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On the influence of space holder in the development of porous titanium implants: Mechanical, computational and biological evaluation

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ABSTRACT

Commercially pure titanium (cpTi) is frequently used for bone replacement in both dentistry and orthopedics applications, primarily due to its bioinert behavior, but also for providing optimum biomechanical behavior. It is recognized that the high elastic modulus of cpTi is associated with the stress-shielding phenomena, which promotes bone resorption. Development of implants with low elastic modulus, providing suitable mechanical strength and optimum osseointegration, is the focus of emergent research in advanced Ti-based alloy biomaterials. Porous metals and, in particular, porous titanium can provide the optimal combination of biocompatibility, high strength and minimal stiffness that best mimic bone. Synthesis of porous Ti, controlling porosity and interfacial properties, which optimizes in-vivo integration, remains the subject of recent research. In this work, porous samples of cpTi grade IV obtained by space-holder technique with ammonium bicarbonate (NH_4HCO_3) have been studied. For a fixed value of compaction pressure, evaluation of porosity and mechanical properties were performed to determine the influence of space-holder content (from 30 to 70 vol.%) in the global performance of the cpTi porous samples. Porous materials with enhanced mechanical behavior has been achieved, exhibiting an appropriate Young's modulus, which reduces stress shielding, as well as reasonable good mechanical strength. Additionally, in order to go in-depth on the study of these porous cpTi samples, a finite element model has been proposed and the macroscopic mechanical response of porous compacts analyzed, showing a good agreement with experimental results. In addition, stress distribution around pores of the compacts has been analyzed, and the influence of the compact microstructure on the obtained stress distribution has been studied. Finally, biological tests in the obtained porous compacts have been carried out. Adhesion of bone cells inside the pores has been analyzed, which is a good indicator of their potential improvement of osseointegration. Cells behavior inside the pores appears to be clearly sensitive to roughness and geometry of pores (diameter and curvature).
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1. Introduction

Bone tissue problems and correlated diseases are still considered a public health issue in many countries. Real alternatives to these problems through clinical products from tissue engineering or by regenerative medicine practice can be considered as part of a promising future nowadays [1]. Therefore, the necessity to improve the current materials used for bone replacement is an unquestionable issue [2]. Within this context, it is well known that commercially pure Titanium (cpTi) and Ti6Al4V alloy are the best metallic biomaterials for bone replacement, basically due to their excellent biofunctional equilibrium, reflected in their high clinical success. This equilibrium is the result of a high biocompatibility, related with their bioinert response, and a good mechanical behavior with respect to biomechanical solicitations. These recognized advantages are not a sufficient guarantee for an optimum reliability of the implants and prosthesis, due to a series of drawbacks in the use of full dense titanium. Firstly, titanium is typically surrounded by a thin film of fibrous tissue, which is related to many loosening events that leads to failure. Secondly, Young's modulus of titanium is higher than bone, which produces the stress-shielding phenomenon, increasing bone resorption around the implant.

Solving the stress-shielding problem requires the development of new implants and prosthesis with a lower stiffness than conventional designs, without any critical detrimental effect on the mechanical strength. Within the effort to obtain implants with a better stiffness match with the cortical bone, there are several important advances already reported [3]. One of those explored routes to obtain porous titanium is the space-holder technique, which is a modification of

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conventional powder metallurgy. This technique consists of mixing the metal powder with a special additive to be removed before sintering. Several authors [4–9] have obtained promising results by using this technique with different additives like ammonium bicarbonate, carbamide, PVA and sodium chloride. Despite their relevant works, there is still a lack of knowledge about the space-holder role in achieving an optimum mechanical equilibrium between stiffness and mechanical strength, as well as in the improvement of osseointegration.

With the aim of achieving a better understanding of the space-holder technique, a very comprehensive investigation has been developed in this work, which comprised of three parts: mechanical testing, numerical analysis and in vitro biological testing. A series of porous compacts of cpTi GIV has been obtained by this technique, using ammonium bicarbonate (NH_4HCO_3) as space-holder. By varying its relative content, five different porous materials have been obtained, with 30, 40, 50, 60 and 70 vol.% space-holder concentration. Processing conditions, such as blends mixing parameters, sintering time and temperature, as well as the compaction pressure, have been fixed according to some previous works of the authors [10,11].

In the first part of the paper, the influence of space-holder content in the global performance of the cpTi porous samples has been analyzed: porosity and mechanical properties.

In order to complement the study, the second part of the work is focused on the numerical simulation of the mechanical response of porous compacts, by the Finite Element Method (FEM). Conventional tools of mechanical experiments sometimes are not sufficient to enable understanding of relationships between processing, microstructure and mechanical response of a porous sample. Analytical models are capable of predicting the overall material response under idealized conditions or simplifying assumptions [12]. In contrast, finite element models are able to consider more realistic structures. With that aim, Li et al. [13] made an attempt to describe the effect of pore morphology on the mechanical properties of titanium foams by using a 2D finite element model based on periodically distributed circular pores geometries. Shen et al. [14,15] developed a finite element model to study the macroscopic and microscopic response of titanium foam with a porosity of 15%, by generating a 3D porous geometry based on minimal 2D microstructural information from a metallography: pore size and site distribution. This model was only used for titanium foams with spherical pores. Furthermore, due to its computational complexity, only one case with 15% of porosity was studied. However, in porous titanium obtained by the space-holders technique, the pore morphology is different: more elongated pores, which lead to higher stress concentrations and, consequently, a decrease of the mechanical strength of the porous material. In order to overcome this difficulty, a 2D finite element model, based on geometries generated from information of the pore morphology, has been proposed in this work. By using the proposed model, suitable for the study of porous titanium by space-holder technique, a conscientious FEM analysis has been included.

