MI-1037 Development of X-ray imaging

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The long term proposal MI1037 failed to be granted as the panel felt that the proposed project aims could be met by the standard proposal route and consequently 18 shifts of ID15 were awarded. Our aim in the project is to develop the X-ray imaging methods (described in the application) and to use these for studying the preparation and operation of supported catalysts. We proposed a mixture of different tomographic methods. We report here our initial progress on this project.

Collimator testing: We successfully tested a variety of collimators for coupling to array energy-dispersive detectors.

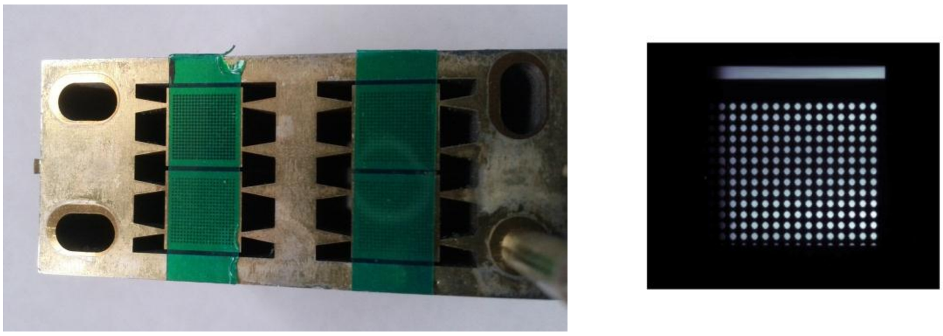

Figure 1 Left, array collimators of different sizes (200,150, 100 and 75µm); right, radiograph of portion of 200 µm array collimator at 120 keV showing that it does indeed collimate. Such pictures are obtained for all collimators at a range of energies. An 80×80 array collimator is now being manufactured using the same construction technique for coupling to the 80×80 pixelated energy dispersive device.

Preparation of Molybdenum Oxide catalyst bodies: We investigated the preparation of such catalysts by a combination of standard micro-CT and XRD-CT. In situ studies were performed on samples wetted (so called impregnation) with various metal pre-cursor solutions followed by a fixed drying periods, in order to emulate the industrial preparation processes.

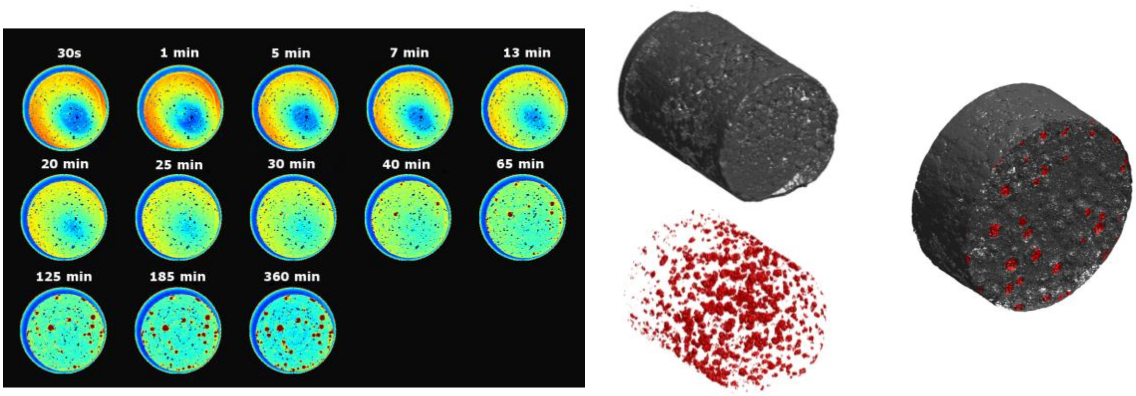

Figure 2 Left, shows a cross-section slice from time resolved micro-CT of an alumina extrudate (extruded alumina cylinder) impregnated with 1.5 M ammonium hepta-molybdate sample solution. At this concentration and conditions, formation of hot-spots (high catalyst concentration) is found to occur. These are found to be distributed down the length of the extrudate; the 3D images on the right indicate the hotspots. Since we have a full 3D information at each time point we can monitor how these hot spots form and grow. The images in the **centre-right**, indicate the distributions of the hotspots in the complete extrudate body where the contribution of the alumina is mapped in gray and from the Mo in red. The image on the **right** shows a thick section through the body. We are able to identify the Mo species using XRD-CT (see figure 3 below).

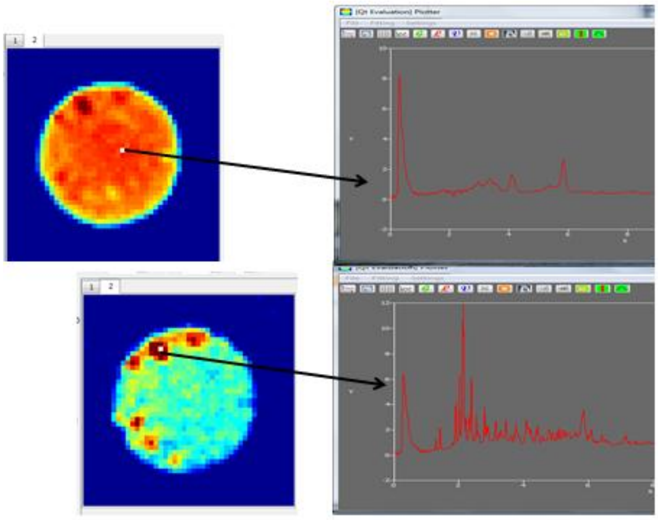

Figure 3. Some XRD-CT results from a catalyst body where hot-spots have formed. Both images correspond to the same recorded slice but showing the distribution of two selected features in the recorded diffraction. Though XRD-CT currently offers limited spatial resolution (since there is a trade-off between this and collection time) the method is extremely powerful, as the solid-state phases found in each volume element can be identified/quantified from their characteristic diffraction signature and subsequently mapped. This was used to identify/quantify the material in our hotspots and correlate the distributions to the micro-CT images described above. The XRD-CT slice described in the figure took ~600 s to acquire.