QNANO Research Infrastructure

| ANKA-PDIFF: Polycrystalline X-ray Diffraction | Category: |
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## ANKA-PDIFF: Polycrystalline X-ray Diffraction

**Technology:**
*X-ray diffractometry*
*Bulk and thin-film nano/microstructure analysis*
*Chemical-crystallographic structural characterisation*

**Equipment:**
- *3-circle powder diffractometer for large loads*
- *4-circle single crystal diffractometer for low loads*

**Category:**
**C. Particle Characterization in- and ex-situ**

**Institute:**
**Karlsruhe Institute of Technology (KIT)**

**Location:**
Karlsruhe Institute of Technology
Institute for Synchrotron Radiation (ISS)
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**Short technology description/Overview** *(approx 300 words)*:

Hard X-ray (6-21keV) synchrotron beamline for the structural investigation of polycrystalline materials under varying in-situ conditions and for high-resolution powder diffraction and residual-stress and texture measurements. The beamline is also well suited equipped for high-resolution scattering studies on single-crystals, epitaxial layers and bulk single-crystalline materials under ambient conditions. The experimental facilities consist essentially of a heavy duty 3-circle powder diffractometer with detectors and sample conditioning chambers and a second 4-circle kappa-goniostat optimised for high-resolution scattering experiments.

Typical applications of both experimental set-ups are:

- Real-time in-situ characterization of nanostructural & microstructural properties of metals and alloys,
- In-situ powder studies of crystallographic phase changes in bulk polycrystalline materials,
- High-resolution powder diffraction for chemical structure determination/characterization (e.g. pharmaceutics)
- Residual-stress and texture analysis in functional polycrystalline thin films

**Main Features (Equipment Capabilities):**

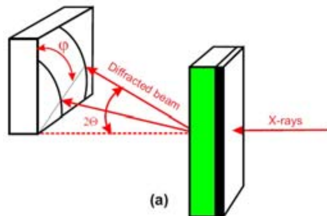
- tuneable X-ray energy between 6 and 21keV (wavelength 2.1-0.62Å), focal spot size (fwhm) 0.6mm x 0.3mm, energy resolution ($\Delta E/E$) ≈ $2x10^{-4}$ (at 10 keV)
- 3-circle powder diffractometer ($2\theta_1$, $2\theta_2$, $\theta$) with 2D and 1D detectors, Kappa-diffractometer with 3 sample orientation circles ($\Omega$, K, $\Phi$) and detector circle ($2\theta$) equipped with Ge111 or Si 111 crystal analyser
- Spatial resolution approx. 100μm
- Diffraction experiments in either transmission or reflection between 77K & 1300K
- Various cells/sample holders for solid & liquid samples

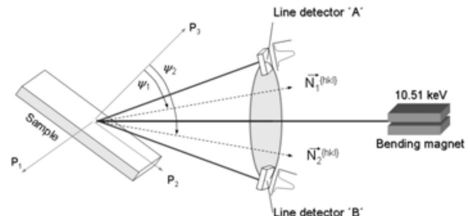
**Limitations / constraints**

- Detection/Measuring limits: bulk sample quantities down to approx 5 mg, thin film samples down to a few 10's of nanometers thickness depending upon surface quality

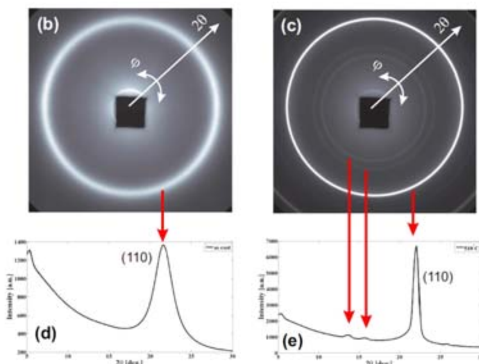
- Time resolution for real-time investigations 0.1s to 10s depending on sample scattering power/quantity
- Some possibility of radiation damage depending on sample type (organic/biological samples more prone to X-ray damage)

**Typical structures & designs**

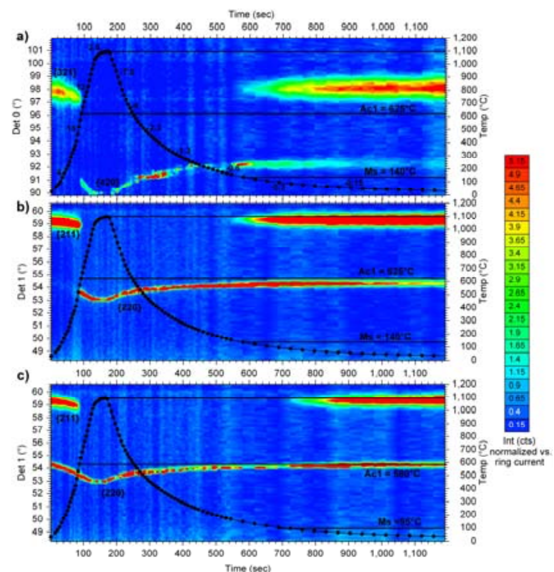



*Schematic view of single exposure set-up for fast in-situ strain analysis*




*Diffraction patterns of a metallic alloy in the glassy state (b, d) and after annealing at 500°C (c, e), showing the development of nanocrystalline structure (particle size approx. 10nm) with temperature.*




*Evolution of lattice parameter change in α-Fe as a function of temperature: diffraction pattern sampling rate 1Hz.*

*Any further Information*: