e^{-\mu' x} \]  \hspace{1cm} (1)

where \( I_0 \) and \( I \) are the incident and emergent gamma ray beam intensities respectively (cont s\(^{-1}\)), \( \mu' \) is the linear attenuation coefficient of the sample (cm\(^2\) g\(^{-1}\)), and \( x \) is the thickness of the sample (cm). The linear attenuation coefficient (\( \mu' \)) can be written as:

\[ \mu = \frac{\mu'}{\rho} \]  \hspace{1cm} (2)

where \( \mu \) is the mass attenuation coefficient (cm\(^2\)/g), and \( \rho \) is the density of the sample (g/cm\(^3\)).

The porosity determination of the sample, in relation to his linear attenuation coefficient [7], is done by the following equation:
\[
\phi = 100 \left( \frac{\mu_p - \mu_s}{\mu_p} \right)
\]  

(3)

where $\phi$ is the porosity, $\mu_p$ is the linear attenuation coefficient of the sample totally solid (same material of the sample but without pores) and $\mu_s$ is the linear attenuation coefficient for the porous sample, both in cm$^2$/g.

The $\mu$-CT is an inspection method that provides detailed images by the linear attenuation coefficient mapping of the sample crossed by the x-ray beam. The X-ray attenuation results in projections along a measure line, these projections of the material internal structure are accomplished, transversally of the sample longitudinal axis, originating 2D slices of the whole sample that are analyzed with appropriate images analysis software.

### 3. MATERIALS AND METHODS

Porous titanium samples, with density 4.5 g/cm$^3$, were processed by a powder metallurgy route. Pure titanium powder grade 2 (Micron Metals-EUA) made by HDH-hydrogenation-dehydrogenation process was used. The sintered samples dimensions are approximately 15.3x15.3x5 mm$^3$. Figure 1 shows the Titanium sample.

![Figure 1. Porous Titanium sample.](image)

The Figure 2 presents the experimental setup of gamma ray transmission consisting of a 2" x 2" NaI(Tl) detector, a 241-Am radioactive source (59.53keV, 100mCi), an automatic micrometric table for the sample movement and a standard gamma spectrometry electronic chain. The gamma ray apparatus is localized at the Applied Nuclear Physics Laboratory at the Londrina State University-Londrina/PR.

The X-ray microtomographic system used (Figure 3), localized at the CENPES/PETROBRAS-Rio de Janeiro/RJ, was a SKYSCAN 1172 scanner consisting of an X-ray tube (20-100kV, 0-250µA), a 10Mpixel CCD detector and system for sample and detector movement. The equipment reaches ~0.8µm image resolution.
Calculations of the mass attenuation coefficients of the samples were carried out by the WinXCOM program [5]. The software can generate cross sections and attenuation coefficients for elements, compounds or mixtures in the energy range between 1 keV and 100 GeV, in the form of total cross sections and attenuation coefficients as well as partial cross sections of the following processes: incoherent scattering, coherent scattering, photoelectric absorption, and pair production in the field of the atomic nucleus and in the field of the atomic electrons. For compounds, the quantities tabulated are the partial and total mass interaction coefficients.

4. RESULTS AND DISCUSSION

Three samples of Ti foam were measured by GRT, and one of these (Ti-1) was also analyzed by µ-CT. The linear attenuation coefficient of the porous sample was obtained by beam transmission at 27 random points along each sample with 300 s measurement time. The mass attenuation coefficients of the titanium foams were determined with WinXCOM. The average porosity results for the samples are showed at the Table 1.

With the GRT technique it was also determined the porosity profiles for the samples. Figure 4 shows these profiles for the Ti-1, Ti-2 and Ti-3 samples.
### Table 1. Average porosity data by GRT technique.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ti-1</th>
<th>Ti-2</th>
<th>Ti-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity (%)</td>
<td>53.47</td>
<td>55.95</td>
<td>56.80</td>
</tr>
<tr>
<td>Deviation</td>
<td>0.21</td>
<td>0.23</td>
<td>0.33</td>
</tr>
<tr>
<td>95% of confidence</td>
<td>0.36</td>
<td>0.40</td>
<td>0.56</td>
</tr>
</tbody>
</table>

#### Figure 4. Porosity profile of the Titanium samples by GRT technique.

The tomography measurements were accomplished with the following scan conditions: 60kV, 167µA, Al filter (1 mm), each 5 frames averaged, 1475 ms integration time per frame, 180.0° total rotation angle with 0.45° step size of rotation angle. 815 images of sample slices were reconstructed with 5 µm spatial resolution.

In order to characterize the structure of samples using the IMAGO software [8], it is necessary to treat the two dimension images. The binarization, part of this treatment, is performed based in a gray level histogram. Figure 5 (a) shows a 2D image of Ti-1 sample, and Figure 5 (b) presents the same binarized slice.

#### Figure 5. (a) 2D slice (gray level) from the Ti-1 sample and (b) same slice binarized.
The porosity determined to Ti-1 sample by µ-CT method is $\phi_1 = 53,38 \pm 0,14\%$. The porosity estimations for the Ti-1 sample obtained from both techniques show good agreement. The size pore distribution, from µ-CT technique of the Ti-1 sample, shows that 57% of porous phase have porous with radius in 20 to 80 µm range. Figure 6 shows the pore size distribution. A fraction of the 3D real volume of this sample is showed at Figure 7 (a) and the 3D model generated with the Imago software is showed at Figure 7 (b). This model can be use in computational simulations methods. Table 2 shows the porosity values from the Titanium foams obtained by the two nuclear methodologies, gamma ray transmission an X-ray microtomography.

**Figure 6. Pore size distribution from the Ti-1 sample.**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Gamma Ray Transmission (%)</th>
<th>X-Ray Microtomography (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-1</td>
<td>53,47 $\pm$ 0,36</td>
<td>53,38 $\pm$ 0,14</td>
</tr>
<tr>
<td>Ti-2</td>
<td>55,95 $\pm$ 0,40</td>
<td>-</td>
</tr>
<tr>
<td>Ti-3</td>
<td>56,80 $\pm$ 0,56</td>
<td>-</td>
</tr>
</tbody>
</table>

**Table 2. Titanium foams porosity.**

**Figure 7.** (a) fraction of the 3D real volume of Ti-1 sample and (b) 3D model performed using the Imago.
3. CONCLUSIONS

The gamma rays transmission technique showed itself a powerful tool to the non destructive quantification of the porosity of the Ti porous foam samples analyzed in this work. Similar results of total porosity were obtained by microtomography.

The employed microtomography system showed good capacity to study the structural parameters of the considered sample. The Ti-1 sample images permitted to quantify the porosity and the pore size distribution, showing that approximately 1.3 % of the porous phase consists of porous with radius smaller than 5.0 µm (the system spatial resolution).

The Imago software was an important tool on structural characterization for determinations of porosity and size porous distribution, allowing also the construction of a 3D model to be used in computational simulations.

REFERENCES