Poster

Design, Implementation, and Performance of Operando Anomalous Powder X-ray Diffraction (AXRD) Interleaved with X-ray Absorption Spectroscopy (XAS) Using a Scanning 2D Imaging Detector at the XMaS Beamline

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We present a method for achieving high-quality anomalous powder X-ray diffraction (AXRD) by employing a flat panel imaging detector scanning over a large angular range, coupled with transmission X-ray absorption spectroscopy (XAS) (Fig. 1). Maintaining an adequate Q-range becomes particularly challenging in experiments involving the collection of AXRD data at atomic absorption edges, which are often found at relatively low energies. However, these techniques can be highly valuable for in situ experiments as they enable the isolation of specific elements within a crystal structure and the identification of active sites involved in specific reactions.

Our proposed method allows for the collection of a Q-range ($Å^{-1}$) that is not achievable using static flat panel detectors in the absorption energy regimes utilized (8.94 – 17.5 keV). It simultaneously compensates for preferential orientation and graininess, which static flat panels are less sensitive to than 1D detectors. This approach has demonstrated its ability to produce high-quality data, which enables determining the atomic positions of reactive copper atoms in a Cu-MAZ zeolite crystal structure during variable temperature and aerobic activation. Additionally, we have shown that this method can be easily combined with XAS, allowing for quantitative establishment of the chemical speciation of copper, particularly the partitioning of Cu(II) in both hydrated and dehydrated states, at each step.

Compared to static flat panel detectors, multi-crystal arrays, or arc detectors, our method offers several distinct advantages. We believe that this approach to operando (A)XRD could find widespread application in many fields of material research where precise and operando understanding of structure and speciation is highly sought after.

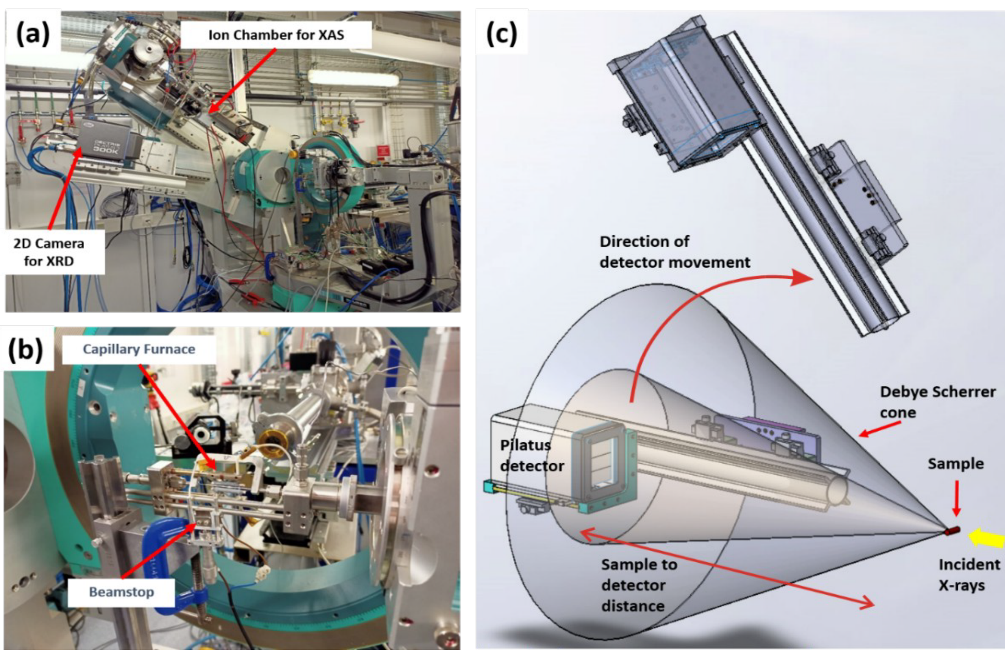

Figure 1. The experimental setup for the measurement of AXRD using a scanning flat panel detector, as well as its interleaving with transmission XAS. Panel (a) shows the mounting of the flat panel detector and ion chambers on independent arms along one axis (in the direction of the X-ray beam) of a 6-circle Huber diffractometer. Panel (b) depicts the positioning of the sample reactor at the centre of the axis of rotation, along with the placement of the beam stop. Panel (c) provides a schematic representation indicating the direction and range of movement of the Dectris Pilatus 3 300K detector.