Electron crystallography – an introduction

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Everything in Nature, macroscopic or microscopic, inorganic, organic or biological, has its specific properties. Most properties of matter depend on the structures, and many techniques have been developed over the centuries for structure analysis. The greatest of them all, structure analysis of single crystals by X-ray diffraction, X-ray crystallography, was founded in 1912 and is still today the most important technique for studying microstructures of periodically ordered objects. Electron diffraction of single crystals was discovered fifteen years later and thereafter the wave property of electrons was exploited in the invention of the electron microscope. Since then, electron microscopes have been used in many fields as a tool for exploring and visualizing the microscopic world in all its beauty. Between the first blurred images and today's sharp atomic resolution lies fifty years of untiring engineering. More recently, the unprecedented power of computers has made it possible to analyze quantitatively, and even further improve, these images. The amalgamation of electron diffraction and atomic resolution electron microscopy with crystallographic image processing has created a new powerful tool for structure analysis - electron crystallography.

1.1. The history of electron crystallography

1.1.1. Electron diffraction analysis started early in Moscow

In 1976, Dorset and Hauptmann in Buffalo for the first time applied the direct methods to electron diffraction data.

1.1.2. Electron microscopy can be combined with image processing

At the time when the different phasing methods developed for X-ray crystallography were applied for finding the lost phase information in electron diffraction, DeRosier and Klug (1968) at the MRC laboratory of Molecular Biology in Cambridge introduced a method of reconstruction of three-dimensional objects from a set of electron microscope images. This 3D reconstruction method is based on the fact that both phase and amplitude information are present in electron microscopy (EM) images and can be extracted from the Fourier transform of images by digitized image processing. They compared the 3D reconstruction method with the structure analysis method in X-ray diffraction and wrote: "*The difference is that the 'phase', which together with the amplitudes of the Fourier components allows the reconstruction of a three dimensional map, is lost in recording the X-ray diffraction data. It is preserved, however, by the focusing of the diffracted electron beam into an image*".

The 3D reconstruction method introduced by DeRosier and Klug created a revolution in structural molecular biology. Hundreds of macromolecular structures, including membrane proteins and viruses, were determined by this method. A most significant result was the 3D structure of purple membrane, solved to a resolution of 7 Å from EM images and electron diffraction data (Henderson and Unwin, 1975). This was the first study giving information on the internal structure of a membrane protein. It was to take another ten years before the first X-ray crystal structure of a membrane protein, the photosynthetic reaction center, was solved, a work for which they were awarded the Nobel Prize in Chemistry in 1988.

Most inorganic crystals give much higher resolution than biological samples do, due to smaller unit cells, better ordering and less radiation sensitivity. It is relatively easy to get high resolution electron microscopy (HRTEM) images and diffraction patterns from them. The first EM images near atomic resolution (4 Å) showing details within a unit cell were obtained from a thin crystal of $Ti_2Nb_{10}O_{29}$ by Ijima (1971).

1.2. Electron crystallography has some advantages over X-ray crystallography

1.3. The fear of multiple scattering hampered the development of electron crystallography

1.4. New developments in electron crystallography

The last decade has seen a dramatic increase in new techniques for electron crystallography.
These include Cs-correctors for improved HRTEM images and precession and rotation methods for getting improved intensities and higher resolution in 2D and 3D, respectively.

Electron Crystallography of Three-Dimensional Protein Crystals

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When protein crystals fail to grow to a size sufficient for X-ray crystallography, electron diffraction may be a viable alternative. The reason is that, relative to the number of elastically diffracted quanta, electrons on average deposit less energy in the crystal than X-rays, while radiation damage is the limiting factor in data collection. As a consequence, solving the structure of 2D crystals that are just one unit cell thick, is possible with electron diffraction, but not with X-ray diffraction. However, there are still practical problems with sample handling, diffraction data collection and data processing of 3D protein crystals. Furthermore, in the case of 3D protein crystals that are more than one unit cell thick, dynamical scattering, caused by electrons scattering multiple times within the sample, may prevent structure solution.

