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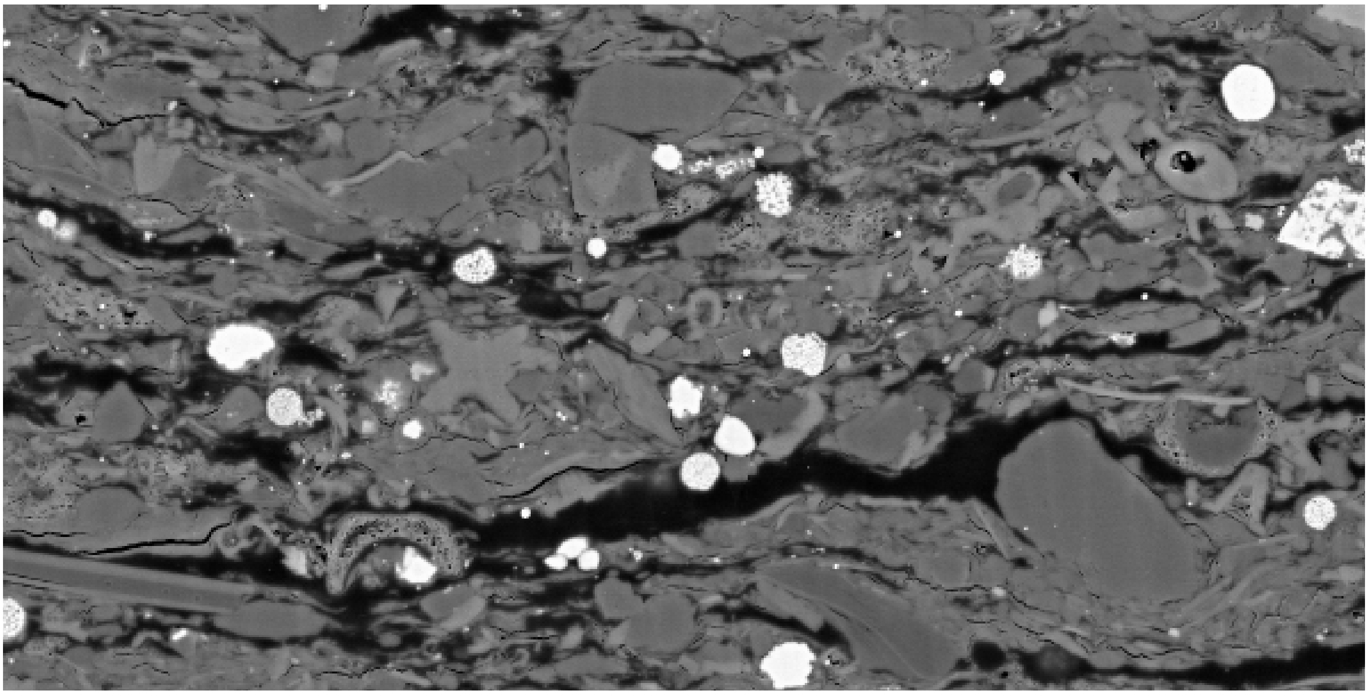
Pore Space Quantification and Distribution in an Organic-rich Shale



High Resolution Backscatter Electron Map of an organic-rich shale

Summary

Physical porosity is a crucial property in shales as it controls the storage capacity to a large extent and forms the transport system. Imaging the visible pore space, the associated mineralogy, and organic matter provide insight into the relevant phases for storage and transport and allow for upscaling. However, it is a challenge to measure the physical porosity in a representative way and at high resolution because of sample heterogeneity as well as small pore sizes, which are typically below the micrometer in those fine-grained rocks. Using our BIB-SEM technology we are able to assess the sample heterogeneity and image the typical pores at nanometer scale resolution, commonly employing a pixel size of 15 nanometer, in a representative way. Moreover, core damage like cracks can be quantified and corrected for bulk porosity and permeability measurements.

Aim

The aim of this study is to measure the pore geometries of the physical pore space in representative elementary area (REA) of an organic-rich shale sample and to derive pore-mineral associations. In addition, cracks due to core damage and shrinkage are quantified.

