Karlsruhe Institute of Technology

Institute for Applied Materials
Energy Storage Systems
AK Ehrenberg

Bundesministerium für Bildung und Forschung

High-resolution and stroboscopic XRPD

at the P02.1 beamline of PETRA III (DESY)

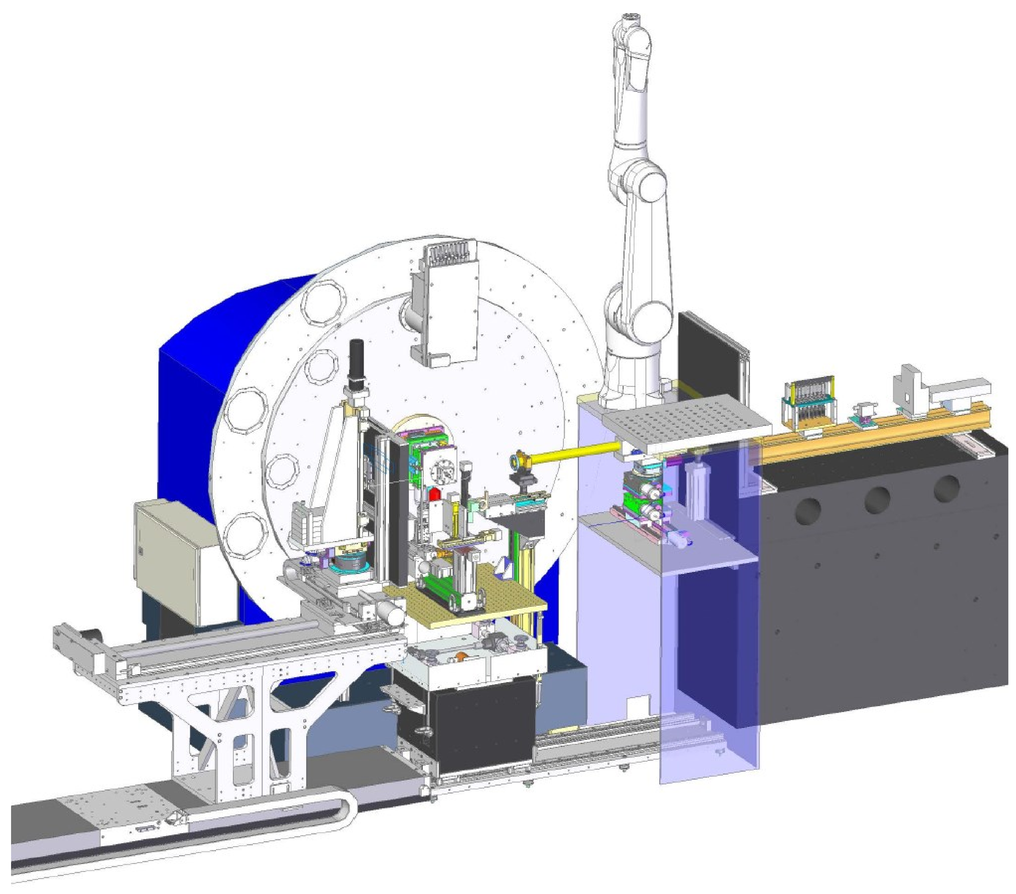
A. Schökel[1], A. Berghäuser[2], M. Etter[3], S. Gorfman[4], M. Hinterstein[1], M. Knapp[1]

[1] Karlsruher Institut für Technologie, [2] Universität Hamburg, [3] Deutsches Elektronensynchrotron, [4] Universität Siegen

