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Investigation of structural defects and beam induced transitions in MgV_2O_4 nanocrystals using atomic resolved scanning transmission electron microscopy.

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As our energy needs grow, so too will our needs for portable energy storage. Today, most energy storage technology is based on Li ion transfer between a cathode and anode material. Despite their abundance, Li batteries have room for improvement due to low ion charge density, dendrite formation upon cycling, as well as environmental concerns surrounding disposal[1]. Thus, researchers are investigating the materials needed for the next generation of batteries. In this contribution, we will present a study of vanadium oxide nanocrystals, which, despite a variety of structural defects as suggested by X-ray measurements, has been shown to have a high cycling capacity of Mg^{2+} ions [2]. Specifically, we will report an atomic-resolved characterization of the structural defects in spinel V_2O_4 nanocrystals and their potential effect on Mg^{2+} cycling. Additionally, we will report the effects of electron beam exposure, such as a spinel to rock salt transformation.

This study will be conducted using an aberration-corrected cold field emission JEOL ARM200CF operated at 200kV primary electron energy. Imaging and spectroscopic measurements will be conducted with the emission current at 15μA. The electron probe will be operated at 24 mrad convergence semi-angle and the inner angle detector will be set to 75 mrad [30mrad] for high angle annular dark field [low angle annular dark field] imaging. The ARM200CF is also equipped with an Oxford XMAX100TLE X-ray detector and a post-columns Gatan Continuum GIF spectrometer.

Figure 1 shows the spinel to rock salt transition along [110] that occurs upon beam exposure. In this contribution, we will report a comparison of acquired images to simulated STEM images, in order to understand the changes in occupancy of different atomic column. Additionally, we will correlate these structural defects to changes in the ELNES fine structure of the oxygen K-edge. In order to aid our interpretation of our results, we will use FEFF9- a real space, multiple scattering code- to simulate O *K*-edge spectra of a variety of defects structures. Figure 2 compares (a) experimentally acquired O *K*-edge spectra of MgV_2O_4, simulated spectra of (b) rock salt structure, (b) twin grain boundary defects, and (c) MgV_2O_4.

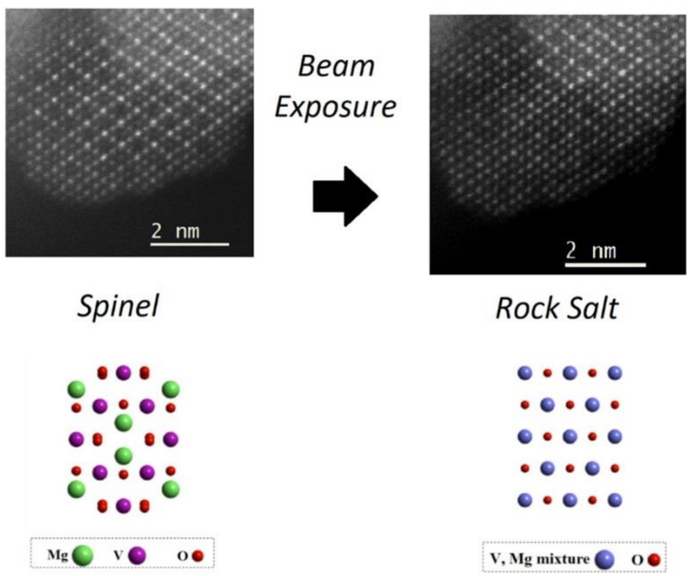

Figure 1. Particle oriented along (110). Upon electron beam exposure MgV2O4 nanocrystals undergo a spinel to rock salt transformation.

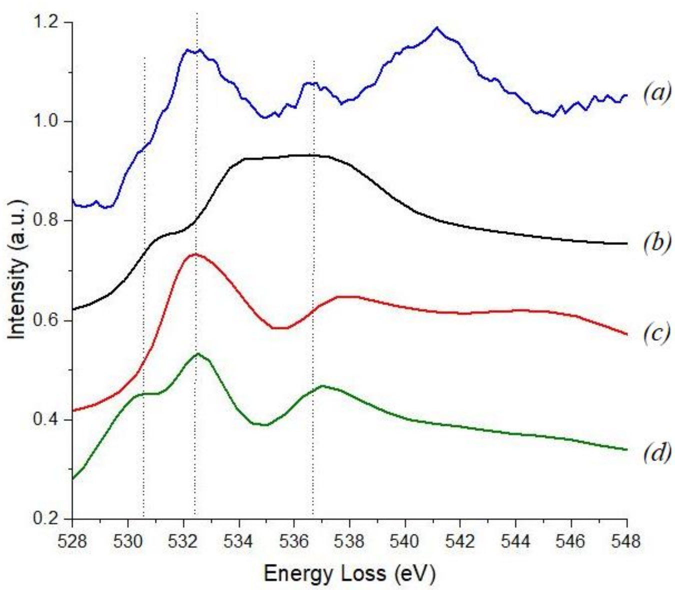

Figure 2. EELS oxygen K-edge spectra (a) experimentally acquired from MgV2O4,(b) simulated rock salt structure, (c) simulated twin grain boundary defects, (d) simulated MgV2O4.

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