β-TCP porous scaffold: part 1. Casting method and structure characterization

member of EMRA

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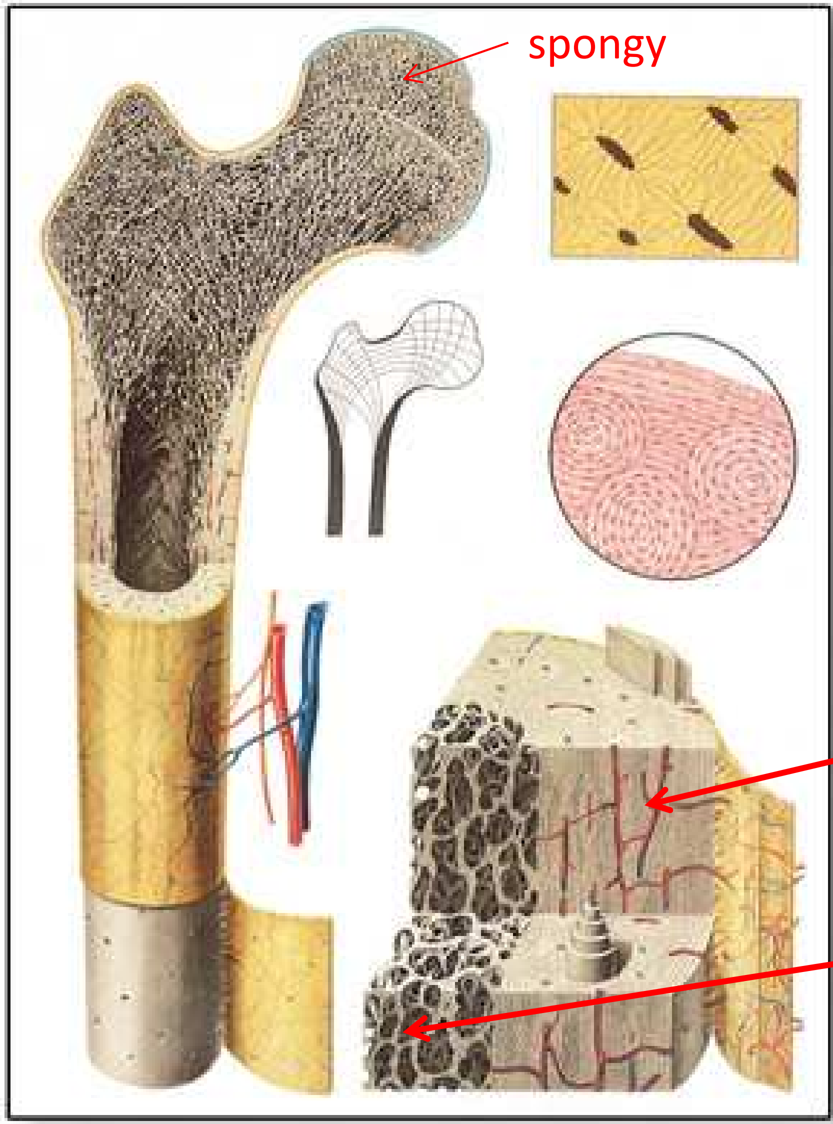


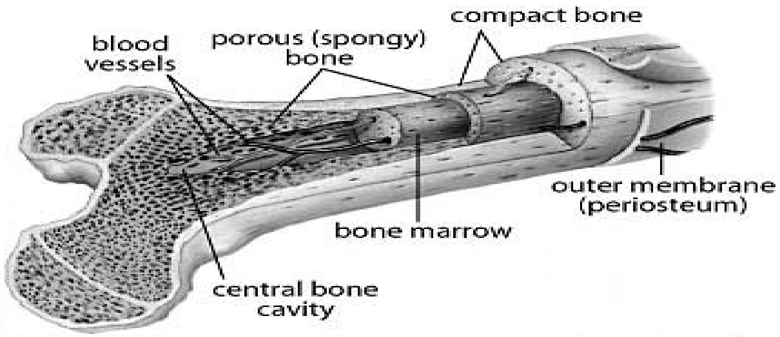
Bone structure

Natural composite of collagen and mineral.

2 types of bone:

- cortical with dense structure → compact bone

-cancellous or trabecular bone with high porosity → spongy bone



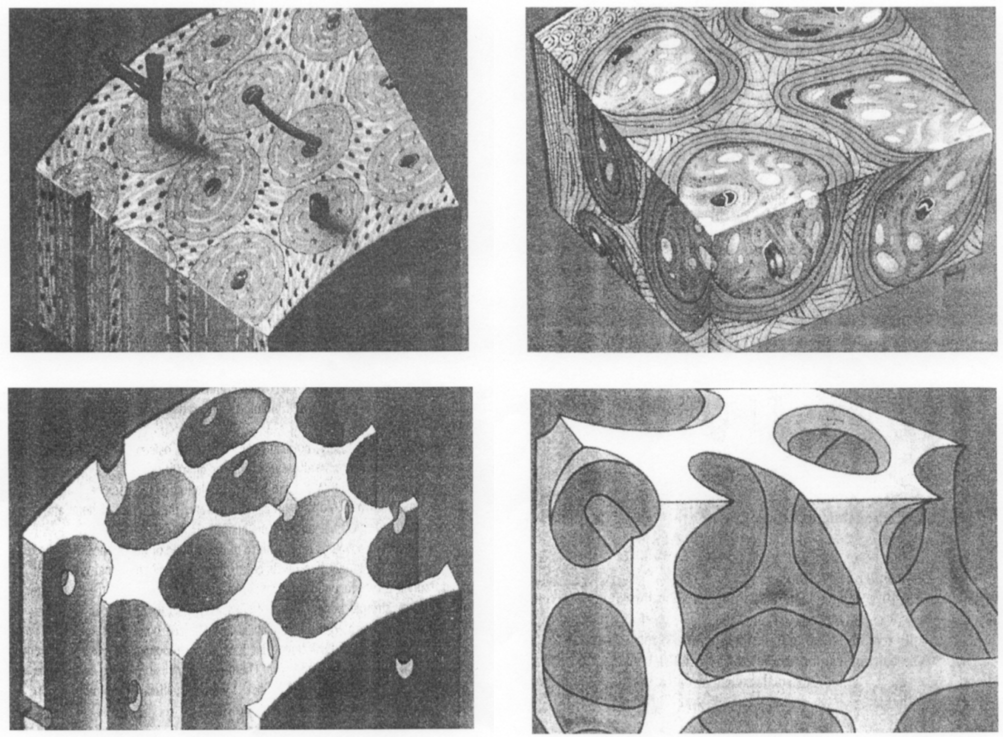
Compact bone

Porosity

Φ : 190-230 µm

%P : 65%

σ_c = 80-200 MPa



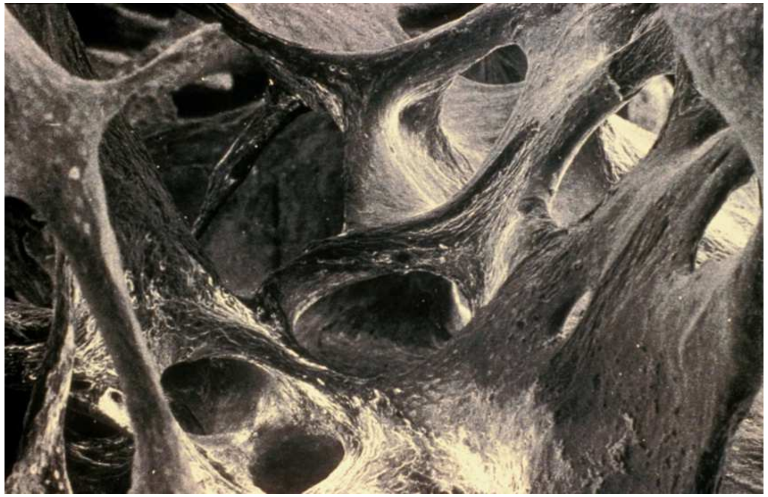
Spongy bone

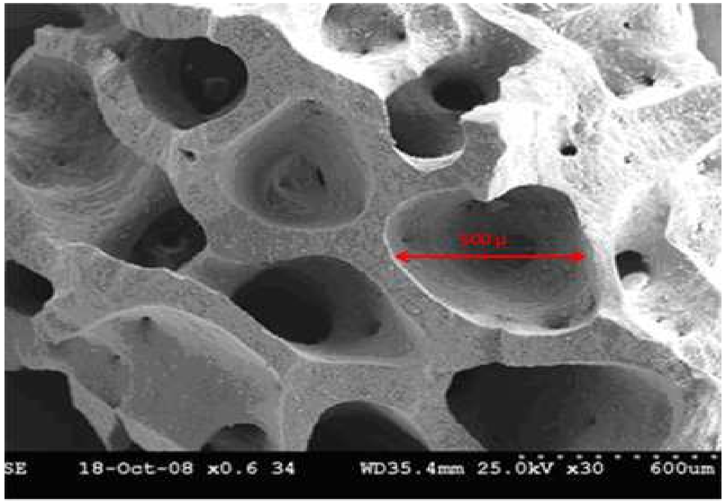
Porosity

Φ : 500-600 µm

%P : 80%

σ_c = a few tenths MPa





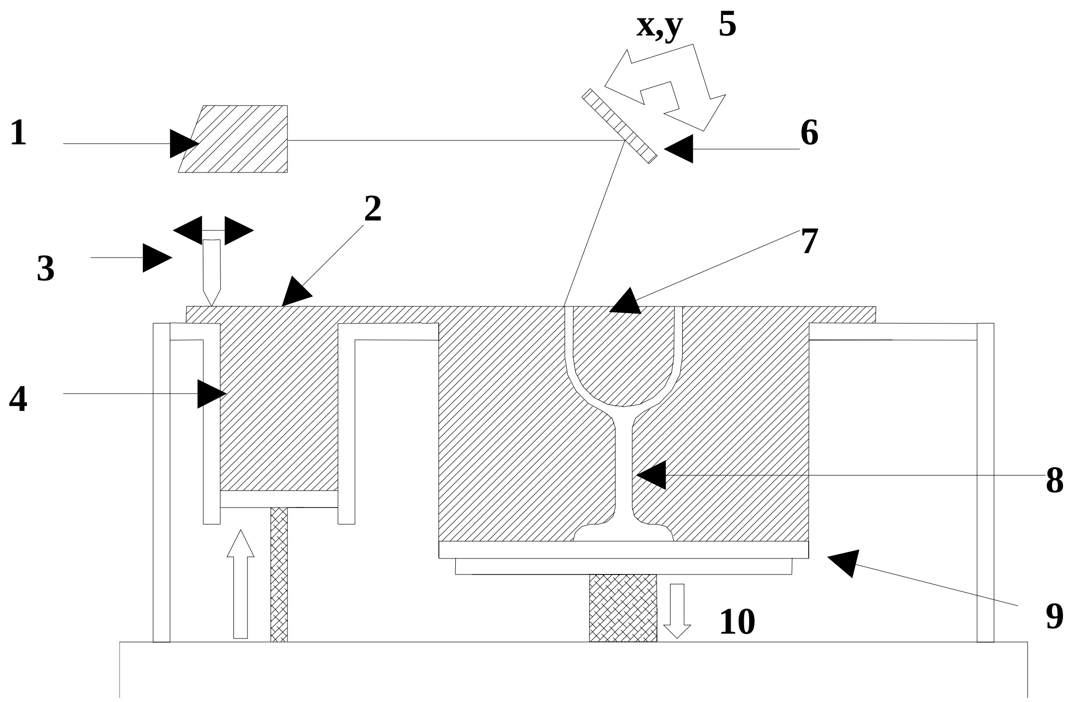
How to mimic natural bone for substitute processing?

