Imaging a supercritical water crystallisation vessel using in-situ diffraction (MA387)

Our aim in this experiment was use a combination of high-energy angle- and energy-dispersive diffraction to probe the interior of a continuous hydrothermal flow synthesis (CHFS) reactor *in-operando*; CHFS provides a route for the controlled synthesis of nano-particle ceramics. In the beamtime awarded we achieved a number of objectives which are described below.

With a great deal of help from the ID15 staff, the desired experimental set-up was achived, including the integration of our large movement *xyz* stage with the station software. The setup allowed fast switching between angle and energy-dispersive modes and provided rapid and large sample translation for spatial studies. The setup took slightly longer than expected, but was, nevertheless, a requirement of the experiment; the experience will significantly shorten the setup time of our forthcoming experiment (CH2645, which requires an identical setup). The experimental setup was used to monitor the controlled synthesis of CeO_2. TEDDI (tomographic energy-dispersive diffraction imaging) was used to image both diffraction and fluorescence signals from within the CHFS reactor. The technique was shown to be sensitive to material depositing at specific locations on the interior of the reactor steel tubing. Some example results are given below. Spatial scanning angle-dispersive diffraction was also carried out. This was shown to be much more sensitive to material crystallising and derived maps of the changing crystallite size within the reactor have been defined.

The study represents the first comparison of the two imaging techniques and the first '*look*' inside an in-operando CHFS reactor. The results are impressive and these are being prepared for submission to a high ranking journal. **Dr Simon Jacques. March 2008**

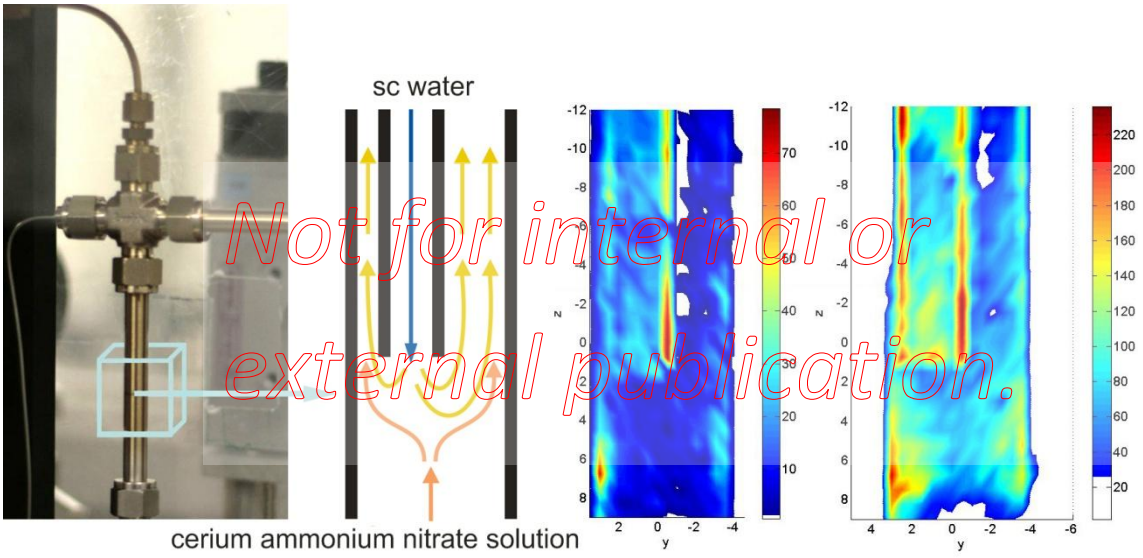

Fig. The *CHFS* counter-current mixer and *TEDDI* results: **(A)** Photograph and schematic illustration showing the counter-current mixer design for continuous production under supercritical water conditions (22.1 MPa and 374 ºC) of nano-sized CeO_2 particles. The inner and outer stainless steel tubes have diameters of 3.2 and 9.5 mm respectively. **(B)** YZ *TEDDI*-image based on intensities from the ceria *113* reflection observed at 108.5 keV [*TEDDI* scan = 23 x 29 points, pixel size = 0.5 x 0.75 mm, collection time = 5 sec per point, total scan time 68 minutes]. **(C)** Similar scan to (B) but obtained during the post-production water-wash cycle. The build up of ceria product on the walls of the inner steel tube and some regions of the outer pipe (inner wall) is clearly evident in B and C.