We have made substantial progress in tackling these issues: we now have developed procedures that allow rotation data collection of protein crystals by using a quantum area detector which has a substantially higher signal to noise ration than other detectors. We can accurately determine the unit cell even when only single frames with unknown angular relationships are available and can index and integrate these patterns. We also demonstrated that it is theoretically possible to use the dynamic effect for ab initio phasing. An overview of these recent developments will be presented.

Energy filtered Nano Beam Diffraction (NBD) - the prerequisite for highly resolved crystallography

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Since a few years, besides HR imaging electron diffraction (ED) is used as stand alone technique to solve nanostructures. Precession Electron Diffraction (PED) is a recent technique [1] which allows solving “ab-initio” crystal nanostructures, by using a collection of PED patterns acquired from different Zone Axes under quasi-kinematical conditions, like for X-ray Diffraction, but with a lateral resolution down to nm range.
The Köhler Illumination system [2] of the LIBRA 200 offers the best requisite for Nano Beam Diffraction (NBD). Nevertheless it is a huge optical challenge

- to generate a field of view from a few tenth of nm down to 1 nm with small convergence angle (0.1-1 mrad). and
- to make sure that neither the illuminating field nor the specimen image is moving when precession is applied.

We have developed a new ray path, where the limiting illumination aperture is arranged higher up in the illumination system so that the additional demagnification stage can be used to demagnify the aperture. This results in a high flexibility concerning illuminating field and convergence angle without any need of readjustment of pivot points and refocusing of the diffraction lens. First results will be shown and the physical limits of NBD will be discussed.
The corrected Omega energy filter of the LIBRA 200, besides an ultimate energy resolution of 41 meV [3], allows to perform energy filtered electron diffraction with high acceptance angle (100 mrad at ΔE = 10 eV). In combination with the new modular digital precession instrument DigiSTAR [4] we are able to clearly visualize electron diffraction reflections from the First Order Laue Zone, which is very useful either for space group symmetry determination [4] or to enhance fine structure details for structure determinations using PED.

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Direct methods and electron diffraction data

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Direct Methods are the technique which enabled the practical solution of the phase problem, at least for X-ray diffraction data and up to 200 atoms in the asymmetric unit. Its application to electron diffraction data is not straightforward, mainly because of the large deviations of the diffraction amplitudes from the kinematical values and of the incompleteness of the collected data.

The solution is usually attained via “multi solution” approaches by using random starting phases, in the space group experimentally determined. Lowering the symmetry is always possible if needed. The main tool is the tangent formula which involves the use of triplet and quartet invariants. We will describe the main features of the current version of SIR2010, particularly optimized for electron diffraction data. Usually a fragment is provided which may be completed, chemically interpreted and refined via least squares techniques.

The BEA algorithm: an additional tool for crystal structure solution via PED data

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Strong deviations of the diffraction intensities from the kinematical values are often present in electron diffraction data, owing to many beam dynamical scattering effects. The consequence is a limited capacity of precisely determining the values of the structural parameters. *PED* techniques [1] reduce the number of reflections which are simultaneously excited: however 2-dimensional reflections from a few well oriented zone axes are usually collected. More recently [2,3] the combination of the *ADT* (automated diffraction tomography) technique with *PED* allowed much larger data completeness.
We have studied the statistical features of *PED* and *ADT* amplitudes with particular attention to the effects they produce on the efficiency of the phasing procedures, specifically on Direct Methods approaches. A new algorithm, denoted *BEA*, is proposed: once a structural model is available (even if it is imperfect), the *best* amplitude among the equivalent reflections is used to improve the model. It is shown that *BEA* is able to provide more complete structural models, to make the phasing process more straightforward, and to result in crystallographic residuals that are much better than those usually obtained by electron diffraction.