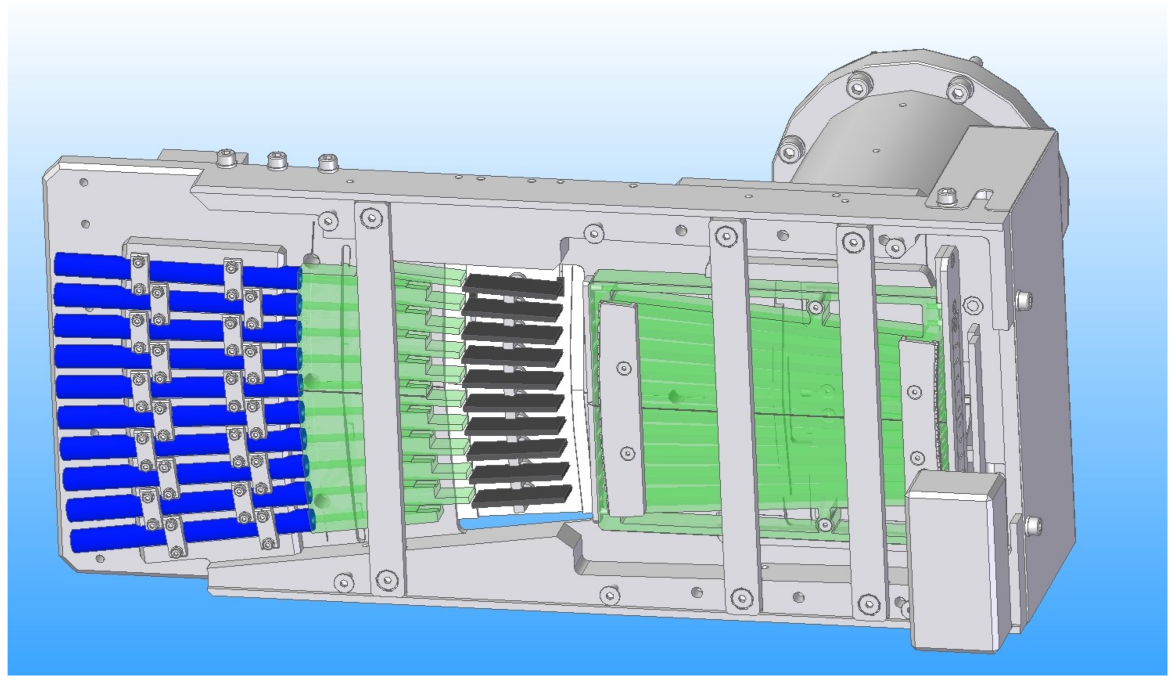
The **P02.1 beamline** at the PETRA III synchrotron is a dedicated diffraction beamline with a fixed photon energy of 60 keV. The high X-ray energy makes the beamline especially suited for in-situ studies and strongly absorbing samples.
Besides a commercial Perkin Elmer XRD1621 2D detector, a high-resolution 10 channel multi analyzer crystal detector has recently finished commissioning. Together these detectors provide a wide range of capabilities from pair distribution function to high resolution X-ray diffraction analysis.

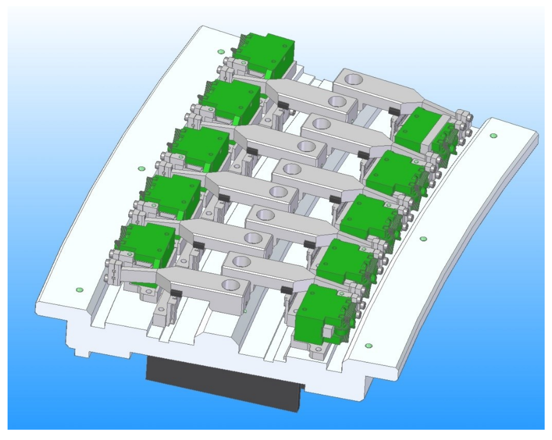
Beam parameter at sample position	
energy / wavelength	60 keV / 0.207 Å
rel. energy bandwidth	2.5×10^{-4}
photon flux	4×10^{10} photons/s
spot size (FWHM)	max. 2.000 x 2.000 µm²
	min.100 x 100 µm²

