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The University of Resources.
Since 1765.

TU Bergakademie Freiberg – Research Highlights with Synchrotron Radiation

Workshop: Saxony-DESY Cooperation Center
23rd November – 24th November

Technische Universität Bergakademie Freiberg, 2023

TU Bergakademie Freiberg (TUBAF)

TUBAF
Die Ressourcenuniversität.
Seit 1765.

- Founded 1765
- Six faculties: Mathematics and Computer Science, Chemistry and Physics, Geosciences, Geotechnics and Mining, Mechanical, Process and Energy Engineering, Materials Science and Technology, Business Administration
- Research and study profile:
    - Climate & Environment
    - Technologies
    - Raw Materials, Materials & Substances
    - Energy Systems
    - AI & Robotics
    - Economics & Law

THINK GLOBALLY, ACT GLOBALLY

Study and research across borders to find solutions for the challenges of our time.

Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

Zentrum für effiziente Hochtemperatur-Stoffwandlung (ZeHS) - Center for efficient high-temperature material conversion

- Central scientific facility of the TU Bergakademie Freiberg
- 40 participating professorships
- Research profile:
  - → Processes optimization at temperatures above 500 °C
  - → Developing of innovative, resource- and energy-efficient technologies and materials

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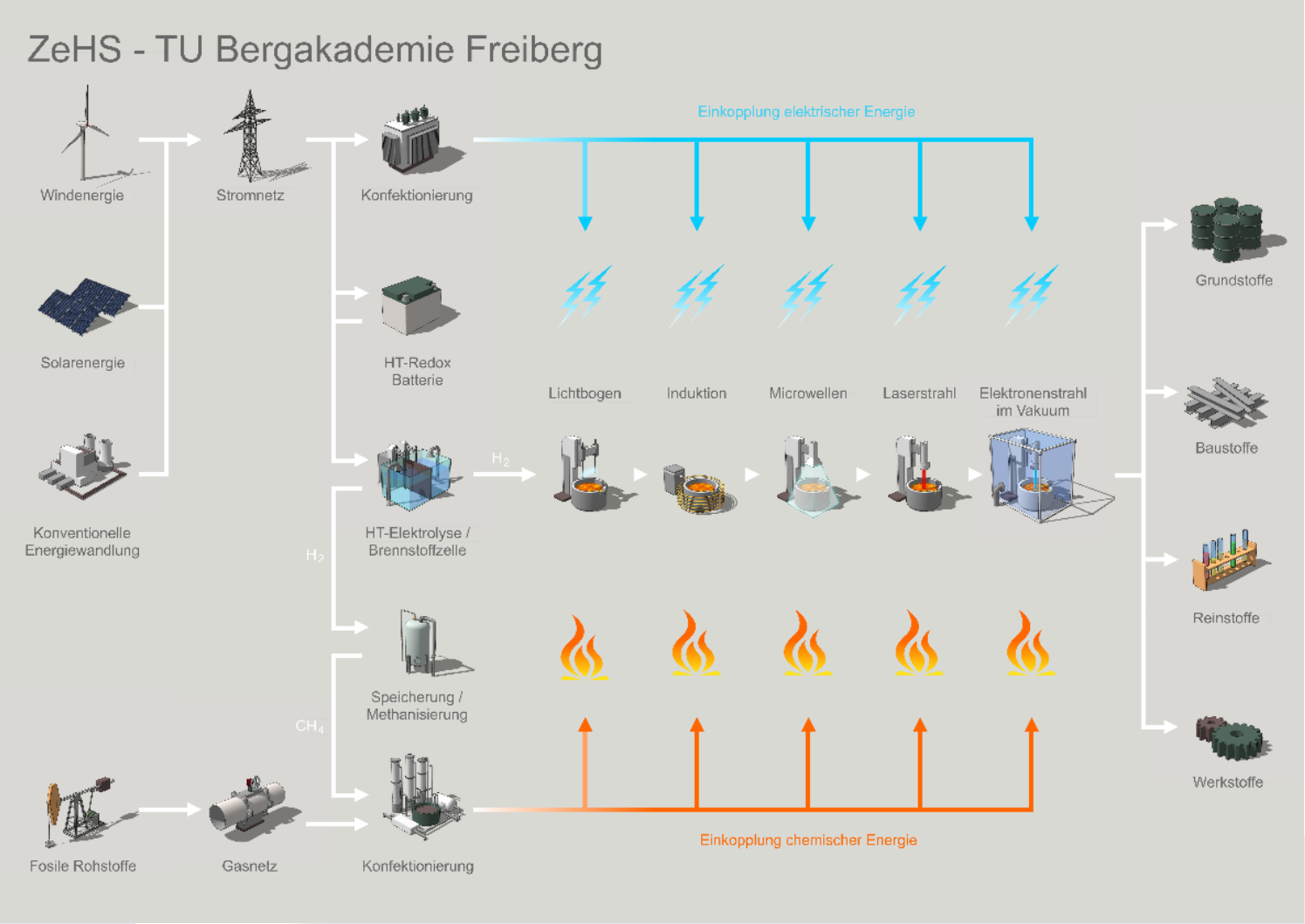
Zentrum für effiziente Hochtemperatur-Stoffwandlung (ZeHS) - Center for efficient high-temperature material conversion



- Processes optimization at temperatures above 500 °C
- Developing of innovative, resource- and energy-efficient technologies and materials
- New technologies for electrification and decarbonization of high-temperature processes in connection with material requirements

Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

TUBAF – Research Fields with Synchrotron Radiation

Finding new materials

- Electronic and battery materials, materials for photovoltaics and optoelectronics: IAP, IEP (Fac. 2), IWW, INEMET (Fac. 5)
- Shape memory alloys: IWW, IWT (Fac. 5)
- High-entropy alloys: IWW (Fac. 5) together with IFW Dresden
- Hard materials/materials for extreme conditions, high-pressure/high-temperature synthesis of novel materials: Freiberg Centre for High Pressure Research, IAC (Fac. 2), IOM (Fac.3), IWW (Fac. 5)
- Nanomaterials and polymers: ESM (Fac. 4), IPC (Fac. 2)

Process optimization

- High-temperature processes: ZeHS, Fac. 4, Fac. 5
- Physical layer deposition of functional materials: ZeHS, IEP (Fac. 2)
- Holistic materials design including development of materials technologies: Fac. 5
- Additive manufacturing: Fac. 4 and 5

Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

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Synchrotron Radiation for Materials Science and Engineering

Institute of Materials Science
Prof. Dr. David Rafaja (rafaja@iww.tu-freiberg.de)