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Liquid nitrogen free EDS for Scanning Transmission Electron Microscopy

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Silicon drift detectors (SDD) were originally developed to avoid the use of liquid nitrogen in devices for space research. More than a decade ago Roentec, now Bruker Nano GmbH, was the first company to introduce SDDs for energy dispersive X-ray spectroscopy (EDS) in scanning electron microscopy (SEM) [1]. Today silicon drift detectors represent established technology in SEM and are about to revolutionize EDS for transmission electron microscopy (S/TEM). Within the geometrical constraints of a TEM pole piece medium size detector areas are well suited to cover enough solid angle for radiation collection and to provide a good take off angle for a high peak to background ratio. Additionally they offer higher efficiency, better energy resolution and easier cooling and thus more stable measurement conditions than large detector areas. Using one $30mm^2$ SDD at 0.12sr solid angle, atom column EDS in case of an aberration corrected instrument [2] and nm resolution EDS for a conventional TEM [3] have been shown. Aberration correction and high brightness sources can offer high beam currents within atomic size probes. Electronics adapted to utilize multiple detector systems offers exciting new possibilities for the fast detection of even smaller amounts of matter [4]. Results achieved so far give an idea of exciting new possibilities for speed and data quality in modern TEM EDS.

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Rotation diffraction method for the collection of 3D data sets

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Rotation diffraction is a new method for collecting complete three-dimensional (3D) electron diffraction data. Diffraction data is collected by combining electron beam tilt at many very small steps, with rotation of the crystal in a few but large steps. A number of practical considerations are discussed, as well as advantages and disadvantages compared to other methods of collecting electron diffraction data.

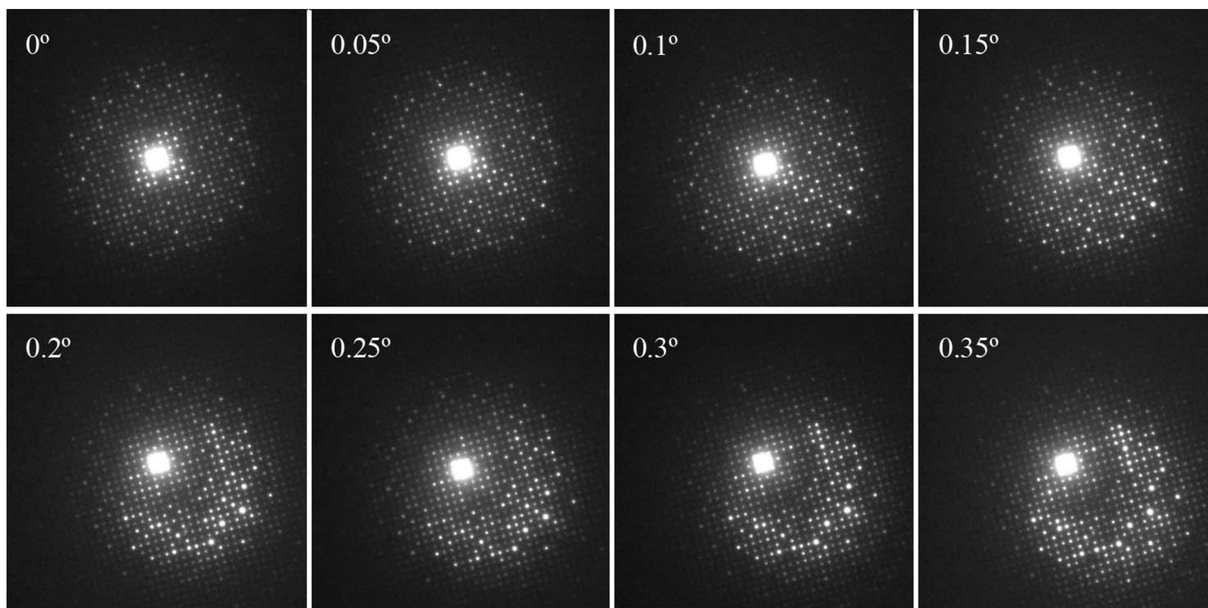

Fig. 1. High precision beam tilt series of 0.05º interval performed by the rotation method. The electron beam was tilted slowly away from the [001] zone axis at 0º, while the direct beam was kept at the same position.

While precession electron diffraction is superb for obtaining high-quality near-kinematical data for individual zone axes, rotation electron diffraction is better suited for collecting complete 3D diffraction data. We present a new data collection method, combining goniometer rotation for large increments and beam rotation with very fine steps.

