Thermal Characterization of As Synthesized Nano Ceria

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Nano ceria was synthesized using the hexamethylene-tetraamine (HMT) method [1]. Laboratory thermal cycles to 400°C showed that the lattice parameter after the first heating ramp was smaller than the initial cell dimension. This was attributed to surface adsorbates on the fresh sample.[2] These measurements were repeated at PDF (28-ID-1) beamline at NSLS II [3] with smaller temperature increments, more cycles, and with data for standard powder profile refinement [4] and for pair distribution function (PDF) analysis [5]. The NSLS II measurements allowed for more complete characterization of the structural changes during heating. The plot of the cell dimension during the first cycle (Fig. 1) shows a large difference in the initial and final cell dimension as in the laboratory measurement. The difference electron density map (Fig. 2) suggests possible defects in the as synthesized ceria that could contribute to the cell dimension effect. Figure 1 also shows a peak during the initial ramp which could be attributed to reduction of the ceria. This reduction may arise from the presence of HMT or HMT decomposition productions left on the sample.

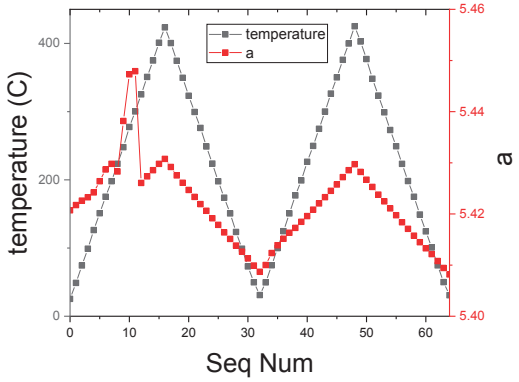

Fig. 1 Lattice parameter variation during heating cycle 1 and 2 for 8nm nano ceria.

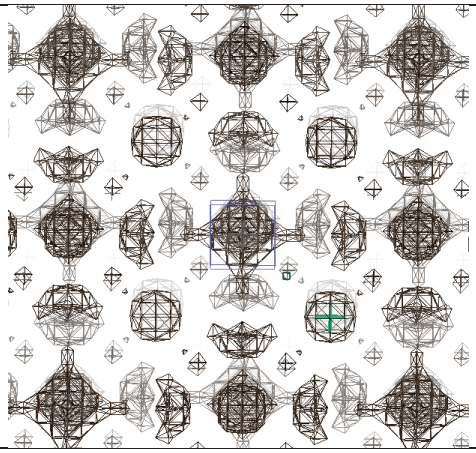

Fig. 2 Difference electron density of as synthisized 8nm CeO_2 sample.

We have tentative models for the defect from the difference electron density maps and PDF's. We are doing additional analysis with demodulation of cyclic data [6] and multivariate curve resolution alternating least squares (MCR-ALS).[7] We will present this analysis to demonstrate the new techniques and our current models for the defect.

References

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