Technologies to process porous ceramic :

- Stereolithography
- PMMA templating
- Freeze casting

Mechanical properties

1) Stereolithography



1: laser UV

2: feed zone

3: wiper

4: paste tank

5: rotation XY

6: galvanometric mirror

7: photosensible paste

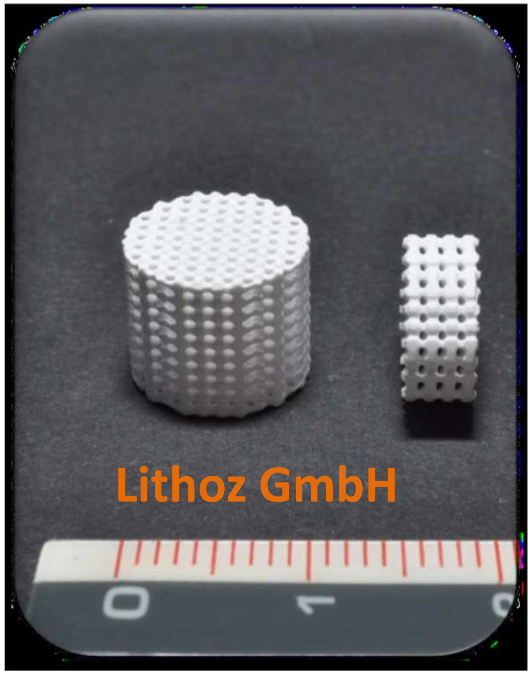
8: polymerised prototype

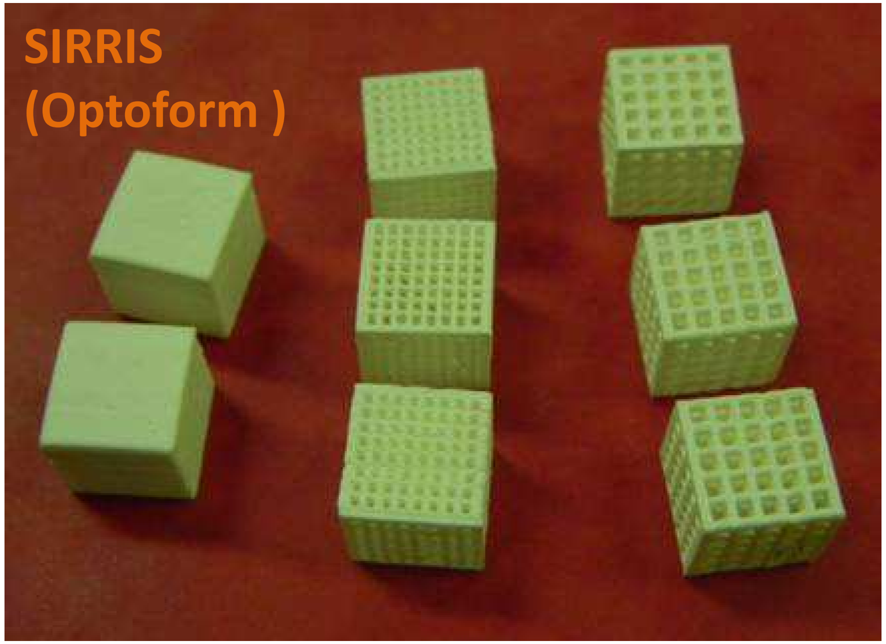
9: manufacturing plate

10: z control

Technologies to process porous ceramic

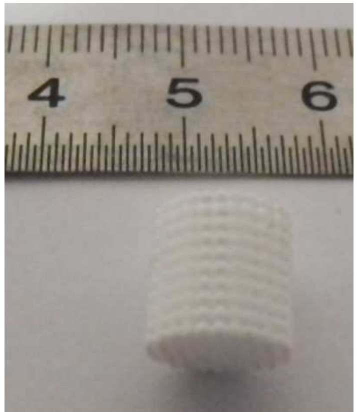
- Manufacturing of parts with complex external and internal shape, with controlled (size and shape) porosity.

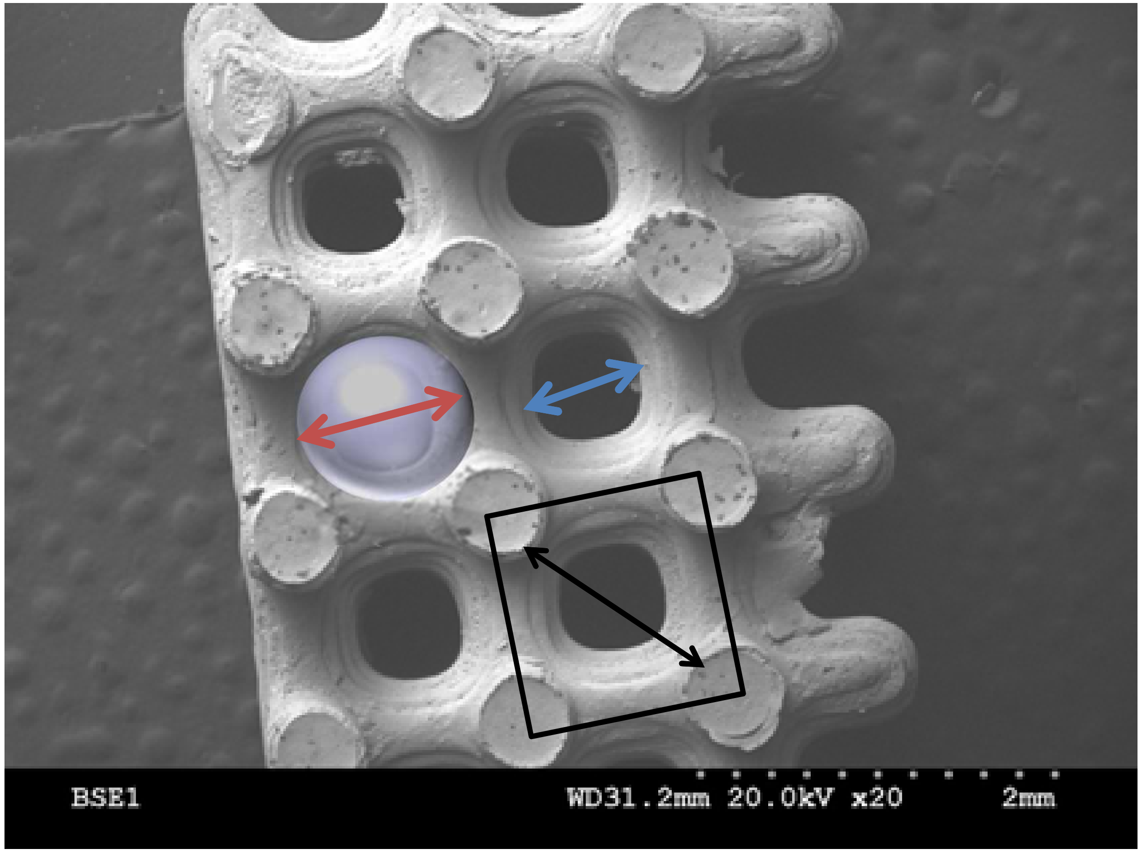




- Debinding and sintering to densify the parts

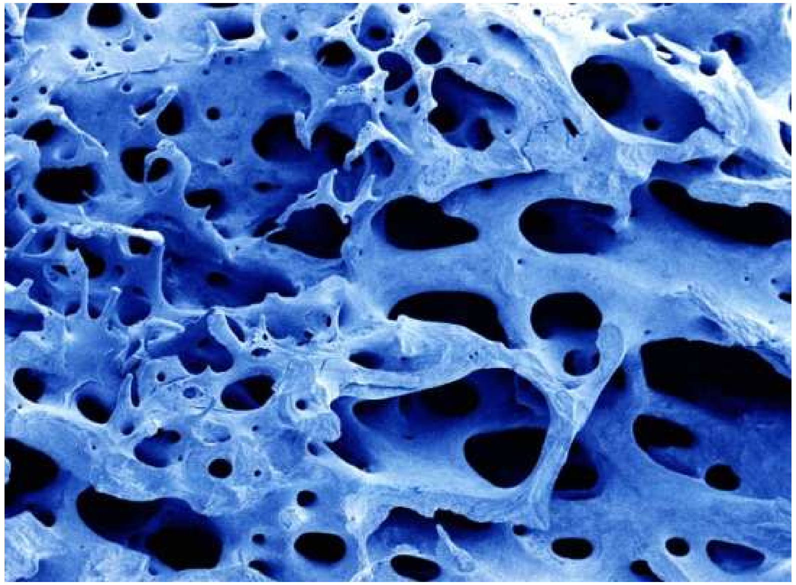
Lithoz GmbH

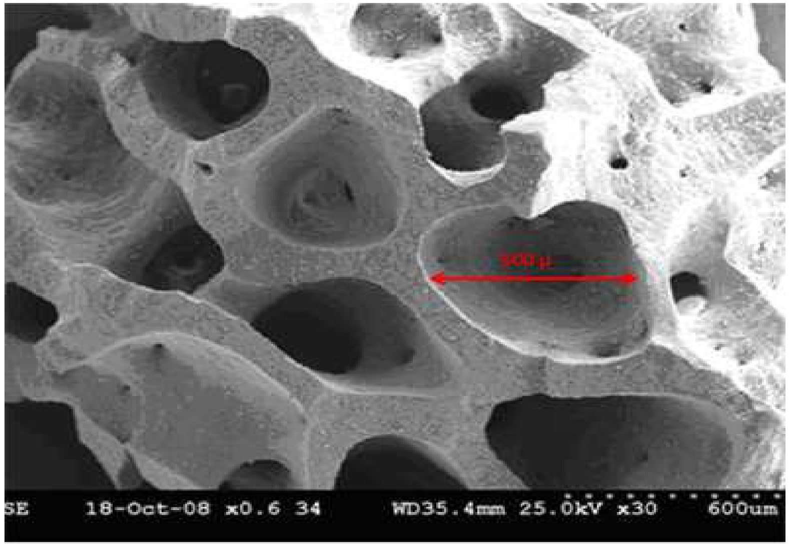




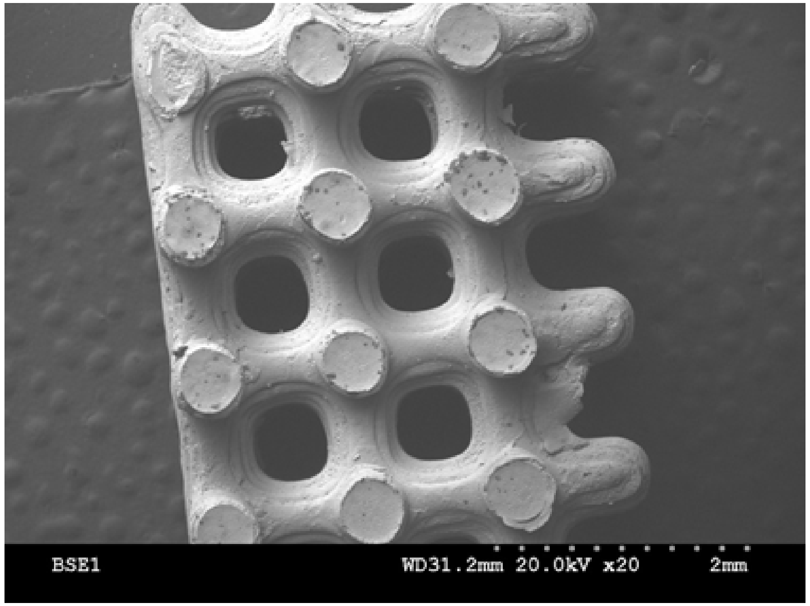
Pore type	interconnection	Spherical	channel
Pore size	400 µm	800 µm	1 mm

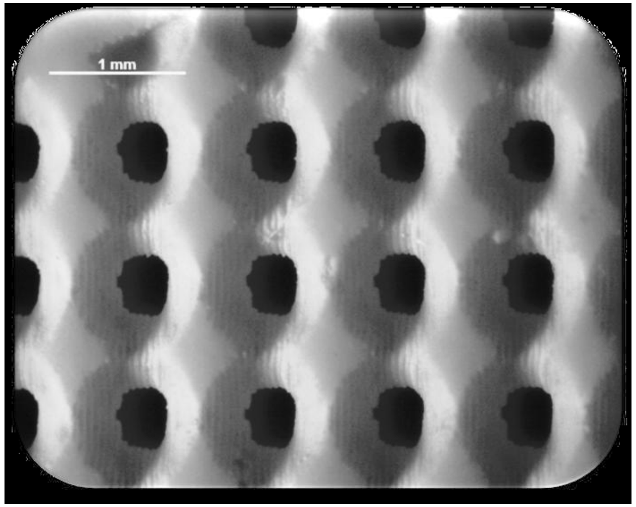
Technologies to process porous ceramic





Human bone



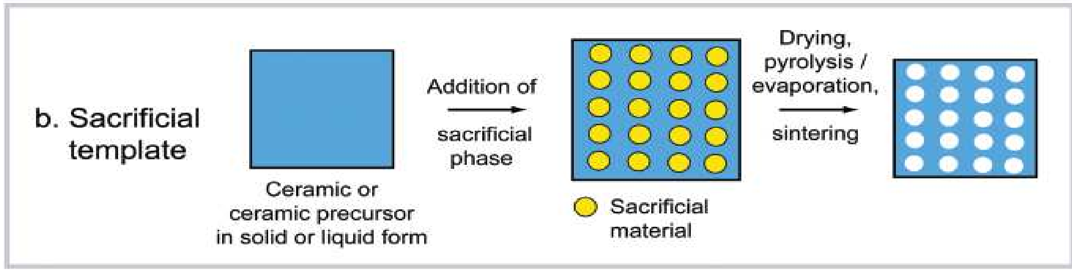


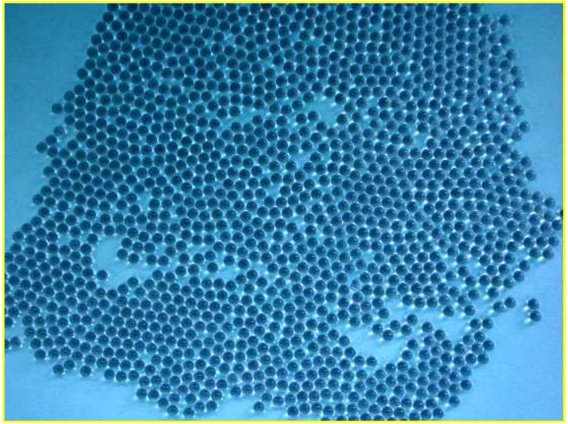
Technologies to process porous ceramic :

