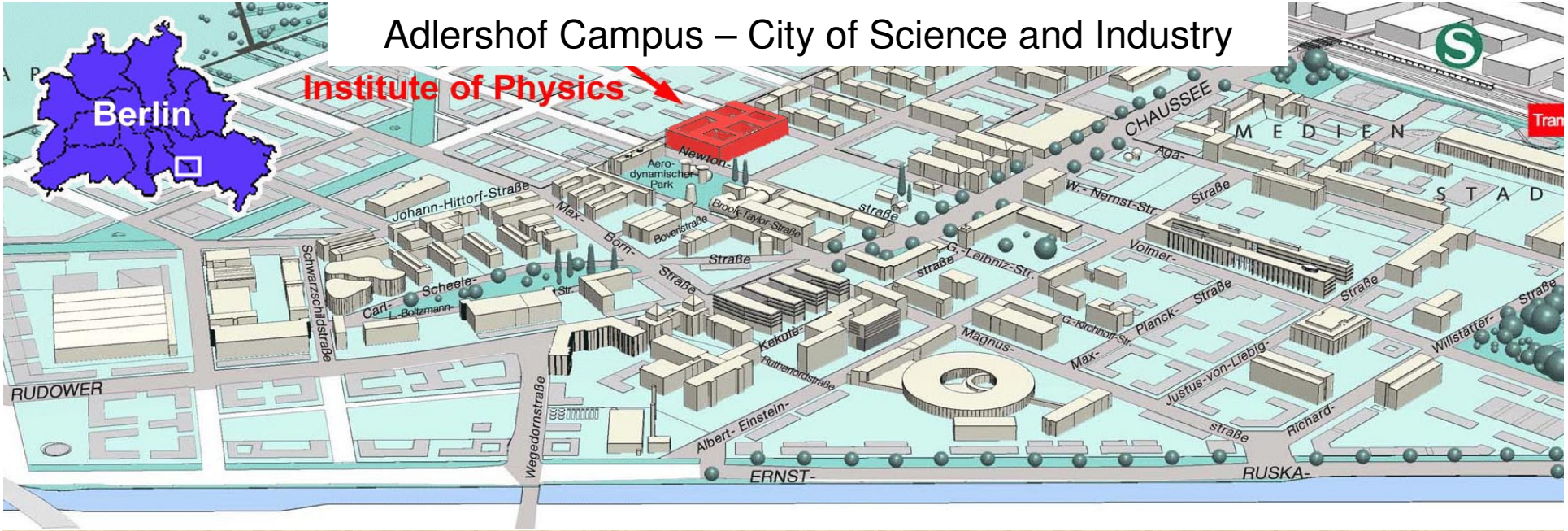
International Workshop
"Facets of Electron Crystallography"
Berlin, Germany
7-9 July 2010

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

W. Neumann,
I. Häusler, A. Mogilatenko, H. Kirmse

Humboldt University of Berlin,
Institute of Physics, Chair of Crystallography
Newtonstrasse 15, D-12489 Berlin, Germany
Phone ++49 30 20937761, Fax ++49 30 20937760
Email: wolfgang.neumann@physik.hu-berlin.de
Web: http://crysta.physik.hu-berlin.de

Adlershof Campus – City of Science and Industry



Humboldt University of Berlin

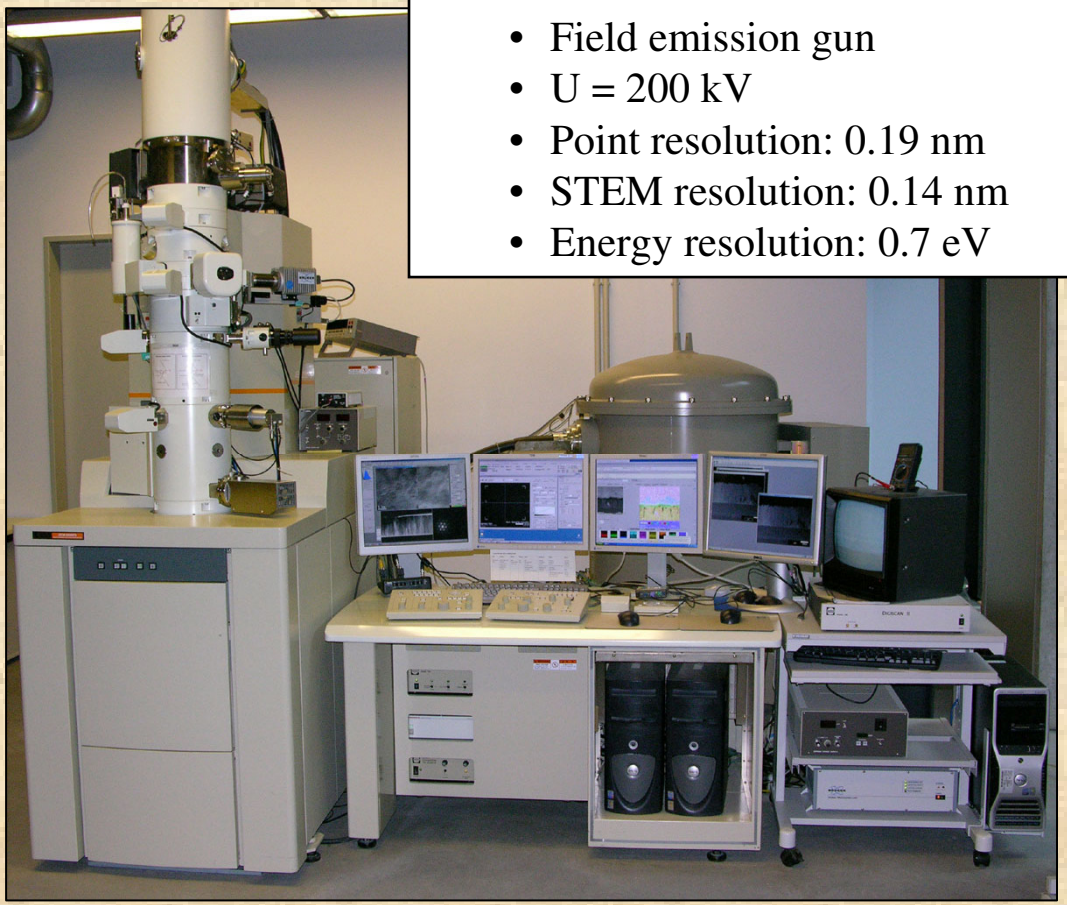
Institute of Physics
Chair of Crystallography

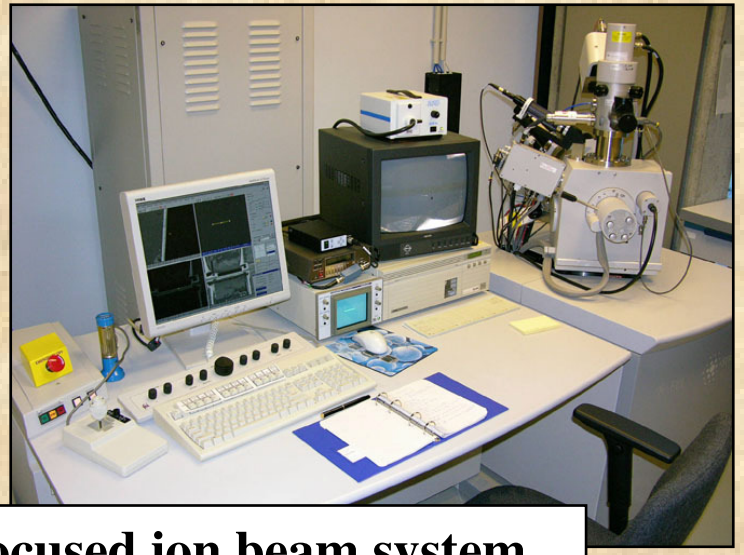
Newtonstrasse 15
D-12489 Berlin
Germany

Joint Laboratory for Electron Microscopy Adlershof (JEMA)

TEM/STEM JEOL 2200 FS

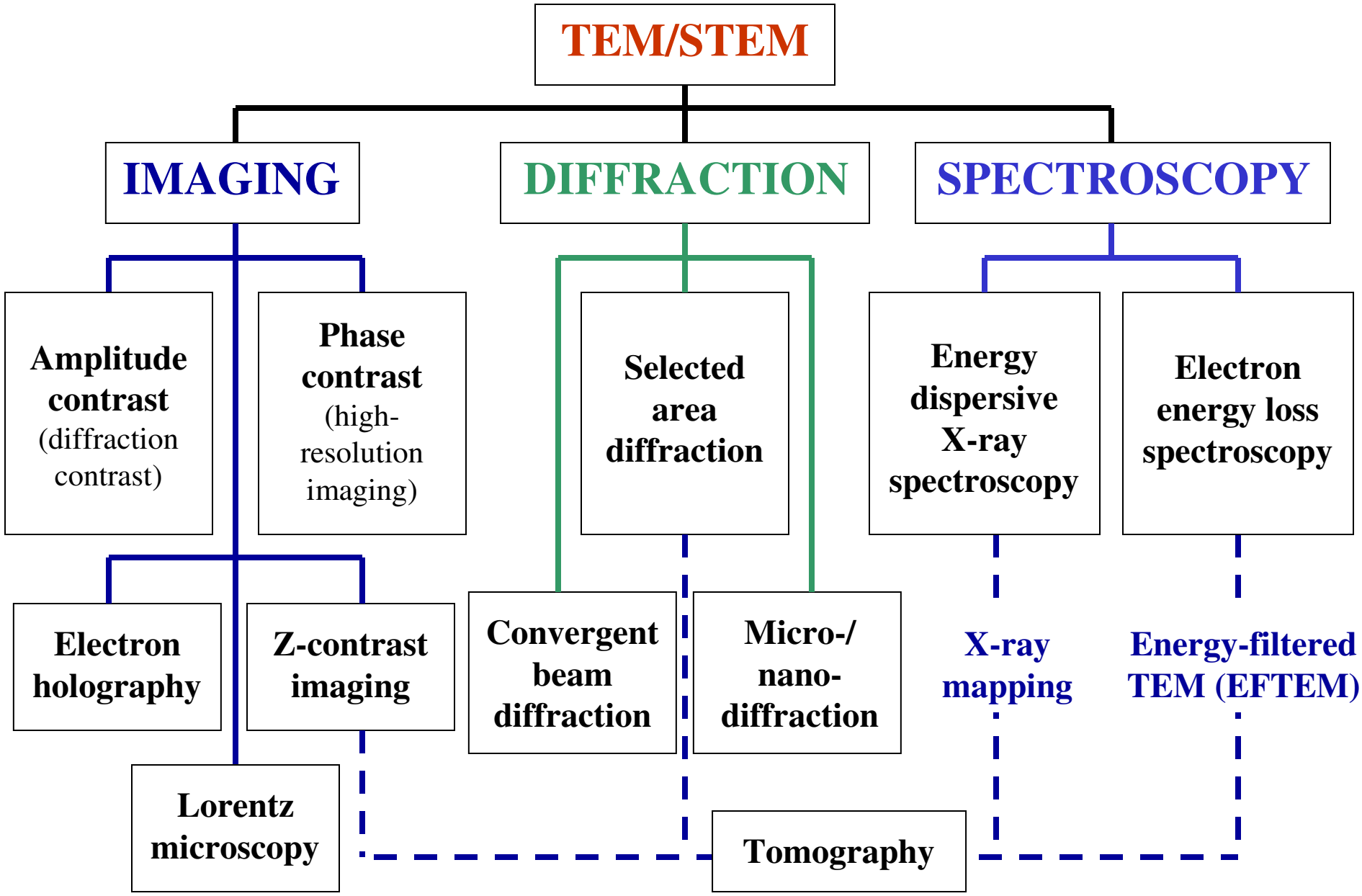
- Field emission gun
- U = 200 kV
- Point resolution: 0.19 nm
- STEM resolution: 0.14 nm
- Energy resolution: 0.7 eV

