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A new challenge: in-situ investigation of the elusive nanostructures in wet halite and clay using BIB/FIB-cryo-SEM methods

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Mudrocks and saltrocks form seals for hydrocarbon accumulations, aquitards and chemical barriers. The sealing capacity is controlled either by the rock microstructure or by chemical interactions between minerals and the permeating fluid. A detailed knowledge about the sealing characteristics is of particular interest in Petroleum Sciences. Other fields of interest are the storage of anthropogenic carbon dioxide and radioactive waste in geologic formations.

A key factor to the understanding of sealing by mudstones and saltrocks is the study of their porosity. However, Halite and clay are so fluids sensitive that investigation on dried samples required by traditional methods of investigations (metal injection methods [6],[3]; magnetic susceptibility measurement [4]; SEM imaging of broken surfaces [5] and CT scanner computing [7]) are critical for robust interpretation. In one hand, none of these methods is able to directly describe the in-situ porosity at the pore scale and on the other hand, most of these methods require dried samples in which the natural structure of pores could be damaged due to the desiccation, dehydration and dissolution-recrystallisation of the fabric.

SEM imaging is certainly the most direct approach to investigate the porosity but it is generally limited by the poor quality of the mechanically prepared surfaces. This problem is solved by the recent development of ion milling tools (FIB: Focussed Ion Beam or BIB: Broad Ion Beam, which allows producing in-situ high quality polished cross-sections suitable for high resolution pores SEM imaging at nano-scale. More over, new and innovative developments of the cryo-SEM approach in the Geosciences allow investigating samples under wet natural conditions.
Thus, we are developing the combination of FIB/BIB-cryo-SEM methods ([1],[2]), which combine in one machine the vitrification of the pore fluids by very rapid cooling, the excavation of the sample by ion milling tool and SEM imaging. By these, we are able to stabilize the in-situ fluids in grain boundaries or pores, preserve the natural structures at nano scale, produce high quality polished cross-sections for high resolution SEM imaging and reconstruct accurately the grain boundary and the pore space networks in 3D by serial cross sectioning.

Our first investigations on wet halite and wet clay materials produced unprecedented high quality images of fully preserved fluid-filled pore space as appear in nature. We have thus validated the use of the FIB/BIB-cryo-SEM technology for the in-situ investigations of the elusive structures in wet geomaterials paving the way towards a fuller understanding of how pore geometry can affect physical properties of rocks.

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