Nanoindentation: A new workflow for spatially-resolved micromechanical investigations on sedimentary organic matter

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As a source for thermally generated oil and gas, sedimentary organic matter plays an important role in geoenergy-related research. Its pore structural and micromechanical properties need to be understood in order to predict matrix and fracture transport processes in organic matter-rich rocks including coals, which may serve as unconventional hydrocarbon and potential secondary gas storage (e.g., CO_2) reservoirs. This contribution introduces nanoindentation as a novel technique to obtain spatially-resolved micromechanical properties (e.g., hardness H and elastic modulus Er) at a micro- to nanometer length scale for both coals and organic-rich shales. A nanoindentation study on Carboniferous coals from the Ukrainian Donets Basin (vitrinite reflectance range from 0.62 to 1.47 %Rr) shows highly variable properties for the three different maceral groups (vitrinite, liptinite, inertinite). While inertinite H and Er are exclusively controlled by the conditions during primary inertinite formation (paleo-wildfire temperature), vitrinite and liptinite are affected by both primary depositional (e.g., sulphur content, mineral matter inclusions, primary pore structure) and secondary thermal maturation-related (e.g., bitumen generation) processes. A pore structural control on micromechanical properties of vitrinite is indicated by the correlation between Er and average nanopore diameters obtained by high-resolution transmission electron microscopy, which in some cases is obscured by pore occlusion by bitumen impregnations particularly at peak oil maturity (~0.9 %Rr). While nanoindentation of coals is relatively straight forward due to their organic richness and the easily identifiable maceral types, an improved approach was necessary to establish nanoindentation as a characterization tool for the finely dispersed organic matter in shales. An overmature sample set (vitrinite reflectance range of 1.33 to 2.23 %Rr) from the Lower Cretaceous Shahezi Formation in the Songliao Basin was selected in order to test for pore structural and resulting micromechanical changes of vitrinite and secondary pyrobitumen macerals. High-speed nanoindentation mapping was combined with multiple high-resolution imaging techniques and a new preparation approach by femtosecond laser treatment, and the results were analysed by k-means clustering in order to eliminate grain boundary and heterogeneity (e.g., mineral inclusion) effects. Adjusted for these influencing factors, a declining Er trend of organic matter with increasing thermal maturity is visible for samples up to 1.96 %Rr, at which point Er remains relatively constant with a slight indicated increase towards the overmature range arguably as an effect of severe compaction. The representative Er clusters determined for vitrinite and solid bitumen range significantly lower compared to previous nanoindentation studies on organic constituents in shales, and clearly show that nanoindentation mapping is capable of providing reliable micromechanical properties even for disseminated organic matter at a particle size in the range of micrometers. The established femtosecond laser marking technique enabled a comparative imaging study, which helped to explain outliers (e.g., due to indentation on cracks or at positions with poor surface quality) and systematically shifted clusters (e.g., areas near grain boundaries with harder mineral matter) identified by the machine learning algorithm.