Sample material and method

The Posidonia Shale sample has a total organic carbon (TOC) of 8 wt. % of mostly kerogen type II (Stockhausen et al., 2015). The mineral composition consists mainly of carbonate grains and fossils, quartz and feld-spars, clay minerals and framboidal and euhedral pyrite grains. Samples were Argon ion-polished by Broad Ion Beam (BIB) milling, which produced a 1.5 mm² flat, damage free cross-section that was subsequently imaged in the SEM (Figure 1). The REA was determined by box-counting of the Back Scatter Electron (BSE) image which was segmented in four phases, namely: 1) Organic matter, 2) Clayey Matrix, 3) Carbonates and 4) Pyrite and other relatively dense minerals (Figure 2). A large carbonate shell was excluded from the analyses (highlighted in Figure 1). High reso-

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lution mapping of the pore space was performed on the area of interest at a pixel size of 15 nm using the Secondary Electron (SE) detector. The pore space was segmented from the SE map by applying advanced BIB-SEM image processing algorithms (Jiang et al., 2015). Mineral related porosity was determined by combining segmented pore space with high resolution BSE (30 nm pixel resolution) and EDS (60 nm pixel resolution) maps.

REA and Porosity

The box-counting shows that the REA for the BIB cross-section is in the order of 100 × 100 µm^2 (Figure 3). Segmented organic matter is 18 ± 1 %, which corresponds to the average TOC of 8 wt. %. The mineral composition of the area of interest has a relatively higher amount of organic matter and clayey matrix and a relatively smaller amount of carbonates and pyrite compared to the whole sample (circles Figure 4). However the differences are relatively small, below 5 %.

The visible porosity in the segmented map (Figure 5) is 2.6 % and contains over 20,000 of pores and about 750 cracks. These cracks acount for about $^1/_3$ of the visible pore space. The pores are mainly below 1 µm in equivalent diameter and follow a power-law distribution indicating fractal geometry (Figure 6). The fractal geometry allows extrapolation of the power-law distribution to smaller pore sizes and comparison to Mercury Intrusion Porosimetry (MIP). It shows that extrapolation of the pore and crack porosity leads to comparable po-