2D XRD + deformation experiment

TRIP/TWIP steels
High entropy alloys (IFW DD)
Shape memory alloys

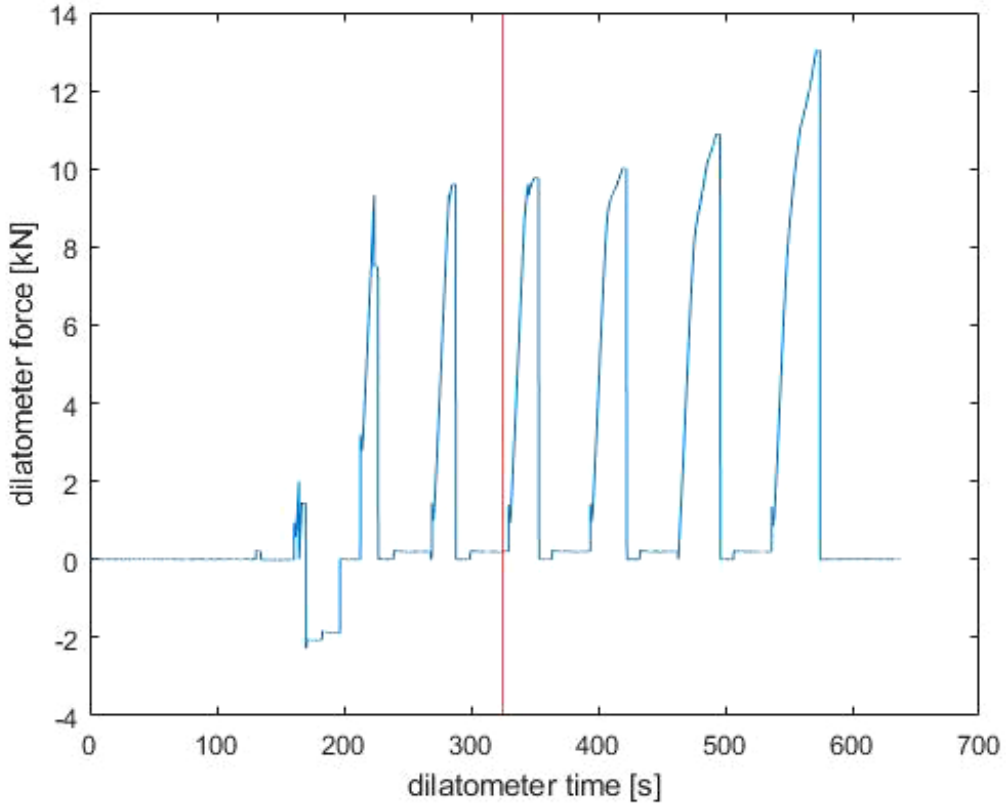
to come: Additive Manufactured samples

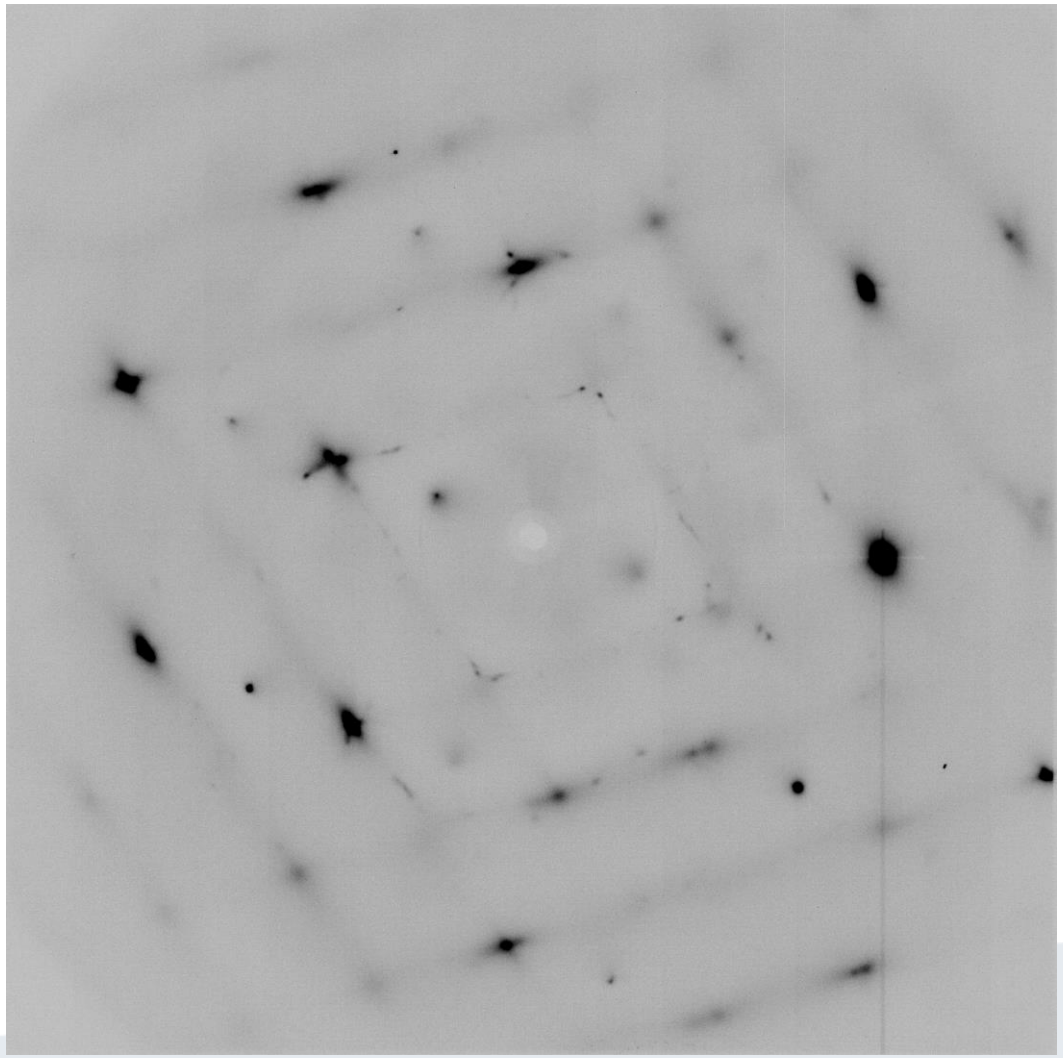
Petra III / HEMS P07

- E = 100 keV (l = 0.01235 nm)
- sample thickness: up to 5 mm (steel)
- Transmission geometry
- 2D detector (Perkin Elmer, 2048 x 2048 pixel)
- Compression/tension
- high temperature

Prof. David Rafaja (rafaja@iww.tu-freiberg.de), TU Bergakademie Freiberg

2D XRD + deformation experiment - Fe-34.5Mn-14.5Al-6Ni (deformation at RT)





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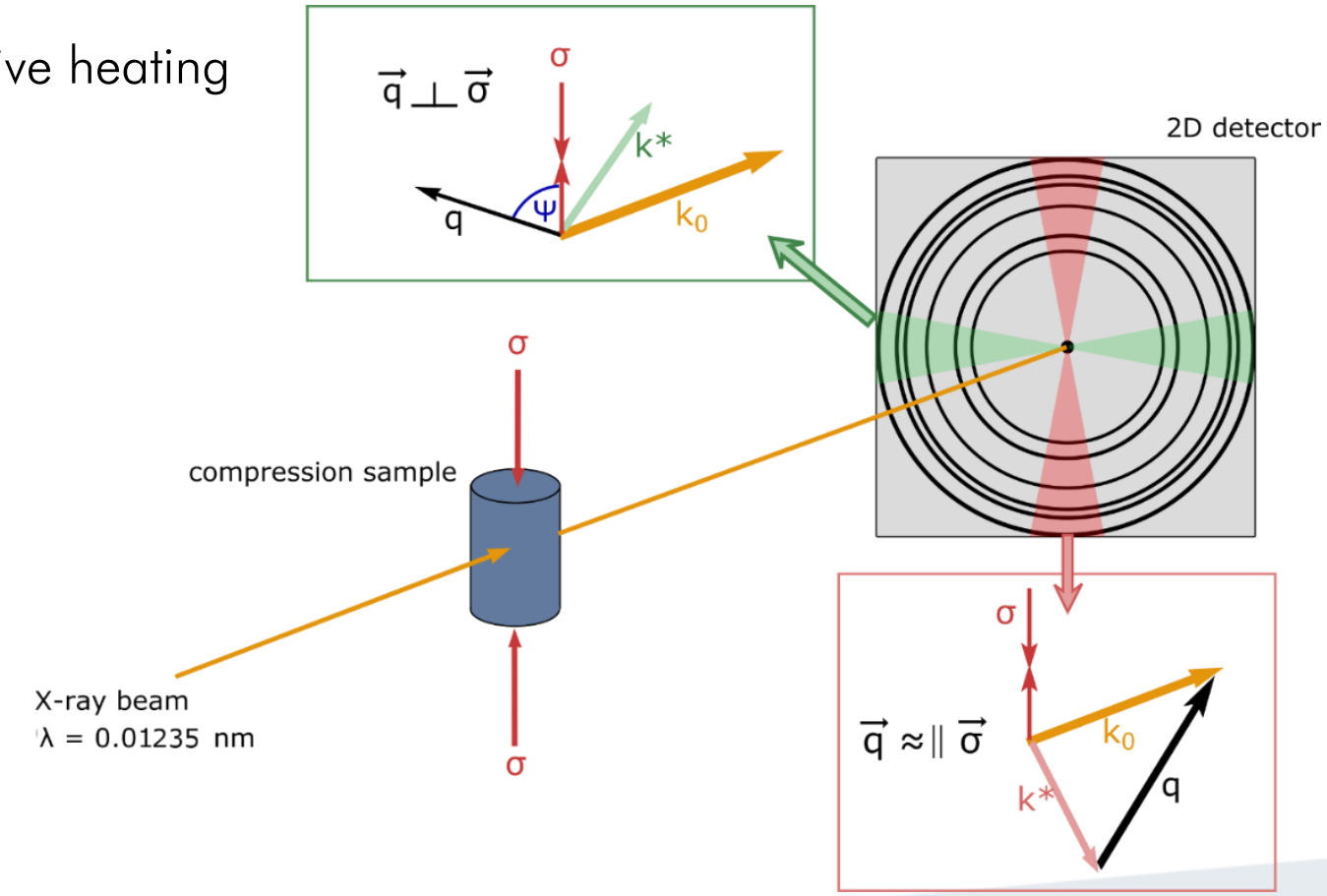
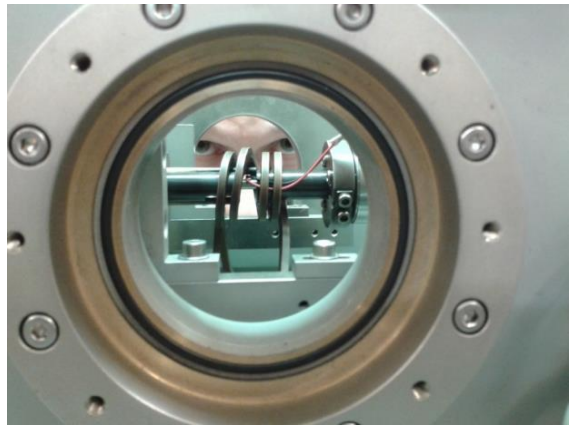
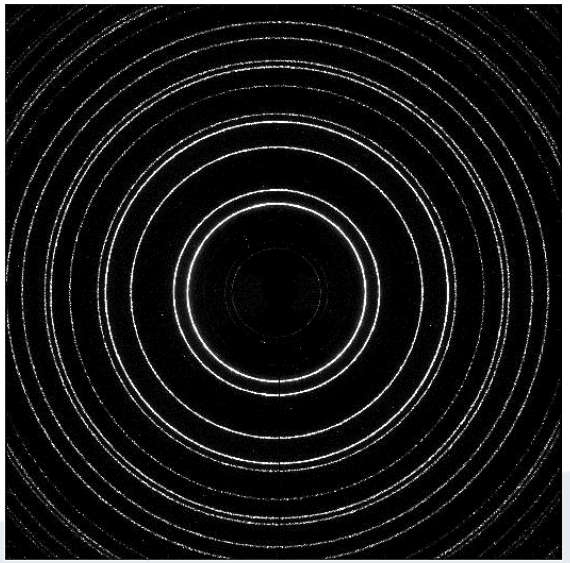
In situ Synchrotron Diffraction Experiments @ Petra III / HEMS P07

- *In situ*- compression deformation dilatometer, compression mode, F_{max} = 20 kN
- Inductive heating







Prof. David Rafaja (rafaja@iww.tu-freiberg.de), TU Bergakademie Freiberg

D. Rafaja, *et al.*:, Springer Series Mater. Sci. 298 (2020) 325-377.

C. Ullrich, S. Martin, C. Schimpf, A. Stark, N. Schell, D. Rafaja, Adv. Eng. Mater. 21 (2019) 1801101.

C. Ullrich, S. Martin, C. Schimpf, H.-G. Brokmeier, N. Schell, A. Stark, D. Rafaja, Mater. Char. 176 (2021) 111132.

2D XRD + deformation experiment

Line positions and intensities

- fast acquisition of phase composition -> mapping of changes in phase fractions upon deformation
- analysis of preferred orientations of crystallites

Dependence of the line positions on the macroscopic and crystallographic directions

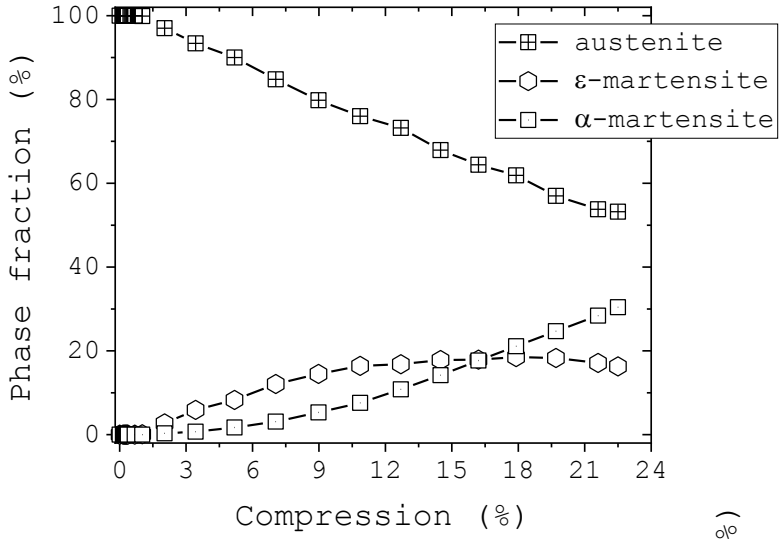
- Intrinsic (stress-free) lattice parameters
- Stacking fault probability
- Residual stresses

