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Accessing New Dimensions of Nanomaterials: EEL Spectroscopic Tomography

Lluis Yedra[1,2], Alberto Eljarrat[1], Raul Arenal[3,4], Moises Cabo[5], Alberto Lopez-Ortega[6], Marta Estrader[6], Eva Pellicer[5], Maria Dolors Baro[5,6], Sonia Estrade[1,2], Francesca Peiro[1]

1. Laboratory of Electron Nanoscopies (LENS)- MIND/IN2UB, Dept. d'Electrònica, Universitat de Barcelona, Barcelona, Spain, 2. CCiT Scientific and Technological Centres, University of Barcelona, Barcelona, Spain, 3. Laboratorio de Microscopias Avanzadas (LMA), Instituto de Nanociencia de Aragon (INA), Universidad de Zaragoza, Zaragoza, Spain, 4. Fundacion ARAID, Zaragoza, Spain, 5. Departament de Física, Facultat de Ciències, Universitat Autònoma de Barcelona, Barcelona, Spain, 6. CIN2(CIN-CSIC) and Universitat Autònoma de Barcelona, Catalan Institute of Nanotechnology, Barcelona, Spain

Electron tomography is a widely spread technique for recovering the three dimensional (3D) shape of nanostructured materials. Using a spectroscopic signal to achieve a reconstruction adds a fourth chemical dimension to the 3D structure. Up to date, energy filtering of the images in the transmission electron microscope (EFTEM) is the usual spectroscopic method even if most of the information in the spectrum is lost. Unlike EFTEM tomography, the use of electron energy-loss spectroscopy (EELS) spectrum-images (SI) for tomographic reconstruction retains all chemical information, and the possibilities of this new approach still remain to be fully exploited. In this work we prove the feasibility of EELS tomography at low voltages (80kV) and short acquisition times thanks to the recent advances in TEM and the use of Multivariate Analysis (MVA), applied to FexCo(3-x)O4@Co3O4 mesoporous materials. This approach provides a new scope into materials: the recovery of full EELS signal in 3D. Data acquisition was carried out on a probe Cs corrected FEI Titan operated at 80 kV acceleration voltage. Afterwards, MVA methods were applied, namely principal component analysis (PCA) and independent component analysis (ICA). From the noisy raw spectra, enhanced O (K), Fe (L3,2) and Co (L3,2) edges were retrieved after PCA analysis. ICA successfully retrieved the Fe oxide and Co oxide signals of the sample as well as the background signal before the oxygen K edge. Reconstruction of those signals was achieved, leading to volumes not only containing structural information, but also chemical information. Regarding chemical information, an interesting result was revealed: the comparison between iron and cobalt signals showed that some of the iron which was intended to penetrate into the structure remains instead on the outer surface. The particles are richer in iron at the border and therefore, iron related chemical signals give a sharp interface between the particle and the background, where HAADF signal is very low and has fallen to background levels due to the small thickness. These results show that iron signals reconstruct more precisely the edge of the particles than HAADF. On the other hand, the thickness signal has the drawback of underestimating the border more than the HAADF signal. However, the most interesting feature of this signal is that it is insensitive to the chemistry of our sample and independent of multiple scattering, a characteristic not found in any other signal used for electron tomography. As a conclusion, EELS SI tomography is shown to be able to reconstruct chemical information of a sample in three dimensions. Moreover, the application of MVA to the data opens a new range of applications, reducing the limitations due to beam sensitive materials or samples with components with overlapping edges, where core-loss extraction using background estimation fails.