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Magnetic Resonance Imaging of Convection in Phase Changing Materials

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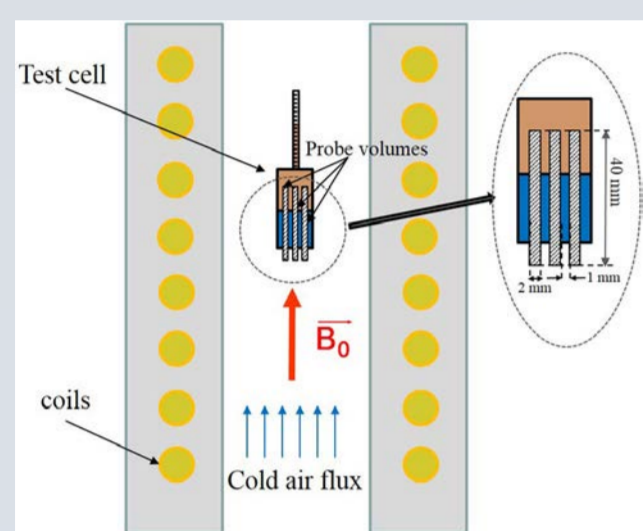
Objectives

Phase changing materials (PCM) are systems with a state change (solid/liquid or liquid/gaz) around ambient temperature. Most of time, studies deals with solid/liquid transitions. This materials are studied a lot for their applications in a lot of domains, and notably energy storage.

However, these materials have relatively low thermal conductivity values which lead to poor thermal exchange during destocking processes. One way to improve these exchanges is to perform the phase change in a convective regime. A good comprehension of the mechanisms at play is therefore necessary to the optimization of the processes.

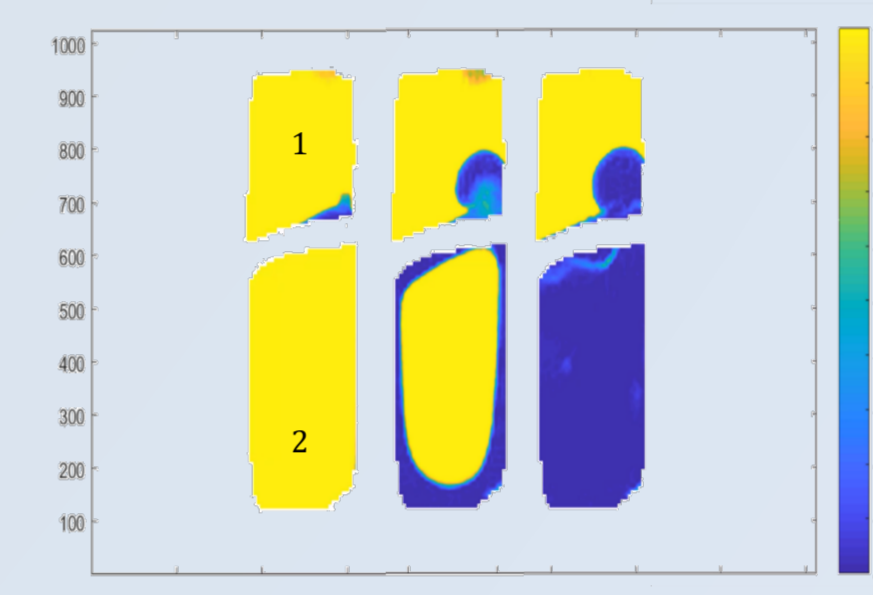
MRI is an interesting tool to study these materials : most of time, the solid phase does not give any signal due to the low relaxation times. Therefore, it is easy to discriminate the two phases and to measure for example the proportions of solid and liquid. Moreover, it is possible to map temperatures inside the system in order to check and improve the mathematical models used to describe theses systems.