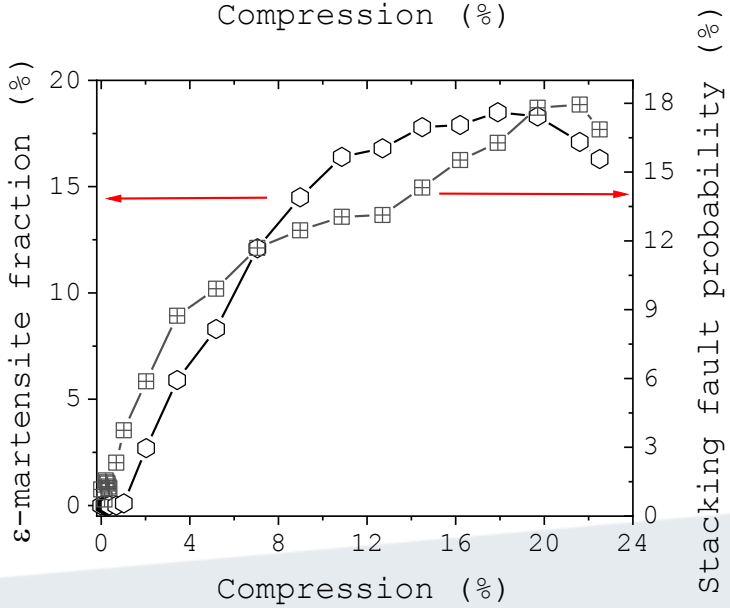
Dependence of the line broadening on the macroscopic and crystallographic directions

- Kind/density of microstructure defects (dislocations, stacking faults)
- Dependence of the defect densities on macroscopic direction

Outcome

- Deformation mechanisms as function of steel composition, applied load, temperature, …
- Tailoring of mechanical properties via thermomechanical treatment





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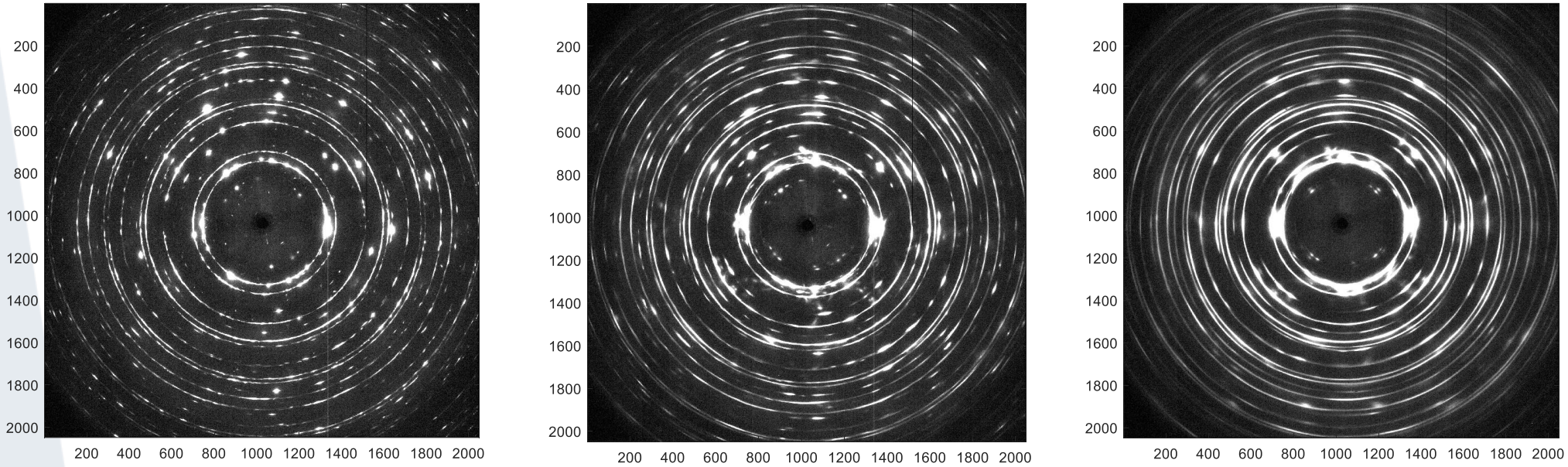
Examples of Further Materials - Fe-33Mn-17.5Al-8Ni

Deformation



Benefits → fast acquisition of:

- phase composition
- residual stress
- crystallographic anisotropy/microstructure defects
- preferred orientation

IN A SINGLE TIME RESOLVED DATASET

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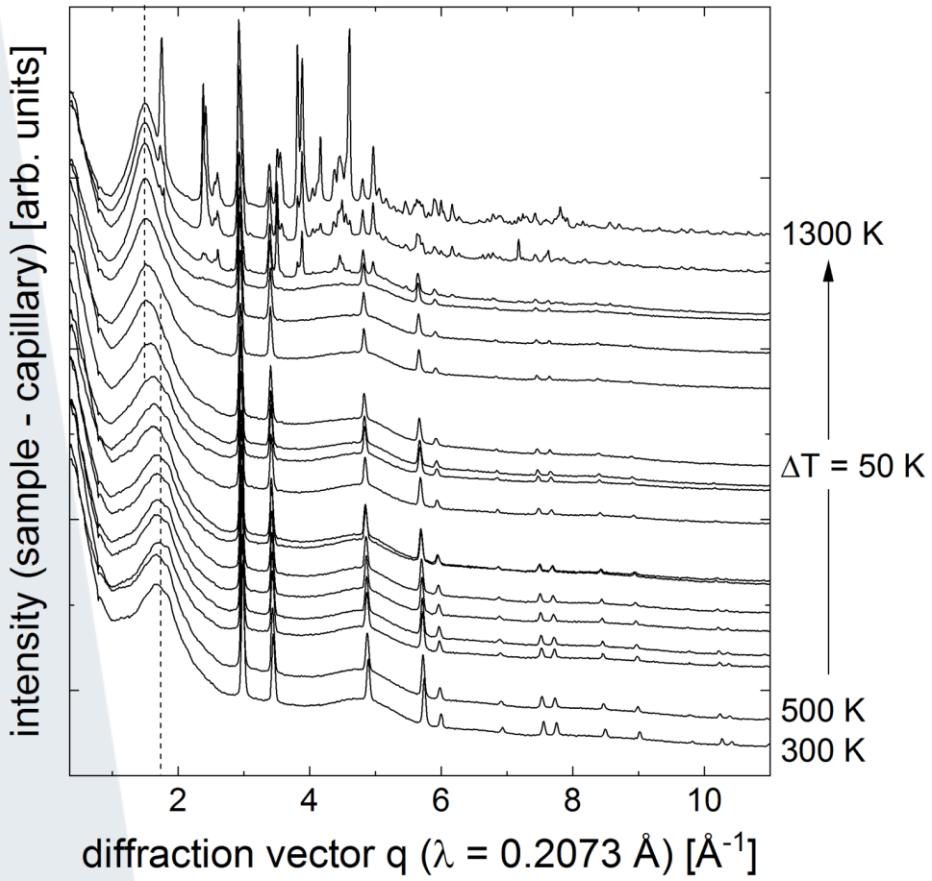
Total Scattering

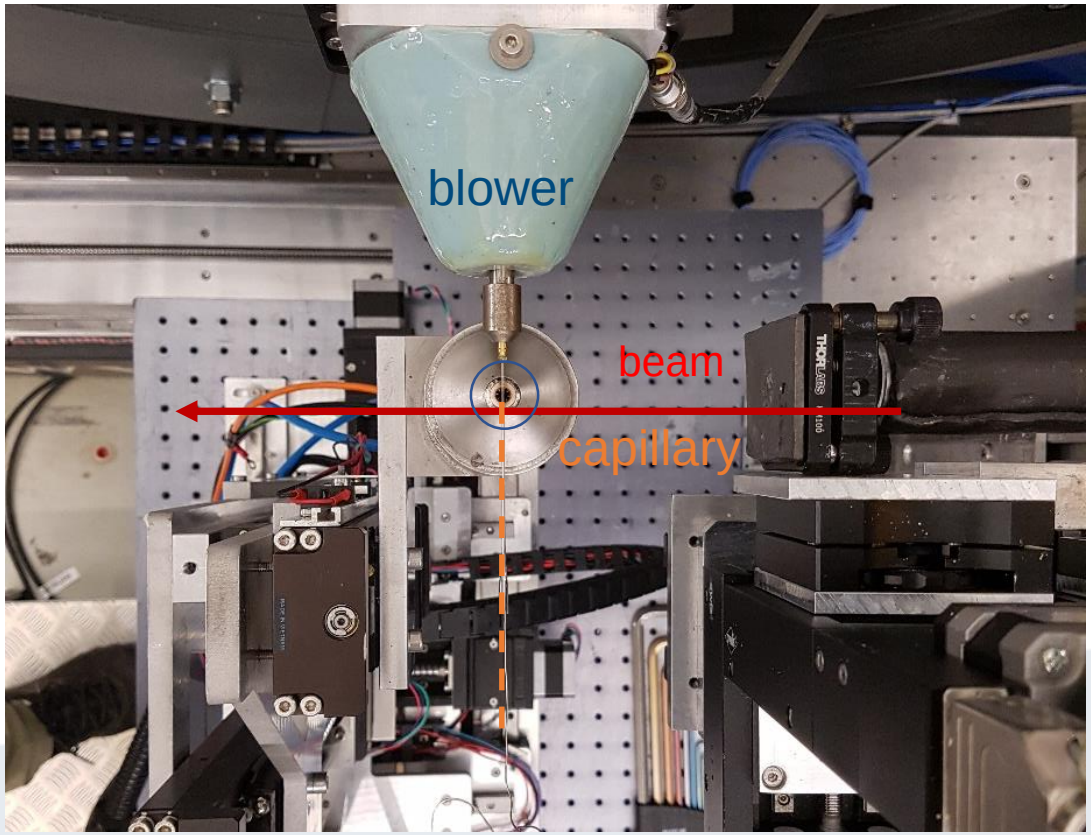


Relaxation and crystallisation of the amorphous structure of shock-compressed SiO_2 at elevated temperatures

Explanation of the mechanisms stabilizing the residual compression in amorphous SiO_2.

