Development of a Flexible In-Situ Cell for Structural Studies of Hydro/Solvothermal Chemistry under Sub- and Supercritical Conditions

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Traditional solid-state structural studies have depended on an examination of samples using diffraction and/or spectroscopic techniques under ambient conditions following synthesis. However, advancements in sample environments and new instrumentation over the last decades have enabled the feasibility of *in situ* and *operando* structural probes. Experiments of this type are routinely performed at large scale user facilities [1], but also are increasingly possible with increasingly performant laboratory-based equipment [2-3]. This work presents the development and application of a flexible in situ cell designed for structural studies of hydro/solvothermal chemistries across a wide range of conditions, from static ambient to supercritical flow. Details of the technical challenges and experimental compromises required for successful lab-based *in situ* analysis are detailed. The design incorporates modularity for adaptability for various probes and beamline experiments.

Recent scientific examples are presented, spotlighting the types of chemistry which can be explored between sub- to supercritical regimes under both batch and flow conditions. In particular, the continuous supercritical flow synthesis of nano-scale ceria-yttria-zirconia materials is examined; with a goal to better optimize polymorph formation of these materials for enhanced catalytic properties [4]. Additionally, the synthesis and phase transformations of mixed anion compounds (hydroxides, carbonates, halides, etc.) under hydro/solvothermal conditions are investigated, with a specific focus on elucidating the rich structural diversity and stability of phases within these systems. Finally, ongoing efforts to extend the modular functionality of this cell for spectroscopic and synchrotron methods (including total scattering) will be discussed.

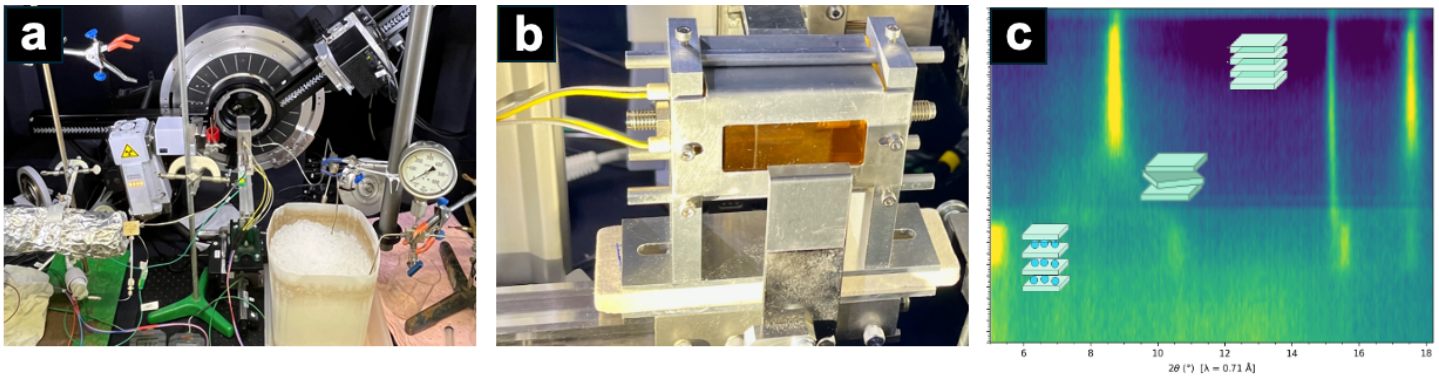


Figure 1. (a) Image of in situ lab diffraction setup under supercritical flow conditions, (b) Zoomed image of mounted in situ cell assembly, and (c) contour plot (temperature *vs* 2θ) revealing structural phase evolution as a function of heating.

[1] M. Roelsgaard, et al., Journal of Applied Crystallography (2023) 56, 581-588.
[2] F. H. Gjørup, et al., Review of Scientific Instruments (2019) 90, 073902.
[3] S. Vornholt, et al., Journal of Applied Crystallography (2025) 58, 1852-1858.
[4] A. Auxéméry, et al., Chemistry of Materials (2020) 32, 8169-8181
[5] H. Hyrondelle, et al., Chemical Reviews (2025) 125, 4287-4358.

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