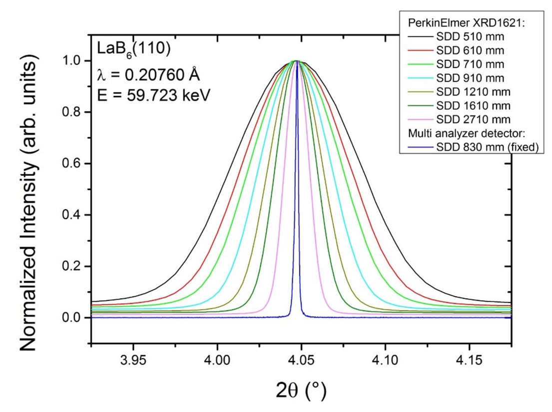
High-resolution detector	
type	multi analyzer crystal detector
crystals	Si 111 in Bragg geometry
channels	10
detectors	YAP scintillator + photomultiplier
resolution	3.5×10^{-5} degrees (mechanical)



Overview of the P02.1 beamline layout.





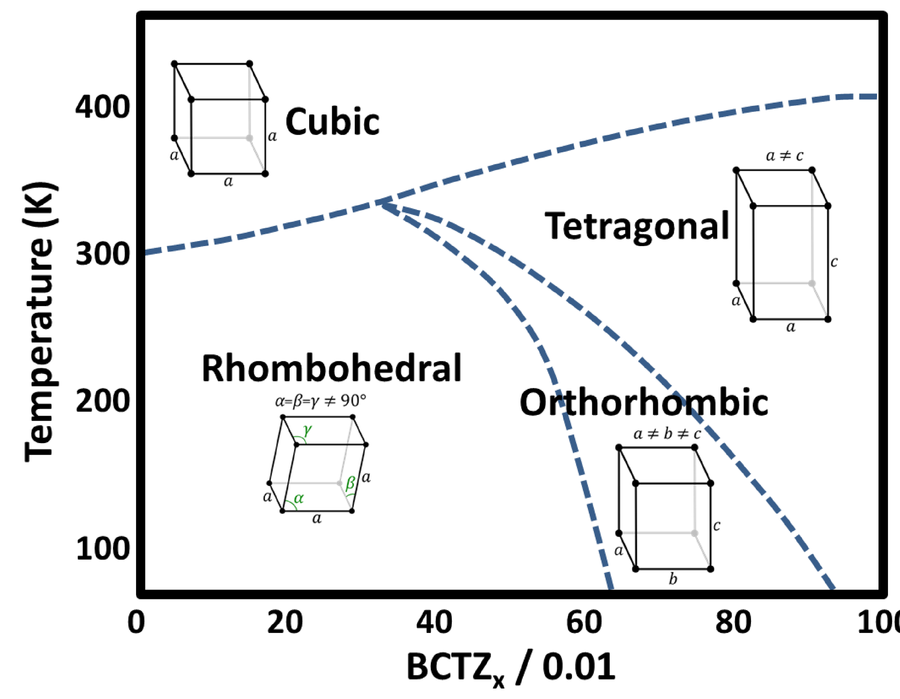


(top) overview of the multi analyzer crystal detector, depicting the tungsten collimators (green), analyzer crystals (black) and detectors (blue). **(bottom left)** detailed view of the piezo actuators driving the 10 silicon crystals. **(bottom right)** comparison of the FWHM of the LaB_6 110 reflection using the Perkin Elmer and the high resolution detectors giving a minimum value of ~$2.4 \cdot 10^{-3}$ deg.

