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Development of porous titanium for biomedical applications: A comparison between loose sintering and space-holder techniques

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ABSTRACT

One of the most important concerns in long-term prostheses is bone resorption as a result of the stress shielding due to stiffness mismatch between bone and implant. The aim of this study was to obtain porous titanium with stiffness values similar to that exhibited by cortical bone. Porous samples of commercial pure titanium grade-4 were obtained by following both loose-sintering processing and space-holder technique with NaCl between 40 and 70% in volume fraction. Both mechanical properties and porosity morphology were assessed. Young's modulus was measured using uniaxial compression testing, as well as ultrasound methodology. Complete characterization and mechanical testing results allowed us to determine some important findings: (*i*) optimal parameters for both processing routes; (*ii*) better mechanical response was obtained by using space-holder technique; (*iii*) pore geometry of loose sintering samples becomes more regular with increasing sintering temperature; in the case of the space-holder technique that trend was observed for decreasing volume fraction; (*iv*) most reliable Young's modulus measurements were achieved by ultrasound technique.

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1. Introduction

Bone degradation related to trauma and disease, as well as its consequent replacement, is considered a public health problem today, which affects one in seven North Americans [1]. This tissue degradation is evident by density reduction above the age of 30 years old, which involves a strength reduction of up to 40% which could be increased by both the cyclic loading and surface wear of joints [2]. These facts can be supported through statistics such as those provided by the *American Dental Association* [3] indicating that 113 million North American adults have lost at least one tooth and 19 million are edentulous. It has also been reported that 10–15% of implants fail in the first 10 years, and 20% of the surgeries are carried out to replace failed implants [4]. Furthermore, the demand for implants is still growing as a result of the rise in life expectancy (13% of the population in the USA are aged 65 years or older; an increase of 8% more has been forecasted for 2050 [5]). Moreover, in younger patients the need for prostheses is also rising, which implies that implants will be subjected to higher levels of mechanical loading for longer periods of time. In this context, further research is needed to develop methodologies that can improve in vivo performance of all implantable devices.

Among all biomaterials employed for bone replacement, it is recognized that titanium and its alloys are those with the best in vivo behavior. Despite this, implant fixation to the bone remains an aspect to be improved through alternatives for reducing the stress-shielding phenomenon, which is a consequence of the mismatch between Young's modulus values (Titanium is 110 GPa and cortical bone around 20–30 GPa); this difference has been identified as one of the major reasons for implant loosening [6–8] and bone resorption. Furthermore, it has been suggested that when bone loss is excessive, it can compromise the long-term clinical performance of the prosthesis [9]. This may also be responsible for implant migration, aseptic loosening, fractures around the prosthesis, and can imply technical problems during revision surgery [9].

Although titanium and its alloy (Ti-6Al-4 V) are the metallic biomaterials with the lowest elastic modulus value (50% lower than Co–Cr), the mismatch with respect to bone stiffness remains a challenging problem that still needs to be addressed. Manufacturing of implants with lower stiffness materials could be a solution for the stress shielding phenomenon [10]. In that sense, development of porous materials is an alternative approach to achieve a stiffness reduction. However, an important issue with the use of porous implants for load bearing applications is the risk of reduction of both mechanical strength and fatigue resistance because the material must be able to withstand the loads without failure. Therefore, to get a desired balance between strength and stiffness is the most important challenge of this approach and has to be accomplished.

There is some ongoing work and developments about biocomposites and porous titanium implants that still do not fulfill the suitable

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equilibrium between mechanical and biofunctional properties [11–14]. Several workers have previously shown that it is possible to match the stiffness of cortical bone by using different techniques to produce porous titanium samples [15–25], however, there is a lack of studies about relationships between processing parameters, microstructure and the effect of porosity on the mechanical properties of porous titanium samples.

Porous titanium can be produced by several methods such as loose powder sintering [26,27], slurry foaming [28], reactive sintering [29], hollow sphere sintering [16] and entrapped gas techniques [30]. However, the production of porous materials via the conventional powder metallurgy (PM) route can be cost effective, flexible and lead to the desired design foams [20]. In addition, most of the above-mentioned methods provide limited porosity. Recently, a new powder metallurgy technique using space holder materials (such as carbamide [20,31], NaCl [32,33], K_2CO_3 [34], PMMA [35,36]) has been developed, which has the advantages of great uniformity, adjustable porosity amount, controlled pore shape, and more uniform pore size distribution [37–43].

In this work, loose-sintering and space-holder techniques were used to manufacture porous cpTi samples and the influence of processing on both their microstructural and mechanical properties was investigated. The relationships between morphological features and mechanical properties are also rationalized.

