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Instrumentation to Promote In-Situ Experiments at the Powder X-ray Diffraction Beamline (PAINEIRA) at SIRIUS

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PAINEIRA beamline at the SIRIUS synchrotron laboratory in Brazil is a facility dedicated to the X-ray diffraction characterization of polycrystalline materials. In addition to the high photon flux, it presents an arc-shaped 2D detector covering 109° in 2θ (in-house development). These provide high-quality and fast acquisition of the XRD pattern at the beamline, which is suitable for in-situ experiments. Thus, several systems were designed and implemented to enable kinetics while acquiring the structural data. First, two types of capillary cell reactors were projected to be installed on the center of the 3-circle diffractometer of the beamline. One operates for ambient pressure experiments and the other for up to 100 bar. Second, a module (Fig. 1 and 2) presenting five mass flow meters, pneumatic valves with automatic actuators, one-way valves, a saturator, a heating system, pressure sensors, and back pressure (Fig. 2) was developed to select and control fluids used in the experiments up to 100 bar. It is installed close to the diffractometer (Fig. 1) and connected to the capillary cells via peek tubes. A vapor phase can be delivered to the experiment through a controlled flow of He, which carries the vapor pressure of a liquid inside the saturator. A liquid phase can be supplied with an injection pump. All fluids can flow through the capillary cell or bypass. A PyQt interface from the beamline controller computer automatically controls the module. Finally, open-source software (Iguape) is being developed to allow instantaneous visualization of the XRD patterns acquired during kinetic or static experiments. Iguape will also plot auxiliary graphs exhibiting the peak position, the full width of the height maximum (FWHM), and the peak area evolution of a specific diffraction peak selected by the user. This set of instruments is a user-friendly system that improves the efficiency of in-situ experiments at the Paineira beamline.

I plan to submit also conference proceedings

Yes

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