	Experiment title: Supercritical microfluidic study for CO2 geological storage	**Experiment number**: **EV-132**
Beamline: ID19	**Date of experiment**: from: 15/04/2016 to: 17/04/2016	**Date of report**: 02/09/2016
Shifts: 6	**Local contact(s)**: Lukas Helfen	*Received at ESRF:*

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Report:

In July 2015 we acquired 65 scans, but it has been impossible to reconstruct correctly a single 3D volume from these data sets (cf. first report for EV-132). A test performed in December 2015 with an empty microfluidic device produced a data set for which no problem of reconstruction occurred. Consequently, the problem was not linked to the samples properties. A new experiment has been scheduled for April 2016.

We improved our experimental protocol on two aspects: 1) The shape of the reactive particles: In July 2015, spheroidal reactive particles were prepared at ICMCB a few days before the experiment, but when injected in the microfluidic device the particles appeared to be needles instead of spheres: during the transport calcite became aragonite. *For the new experiment, reactive particles have been transported dry and put into the solution at equilibrium shortly before injection*. 2) The stability of the reactive medium during the scans: The transition between the reactive fluid and the fluid in equilibrium was not sharp enough and transport by diffusion sustained the reaction during the scans. The main consequence was the production of gas bubbles. *For the new experiment, a multi-way valve has been used with a calibrated by-pass containing the reactive flow (Fig. 1).*

The reconstruction procedure has been carefully checked before the series of reactive fluid injections. A section through the system in its initial state is shown Fig. 2.

We injected a calibrated volume of reactive solution with a slowly decreasing pH in order to control the quantity of dissolved carbonate particles. The problem is that the system was not changing at all. Reconstructed images were identical at each step from the initial one (0) to the step number 17. Suddenly a huge change occurred (Fig. 3).

After long discussions, the interpretation was that a large number of particles were blocked within the connexion bloc. The injected reactive fluid dissolved some of these particles during the injection steps 1 to 17. Injection pressure increased at each step because of the compaction of the packing composed of partly dissolved particles, and suddenly, during step 18, the fluid broke this internal packing causing a rapid flow of very reactive fluid within the micro fluidic system itself. The catastrophic change was the consequence of this sudden flow.

Conditions of injection-step N°19 have been defined as initially estimated, and a "normal" behaviour happened: the fluid dissolved a limited fringe of carbonate particle around the packing (Fig. 4). This proved that small dissolution steps could be obtained and scanned. For a complete characterization of these evolutions, 3D differences between images are required. Because of the laminography properties (weak contrast in the horizontal planes, big phase contrast in our acquisition conditions), an adapted procedure must be developed. We began the corresponding developments by first looking for an optimal estimation of the upper and lower limits of the microfluidic device, and second building a precise mask corresponding to the lateral boundaries of the device (Fig. 5). These parts are fully operational and different 3D registration approaches between the different 3D images are under evaluation in order to reach a registration precision around 0.25 voxel.

Fig. 1: Microfluidic device fixed on the new sample holder and with the multi-way valve.

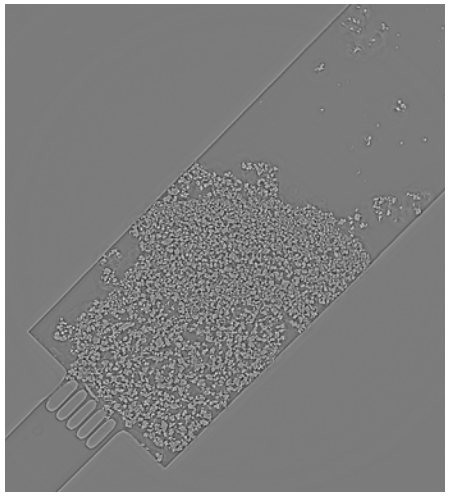

Fig. 2: Section through the microfluidic device at the initial state.

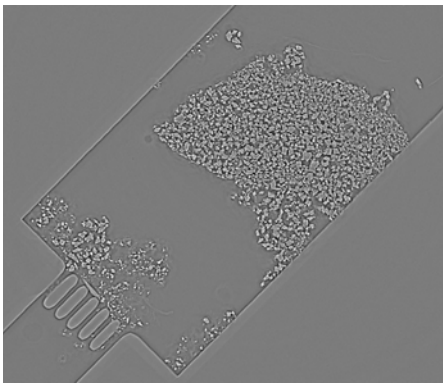

Fig. 3: Catastrophic change of the particle packing that occurred during the injection step N°18.

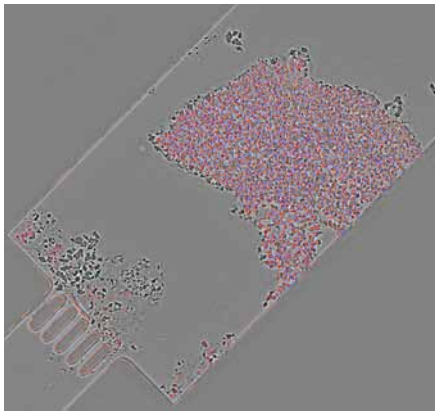

Fig. 4: Rough difference between states 18 and 19. The black particles at the limit of the red packing have been dissolved during injection step 19.

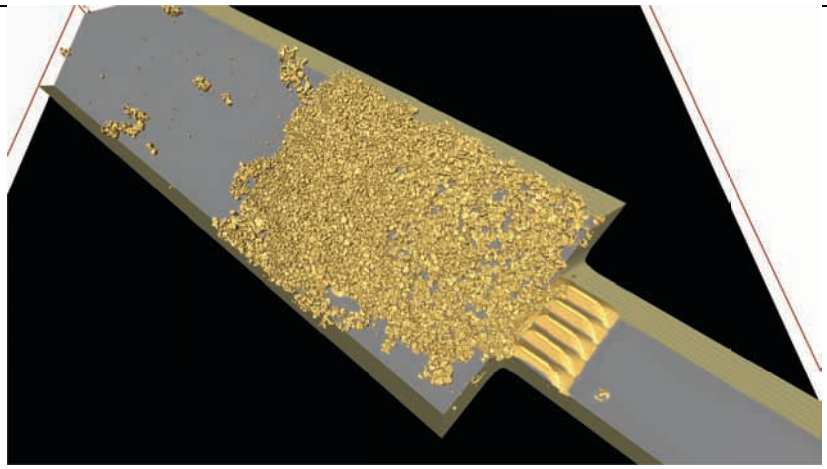

Fig. 5: 3D rendering of the interior of the microfluidic device after determination of the lateral boundaries (in yellow).