ESRF	**Experiment title:** **In-situ high resolution CT study of the healing process in fractured cement under elevated temperature and pressure.**	**Experiment number**: MA 3230
Beamline: ID-19	**Date of experiment**: from: April 29, 2017 to: May 01, 2017	**Date of report**: September 13,2023
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Names and affiliations of applicants (* indicates experimentalists):

Laboratory Norwegian University of Science & Technology Department of Physics TRONDHEIM

- Dr CHAVEZ PANDURO Elvia Anabela; Phone- 004798764815; Email- elvia.a.c.panduro@ntnu.no
- Prof. BREIBY Dag Werner; Email- Dag.Breiby@ntnu.no

Laboratory Riso National Laboratory Materials Research Department P.O. Box 49 - Building 228 DK - 4000 ROSKILDE

- Dr OSHOLM SØRENSEN Henning Phone +45 4677 5783 Email-henning.sorensen@risoe.dk

Laboratory SINTEF, S. P. Andersens vei 15 B NO - 7031 TRONDHEIM

- Dr. TORSAETER Malin Phone +4748111159 Email malin.torsater@sintef.no

Laboratory Universite du Maine Laboratoire de Physique de l Etat Condensé Avenue Olivier Messiaen FR - 72085 LE MANS Cedex 9

- Prof. GIBAUD Alain Phone 02 43 83 32 62 Fax 02 43 83 35 18 Email alain.gibaud@univ-lemans.fr

Laboratory University of Angers MINT INSERM UMR-S 1066 IBS-CHU ANGERS 4 rue Larrey FR - 499 ANGERS 9

- CALVIGNAC Brice Phone 06 16 97 07 37 Email brice.calvignac@univ-angers.fr

Laboratory University of Copenhagen Department of Chemistry Nanoscience Center The H. C. Ørsted Institute Universitetsparken 5 DK - 2100 COPENHAGEN

- Dr YANG Yi Email yiyang@nano.ku.dk

Report:

Depleted oil reservoirs are considered a viable solution to the global challenge of CO2 storage. A key concern is whether the wells can be suitably sealed with cement to hinder the escape of CO2. Under reservoir conditions, CO2 is in its supercritical state, and the high pressures and temperatures involved make real-time microscopic observations of cement degradation experimentally challenging. Here, we present an in situ 3D dynamic X-ray micro computed tomography (μ-CT) study of well cement carbonation at realistic reservoir stress, pore-pressure, and temperature conditions. The high-resolution time-lapse 3D images allow monitoring the progress of reaction fronts in Portland cement, including density changes, sample deformation, and mineral precipitation and dissolution. By switching between flow and nonflow conditions of CO2-saturated water through cement, we were able to delineate regimes dominated by calcium carbonate precipitation and dissolution. For the first time, we demonstrate experimentally the impact of the flow history on CO2 leakage risk for cement plugging. In-situ μ-CT experiments combined with geochemical modeling provide unique insight into the interactions between CO2 and cement, potentially helping in assessing the risks of CO2 storage in geological reservoirs.(Abstract)[1]

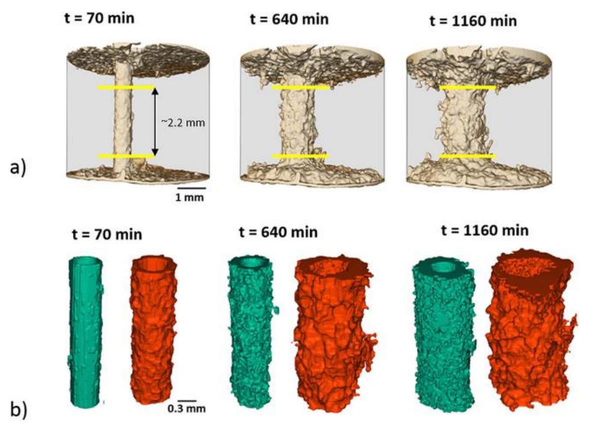

Figure 1. Reaction fronts in the cement as a function of time retrieved from μ-CT analysis. (a) 3D volume rendering of the whole specimen, showing reacted cement in yellow, at 70, 640, and 1160 min, (b) 3D rendering of carbonated (red) and porous silica zones (green).[1]

