

Application of Simultaneous Energy-Filtered High-Speed 4D STEM Diffraction Imaging and EDS to Study the Strain and Elemental Distribution at the Interface

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With the competitive design and production of new electronic materials, these days understanding the mechanics at the interface and being able to accurately and reliably correlate this information to the elemental distribution and chemistry measured with other devices is critical. Four-dimensional scanning transmission electron microscopy (4D STEM) uses a parallel or convergent electron beam to generate diffraction patterns at each scanning point (two-dimensional (2D) diffraction patterns, for a 2D STEM rastered area, results in a 4D data cube). The diffraction pattern contrast and interpretability of such data can be increased, when the camera used is fitted at the end of an energy filter. Energy dispersive x-ray spectroscopy (EDS) is also a key technique for elemental composition analysis of materials, that has been used in STEM for several years.

In this work, we present a new approach where energy-filtered 4D STEM and EDS datasets are collected simultaneously at high speed. The direct detection CMOS camera (K2 IS, installed at the end of an energy filter) leads the acquisition as the master and provides synchronization by sending a signal out at the end of each frame; this signal informs the DigiScan unit to advance the beam to the next pixel position and the EDS unit that the beam has moved. EDS, STEM and 4D STEM diffraction datasets are all acquired in DigitalMicrograph™ and can be stored on disk for future data analysis. Using this approach, we studied the interface region between yttria-stabilized zirconia (YSZ) and Fe₂O₃. 4D STEM dataset was used to calculate the strain variation. The EDS maps showed the interface to be quite abrupt although a careful observation of the O map showed some decrease of contrast right across the interface indicating a possible deficiency in oxygen. In order to obtain a better understanding of the elemental distribution across the interface, high-energy resolution core-loss EELS analysis was carried out; a significant change in the Fe L₂ and L₃ ratio, as well as a chemical shift, were present, all these effects being consistent with changes in the valence state. We think that the strain variation measured at the interface could be the reason for such changes in the chemistry and composition across the interface.