

Chemical imaging of single catalyst particles with scanning μ -XANES-CT and μ -XRF-CT: Supplementary Information

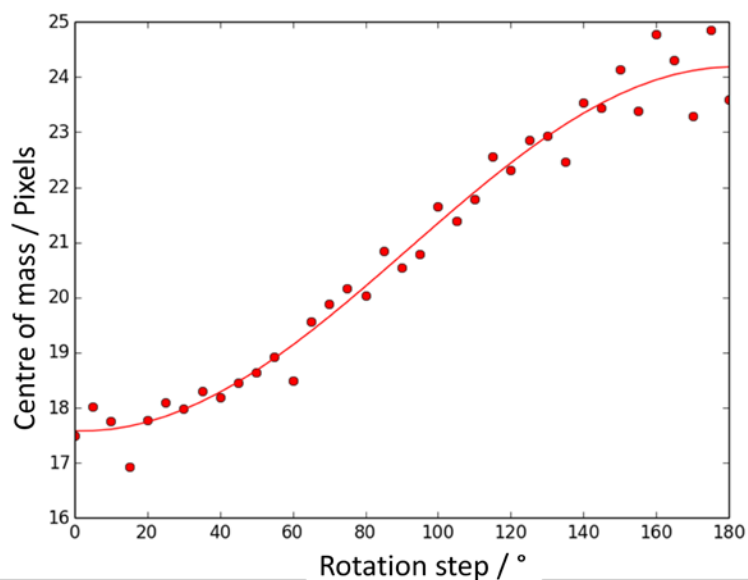


Fig 1: Determination of centre of mass shift and centre of rotation adjustment for sinogram correction.

XRD collection

Powder XRD data were collected on a Bruker AXS D8 Advance diffractometer in parallel beam mode with a 60 mm Gobel mirror, and a Ni filtered Cu K_{α} X-ray source. XRD patterns were collected over a scan range of 20-80 $^{\circ} 2\theta$ with a 0.022 $^{\circ}$ step size at a scan rate of 0.264 2θ per minute using a Vantec Position Sensitive Detector. Collected pattern in Fig 2.

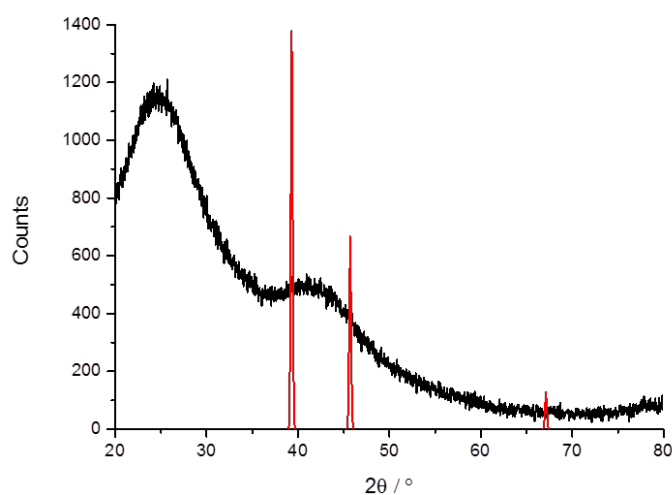


Fig 2: Powder XRD pattern of cPt + Mo/C catalyst (black). Position of Pt reflections in red (ICSD collection code 41525).

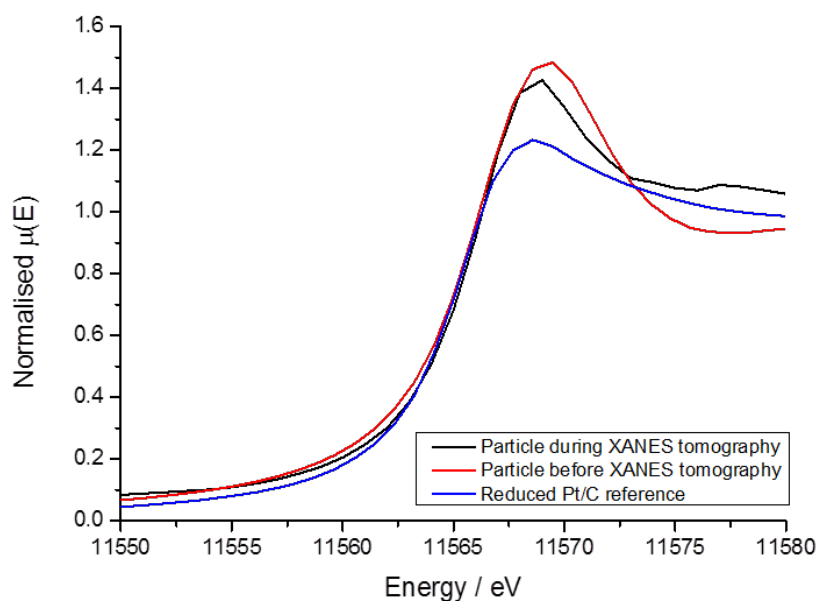


Fig 3: Loss of white line intensity (at 11568 eV) and increase of post edge intensity (at 11578 eV) of sample illustrating reduction by X-ray beam before (red) and during (black) XANES-CT experiment; total time exposed = 14 hrs (9 hrs for XRD-CT + 5 hrs to peak of white line 11568 eV). The photon flux to the sample is of the order of 10^{10} photons/second. The spectra were normalised by fitting a pre-edge and post-edge line to the calibrated XAS data using Athena. A lower absorption edge peak indicates a greater electron density in the 5d 5/2 orbital.

