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Hydrothermal sintering: a low temperature densification process of ceramics

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Low temperature densification of high performance ceramics

Conventional sintering

1000°C

900°C

Nanopowders High pressure

- High pressure
- ↑ Densification
- ↑ Plastic deformation
- ↑ Pore shrinkage

- Nanopowders
- ↑ Reduction of surface free energy
- ↑ Enhancing diffusional process

Hydrothermal sintering

400°C

Hydrothermal sintering is a relevant and affordable solution to overcome these technological barriers. These low temperatures specific conditions permit the densification of ceramics without solid state diffusion and prevent grain growth or high temperatures phase transition.

An energy and cost-efficient process

Most of advanced ceramic materials are currently fabricated from powders and sintered above 1000°C to reach 95% of their theoretical densities.

→ several technological barriers

grain growth / sintering of metastable materials / co-sintering of multimaterials.

Main features

Hydrothermal sintering apparatus [1]

Sintering parameters :

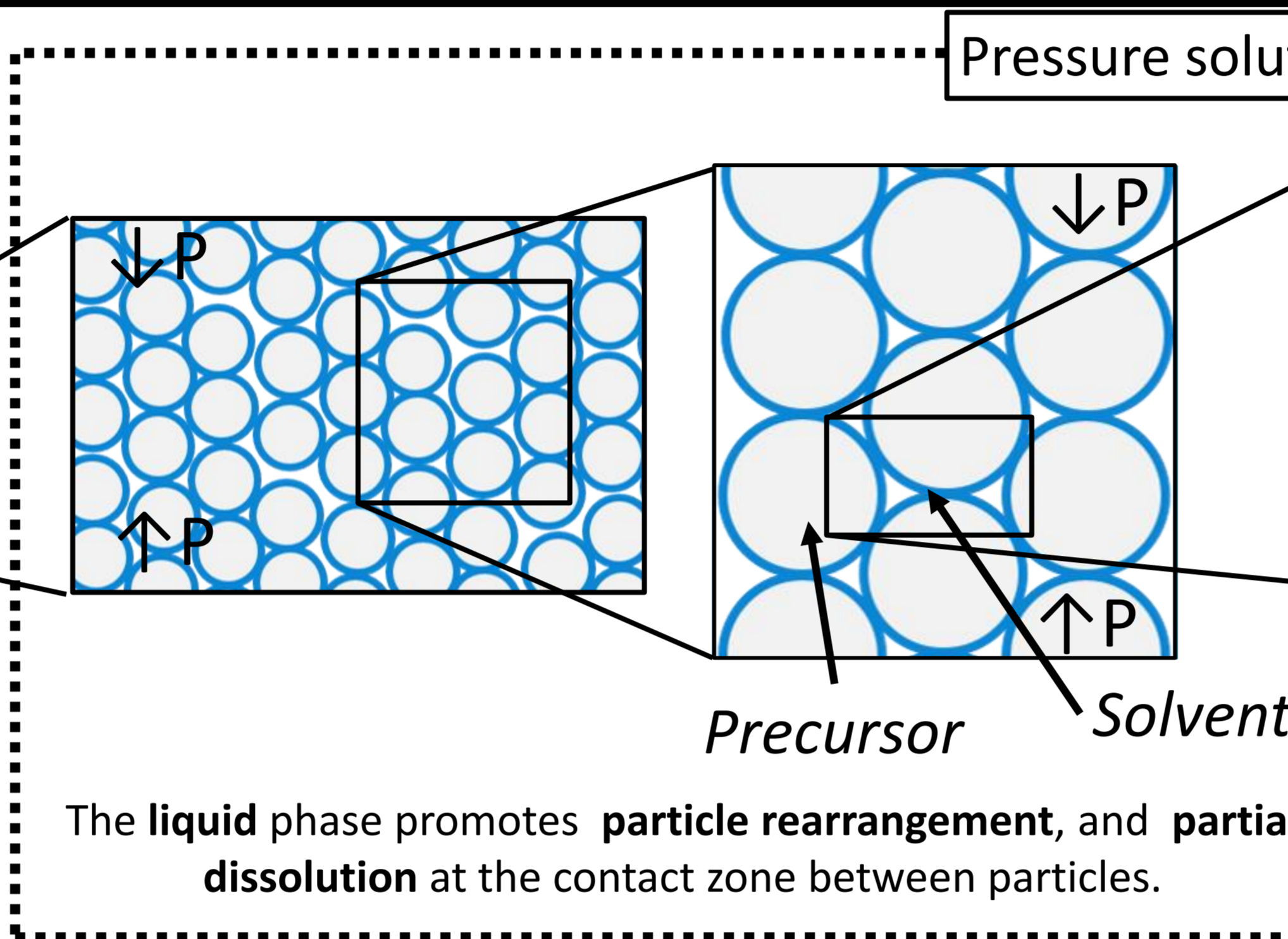
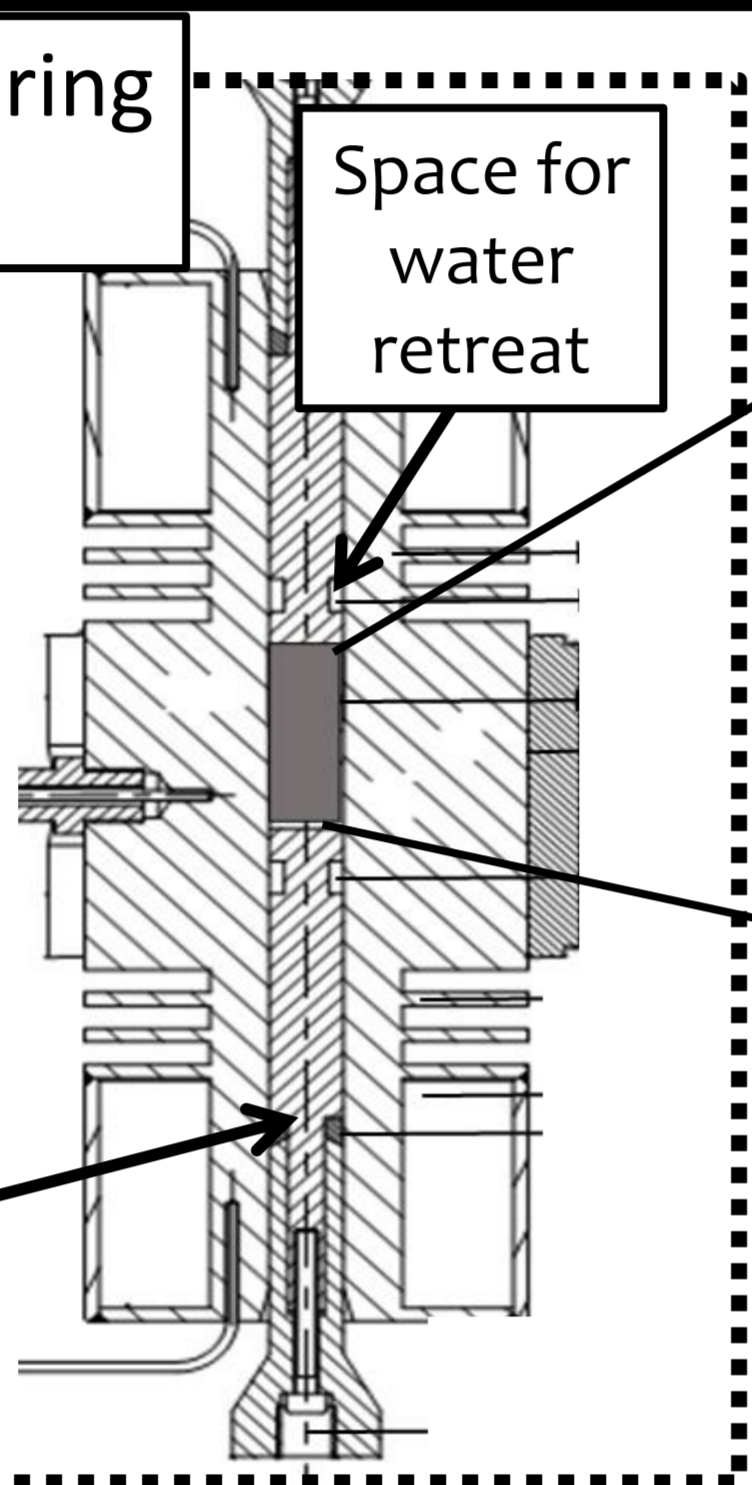
Temperature : 25°C < T < 500°C

Pressure : 20 < P < 350 MPa

Solvent %wt : 1 – 30 %wt

From few minutes to few hours.

