1. Introduction

Damage to human bone tissue is highly influenced by the age and gender of the patient, pathologies (genetic, dietary and acquired infections), and injuries due to accidents, among others. In addition, the trend towards increased life expectancy increases the probability of bone fracture, wear, loss of density, etc., which involves the necessity to use implants for total or partial bone replacements [1-4]. In this context, the clinical use of commercially pure titanium (Ti c.p.) and $TiAl_6V_4$ alloy are the bio-metallic materials more widely employed. However, problems associated with the bone resorption phenomenon, due to the stiffness mismatch between the cortical bone (20-25 GPa) and the implant of titanium (100-110 GPa), have been reported [5,6]. The use of porous implants to solve the stress-shielding problem has been widely addressed by the scientific community. In this context, the authors of this work present in the Table 2 a new classification of up to 34 different techniques to produce porous metallic materials (also named foams or cell materials) [10-125], classifying these processing routes by the raw material presentation: liquid metal, metal powder, metal preform and, metal powder suspension. On the other hand, other great goal is to replicate the hierarchical structure of bone tissues. Moreover, radial graded porosity materials are also needed in a great many applications, such as self-lubricated parts, CO_2 capture systems, substrates for catalysis, high efficiency heat sinks and surrogate materials to simulate irradiated nuclear fuel, among others.

Table 2. Classification of the different techniques to develop porous metallic materials.

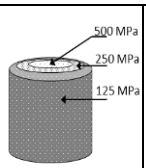
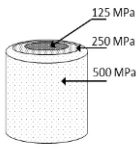
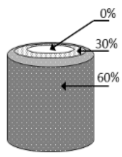
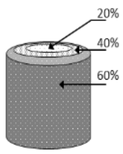
State of the starting material	Name of the Technique			At what stage are the pores generated?	Heat source	Gradient Porosity (G) Monolithic (M)
Liquid Metal	Space-Holder	Granular particles [10-21]		During pouring into the mould. It requires subsequent removal by a leaching or thermal process.	Cooling of liquid	G, M
		Replica of polymeric skeletons [22-24]				
	Direct injection of gas into liquid [25-31]			During the generation of the gas bubbles, by trapping		M
	Foaming agent (H2) [7,9,32-36]					M
	Eutectic Hydrogen GASARs [37]					M
Powder Metal	Different particle diffusion coefficients [38-42]			During sintering	Furnace	M
	Different sized particles [43,44]			During compaction		G
	Compacting at low pressures [8,45-47]					M
	Loose Sintering [48-53]			During non-compaction		M
	Loose Sintering and different sized particles [48-52]					G
	Injection moulding of metallic powder (MIM) [54]			During powder injection		M
	Space-Holder	Granular particles	Similar proportions and / or sizes [55-66]	During compaction. Requires subsequent removal of the spacer by a leaching or thermal process		M
			Different proportions and /or sizes			G
			Isostatic Hot Pressure [67,68]			M
		Replica of polymeric skeletons				G, M
	Ar gas trapped in powder [69-74]			During compaction bubbles are generated by gas trapped or by expansion		M
	Foaming agent mixed in powder					M
	Foaming agent mixed in powder and subsequent compaction [75-82]					M
	Rapid prototyping [83-86]			During manufacture of the green by stratification of layers according to previous model		G, M
	Self-propagation high temperature synthesis (SHS) [87-90]			During propagation of heat wave added after compaction and preheating		M

	Superimposition of different sheets [91,92]	Porous sheet on solid cores	During compaction using spacers. Subsequent removal of the spacer by leaching or a thermal process		G
		Sheet metal foams	During compaction bubbles are generated by expansion of the foaming agent		G
	Selective laser sintering (SLS) [93-98]		During sintering layer by layer by the laser beam	Laser	G, M
	Selective laser melting (SLM) [99]				G, M
	Direct metal laser sintering (DMLS)		During sintering of a single layer by the laser beam		M
	Electric current assisted sintering (ECAS) [100-105]		During powder compaction and electric field application at the same time	Electric Field	M
	Electro-discharge capacitors [104-106]		During powder compaction with electro-discharge		
	Electron beam melting (Arcam) [107]		During manufacture of green and sintering layer by layer	Electron Beam	G, M
	Combined electron beam melting [108]		In the manufacture of green by use of a polymeric foam scaffold		G, M
Preform	Hollow metal spheres [109-110]		During the manufacture of the metal preform and the compaction.	Furnace or Electric Field	G, M
Powder Metal Suspension	Polymer scaffold immersion [83,111,112]		During the scaffold coating	Furnace	G, M
	Directional freezing [113,114,118]		During the dendritic growth of conductive liquid of the process		G
	Electro deposition of metal on polymeric substrate [119-124]		During deposition of metal ions on the foam scaffold		G, M

Some available works show the limitations in controlling the quantity, size, distribution and morphology of the pores by conventional routes. Other works indicate the high cost, and the great difficulty in obtaining reproducibility and versatility of the new processing routes (laser sintering, ion beam milling, field assisted sintering technology, etc.). In this work, the authors develop the optimisation and validation of a novel sequential uniaxial compaction device, to produce cylinders of radial graded porous materials.