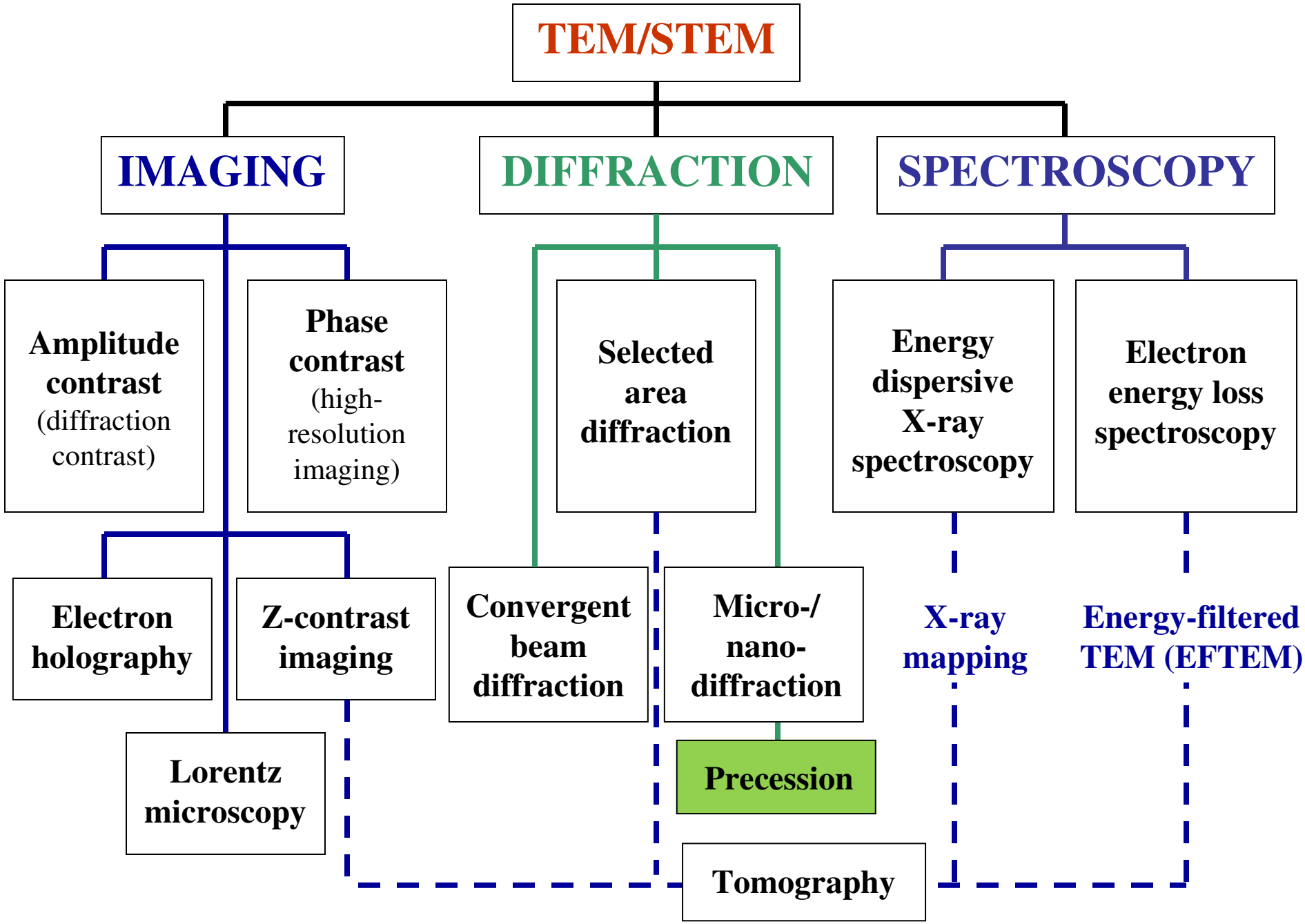




Focused ion beam system FEI FIB Strata 201

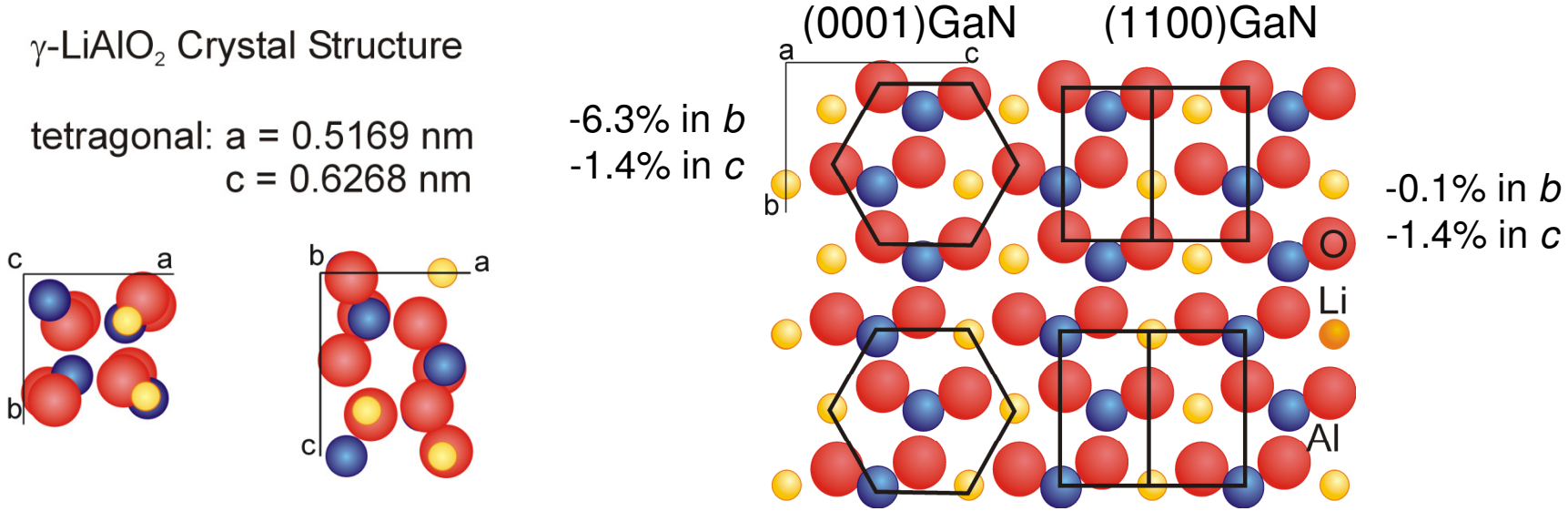
- TEM specimen preparation
- Cross sections
- Target preparation
- Surface morphology tailoring
- Ion beam diameter: 20 nm

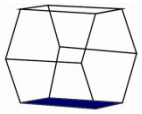
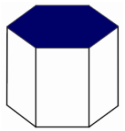

TEM/STEM
IMAGING
DIFFRACTION
SPECTROSCOPY
Amplitude contrast (diffraction contrast)
Phase contrast (high-resolution imaging)
Selected area diffraction
Energy dispersive X-ray spectroscopy
Electron energy loss spectroscopy
Electron holography
Z-contrast imaging
Convergent beam diffraction
Micro-/ nano-diffraction
X-ray mapping
Energy-filtered TEM (EFTEM)
Lorentz microscopy
Tomography


TEM/STEM
IMAGING
DIFFRACTION
SPECTROSCOPY
Amplitude contrast (diffraction contrast)
Phase contrast (high-resolution imaging)
Selected area diffraction
Energy dispersive X-ray spectroscopy
Electron energy loss spectroscopy
Electron holography
Z-contrast imaging
Convergent beam diffraction
Micro-/ nano-diffraction
X-ray mapping
Energy-filtered TEM (EFTEM)
Lorentz microscopy
Precession
Tomography

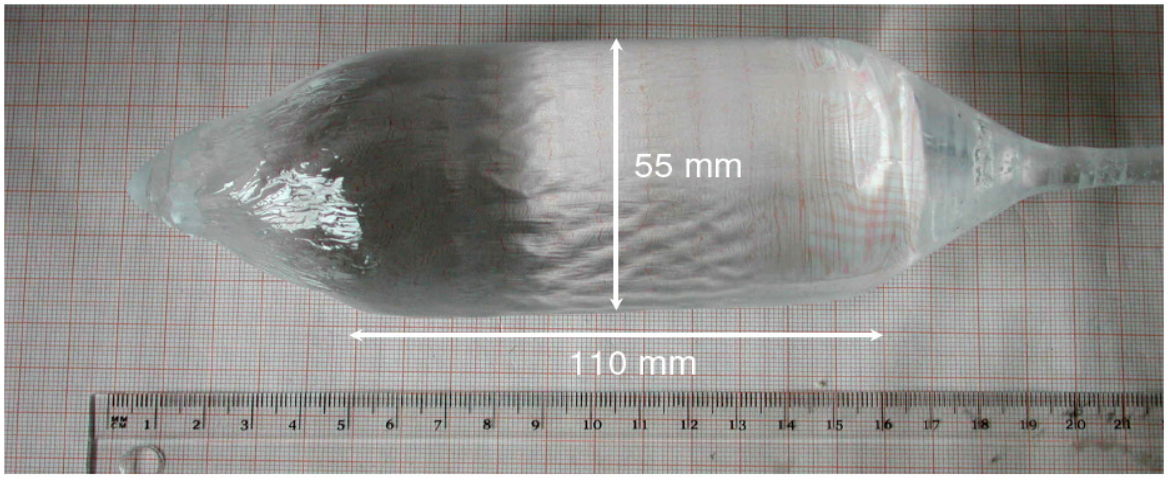
Characterization of single crystalline $LiAlO_2$ substrates for subsequent GaN epitaxy

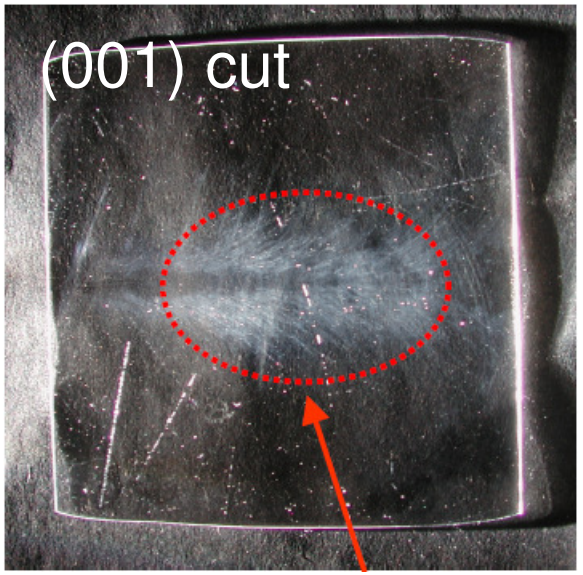
$LiAlO_2$(100) substrates for GaN based optoelectronics



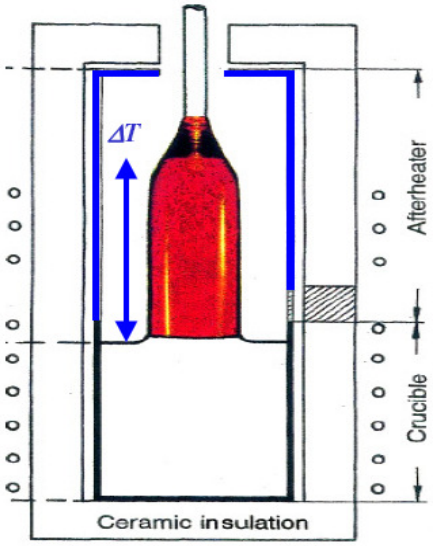
- Almost a lattice matched substrate for GaN epitaxy;
- $LiAlO_2$(100) allows the growth of both polar c-plane and non-polar m-plane  GaN; 
- Fabrication of free standing GaN waffers, which can be used as substrates for subsequent homoepitaxy;
- $LiAlO_2$ self-separation from thick GaN layers

Growth of γ-$LiAlO_2$(100) single crystals by Czochralski technique





inclusions



FWHM < 40 arcsec

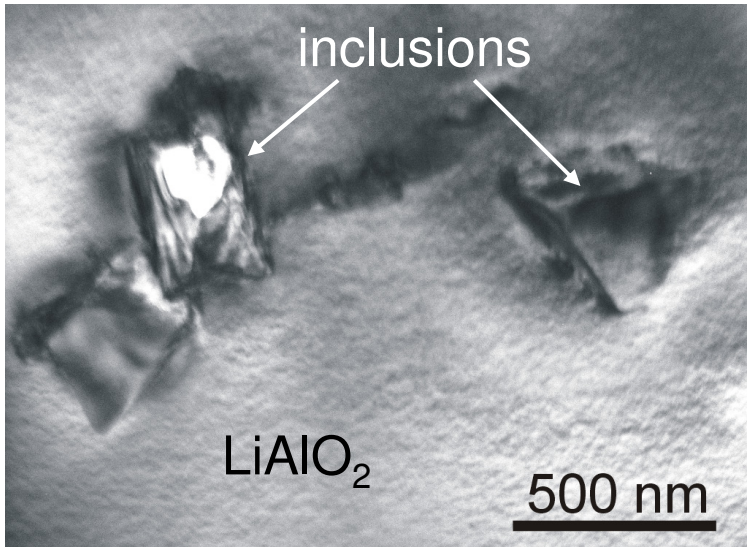
Problem:

Li_2O evaporation from the surface of the growing crystal or melt during the single crystal growth

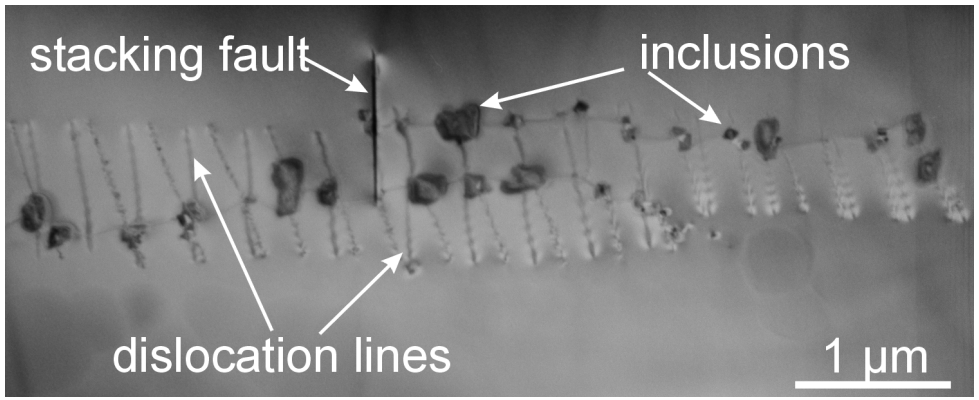
B. Velickov et al.,
Journal of Cryst. Growth 310 (2008) 214

