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A simple and economical device to process Ti cylinders with elongated porosity by freeze-casting techniques: design and manufacturing

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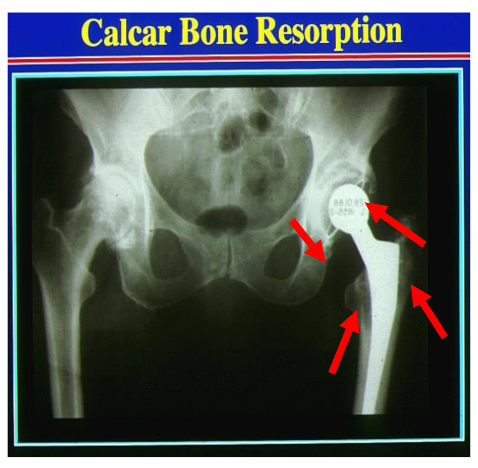
Introduction and Objectives

Disadvantages of Titanium for bone tissue replacement:

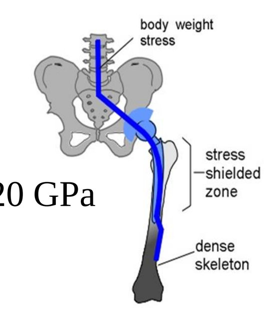
STRESS-SHIELDING PHENOMENON



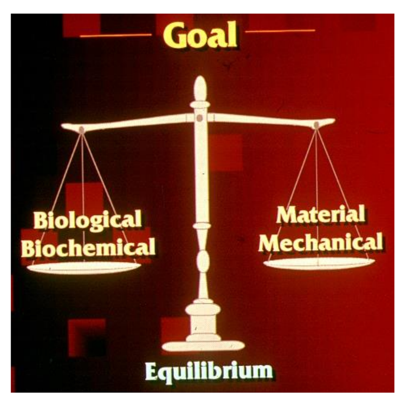
Mechanical incompatibility with the bone: due to big difference in Young´s modulus

E (Ti) ~ 110 GPa

E (Cortical bone) ~ 20 GPa



What is the aim?



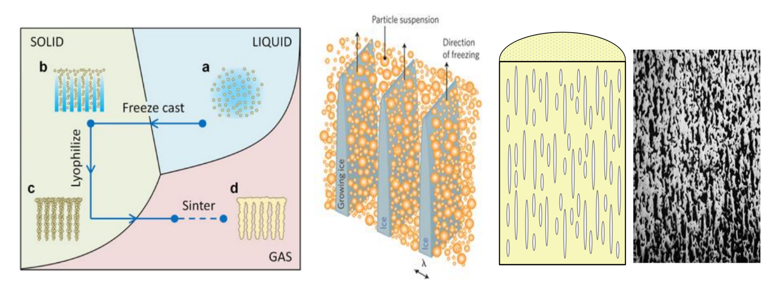
One possible solution: porous titanium manufactured by different techniques

Objective:

Design, manufacture and validation of a simple and economic device that allows producing Ti cylinders with directed porosity applying the freeze-casting technique, and the study of the influence on the internal structure of the Ti porous samples when different materials are used for the vessel (alumina or Teflon).

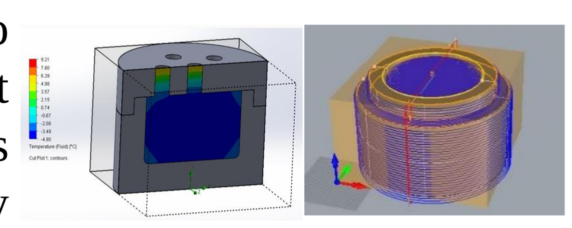
Materials and Experimental Procedure

Freeze-casting technique allows the generation of elongated pores oriented to the direction of the thermal gradient, that is generated during freezing. The process starts with the production of a slurry which is poured into a vessel. It directionally solidifies when a temperature gradient is applied. The growth of the ice dendrites produces the rejection of the metallic particles, generating alternating regions of solidified liquid and regions of compacted particles.



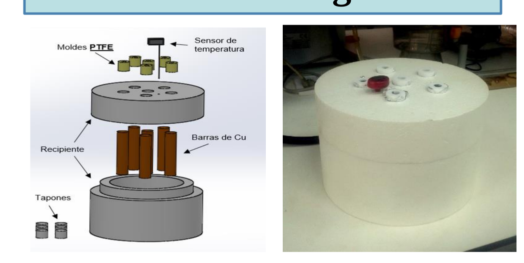
The frozen liquid dendrites are then removed by sublimation, and an elongated pore is generated instead. Finally, the green sample must be sintered under an appropriate atmosphere.

