

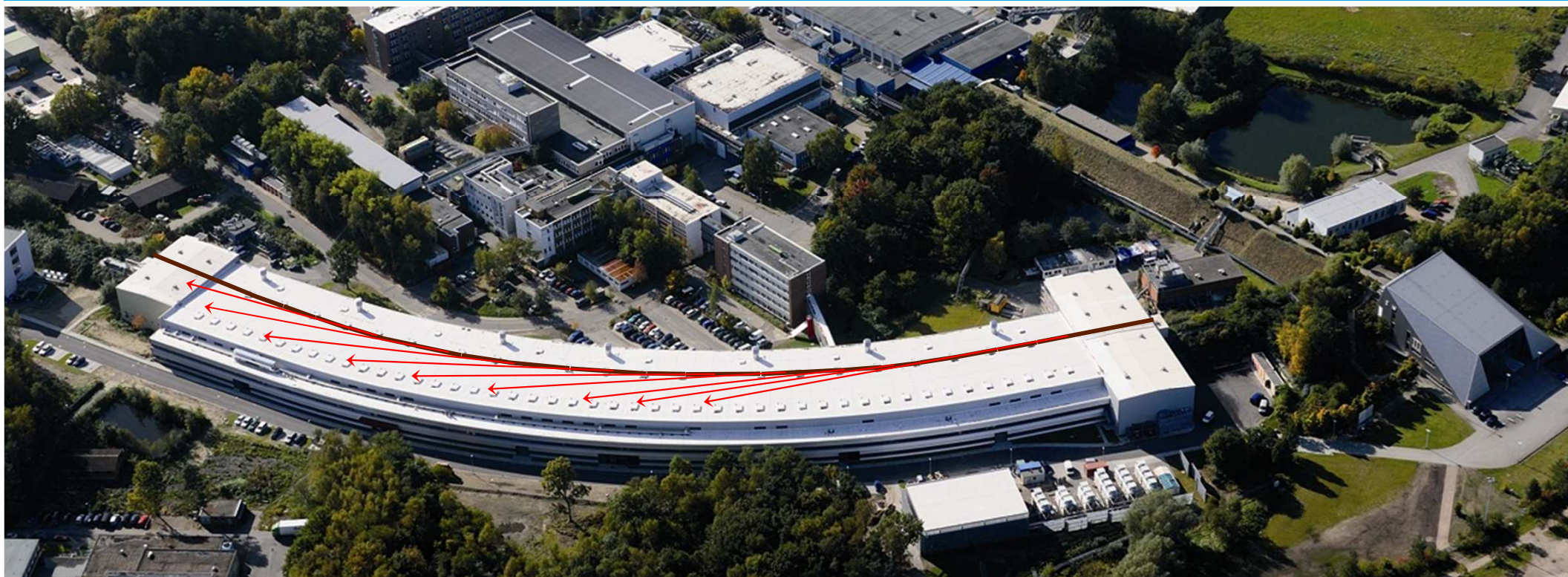
PETRA III

Materials science opportunities



Hermann Franz
DESY/KEK Meeting
March 20 2012

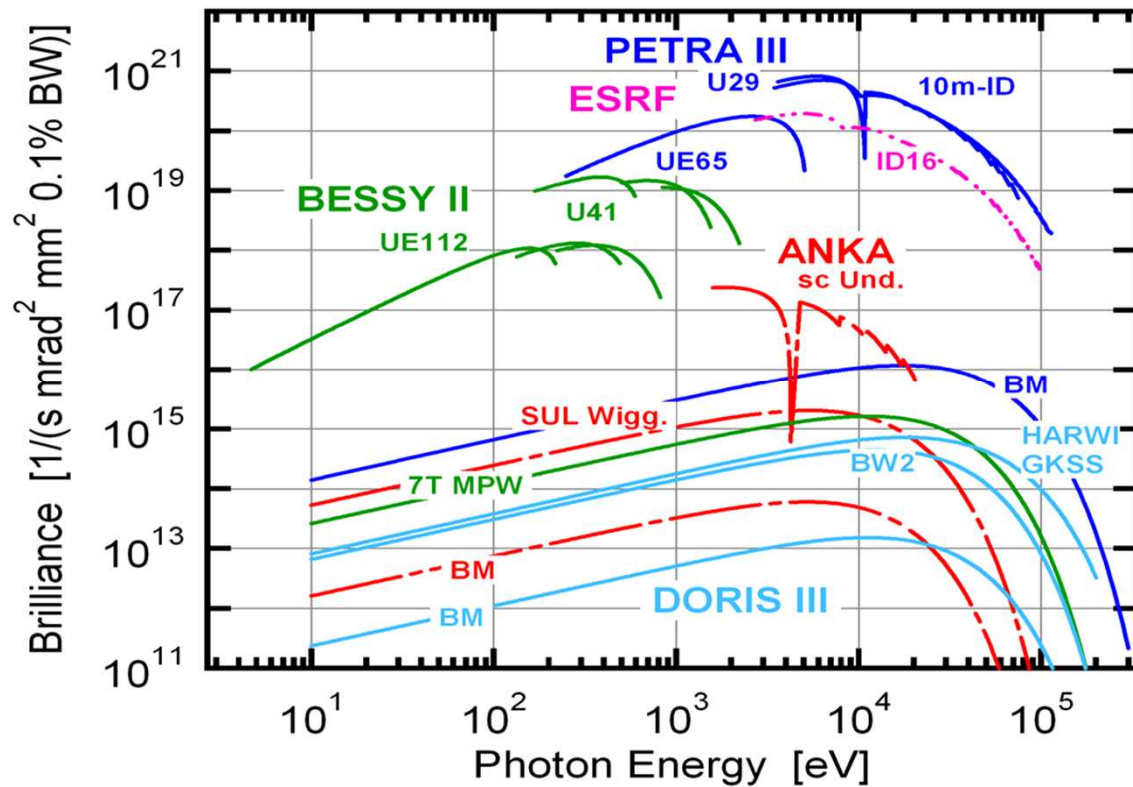
PETRA III: DESY's brilliant X-ray source