P02.1

- glass capillary + hot air blower (up to 900°C)
- $q_{max} \sim 20$ Å^{-1}
- analysis of fully amourphous structures (PDF calculation)



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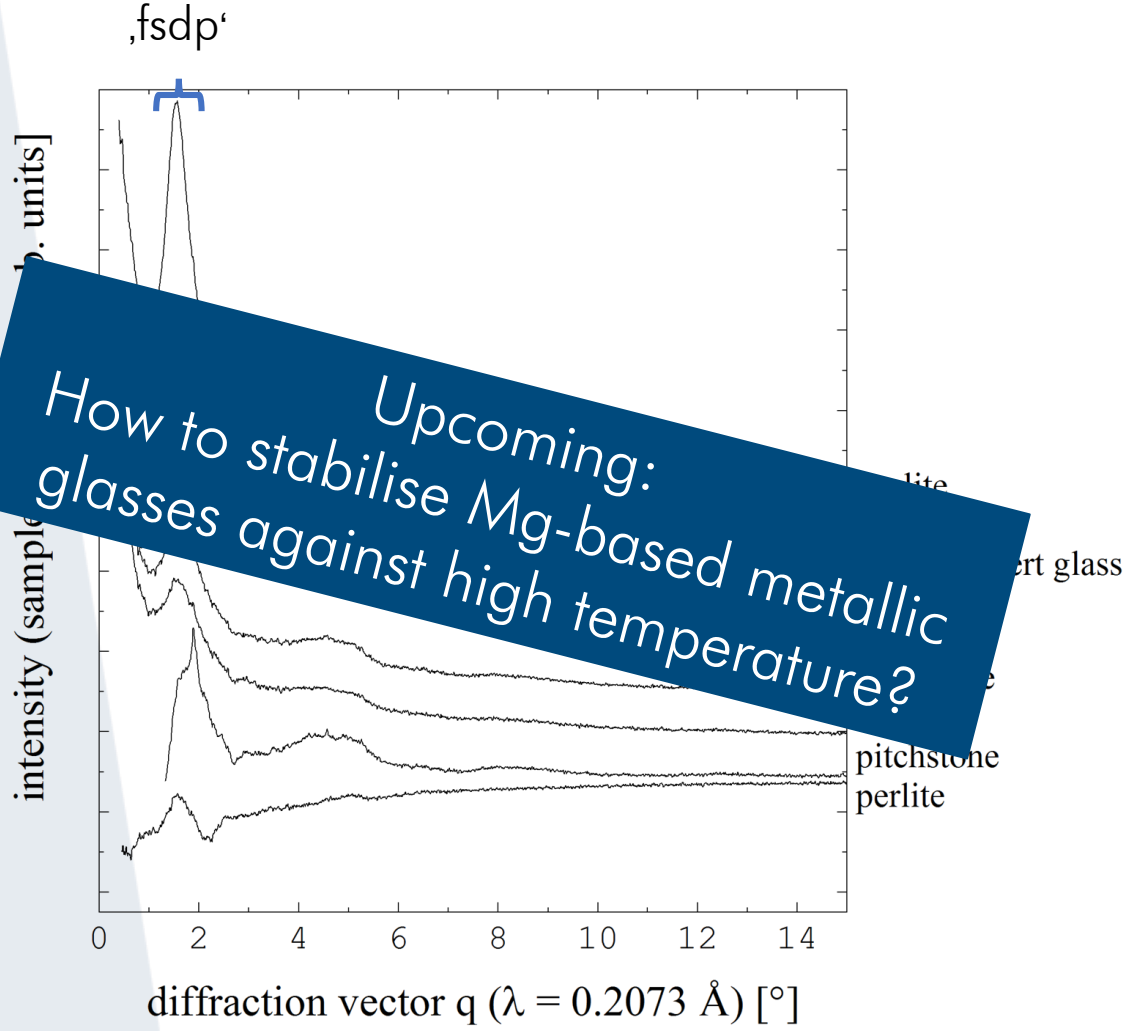
Total Scattering



Upcoming:
How to stabilise Mg-based metallic glasses against high temperature?

Differentiation among different natural amorphous silicon oxides
highly interesting for mineralogists!

(genesis of these materials: volcanic eruption, meteorite impact, lightning strikes,…)

comparison with artificially synthesized amorphous SiO_2 → implication for their stability

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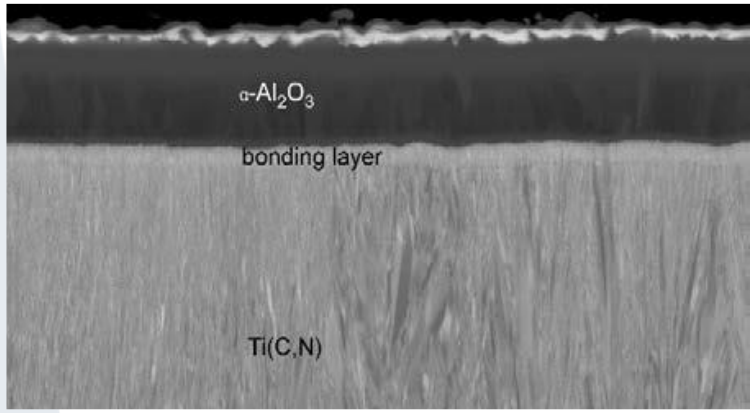
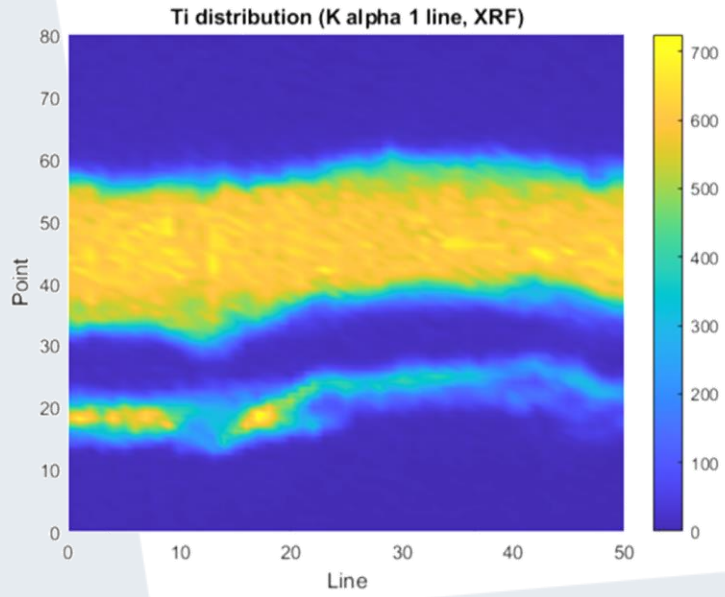
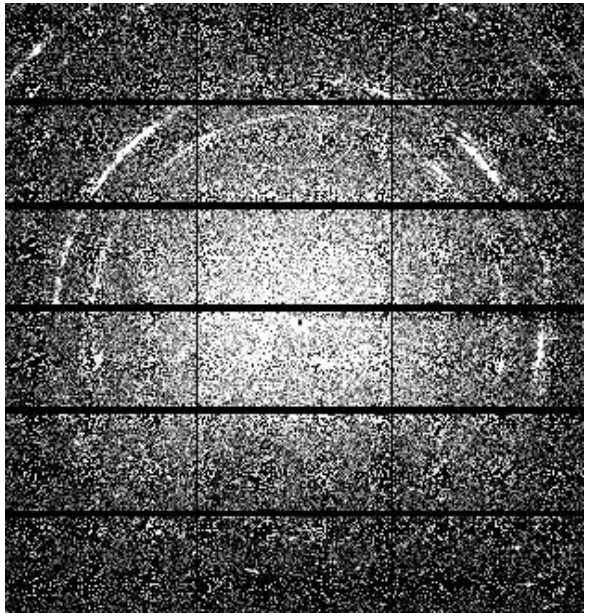
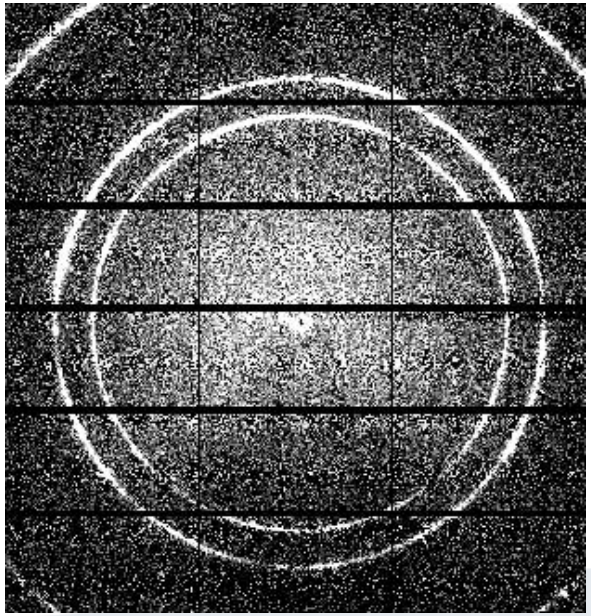
Nanobeam Diffraction on Thin Films





P03, nanofocus endstation
Transmission mode
Diffraction & X-ray fluorescence simultaneously
(control of thin film architecture/design)