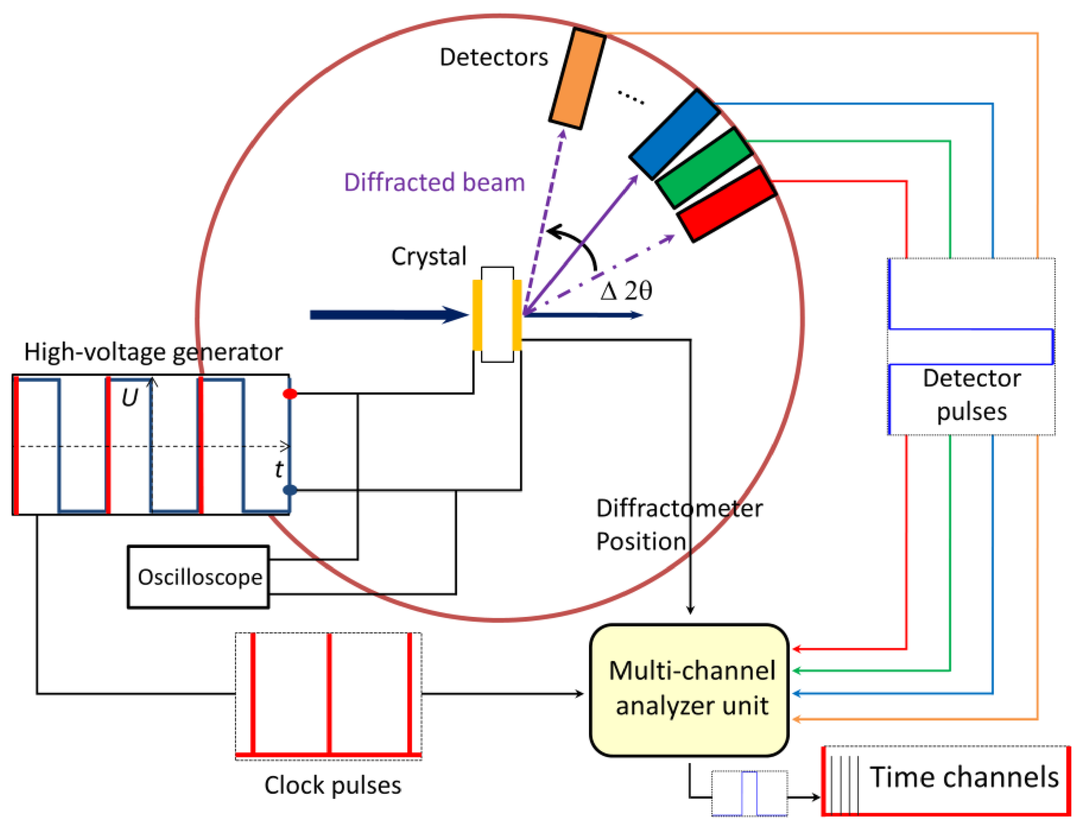
Using a FPGA based data acquisition system developed by the Universität Siegen, diffraction data can be collected with a temporal resolution down to 10 ns. For the measurement of a piezo ceramic sample under unipolar excitation by a 10 kHz, 500 V/mm electric field the diffraction patterns were binned to 1 µs time resolution to improve statistics. The total measuring time was about 10h.
Our stroboscopic pump-probe measurements on doped barium titanate (BZT-BCT) showed:

- Unexpected and highly complex behavior on the low µs scale in the morphotropic region
- 111 and 200 reflections show different behavior in time
- Effects of phase transition and lattice distortion can be separated
- Resonant lattice vibrations