- particle energy: **6 GeV**
- stored current: **100 mA (top-up)**
- emittance: **1.0 nmrad**
- circumference: **2304 m**
- # of undulators: **14 (incl. canted)**
- # of experiments: **30**
- X-ray wavelength: **10 – 0.05 Å**
- beamline length: **70-100 m**
- annual operation: **5000 h**

- built in 1978
- rebuilt as a synchrotron radiation source in 2007/2009
- commissioning since 2009
- user operation since 2010
- largest storage ring of its kind

PETRA III – a high brilliance machine



- coherent flux @12keV ($B(\lambda/2)^2$):
 - 2m ID: $\sim 4 \times 10^{10}$ ph/s/0.01%BW
 - 5m ID: $\sim 1 \times 10^{11}$ ph/s/0.01%BW
 - 20m ID: $\sim 2.5 \times 10^{11}$ ph/s/0.01%BW

Main goal of PETRA III

Production and use of
nanometre sized X-ray
beams

Photon beam parameters at 12keV:

	β_x [m]	β_y [m]	σ_x [μ m]	σ_y [μ m]	$\sigma_{x'}$ [μ rad]	$\sigma_{y'}$ [μ rad]	ID-length [m]
low- β 5 m	1.3	0.6	35.9	5.7	28	5.0	5
high- β 5 m	20	2.38	141	5.2	8.6	5.2	5

Horizontal β -function of each straight section can be selected individually
and is changeable ($\beta_x = 1.3\text{m}$ or $\beta_x = 20\text{m}$)



PETRA III beamline list (phase I)

Number	Name	ID type	Energy range	Contact
P01	Dynamics beamline, IXS, NRS	10 m U32	5 - 40 keV	H.-C. Wille, DESY
P02	Powder and Extreme Conditions	2 m U23	20 - 100 keV	H.-P. Liermann, DESY
P03	Micro and Nano SAXS/WAXS	2 m U29	8 - 25 keV	S. Roth, DESY
P04	Variable Polarization XUV	5 m UE65 (APPLE II)	0.2 - 3.0 keV	J. V. Maas, DESY
P05	Micro- and nano-tomography	2 m U29	8 - 50 keV	A. Haibel, HZG
P06	Hard X-ray nano probe, imaging	2 m U32	2.4 - 100 keV	G. Falkenberg, DESY
P07	High energy materials science	4 m U19 (IV)	50 - 300 keV	N. Schell, HZG
P08	High resolution diffraction	2 m U29	5.4 - 30 keV	O. Stock, DESY
P09	Resonant scattering/diffraction, HAXPES	2 m U32	2.4 - 50 keV	J. Stempfer, DESY
P10	Coherence	5 m U29	4 - 25 keV	M. Sprung, DESY
P11	Bio imaging and diffraction	2 m U32	3 - 35 keV	A. Menets, DESY
P12	BioSAXS	2 m U29	4 - 20 keV	M. Rößle, EMBL
P13	Macro molecular crystallography I	2 m U29	5 - 35 keV	M. Cianci, EMBL
P14	Macro molecular crystallography II	2 m U29	5 - 35 keV	G. Bourenkov, EMBL

high beta section

low beta section

142 x 5 μm

35 x 6 μm

Main research topics

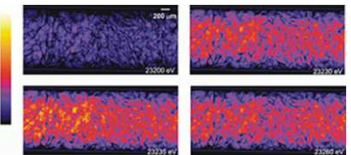
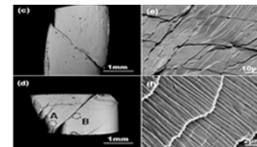
> Surface science

- Thin film technology (polymer-metal)
- Wetting phenomena
- Phase transitions
- NRS (P01), GISAXS (P03), XRR&GID (P08), XPCS (P10)



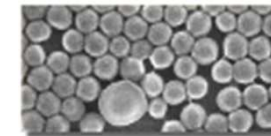
> Materials science

- Catalysis, joining technology
- Magnetism, superconductivity
- Metallic glasses, Battery research
- HEMS (P07), RXRD & HAXSPES (P09), NRS (P01), XRD (P02), IXS (P01,P04)



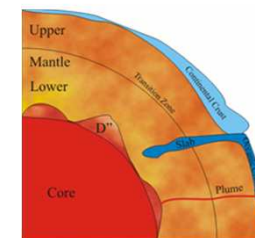
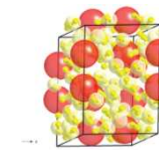
> Soft matter research

- Properties of colloids, glass transition
- NRS (P01), Diffraction (P08&P09), SAXS (P03), XPCS&Rheology (P10)



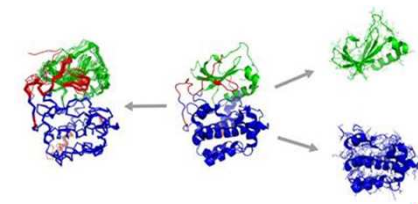
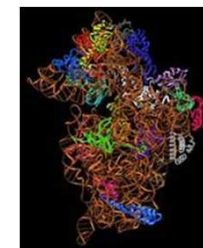
> Earth science

- High pressure research, Geophysics, Mineralogy
- Trace element analysis
- Extreme conditions (P02), IXS&NRS (P01), Diffraction (P08&P09)