- Stereolithography
- **PMMA templating**
- Freeze casting

Mechanical properties

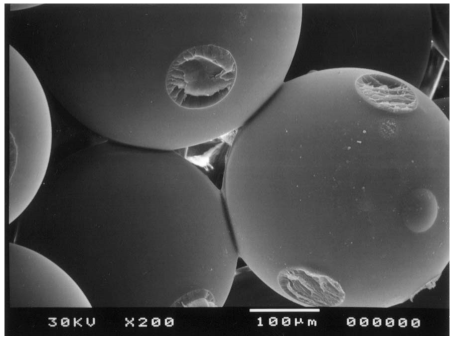
2) Ceramic slurry infiltration of organic bead skeleton





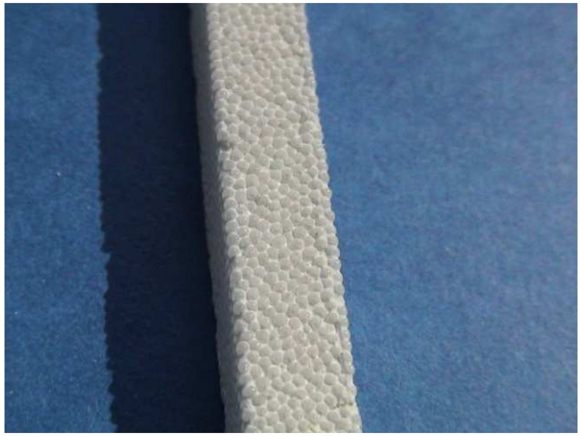
Chemical forming with acetone

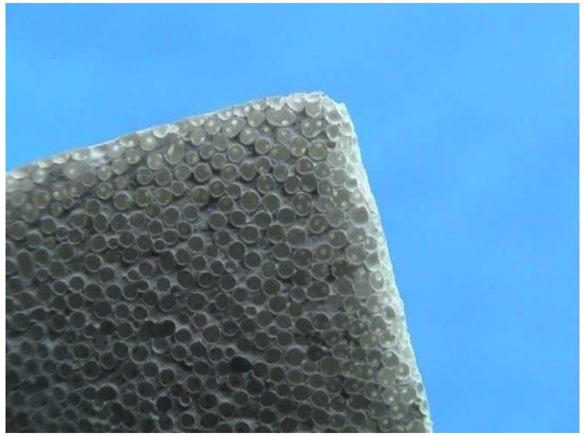
under pressure



- Bonding between PMMA beads (scaffold)
- Controlled diameter bonding (Interconnection) depends on time, temperature, pressure

Technologies to process porous ceramic

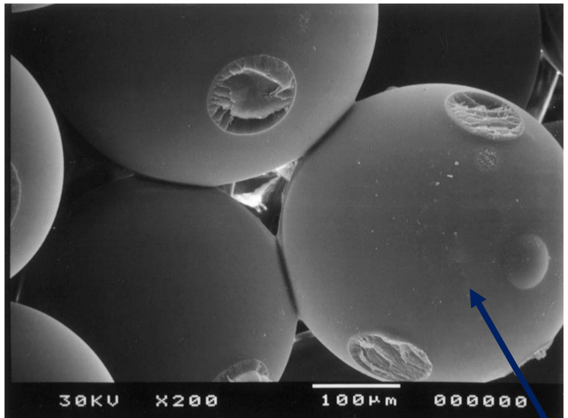


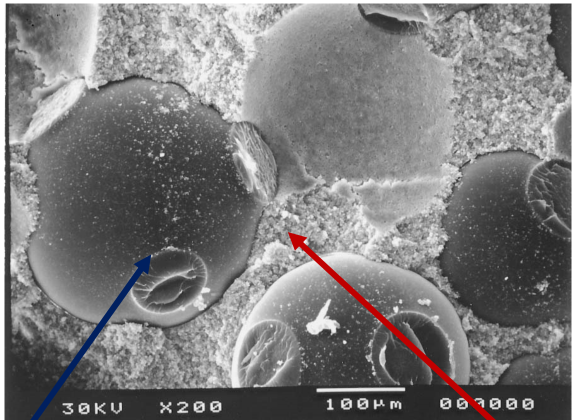


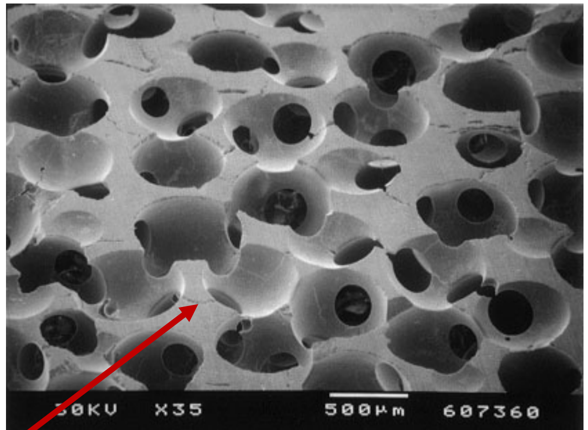
1 **Shaping**

2 **Impregnation**

3 **Debinding**



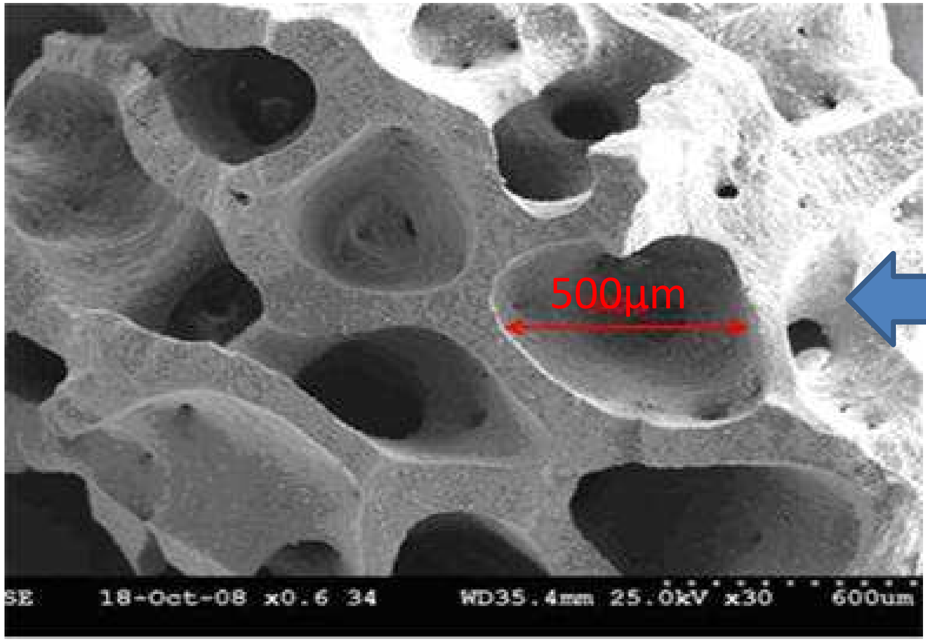


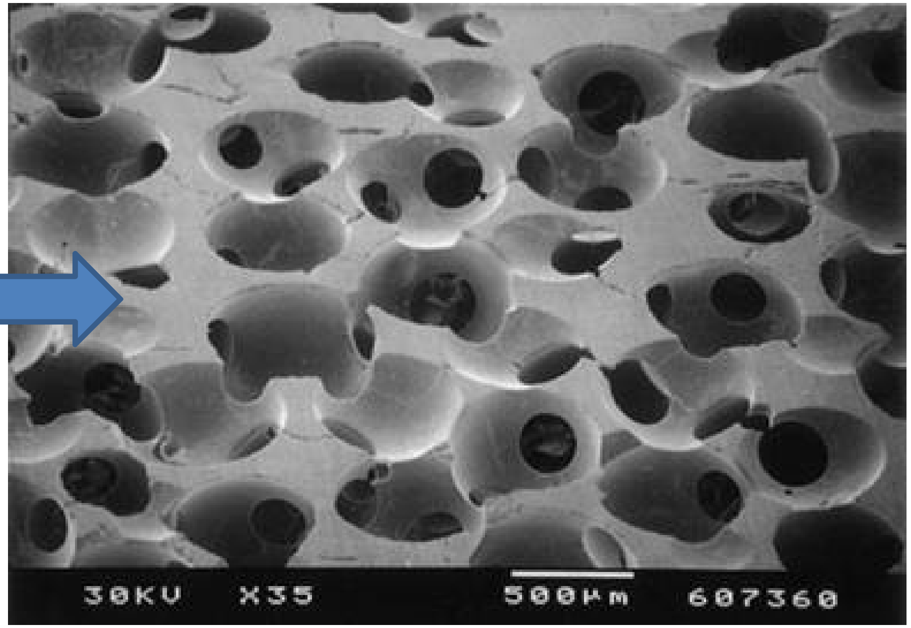


PMMA beads

ceramic

M. Descamps et al., JECS. 28 (2008) 149





Natural bone

β-TCP Scaffolds - initial bead size 500-600µm

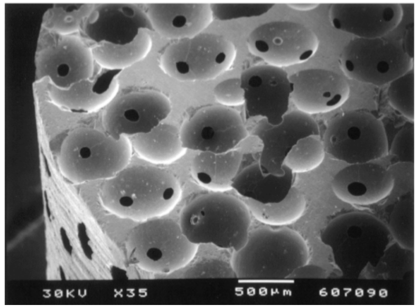
Technologies to process porous ceramic

- Control of pore size depending on PMMA beads size

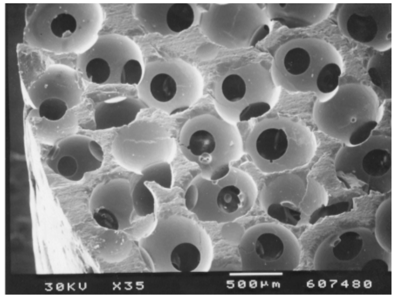
- Control of interconnection diameters: id

PMMA beads (500 - 600 µm)

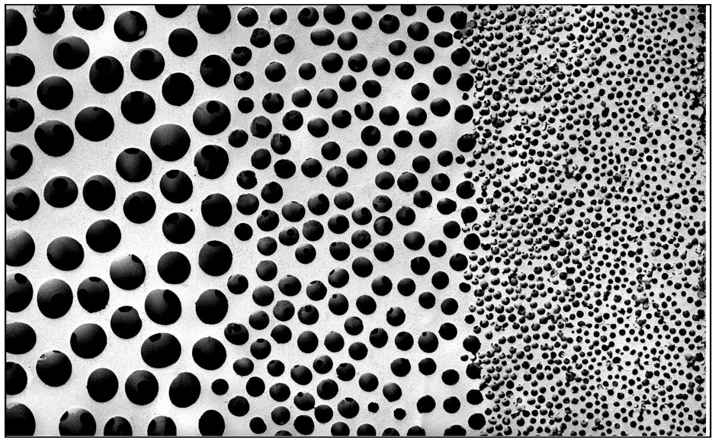
id : 60 µm

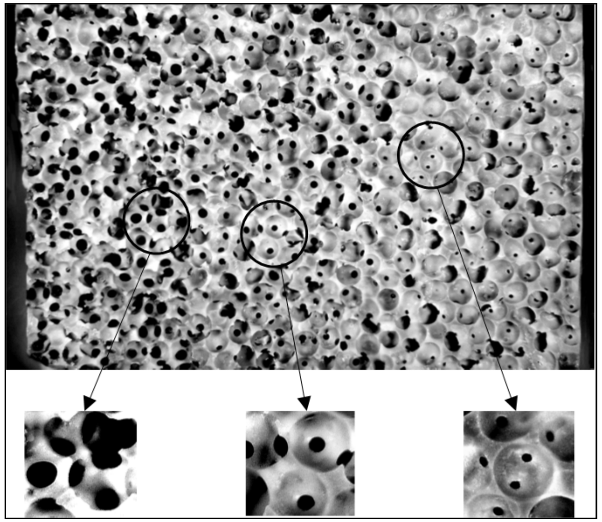


id: 260 µm



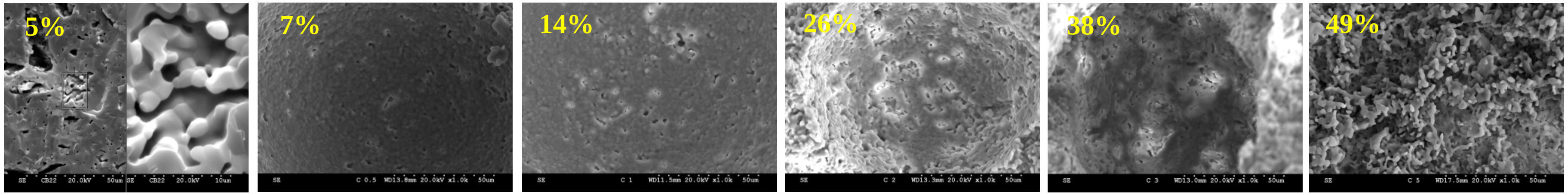
- Control of porosity gradient in pore size and interconnection size