Figure 1. Components of the compaction device: a) die, b) set of compaction punches, c) set of extraction punches and d) centring tools.

Table 1. Radial graded porosity designs developed, and their corresponding manufacturing conditions

<table>
<tr><td></td><td colspan="2">Conventional Powder Metallurgy (PM)
Different compaction pressures 125-250-500MPa; No spacer</td><td colspan="3">Space-holder Technique (SHT)
Compaction pressure 800MPa; Different contents of spacer</td></tr>
<tr><td>Porosity distribution</td><td>
Increasing Gradient (IG)</td><td>
Decreasing Gradient (DG)</td><td>
Hard Gradient (HS)</td><td colspan="2">
Soft Gradient (SG)</td></tr>
<tr><td rowspan="2">Spacer size</td><td colspan="2" rowspan="2">0 µm</td><td colspan="2">NaCl</td><td>$NH_4(HCO_3)$</td></tr>
<tr><td>D[4,3]= 445µm</td><td>[4,3]= 206µm</td><td>D[4,3]= 265µm</td></tr>
<tr><td>Lubricant removal</td><td colspan="4">100ºC (2h), 300ºC (4h) and 500ºC (4h)</td><td>-</td></tr>
<tr><td>Spacer removal</td><td colspan="2">-</td><td colspan="2">Immersion in water at rest, at 45-55ºC: 4 cycles of 4h, and finally drying at 100ºC (1.5h)</td><td>60ºC (10h), 110ºC (12h), both stages at 10^{-2} bar</td></tr>
</table>

Sintering	1250ºC during 2h; high vacuum ≈10^{-5} bar

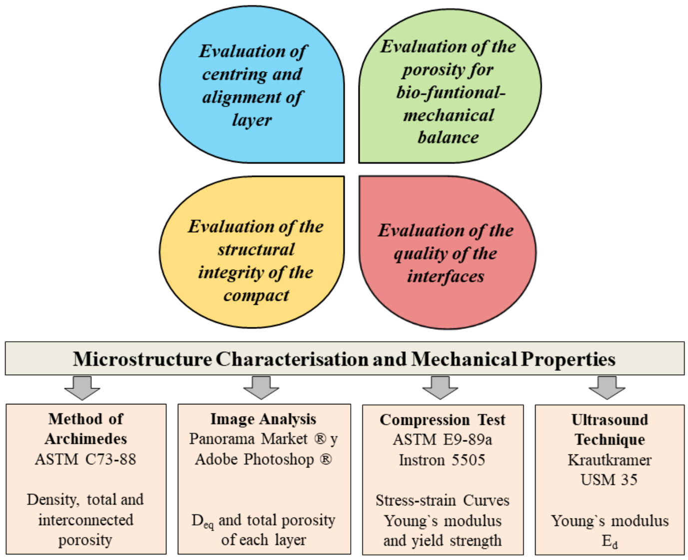


Figure 2. Diagram of the methodology used.

