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Development of an in-situ apparatus for synchrotron-based surface scattering measurements at mineral–water interfaces in elevated pressure and temperature conditions

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Mineral–water interfaces are primary sites for various chemical reactions in nature. Many of these reactions occur in subsurface environments where elevated pressure and temperature can alter both the chemistry of solutes and the stability of mineral surfaces, which in turn influence interfacial reactivity. Here, we report a new in-situ apparatus that is designed for synchrotron-based surface scattering measurements in these nonambient conditions. This custom-built cell, made of an anti-corrosive Ni-based alloy (Alloy 625), has a miniaturized reaction chamber that contains a small volume (~10 µL) of a fluid in contact with a small active area (~5 mm^2) of a mineral surface. This interfacial environment is encapsulated with a pair of diamond discs as X-ray windows, which are stable under operation pressures up to ~40 MPa (= ~6,000 psi) and temperatures up to ~400 °C. The cell performance is validated by collecting a series of in-situ high-resolution X-ray reflectivity (XR) at the barite (001)–water interface as a function of temperature from 25 °C to 200 °C. Challenges in the measurement and data analysis are also discussed based on the comparison with those using other XR cell setups. Overall, the development provides us a new opportunity for the research on this currently unexplored area of geochemistry at mineral–water interfaces in extreme conditions.

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