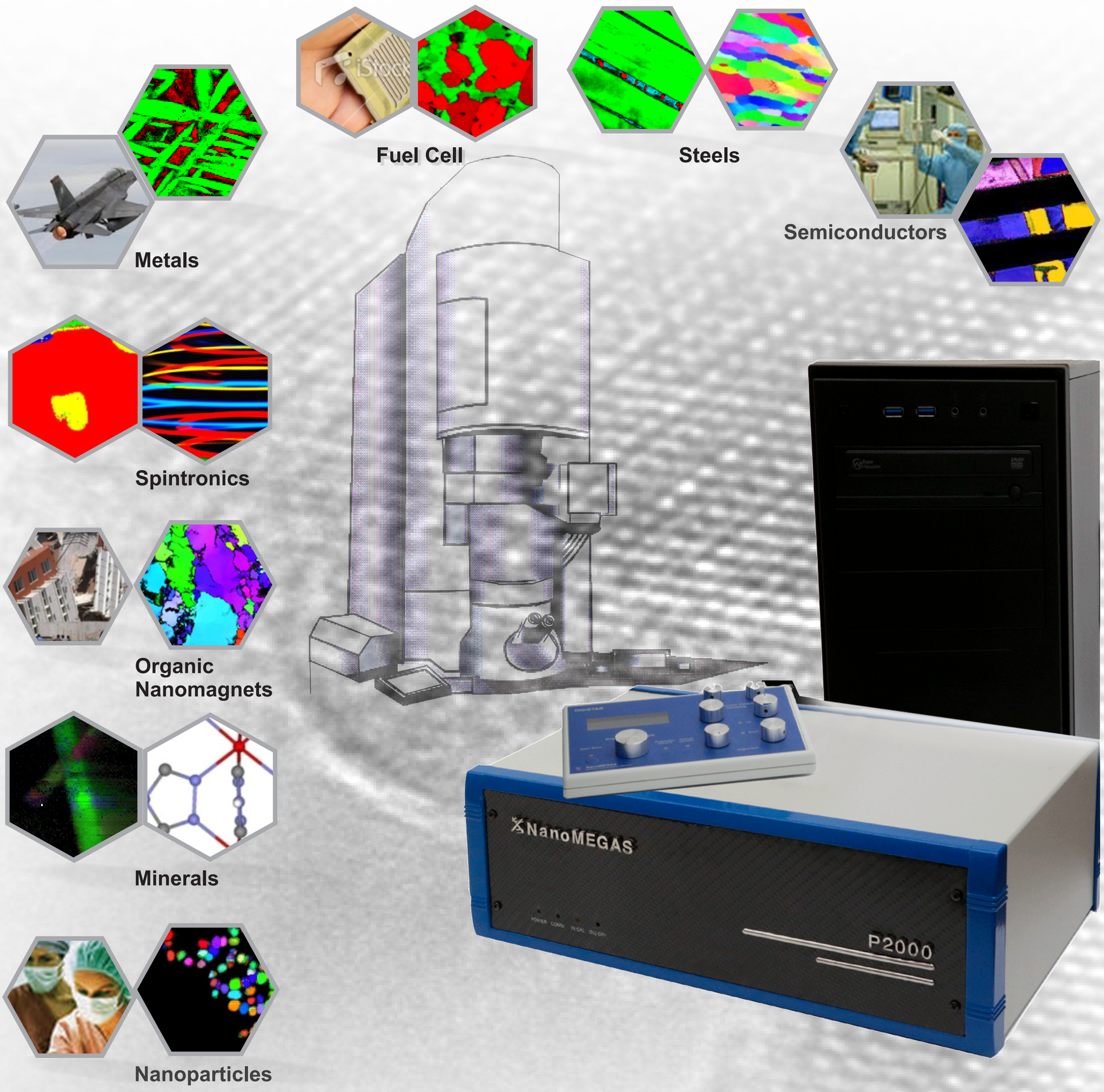


Applications

- **Precession Electron Diffraction (PED)**
- **EBSD TEM Like Orientation & Phase Mapping (ASTAR)**
- **3D Diffraction Tomography (ADT-3D)**
- **Electron Pair Distribution Function(e-PDF)**
- **Auto Strain Mapping**
- **Enhanced EELS & EDS Spectroscopy**

NanoMEGAS was created in 2004 by a team of scientists and experts in the field of electron crystallography and catalysis and was the first to commercialize a universal precession device (spinning star) for TEM.

Being an important part/contribution of this scientific success story almost 13 years later precession diffraction/electron crystallography applications are present in almost every major electron microscopy/X-Ray crystallography scientific congress, with more than 200 publications from various laboratories worldwide and dedicated issues of major scientfic microscopy journals.

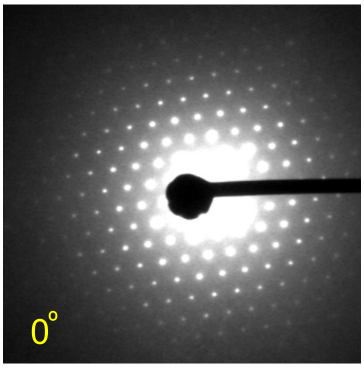
Precession Electron Diffraction (PED)

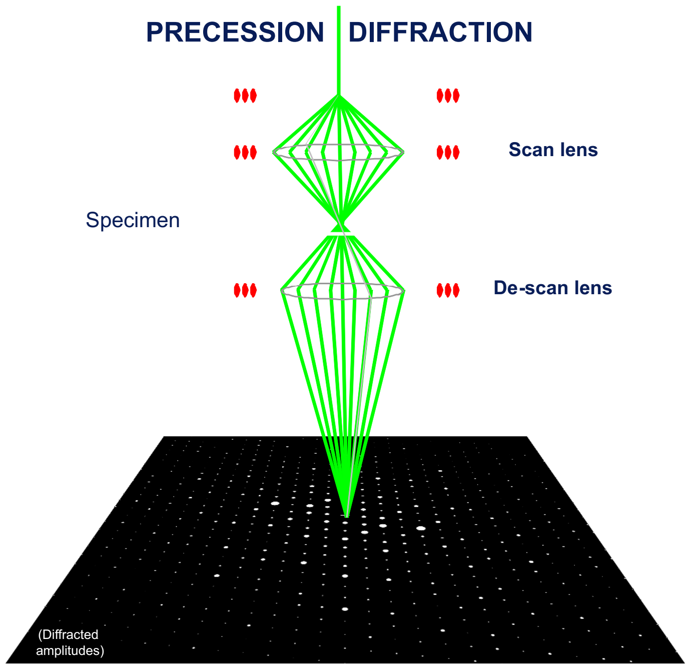
Transmission Electron Microscope (TEM) allows to study nm size crystals. By tilting (manually/automatically) around an arbitrary axis a single nanocrystal (tilt range usually > 120º e.g. 120 diffraction patterns with 1º) in combination with precession electron diffraction (PED), the reciprocal cell can be reconstructed and crystal cell parameters can be evaluated automatically and precisely (error 2-5%). Reflection intensities can also be measured automatically (Completeness of reciprocal space>60%) to enable the solution of the crystal structure.

Any TEM (LaB6/FEG, 120-300 kV) can be used in TEM/STEM mode using a small beam (e.g. 50nm or less). Selected Area Electron Diffraction (SAED) or Nanobeam Electron Diffraction (NED) can be used with combination with any standard CCD Camera (1K X1K or higher) to collect data.