Institute of Crystal Growth - Berlin

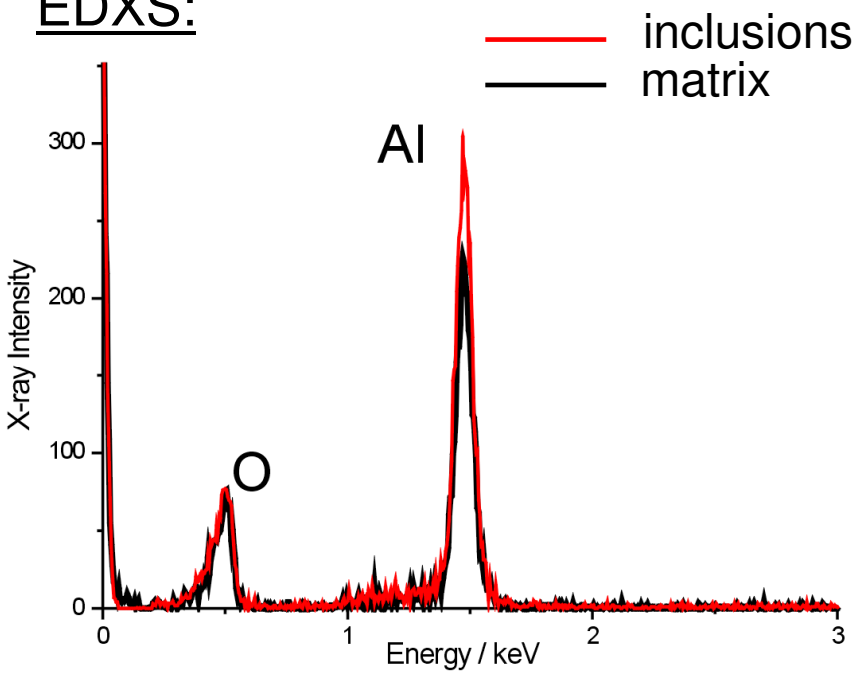
Inclusions in γ-$LiAlO_2$



- no common orientation relation to matrix
- idiomorphic shape



EDXS:



Phase analysis of inclusions in γ-$LiAlO_2$

Problems:
- a large number of possible phases, i.e. **$LiAl_5O_8$**, **Al_2O_3**, **α-**, **β-**, **δ-$LiAlO_2$** modifications
- inclusions are not homogeneously distributed in the γ-$LiAlO_2$ matrix, so that it is difficult to localize them during the specimen preparation

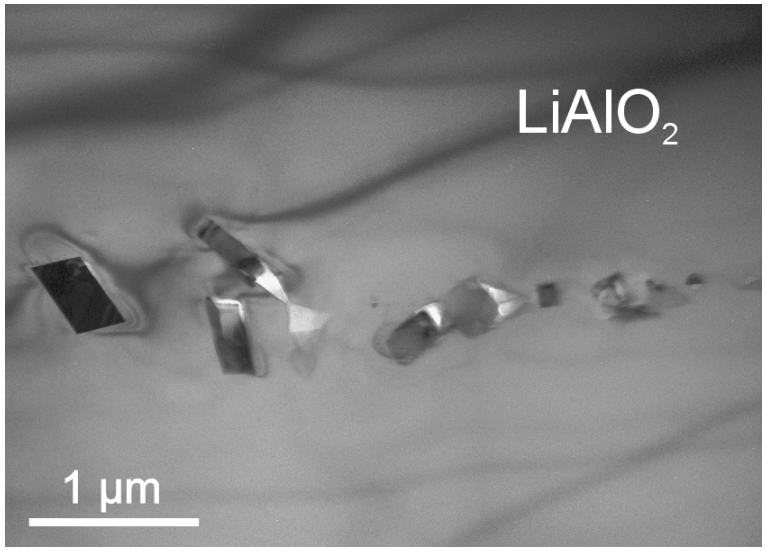
Solutions:

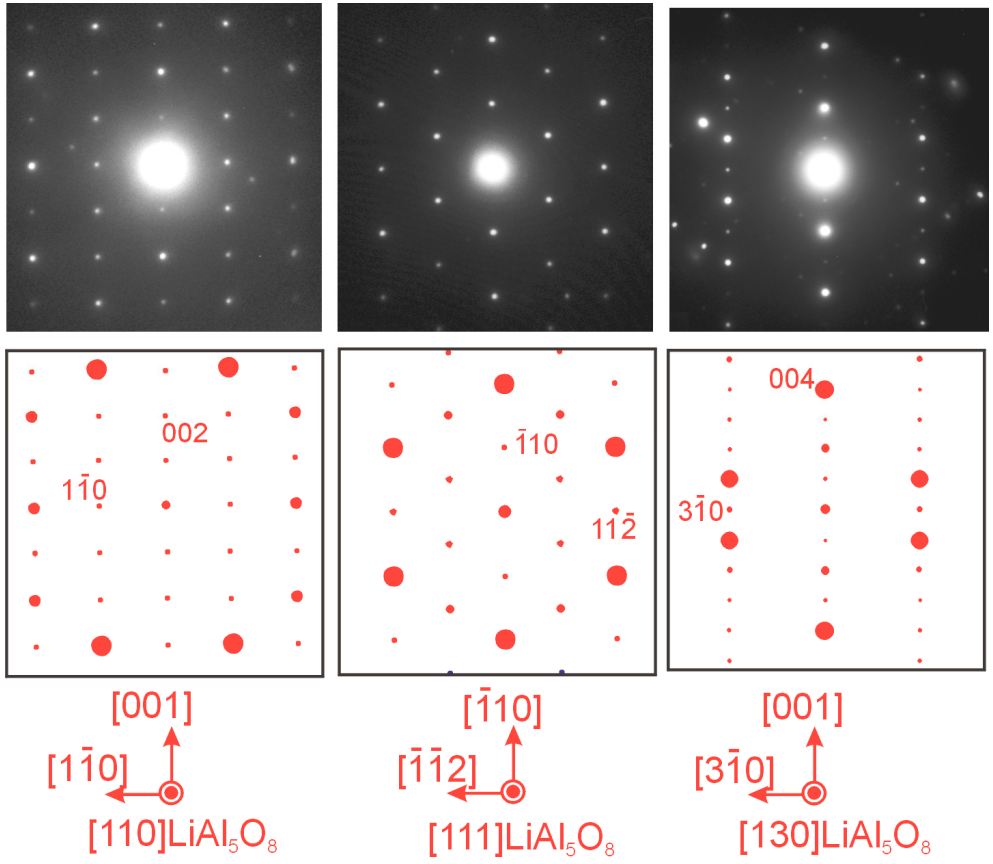
1. electron diffraction analysis along a number of low index zone axes
2. possibly ELNES- analysis of oxygen K-edge

Way:

1. prepare a ***large number*** of specimens (time consuming) and tilt, tilt, tilt
2. simulate fine structure of O-K edge for different phases and look if you can distinguish between them with energy resolution available at your TEM

Electron diffraction evidence for formation of $LiAl_5O_8$





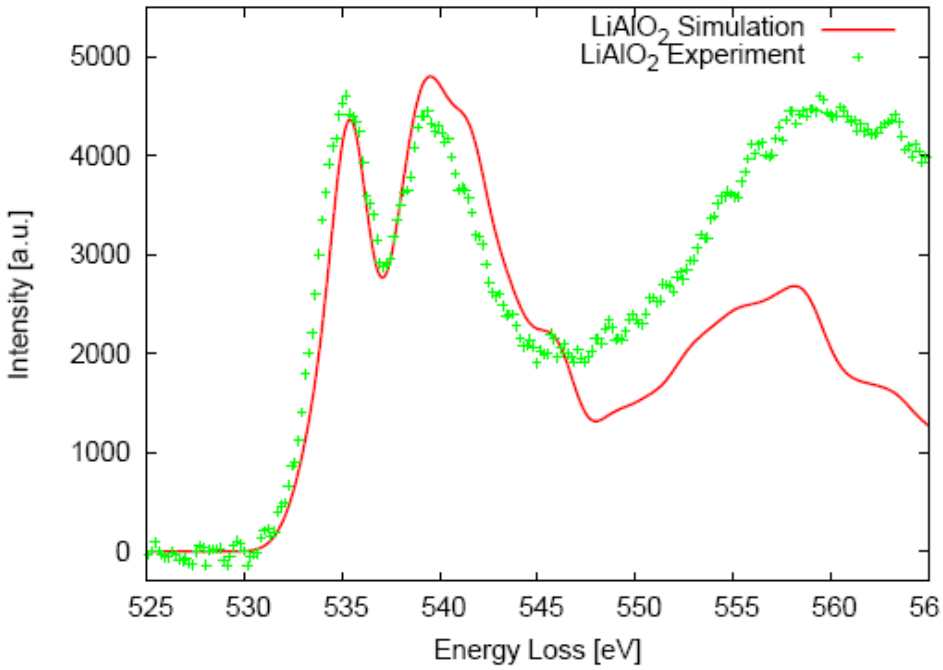
Explanation: Li_2O loss from the melt resulting in formation of unsolvable $LiAl_5O_8$ inclusions.

- number of prepared specimens: **13**
- invested time: **1 year**

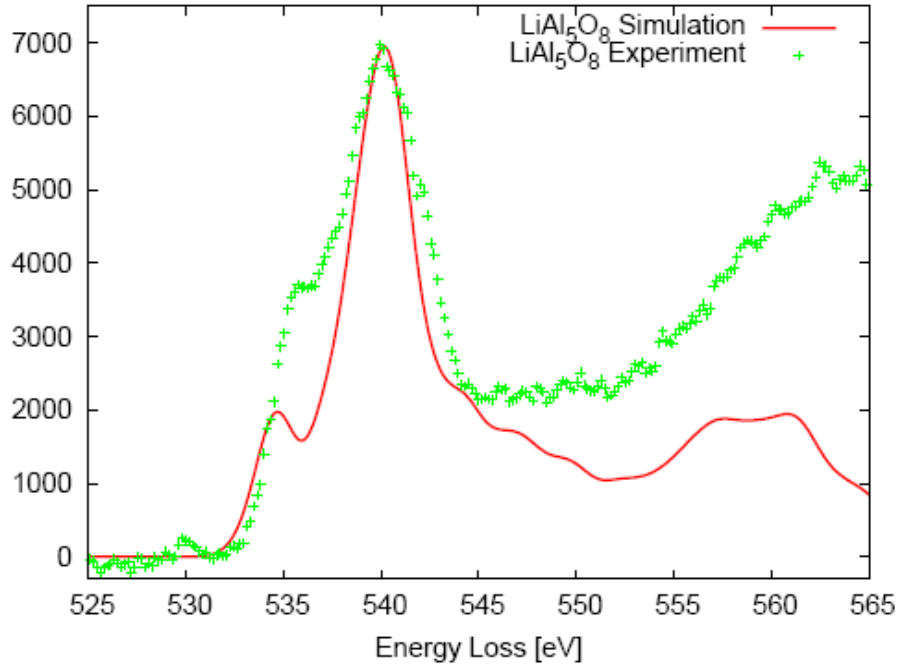
B. Velickov et al.,
Journal of Cryst. Growth 310 (2008) 214

ELNES evidence for formation of $LiAl_5O_8$

$LiAlO_2$ matrix



$LiAl_5O_8$ inclusion

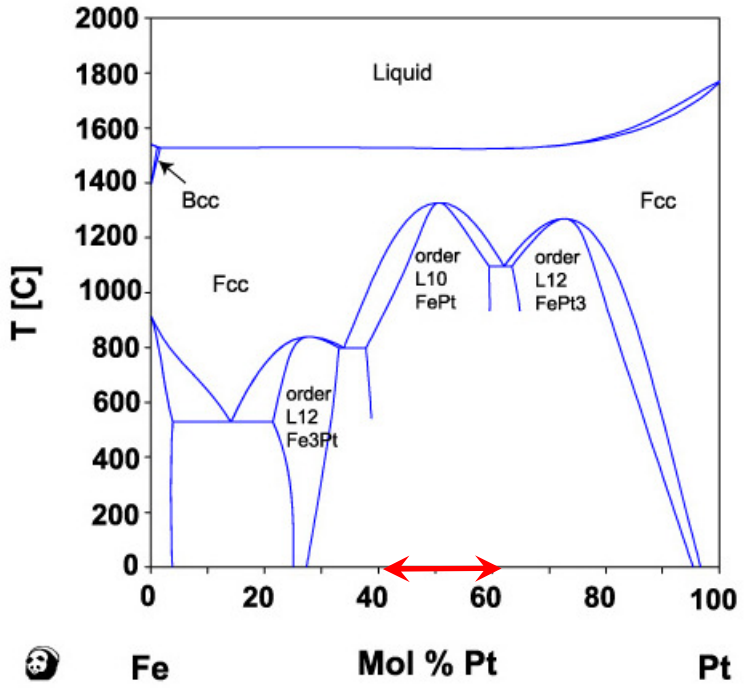


- electron energy filter with **a proper energy resolution** is necessary
- **time consuming simulations** are necessary

W. Hetaba et al., Micron 41 (2010) 479

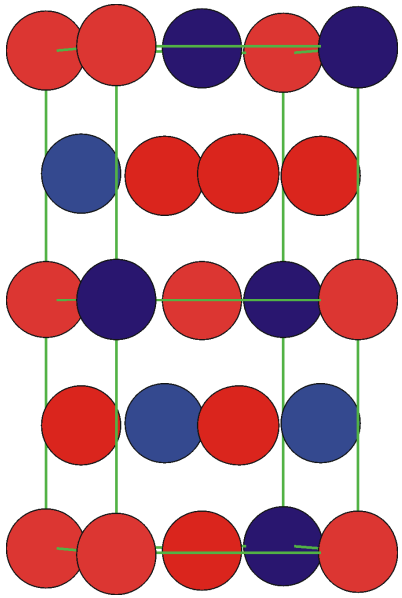
FePt crystallites on self-assembled SiO_2 nanospheres