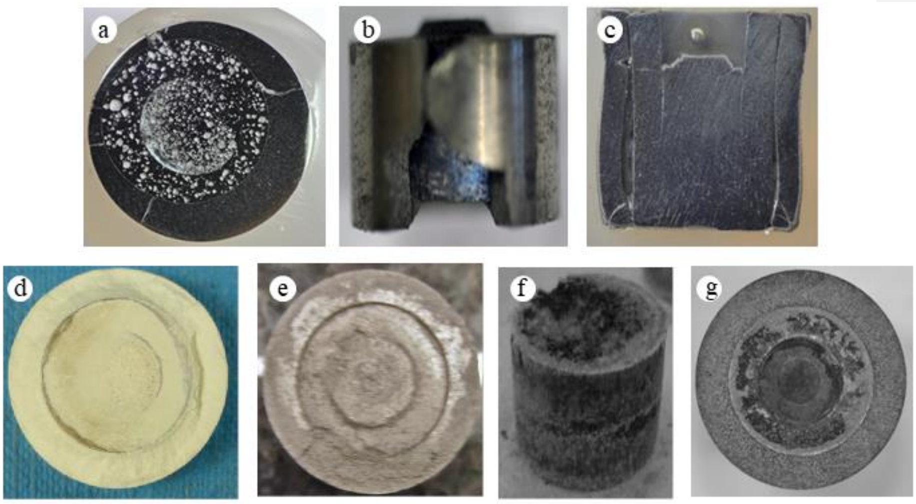


Figure 3. Images of defective pieces, due to the pressing stage and elimination of the lubricant and the spacer, and an image of a correct piece produced with the application of the optimised conditions for the sequential compaction process (three concentric zones designed with a radial graded porosity structure). Titanium cylinders with: a) displaced core; b) break in the intermediate layer in the green body; c) lack of adhesion between layers after sintering; d), e) and f) with loss of material after removal of both lubricant (EBS) and the spacer from the green body (ammonium bicarbonate or NaCl); and, g) optimal structural integrity and concentricity of the layers.

Table 3. Lubricant (EBS), $NH_4(HCO_3)$ and NaCl space-holder removal protocols used.

	Incorrect Removal Protocol	**Correct Removal Protocol**
EBS	500ºC (1h)	100ºC (2h), 300ºC (4h) and 500ºC (4h)
$NH_4(HCO_3)$	110ºC (2h)	60ºC (10h), 110ºC (12h), both stages at 10^{-2} bar
NaCl	Immersion in water with moderate agitation, at room temperature (2h).	Immersion in water at rest, at 45-55ºC: 4 cycles of 4h, and finally dried at 100ºC (1.5h)

Figure 4. Macro- and micrographs of the longitudinal section in the designs manufactured by conventional PM with increasing and decreasing gradients.

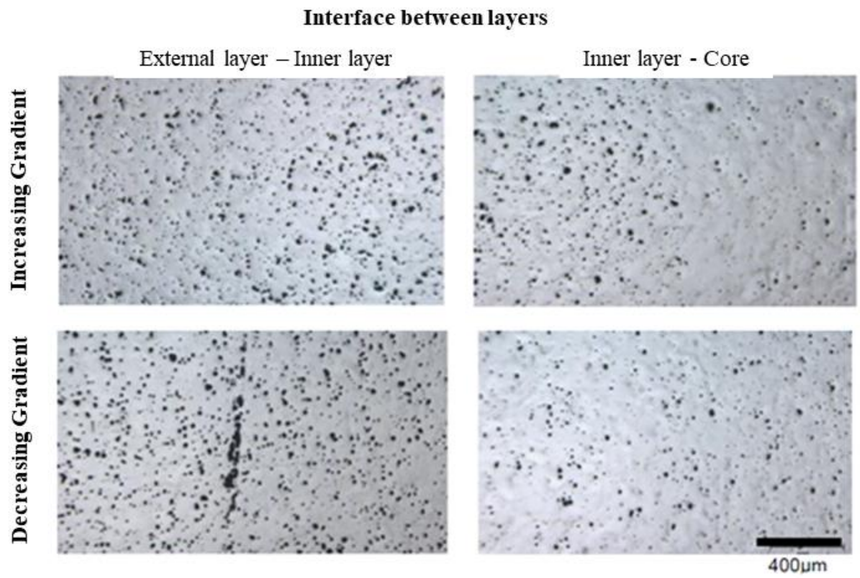


Figure 5. Micrographs of the longitudinal section made in each transition zone in the increasing and decreasing designs manufactured by conventional PM: IG and DG, respectively.