Ab-initio structure solution of nano-crystalline material by Automated electron Diffraction Tomography (ADT)

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Electron diffraction is a well known technique to determine structural information from nano-crystalline material where single crystal or powder X-ray diffraction fails. Traditionally, 3D electron diffraction data is collected by a tilt of a pre-oriented nano-crystal around a crystallographic axis. Using automated diffraction tomography (ADT), a technique recently developed at the University of Mainz [1, 2], it is now possible to collect improved diffraction data, both in terms of quantity and quality, through a tilt around an arbitrary axis.
ADT has been performed so far on crystals down to 50 nm size and allows the access of strongly agglomerated or embedded particles as well as multiphase systems. Application of STEM imaging and nano-diffraction makes the technique particularly suitable for beam sensitive samples. Collecting ADT data from non-oriented nano crystals and coupling with precession electron diffraction (PED) [3] delivers data suitable for "ab-initio" crystal structure analysis by standard software available from X-ray crystallography. The necessary time for a full data acquisition and structure solution procedure has been reduced down to 1.5 days bringing the ADT technique in the range of X-ray structure analysis methods.
A introduction into ADT and survey of ADT structure analysis will be given for a wide range of material such as high pressure materials, minerals, natural and synthetic zeolites, metal organic frameworks, covalent organic frameworks, organic-inorganic hybrid material, small organic molecular crystals (e.g. polymorphic systems: pigments and drugs) [4-9].

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Imaging and analysis with electrons – where are we today and what are the possibilities for tomorrow?

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The ability of electron microscopes to identify atomic positions in individual nanostructures is limited by lens aberrations and by electron beam sample interaction which leads to modifications of the investigated structure. As Abbe imaging theory describes structure investigation can be done via imaging of the sample directly (HRTEM) or via transferring a magnified diffraction pattern (SAED, CBED, etc). In both cases the resolution is limited by aberrations of the objective lens.
The resolution of present-day spherical-aberration corrected TEM is limited by the chromatic aberration of the objective lens to about 0.07 nm at 300kV [1,2]. In case of applying HRTEM technique structure investigation using the combination of a small defocus with a small negative spherical-aberration value, an optimum contrast at a minimal point spread can be achieved in such TEM's [3]. With this method the precision at which the separation of well-isolated atoms can be measured is better by more than an order of magnitude of the TEM Rayleigh-resolution. Therefore metrology of atomic position using Cs-corrected (S)TEM is possible with a precision of a few pm. For example Jia et al. could measure atomic position with a precision of 5pm using an aberration-corrected TEM with an information limit of 80 pm [4].
On the other hand electron precession technique [5] for crystal structure investigation techniques provides the possibility to measure integrated diffraction beam intensities which do not require precise tilt adjustment of the crystal under investigation. At the same time this method allows to image not only the zero-order Laue zone (ZOLZ) but also higher-order Laue zones (HOLZ) which gives three dimensional information of the sample. Since image spherical aberration corrected TEM is available large-angle convergent-beam electron diffraction (LACBED) is a promising alternative method to investigate crystal structure in the nm range [6].
In the talk new developments on equipment view of point will be presented.

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Structural fingerprinting in the TEM and open-access crystallographic databases

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Electron crystallography was divided (*"roughly speaking"*) by Sven Hovmöller *"into two main categories; 1. Identify known phases and 2. Determine 3D structure of new unknown phases"* [1]. The comment *"while the first is of more interest to industry, the second is more exciting in academia"* was also made [1].

While most well established electron crystallographers in academia were pursuing more exciting activities, a small group of newcomers at Portland/Oregon has over recent years worked on the utilization of recent developments in electron crystallography for the identification of known phases in a transmission electron microscope (TEM) [2-5]. This talk gives a brief overview over this body of work and also deals with recent developments in comprehensive open-access crystallographic databases.

There are now data for more than 130,000 crystal structures in open access [6]. The Crystallography Open Database [7] (COD) alone features currently more than 100,000 entries (at its main site and four mirror sites) [8] and is rapidly growing. All of the entries of the COD can be visualized interactively over our web portal [8]. An about 20,000 entry subset of the COD [9] supports structural fingerprinting from the projected reciprocal lattice geometry of single crystals by the calculation of so called "lattice-fringe fingerprint plots" [2,3]. These plots are applicable to the amplitude part of the Fourier transform of high resolution TEM images as well as to single-crystal electron diffraction spot and Kikuchi patterns.

