CHARACTERIZATION OF THE POROSITY IN BOOM (MOL SITE, BELGIUM) AND OPALINUS (MONT TERRI, SWITZERLAND) CLAYS – ABOUT THE BENEFIT OF USING ION BEAM MILLING TOOLS AND CRYO-SEM

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Claystones 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 the storage of anthropogenic carbon dioxide and radioactive waste in geologic formations. A key factor to understanding the sealing by claystones is a detailed understanding of the morphology of the pore space. The morphology of porosity has a strong effect on many mechanical and transport properties of claystones. Though the bulk expression of these properties are relatively well known for a number of claystones, the relation between nanostructures and macroproperties are poorly studied for a complete understanding of the fluid-rock interactions.

There is a considerable body of literature on the characerization of porosity in claystones using many different techniques (metal injection porosimetry; magnetic susceptibility measurement; SEM; TEM; neutron scattering; NMR spectroscopy, CT scanner; ESEM…). However, the pore space characterization has been mostly indirect until now.

To investigate directly the meso-porosity (from mm to about ten nm in pore size) in claystones, SEM imaging is certainly the most direct approach but, in one hand, it is limited by the poor quality of the investigated surfaces (mainly broken or mechanically polished surfaces including the decoration of porosity by colored resin embedment), which make observation difficult, and the interpretation of the microstructures complicated; and on the other hand, most of conventional methods require dried samples in which the natural structure of pores could be damaged due to the desiccation and dehydration of the clay minerals.

Some recently developed alternatives are able to solve these problems of sample preparation suitable for SEM imaging of pore space at high resolution. The first of them is to use ion beam milling tools able to prepare smooth, damage free cross-sectioned surfaces. Two main ion source are available: (1) a broad ion beam (BIB, up to 3.5mA) is suitable to produce large polished cross-sections area of few mm^2, while the focused ion beam (FIB, 1pA - 45nA) is better used for fine and precise polished cross-sections area of about few μm^2. The second alternative is to preserve the in-situ nanostructures of wet claystones by using cryogenic techniques in order to stabilize in-situ fluids at temperature of liquid nitrogen. A FIB/BIB-cryo-SEM instrument combines cryo-techniques to preserve wet samples, in-situ sample preparation by ion beam cross-sectioning (BIB or FIB) and observations of the stabilized microstructure at high resolution (SEM). Since the ion beam is directly embedded into the SEM, this instrument offers a unique way to get a direct access of the pore space in 3D by using a “slice and view” procedure equivalent to nano-serial-sectionning. This procedure allows us investigating the natural in-situ pore space as a 3D matrix.

This contribution reports on a study of Boom and Opalinus clays from reference sites for research (respectively at Mol site, Belgium and Mont Terri, Switzerland) using cryo-SEM at cryogenic temperature, with ion beam cross-sectioning to prepare smooth, damage free surfaces. Pores commonly have crack-like tips, preferred orientation parallel to bedding and power law size distribution. We define a number of pore types depending on shape and location in the microstructure. 3-D reconstruction by serial cross-sectioning shows 3-D connectivity of the pore space and provide natural pore matrix for simulation of permeability using Lattice Boltzmann method. These findings offer a new insight into the morphology of pores and quantification of porosity down to nano-scale and provide the basis for microstructure-based models of transport in clays.