Disorder-order transformation in FePt



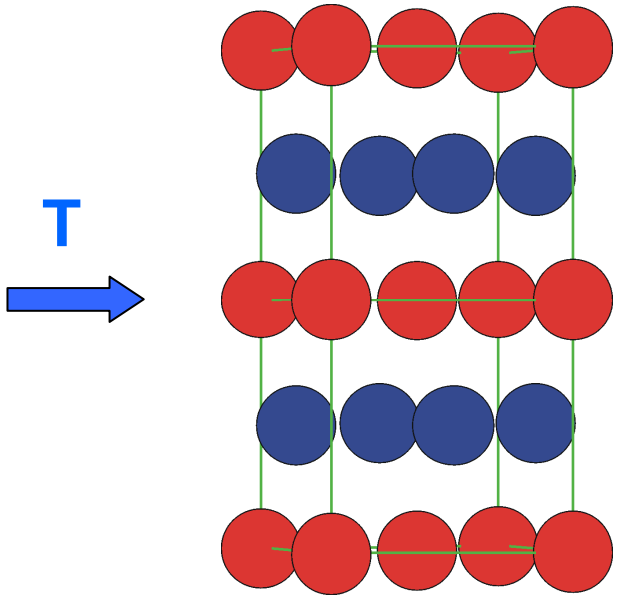
Calculated Fe-Pt phase diagram assessed by 2001 P.Fredriksson

disordered phase
FePt, A1 (fcc)



a = 0.380 nm

T



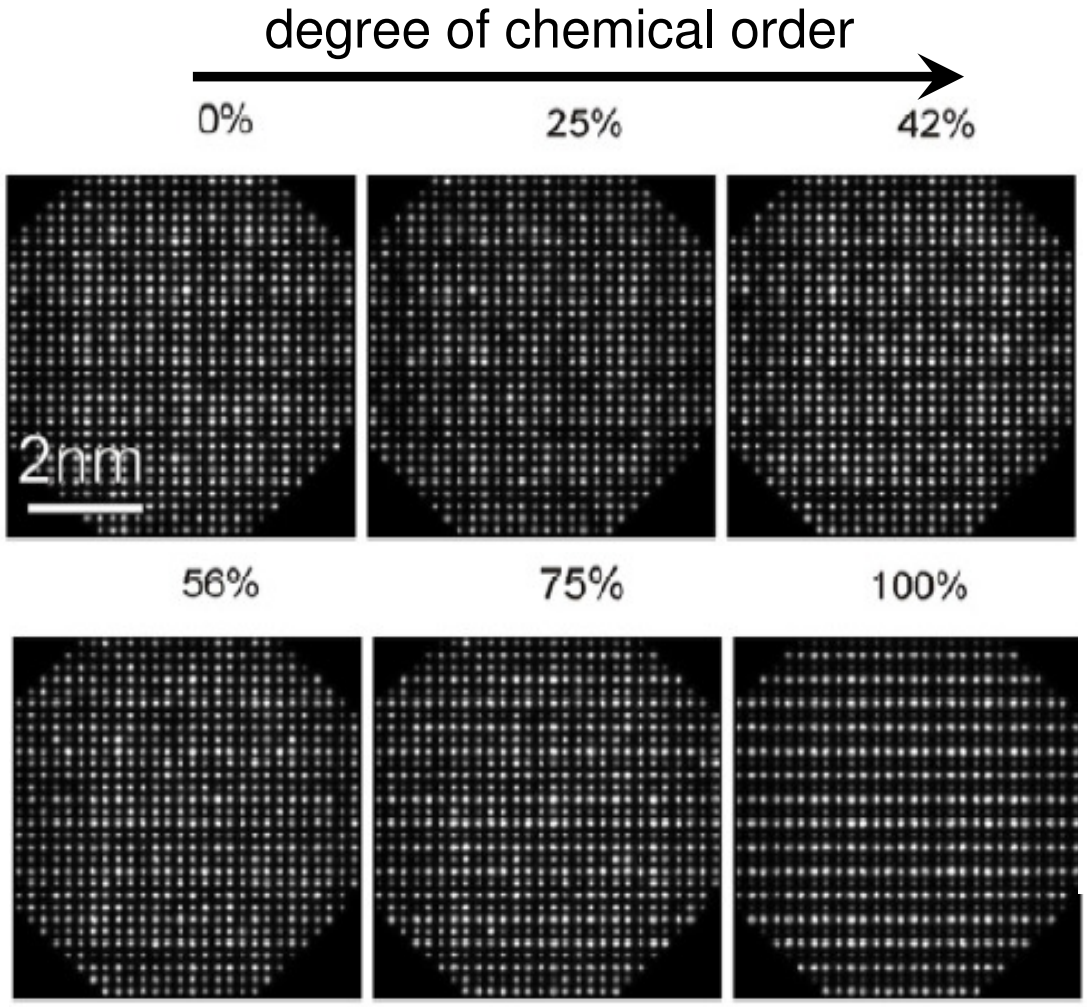
chemically ordered phase
FePt, $L1_0$ (fct)

$a \neq c$
a = 0.385 nm
c = 0.371 nm

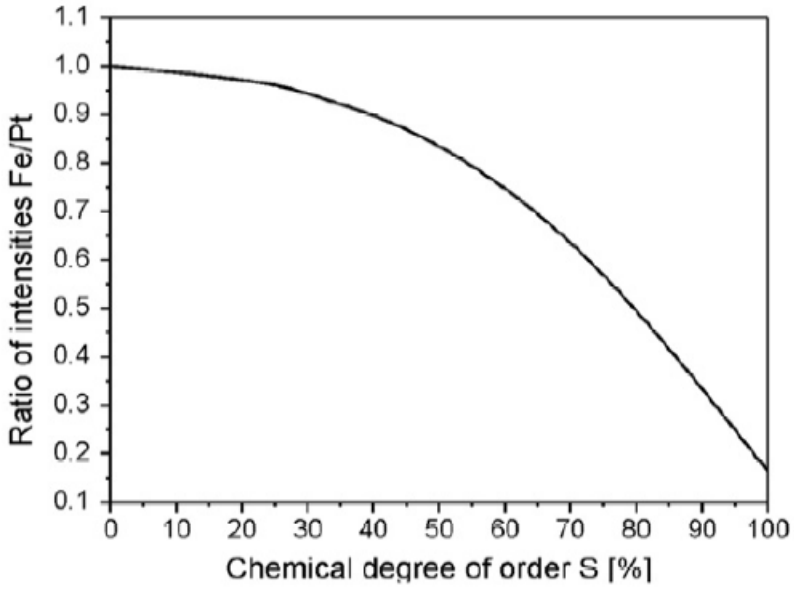
Chemically ordered $L1_0$ phase shows a high uniaxial magnetic anisotropy
→ promising candidate for high-density magnetic recording media

Phase determination in single crystalline FePt nanocrystals

HAADF STEM:



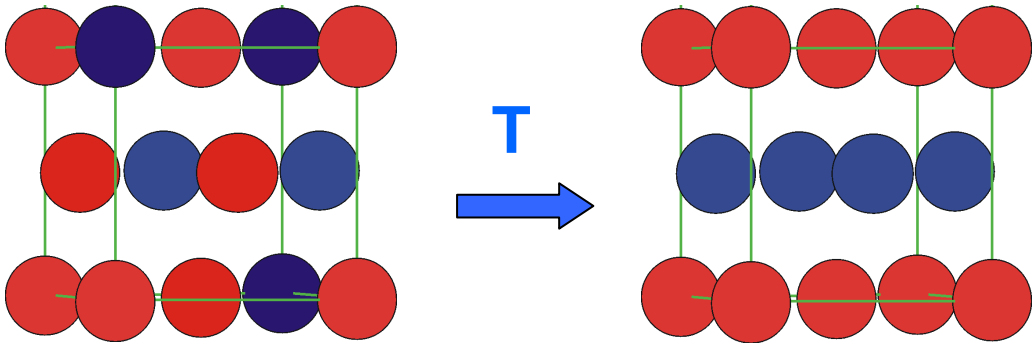
J. Biskupek et al.,
Ultramicroscopy 110 (2010) 820



Phase determination in single crystalline FePt nanocrystals

disordered phase
FePt, A1 (fcc)

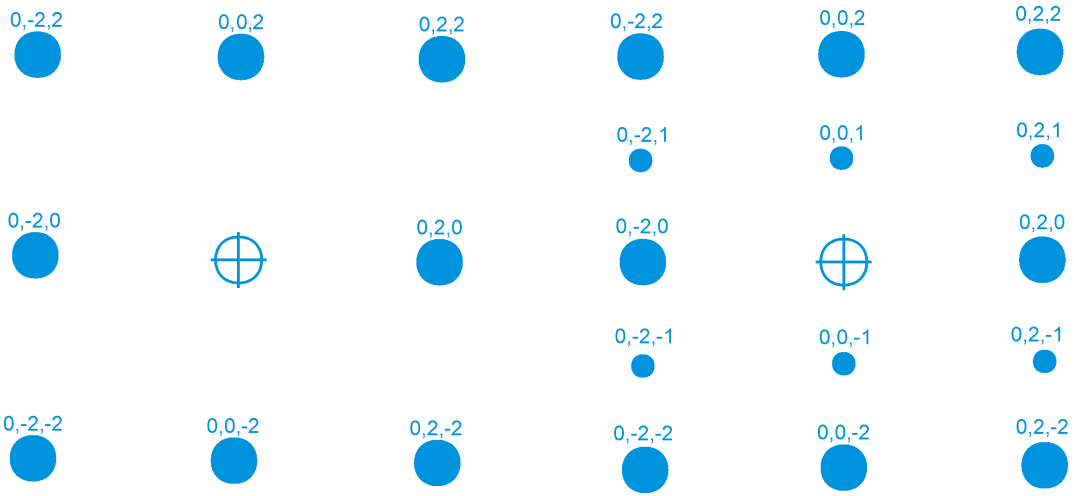
chemically ordered phase
FePt, $L1_0$ (fct)



Structure factor:

$$F_{hkl} = \sum_j f_j \cdot e^{2\pi i \cdot (hx_j + ky_j + lz_j)}$$

Electron diffraction: in [100] zone axis



$$I_{001} \propto F_{001}^2 = 0$$

- kinematically forbidden for a random phase.

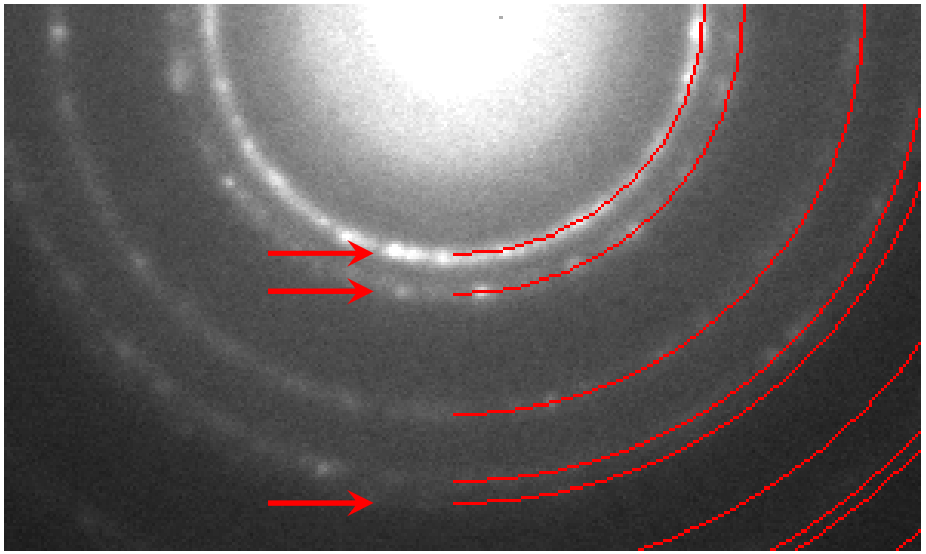
$$I_{001} \propto F_{001}^2 \neq 0$$

- allowed for a chemically ordered phase

Electron diffraction analysis of polycrystalline FePt layers on Si

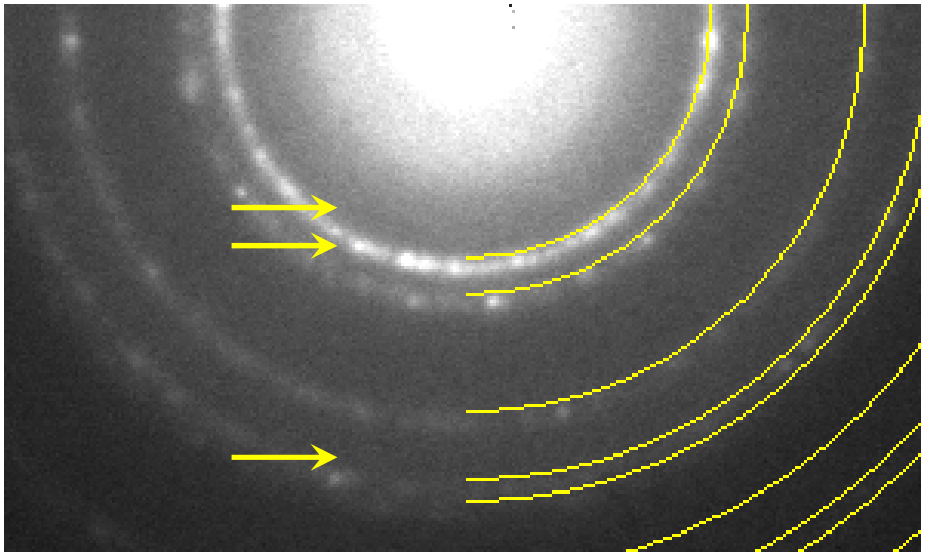
as-deposited FePt layers → chemically disordered (fcc) FePt

