HOLZ-STEM Investigations of Ceramic Thin Films via Direct Electron Detection

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We report the observation of a 'lattice doubling' effect within thin films of La_2CoMnO_6 (LCMO) using a medpix3 direct electron detector (DED)[1]. Multiple HOLZ rings were observed and then analysed in post-acquisition processing. The collection of diffraction patterns on a per pixel basis in STEM mode (HOLZ- STEM imaging) enables the observation of 3D-ordering within the specimen as the periodic modulation of atomic positions along specific atomic columns alters the radial intensity of the observed HOLZ rings [2,3].

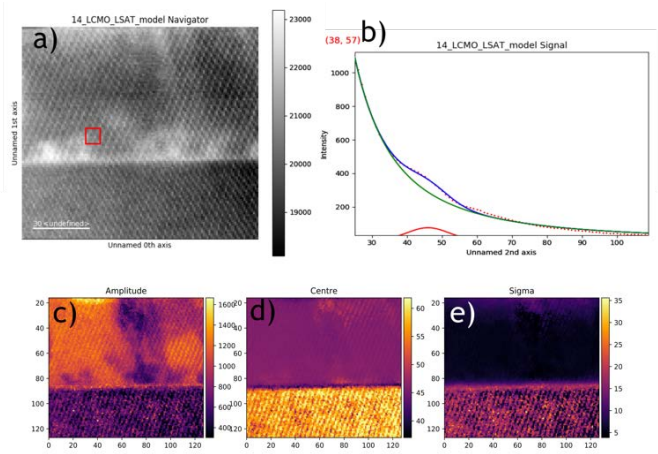

Figure 1: Model (blue) fitting of LCMO showing the powerlaw (green) and Gaussian (red) components.

After locating the central bright disk in each diffraction pattern, the coordinates of this point are used as a starting point for radial integration. This analysis produces a 1D profile of intensity versus radius (either in pixels or calibrated into units of angle). After cropped this profile to remove the influence of zero order reflections the remaining model was suitable for HOLZ-STEM investigations. We fit this radial profile to a multi-component model as shown in Figure 1.

After selecting suitable regions before and after the Gaussian fit is apparent in the data a power law model was developed and then applied to the length of the radial profile. The Gaussian component was fitted and each of its components, the amplitude, the centre of the fit and its width (termed sigma) was determined, the per pixel values of each of these parameters was then plotted graphically as shown in Figure 1 c-e.

The variation in the Amplitude and Centre parameters are indicative of movement of the HOLZ rings with the specimen thin film; we relate this to the atomic modulation of the La atoms in the A sites of the material. The real space plots show that are two domains present within the material. We will present study of the material and other ceramic and outline how HOLZ-STEM measurements greatly aid structural investigation of these materials.

1. Nord, M. *et al.* Three-dimensional subnanoscale imaging of unit cell doubling due to octahedral tilting and cation modulation in strained perovskite thin films. *Physical Review Materials* **3**, 063605 (2019).
2. Huang, F.-T. *et al.* Scanning Transmission Electron Microscopy Using Selective High-Order Laue Zones: Three-Dimensional Atomic Ordering in Sodium Cobaltate. *Physical Review Letters* **105**, 125502 (2010).
3. Randle, V., Barker, I. & Ralph, B. *Measurement of Lattice Parameter and Strain Using-Convergent Beam Electron Diffraction. JOURNAL OF ELECTRON MICROSCOPY TECHNIQUE* **13**, (1989).