A closed system



Pressure solution creep

- 1a) Dissolution
- 1b) Diffusion
- 1c) Precipitation
- 2) Plastic deformation

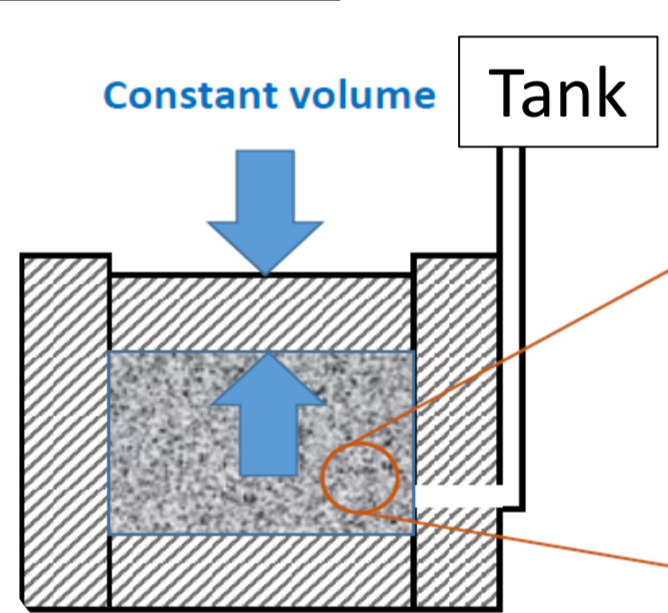
Scheme of the mass transport mechanisms at the particle-particle contact zone [2]

The main driving force is the stress gradient within particles, which allows the activation of the dissolution/precipitation phenomena at the solid/liquid interface. [3]

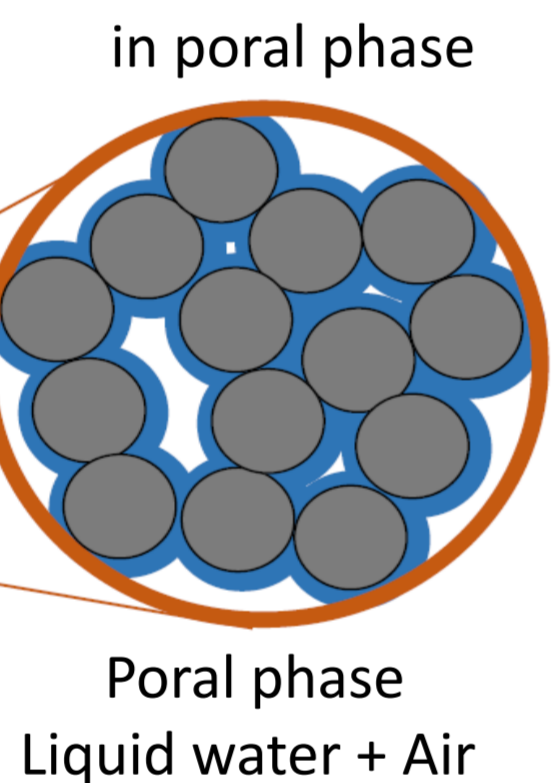
Modelling

The modelling aims at describing the state of water in the system when temperature is increasing before applying pressure:

Initial state of water in poral phase

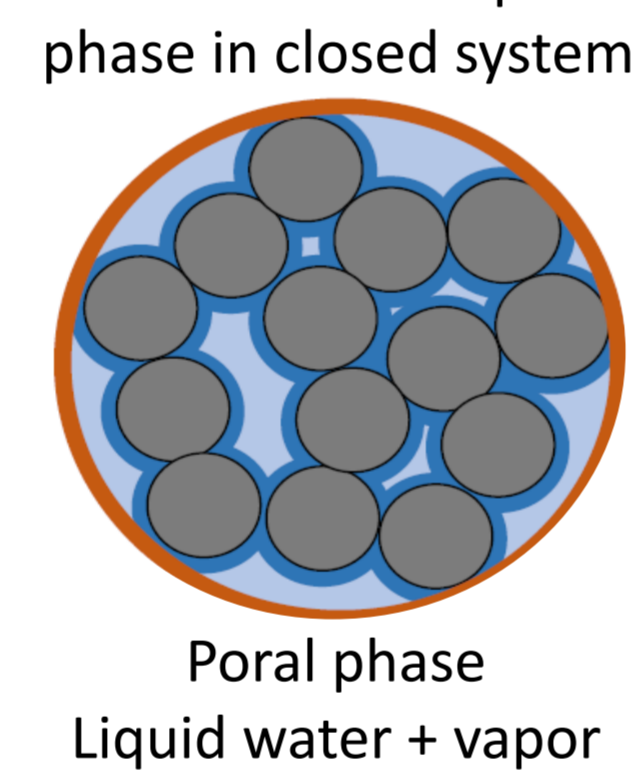


Initial state of water in poral phase



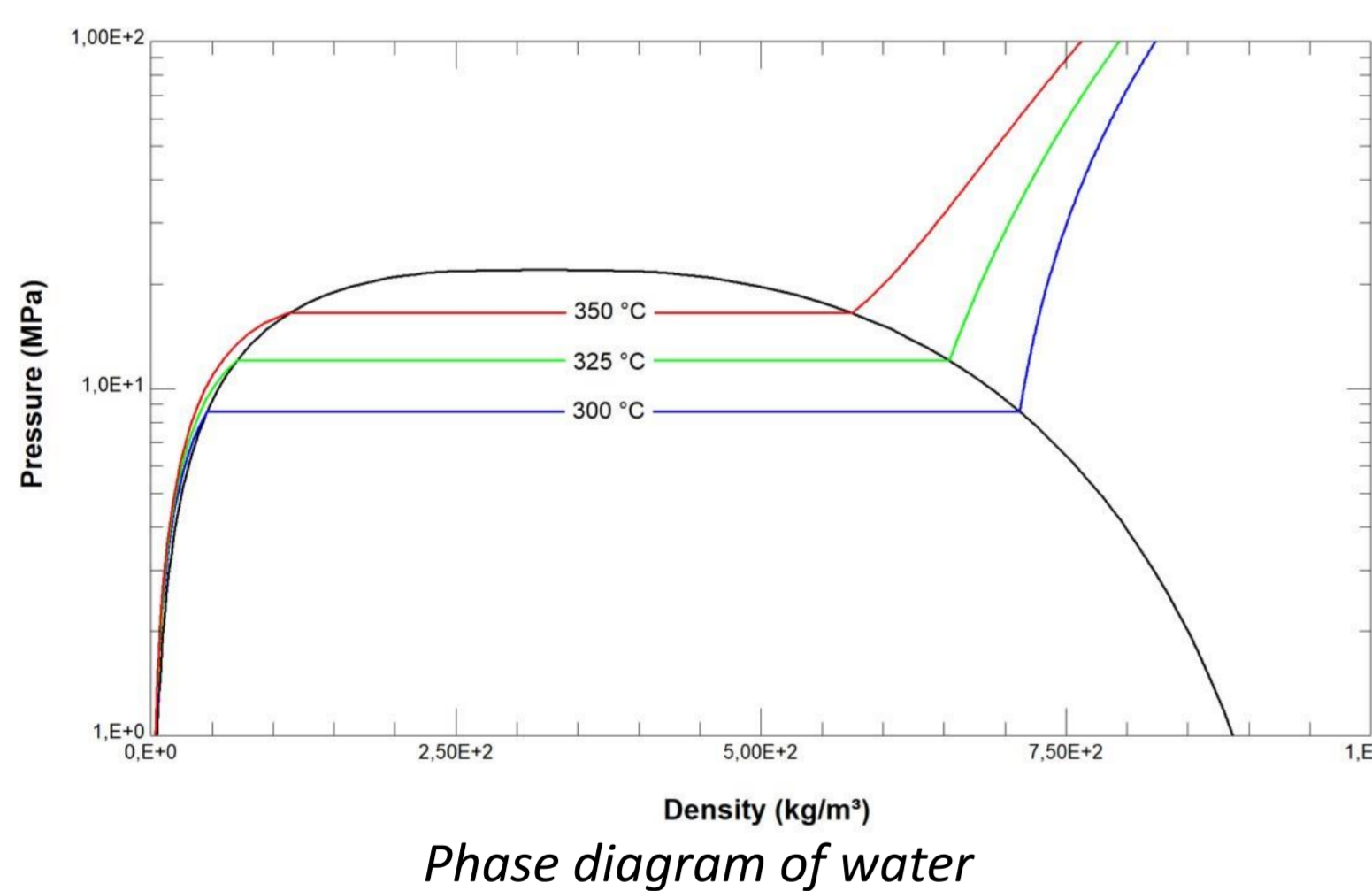
Poral phase
Liquid water + Air