experimental pattern with FePt simulation



FePt:
a = 0.380 nm

experimental pattern with Pt simulation



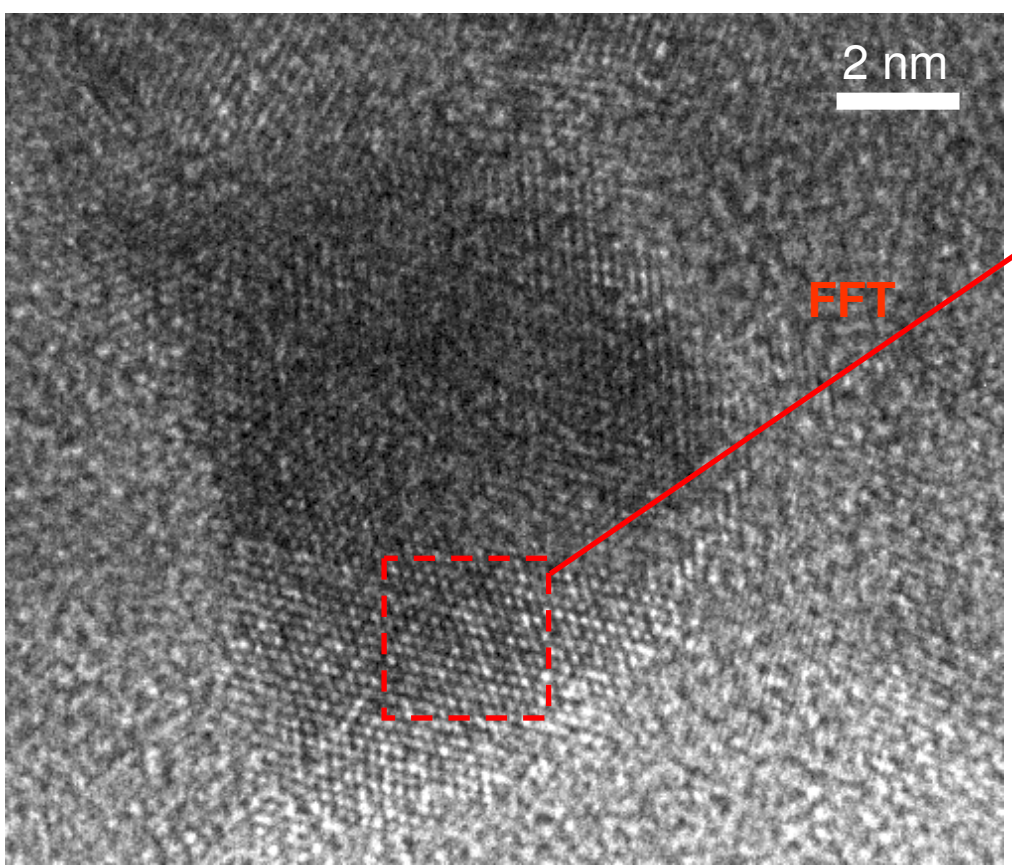
Pt:
a = 0.391 nm

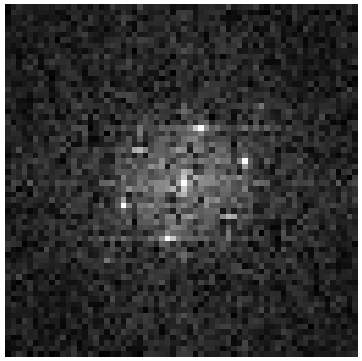
Result: no Pt!

CHEMNITZ UNIVERSITY
OF TECHNOLOGY
Group of Surface and Interface Physics

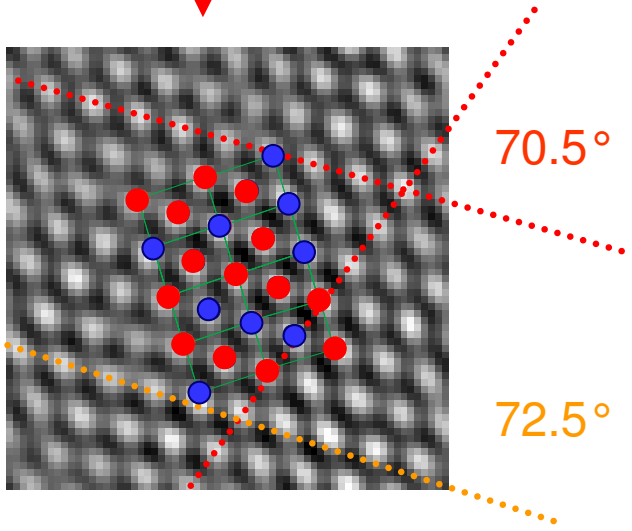
HRTEM analysis of polycrystalline FePt layers

as-deposited FePt layers → chemically disordered (fct) FePt





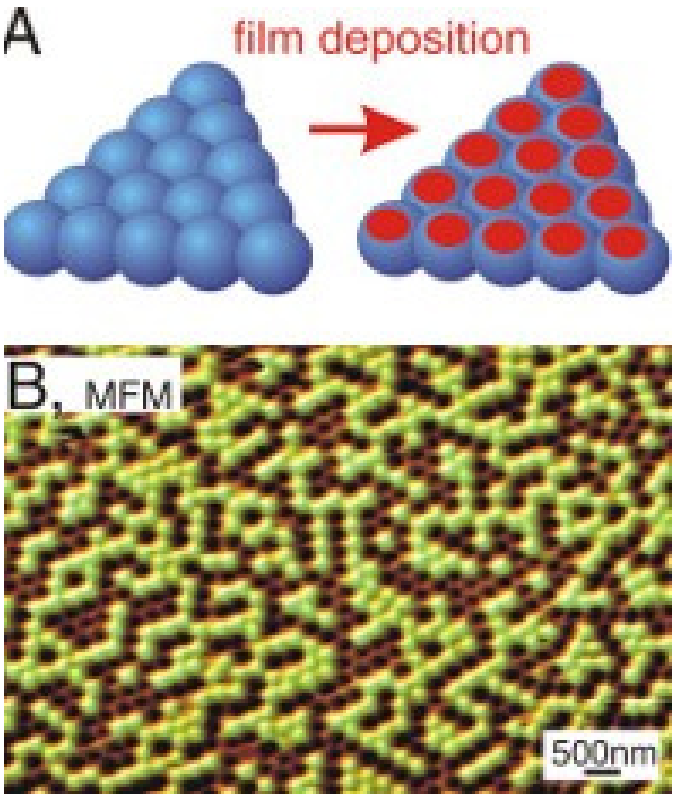
IFFT



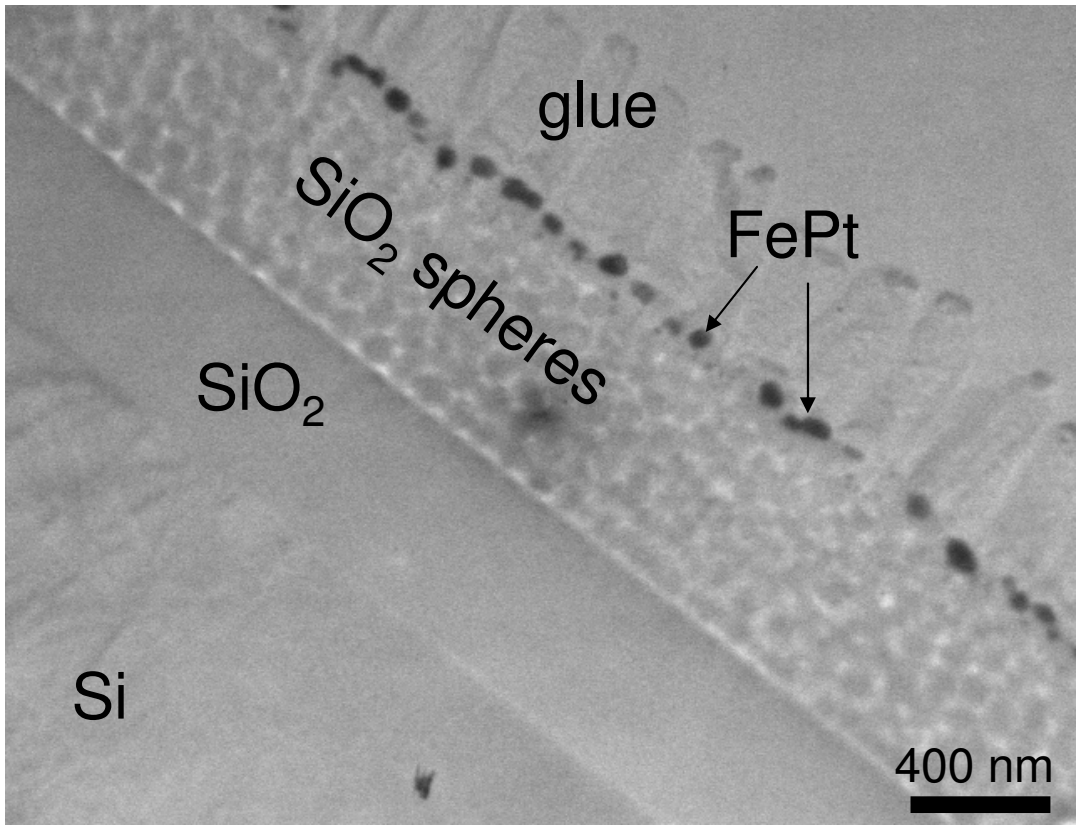
FePt fcc: angle beween (1-11) and (1-1-1) is 70.53°

FePt fct: angle beween (1-11) and (1-1-1) is 72.54°

FePt crystallites on self-assembled SiO_2 nanospheres



FePt on the 100 nm SiO_2 spheres
+ annealing

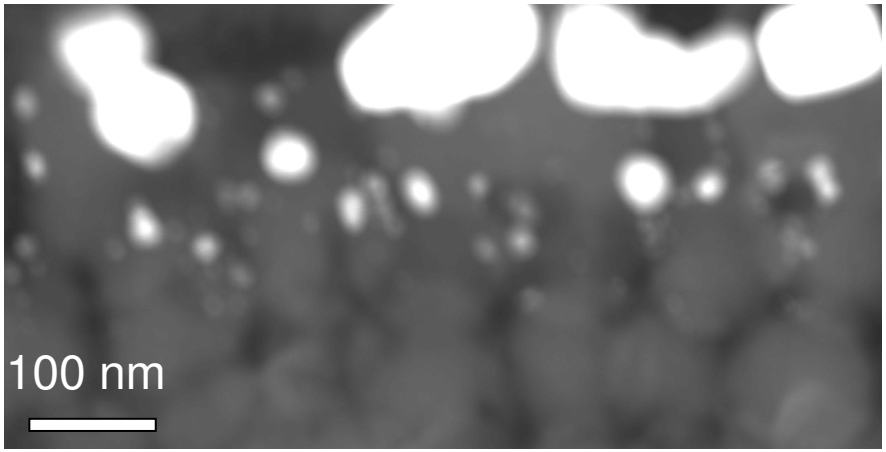


annealing should initiate the formation of chemically ordered fct phase!

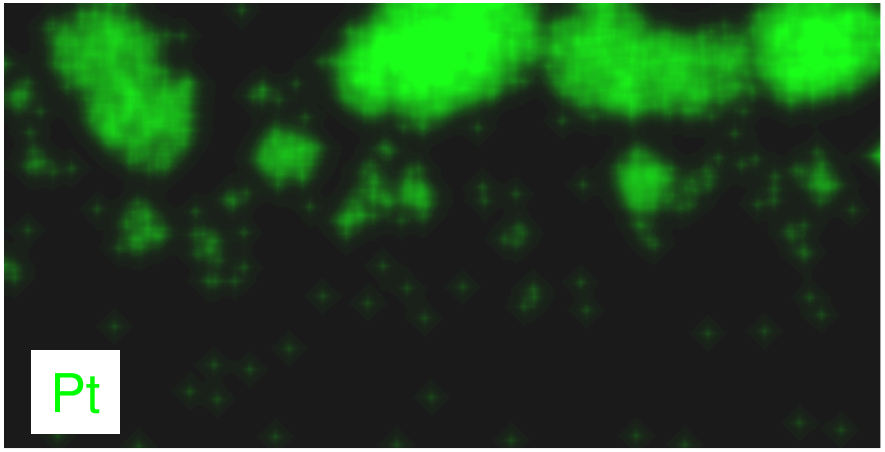
CHEMNITZ UNIVERSITY
OF TECHNOLOGY
Group of Surface and Interface Physics

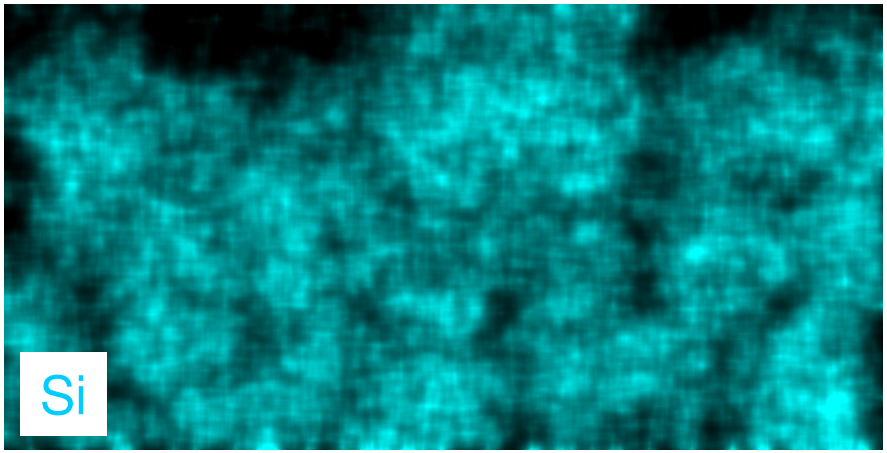
FePt crystallites on self-assembled SiO_2 nanospheres

