	Experiment title: NANO-CONFINEMENT IN ZEOLITES: HIGH PRESSURE INDUCED POLYMERIZATION FOR INNOVATIVE GAS SENSING MATERIALSPEPTIDE SYNTHESIS	**Experiment number**: CH5445
Beamline: ID15B	**Date of experiment**: from: 07/5/18 to: 11/5/2018	**Date of report**:
Shifts: 12	**Local contact(s)**: Michael Hanfland	*Received at ESRF:*

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Report:

All silica zeolites with monodimensional channel systems were selected for this experiment, in particular TON and MOR framework type.
The zeolite were pre-loaded in laboratory via vapor phase with hexadine (Hexa) and two series of experiments were performed: 1) using liquid unsaturated hexadiene acting as penetrating PTM; 2) using DAFNE oil as non penetrating PTM.
X-ray powder diffraction patterns have been collected during the compression stages to follow *in situ* the polymerization reaction. At the maximum pressure reached, for selected sample HT was applied to favour the polymerization.
Here the pressure reached for the various ramp.

MOR+hexa compressed in hexa (2.8 GPa P max, 200°C T max)
MOR+hexa compressed in Dafne (1.94 GPa P max, 200°C T max)
TON+hexa compressed in hexa (2.52 GPa P max, 200°C T max)
TON+hexa compressed in Dafne (3.3 GPa P max, 185°C T max)
TON sample was not tested with Ph since it was previously demonstrated that the penetration of the molecules was not allowed in the pores.

The data relative to MOR + hexa has been successfully refined.

The structural refinement allow to locate the hexadiene molecules and the water molecules in MOR cavities at P amb: **6 carbon** atoms in each channel 2.4 water molecule in each cell, positioned in the pocket.
In figure 1 the representation of the structure

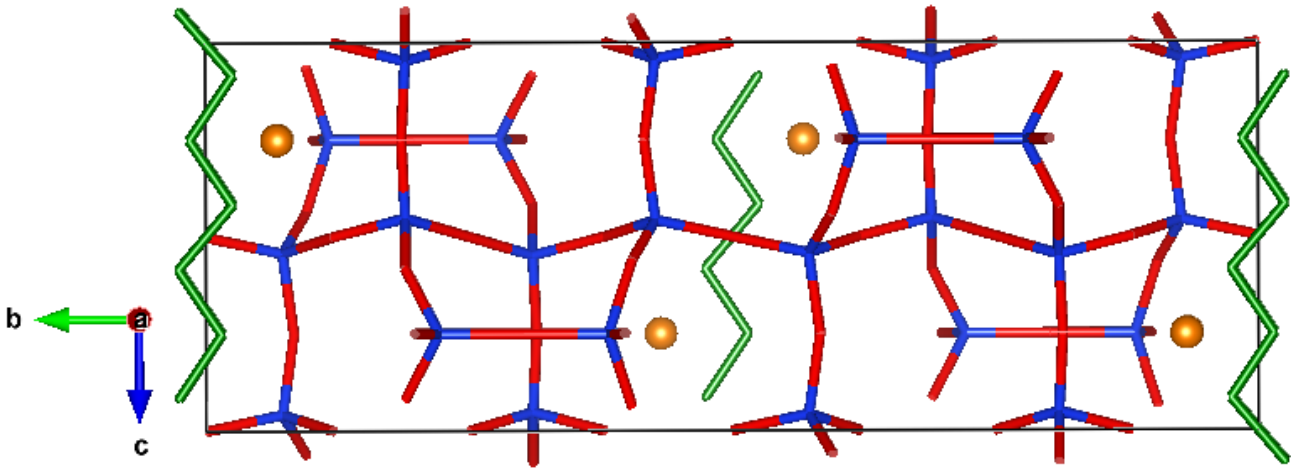

Figure 1. Structure of MOR hexa at P amb

The compression of the sample in Dafne oil induces variation in the unit cell, but the structure remains almost unchanged (Figures 2and 3).