MRI of supercooled water

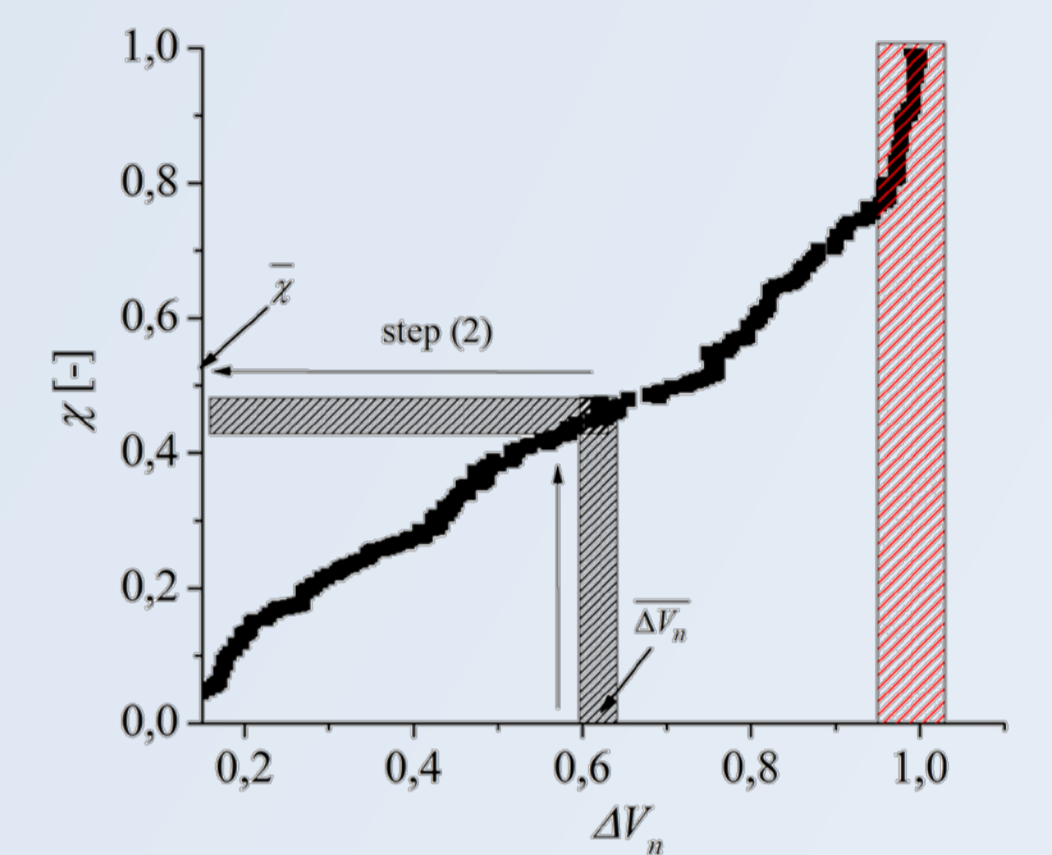


Experimental setup

MRI was used in order to distinguish solid and liquid phase in supercooled water. During freezing, there are two phases : a fast one (few ms) where ice dendrites appears in water, the amount of dendrites being linked to the difference between the actual freezing temperature and 0°C. Up to 15% of the amount of water can freeze nearly instantaneously. A second slower phase corresponds to the freezing of the remaining water. We followed the evolution of the ice fraction with a succession of fast images (on image every 4 s during 30 to 60 minutes) acquired on a 14T vertical magnetic resonance imager.



Three steps of the freezing process (1) Oil - (2) water



Evolution of the ice fraction χ with the normalized ice volume

The increase of volume was extracted from each images using pattern recognition algorithms. We then linked the evolution of the ice fraction with the ice volume. These results helped us to calibrate LIF (laser induced fluorescence) experiments on the same systems in order to measure temperature with this technique.