State of water in poral phase in closed system



Poral phase
Liquid water + vapor water

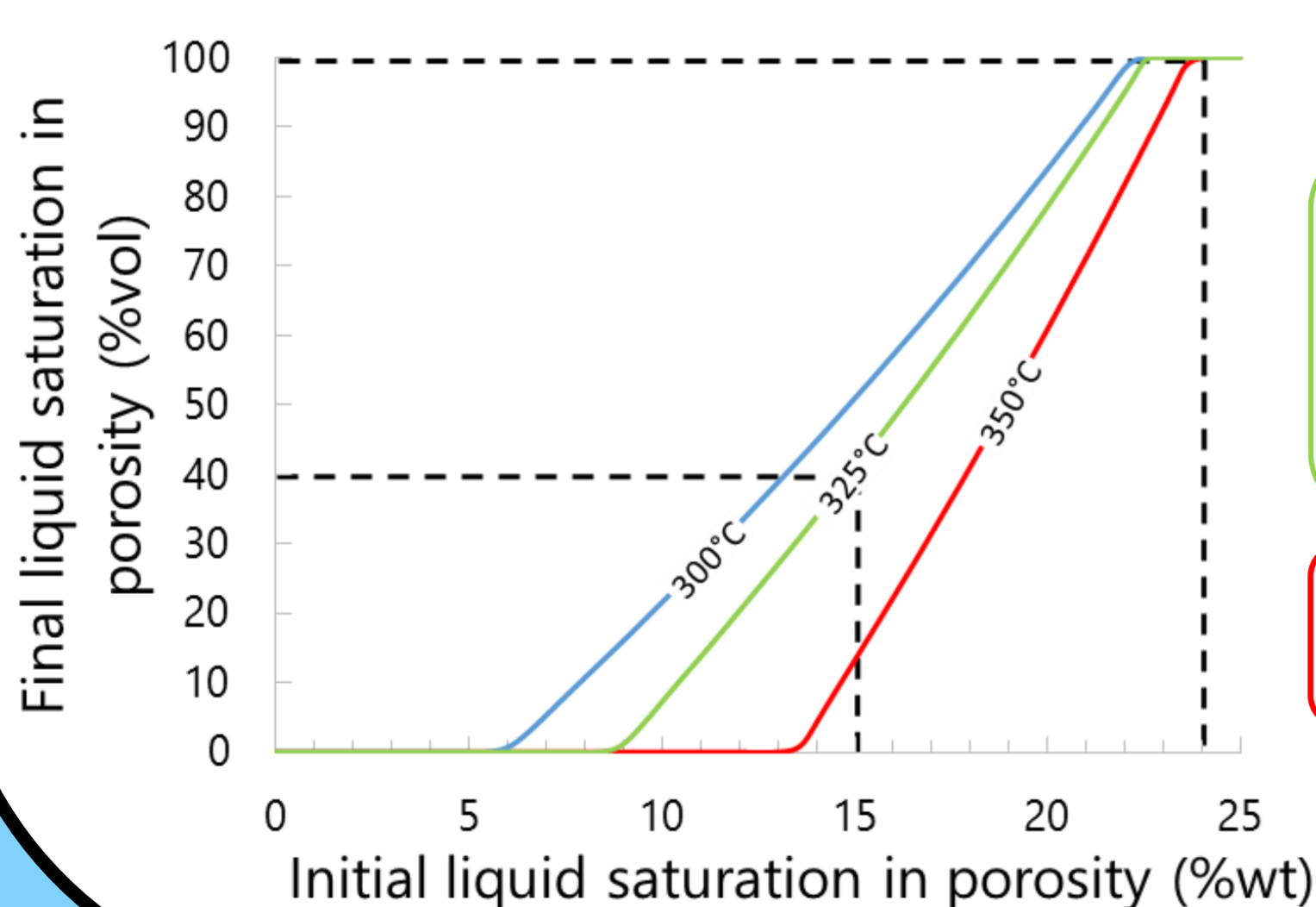
Temperature increasing



This phase diagram of water describes the evolution of pressure as a function