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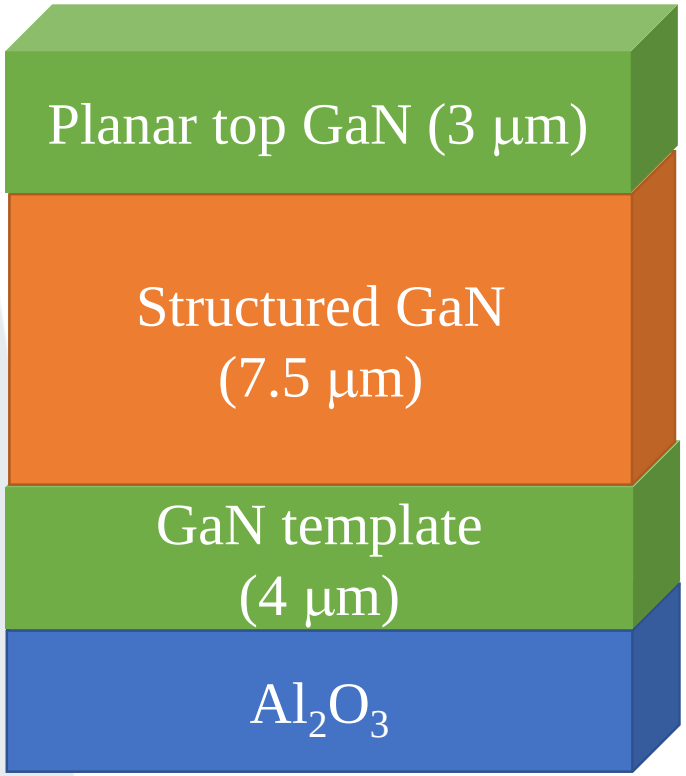
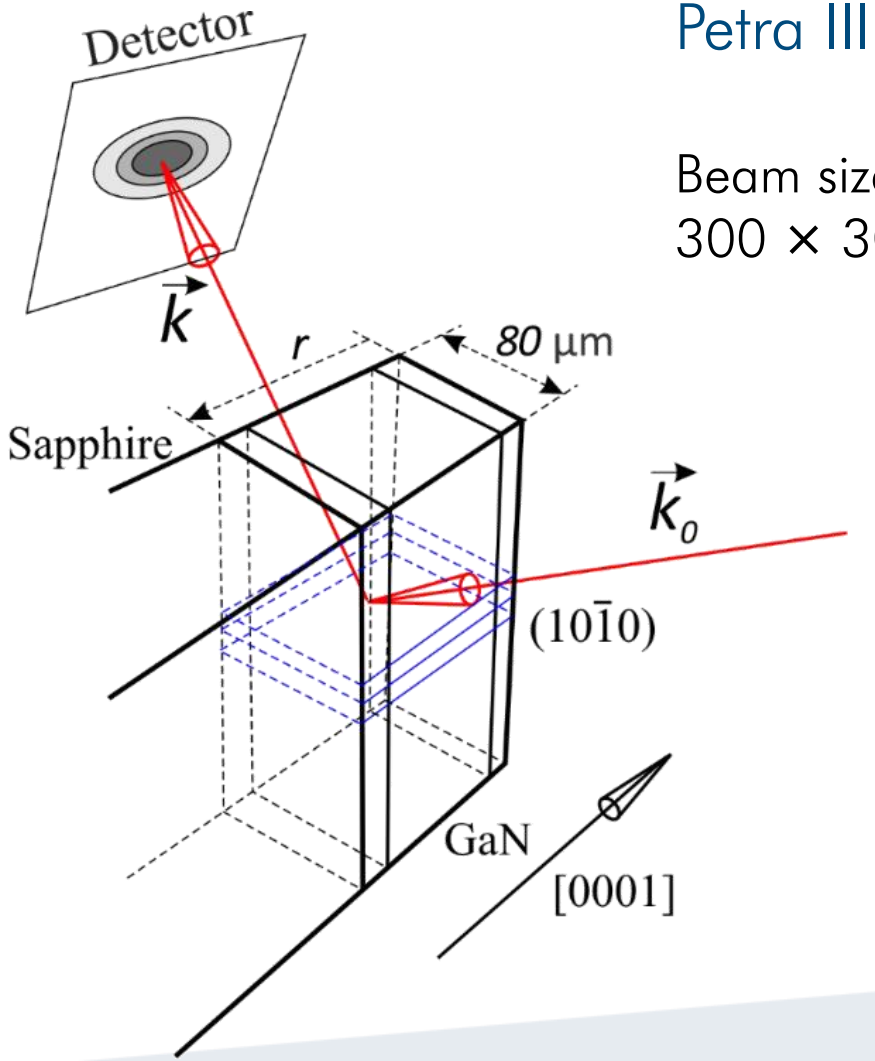
Nanobeam Diffraction on Thin Films

Structured GaN Crystals



Petra III / P03

Beam size
300 × 300 nm²



M. Barchuk, M. Motylenko, T. Schneider, C. Röder, A. Davydok, S. Lazarev, C. Schimpf, C. Wüstefeld, O. Pätzold, D. Rafaja: Defect-rich GaN interlayer facilitating the defect annihilation in GaN crystals grown by high-temperature vapor phase epitaxy, J. Appl. Phys. 126 (2019) 085301.

Prof. David Rafaja (rafaja@iww.tu-freiberg.de), TU Bergakademie Freiberg

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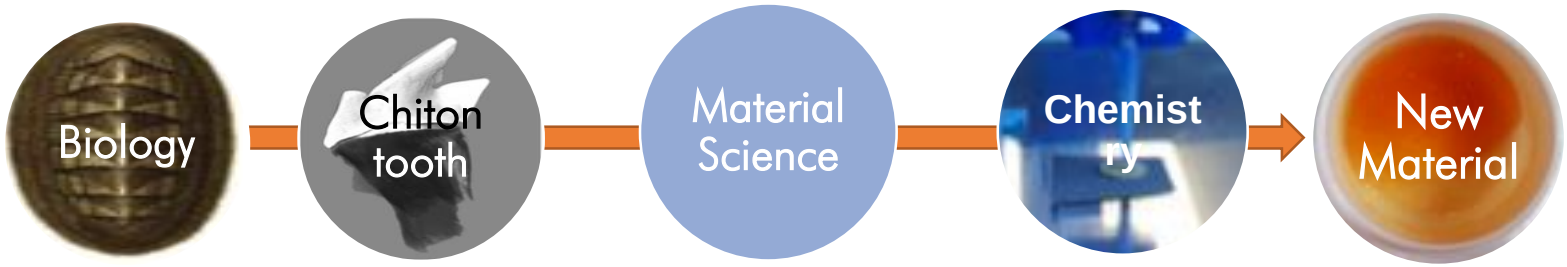
Synchrotron Radiation for Characterization of Biominerals

Professorship Biogenic Technical Materials

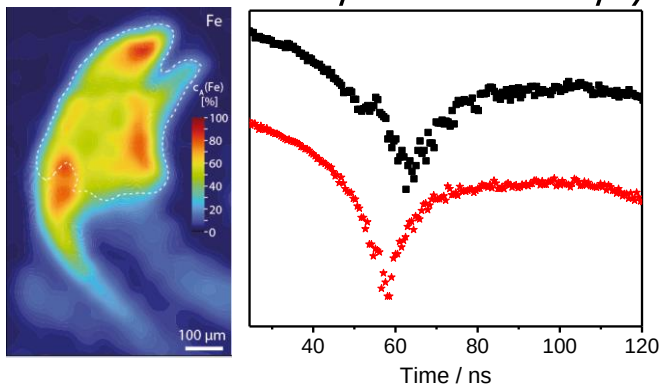
Juniorprof. Dr. rer. nat. Linus Stegbauer (Linus.Stegbauer@esm.tu-freiberg.de)

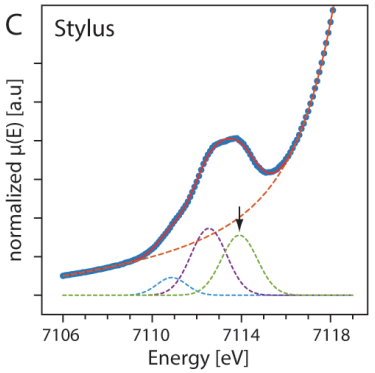
Past: Studying amorphous iron biominerals at the synchrotron: Spatially resolved on sub μm

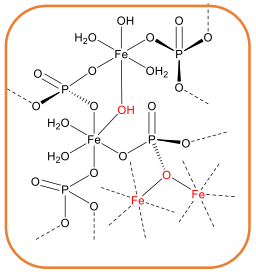
Jun.-Prof.
Linus Stegbauer
TUBAF

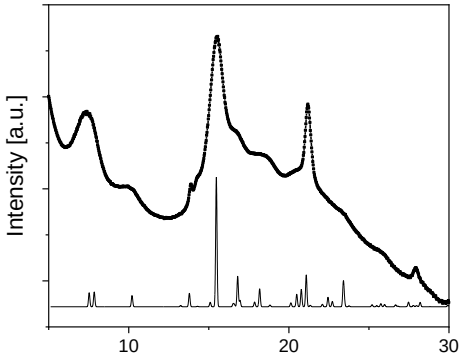


μCT → *Synchrotron Mößbauer Spectroscopy* → *Near edge X-ray absorption* → *WAXS*









Experimental work conducted at the APS (Argonne) at Beamlines 3ID-B, DnD Cat, 6-ID
17 days of beam time (2017-2019).

L. Stegbauer, E. E. Alp, P. Smeets, R. Free, S. Wallace, M. Hersam, D. Joester, PNAS, 2021, 23, e2020160118.

Future: Studying amorphous calcium biominerals at the synchrotron: Spatially resolved on sub μm

Jun.-Prof.
Linus Stegbauer
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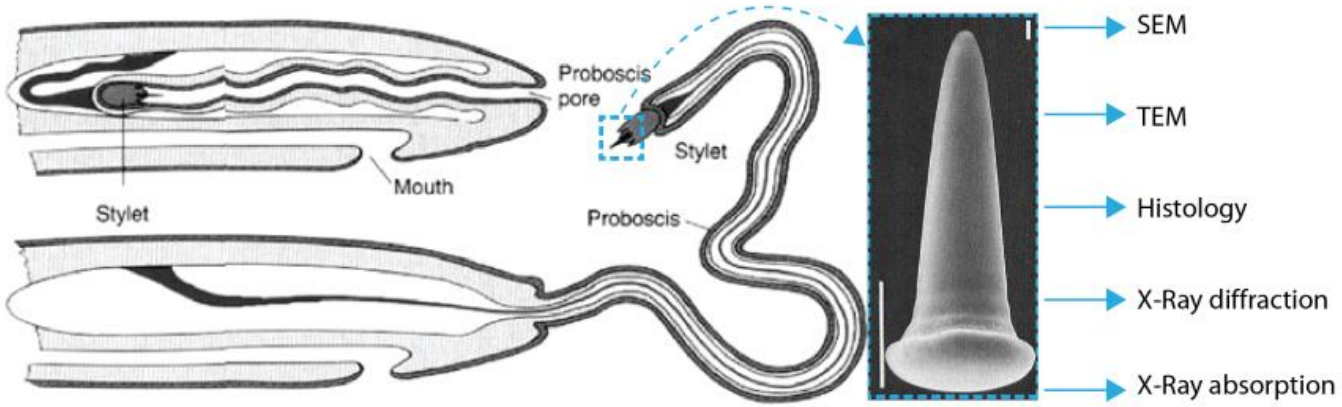