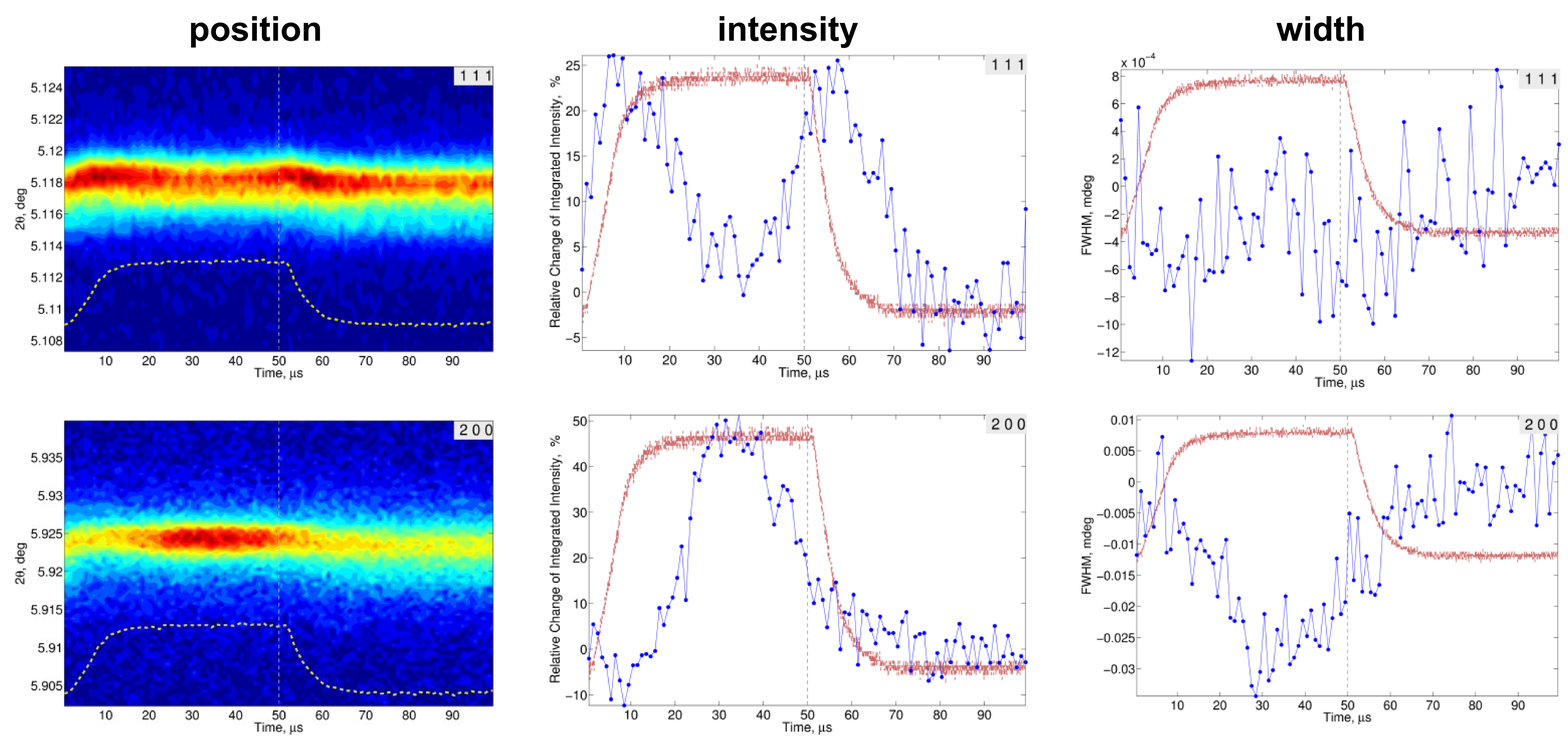
Depending on frequency the sample will heat up. A future design will implement a temperature controlled sample environment.



Phase diagram of BTO solid solutions.
[Keeble et al., Applied Physics Letters 102, 092903 (2013)]



Schematic of the experimental setup for stroboscopic powder diffraction on a piezo ceramic sample under electric field.



Diffraction data of a BZT-BCT piezo ceramic sample under periodic excitation by an 10kHz, 500V/mm unipolar electric field. **(on the left)** plots of the intensities of the 111 and 200 reflections in relation to the electric field (dotted line). **(middle and right)** intensities and widths of the reflections (blue) in relation to the electric field (red).

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