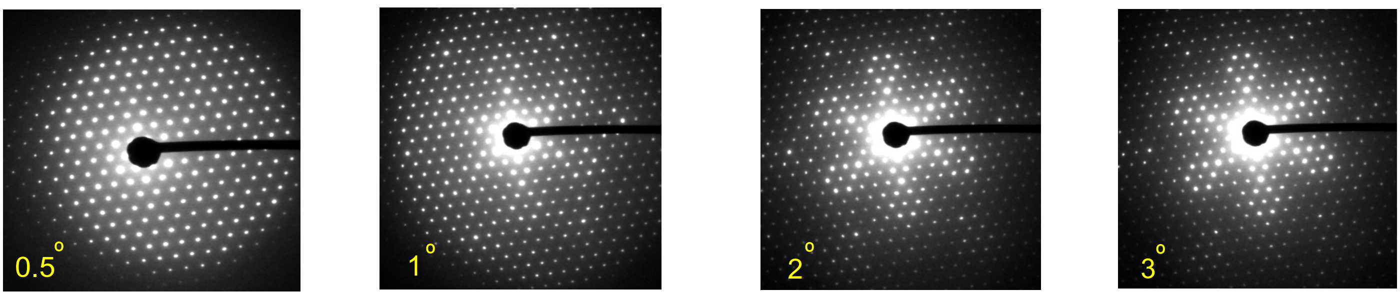
DigiSTAR hardware from NanoMEGAS enables precession diffraction to obtain high quality quasi-Kinematical integrated intensities for accurate structure solution.

Non - Precessed

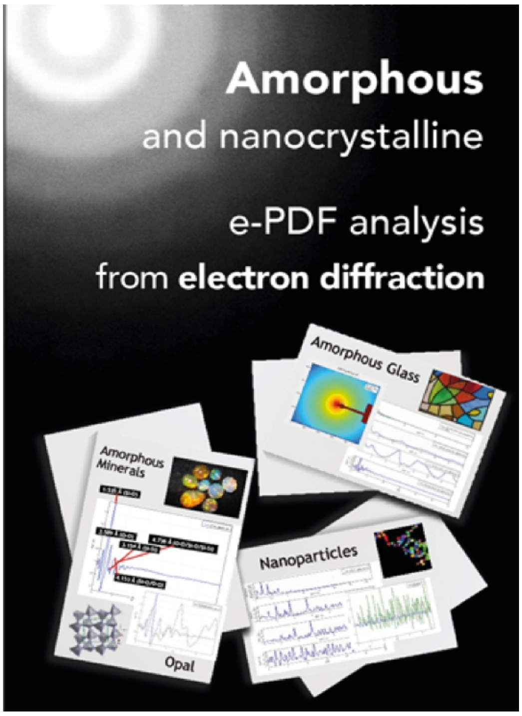




Precessed



As the precession angle increases from 0º to 3º, the diffraction pattern goes to higher resolution (i.e. more diffraction spots are seen).



Software solution for structure analysis of nanocrystalline and amorphous materials via Electron Pair Distribution Function(ePDF). Possibility of identify/fingerprint amorphous or nanocrystalline phases using PDF spectra , estimation of interatomic distances and direct estimation of short range order in metallic glasses, amorphous metals/alloys,nanoparticles and minerals. Electron Diffraction data suitable for ePDF analysis can be acquired with CCD camera (12 bit minimum) and Image plates.

Advantages

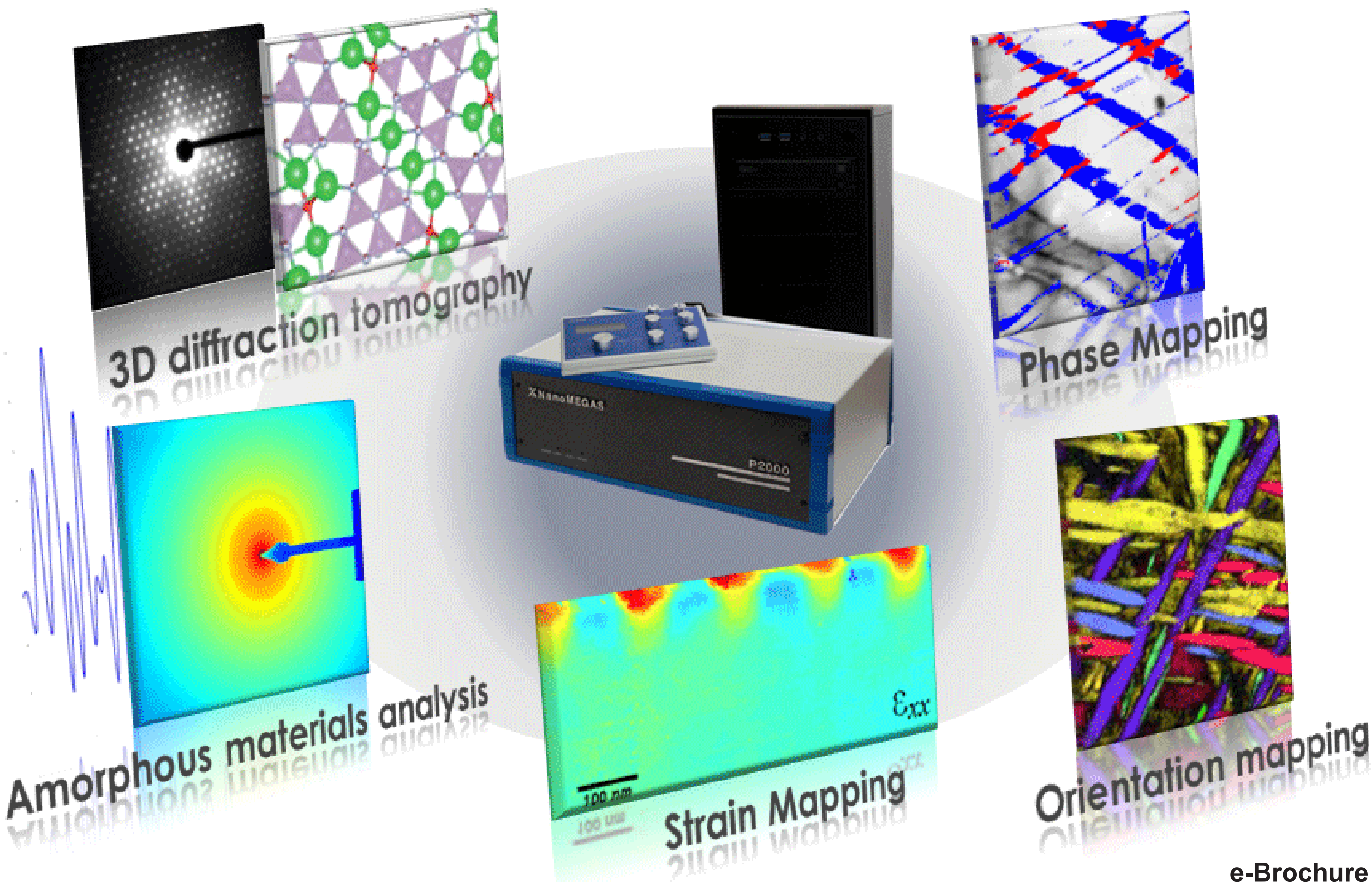
Diffraction data can be collected in any TEM microscope (FEI, JEOL, Zeiss etc.)

Any CCD/IP can be used for Data collection

Data can be collected in either NBD or SAD mode

ePDF acquisition time is much shorter then X-Ray PDF (Few seconds Vs almost 24 hrs)

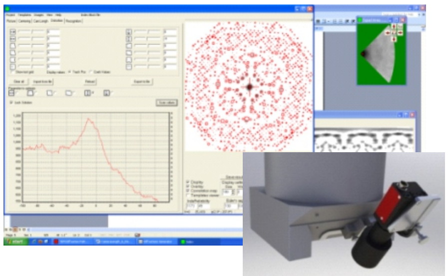
PDF obtained from election diffraction data and PDF obtained from X-ray are fully comparable, using e-PDF very small amorphous areas can be studied

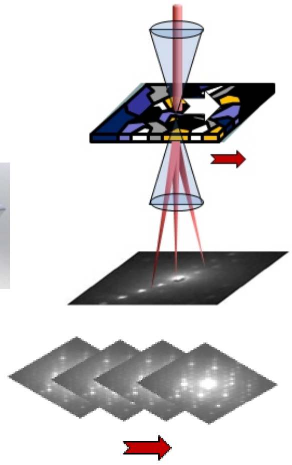


Automatic TEM Orientation/Phase Mapping Precession Unit

ASTAR is an automatic crystallographic indexing and orientation/phase mapping tool, developed for any TEM.

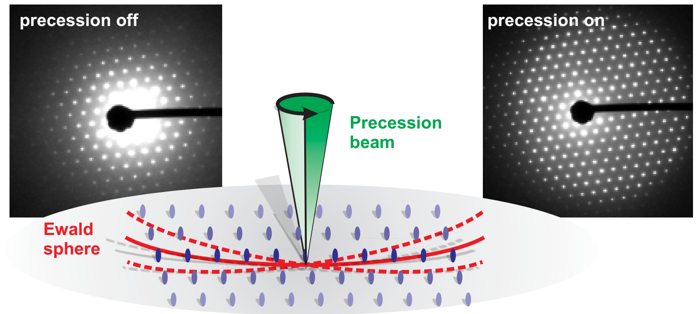
The electron beam is scanned in combination with beam precession through the sample area of interest; beam scanning is done by the NanoMEGAS DigiSTAR precession unit without using an inbuilt STEM mode.



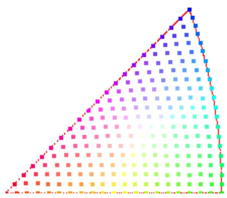


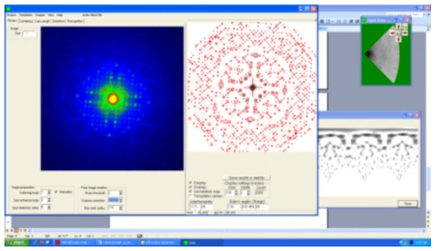
A number of electron diffraction (ED) spot patterns from several sample locations are acquired at high speed, using a dedicated fast CCD camera placed in front of the TEM screen; local crystal orientation(s) are obtained by comparing all individually obtained ED spot patterns via cross-correlation matching techniques with pre-calculated ED templates.

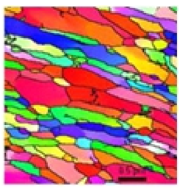
Detection and orientation/phase mapping of different (known) crystallographic phases and orientations in a crystal structure requires collection of high quality ED patterns. Electron beam Precession diffraction is extremely useful for obtaining patterns with a large number of spots almost twice as many compared to conventional selected area electron diffraction (SAED) and without dynamical effects (e.g. Kikuchi lines).

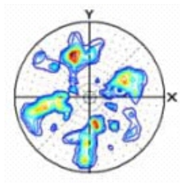


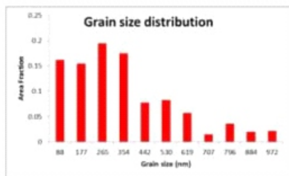
Therefore, phase and orientation identification based on pattern matching between experimental precession patterns with simulated templates is very reliable and precise. Required diffraction templates are generated every 1º through the respective symmetry invariant section (f) of orientation space (stereographic triangle for cubic crystals). Resulting colour maps show with nm detail grain structure including boundaries (g), grain size distribution, and relative pole figures



