



Three dimensional analysis of the pore space in fine-grained Boom Clay, using BIB-SEM (broad-ion beam scanning electron microscopy), combined with FIB (focused ion-beam) serial cross-sectioning, pore network modeling and Wood's metal injection

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The Boom Clay is, besides the Ypresian clays, one of the potential host rock materials for radioactive waste disposal in Belgium (Gens et al., 2003; Van Marcke & Laenen, 2005; Verhoef et al., 2011).

To access parameters, which are relevant for the diffusion controlled transport of radionuclides in the material, such as porosity, pore connectivity and permeability, it is crucial to characterize the pore space at high resolution (nm-scale) and in 3D. Focused-ion-beam (FIB) serial cross-sectioning in combination with high resolution scanning electron microscopy (SEM), pore network modeling, Wood's metal injection and broad-ion-beam (BIB) milling, constitute a superior set of methods to characterize the 3D pore space in fine-grained, clayey materials, down to the nm-scale resolution.

In the present study, we identified characteristic 3D pore space morphologies, determined the 3D volume porosity of the material and applied pore network extraction modeling (Dong and Blunt, 2009), to access the connectivity of the pore space and to discriminate between pore bodies and pore throats. Moreover, we used Wood's metal injection (WMI) in combination with BIB-SEM imaging to assess the pore connectivity at a larger scale and even higher resolution.

The FIB-SEM results show a highly ($\sim 90\%$) interconnected pore space in Boom Clay, down to the resolution of $\sim 3E+03\text{ nm}^3$ (voxel-size), with a total volume porosity of $\sim 20\%$. Pore morphologies of large ($> 5E+08\text{ nm}^3$), highly interconnected pores are complex, with high surface area to volume ratios (shape factors $G \sim 0.01$), whereas small ($< 1E+06\text{ nm}^3$), often isolated pores are much more compact and show higher shape factors (G) up to 0.03. WMI in combination with BIB-SEM, down to a resolution of $\sim 50\text{ nm}^2$ pixel-size, indicates an interconnected porosity fraction of $\sim 80\%$, of a total measured 2D porosity of $\sim 20\%$. Determining and distinguishing between pore bodies and pore throats enables us to compare 3D FIB-SEM pore-size distributions to 2D BIB-SEM data, as well as MIP data. Results show a good agreement between the 2D BIB-SEM and 3D FIB-SEM inferred pore-size distributions, indicating the representativeness of our investigations, since BIB-SEM has been carried out on representative elementary areas (REAs) (Hemes et al., 2013).