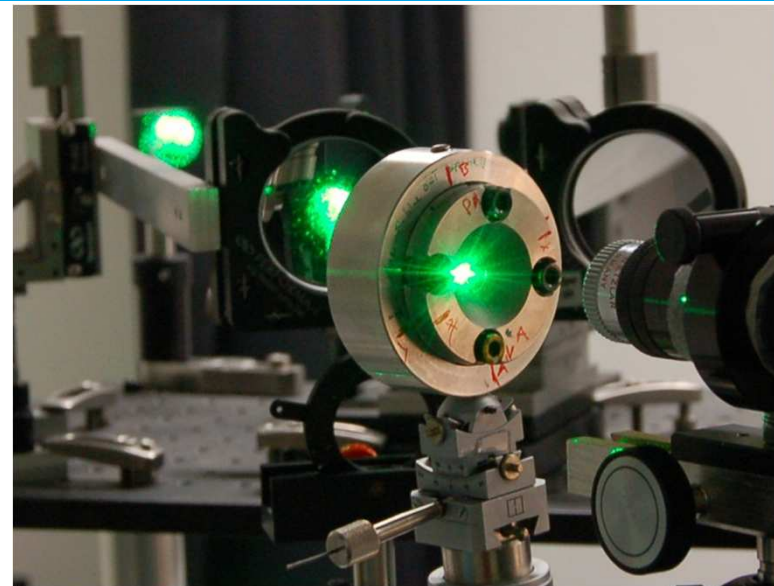
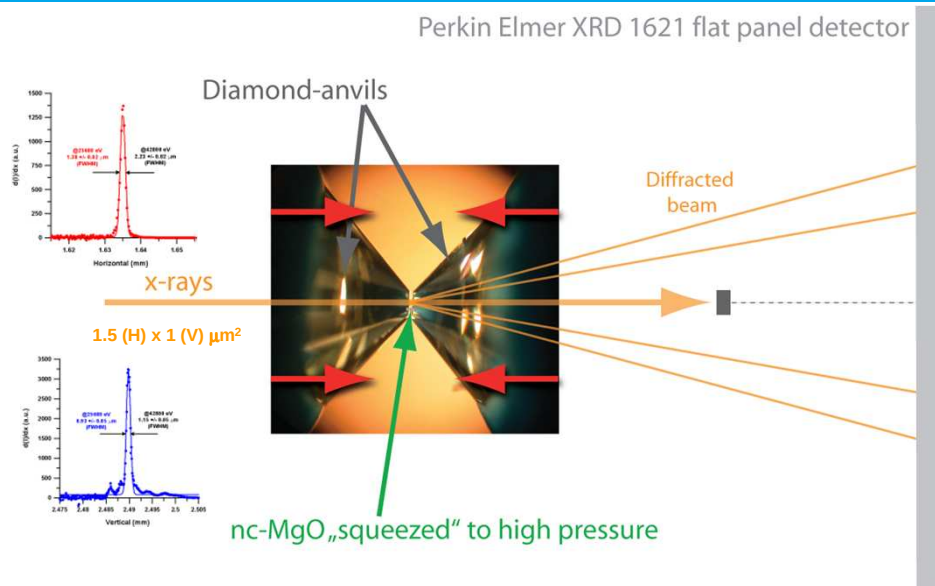
> Life science

- Protein structures
- Drug development
- BIOSAXS (P12), MX (P11,P13, P14), Imaging (P11)

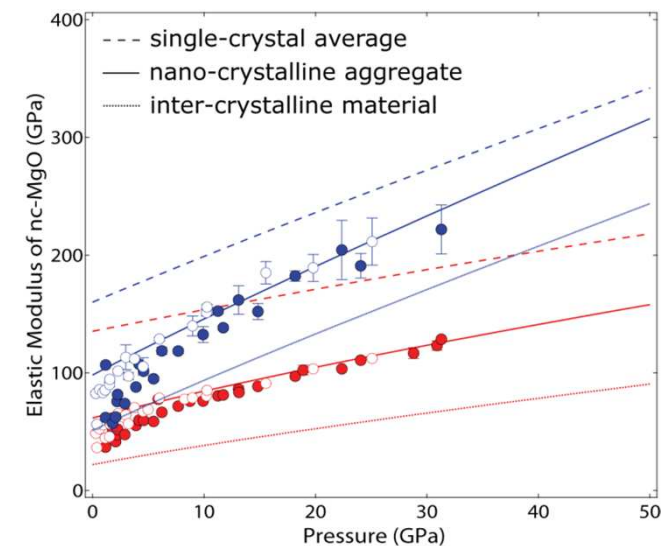
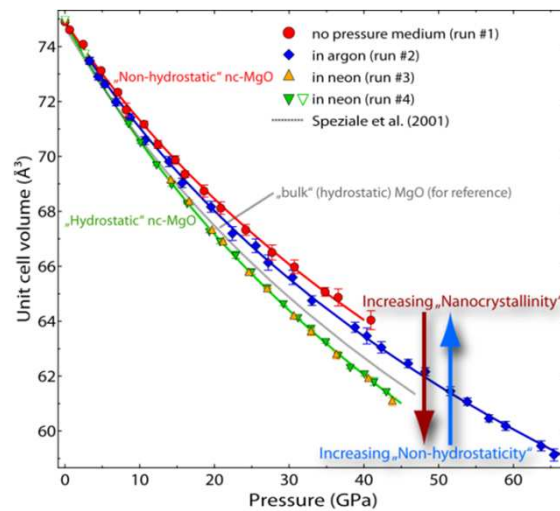
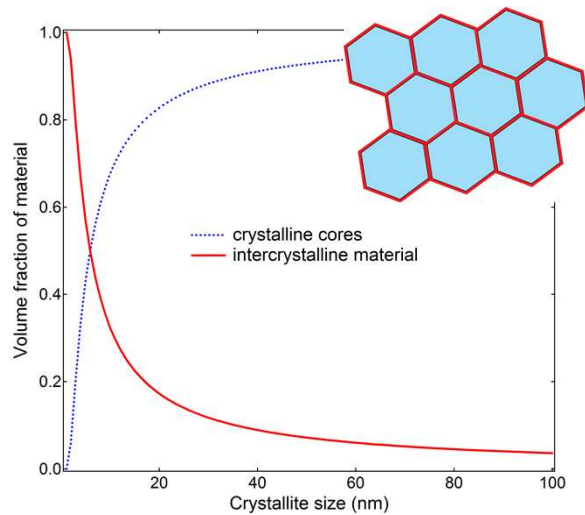