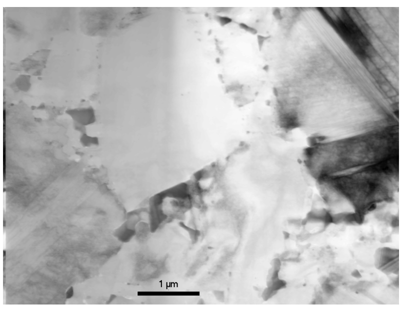




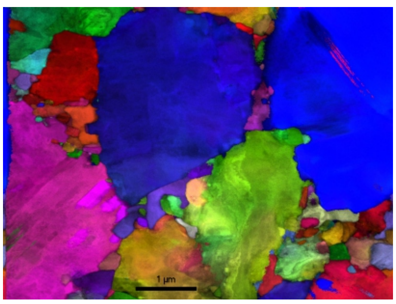




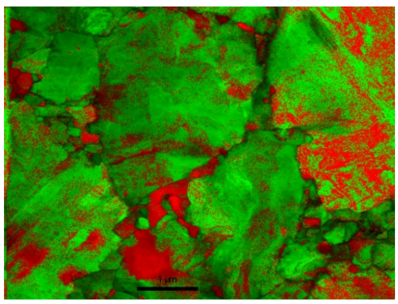
ASTAR resolution on a TEM is determined by the electron probe size and can reach 1 nm on orientation maps with TEM-FEG microscopes.



Virtual Bright-Field



Orientation Map



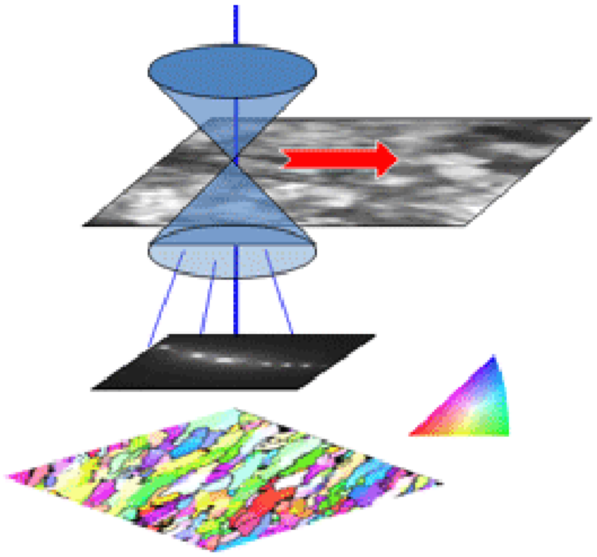
Phase Map

Advanced Tools For Electron Diffraction

TOPSPIN

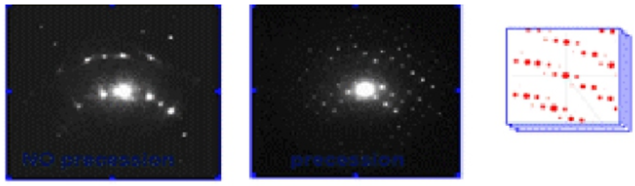
A next-generation TEM application experiment framework for materials science characterization enabled by synchronized beam scanning, beam precession and advanced analysis

ASTAR/Topspin acquisition

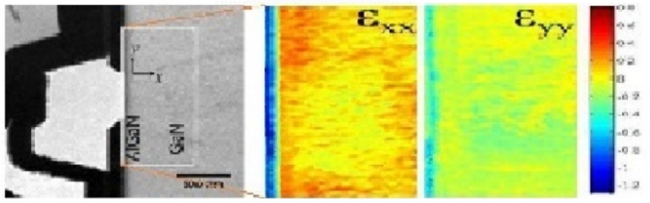


Orientation map

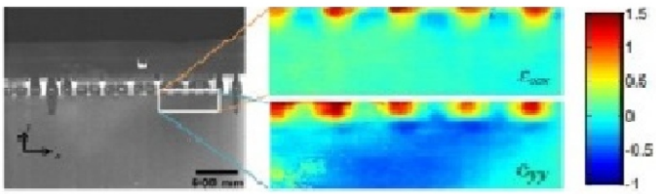
Precession electron diffraction spot patterns are acquired to correlate with local crystal orientations



Using precession diffraction the number of ED spots observed increases (almost double);is very reliable compare PED patterns with theoretical templates