2. Experimental

2.1. Materials

Commercially Pure titanium powder (cp Ti) produced by a hydrogenation/dehydrogenation process was used as the starting powder. The particle size distribution, according to the supplier SE-JONG Materials Co. Ltd., Korea, exhibited particles with irregular morphology and sizes, corresponding to 10%, 50% and 90% passing percentages, of 9.7, 23.3 and 48.4 μm, respectively. The chemical composition is equivalent to commercially pure titanium ASTM F67-00 Grade IV. The space holder used was NaCl (Panreac, purity > 99.5%) due its undemanding decomposition. The concentration of NaCl was within a range of 40–70vol.%, which was similar to that used in previous papers [14,20,44]. The NaCl granules employed as space-holder presented particle sizes corresponding to 10%, 50% and 90% passing percentages, of 183, 384 and 701 μm, respectively. The authors chose a space holder (NaCl) with a large particle size, and cpTi with a fine particle size; there are several reasons for this: (i) A wider NaCl particle distribution (which promotes a higher degree of interconnectivity of the pores), and a high average size of space-holder (>100 μm) would fulfill the requirements to ensure the growth of bone into the implant (ingrowth); (ii) On the other hand, the choice of a titanium powder of irregular shape and small average size would improve the sinterability of the compact (quality of the neck and lower grain size); this will also help to offset the loss of mechanical strength inherent in increased porosity.

2.2. Processing of porous titanium

Two different PM techniques were used: loose-powder sintering and space-holder technique. A sketch illustrating the processing stages for both methods is shown in Fig. 1.

Loose-powder sintering is a method in which the metal powder is poured or vibrated into a mold which is then heated to the sintering temperature in an appropriate atmosphere [27]. Fig. 1a represents the loose-sintering process applied to porous cpTi powder blended for 40 min in a TURBULA T2C mixer. Subsequently, the powder was vibrated into a cylindrical mold of alumina for 2 min. This was a measure of densification of the powder. Finally, the sample was heated to 1000 °C and 1100 °C for 2 h, in a CARBOLYTE STF 15/75/450 ceramic furnace with a horizontal tube under high vacuum (~5×10^{-5} mbar). The above processing conditions were chosen in order to obtain mechanical properties (Young modulus and yield strength) similar to the cortical bone [45].

The space-holder technique is a recently developed PM method that allows porous structures to be obtained with controlled porosity and an enhanced homogeneity. A sketch of the aforementioned process is shown in Fig. 1b. The powder metallurgy technique used to manufacture these samples consisted of a conventional process: 1) mixing of the titanium powder and NaCl particles for 40 min in order to ensure a good homogenization, 2) compaction of the mixture (pressure of 800 MPa, defined according to compressibility curves of materials and the results of a previous work [2,46]), 3) subsequently, the salt was dissolved with distilled water (50–60 °C), in cycles of 240 min (see details in previous work [2,46]), and 5) finally, the sintering temperature was fixed at 1250 ºC for 2 h under high vacuum [2,46].

In both methodologies, the powder mass used to obtain specimens with dimensions suitable for testing compression (height/diameter = 0.8) was overestimated and varied between 2.0 and 2.1 g in order to ensure the desired ratio for compression testing, as well as to avoid the effect of some losses during handling and cutting of the samples. The compaction step was carried out by using an INSTRON 5505 universal machine to apply the pressure needed for the desired porosity, followed by a MALICET ET BLIN U-30 universal machine in order to remove the samples from the matrix. The compaction loading rate was 600 kgf/s, the dwell time was 2 min and the unloading time was 15 s for decreasing loads up to 15 kgf.

2.3. Microstructural and mechanical characterization

The density measurement was carried out by using the Archimedes method with distilled water impregnation, due to its experimental simplicity and reasonable reliability (ASTM C373-88). The total porosity P(Arch) and interconnected porosity (Pi) were calculated from the density measurements by using well known mathematical expressions [47].

For the image analysis, the sectioned parts were prepared by a sequence of conventional metallographic steps (resin mounting, grinding and polishing) followed by a mechano-chemical polishing with magnesium oxide and hydrogen peroxide. Conventional optical microscopy (OM) was also used for the basic observation of the microstructural features of the samples. The porosity evaluation by image analysis was performed by using an optical microscope NIKON EPIPHOT coupled with a JENOPTIK PROGRES C3 camera, and suitable analysis software (IMAGE-PRO PLUS 6.2). The following morphological pore parameters were estimated by this method: the total porosity (P_{IA}), equivalent diameter (D_{eq}) [48] defined as the average diameter measured from the pore centroid, and aspect ratio ($F_f = 4\pi A/(PE)^2$) [48], where A was the pore area and PE was the experimental perimeter of the pore representing a measure of roundness of the pores. Zero values correspond to maximum irregularity and values closer to 1 match with more spherical pores. The mean free path between the pores was defined as the average size of the necks between the pores (λ) [49], and the pore interconnectivity (C_{pore}) defined as the fraction of connected pores of the total reference line length [50].

For mechanical compression testing, the specimen dimensions were fixed according to those recommended (height/diameter = 0.8) in Standard ASTM E9-89A [51]. The yield strength, relative strength (defined as the ratio between the strength of porous material and the solid material) and the Young's Modulus were also obtained. Furthermore, the dynamic Young's modulus measurements using the ultrasound technique were performed with KRAUTKRAMER USM 35 equipment which was used to estimate both longitudinal and transverse propagation velocity of acoustic waves (ASTM E494-10). For each case the PANAMETRICS probe and a suitable ultrasonic couplant fluid was used. Once the acoustic wave velocities were measured, the dynamic Young's modulus was calculated using a known mathematical expression [52].

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