In situ study of chemical synthesis using high-energy X-ray diffraction at beamline I12 (Diamond Light Source)

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The crystallisation of various materials from solution is an important area of study in the field of *in situ* X-ray diffraction. Beamline I12 at Diamond synchrotron offers the improved experimental capabilities for *in situ* investigation of the large-scale synthesis process in unprecedented detail [1]. Time-resolved monochromatic high energy X-ray diffraction on the Beamline I12 is a fast and efficient method for investigation of crystallization allowing the detection of crystalline intermediates, formulating an idea about the crystallization mechanism, and the assessment of individual reaction parameters, i.e., reaction rate constants and activation energies. Thus, the optimization of the synthesis conditions of new compounds can be achieved. The high X-ray flux on the beamline I12 allows real-time monitoring the synthesis in the large containers, including standard laboratory metal autoclaves. Using monochromatic X-rays for the synchrotron experiments produces the high-quality diffraction data that permits the full structural refinements to be undertaken on metastable materials observed during the reaction.

The simplest experimental setup for low temperature *in situ* diffraction experiments is a metal heating block, which allows measurements during the synthesis from room temperature to approx. 90^{o}C. It can be used with magnetic hotplate stirrer, allowing to mix substance during the measurements providing the homogeneous distribution of material in the reaction tube. For synthesis at temperatures close to the room temperature, the remotely controlled syringe pump can be used allowing simultaneous or sequential adding the reactants, thus permitting the investigation of the reaction in the controlled way from the very early stages [2].

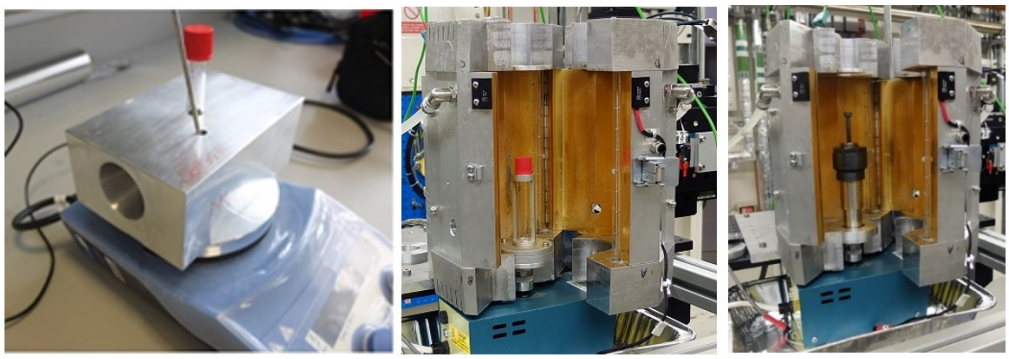

Figure 1. Custom design metal heating block for in situ chemistry measurements with magnetic hotplate stirrer (left); ODISC furnace with quartz tube inside and magnetic stirrer below (middle); ODISC furnace with metal autoclave inside and magnetic stirrer below (right).

For more demanding *in situ* synthesis – at temperatures above 100^{o}C or in metal autoclaves – the custom designed furnace ODISC was developed on the beamline I12 [3]. The furnace is very versatile with integrated heating, stirring, and precise sample centring and it can be used for a wide range of *in situ* experiments on the beamline. On the beamline I12, the furnace ODISC can be used in two configurations: *1) in situ* measurements of reaction kinetic during solvothermal synthesis experiments, which performed at temperatures below boiling temperature of the solvent. In this case simple quartz tubes are used as a container during large-scale *in situ* synthesis [4]. *2) in situ* measurements of reaction kinetic during hydrothermal synthesis, which should be performed in metal autoclaves. Despite measurements during the crystallization were performed in the metal autoclave, the data quality recorded on the beamline I12 allowed the refinement of the diffraction data and subsequent analysis of crystallization kinetic [5].The references should be in Heading 4 style (Times New Roman 9 pt, shortcut CTRL + NUM 4) and listed immediately at the end of the text without a heading.

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