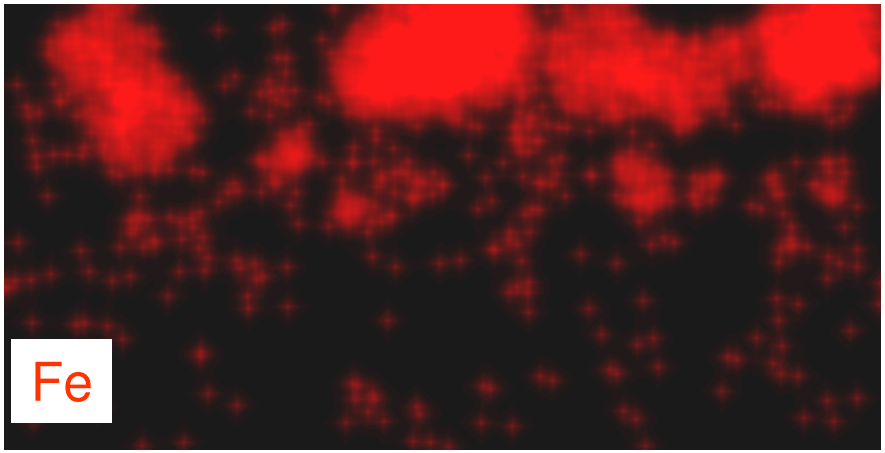
HAADF STEM:



EDXS mapping:



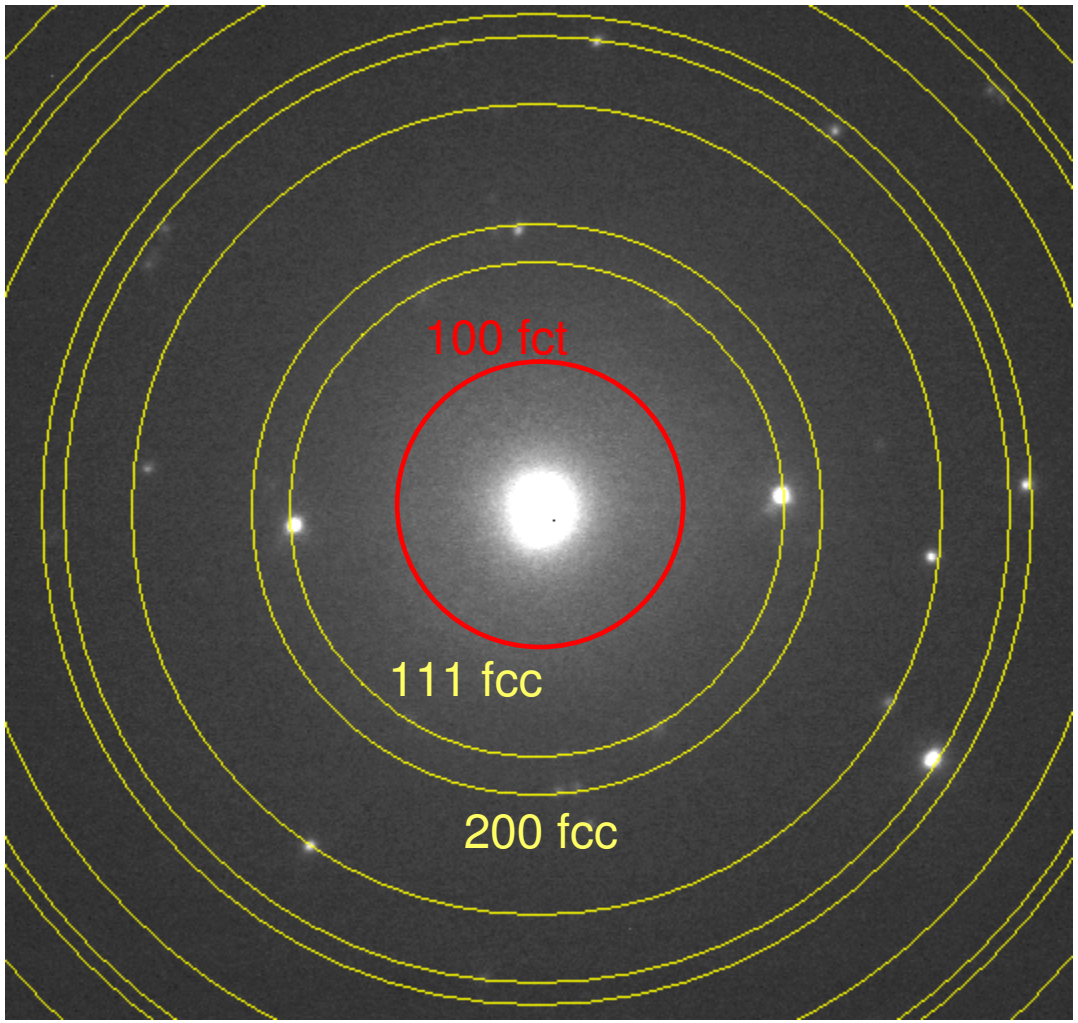




55 at. % Pt, 45 at .% Fe ± 5 at.%

Phase determination in FePt nanocrystals on self-assembled SiO_2 nanospheres

Electron diffraction:



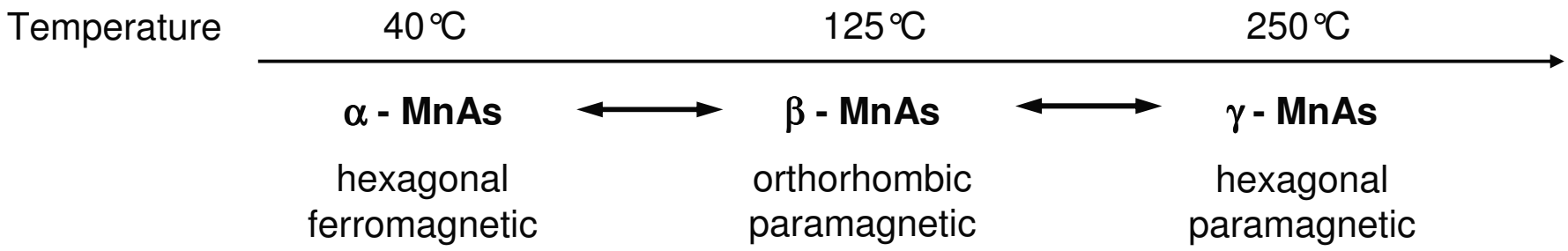
Problem:
low number of diffraction reflections

Possible solution:
precession electron diffraction

Crystallite phase and orientation mapping of MnAs in GaAs

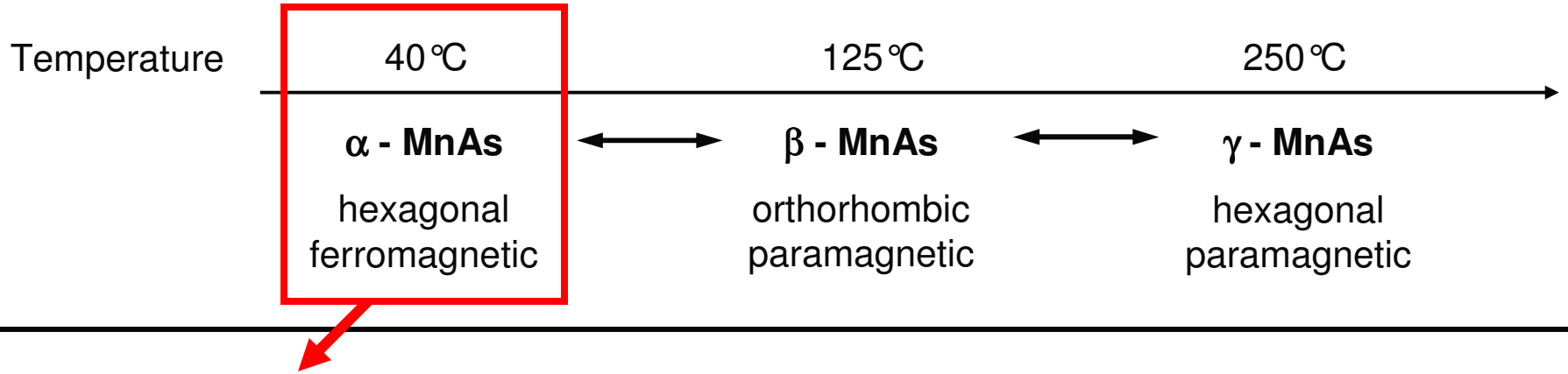
Material system: MnAs/GaAs
Motivation

Phase transformation of MnAs:



Material system: MnAs/GaAs
Motivation

Phase transformation of MnAs:



Spintronic devices :

- Exploitation of the intrinsic spin of the electron and its associated magnetic moment, in addition to its fundamental electronic charge

Advantages over conventional electronic devices:

- Faster and more efficient devices
- Processing and handling of an higher information density
- Low heat development

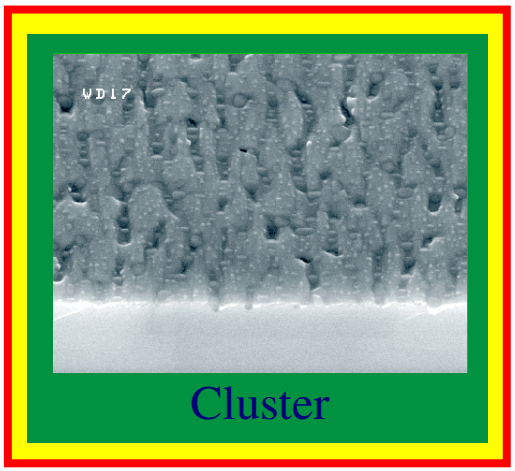
Philipps Universität Marburg

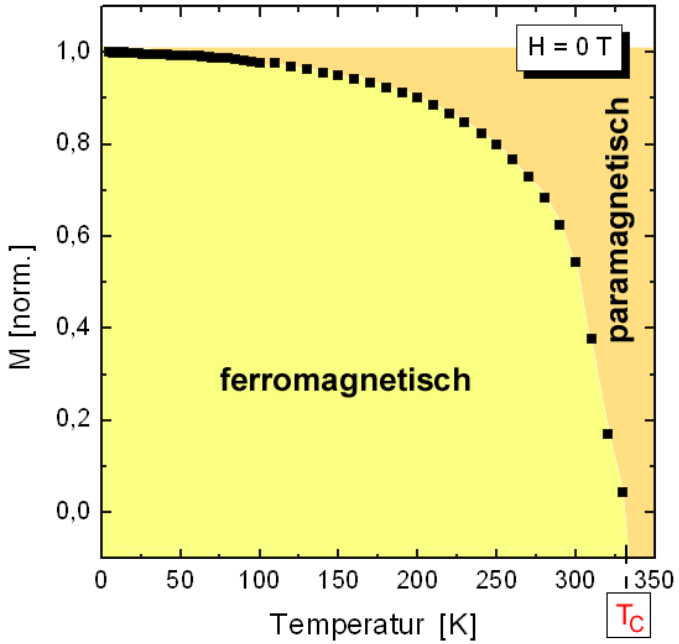
f MnAs/GaAs

Structure	Growth technique	Properties
1-dim: MnAs/GaAs Nanowires	MOCVD	non magnetic
2-dim: MnAs/GaAs Layers	MBE	α-MnAs (ferromagnetic)
3-dim: MnAs/GaAs crystallites	MOCVD	α-MnAs (ferromagnetic)

Growth of MnAs/GaAs

Structure	Growth technique	Properties
1-dim: MnAs/GaAs nanowires	MOCVD	non magnetic
2-dim: MnAs/GaAs layers	MBE	α-MnAs (ferromagnetic)
3-dim: MnAs/GaAs crystallites	MOCVD	α-MnAs (ferromagnetic)



