Experimental report

14/02/2023

Proposal: 6-07-88 **Council:** 10/2020

Title: intrusion of LiCl solutions in hydrophobic materials

Research area: Materials

This proposal is a new proposal

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Samples: LiCl water solutions
Silicates , SiO2, methyl groups at the surface

Instrument	Requested days	Allocated days	From	To
D16	6	4	07/06/2021	11/06/2021

Abstract:

In a very recent work (fall 2019), we have studied by neutron scattering the thermodynamic states of water confined in very hydrophobic material. Experiments performed by neutron imaging (IMAGINE,LLB) were carried out and the intrusion pressure for some hydrophobic materials was measured; then diffraction experiments performed on the G44 (LLB) have revealed very surprising and interesting results on the cristalline states of water. The present proposal focuses now on ionic solutions confined in hydrophobic materials. It appears in the literature that LiCl solutions strongly confined present interesting energetic performances, allowing the storage of higher energy quantity than pure water in the same material. We would like then to explore the amount of solutions intruded in the new very hydrophobic material we have synthesized . We are expecting larger energy storage capacity and more exotic phase transitions of the solutions when the T and P are changed. Therefore we ask for 6 days of experiment on the instrument D16.

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Experiment was performed on D16 from 06/07/2021 to 06/11/2021 (λ~4.5Å, Orange cryostat + pressure cell). The object of the measurements was to study the behavior of water (D_2O) confined in hydrophobic MCM mesosilica, in particular the stabilization of ice phase in "abnormal" Pressure/Temperature conditions. The pressure/Temperature domain correspond to that of hexagonal ice Ih in ambient conditions, and close to cubic ice Ic and ice II and III polymorphs (Fig. 1)

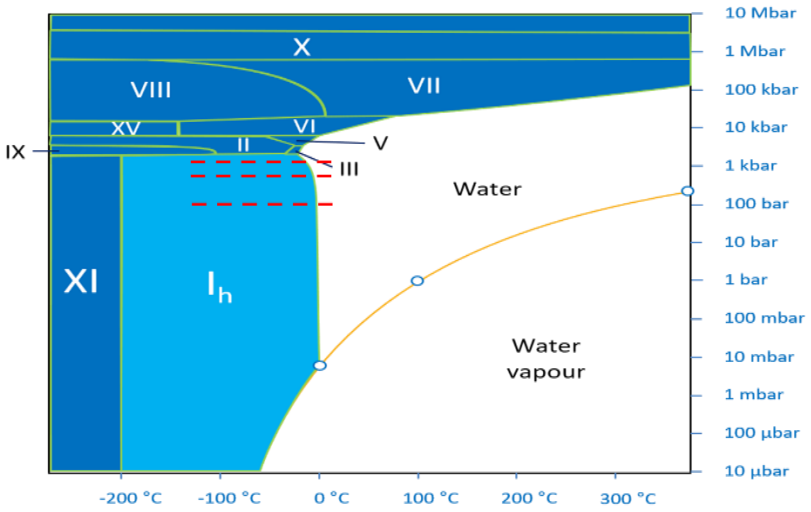

Figure 1: Typical phase diagram for water - The thermodynamic paths explored are shown as red lines

The experiment clearly shows up the stabilization of additional Ice phases, in addition to the expected ice Ih, present inside and around the hydrophobic MCM grains in the pressure cell (Fig. 2 & Fig. 3)