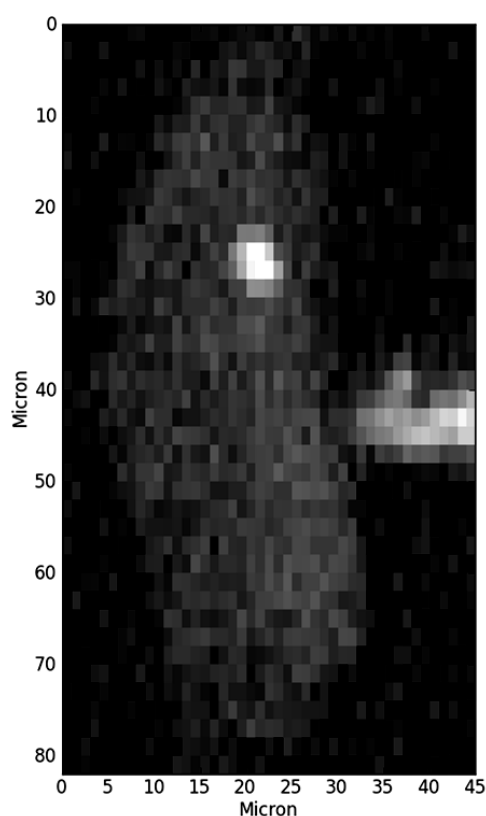


Fig 4: Fe distribution map from XRF-CT. Two brighter regions are concentrations of Fe outside of support, but trapped in epoxy resin used to mount particle to Kapton grid.

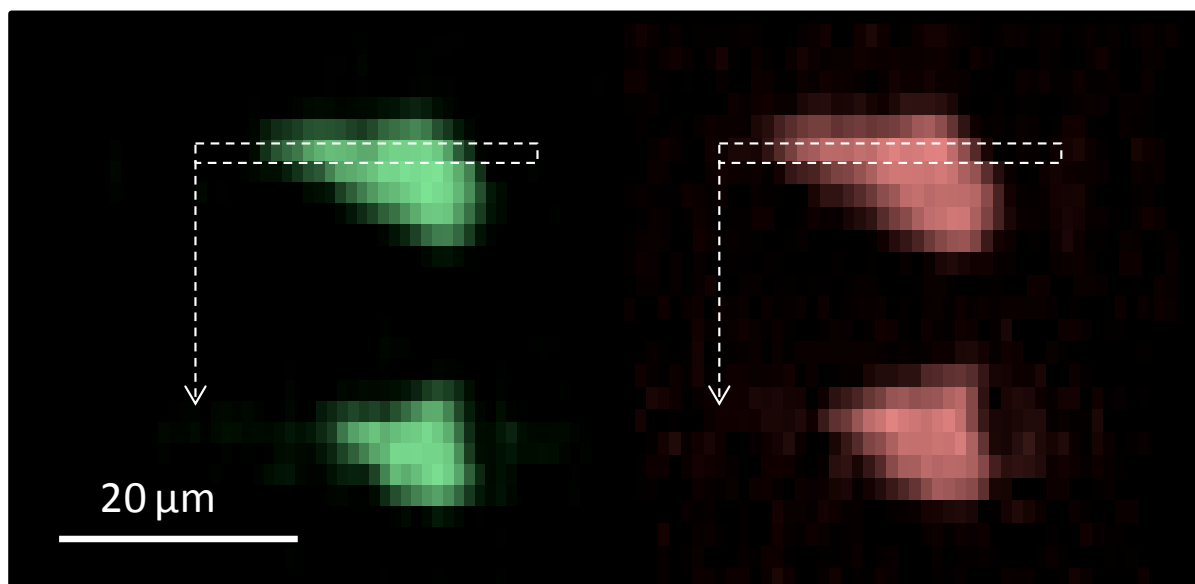


Figure 5: Horizontal and vertical cross-sections from XRF tomography on a second, smaller, particle showing Pt (red) and Mo (green) distributions throughout the carbon support. Pixel size was 2 micron.

Cluster analysis of XANES tomography stacks

Cluster analysis of the Pt XANES stack reveals five clusters (Fig 8 in paper), one of which contains no Pt signal outside of the particle. There is a low intensity cluster, approximately 1 pixel wide, running around the outside edge of the particle is a result of the beam being approximately Gaussian in shape and the size is defined as the FWHM. Whilst the centre of the focussed beam is missing the particle, some of the tails are striking the edge and causing the false positive cluster (Fig 8 (paper), green). Reducing the number of clusters calculated by 1 prevents this false positive occurring however it then results in a change in the distribution of clusters calculated within the particle. Therefore, this extra cluster must be included during the cluster calculation stage, but can be disregarded during analysis.