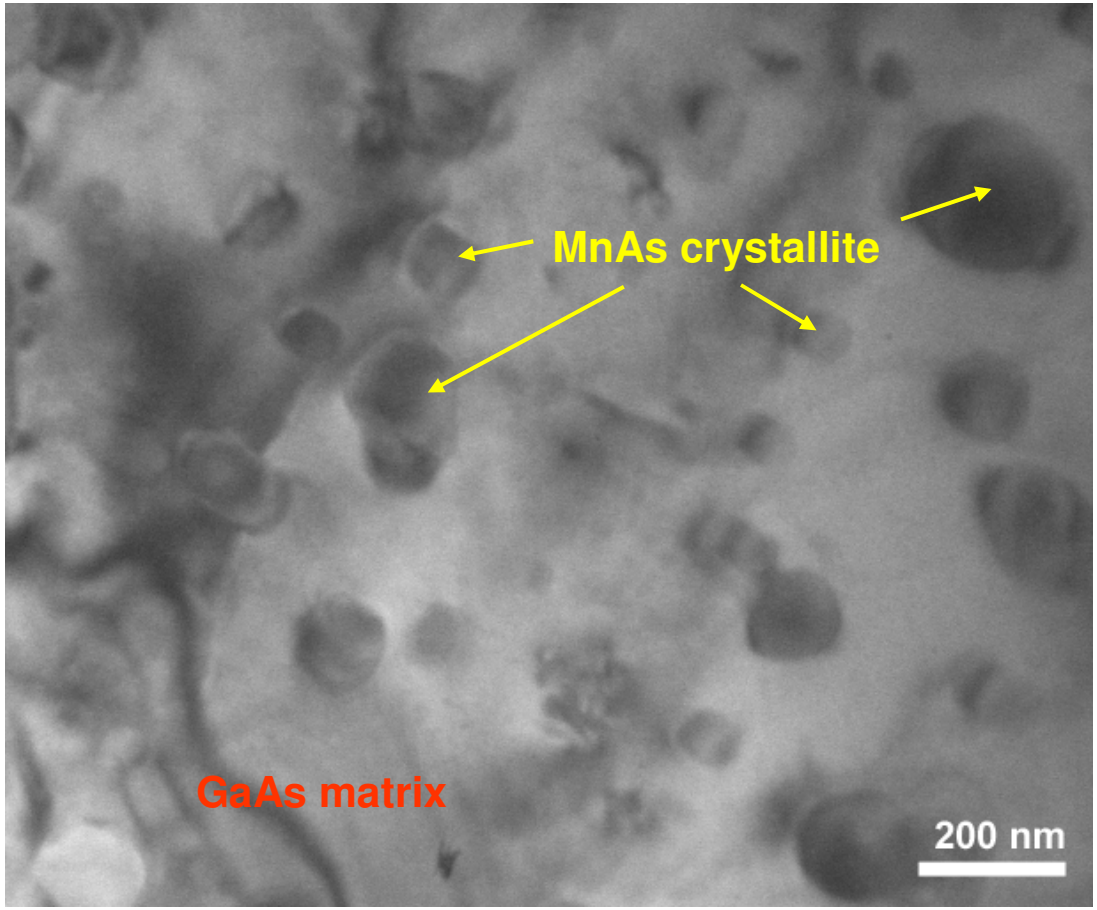


Curie-Temperatur

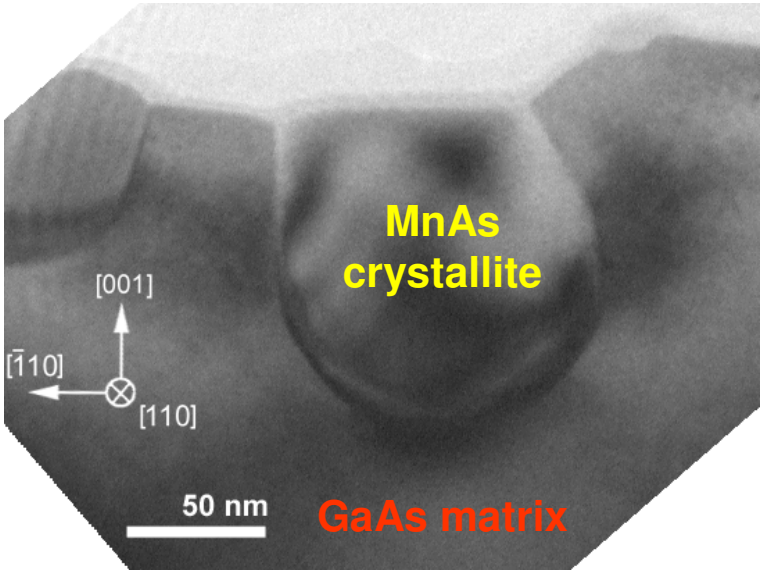
T_C > 330 K

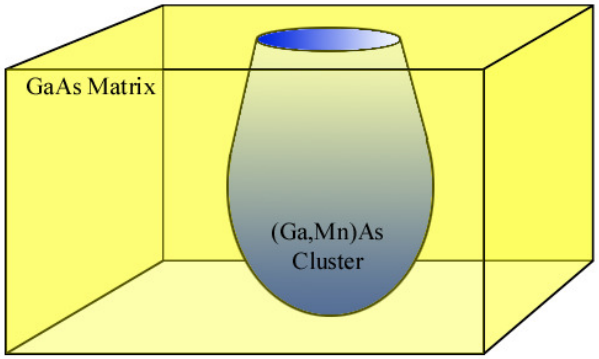
Material system: MnAs-crystallite / [001] GaAs

plan view bright field TEM image

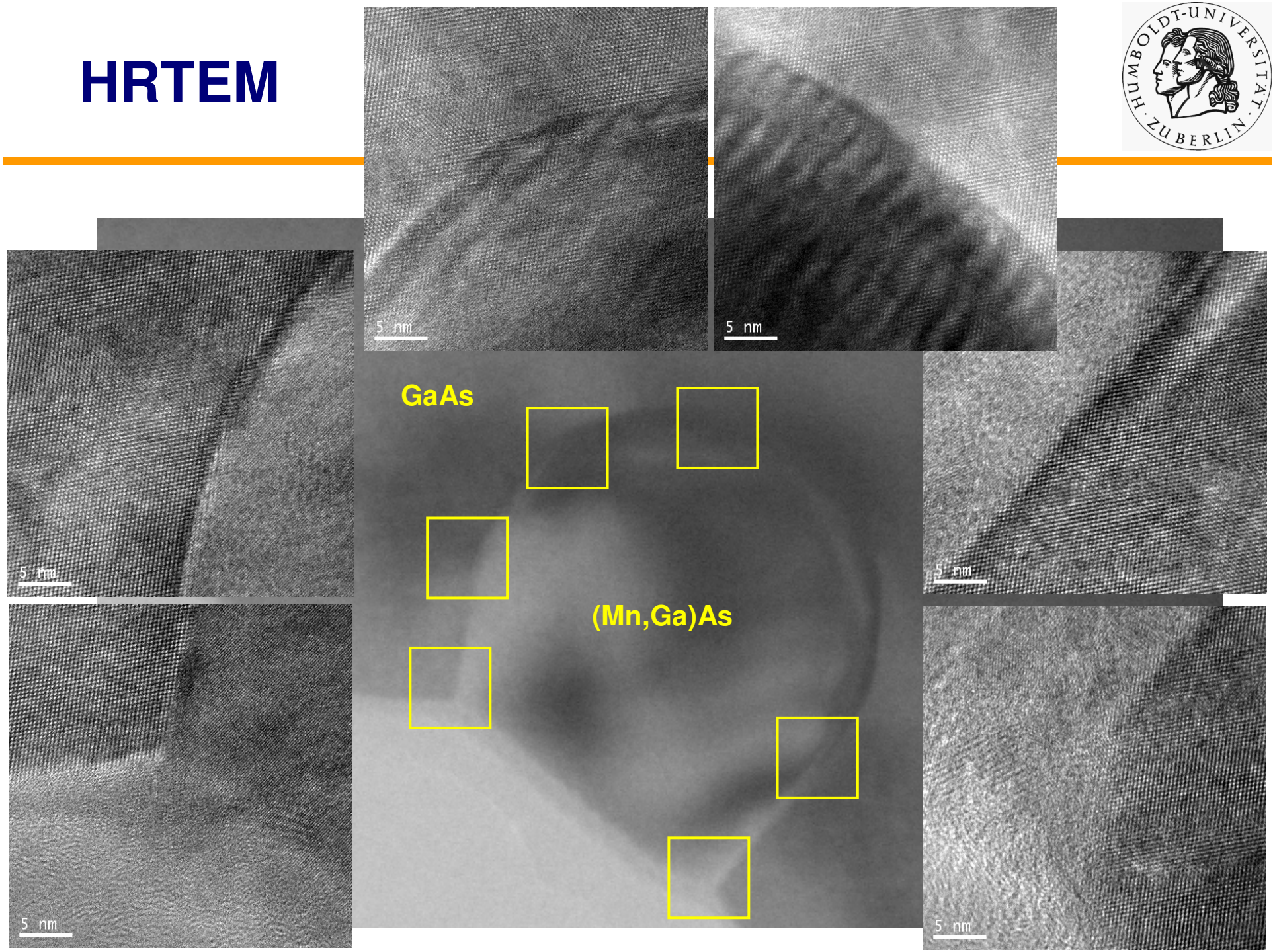


cross section bright field TEM image

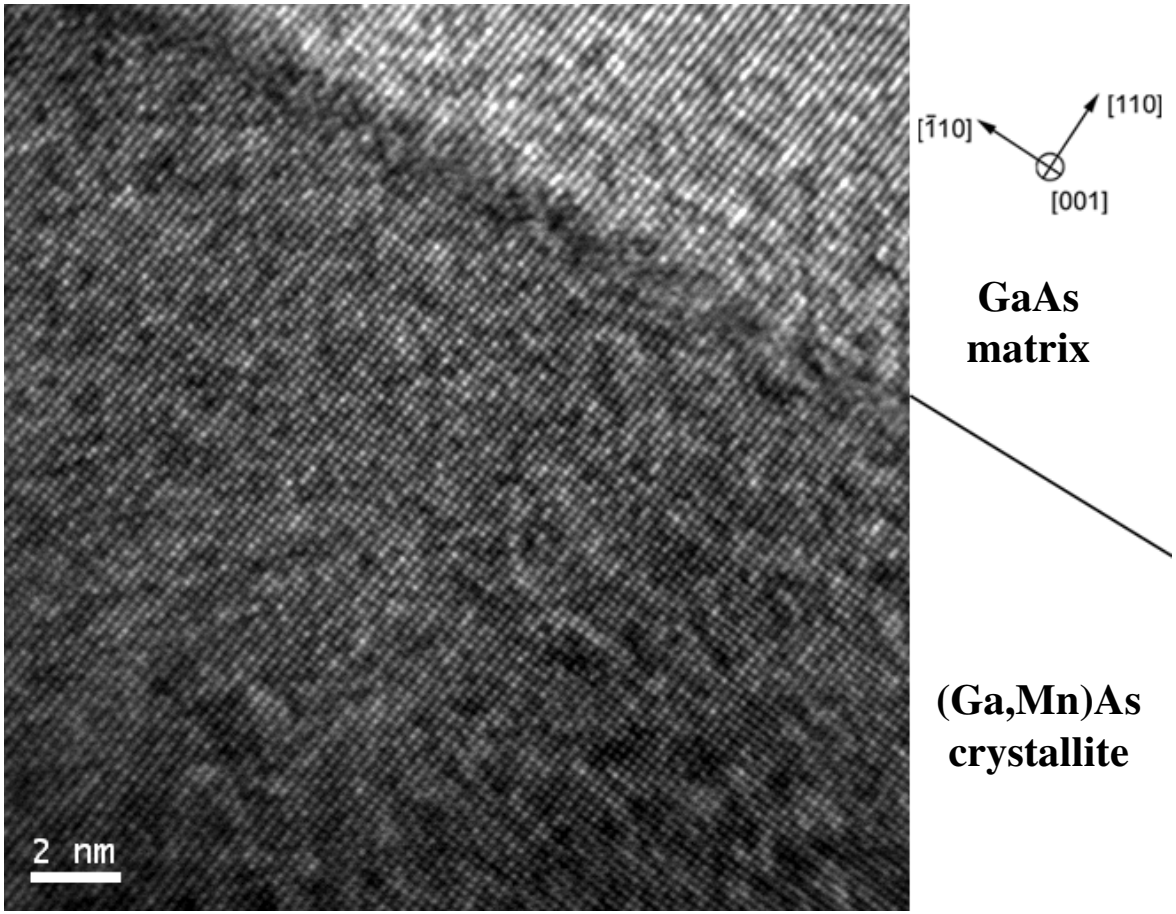




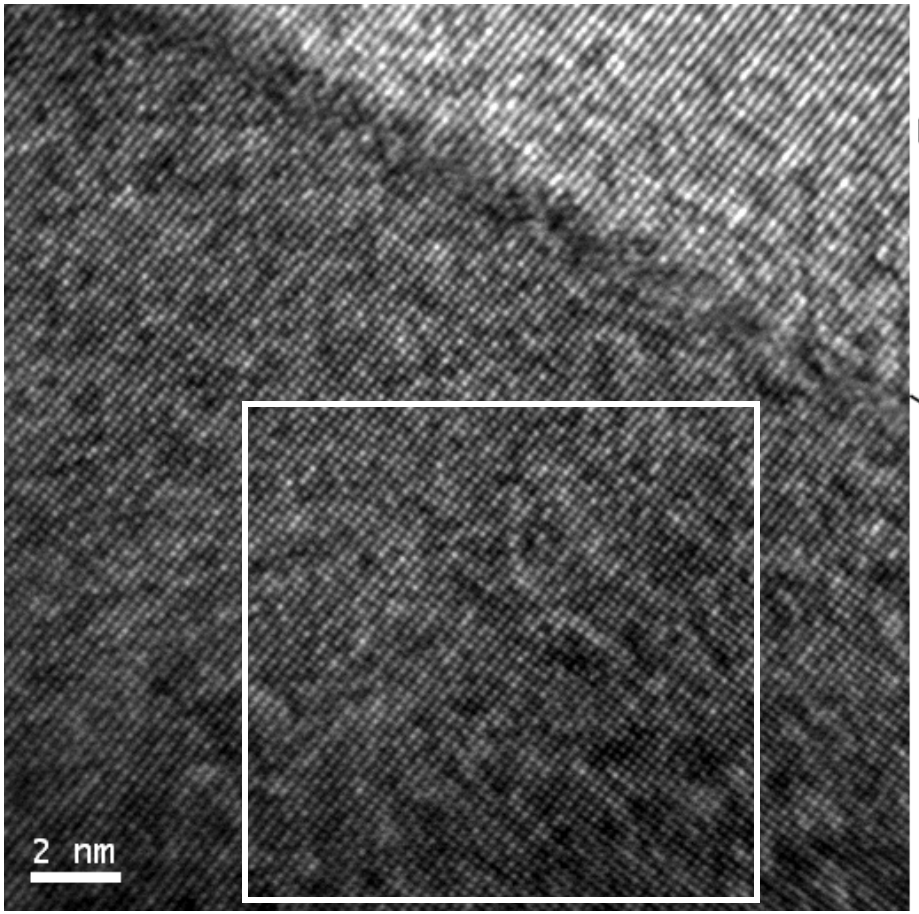
HRTEM

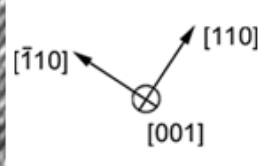


Material system: MnAs-crystallite / [001] GaAs



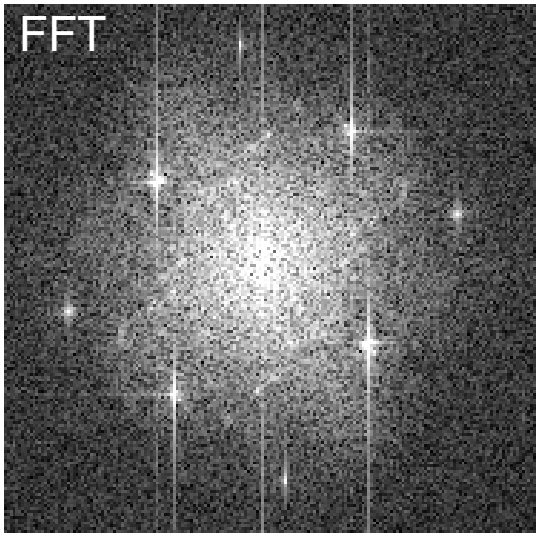
Material system: MnAs-crystallite / [001] GaAs





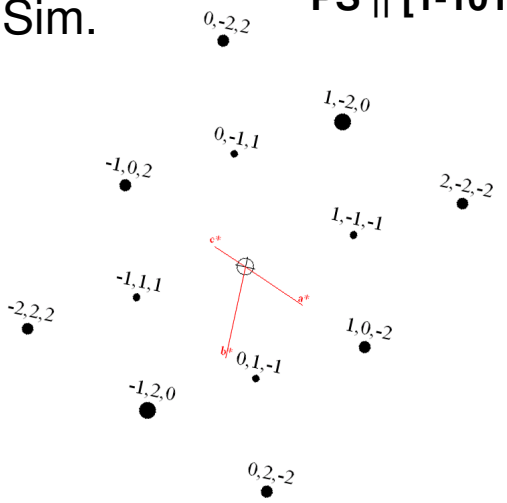
GaAs matrix

(Ga,Mn)As crystallite



PS || [1-101]

Sim.

