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Supporting Information

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~~**Supporting Information to the *Angewandte Chemie* communication:**~~

Tomographic Energy Dispersive Diffraction Imaging as a Tool to Profile in Three Dimensions the Distribution and Composition of Metal Oxide Species in Catalyst Bodies

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Below we include additional information on the set-up, data collection process and data of the calcined Co-Mo/Al_2O_3 catalyst body.

1. Experimental set-up and data collection process

Experiments were performed on Station 16.4 at the SLS Daresbury Laboratory (UK), which operates at 2 GeV with a typical current of 150 – 250 mA. A schematic and photograph of the set-up is given in Figure 1. Since both the detector positions and lozenge sizes can vary, those that were used for this work are given in Table 1.

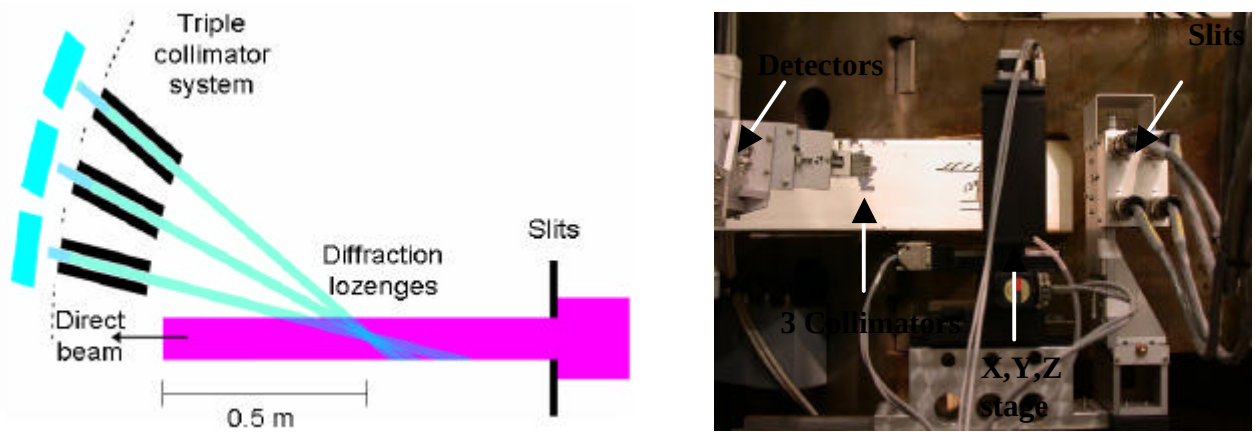


Figure 1. *Schematic (left hand side) and photograph of the energy dispersive diffraction set-up on Station 16.4 at SRS. The set-up utilizes a white beam to collect diffraction data using a 3 element detector arrangement at various fixed 2q positions (separated by ca. 2.9°). The beam passes through pre-sample*

collimators, the catalyst body and post-sample collimators (30 cm long and 15 mm wide) before striking the energy discriminating Ge detectors.

Table 1. *Typical detector 2q angles and lozenge lengths for a 100 mm circular cross section beam used to record the data.*

	Top	Middle	Bottom
Detector Angle (2?°)	7.5170	4.641	1.745
Lozenge length(mm)	1.5222	2.468	6.566

The sample was positioned longitudinally and parallel to a pin attached to a goniometer (Figure 2), the latter, itself mounted to the *xyz* stage. The goniometer was adjusted so that it would lie:

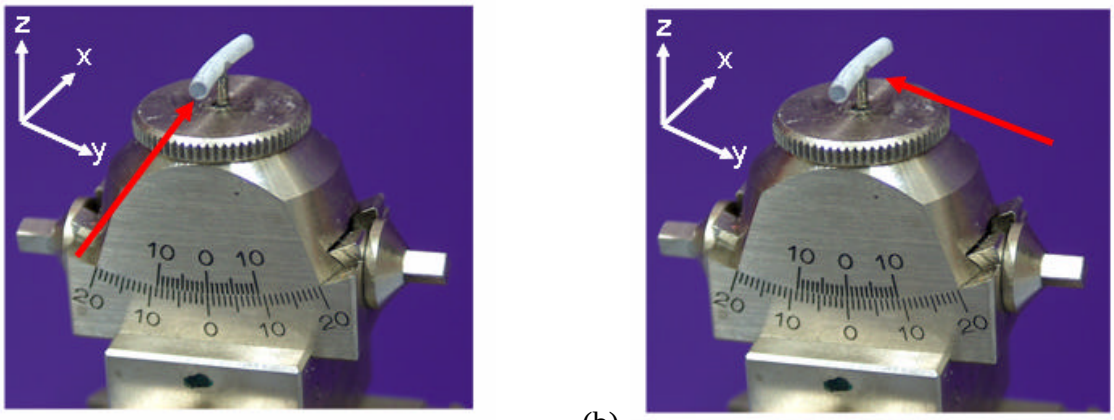
(a) mounted with cylindrical axis parallel to the beam, i.e. sample face normal to the beam (see Figure 2(a)). Scans were then performed in '2D' by moving the sample (with a fixed position in X) in the Y and Z directions.

