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Time-resolved micro-tomography of dynamic systems at the Australian Synchrotron

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The high x-ray flux of synchrotron sources offers the possibility of time-resolved micro-CT experiments to explore the 3D structure of systems which are changing with time. At the Imaging and Medical Beamline at the Australian Synchrotron, hardware upgrades have made it possible to acquire a micro-CT scan in as little as 10s. This capability is illustrated with two examples showing evolution of 3D structures in time.

The first example is a study of gas diffusion into coal. Coal is an important source rock for methane and also a potential reservoir for carbon storage. Key to these applications is an understanding of how gases move through the micro-structure of this very complex and heterogenous material. This study was performed by observing the flow of xenon gas into a selection of coal samples. K-edge subtraction was used to separate the 3D images of the coal from the xenon in order to get a quantiative understanding of the spatial and temporal variation in gas take-up .

The second example is a study of the rising and baking of a range of bread doughs made with different formulations. These experiments required the fastest scanning speeds the IMBL was capable of at the time, taking about 20s per scan. The dough structure was observed during rising and then baking to investigate the differences between the behaviour of dough made from low and high protein flour, and with different salt additives..

Rapid time-resolved scanning presents an additional challenge compared to more typical micro-CT experiments as the quantity of data generated in a single 3 day experiment can easily be several terabytes. The dough experiment for example generated over 460 individual CT scans requiring processing and analysis. Approaches to dealing with these challenges will be addressed.

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In situ applications of soft X-ray ptychography

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Coherent diffractive imaging (CDI) with synchrotron X-ray beams allows extended objects to be characterised at high spatial resolution (<30 nm) and high energy resolution (0.1 eV). In an implementation of CDI known as ptychography, a far-field diffraction pattern is measured from many overlapping regions as the sample is scanned through a coherent X-ray beam. Quantitative images of an object are then obtained from the far-field intensity pattern through iterative reconstruction algorithms. This provides a unique method of studying the elemental and chemical-state distributions in relatively thick materials and their relationship to nanoscale morphology. The high coherent flux offered by synchrotron X-ray sources can also potentially allow high temporal resolution through the use of emerging detector technology and advanced image reconstruction algorithms. This in turn allows the nanoscale structure of functional materials to be studied under non-equilibrium real-time conditions. In this work, we review recent efforts to apply soft X-ray ptychography to in situ and operando applications at several synchrotron facilities. We emphasise studies of functional materials that are characterised by heterogeneity over a range of relevant length scales, including energy storage materials based on polypyrrole nanocomposites and inorganic, aluminosilicate based ceramics. Finally, a perspective on the future prospects of the method will be given, with particular attention to how experimental challenges can be overcome to achieve the spatiotemporal resolution limits defined by the available coherent flux from synchrotron light sources.

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Hydrogen release mechanisms in bulk MgH_2 by ultra-high voltage transmission electron microscopies

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Ultra-high voltage transmission electron microscopy (UHV-TEM) has significant advantages compared with conventional TEM including (1) higher resolution due to the shorter wave length of incident electron beams, (2) the minimization of surface effects and the ability to image a thicker specimen, and (3) the inelastic incident beam interaction with the sample atoms is less than occurs at lower voltages and, as a result, less pronounced 'electronic excitation' occurs. Here we report on the real-time atomic level observation of hydrogen release behavior in MgH_2 particles at a grain size of a few micrometers, which is at a scale applicable to real industrial hydrogen storage technologies. Techniques used included temperature controlled UHV-TEM with supporting synchrotron atmospheric and temperature controlled X-ray diffraction, X-ray absorption fine structure analysis and DSC. These observations help elucidate the fundamental mechanisms of hydrogen release and help with the development of high performance and safe hydrogen storage stations for fuel cell vehicles in the form of solid metal hydrides. This paper focuses on characterising and comparing the hydrogen release behavior in MgH_2 particles using two different TEMs with acceleration voltages of 1,000kV (JEM-1000, EM-HSTH heating holder) and 200kV (JEM-2100HCLM, GATAN Model 652 double tilt heating holder). The differences of the obtained hydrogen release behaviors were, (1) the hydrogen release mechanism from bulk (2 μm) MgH_2 particles observed at high voltage is based on the growth of multiple pre-existing Mg crystallites within the MgH_2 matrix, present due to the difficulty of fully transforming all Mg during a hydrogenation cycle. In comparison in thin samples (2) analogous to nano-powders, dehydriding occurs by a 'shrinking core' mechanism. The sample thickness and electron