form an angle of 120 degrees due to the crystallgraphic symmetry, indicating a possible DNA looping mechanism during transcription activation and inhibition.

Keywords: crystal_structure, circadian_rhythm, transcription_control

MS.78.5

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Crystal structure analysis of release factor 3

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Translation of an mRNA is terminated when a stop codon is encountered. Since tRNAs do not recognize stop codons, this event is performed by proteins called release factors. Prokaryotes have two class I release factors (RFs), RF1 and RF2, and one class II release factor, the G protein RF3. RF1 and RF2 hydrolyze and release the completed polypeptide from the peptidyl-tRNA at the ribosomal P-site in response to a stop codon. RF3 binds to the ribosome to promote rapid dissociation of RF1 or RF2 from the A-site in a GTP-dependent manner. We have studied the structure-function relationship of the RF3 from sulfate-reducing bacterium, *Desulfovibrio vulgaris* Miyazaki F. Here we present the high resolution crystal structures of RF3 complexd with GDP and guanosine 3',5'-(bis) diphosphate (ppGpp).

ppGpp is known as an alarmone which is involved in stringent response in bacteria. In cells growing under optimal conditions, the concentration of GDP is much dominant over that of ppGpp. Under stress conditions, however, the concentration of ppGpp increases strikingly, and attains levels over that of GDP. In the structure of RF3 complexed with ppGpp, ppGpp binds at the same nucleotide-binding site in an almost identical manner with GDP, suggesting that GDP and ppGpp is two alternative physiologically relevant ligands to RF3. We have found that ppGpp blocks the recycling of RF1 or RF2 by RF3 in bacterial ribosome. It is probably because ppGpp interferes either binding of RF3 to ribosome or replacement of GDP by GTP in the RF3 ribosome complex. These lines of evidences suggest that RF3 would have functions of a cellular metabolic sensor and/or regulator that switches between the active GDP-bound form which allows active protein syntheses under the normal condition and the low-active ppGpp-bound form when shortage of nutrients are detrimental.

Keywords: structure_function_relationship, stress, translation_factor

MS.79.1

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Electron crystallography: harder, better, faster, stronger

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The stronger interaction of electrons with matter allows nanoscale crystals to be investigated, which is of increasing importance, academically and commercially, given the growth of nanotechnology research and application during the last decade. Electron crystallography fills the void between crystals which are too small for single crystal X-ray studies and powder diffraction experiments that fail to yield a structure. However, it is often viewed as a technically difficult and time-consuming method for structure solution.

The development of the Automated Diffraction Tomography (ADT) [1] for electron crystallography has led to a number of distinct advantages over traditional methods, in terms of data collection, quality, quantity and the ability to solve structures *ab initio via* direct methods. The most appealing advantage, when compared to traditional approaches which require zonal diffraction data and complementary real space image, is the vastly reduced time it takes to solve a structure from diffraction intensities only, in favourable cases data collection and structure solution can be completed within a single day.

The details of how ADT methodology performs data collection from nanocrystals shall be outlined. The new data collection geometry has some striking advantages, it provides a vastly improved coverage of reciprocal space, when compared to zone axis data sets, as well as reduced dynamical effects. The new processing requirements [2], for extracting the intensities for structure solution, these will be discussed in detail.

The improvements in data quality gained by using ADT for electron crystallography have two distinct advantages. 1. Direct Methods can be used routinely for structure solution [3]. 2. Structures more difficult and complex can be solved by electron diffraction data alone than previously thought possible. Examples of structures solved using ADT, which were not possible by any other method, will be presented.

We hope to convey the benefits of using the ADT approach for electron crystallography and appeal to crystallographers who would not normally consider using electron crystallography that it may be a viable approach to consider in the future for solving problematic nanocrystals which would not yield an answer to their tried and trusted methods.

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Keywords: automated diffraction tomography, nanocrystal, software

MS.79.2

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Modulated structures and TEM's: from relaxor ferroelectrics to nano-chessboards

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Functionally useful materials are often modulated and frequently inherently flexible [1] *i.e.* materials whose local structures and properties are finely balanced and hence able to respond to the application of external signals *e.g.* electric and/or magnetic fields, strains, changes in temperature, composition *etc*. Materials of this type (piezoelectrics, relaxor ferroelectrics, ionic conductors, solid solutions *etc.*) are ubiquitous in devices all around us *e.g.* mobile phones, sensors, solid oxide fuel cells. A detailed understanding of structure, both average as well as local (on the relevant length and time scales, see *e.g.* Fig.1 below) of such materials is essential for an understanding of their properties and of methods to optimize and manipulate them.

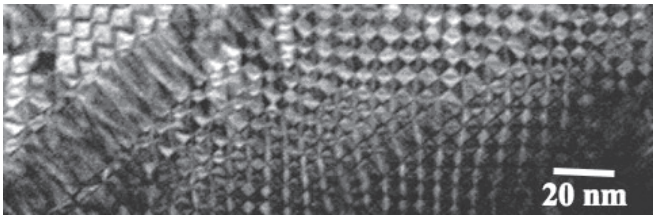

Fig.1. Low mag image of an x = 0.1, $Li_{3x}Nd_{2/3-x}TiO_3$, *A*-site ordered, 'defect' perovskite exhibiting complex ordering on multiple length scales.

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

MS.79.3

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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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Keywords: charge-density, electric-field, CBED

MS.79.4

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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