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Hard X-ray Diffraction & Brillouin Spectroscopy at P02

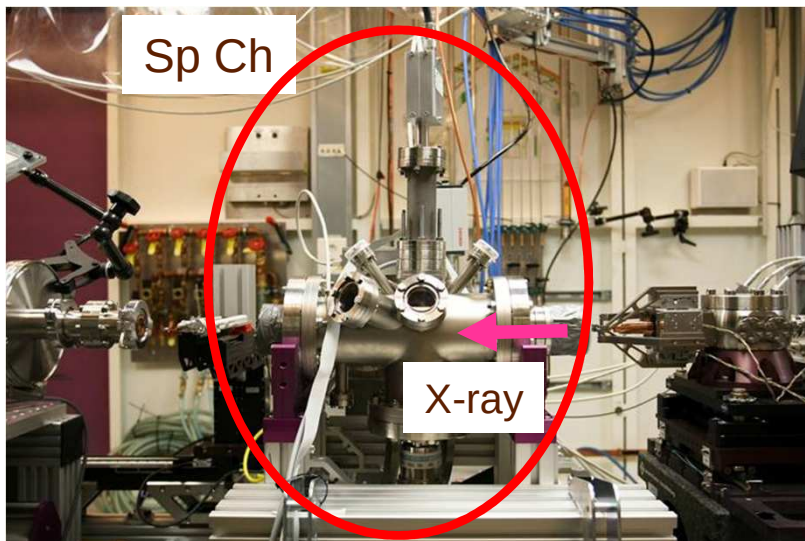
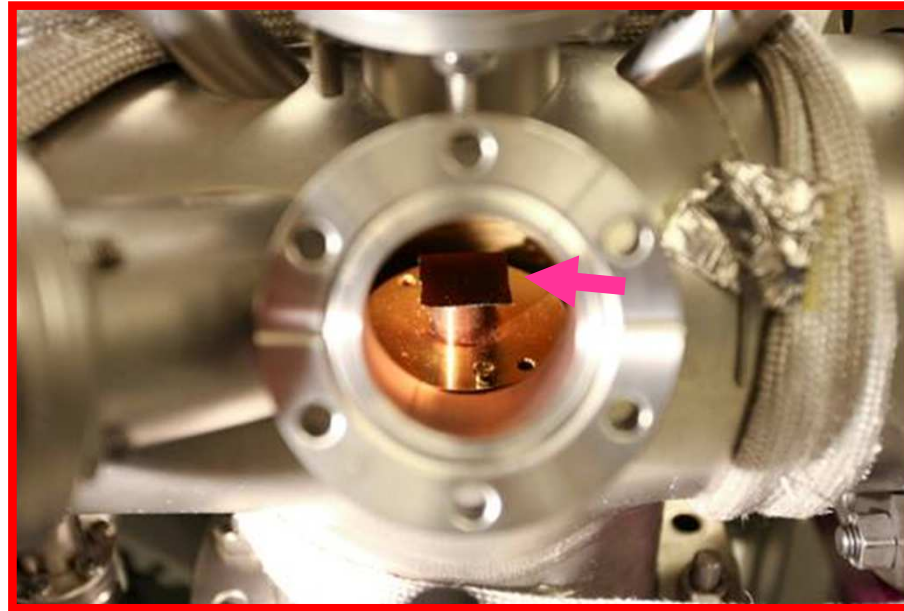
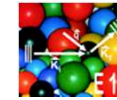


Nanomaterials (MgO) are “softer” than their bulk counterparts



P03: Micro and nano Small-angle scattering

- In-situ sputter deposition & thin films
- Nano-structured polymer thin film
- Metal sputter deposition
 - Rate: $(0.23 \pm 0.03) \text{ nm/s}$
 - $d_{\text{final}} = 35 \text{ nm}$
- Beam size: $38 \times 19 \mu\text{m}^2$ (SAXS)
- Temporal resolution 15 ms



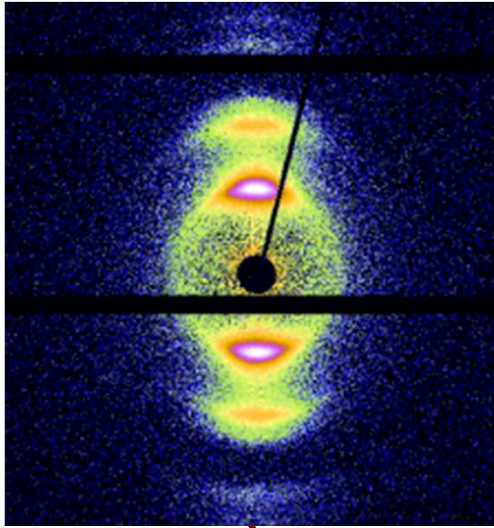
DESY: Roth, Schwartzkopf, Buffet, Abul-Kashem, Herzog, Gehrke, Perlich, Röhlberger, Rothkirch, Schlage, Benecke (*DESY/MPI Golm*)