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Introduction to electron precession diffraction

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Electron precession is a new electron diffraction technique proposed by Vincent and Midgley in 1994. This technique strongly increases the possibilities of electron diffraction since it allows taking into account the integrated intensity of the diffracted beams. In addition, the precession patterns display a much larger number of reflections than the conventional microdiffraction or selected-area electron diffraction patterns and "few beam" conditions are observed so that the kinematical forbidden reflections can be identified from the allowed reflections. Therefore, this method is very well adapted to electron crystallography.
A description of the technique will be given and the precession patterns will be interpreted by means of the Ewald sphere and the LACBED patterns.

Space Group Identification from Electron Diffraction

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Zone axis electron diffraction patterns display some typical features which can be used to identify the space group. They are:

- The "net" and "ideal" symmetries of the Zero-Order Laue Zone (ZOLZ) and of the Whole Patterns,
- The shift between the ZOLZ and the First-Order Laue Zone (FOLZ),
- The periodicity differences between the ZOLZ and the FOLZ,
- The kinematical forbidden reflections located in the ZOLZ and in the FOLZ.

Two diffraction techniques a very well adapted to the observation of these features: electron precession and CBED. A systematic method involving a few zone axis patterns and taking into account the extinction symbols will be given. It is based on comparison of experimental precession and CBED patterns with theoretical patterns drawn for all the extinction symbols.

Microstructure diagnostics of modern materials by transmission electron microscopy – need for advanced diffraction techniques

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Transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) in combination with analytical methods allow for detailed insights into the characteristics of crystalline materials. In order to correlate the structure, chemistry and physical properties of micro- and nanoscaled materials, the various TEM techniques for imaging, diffraction and spectroscopy have to be combined. The increasing use of micro- and nanoscale materials where the reduced dimensionality may drastically change the physical properties demands imaging and composition analysis with high spatial resolution for correlating the structure and properties of the materials investigated. In the case of single crystals, the classical parallel beam electron diffraction technique, i.e. selected-area electron diffraction, is commonly used to gain information about the degree of crystallinity of the materials as well as of basic parameters of the crystal structure (e.g. lattice parameter, type of Bravais lattice) and specimen orientation. For specimens containing numerous differently oriented crystallites, the application of the classical electron diffraction techniques using one zone-axis orientation at a time for the determination of the crystallite structure and orientation can be very time consuming. Typically, scanning electron microscopy based electron backscattered diffraction (EBSD) is applied to the orientation determination of larger crystallites with smooth morphology. A novel characterization tool that provides similar information for nanocrystalline materials of arbitrary shape is available with precession electron diffraction (PED) add-on devices to TEMs.
First, we will illustrate the time-consuming and limited application of classical TEM methods for the structure characterization of differently oriented inclusions in $LiAlO_2$ single crystals as well as for the phase determination of FePt crystallites. Finally, crystallite phase and orientation mapping of MnAs crystals embedded in a GaAs matrix by means of automatically analyzed PED spot patterns will be presented.

New techniques for TEM nano-analysis: precession electron diffraction for structure determination and ("EBSD-TEM like") high resolution crystallite phase/orientation mapping

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Precession electron diffraction (PED) is a promising technique for electron diffraction pattern collection very close to kinematical condition (like in X-ray diffraction) allowing this way to solve ab-initio crystal structures of nanocrystals. PED intensities help to solve nanocrystal structures (inorganic metals, ceramics, minerals up to polymers, organic structures, pharmaceuticals, etc.), even in cases where X-ray synchrotron data may fail to solve the structure. A new exciting application has also been developed for an "EBSD-TEM like" phase and orientation mapping of nanocrystals. The PED precession interface may control a scanning with a small step (1-35 nm, depending on TEM electron source) through a sample area (for example 5 x 5 μm^2), resulting in the collection of a large number of diffraction patterns which are compared one by one by cross-correlation techniques with a series of generated diffraction patterns (templates) of all possible orientations of known phases existing on the scanned area. The resulting high quality, high resolution (1-2 nm) orientation and phase maps obtained in TEM are superior to equivalent EBSD-SEM orientation maps. Moreover, there is no need for specific surface specimen preparation (like in EBSD-SEM), because with this technique all diffracting crystals have enough signal to produce high resolution orientation maps. Such orientation and phase maps may be produced in a few minutes for any materials, making the technique highly attractive for high throughput TEM structure analysis.