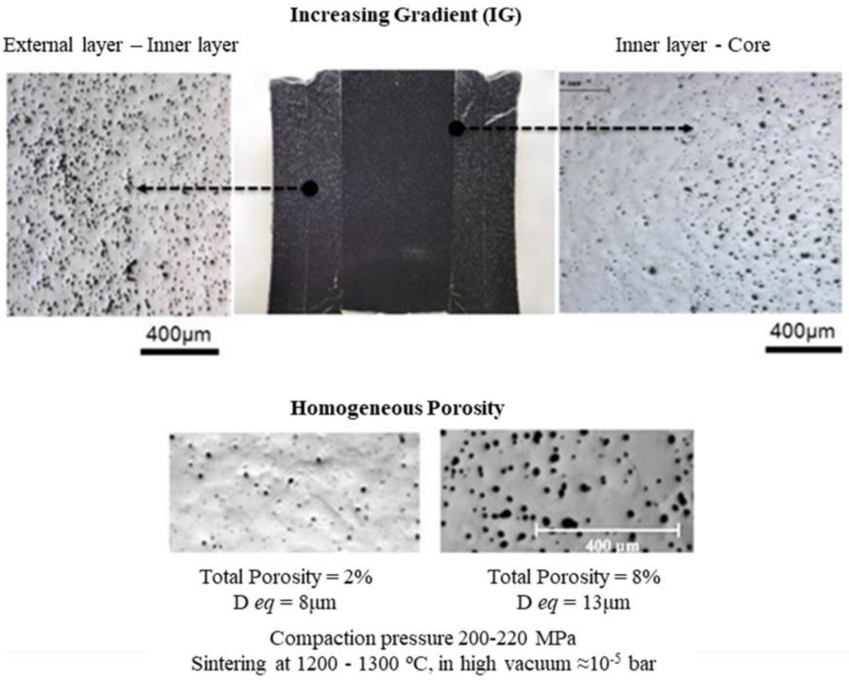


Figure 6. Macro- and micrographs of longitudinal sections made at the interfaces in the IG design and homogeneous porosity cylinders manufactured in similar conditions [128].

Figure 7. Macro- and micrographs of longitudinal sections of Ti cylinders with radial graded porosity obtained by SHT: HG design obtained by NaCl (D [4,3] = 445 µm), and SG designs obtained by NaCl (D [4,3] = 206 µm] and $NH_4(HCO_3)$ (D [4,3] = 265 µm).

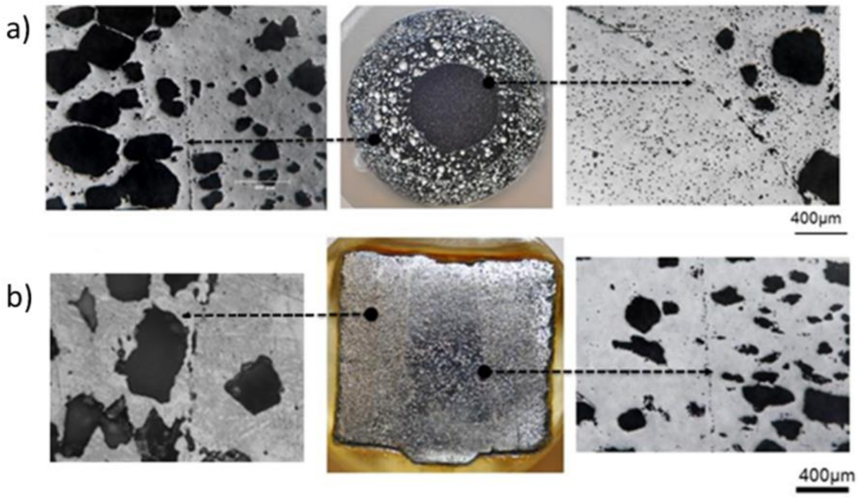


Figure 8. Macro- and micrographs of the Ti cylinder with radial graded porosity obtained by SHT: a) upper base of the HG design obtained by NaCl (D [4,3] = 445 µm)), b) longitudinal section of the SG design obtained by NaCl (D [4,3] = 206 µm).

Table 4. Density, total porosity, interconnected porosity and equivalent pore diameter, by Archimedes' method and image analysis of Ti cylinders with radially graded porosity fabricated according to the SG design by space-holder technique (SHT).

Table 5. Young's modulus and yield strength obtained by uniaxial compression testing and ultrasound technique of Ti cylinders with radial graded porosity fabricated according to the SG design by space-holder technique.

	Ti cylinders, SG design (SHT with 20/40/60 vol. %)			
	Ultrasound		Uniaxial compression test	
	E_d (GPa)	E_d (GPa)	E_c (GPa) - includes machine rigidity	σ_y (MPa)

<table>
<tr><th></th><th>NaCl</th><th>$NH_4(HCO_3)$</th><th>NaCl</th><th>$NH_4(HCO_3)$</th><th>NaCl</th><th>$NH_4(HCO_3)$</th><th>NaCl</th><th>$NH_4(HCO_3)$</th></tr>
<tr><td>Core</td><td rowspan="3">26.0 ± 1.4</td><td rowspan="3">32.3 ± 1.7</td><td>77.5 ± 1.2</td><td>70.2 ± 1.1</td><td rowspan="3">8.3 ± 1.9</td><td rowspan="3">11.5 ± 2.6</td><td rowspan="3">278 ± 18</td><td rowspan="3">312 ± 19</td></tr>
<tr><td>Inner</td><td>31.5 ± 1.4</td><td>51.6 ± 2.3</td></tr>
<tr><td>External</td><td>11.5 ± 1.4</td><td>19.5 ± 2.3</td></tr>
</table>

Figure 10. Stress-strain curves of the Ti cylinder with the SG design: influence of the type of spacer [NaCl vs NH_4 (HCO_3)].

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