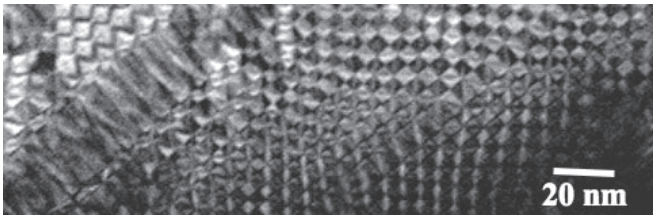

Fig.1. Low mag image of an x = 0.1, $Li_{3x}Nd_{2/3-x}TiO_3$, *A*-site ordered, 'defect' perovskite exhibiting complex ordering on multiple length scales.

The sensitivity of electron diffraction to weak, subtle features of reciprocal space (such as weak additional satellite reflections and/or structured diffuse intensity distributions) coupled with the capacity to also image over a wide range of length scales makes the Transmission Electron Microscope (TEM) an extremely well-adapted instrument for the structural characterization of 'modulated' materials of this type.

In this contribution, the results obtained from several such systems will be described including inherently Pb-free polar functional materials, relaxor ferroelectric systems and the $Li_{3x}Ln_{2/3-x}TiO_3$, $0.047 < x < 0.147$, family of Li ion conductors. The local crystal chemical 'rules' underlying the inherent structural flexibility of such materials will be highlighted along with the characteristic diffraction signatures of such behaviour.

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Keywords: modulated, disordered, electron_diffraction

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Bonding charge density in $srtio_3$ under an electric field measured by electron diffraction

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The precise measurement of charge density when influenced by an electric field is clearly of interest in the understanding of the electrical properties of dielectrics. Considerable practical difficulties have prevented the production of experimental results that could test theoretical calculations of such distortion of charge density. This comment applies to both X-ray and electron diffraction charge density measurements. With X-rays, due to the requirement to use a perfect crystal when an electric field is applied, severe extinction prevents access to the charge density sensitive, low order region of reciprocal space. With electron diffraction, the application of a sufficiently strong electric field while simultaneously cooling the sample is a combination not available in commercial specimen holders. In the present work, modification of an old design of the Gatan 636 double tilting cooling holder has overcome this limitation.

The near zone axis technique of Quantitative Convergent Beam Electron Diffraction, QCBED, as detailed in [1], was used to measure the low order structure factors of $SrTiO_3$ with zero field applied. Some 150 diffraction patterns were recorded over a range of zone axes, accelerating voltages and temperatures between -144C and room temperature. The experiment is planned to repeated with a field of 1 to 4 V/micron applied in the 001 direction, limiting patterns to orientations near the 100 zone axis. The zero field data is a reference against which the field data may be measured as a perturbation. Also, two prior measurements of charge density at zero field, which differ substantially from each other, [2] [3] were available for comparison.

Upon the application of a field in the 001 direction, the 010 mirror line of symmetry in $SrTiO_3$, available in CBED patterns near the 100 zone, should disappear, the crystal symmetry being lowered from Pm3m to P4mm.

Calculated CBED patterns for the above geometry and with an applied electric field are being prepared, using the JEMS program with theoretical Fourier coefficients of the potential. The Fourier coefficients were obtained from the Discrete Fourier Transform of the total electrostatic potential derived from DFT calculations. These were performed with the Abinit program using the projector augmented wave density functional theory method (PAW-DFT), with a PBE density functional. A finite electric field was applied using the Berry phase approach.

Experimentally, samples to which an electric field can be applied are being prepared and the results will be reported at the conference.

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Automated quantitative 3d electron diffraction rotation tomography

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A new method of three-dimensional reciprocal space scanning using automated 3D electron diffraction rotation tomography is developed [1]. Sweeping reciprocal space is implemented by using the electron beam tilt within a given angular range and a small step. The beam tilt is combined with the mechanical crystal tilt in order to cover the full range of tilt angles available for the accessible transmission electron microscope (TEM) goniometer.

The automatic data collection procedure is split into two parts. Firstly, the mechanical tilt is used in order to reach different low-index crystallographic axes. Secondly, the deflection coils of the electron microscope are used for tilting the beam electronically around some axis, thereby sampling and scanning reciprocal space with desired precision. The smallest beam tilt step depends on the TEM machine but can be as small as ~0.0005°.

At present, this method allows the collection of 3D data by sweeping reciprocal space in the range from –43° to +43° (these values are the actual limits for our JEOL 2100 double tilt TEM sample holder) covering ~86° of reciprocal space or from –75° to +75° using the single ultra-high tilt holder. The complete data set contains 36 individual subsets for single tilt holder. Each subset covers 4° and has 80 individual frames, recorded by tilting the beam with 0.05° steps between frames. Every data subset was recorded after physically tilting the sample at an interval of 3.5° introducing some overlap between data subsets. This automated electron diffraction rotation tomography which we have developed allows collecting 2880 individual frames within 90 min (~1 frame per second, ~60 minutes for 2000 frames including crystal tracking). Scanning reciprocal space using rotation tomography allows registering not only the reflections, but also the 3D