New software developments for electron crystallography: eMap and eSlice

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eSlice is a new program within the eMap software package. It is designed for the crystal structure refinement taking dynamical diffraction into account. The calculations are based on the advanced multislice calculation including the exact 3D crystal potential integration method. The program uses a set of electron diffraction intensities extracted from an experimental pattern and a starting crystal structure model.

Solving structures from PED data by charge flipping

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Charge flipping is still relatively new, but increasingly popular ab initio structure solution method. It takes diffracted intensities as an input, and outputs approximate electron density map in the unit cell of the crystal. It has been used mainly for x-ray diffraction data, but it can be applied to other radiation sources (neutrons or electrons) as well. Charge flipping has been also successfully used for solution of several structures from precession electron diffraction data. Solution of crystal structures from PED data poses special challenges for charge flipping. The amount of data is often limited to a couple of measured zones, or to the range of accessible tilt angles of the sample holder. Charge flipping is a Fourier-based technique, and incomplete data represent a serious challenge, as the Fourier maps from incomplete data are obscured by artifacts. This problem can be solved by applying deconvolution techniques to the raw solution, so that the artifacts are removed, and the electron density map becomes clearer and easier to interpret. The algorithm has been implemented in the charge-flipping program Superflip, and applied to several test data sets.

Coupling Template Matching and Precession Electron Diffraction for reliable identification of crystallite orientations and phases in TEM

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The developments of nanomaterials for structural or functional applications lead to an increasing demand for reliable characterization of their structure. For crystallized materials this includes proper measurements of the grain sizes and the local crystallographic texture. The electron backscatter diffraction technique (EBSD) in a scanning electron microscope (SEM), that had played an essential role in the characterization of these features, suffer from an intrinsic limitation when nano-scaled structures are concerned. The transmission electron microscope (TEM), on the other hand, exhibits a higher spatial resolution that appears essential in cases of small crystallite sizes.
A specific procedure that allows orientation maps to be constructed automatically, but also rapidly, starting from the diffraction patterns collected on a TEM operating in nanobeam mode has been developed. The pattern identification is performed using a reverse approach: it consists in (i) generating a series of theoretical diffraction patterns, called templates, that cover all the range of possible orientations and/or phases and (ii) comparing the acquired image to all the templates in order to extract the most approaching one. The crystal orientation is given by the Euler's angles used to calculate the selected template. The association between the theoretical template and the experimental diffraction pattern is performed through an image matching technique that will be described in detail. Essentially, a correlation value is calculated for each template and the highest one determine the adequate orientation.
One of the main difficulties related to electron diffraction is that the scattering by the atoms produces rather complex images. In particular, dynamical effects as well as inelastically scattered electrons lead to strongly orientation sensitive images. Consequently, the aspect of the real diffraction pattern differs significantly when the sample is titled only a fraction of degree. These differences will favor mis-indexing. Fortunately, the electron precession technique, which was proposed by Vincent and Midgley in 1994, is known to improve the quality of the diffraction pattern. More precisely, the scattered beams appear less sensitive to dynamical effects and may be estimated reasonably by integrated kinematic intensities. This leads to patterns that varies less rapidly with the sample orientation and that should be more readily reproduced with a limited number of templates. Therefore, the indexing quality with the pattern matching technique is increased if the electron beam is precessed.
Both template matching and electron precession will be addressed in this talk. The hardware and software aspect of the resulting TEM attachment, called ASTAR, will be described.