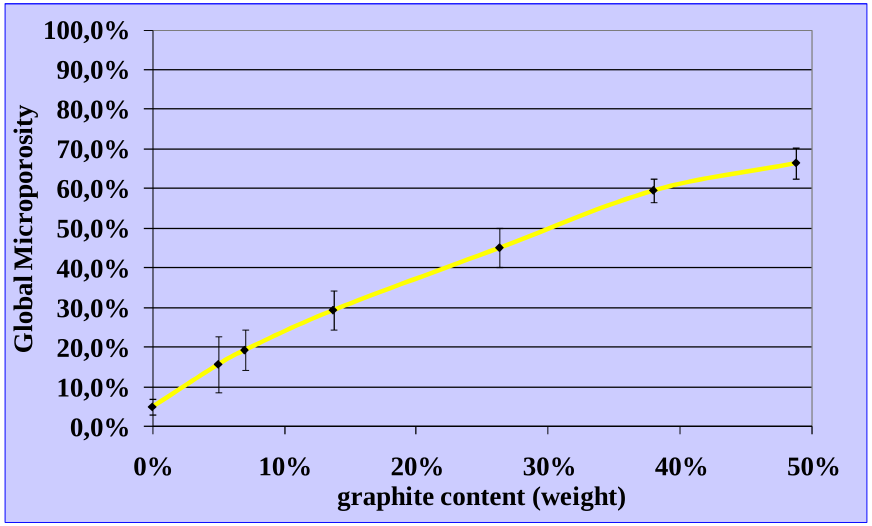


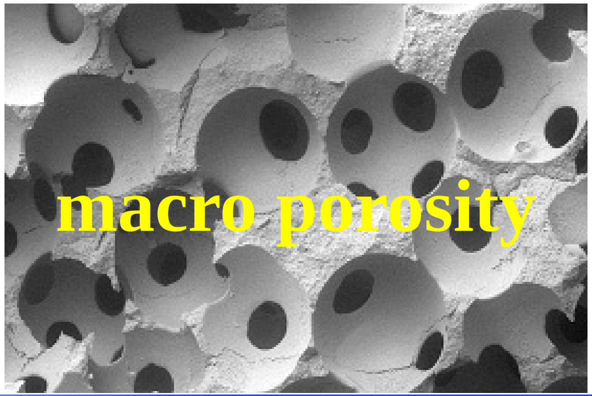


Technologies to process porous ceramic

Possibility to add microporosity by mixing graphite as micropore forming agent.







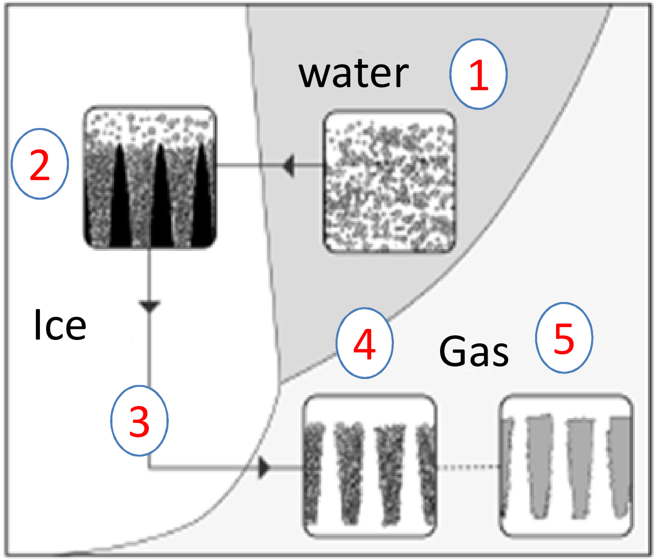
Controlled macro porosity and “micro” porosity

Technologies to process porous ceramic :

- Stereolithography
- PMMA templating
- **Freeze casting**

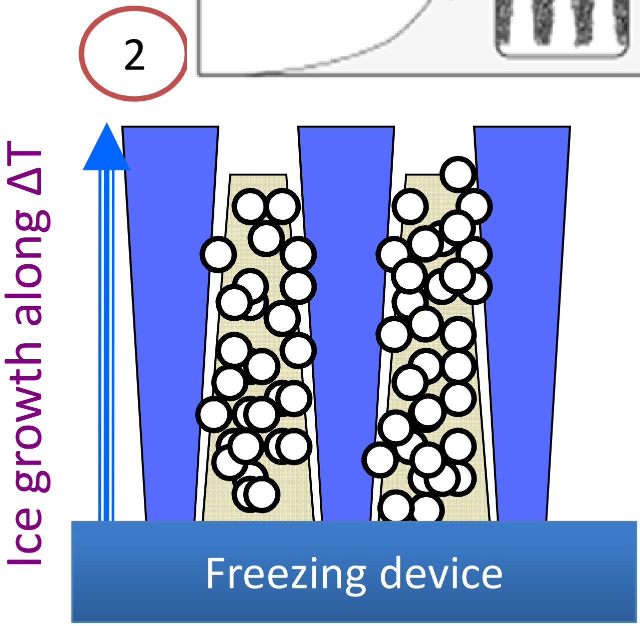
Mechanical properties

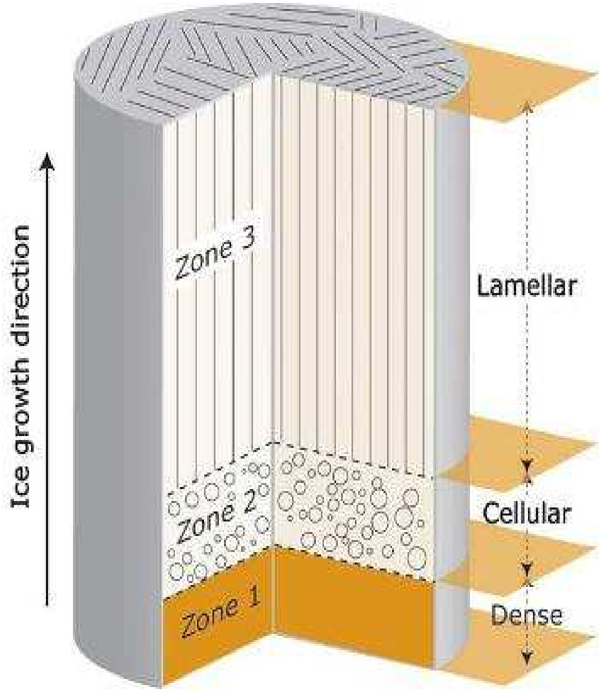
3) Freeze casting by ice-templating



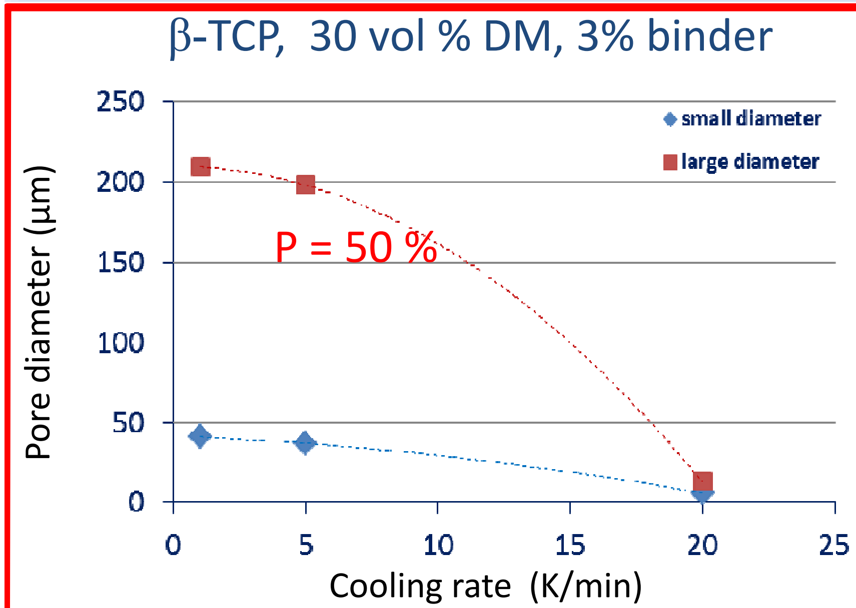
5 steps :

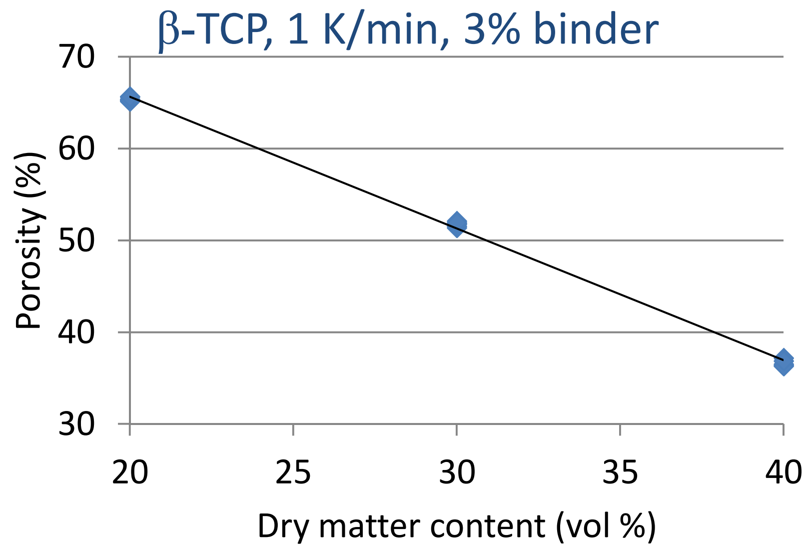
1. Slurry preparation
2. Freezing (anisotropic structure)
3. Demoulding
4. Freeze drying
5. Sintering

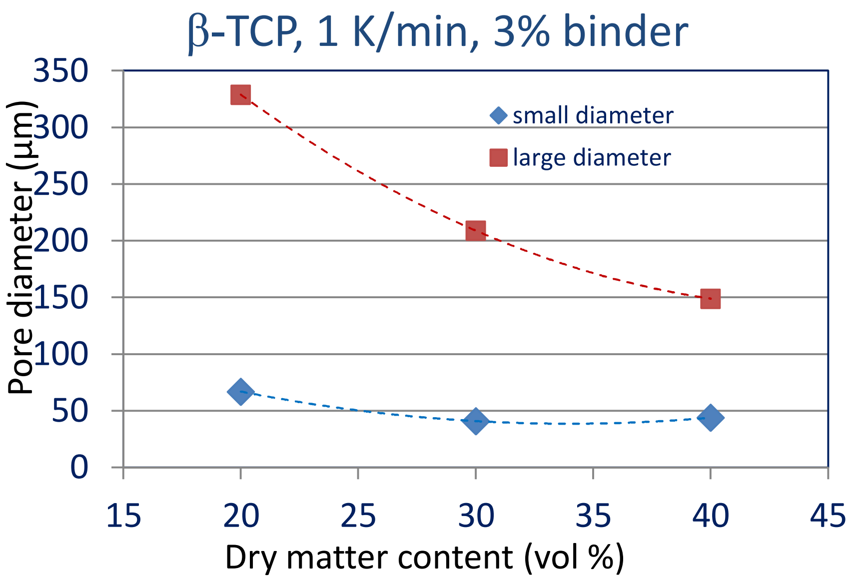




Technologies to process porous ceramic

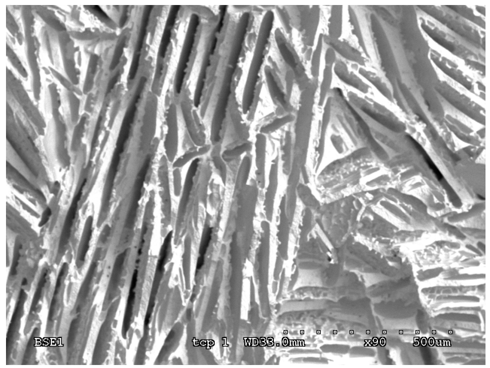


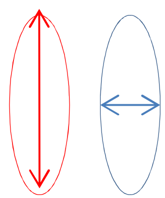




Pore long axis size:

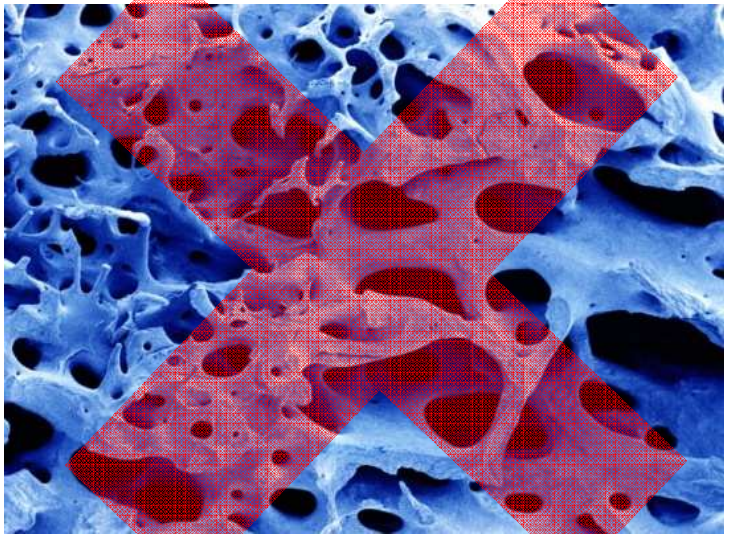
- **between 150 and 340 µm (dry matter content)**
- **between 13 and 210 µm (cooling rate)**

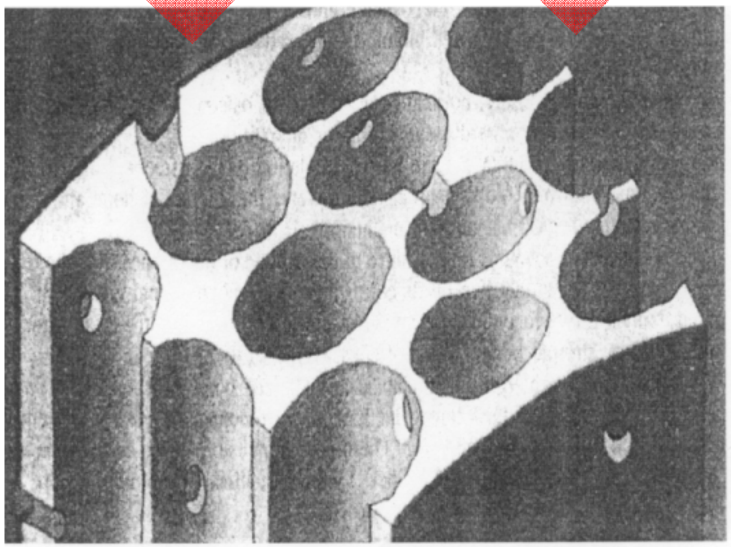




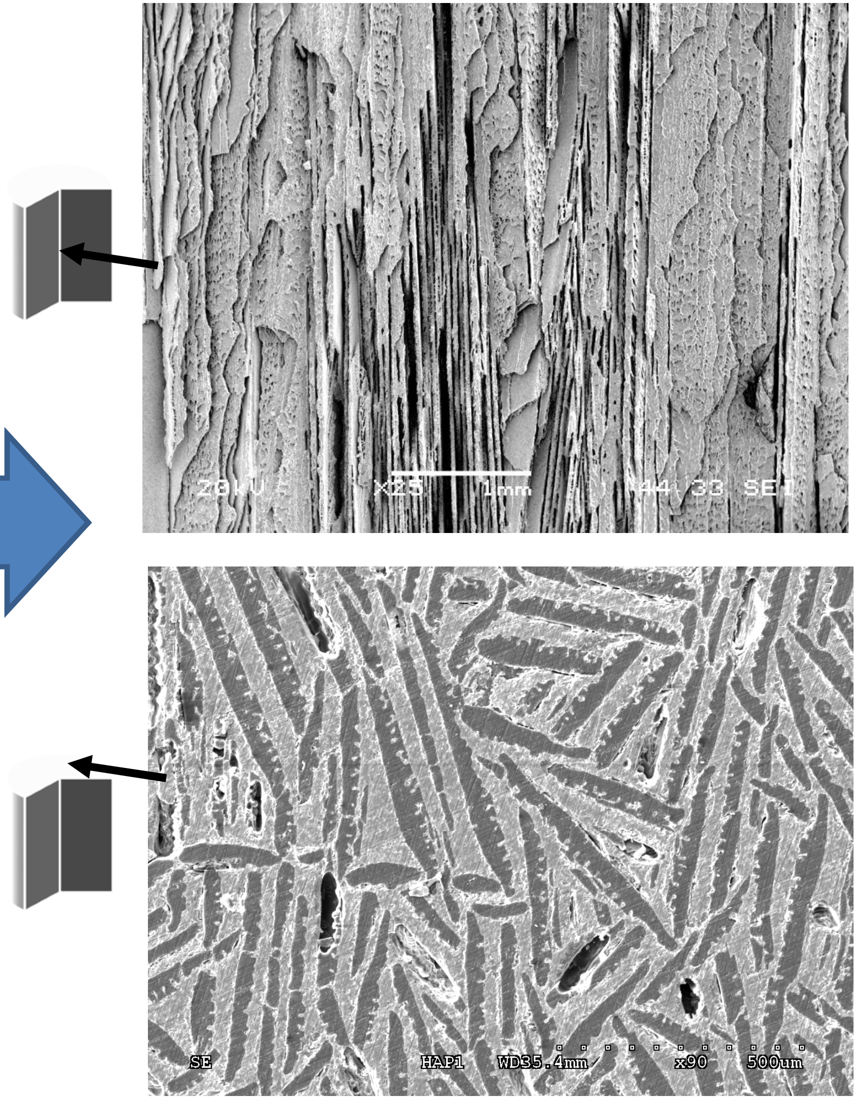
Technologies to process porous ceramic

Spongy bone





Cortical bone



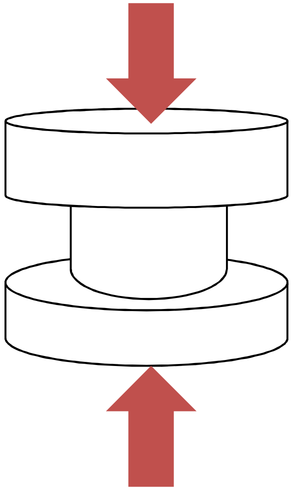
Technologies to process porous ceramic

Techniques	Advantages	Drawbacks
Stereolithography	- Complex designs -High accuracy - Reproducibility	- Expensive photosensitive resins - Cleaning step necessary - Control of the vertical accuracy - Low mechanical properties - Debinding step
PMMA Templating	- Good structure control -Low cost - Easy	- Low mechanical properties -- Complex design - Reproducibility -Debinding step
Freeze casting	- High mechanical properties -Low cost - Good structure control - No debinding step	- Scaling up ? (freeze dryer) -Complex design - Reproducibility

Technologies to process porous ceramic :

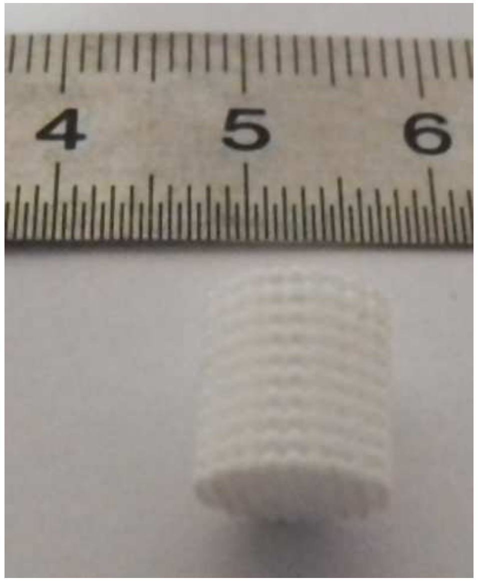
Mechanical properties

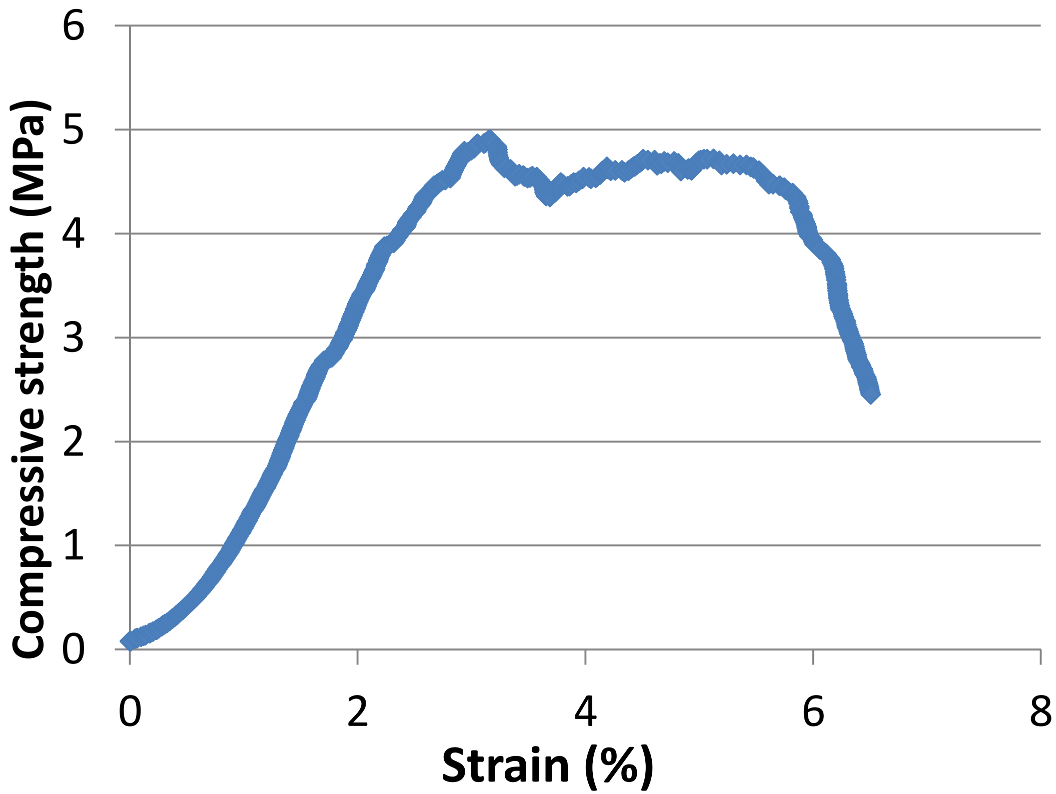
- Experimental: compression test
- 0.5 mm/min and load cell of 10 kN
- Determination of apparent elastic modulus and rupture strength.
- β-TCP sample



Mechanical properties

Stereolithography (from Lithoz)

