Deformation of Boom Clay microstructure, due to compaction during Wood's metal injection, as visualized by high resolution scanning electron microscopy and broad-ion beam milling

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Measuring Boom Clay porosity is of major interest for radioactive waste disposal issues. A well established tool for accessing interconnected porosities down to 3 nm in pore throat diameter is Mercury injection Porosimetry (MIP). However, this method is only indirect and no direct observations exist of Mercury filled pore space. Potentially, the pore space is also being compressed, due to the high pressure applied during MIP. Wood's metal has very similar properties to Mercury, but it is less toxic and can be visualized after the injection, using for example scanning electron microscopy (SEM).

In the present study, we injected several materials with the alloy, using pressures up to 300 MPa, thus in theory being able to access pores down to 4 nm in pore throat diameter. Afterwards, the samples were broad-ion-beam (BIB) milled at 40°C, to produce highly polished, flat sample surfaces and prevent the injected Wood's metal from melting. SEM-investigations down to a resolution of 1.2 nm pixel-size, carried out on an intermediate to coarse-grained sample of Boom Clay after Wood's metal injection, show most of the pore space (~ 90 %) being filled. However, the smallest pores, which account for ~ 10 % of the total SEM-resolved porosity, remain unfilled. The Wood's metal seems to be able to enter pores down to at least 7 nm in pore throat diameter, but some of the more fine-grained, clay-rich areas of the sample show features of deformation, due to compaction, resulting in a compression of the pore space, instead of a filling of the pores by the alloy. From a first 2D image analysis, we found a total porosity of 19 %, with 17 % filled porosity and 2 % unfilled. MIP on the same sample results in a total connected porosity of ~ 35 %, indicating that either some of the measured porosity is due to sample compression during MIP, or that the discrepancy between the filled porosities is due to the resolution of the SEM.