Velocity measurements during phase changing in Butyle Stearate

Rayleigh Benard convection

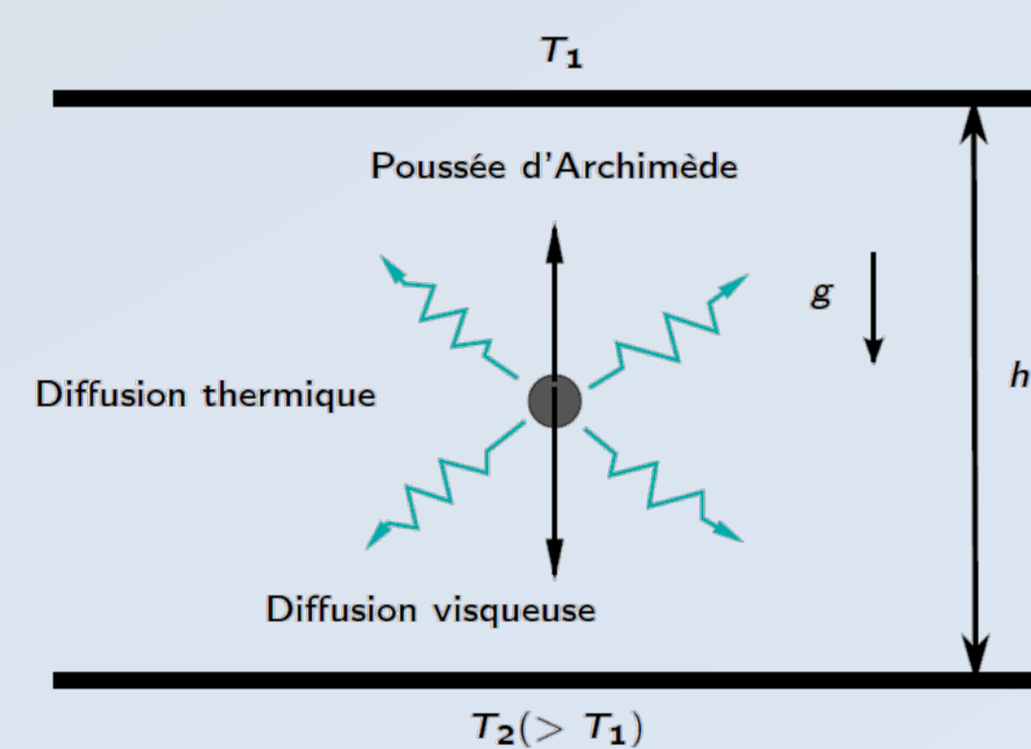
Rayleigh Benard convection is occurring when a fluid is placed between two plates with different temperatures, with heating in the lower part of the setup.

This convection is often described using the Rayleigh number, which takes in account three forces : viscous diffusion, thermal diffusion and buoyancy.

$$Ra = \frac{\rho g \beta \Delta T h^3}{\mu \kappa}$$

with ρ the density of the liquid, g the gravity, β the thermal expansion coefficient, μ the fluid viscosity and κ the thermal diffusivity

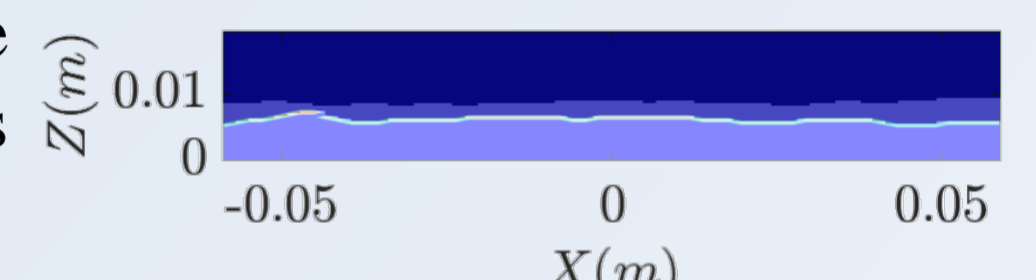
When Ra is lower than 1708, the regime is conductive : there is no convection and temperature decrease with h . When Ra is higher than 1708, convection start and leads to convective patterns (cells, rolls, hexagons...). Velocities increase with the Rayleigh number.



Localization of the convection patterns

During the melting and freezing processes, a waxy phase appears between the solid and liquid ones. This phase is described as “mushy zone” in the literature.