1. Design device



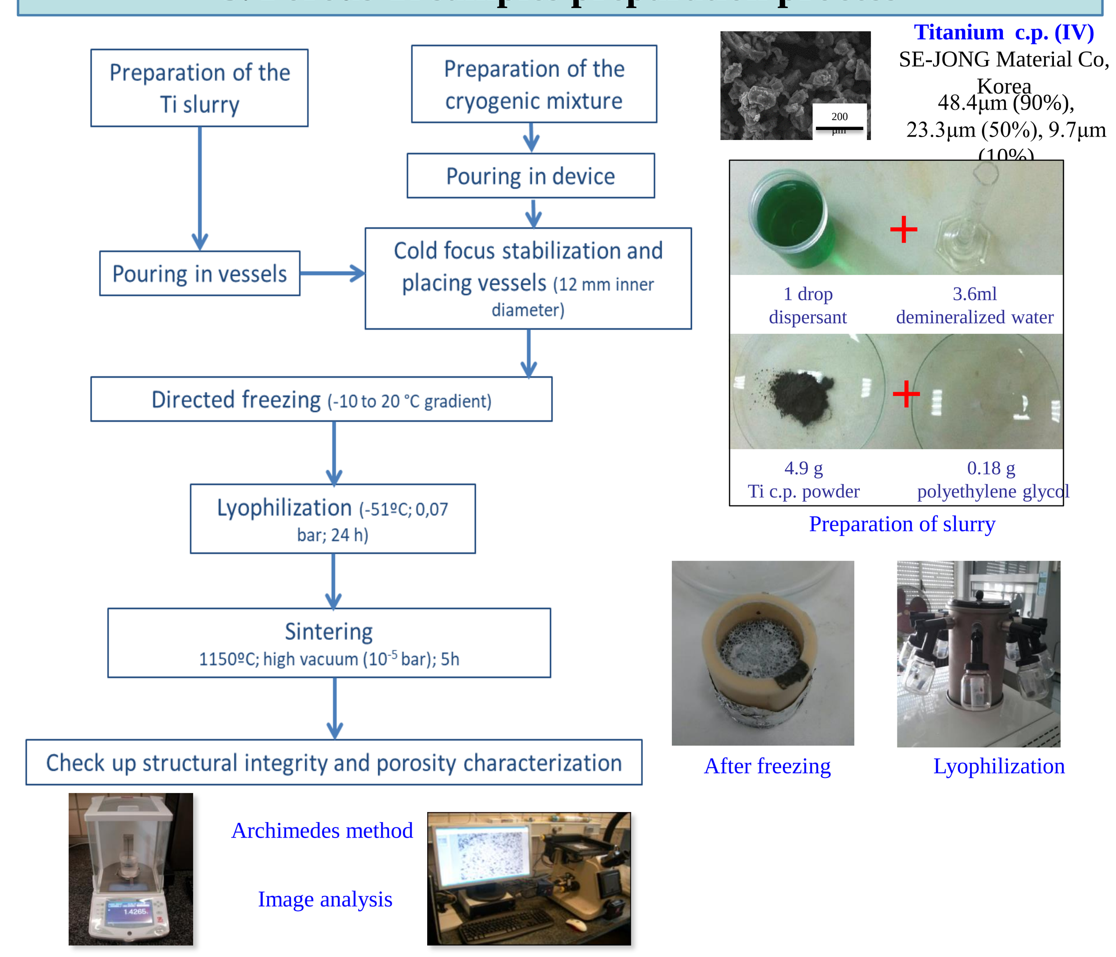
Simulation of the temperature distribution in the cryogenic mixture

2. Manufacturing device



Device already manufactured and assembled

3. Porous Ti samples preparation process



Results and Discussion

Archimedes method ASTM Standard - C373-14 (2014)

Vessel material	Porosity (%) Total, P_T	Interconnected, P_i
Alumina	38.3	37.2
Teflon	38.53	38.44

- ✓ A first global analysis of the total porosity reveals that these values are suitable to solve the stress shielding (~ 40%).
- ✓ According to the overall and interconnected porosity values, the vessel material does not influence them.

