

MS43-P9 New Facility for Long Duration Experiments at Diamond Light Source

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The high brightness beamline I11 at Diamond Light Source is a dedicated powder diffraction instrument which has been in user operation for a number of years. Equipped with multi-analysing crystals (MAC) and fast position-sensitive detectors, it is routinely used for high-resolution and time-resolved experiments [1-3]. Recently, a new facility for long duration experiments (LDE) has been added. Now in operation mode, this facility houses the necessary hardware and equipment for multiple LDE studies. These experiments are mounted on a large sample table equipped with adjustable linear drives to automatically and periodically move sample cells in and out of the beam and the Pixium area detector. LDE are set up and left in place with programmed automated data collections over periods of months or years. Sample environments such as electrochemical cyclers, incubators, heating stages, environmental chambers and high pressure gas cells are accommodated for user operation.

To complement the existing I11 facilities, the LDE hutch opens up new opportunities for those experiments which require weeks to months of periodically monitoring "slow" changes, up to two years. It is of particular benefit to research areas where important information on the development of phases over time cannot be obtained via ex-situ methods. Areas that benefit include studies of crystallization, battery materials, gas storage, mineral evolution, seasonal effects, thermal and electrical cycling and corrosion science. With a versatile design and many automated features such as robotic sample changers, the upgraded beamline is used by many academic researchers from diverse scientific backgrounds and industries. Recent results from LDE experiments on cement have revealed a new type of cement which is up to 50% more effective than previously proposed barrier solutions for containing nuclear waste in deep geological disposal facilities [4].

References:

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Keywords: Powder Diffraction, In-situ Measurements, Synchrotron Instrumentation

MS43-P10 Characterization of amorphous high-surface area magnesium carbonate (Upsalite) using laboratory diffractometer

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Micro- and mesoporous inorganic solids with a large surface areas and controllable pore size are extensively studied due to the numerous potential applications for this type of materials as catalysis and sorption media, for gas storage and separation, as vaccine adjuvants and drug delivery vehicles, in regeneration of bone tissue, in cosmetics, etc. Need for the materials with a disordered interconnected pore network easily accessible for external components, possibility of *in situ* loading of the pores with active agents during synthesis process as well as reduction of production cost and environmental impact are the driving forces for the development of template-free synthesis of porous materials. Recently Frykstrand et al. [1,2] reported the formation of a mesoporous, extraordinary moisture-absorbing, amorphous, high-surface area magnesium carbonate (Upsalite) synthesized without the use of surfactants as pore forming agents. In the attempt to understand the pore formation mechanism in Upsalite and to characterize the properties of this novel material a number of experimental techniques, including electron microscopy, X-ray scattering and spectroscopy techniques, infrared spectroscopy, were used. The outcome of this comprehensive study will be published separately (Cheung et al., in preparation). Here we present the results of small-angle X-ray scattering (SAXS) and total X-ray scattering for pair distribution function (PDF) study of Upsalite.

PDF analysis enabled characterization of short and long-range order in the amorphous Upsalite. The majority of PDF features are explained by magnesium carbonate alone, but the quality of the fitting is improved by including MgO. The PDF results are in agreement with the high-resolution TEM and XPS suggesting composite nature of Upsalite (MgO/MgCO₃).

SAXS experiment confirmed the mesoporous structure of Upsalite. Furthermore, using SAXS at controlled temperature and relative humidity the water sorption process and its effect on the pore structure of Upsalite was characterized *in situ*.

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Keywords: mesoporous, Upsalite, SAXS, PDF, *in situ*