This has an influence on the localization of the convective patterns. We can see different patterns in the two zones, and the velocity are also different.



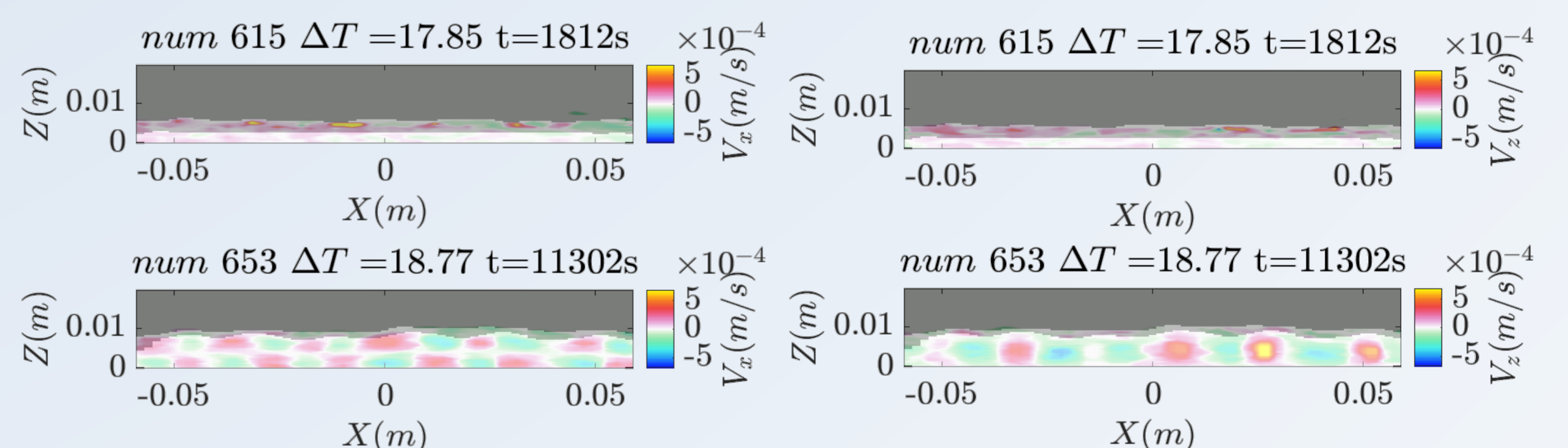
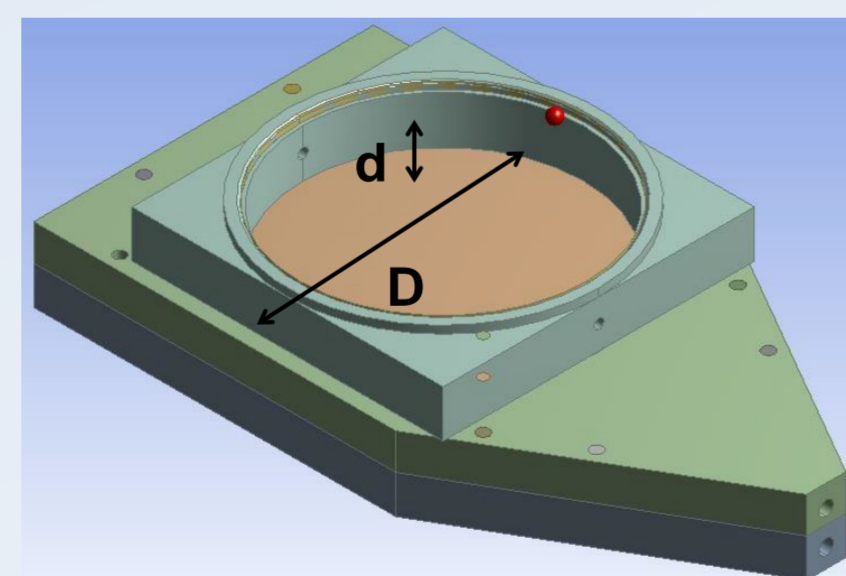
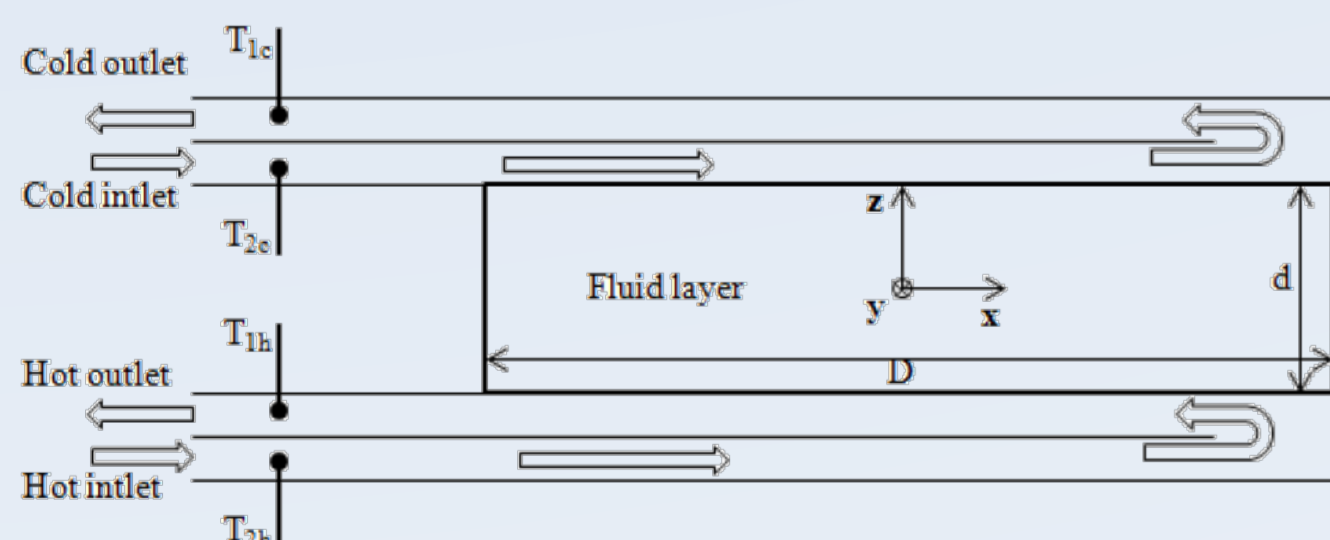
Visualization of the three zones for 15°C (low) and 23 °C (high)