of water density for a given temperature.

For a given temperature there is a specific density of liquid and vapor water for the diphasic domain.

From these thermodynamic data, it is thus possible to calculate the liquid saturation in porosity at the process temperature from the initial liquid saturation at room temperature.



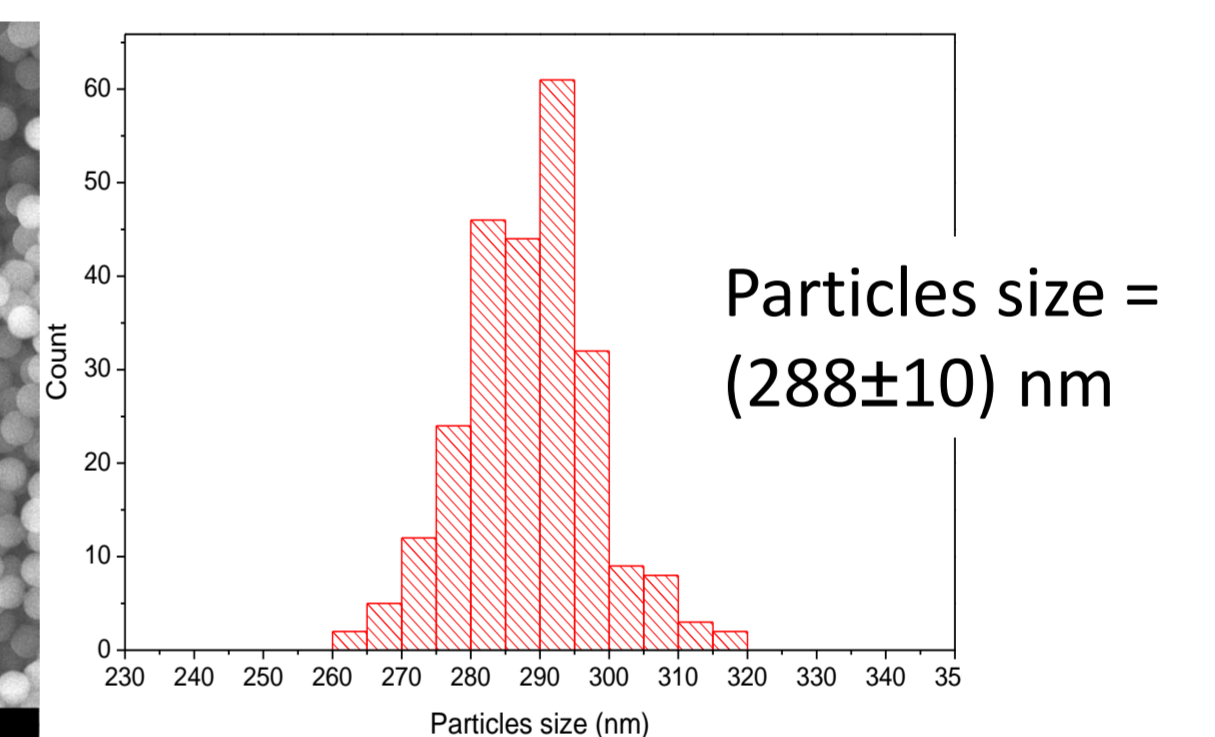
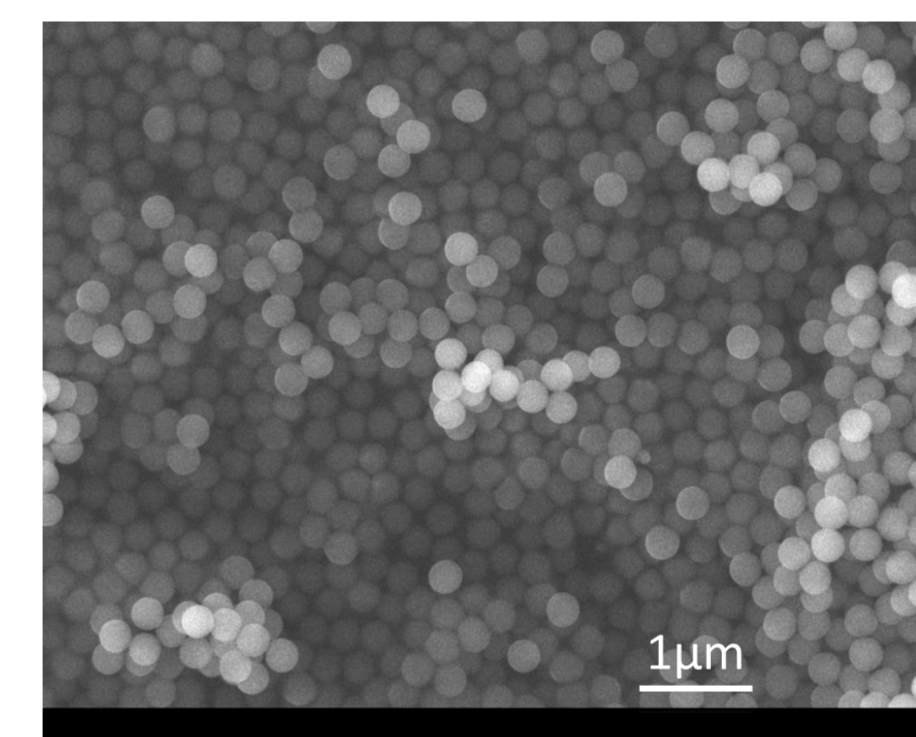
Illustrations :

