Nanoindentation mapping for determining representative mechanical parameters of clay matrix in mudstones: A new tool for top seal characterization

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Nanoindentation is ideal for measuring micro- to nano-scale mechanical properties and structural heterogeneities, and is therefore increasingly applied to fine-grained sedimentary rocks which may serve as top seals of geological reservoirs. Traditional mechanical testing poses a challenge for such rocks, as i) the preparation of a well-defined macroscopic specimen is often impossible and ii) the availability of core material from underground storage prospects is limited. Knowledge of the mechanical behaviour of mudstones seals is important to understand their fracture behaviour which may cause mechanical seal failure due to microfracturing as a response to various geological processes (e.g., buoyancy pressure from the reservoir). In this contribution, first results of high-speed nanoindentation mapping on the clay matrix of top seals from the Vienna Basin are presented. The nanoindentation grids were deployed on clay matrix areas, as matrix-related pores are of great importance for the evaluation of pore network connectivity and overall mechanical strength. The nanoindentation results were then post-processed with machine learning-based tools (e.g., k-means clustering) to remove noise resulting from sub-optimal surface quality or the testing of mixed phases. Feasibility experiments were carried out using a Hysitron TS 77 Select (Bruker) equipped with a Berkovich diamond tip on one sample with two map arrays (7 × 7 indents). To test the depth sensitivity, the indentation loads were increased continuously from 500 µN to 1,500 µN over a total of 49 indentations, corresponding to an increase in indentation depth from ~150 nm to 450 nm, respectively. Indents on the matrix area were selected based on broad ion beam-scanning electron microscopy (BIB-SEM) maps and further analysed in correlation with the load-displacement data to exclude undesired effects such as the pop-in effect by sudden displacement or no loading due to existing cracks. The initial results show that despite the highly heterogeneous phase distribution in mudstones, mechanical parameters obtained from their clay matrix are not sensitive to indentation depths, and hence representative values can be determined from minimum volumes with statistical significance. The resulting average reduced elastic modulus (Er) and hardness (H) values of the clay matrix range at 16.32 ± 7.25 GPa and 0.47 ± 0.35 GPa, respectively for the tested sample. The established workflow was then applied to a vertical top seal section cored in the Vienna Basin well Bo-209 (Bockfließ), and vertical property changes (elasto-plastic deformation behaviour, Er, H) were linked to other rock properties such as bulk mineralogy, bulk geochemical indicators for hydrocarbon leakage (e.g., Rock-Eval S1 and production index) as well as porosity, pore geometry, and grain size distributions calculated from BIBSEM maps. In conclusion, this contribution aims at introducing high-speed nanoindentation mapping as a feasible high throughput tool for the mechanical characterization of mudstones.