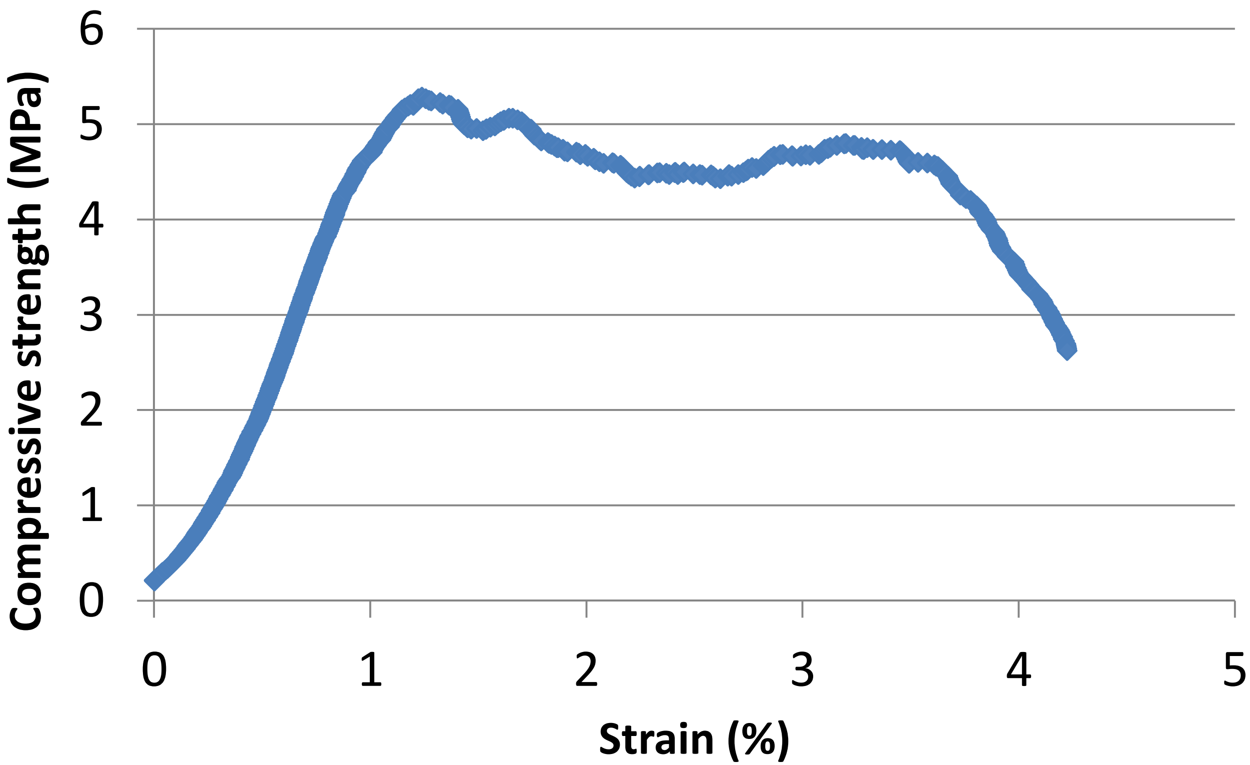




Porosity	Compressive strength	Apparent elastic modulus
45 %	6 MPa	0.4 GPa

Mechanical properties

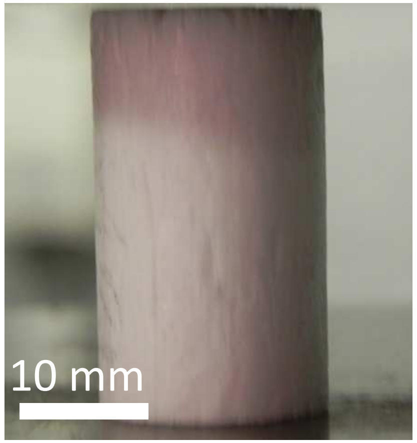
PMMA templating

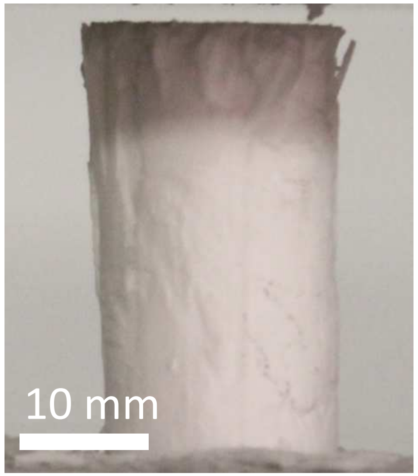


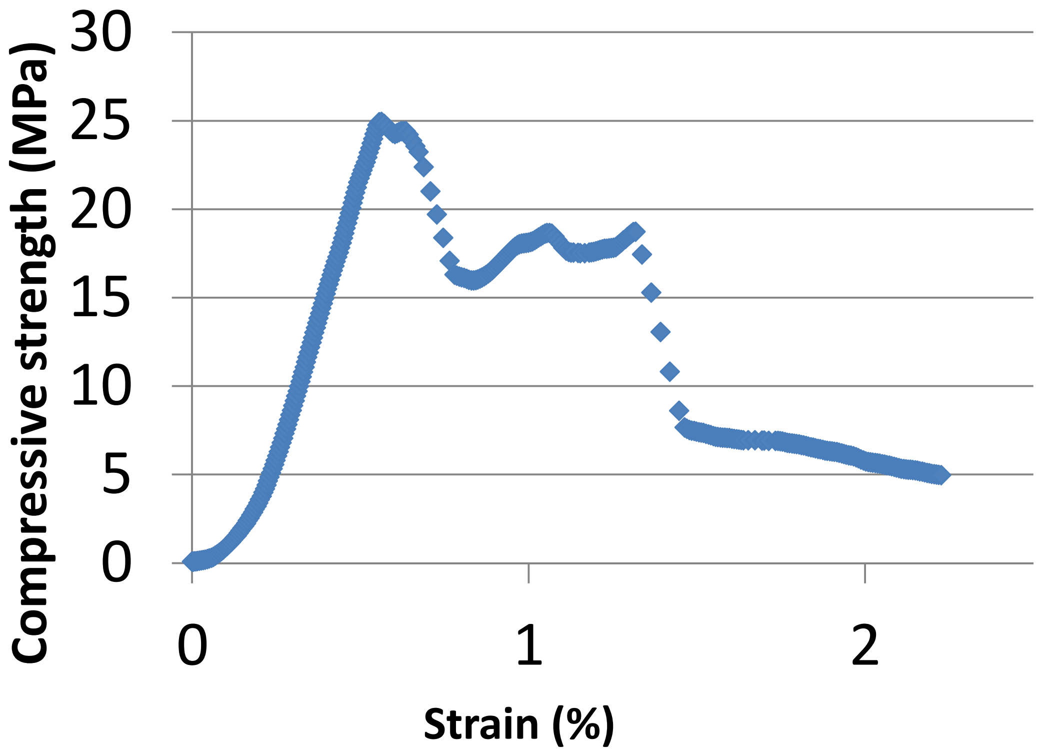
Porosity	Compressive strength	Apparent elastic modulus
65 %	5 MPa	0.6 GPa

Mechanical properties

Freeze casting

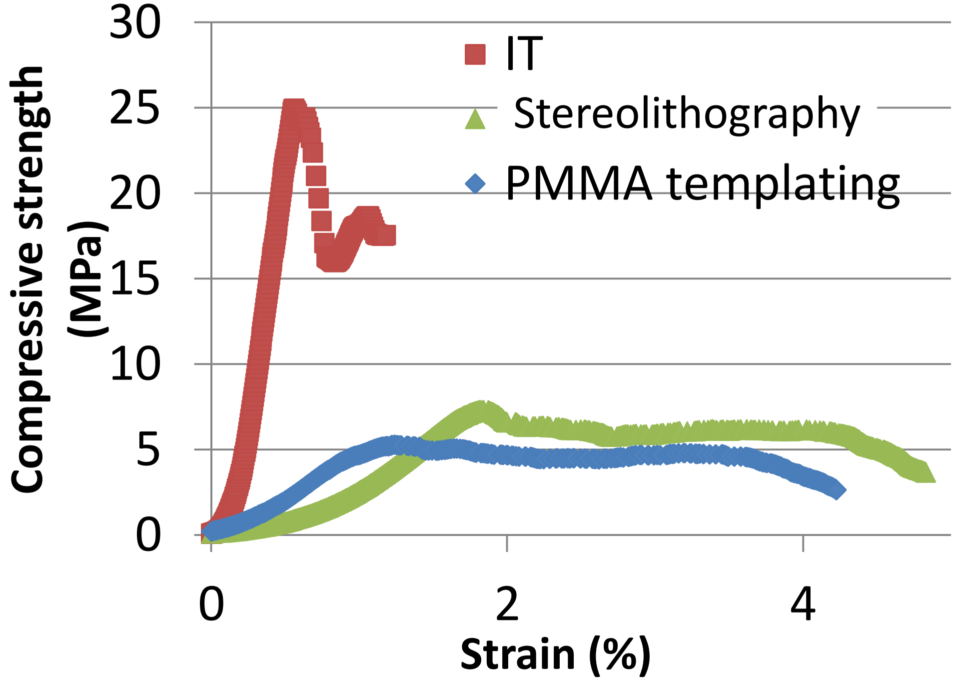


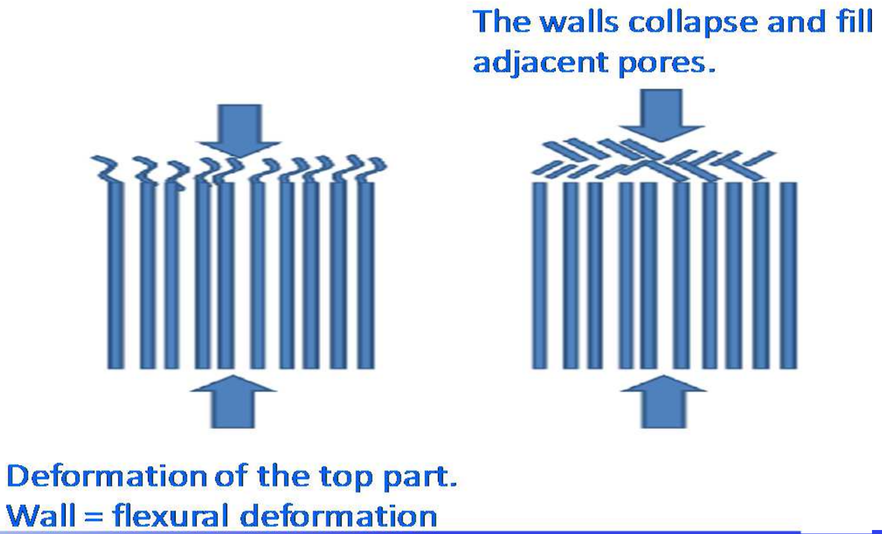


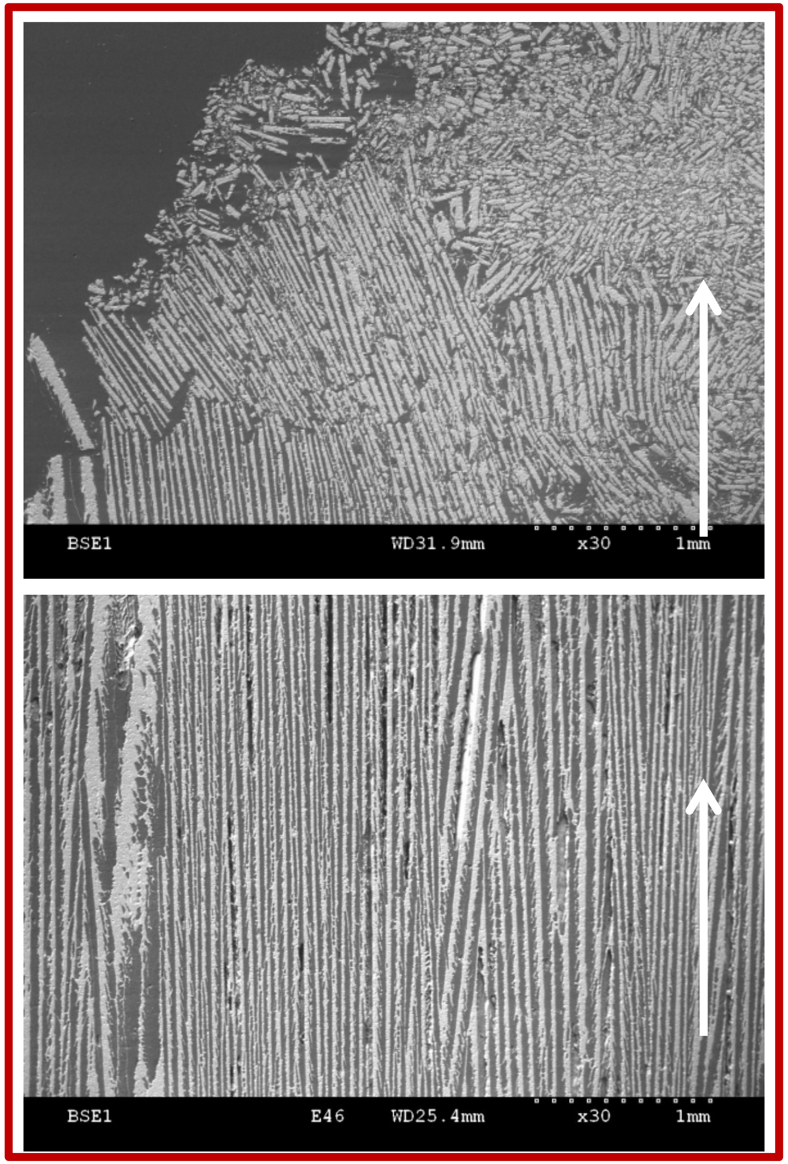


Porosity	Compressive strength	Apparent elastic modulus
50 %	18 MPa	1.5 GPa

Mechanical properties





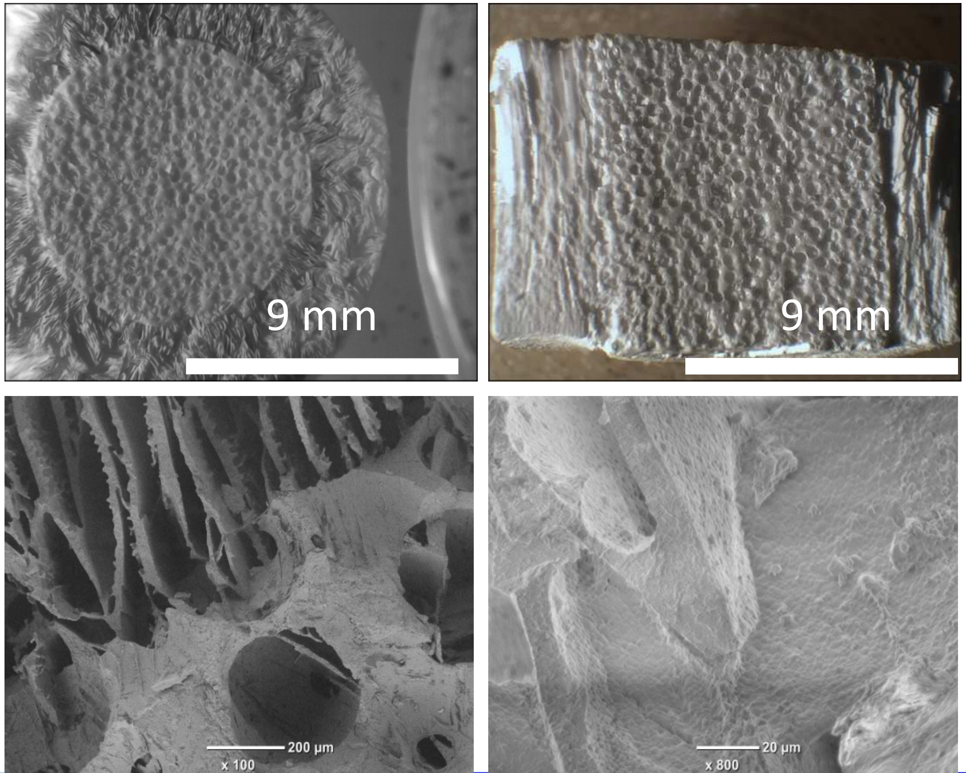


Conclusions and features

- Several techniques are used to produce macroporous bioceramics.
- The choice of the shaping technique has to be done according to the desired pore size range and architecture in relation to the application (implantation site, defect size…).
- Stereolithography (3D-manufacturing) is promising for producing specific scaffold with a complex design, not achievable by the usual techniques.
- Freeze casting is promising owing to the high mechanical properties.