Strain Maps for AlGaN/GaN HEMT



Strain Maps for the Si region of the PMOS device

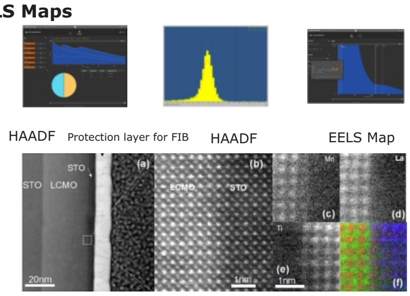
Enhanced EELS Spectroscopy

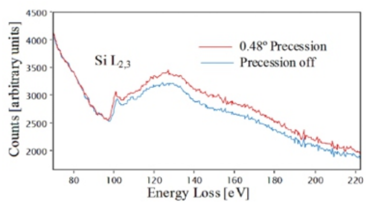
Beam Precession in EELS & EDX spectroscopy enhance the signal reducing channeling effects. Automated quantification with statistical error analysis is available. Multiple scattering derived from an automatically measured relative thickness.

Highly-Automated EELS Elemental Analysis

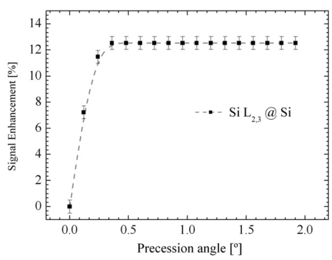
- Minimal user input required
- Specify elements to quanitfy
- Chemical shift of each elemental edge
- Automated quantification with statistical error analysis
- Multiple scattering derived from an automatically measured relative thickness

EELS Maps

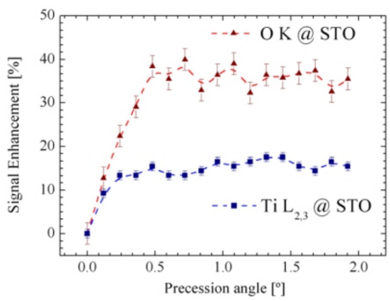


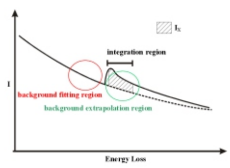


0.48 degree precessed and precession- less EEL spectra showing the Si L2,3 edge in a Si crystal in <110> zone axis condition



Signal enhancement as a function of precession angle for the SiL2,3 edge in a Si crystal in <110> zone axis condition





The signal enhancement (SE)for a given precession angle is given by

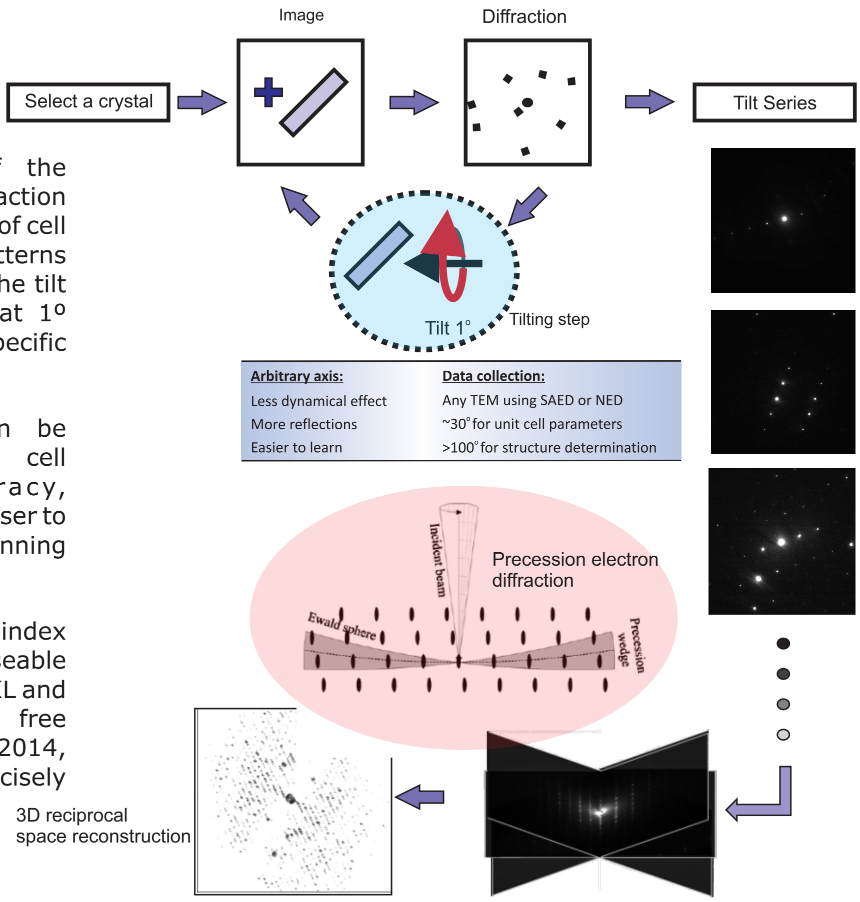
SE = (I (α) – I (0)) / I (0)

