BIB-SEM, Cryo-BIB-SEM and Wood's Metal Injection to Image and Analyze Pore Morphology, Pore Connectivity, and Fluid Distribution in Seals and Reservoirs

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Abstract

Pore geometries and associated mineral phases and fluids are important properties in fine-grained rocks such as a range of cap rocks (e.g., chalks, mudstones and shales) and also reservoir rocks such as gas shales, tight gas sandstone and carbonates. Imaging of the pore space using Broad Ion Beam (BIB) milling and Scanning Electron Microscopy (SEM) allows accurate quantification of the pore space from millimeter to nanometer scale resolution on a relatively large area in these kind of rocks, as shown over the past few years in numerous applied studies (Desbois et al., 2011; Hemes et al., 2015; Houben et al., 2013; Klaver et al., 2015a; Norbisrath et al., 2015). From the mm2 size Argon-ion polished sample area we can obtain the mineral porosity by combining image data from SE, BSE and EDX detectors using dedicated image segmentation algorithms to automate the process of porosity analysis based on SEM-Image data from the cm- down to the pixel size (Jiang et al. 2015). Our workflow allows us accurate segmentation of pores and determination of phase porosity. Based on the porosity segmentation, pore statistics and physical properties of the materials, such as pore size distribution and permeability, can be inferred. It was found that the visible pore sizes in fine-grained rocks follow a power-law behaviour from the micrometer down to the nanometer range (Hemes et al., 2013; Houben et al., 2013; Klaver et al., 2012).

Combining BIB-SEM with Wood's Metal Injection (WMI) enables to visualize the preferred transport pathways and determine the controlling pore throat diameter and infer the sealing capacity. The WMI experiments followed by BIB-SEM illustrated the significant effect of fractures on transport pathways and the low connectivity of the clay-rich matrix in mudstones (Klaver et al., 2015b).

BIB-SEM under cryogenic conditions allows direct study of the oil-water-mineral system in hydrocarbon-bearing reservoirs, at resolutions of 10 nm. We quenched a range of mudstones (Desbois et al., 2014; Desbois et al., 2013) and also sandstone reservoir samples equilibrated with oil and brine (Schmatz et al., 2015), to liquid nitrogen temperature and subsequently sectioned them using BIB-cutting under cryogenic conditions. The flat cross-sections with dimensions of 4 mm2 allow cryo-SEM imaging of oil-brine-mineral interfaces, with high-resolution EDX-mapping for phase identification. 3D-reconstruction of capillary contact angles is done using serial sectioning with a distance of 1 μm. Our results call for improvements in models of multiphase pore-scale flow in digital rocks. Further anticipated applications of the method are i. a. the investigation of pore-level mechanisms of EOR or aging processes; the investigation of oil-sands, gas-hydrates, and other sensitive or wet materials; or the investigation of in-situ fluid distribution reservoir- sandstones and carbonates.