Considering a final temperature of 325°C and an initial water amount of 15%wt the final liquid saturation would correspond to 40%vol of the porosity.

Similarly, considering 350°C and 24%wt the porosity would be full of liquid water.

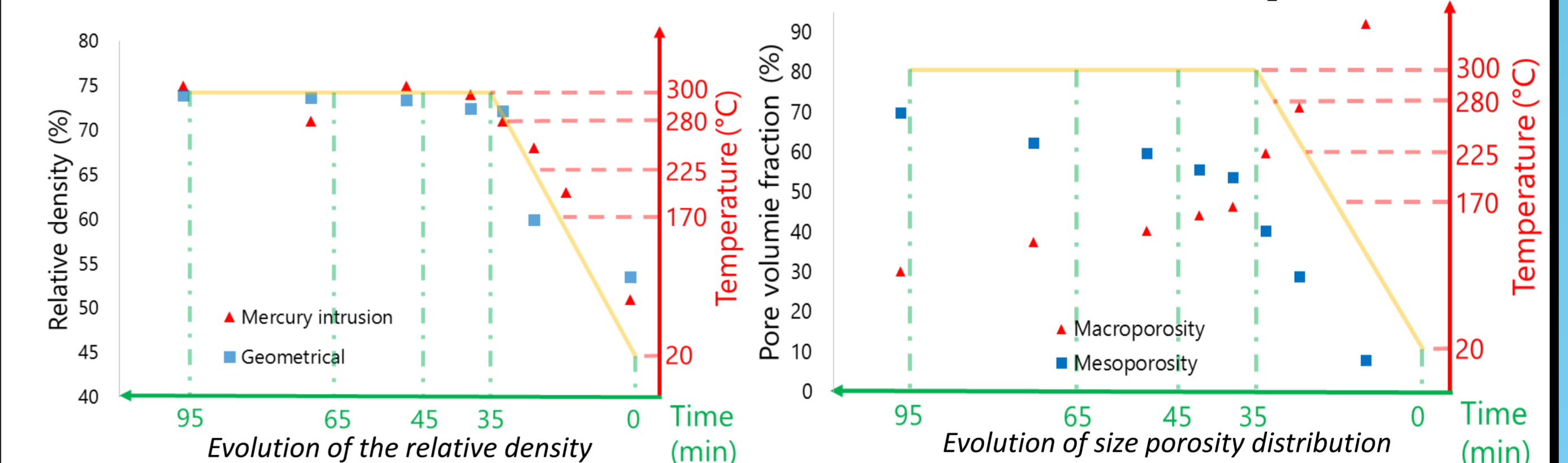
Experimental

Commercial silica nanoparticles :



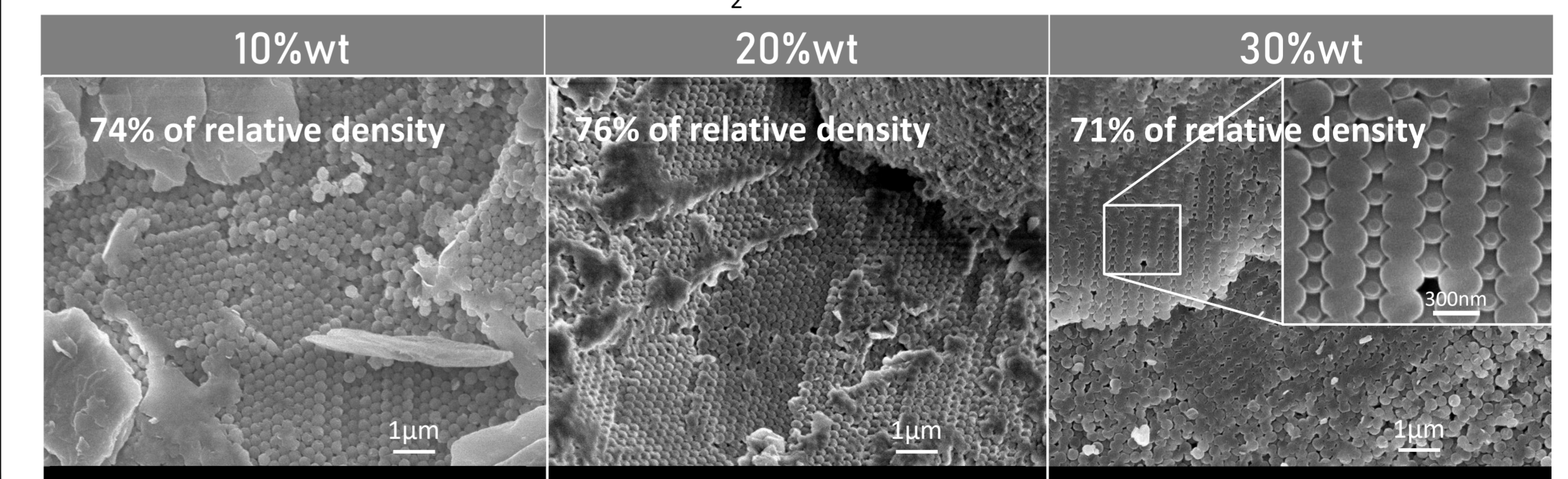
Evaluation of the microstructure during interrupted trials:

Sintering parameters : T = 170 - 300°C (7-8 °C/min), P = 350MPa (start), %wt H₂O = 10%.



Effect of water on sintering :

Sintering parameters : T = 300°C (7-8 °C/min), P = 350MPa (start), dwell time = 60min, %wt H₂O = 10 – 30 %.



Scanning electron micrograph of amorphous silica ceramics

The initial water amount does not significantly impact the relative density but influence the microstructure through change in the distribution of the porosity.

Conclusion and perspectives

Beside the temperature and pressure the microstructure strongly depends on :

- The initial amount of the water
- The dwell time

On the one hand the state (vapor/liquid) of water is controlled by the temperature and the initial water amount in the system. The residual liquid water amount in the pores is a key parameter.

On the other hand the distribution of the porosity is controlled by the time and the initial amount of water

The two parameters will be efficient levers to optimize the densification