Conclusions and features

- What about structure and cell development relation ? (next presentation S. Chamary)
- PMMA templating and freeze casting structure similar to spongy and compact bone : Is it possible to “merge” the techniques ?
- Complex design with freeze casting technique ? LASER machining on the green part



Aknowledgements

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Thank you for your attention !!!

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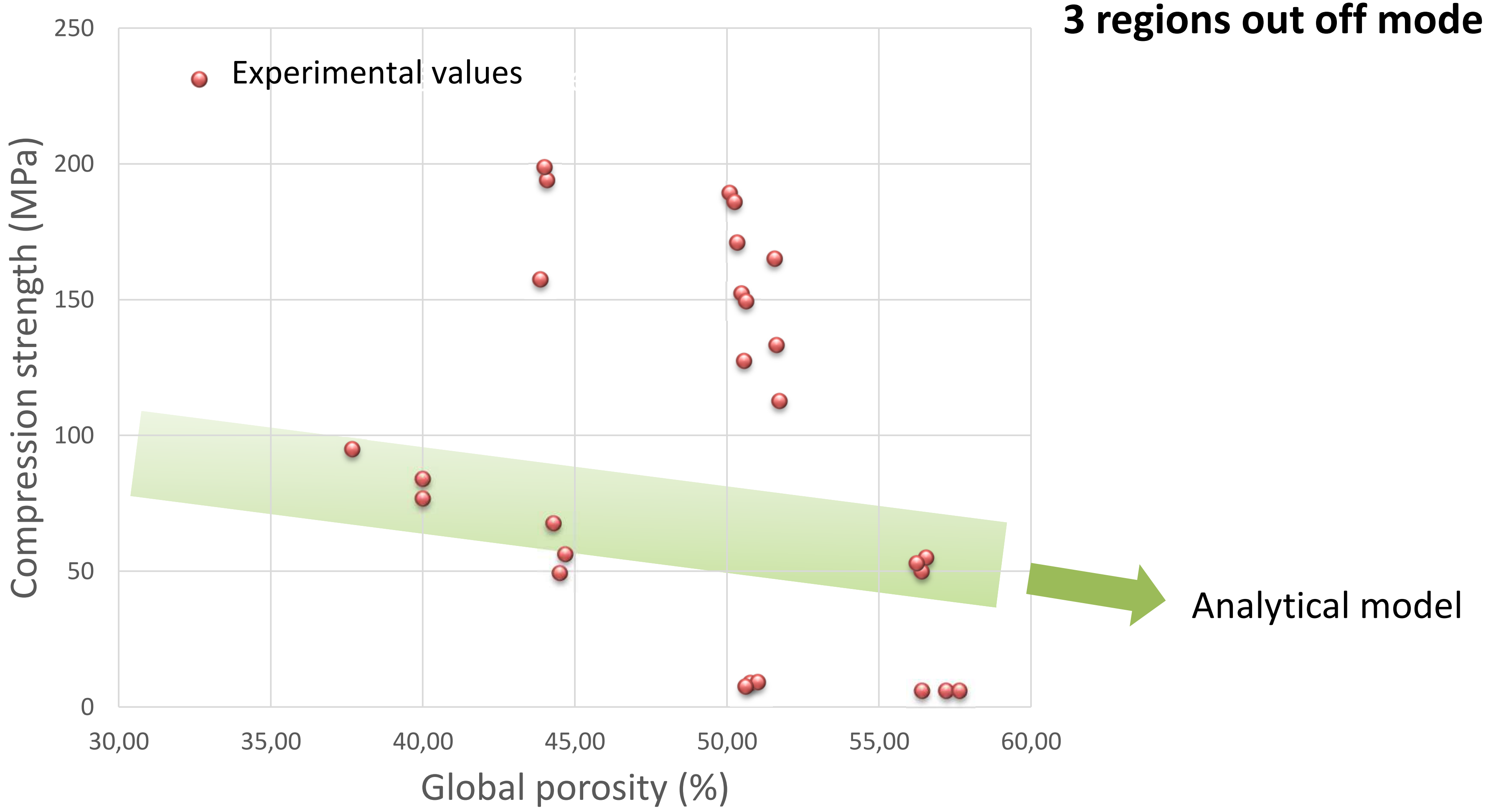
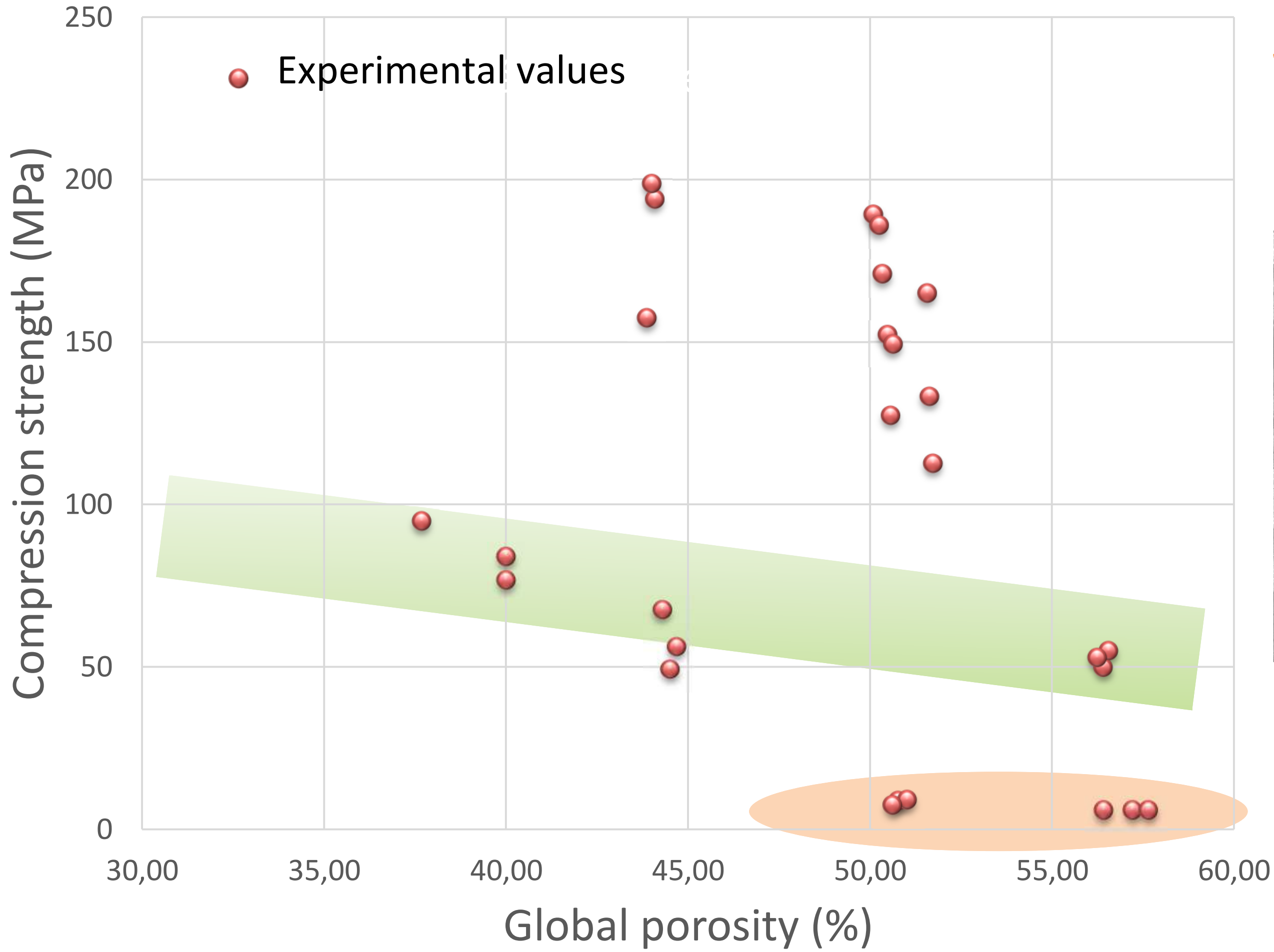
Mechanical properties

3 regions out off model:

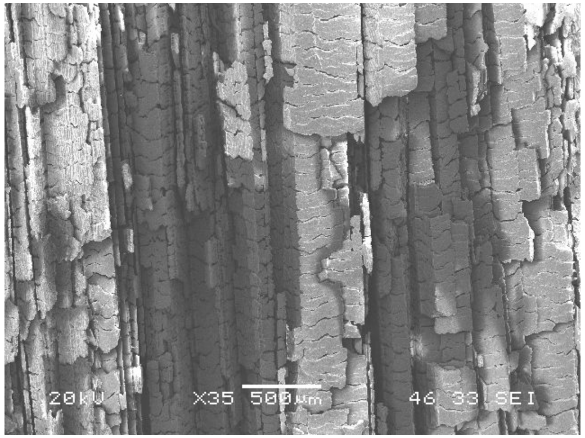


Mechanical properties



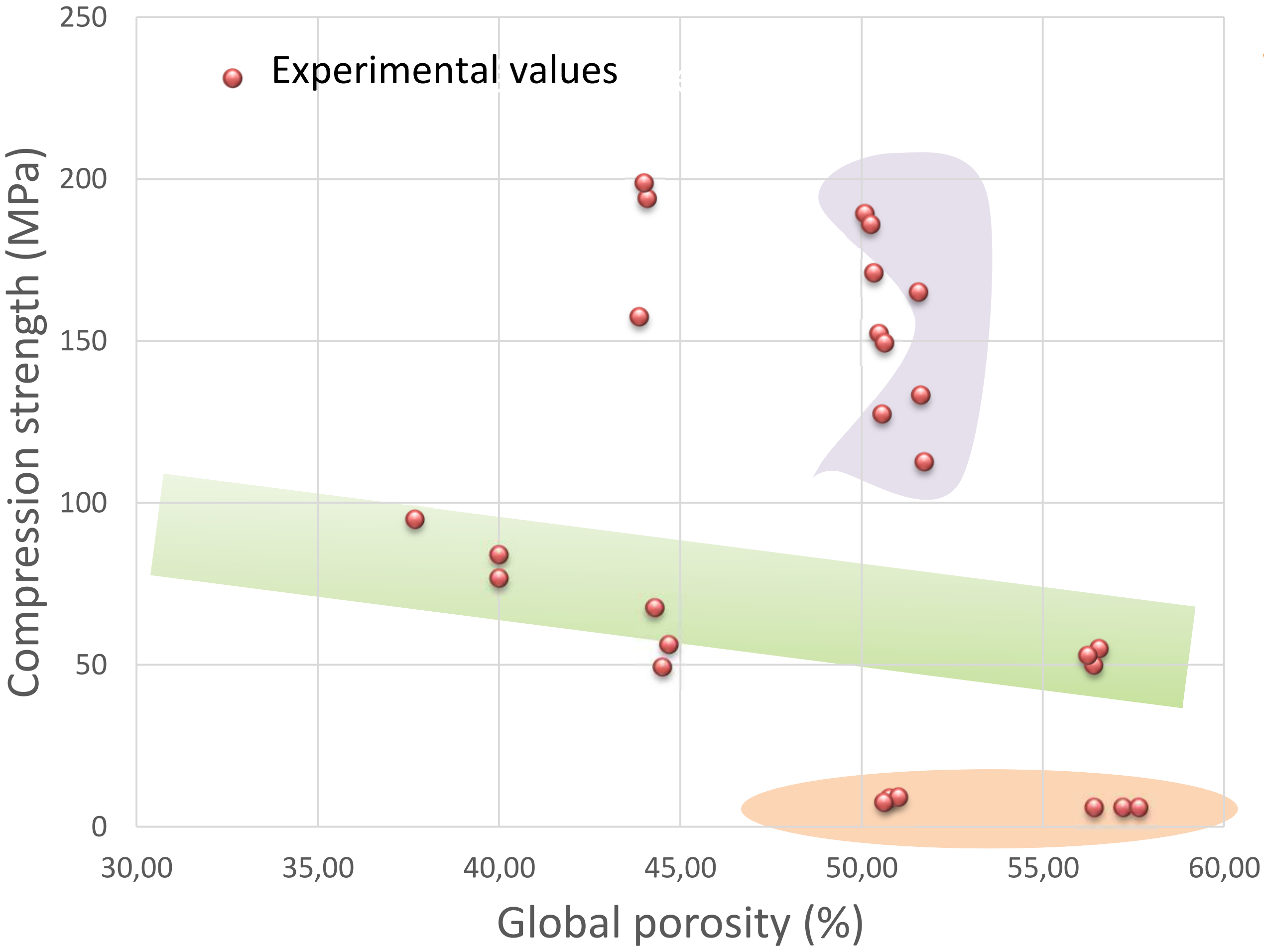
3 regions out off model:

Without binder



Without binder = cracks perpendicular to the freezing direction => low mechanical properties

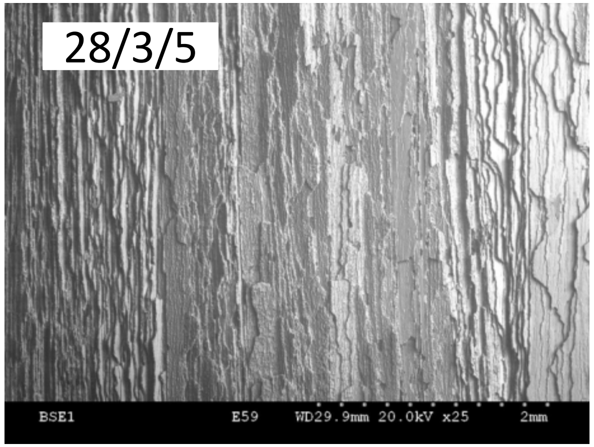
Mechanical properties

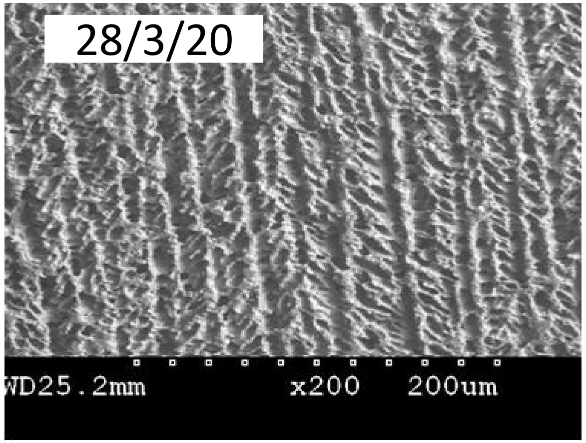


3 regions out off model:

Without binder

High freezing rate

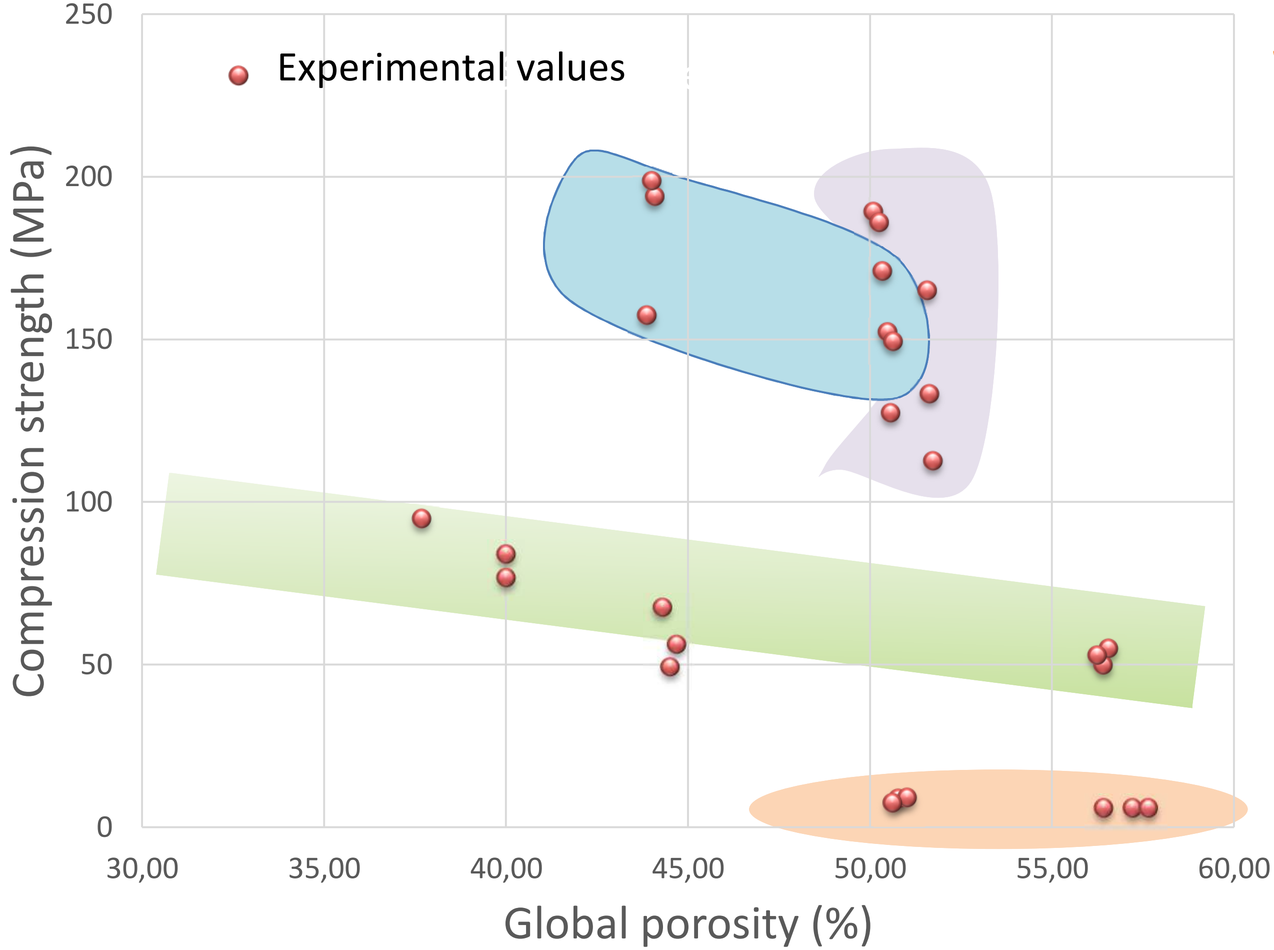




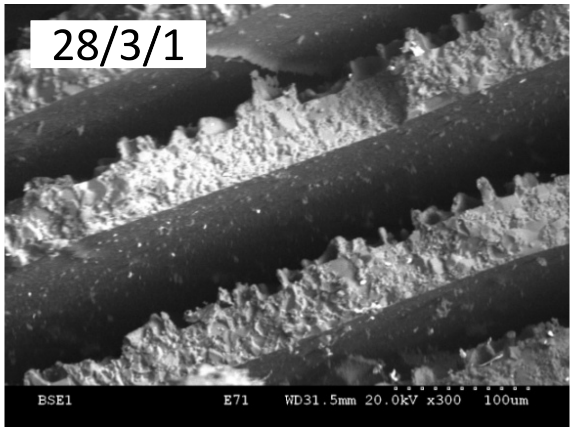
Binder (>3 wt.%) + high freezing rate

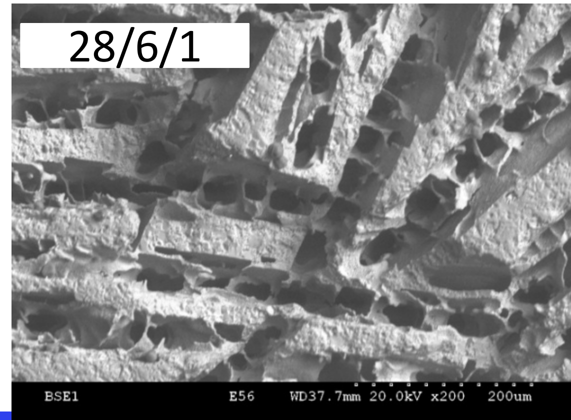
=> morphology modification + smaller pore size

Mechanical properties



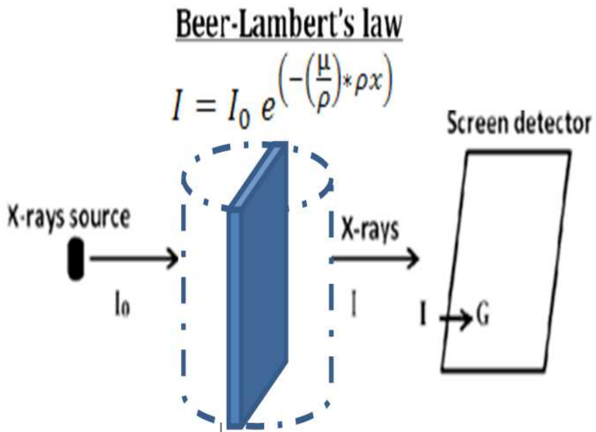
3 regions out off model:
Without binder
High freezing rate
High binder content





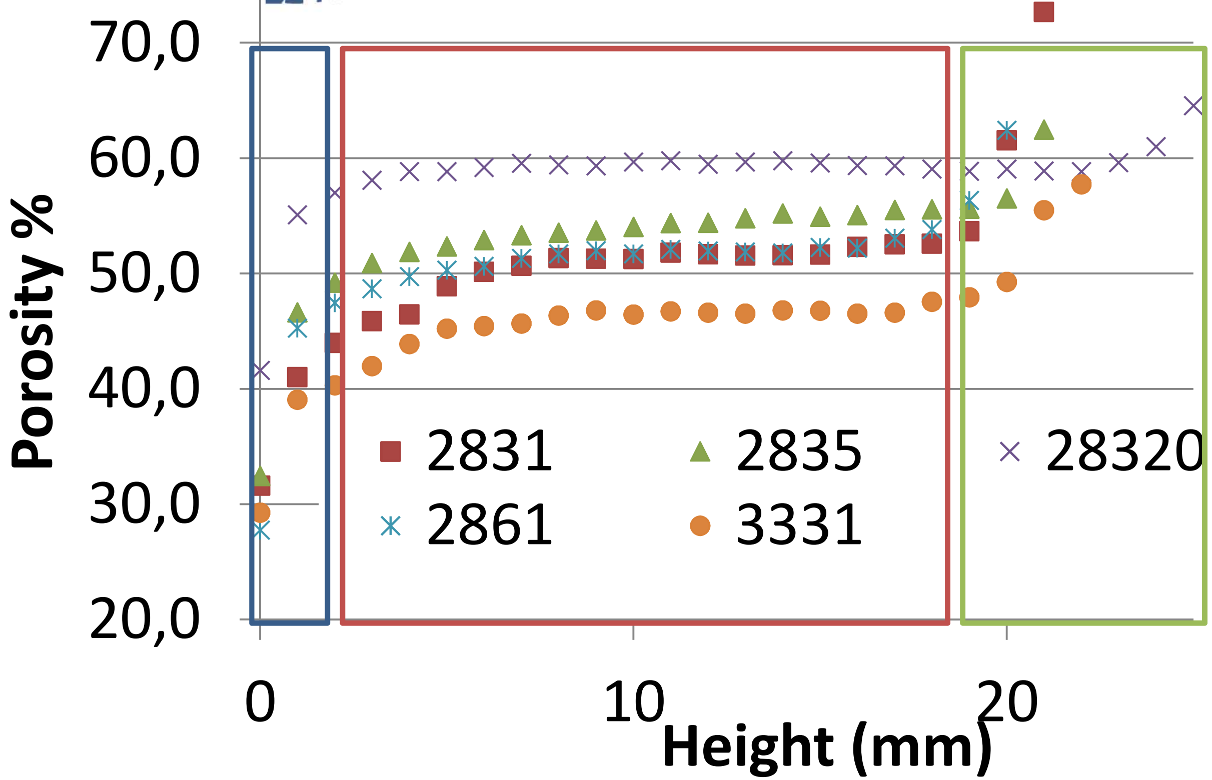
High binder content => structure with bridges

Porosity gradient effect



$$I_i = N_i - N_\infty$$

$$\frac{ln\,\frac{(\bar{N} - N_\infty)}{(N_l - N_\infty)}}{\overline{\left(\frac{\mu}{\rho}\right)}.x} = \rho_l - \bar{\rho}$$



Porosity gradient of about 40% is observed with a plate (3 to 20 mm) where the gradient is about 5%.

The porosity gradient for sample process with different conditions = the same tendencies