After freezing the sample, we applied a high temperature on the lower part of the sample. The sample started to melt in this area. At start the mushy zone is wider than the liquid part and there is no convection. When the temperature difference increases, the liquid zone also increases and convection starts. As the mushy zone is reduced with this temperature increase, we did not observe significant convection in this part of the sample.

Experimental setup

The fluid layer is confined in a cylindrical cell which is $D=120$ mm in diameter and $d=20$ mm thick providing an aspect ratio of 6. The aspect ratio value is limited by the MRI resonator. All materials used in the setup have been chosen non-metallic to do not disturb the MRI measurements. The lateral walls are made of polymethyl methacrylate (PMMA) of 3 mm thick which is an insulated thermoplastic material. The horizontal walls are made of sapphire, a very good thermal conductor. Temperatures at these walls are imposed by using circulating water obtained via refrigeration units.

The experiments were performed on a Bruker Biospec 24/40 (2,34T). The experimental setup was placed inside a 16 cm diameter volume resonator.



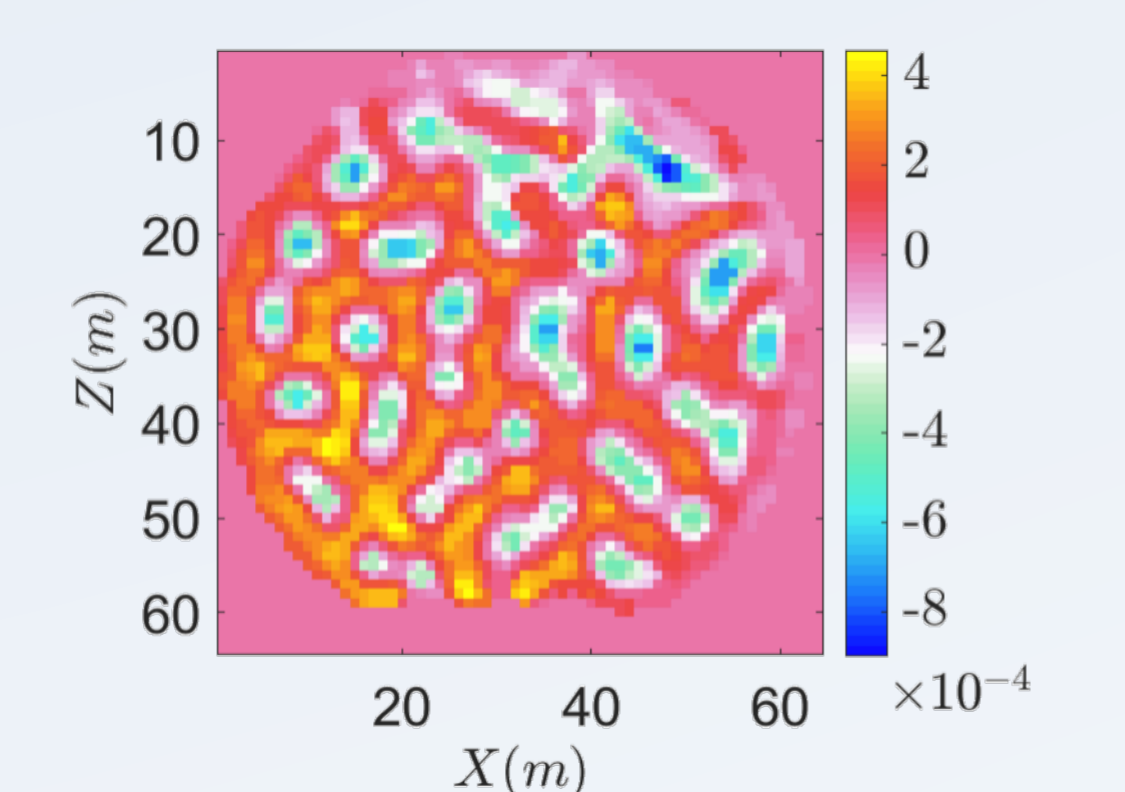
Maps of the horizontal (left) and vertical (right) velocities in the fluid with two temperature differences

The convective flows leads to a deformation of the interfaces (liquid/mushy and mushy/solid). This deformations helps to stabilize the convective patterns, even with relatively high Rayleigh numbers.

Velocity measurements

Velocity images are obtained using a classical spin-echo imaging sequence with additional bipolar gradient pulses in order to encode the velocity. Two images are acquired with different gradient amplitude (0 and 180 mT/m) and the difference of the two images gives the velocity image.

Spin echo sequence is used because it is immune to the chemical shift effect arising from the temperature difference. We have checked that dephasing caused by temperature does not occur due to the bipolar gradients.