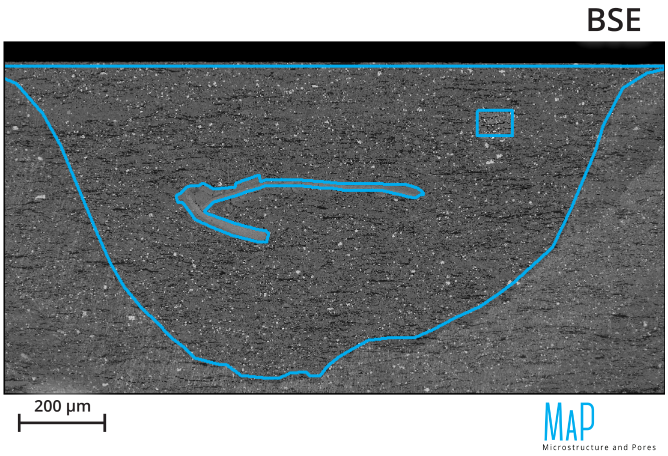

Figure 1: Overview of 1.5mm² cross-section

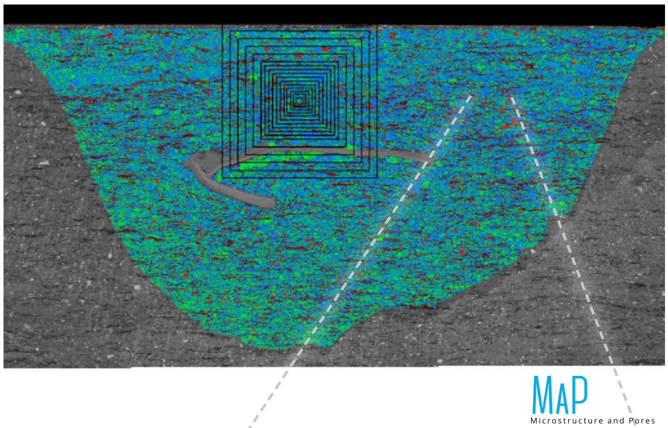

Figure 2: Segmented BSE map

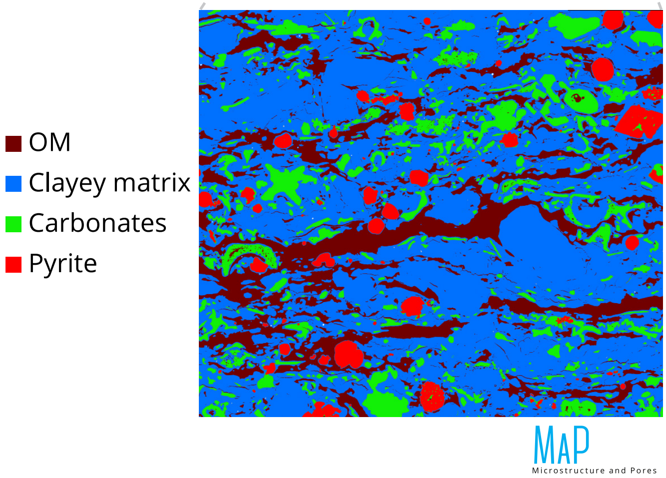

Figure 3: Detail of segmented BSE map

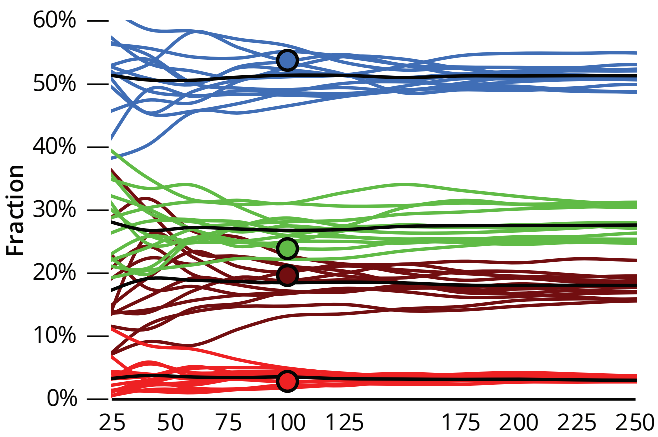

Figure 4: Mineral composition versus box size (lines) and area of interest (circles).

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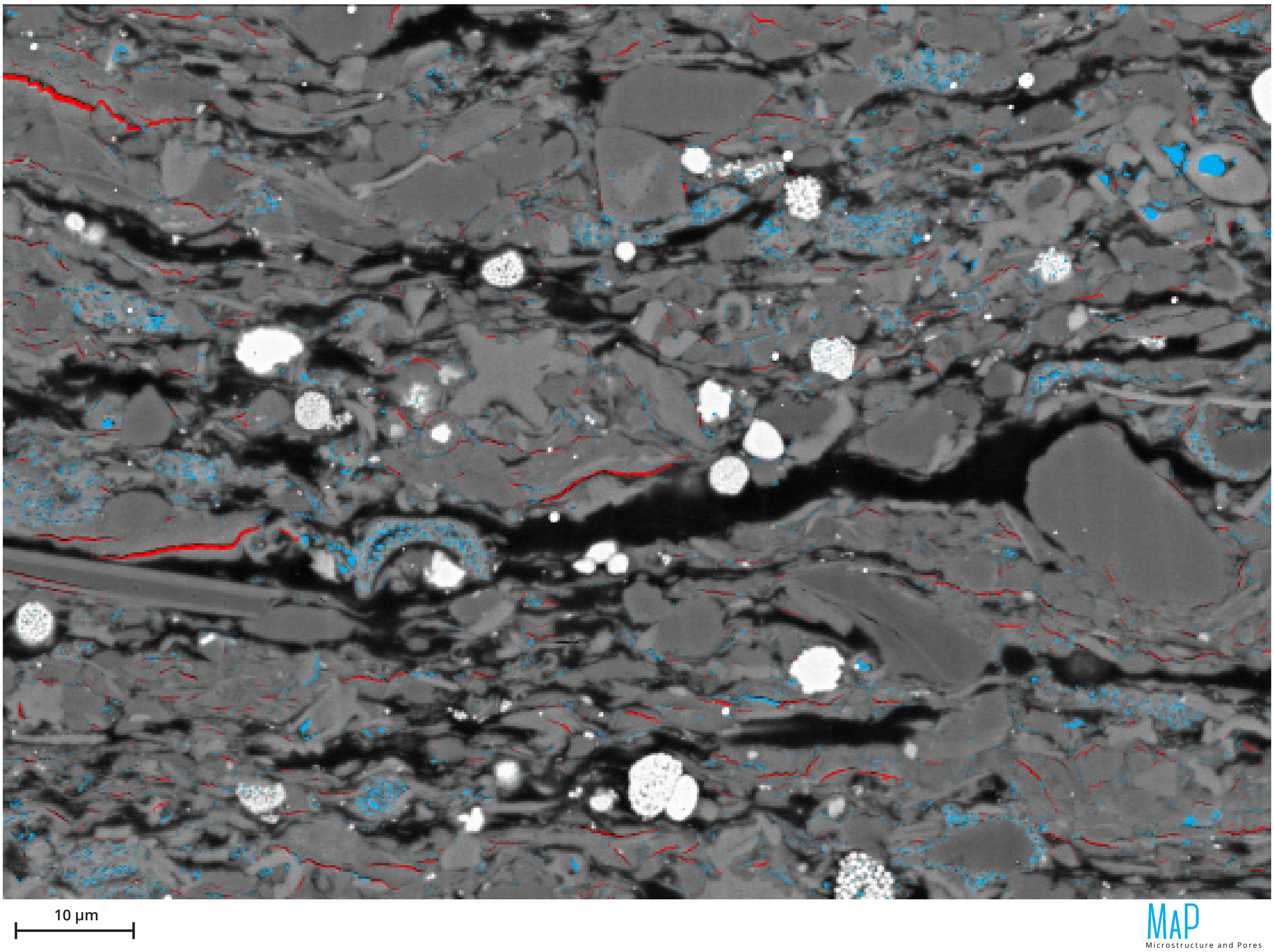