TU München: Müller-Buschbaum, Metwalli, Körstgens

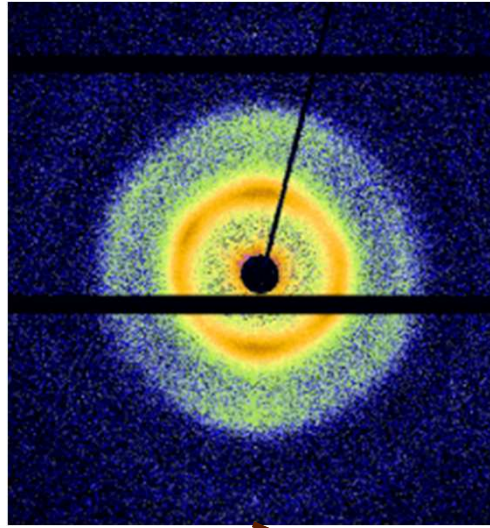


Shear-induced orientation of cylindrical micelles: model system to understand injection molding

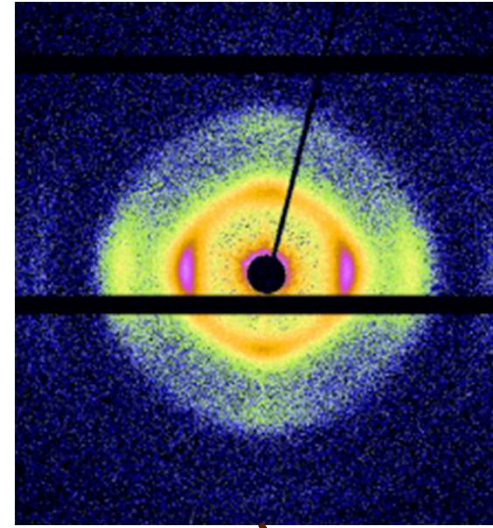
converging flow:
parallel



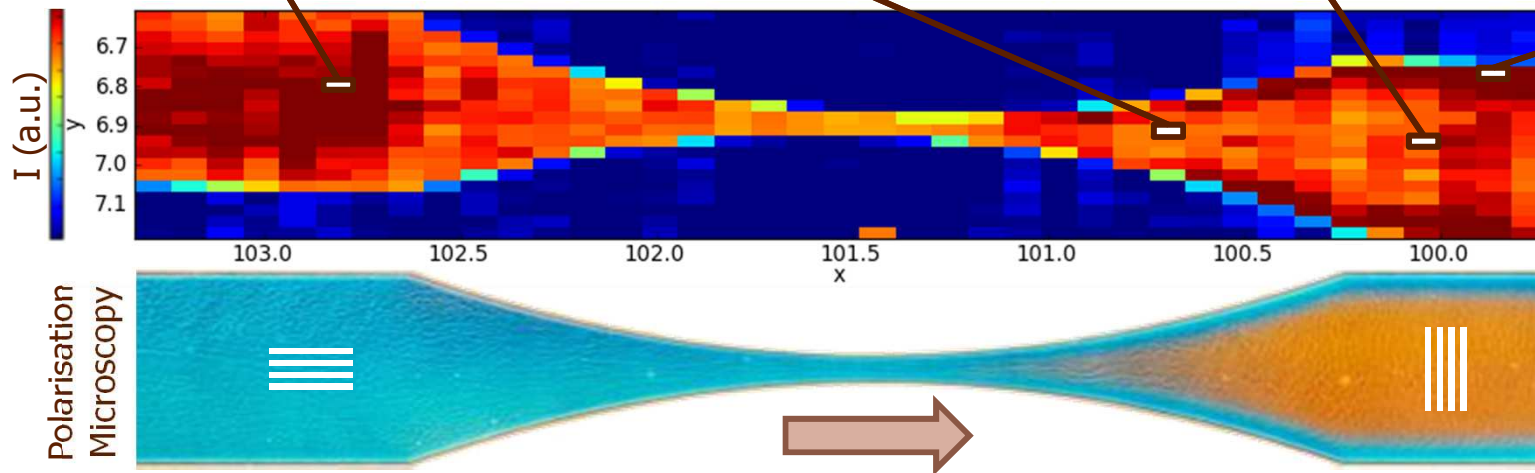
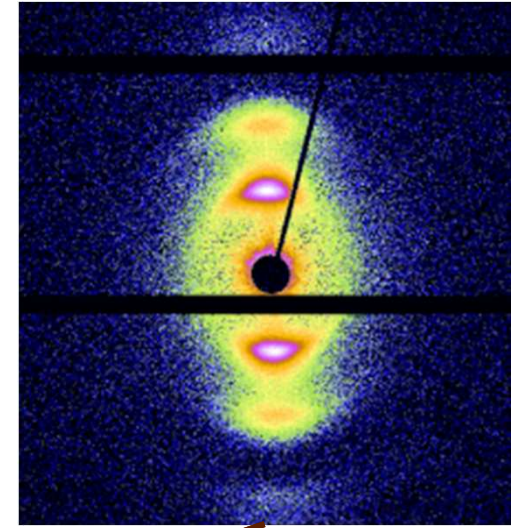
end of tapering:
transition



diverging flow
perpendicular



wall shear:
parallel



500 μm x 67 μm
(**7.5:1** ratio)
flow 300 $\mu\text{L/h}$
PB-PEO cyl. mic.
(30% w/w, water)
Blue: = to X
Yellow: = to Y