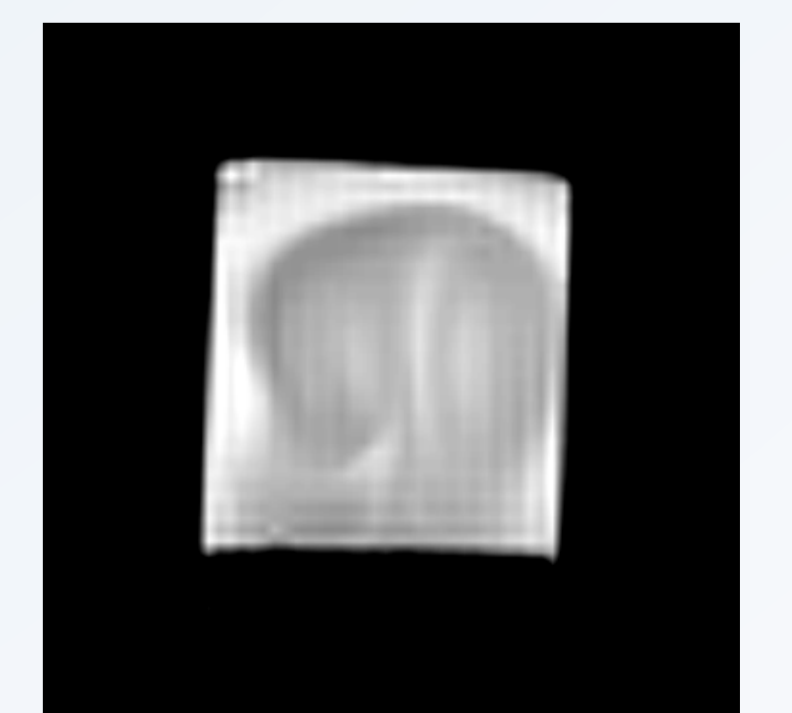
Vertical velocities in butyl stearate (horizontal slice)

For this purpose, the dephasing has been measured in the case $\Delta T = -19.5^\circ\text{C}$ (conductive regime without any motion). In the range of our experimental parameters, we did not observe any significant dephasing caused by temperature variations in the velocity sequences.

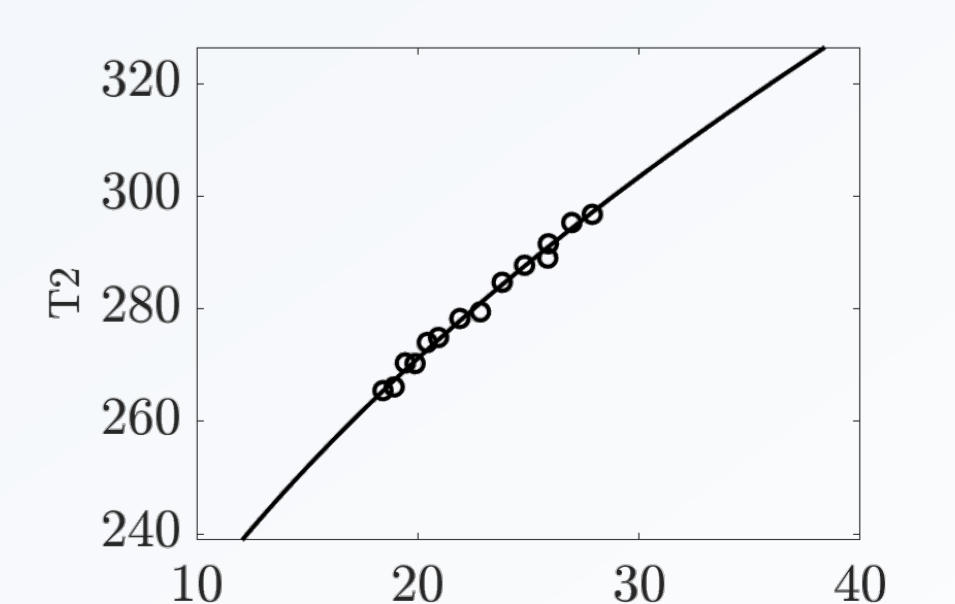
Temperature measurements

There are various ways to measure temperature in MRI. The most commonly used are the proton resonance frequency shift, which leads to a phase shift of the image, and the relaxation times mapping. Proton frequency is accurate in aqueous solvents, hydrogen bonding being very sensible to temperature.

However, in butyl stearate with a 100 MHz imager, we do not see any significant variation of the proton frequency with temperature. Therefore, relaxation times mapping is the only method usable to obtain a temperature cartography. The relaxation measurements can be affected by convection, notably when velocities are not negligible with respect to voxel size and relaxation delays. Therefore we have to take in account these velocities to correct the temperature maps obtained.



Convection effects after ice melting in supercooled water experiments. T2 relaxation imaging



Calibration curve of T2 as a function of temperature.