Figure 5: BSE high resolution map overlaid over the segmented pores in cyan and identified cracks in red.

rosity as found with MIP (Figure 7), indicating that MIP does not take into acount the cracks discovered by SEM imaging (Figure 5). Moreover the extrapolation of the imaged pores only leads to higher porosity compared to MIP, which is due to the intraparticle pores unconnected for the Mercury.

Combining the segmented pore space with the BSE and the EDS maps revealed that only a small fraction of the pores is located within the organic matter (Figure 8). Most of the pores are associated with carbonate grains and fossils. The other part of the pore space is mad eup of interparticle pores between the clay minerals, quartz, feldspar and carbonate grains.

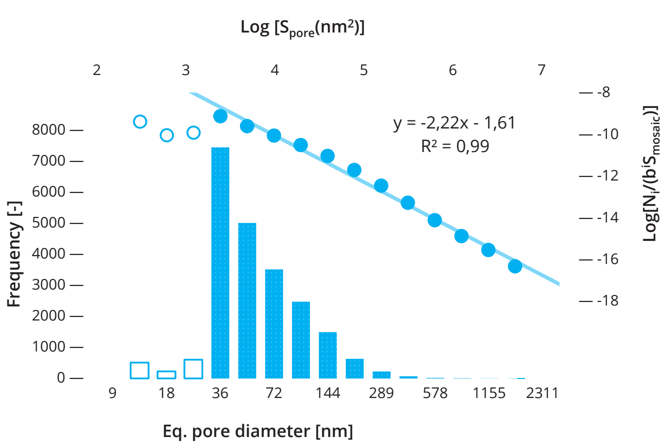

Figure 6: Pore size distribution and normalized pore size distribution (Klaver et al, 2012) on the right.

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Take home messages

→ Using BIB-SEM we are able to analyse microstructure and porosity in a representative area in fine grained materials whilst correcting for core damage.

→ Quantification of the visible pore sizes can be compared to bulk porosity measurements.

→ Combining different types of image data enables advanced pore-mineral associations and analyses.