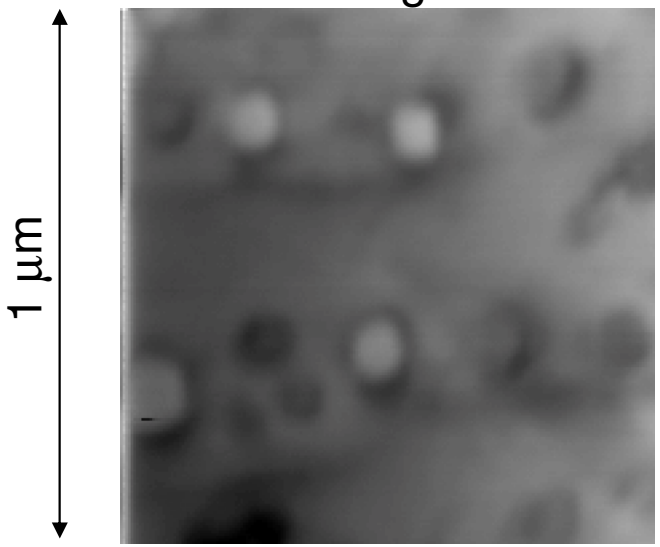


Material system: MnAs-crystallite / [001] GaAs
Phase map

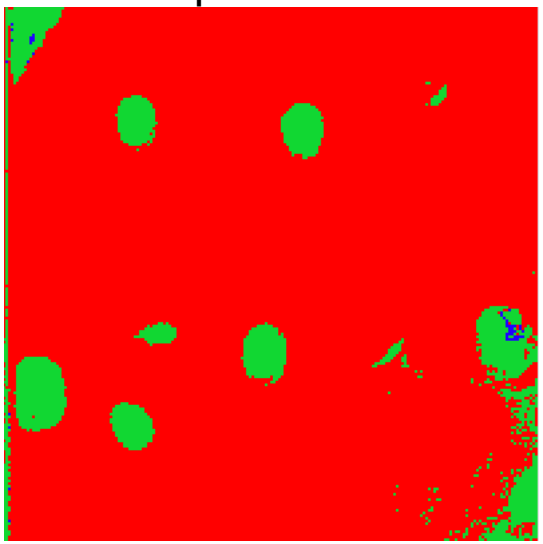
Y: Full precession

Nano beam diffraction
Spot size: 2.4 nm

virtual bright field

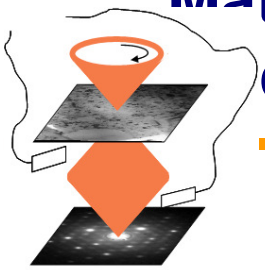


phases

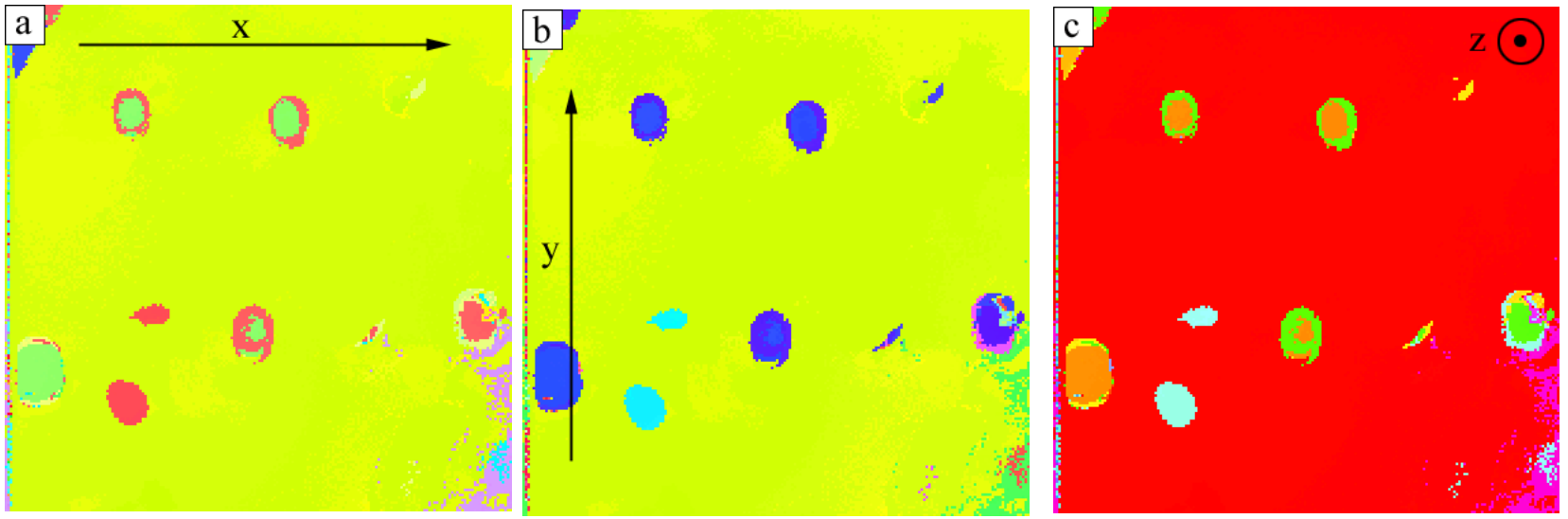


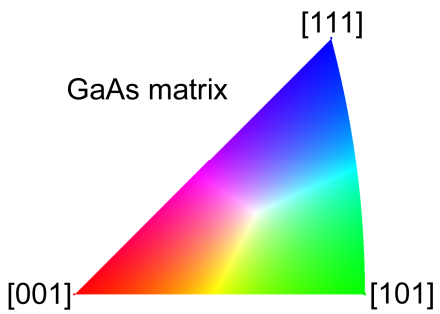
GaAs cubic
MnAs orthorhombic
MnAs hexagonal

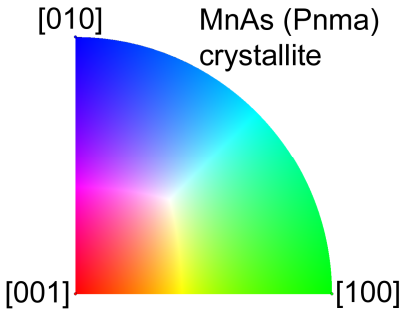
β-MnAs orthorhombic phase
(paramagnetic)



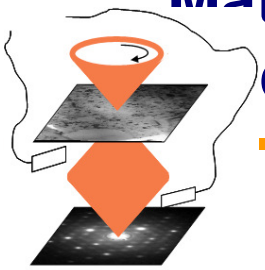
Material system: MnAs-crystallite / [001] GaAs
entation maps

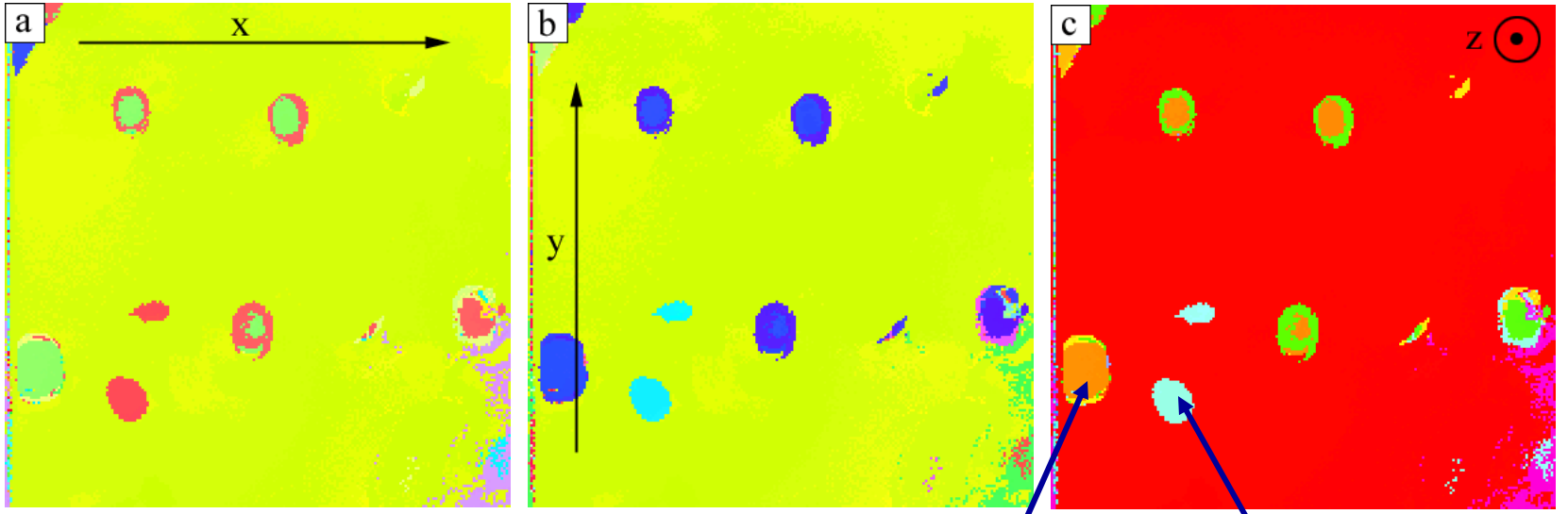




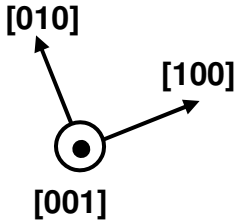


Material system: MnAs-crystallite / [001] GaAs
entation maps

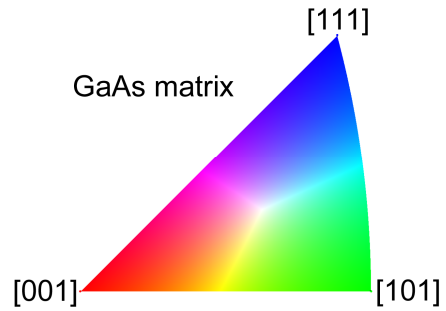


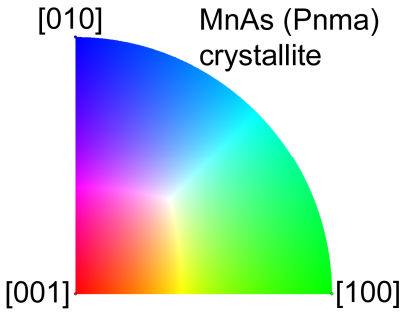


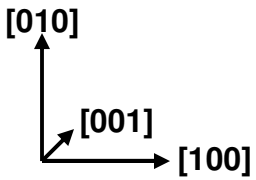
GaAs

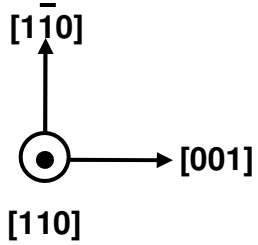


MnAs (orthorhombic)





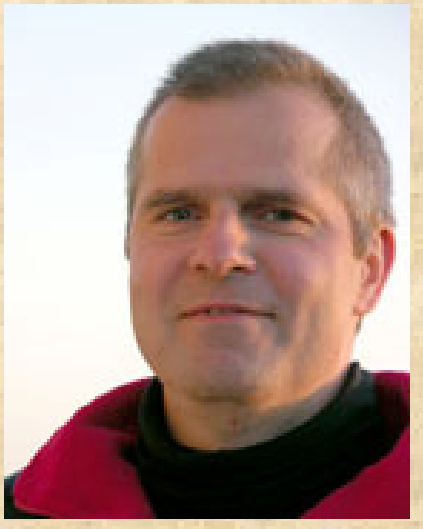




Scientific contributions of

Anna Mogilatenko

Holm Kirmse



Ines Häusler

Thank you for your attention