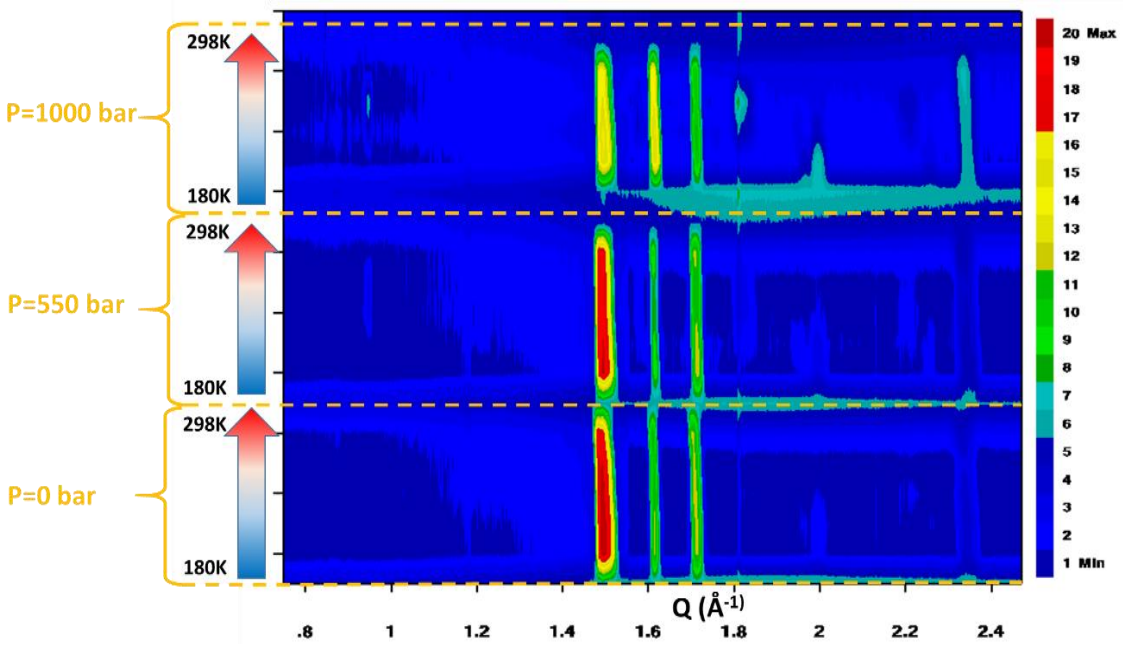

Figure 2: Colour plot of ice phases crystallized during (P, T) cycles

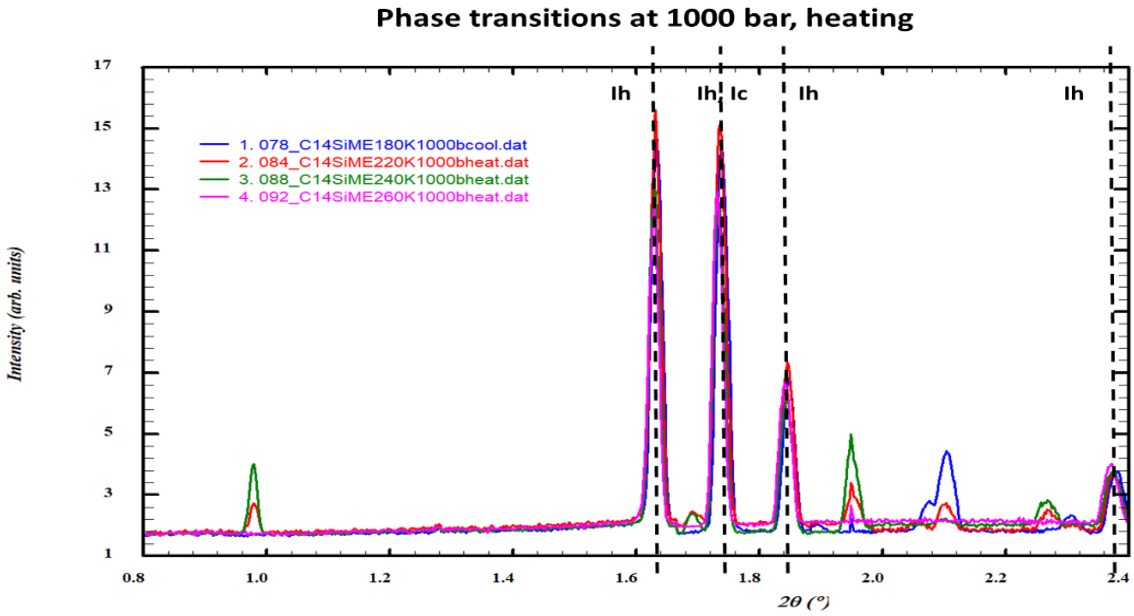

Figure 3: Diffractograms measured on D16 as a function of T at P=1000bar.

Rietveld analysis of the pattern is still underway but shows up a stabilization of ice Ih, Ic, II and to a less extent Ice III, depending on the (P, T) evolution.

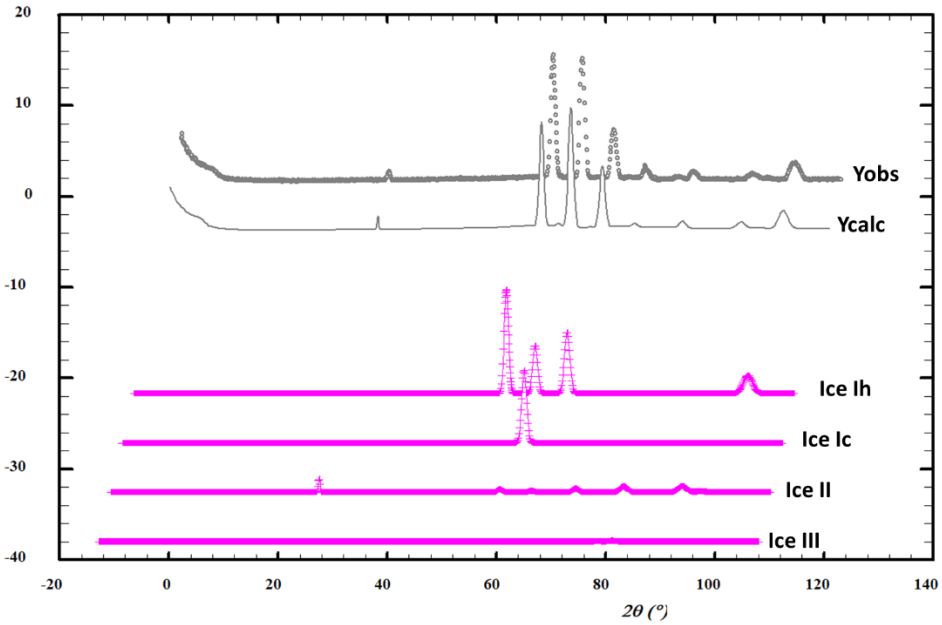

Figure 4: Rietveld decomposition showing the coexistence of ice phases Ih, Ic & II (P=1000 bar & T=220K).