Development of a fast CCD Camera for Electron Diffraction Imaging in Conventional TEM

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The ease of producing diffraction patterns in TEM and using the data to understand crystal structures has made it a powerful analysis tool. Despite previous developments in CCD cameras for recording EM images, viewing and recording diffraction patterns with CCD cameras still remain challenging tasks. As part of our research we have found that many people rated their satisfaction with current CCD cameras and diffraction performance as low, and indicated the importance value as high.
Diffraction patterns as normally observed consist of a bright central transmitted beam and diffracted beams surrounding it. The intensity in the transmitted beam can be several orders of magnitude higher than the diffracted beams. The major concern with using CCD cameras to capture diffraction patterns is damage to the CCD and scintillator by the intense transmitted beam.
This talk will describe our latest effort in developing a fast CCD camera that meets the challenges in imaging electron diffraction patterns.

Atomic scale structural and elemental analysis with the new JEM-ARM200F

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Recently, scanning transmission electron microscopy (STEM) has being strongly progressed owing to a stable field emission gun (FEG) [1] and sophisticated spherical aberration correctors (Cs-corrector) [2]. In STEM, a high angle annular dark-field (HAADF) image is formed with incoherently scattered electrons [3]. It provides a contrast sensitive to the chemistry, which is known as Z-contrast. Therefore the structural image interpretation is easier than in conventional bright-field high resolution transmission electron microscopy (HRTEM). The Cs-corrector for the probe forming lens system enables it to form an electron probe of sub-Angstrom size, resulting in sub-Angstrom resolution imaging. Moreover, a higher intensity of the probe owing to the Cs-corrected STEM provides also an elemental analysis at atomic scale resolution with good signal-to-noise ratio.

In this work, high-resolution structural and elemental analyses were performed on a $SrTiO_3$ perovskite single crystal, with a Cs-corrected STEM. The instrument used for this experiment was the new JEOL JEM-ARM200F (Atomic Resolution analytical Microscope, 200 kV FEG TEM/STEM) equipped with Cs-corrector for probe forming lens system.

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A critical review of orientation microscopy techniques in SEM and TEM

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The term orientation microscopy refers to techniques which use the spatially resolved measurement of crystallographic orientations and phases to reconstruct the microstructure of a sample. The most common techniques are those which employ electron diffraction in SEM or TEM to reveal the crystallographic information. In the SEM it is the electron backscatter diffraction (EBSD) technique which is widely used. The EBSD technique is limited in spatial resolution by the electron-matter interaction volume. The best achievable spatial resolution on a material of medium atomic number is currently in the order of about 30 nm [1].
On the TEM side, several electron diffraction techniques have been explored. The techniques based on Kikuchi diffraction pattern evaluation take the most straight-forward approach [2]. They are, however, limited to samples which do have only a low defect density because of the sensitivity of Kikuchi patterns to lattice imperfections. The present authors have not seen any investigation using this technique which could not have been performed equally well – and with less efforts in sample preparation – by EBSD. On the other hand, spot diffraction techniques can be employed. Here the main obstacle is the determination of orientations using the intensity distribution of the diffraction spots and not only their geometrical arrangement. The template matching technique [3] can be considered as a break-through to overcome this difficulty. The spot diffraction pattern technique can be employed by scanning the beam over the sample and analyzing the recorded spot patterns. This approach is straightforward but may suffer from high beam dose at every investigated point and problems such as sample drift during the measurement. An alternative is based on the collection of a large amount of dark field images, obtained under different primary incident beam directions [4]. From these images, spot diffraction patterns can be reconstructed and analyzed. This technique suffers, however, particularly from the problem of the high parallelism of the primary beam which reveals a lot of dynamic effects in the diffraction pattern and makes the analysis of the diffraction patterns more difficult.
All TEM diffraction techniques compete with the EBSD technique. If they do not show any added value, e.g. in terms of spatial or angular resolution, their use is not justified as difficult sample preparation and a lack of statistical relevance of the observed areas are large disadvantages with respect to the EBSD techniques. In particular the spatial resolution of TEM orientation microscopy techniques must be critically questioned as it is limited by the sample thickness. If this thickness is on the order of 50 to 100 nm, the resolution is frequently not better than that of EBSD. TEM techniques, therefore, imperatively need to deal with the problem of overlapping diffraction patterns arising from several grains across the sample thickness [5].

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