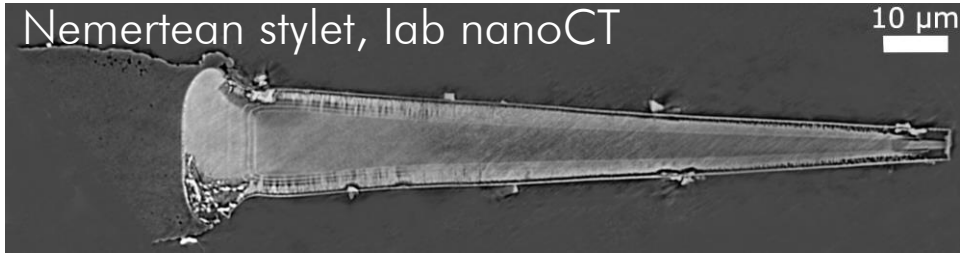

Future work on persistent amorphous calcium phosphate biominerals such as the nemertean stylet: WAXS, nanoCT, XAFS, XRF desired.
Potential to discover new stabilizing mechanisms of inorganic phases in bone replacement materials.

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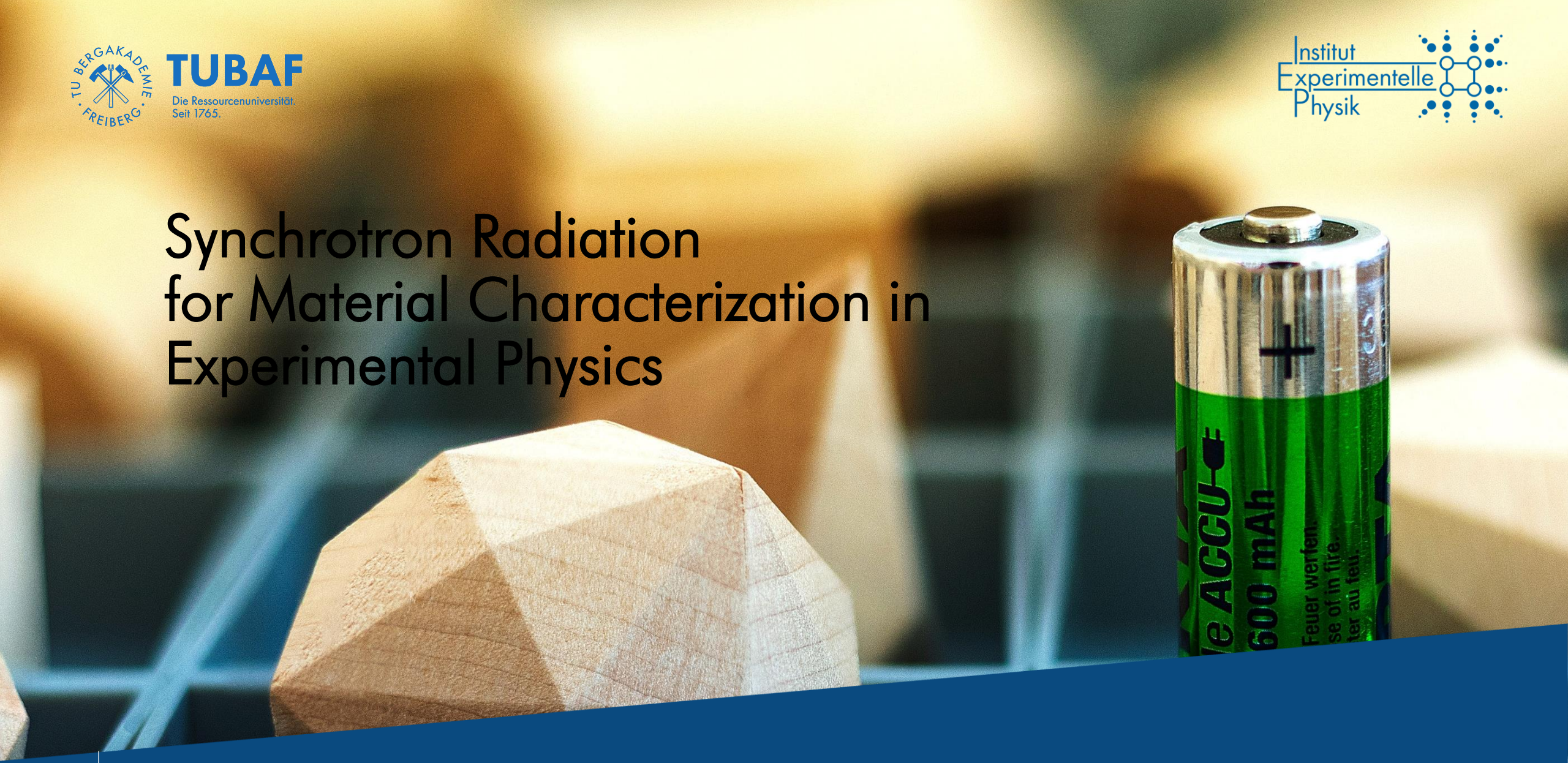
Institut Experimentelle Physik

Synchrotron Radiation for Material Characterization in Experimental Physics

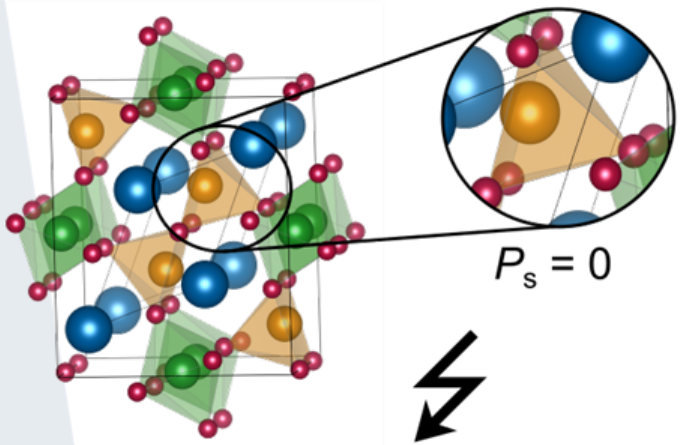


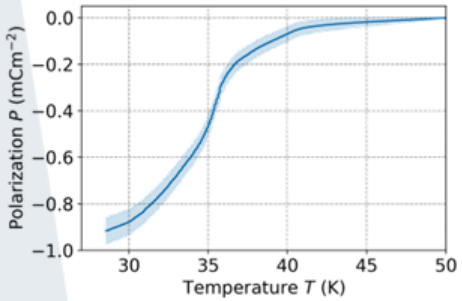
Institute of Experimental Physics
Tina Weigel (tina.weigel@physik.tu-freiberg.de)

Research Highlights with Synchrotron Radiation

Picometer atomic displacements behind ferroelectricity, Experiment @ P23

CM Phase of YMn_2O_5





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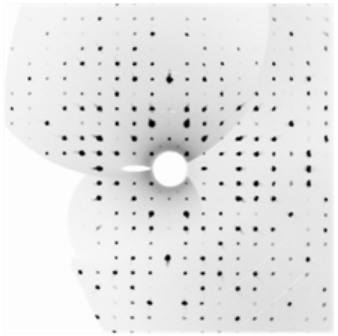
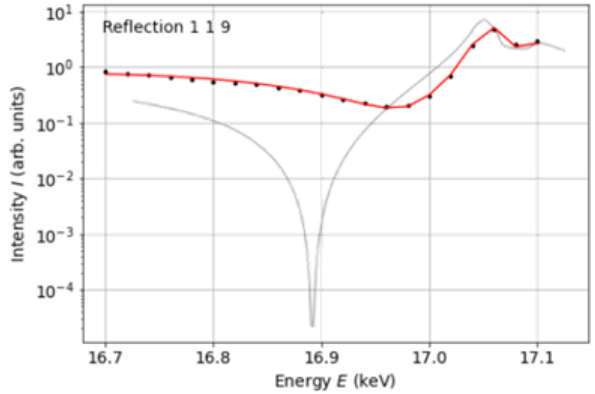
Conventional structure analysis



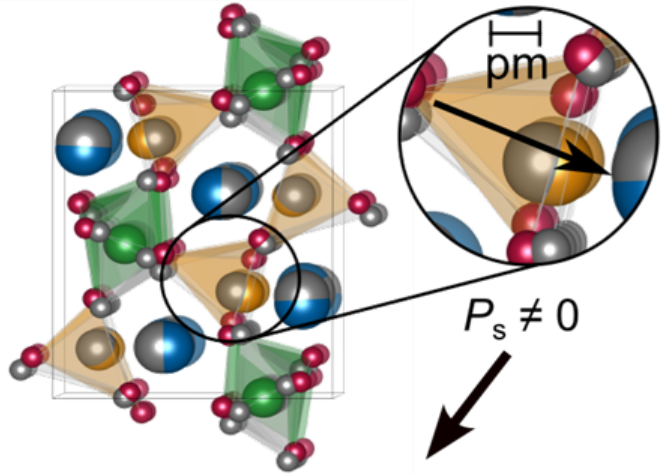
versus

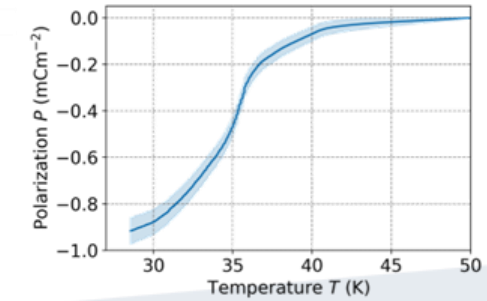


Resonantly Suppressed Diffraction

structural sensitivity

First refined structure of CM phase





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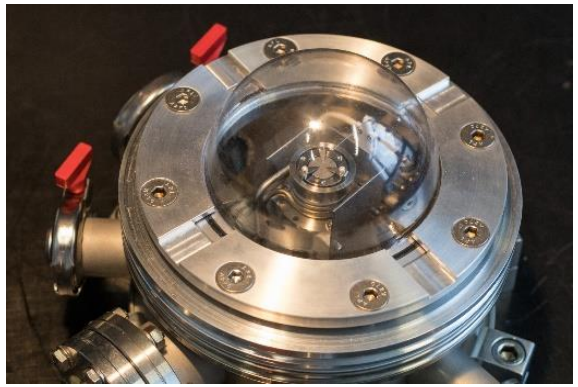
C. Richter, M. Zschornak, D. V. Novikov, E. Mehner, M. Nentwich, J. Hanzig, S. Gorfman, and D. C. Meyer, Nature Communications, 9, 178 (2018).