Finally, in the third part of the paper, biological tests in the obtained porous compacts have been carried out to measure their osseointegration with the bone. Results have allowed addressing the potential to achieve the desired mechanical equilibrium, as well as to have the actual sense of the osseointegration behavior of the samples, by using the analysis of cells adhesion inside the pores as an indicator of the same.

2. Materials and methods

2.1. Sample processing

Powder of cpTi GIV (ASTM F67-13 Standard, [16]), with equiaxial morphology and very rough surface, has been used for the blends [3]. The particle size distribution corresponds to 10%, 50% and 90% passing percentages, of 9.7, 23.3 and 48.4 μm, respectively. Ammonium bicarbonate granules (NH_4HCO_3, Cymit Química S.L., Spain), employed as space-holder, have a particle size corresponding to 10%, 50% and 90% passing percentages, of 73, 233 and 497 μm, respectively, with a purity of 99.9%. They are irregular and also very rough [3]. The blends of cpTi powder and NH_4HCO_3 granules [cpTi + NH_4HCO_3], with a NH_4HCO_3 concentration between 30–70 vol.%, have been prepared by using a Turbula ® T2C blender for ~40 min in order to ensure good homogenization [10,11]. The compacting step has been carried out using an Instron 5505 universal machine (Instron, Norwood, MA, USA) to apply the pressure used (800 MPa). The diameter of compaction die (8 mm) and total powder mass have been selected in order to obtain samples in which the effect of compaction pressure is minimized [10]. Elimination of NH_4HCO_3 from green samples has been thermally performed in two steps: initially placing the samples in an oven at 60 °C for 10 h and then, an additional 12 h at 110 °C, both experiences performed using low vacuum ($\sim 10^{-2}$ mbar). Finally, the sintering process has been performed in a Carbolyte ® STF 15/75/450 ceramic furnace with a horizontal tube at 1250 °C for 2 h using high vacuum ($\sim 5 \cdot 10^{-5}$ mbar), with heating and cooling rates of 10 °C/min.

Following this procedure, five different porous materials have been obtained, with 30, 40, 50, 60 and 70 vol.% space-holder concentration, leading to 28, 37, 47, 57 and 66% total porosity, respectively.

2.2. Porosity characterization

Visual inspection has been used for verifying structural integrity of both green and sintered samples. Density measurements have been carried out using Archimedes' method with distilled water impregnation (ASTM C373-14 Standard, [17]). A detailed description of the procedure can be found in previous works of the authors [3,10,11]. Total porosity has been then calculated from density measurements. Pore morphology has been performed by image analysis (IA) by using a Nikon Epiphot optical microscope coupled with a Jenoptik Progres C3 camera, and suitable analysis software (Image-Pro Plus 6.2). Before the image analysis, the sectioned parts have been properly prepared [3,10,11]. Image analysis has been assessed using 10 pictures of 5× for each porous sample. The main pore characteristics estimated by this method are: the total porosity (θ), the equivalent diameter (D_{eq}), and the pore elongation factor (F_e, ratio between major and minor axis of ellipse equivalent to pore) [18]. Additionally, conventional optical microscopy (OM) and scanning electron microscopy (SEM) have been utilized for the basic observation of the microstructural features of the samples.

2.3. Mechanical testing

For mechanical compression testing, the specimen dimensions have been fixed to standard recommendation from ASTM E9-89a (height/diameter = 0.8) [19]. The tests have been carried out with a universal electromechanical Instron machine 5505 by applying a strain rate of 0.005 mm/mm·min. All tests finished for a strain of 50% and, afterwards, both Young's modulus (E), and yield strength (σ_y) have been obtained. The Young's modulus estimation from the compression stress–strain curves have been corrected with the testing machine stiffness. Three specimens have been employed for each space-holder percentage. These tests have been performed on the samples in which porosity (density) and dynamic Young's modulus were already measured. Measurements of dynamic Young's modulus have been performed by ultrasound technique with a Krautkramer USM 35® flaw detector. This method allows to determine both longitudinal and transverse propagation velocities of acoustic waves. In order to evaluate the longitudinal waves, a Panametric S-NDT® 4 MHz ultrasonic transducer was used with an ultrasonic couplant (Sonotrace grade 30®). For transverse waves, a Panametric S-V153® 1.5 MHz shear wave transducer was used using a shear wave couplant (Panametrics-NDT(TM)). From the measured acoustic wave velocities, the dynamic Young's modulus can be analytically evaluated [20]. The authors described details of the method in some previous works [3,10,11]. Some other

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