(b) with the cylindrical axis normal to the beam; with the length along the sample normal to the beam (see Figure 2(b)). Scans were then performed in '2D' (X-Y (fixed Z) and X-Z (fixed Y) although for the former, the lack of movement in X relative to Y renders the scan almost '1D')

Two types of scan were carried out:

1) '2D' traverses in geometry using motors x, y and z respectively. Traverse steps the same size as the beam cross section, that is 0.1 mm
2) '1D' traverse in geometry type using the motor named *x*. Traverse steps were smaller than the beam cross-section size, that is 10 μm.

Patterns were collected at each point within the traverse for 60 s. A typical '2D' scan lasted approximately 4 - 6 h.



(a) (b)

Figure 2*.Illustrations containing sample mount and an indication of the incoming X-ray beam (highlighted using a red arrow). In (a) the beam approaches from the front of the extrudate (parallel to the incoming beam) and the sample stage is moved in the y and z directions and at each position a measurement is made. In (b) the beam approaches from the side of the extrudate (perpendicular to the sample) and the sample stage is moved in the x and y directions.*

2. Additional data for the calcined Co-Mo/Al_2O_3 catalyst body

Figure 3 shows the TEDDI scans for the calcined Co-Mo/Al_2O_3 sample in the X-Y and Y-Z axes, which accompany the data shown in Figure 3 of the main text of this communication. As was seen for the Mo/Al_2O_3 sample again an ‘egg-shell’ distribution can be observed in both measurements. We note that the apparent ‘brightness’ of the fluorescence signals to the right hand side of the pellet is caused by the differences in density affecting the self-absorption process, although this time caused solely by the marked curvature of the pellet. This may also explain the cause of the diffraction signal intensity observed in the Y-Z scan.

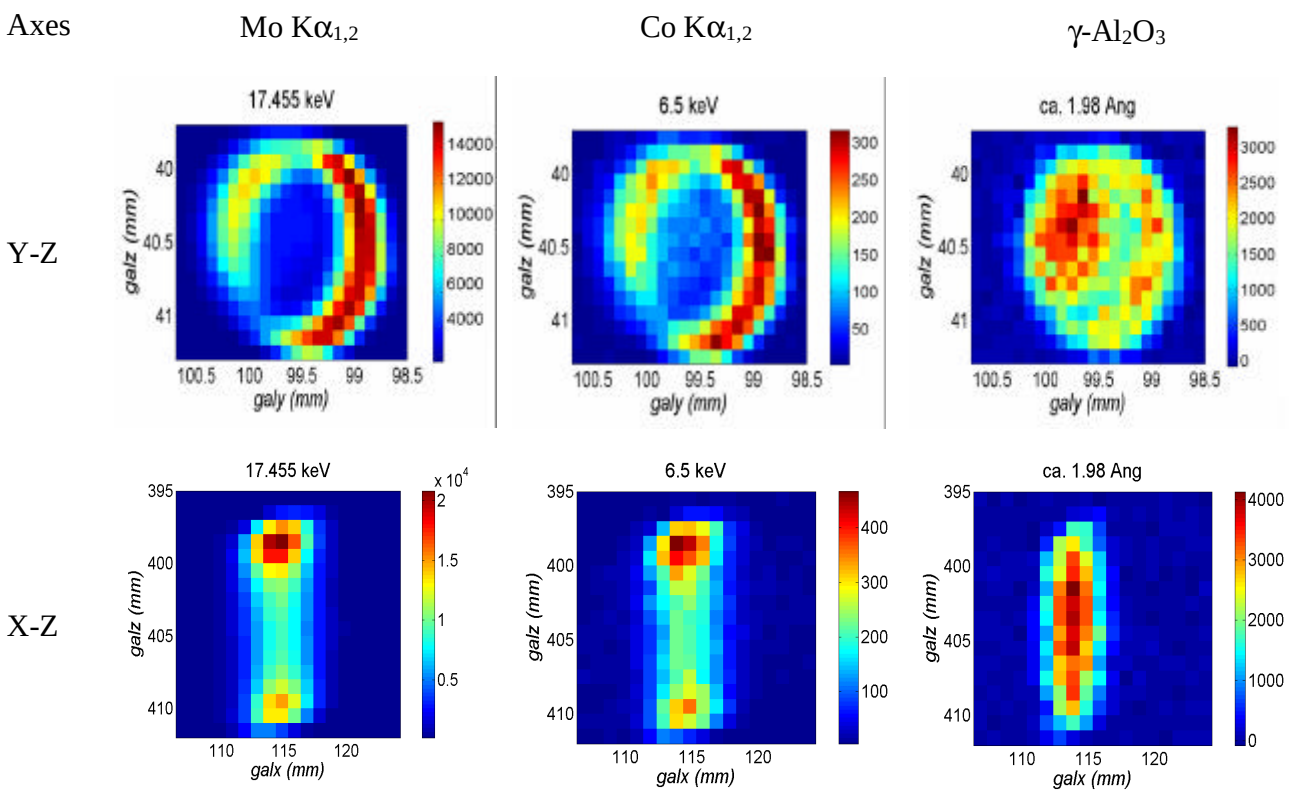


Figure 3. *Concentration maps for Mo $K_{a1,2}$ fluorescence and diffraction data for 1.98 Å (g-Al_2O_3) for the Co-Mo/Al_2O_3 catalyst body. The red (darker) areas represent regions where the signal and therefore the species of interest are most concentrated. Note the scale on the right hand side for each images is in detector counts/15 s.*