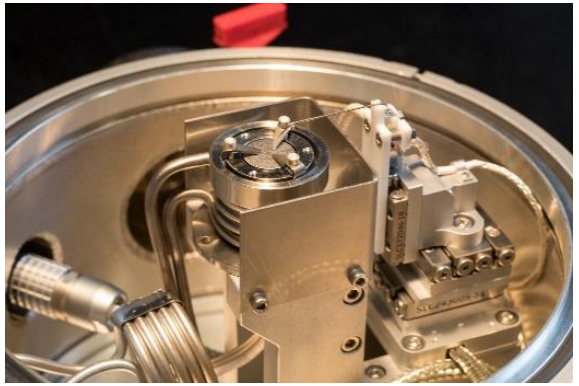
T. Weigel et al. Picometer atomic displacements behind ferroelectricity in the commensurate low temperature phase in multiferroic YMn_2O_5, Physical Review B, under Review

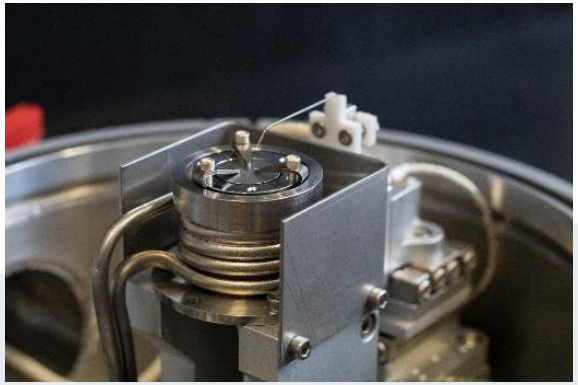
Research Highlights with Synchrotron Radiation

Instrumental development - Nordseekammer

- Customized sample chamber for beamlines P23 and P24 at PETRA III
- Chamber body consists of standard vacuum components
- Operating in vaccum (10^{-6} mbar)
- X-ray transparent dome
- Temperature range from 97 K to1253 K
- Cooling with compressed air, water, or liquid nitrogen
- Electric field up to 10 kV
- Customized *xyz* stage with probing needle for electrical and mechanical contact





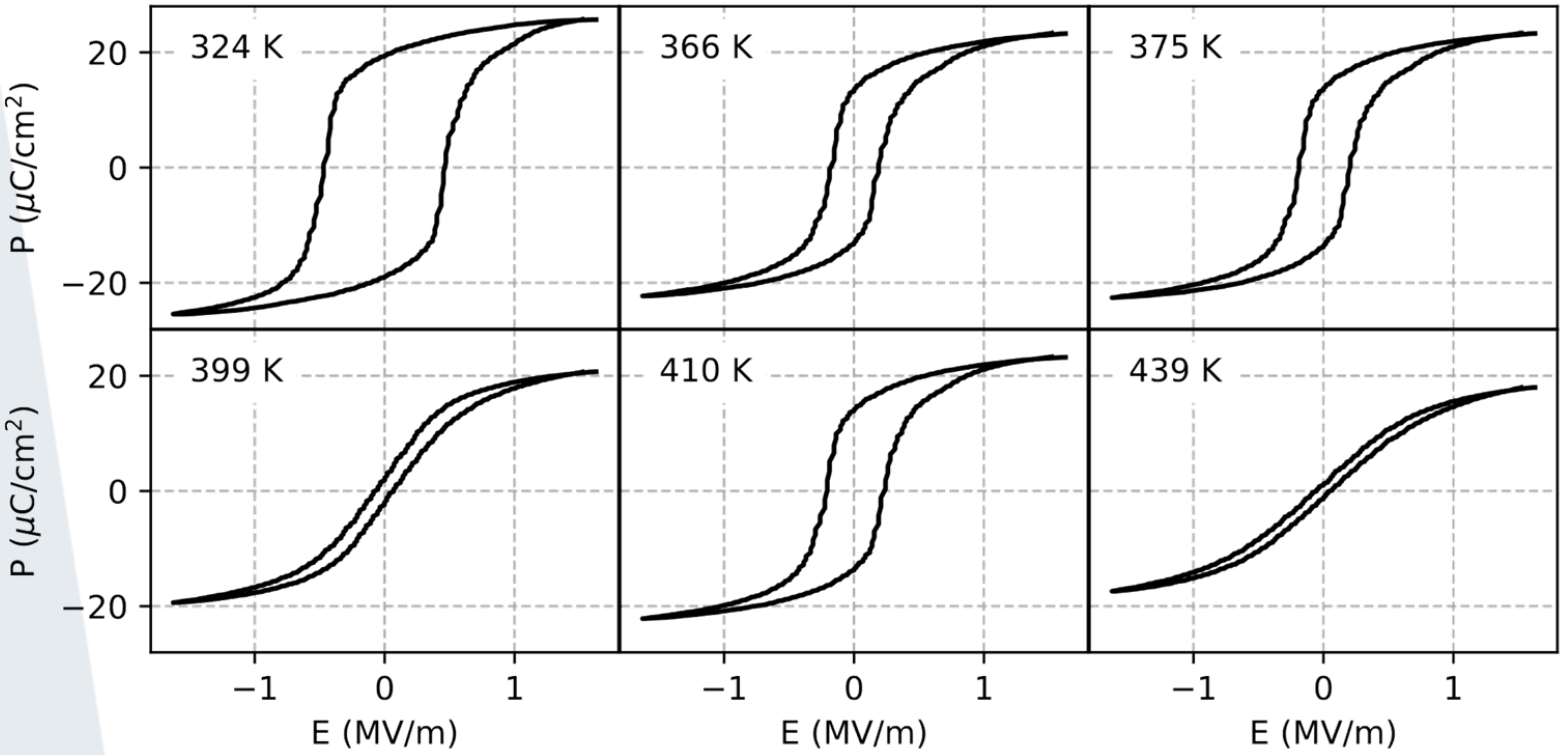


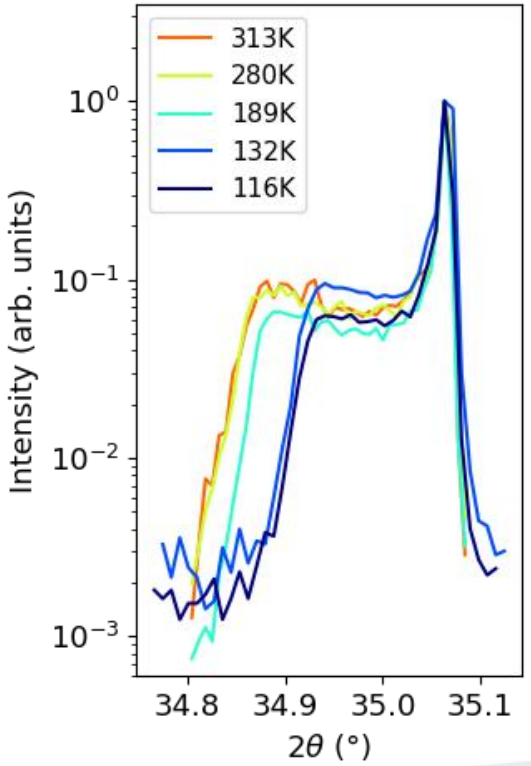
Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

M. Nentwich, T. Weigel, C. Richter, H. Stöcker, E. Mehner, S. Jachalke, D. V. Novikov, M. Zschornak, D. C. Meyer, Journal of Synchrotron Radiation 28158–168 (2021

Research Highlights with Synchrotron Radiation

Nordseekammer - Observation of phase transition in materials under electric field @ P23