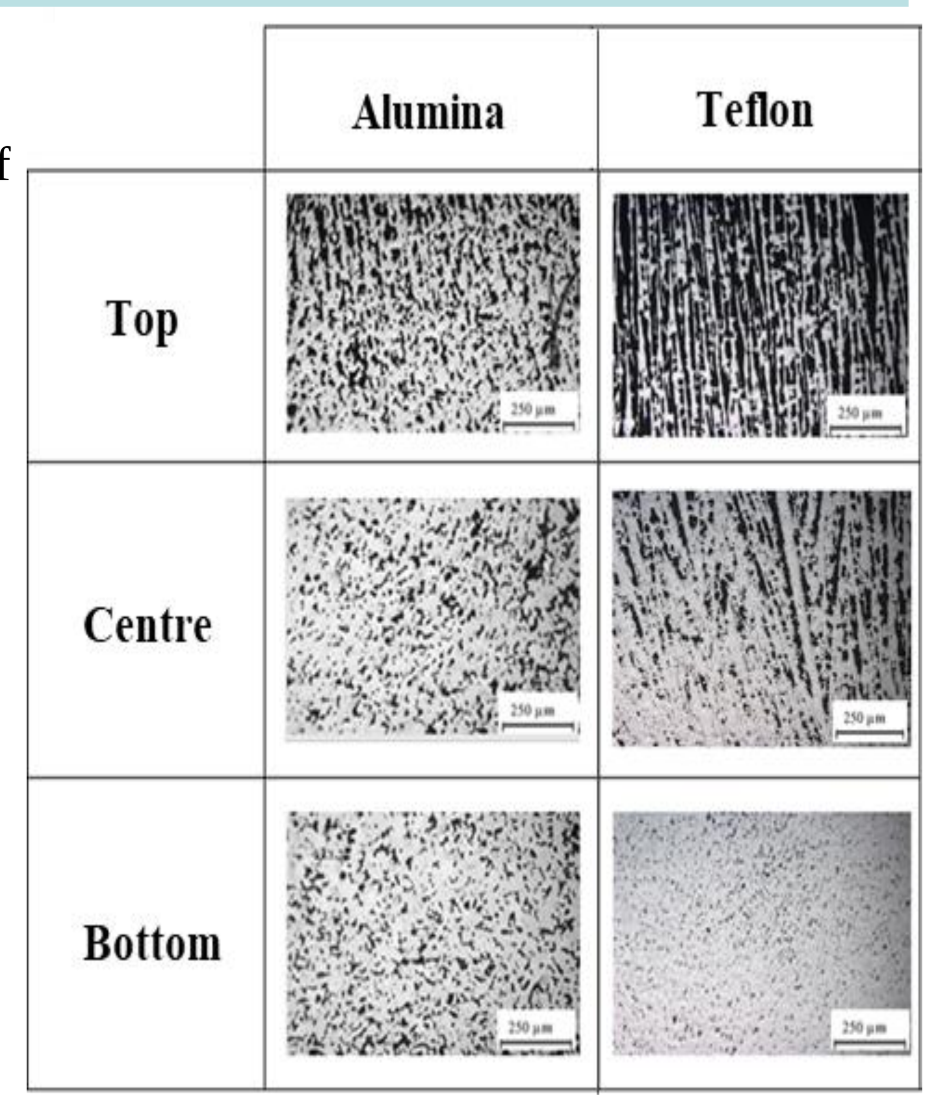
Image Analysis

Vessel material	Zone	Total Porosity (%)	$Ø_{min}$ (µm) (pore width)	$Ø_{max}$ (µm) (pore length)	Elongation factor $Ø_{min}$ / $Ø_{max}$ of equivalent ellipse
Alumina	Top	38.6	15.7	45.2	0.35
	Centre	18.2	13.3	33.4	0.40
	Bottom	24.2	11.4	32.1	0.36
Teflon	Top	39.5	18.8	123.3	0.15
	Centre	16.4	8.8	21.5	0.54
	Bottom	1.7	5.7	10.1	0.56

- ✓ The porosity changes through the direction of the thermal gradient, being greater in the area closest to the hot focus, where both the freeze front velocity and sedimentation are lower.
- ✓ A higher presence of elongated porosity is observed when used Teflon vessel.
- ✓ Pore interconnectivity and the average pore size obtained with Teflon vessels, could be guarantee the ingrowth of the bone tissue (pores ≥100µm) of implants manufactured by this technique.

Images of three zones through longitudinal cross section of the Ti cylinders

The role of the particles sedimentation should be also considered to explain the presence of the three differentiated zones.



Alumina vessel:

- ✓ Porosity at the bottom and the centre is not oriented in the direction of the thermal gradient.
- ✓ At the top it is clearly elongated along that direction.
- ✓ It is related to engulfment of the titanium particles by a fast growth of the ice dendrites during the solidification process.

Teflon vessel:

- ✓ The presence of elongated porosity is much higher..

The elongated porosity appears both at the top and the centre due to a better thermal insulation (Teflon has a thermal conductivity of 0.20 W/mK vs alumina 30 W/mK) and the lower speed of the solidification front and thus, longer freezing time (3 hours using Teflon vessels vs 30 minutes using alumina vessels).

Conclusions

- ✓ An economical and simple device has been designed, manufactured and validated.
- ✓ It allows the implementation of the freeze-casting technique to manufacture cylindrical samples with elongated and aligned porosity.
- ✓ Studies have revealed the advantages of using Teflon instead of alumina vessels to reduce the speed of the freezing front and to achieve a better insulation of the slurry and, thus, a higher amount of directed porosity is obtained.
- ✓ Finally, it is significant that the pore size, average porosity and pore interconnectivity in the titanium cylinders are suitable for their potential use in biomedical applications.

Next works

- ✓ Mechanical characterization of the titanium samples.
- ✓ Influence of different particle size on the results.
- ✓ Study of pouring the titanium slurry into mold by layer.

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