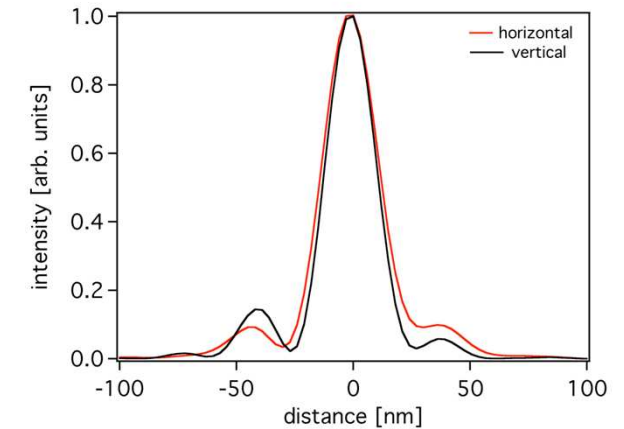
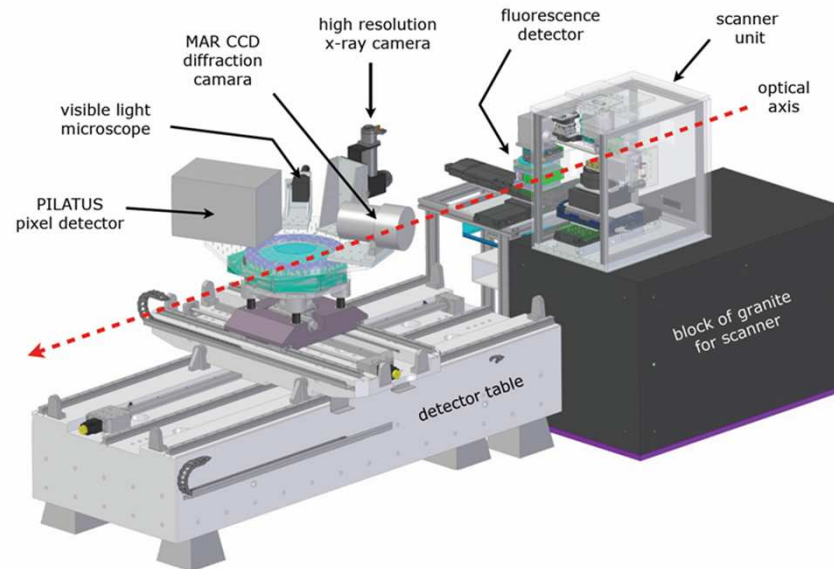
M. Trebbin, G. Benecke, S. With, S.V. Roth, S. Förster et al. **2012**, *in preparation*.

Hermann Franz | DESY/KEK meeting 20.3.2012 | Page 8

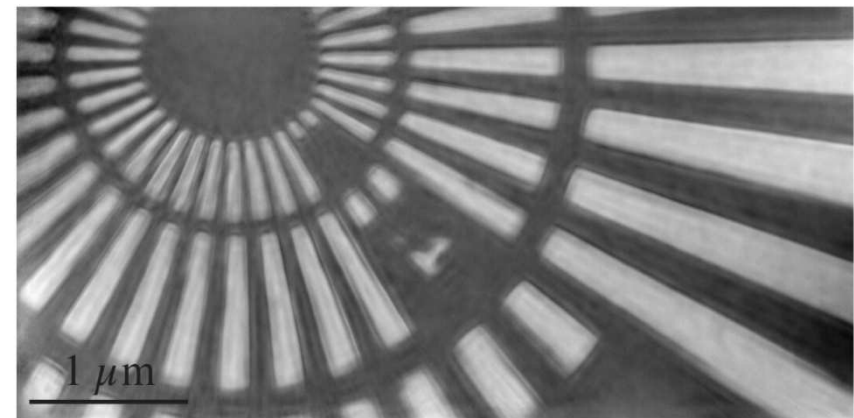
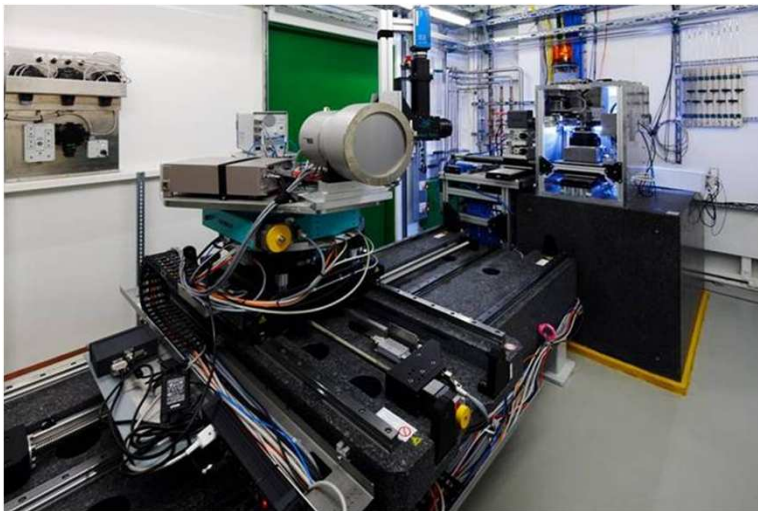


P06: hard X-ray nano- and micro-probe

- Scanning fluorescence microscopy
- Nano-diffraction
- Ptychography
- Tomography
- 5 – 50 keV
- NFLs
- KB system
- FZP