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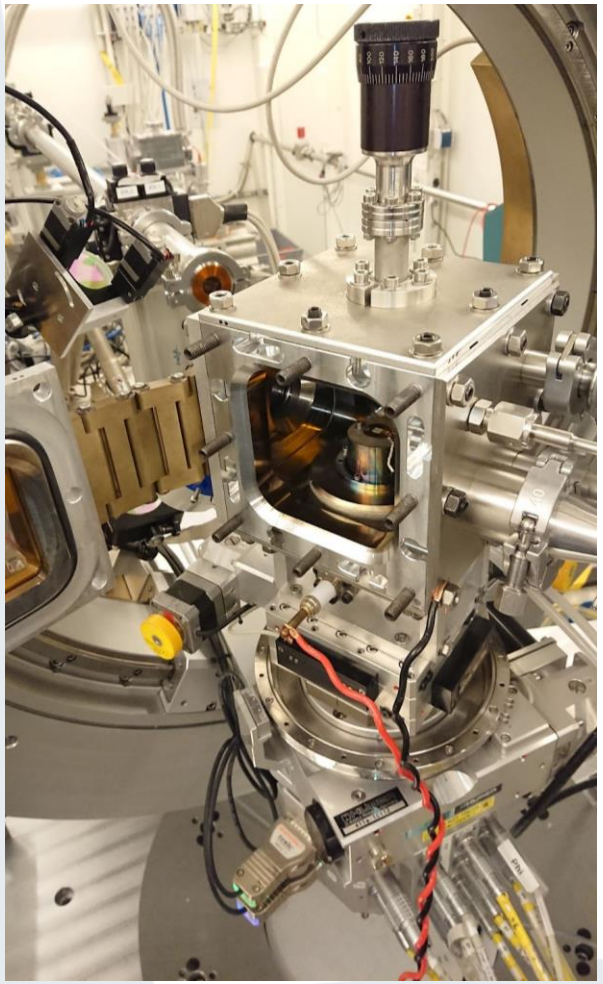
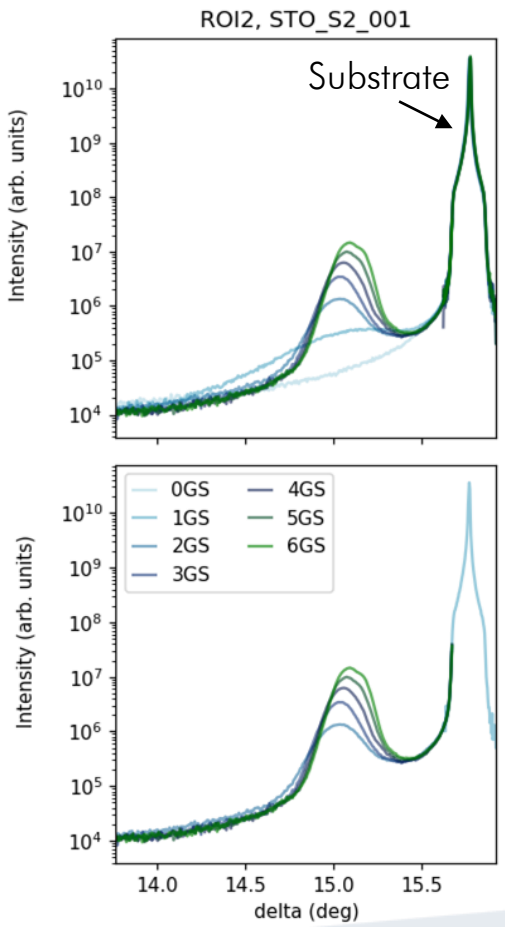
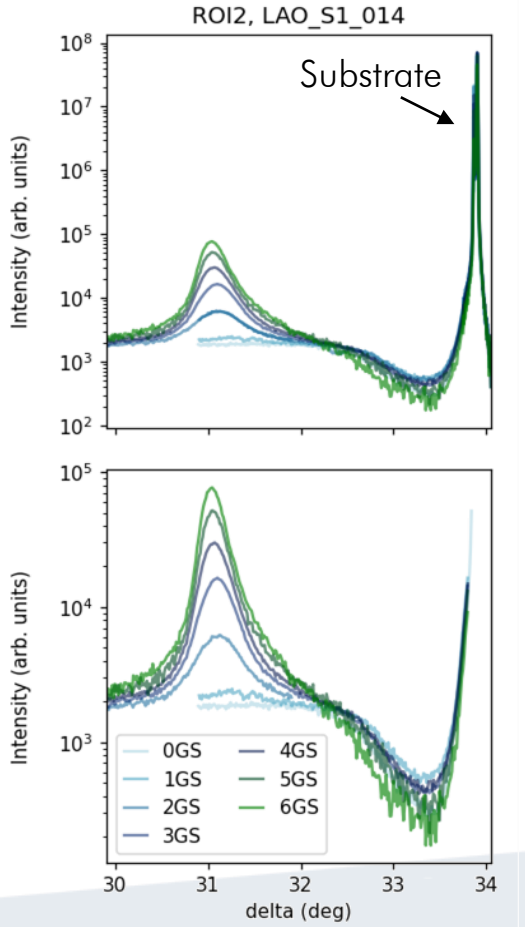
Research Highlights with Synchrotron Radiation

In situ investigation of polarized $BaTiO_3$ film growth by Pulsed Laser Deposition @ P23





$BiTaO_3$ on $SrTiO_3$, 001 reflection



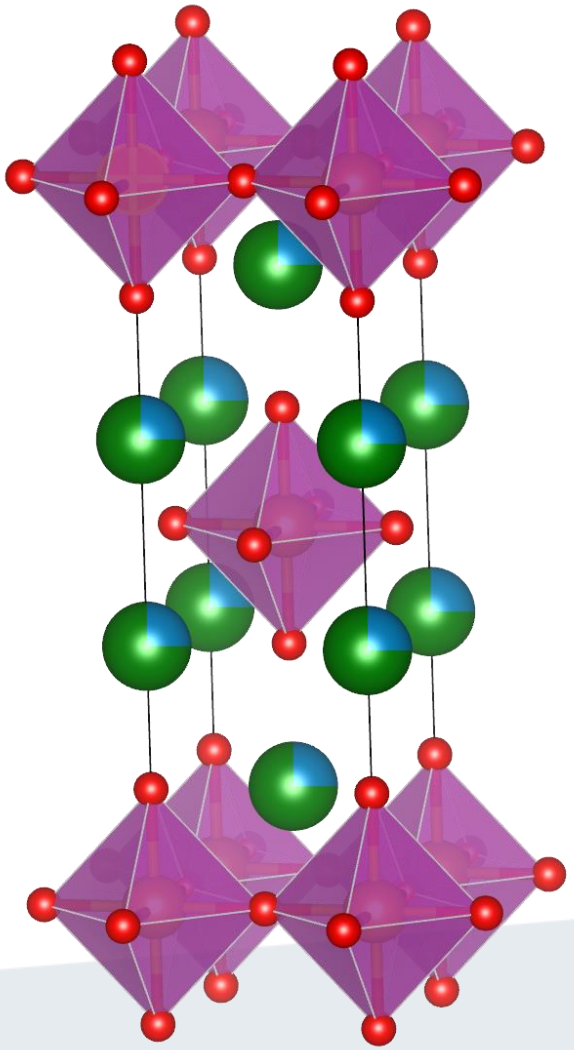
$BiTaO_3$ on $LaAlO_3$, 014 reflection

Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

Research Highlights with Synchrotron Radiation

Fourier-free refinement of the La/Sr split position in $La_{0.5}Sr_{1.5}MnO_4$

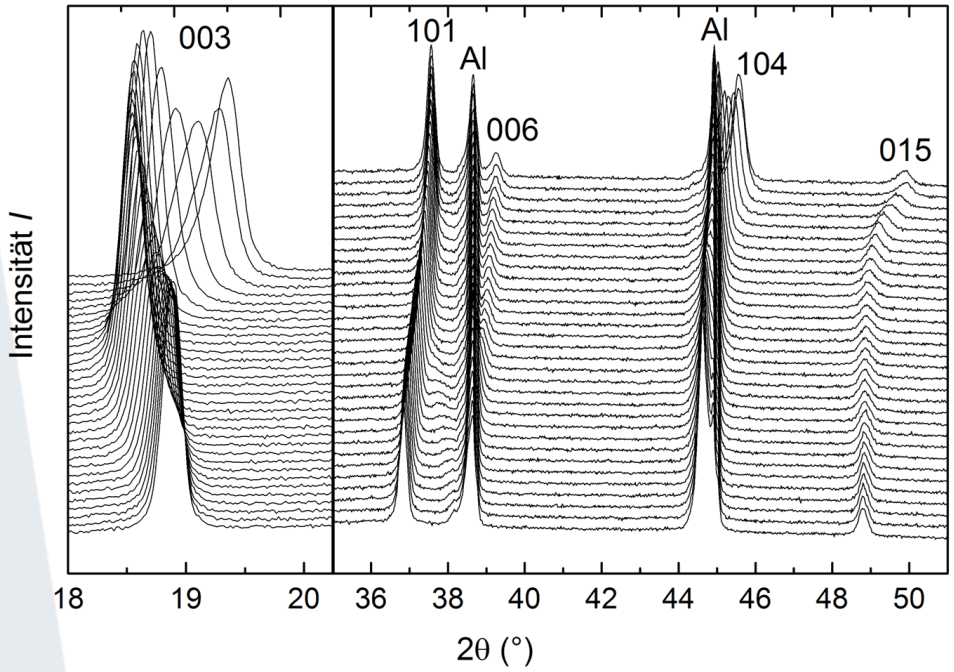
- $La_{0.5}Sr_{1.5}MnO_4$ has been investigated in the potential high-temperature super-conductor (space group *I4/mmm)*
- Positional shift of the cations due to the La/Sr Split in order of $\Delta z \approx 2$pm from the high symmetry position (0, 0, $z = 0.3584$)
- Tuning the resonate scattering contrast of La and Sr by f'_{Sr} to improve the resolution: positional shift of $\Delta z > 0.0034$ (≈ 4.2 pm) with a theoretical precision of up to $z \approx \pm 0.00015$

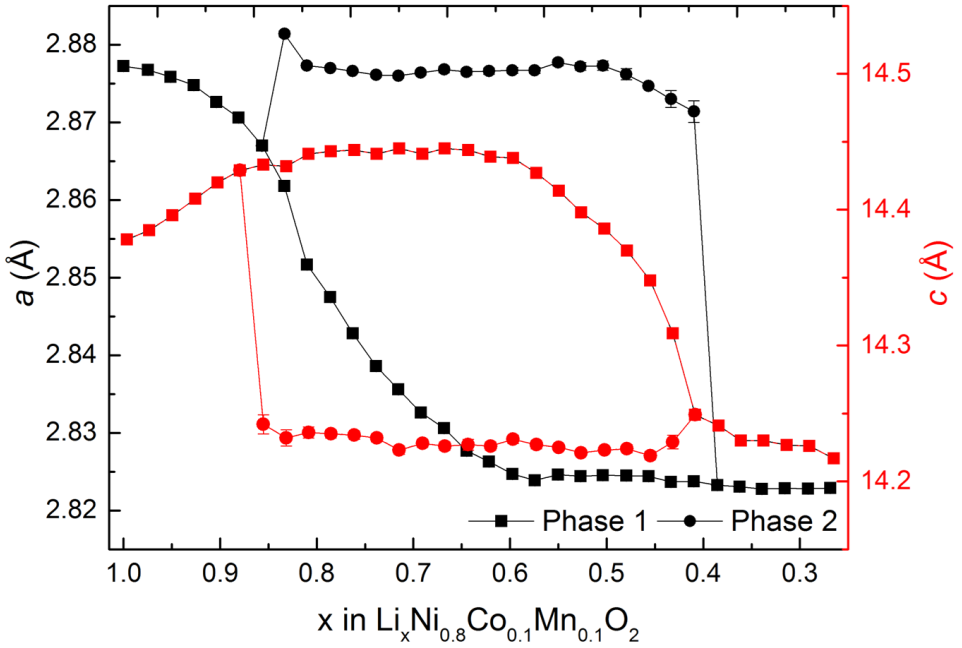


Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

Research Highlights with Synchrotron Radiation

Characterization of new battery materials





- Structural characterization of battery materials during charging/discharging
  - → X-ray diffraction to investigate structural changes
  - → Resonate X-ray scattering to investigate changes of the oxidation state

Tina Weigel (tina.weigel@physik.tu-freiberg.de), TU Bergakademie Freiberg

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THANK YOU
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