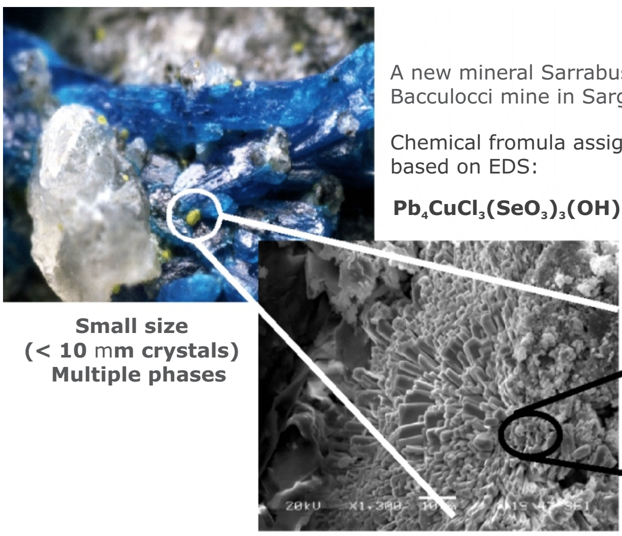
3D Diffraction Tomography

This program covers reconstruction of the reciprocal space based on 3D electron diffraction (ED) data and the subsequent determination of cell parameters. Data consist of precession ED patterns when the holder is tilted (manually) within the tilt limits of the TEM (usually -40º to +40º at 1º angular step with single tilt or of specific tomography holder).

The reconstructed reciprocal space can be visualized to determine possible crystal cell parameters (with in 2-3% accuracy, automatically). The software also allows the user to spot special crystallographic effects like twinning and disorder.

ADT3D software module allows the user to index every reflection and to extract intensities useable for structure analysis procedure. Listing of HKL and intensities allow to be used to various free academic software for structure solution (SIR2014, SHELX, etc.) in order for the user to solve precisely the atomic 3D crystal structure





A new mineral Sarrabusite found in the Bacculocci mine in Sargegna (Italy)

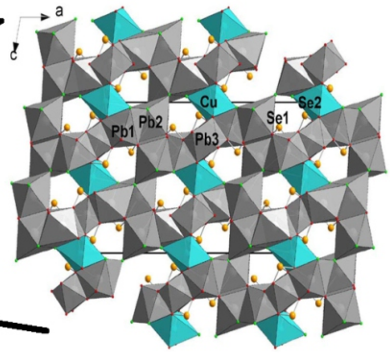
Chemical fromula assigned based on EDS:

$Pb_4CuCl_3(SeO_3)_3(OH)$

Small size
(< 10 mm crystals)
Multiple phases

Same model was obtained by 4 different datasets from different crystals

$Pb_5CuCl_4(SeO_3)_4$



TEM : FEI Tecnai 20F; 1k CCD Gatan

Tilt: ± 46° in steps of 1°
876 independent reflections R_{sym} = 17%

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