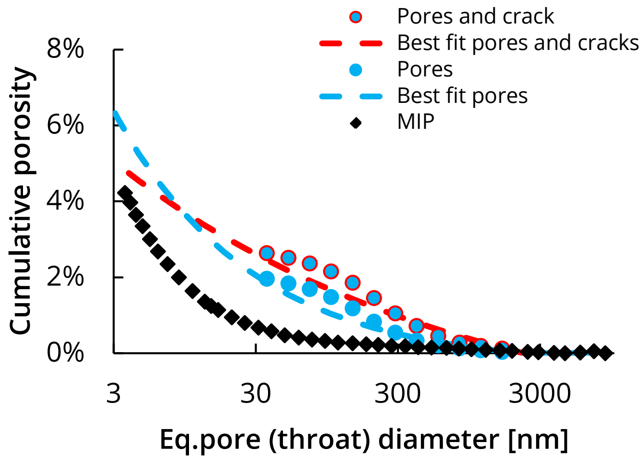

Figure 7: Comparison of MICP, BIB-SEM and extrapolated pore (throat) size versus cumulative porosity.

Acknowledgment

Sample material was provided by Prof. L. Schwark and M. Stockhausen, University of Kiel.

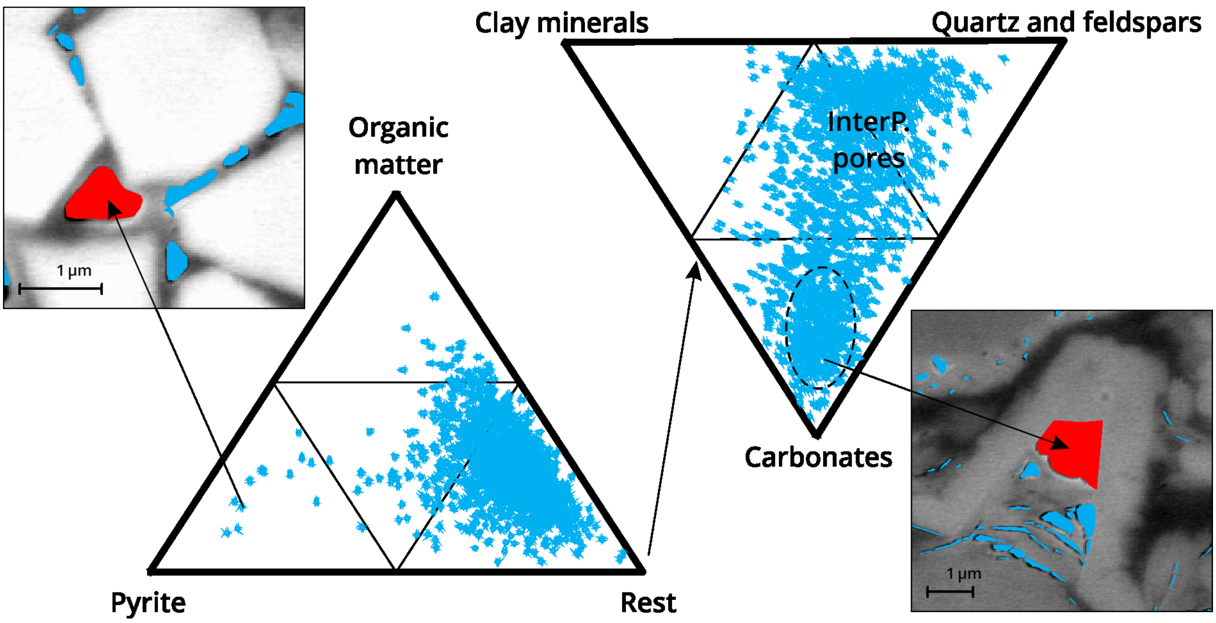

Figure 8: Pore-mineral associations based on EDS data and interpreted mineralogy.

Further reading

Jiang, M., Klaver, J., Schmatz, J., Urai, J.L., 2015. Nanoscale porosity analysis in geological materials, 14th ICSIA. Acta Stereologica, Proceedings ICSIA, Liege, Belgium.

Klaver, J., Desbois, G., Urai, J.L., Littke, R., 2012. BIB-SEM study of the pore space morphology in early mature Posidonia Shale from the Hils area, Germany. International Journal of Coal Geology 103, 12-25.

Stockhausen, M., 2015. Experimental simulation of hydrocarbon expulsion, Dissertation, Kiel University.

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