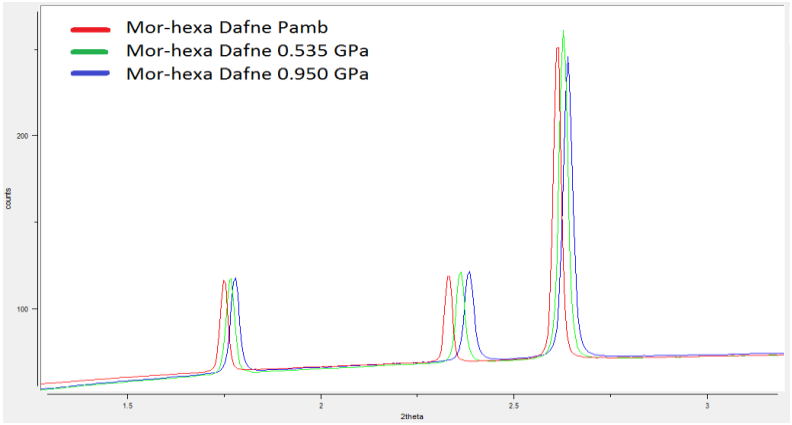

Figure 2. Selected diffraction patterns

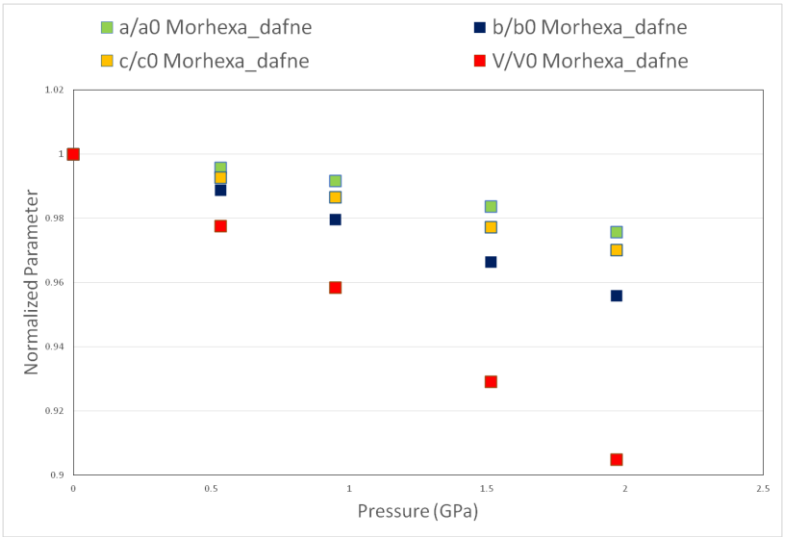

Figure 3. Variation of unit cell when MOR is compressed in Dafne oil

The compression of the systems in hexadiene seems to induce further changes in the structure, but the complexity of the system did no allow a satisfactory structural refinement: the Fourier difference map obtained at high pressure (reported in Figure 4) clearly show the presence of disordered maxima in the channels

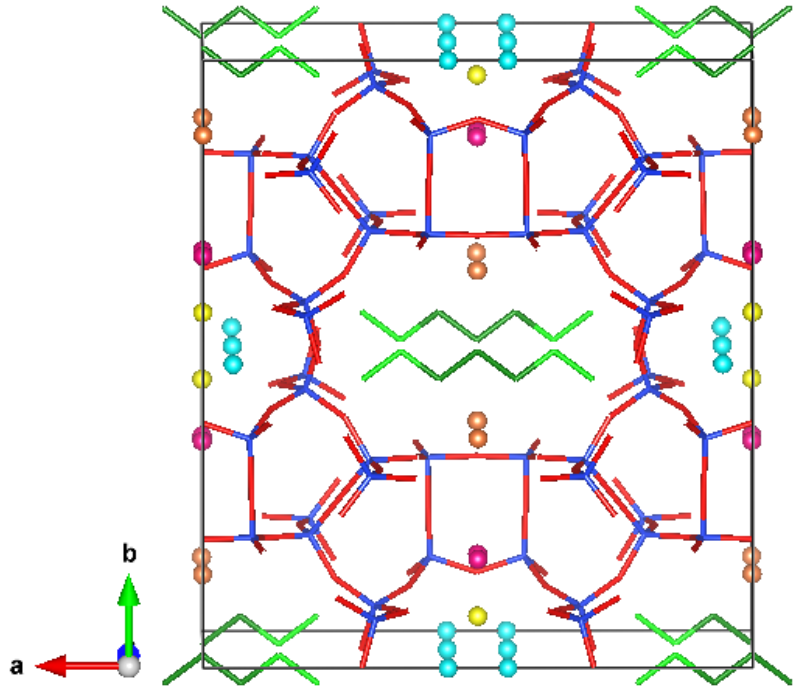

Figure 4. Maxima of the Fourier difference map in MOR-hexa @1.68GPa compressed in hexa.

The thermal treatment at high pressure seem to almost amorphized the sample, making impossible even the unit cell refinement.

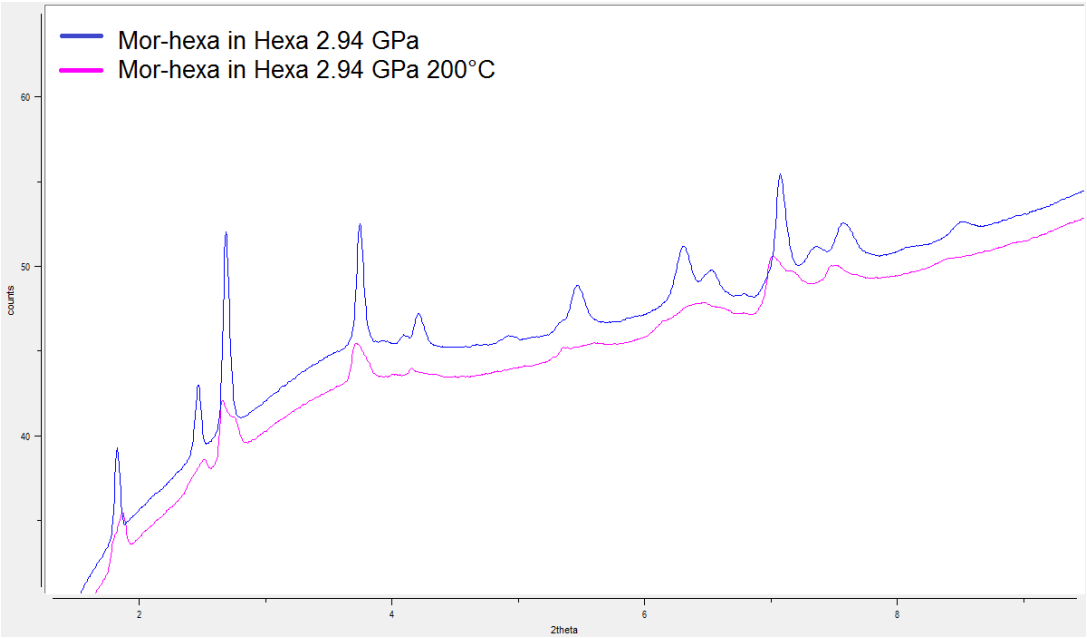

Figure 5. Comparison of XRPD spectra at HP and HP/HT

The data of TON are still under analyses.