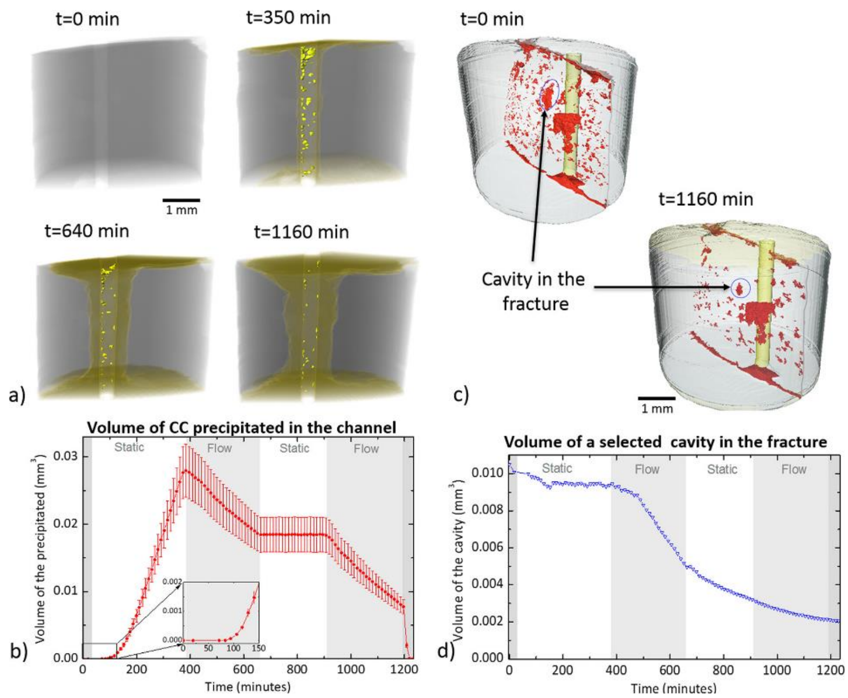

Figure 2. Precipitation in the channel and the fracture. (a) 3D visualization of precipitated $CaCO_3$ in the channel, the crystals are highlighted in sharp yellow, reacted cement in yellow-brown and the unreacted cement cylinder is in semitransparent gray. (b) Time evolution of the precipitated $CaCO_3$ volume in the channel. The inset shows a zoomed-in view of the first 150 min (c) 3D visualization of the fracture (in red) before and after CO_2 exposure. The channel is represented in transparent yellow. (d) Time evolution of the volume of the selected cavity in the fracture shown in part c.[1]

The X-ray measurements were done ID19 beamline, using a high photon energy of 84 keV to enable the X-ray radiation to be transmitted through the titanium walls of the deformation apparatus. The sample–detector distance was 1200 mm, giving edge-enhanced phase contrast. (37) 2D projections were obtained with a PCO Dimax detector with a pixel size of 6.45 μm and an exposure time of 0.05 s. For each time step, a full μ-CT data set consisting of 2000 radiographs (projections) over 180° were acquired. In total, 103 time-steps (full tomograms) were recorded over a period of nearly 20 h. The data were reconstructed using PyHST2 involving filtered back-projection coupled with the phase contrast algorithm.[1]

This study presents unprecedented time-lapse 3D imaging of the progressive degradation of Portland cement and the concomitant precipitation and dissolution of CaCO3 in leakage pathways with exposure to sc-CO2. Importantly, the data was obtained in situ, showing the response to carbonated water at controlled flow rates under realistic reservoir conditions of high pressure and temperature. The alternated application of stagnant and flow-through conditions during the experiment aided delineation of precipitation versus dissolution-dominated regimes: information that can only be achieved through in situ laboratory experiments and potentially can be used for solving imminent environmental challenges related to CO2 storage. The methodology of increasingly realistic in situ imaging experiments combined with advanced modeling is destined to find many applications in the environmental and material sciences in the coming years. In this study, experiments and geochemical modeling confirm that conditions with small CO2 flow rates promote the formation of the carbonated zone and subsequently precipitation of CaCO3 in large cavities. The have been published in high impact journal also got considerably cited[1]. The users are thankful to ESRF for providing beamtime and support and also plan to apply for more beamtimes with high hope of acceptance based on the these results.

[1] Elvia A. Chavez Panduro, Benoît Cordonnier, et al, Real Time 3D Observations of Portland Cement Carbonation at CO2 Storage Conditions, *Environmental Science & Technology*, 2020, 54 (13), 8323-8332.

Note: Figures and most of text is taken from the published article.