Zone plate focus:
(25 nm)²

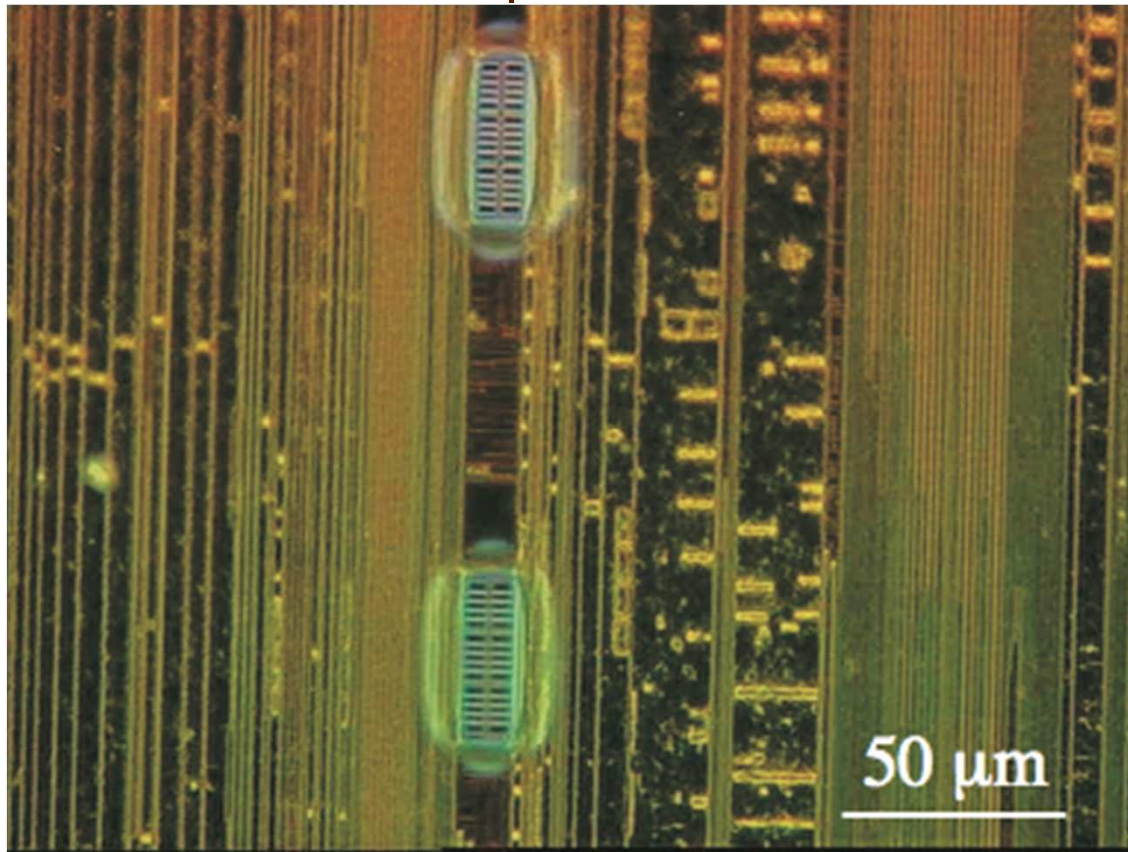


Ch. Schroer et al (TU Dresden), Ch. David (PSI)

P06 Example: Qimonda storage chip

Ptychographic Imaging of Qimonda storage chip

Shared sense amplifier



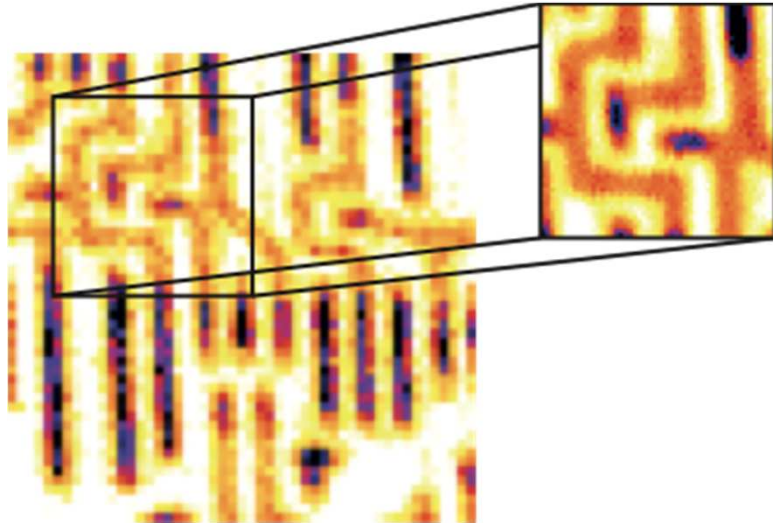
No sample preparation:
passivated chip
(thickness: >100 μm)

512 Mb DDR2 RAM
80 nm technology

A. Schropp, et al., J. Microscopy, 241(1), 9 (2011)

P06 Example: Qimonda storage chip

W-fluorescence map



- ▶ E= 15.25 keV
- ▶ 200nm wide conducting paths

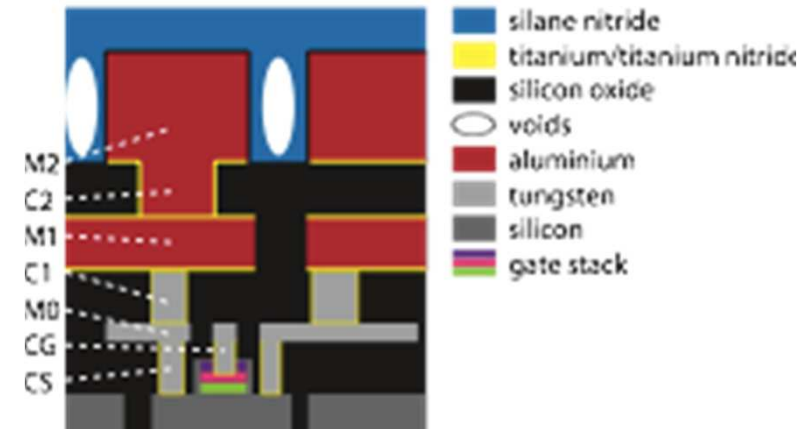
contrast:

- W in SiO₂
- voids

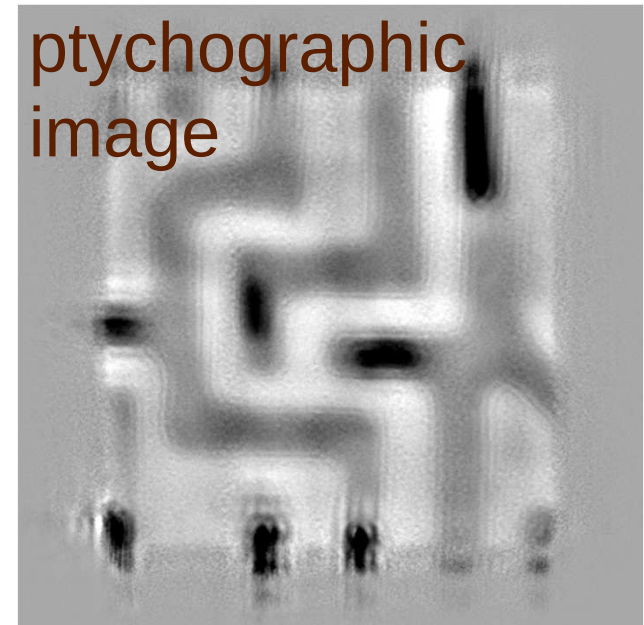
quantitative information:

- thickness via: 90 nm
- two stacked plugs: 570 nm

vertical stacking scheme



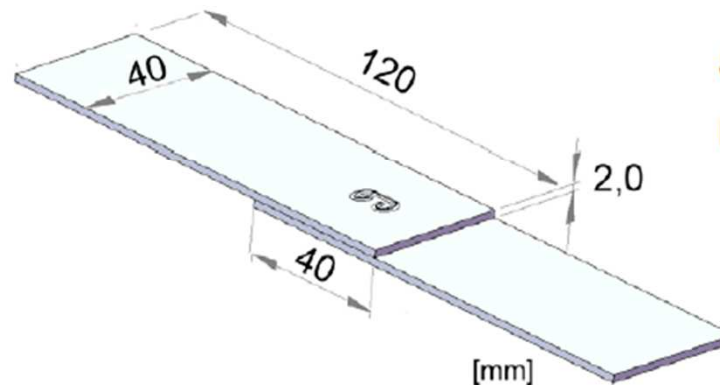
ptychographic image



P07: residual stress in laser spot welds



Residual stress distribution in laser beam welded overlap spot welds (e.g. steel sheets of car bodies) influences fatigue properties.

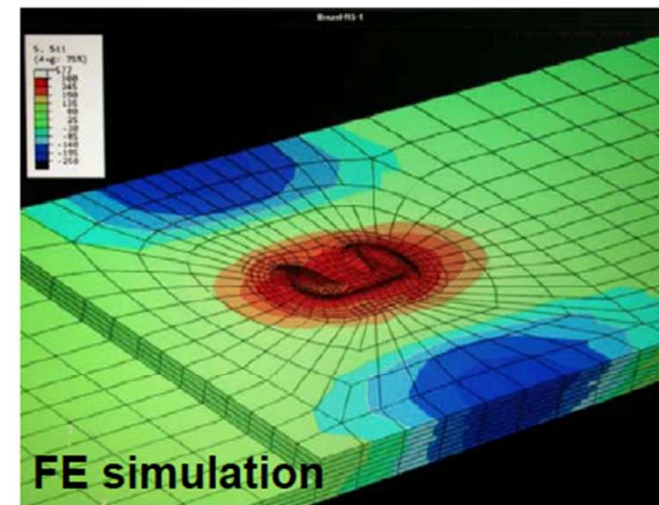
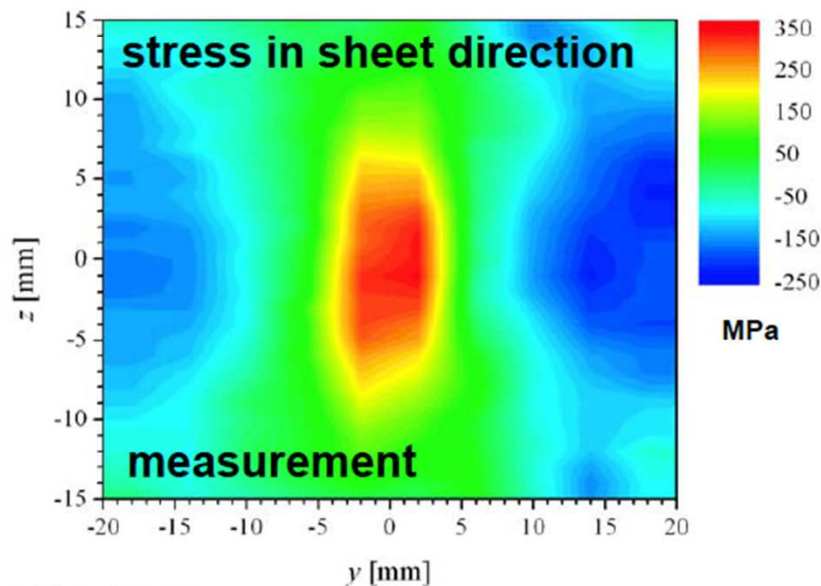


aim: **evaluation of FE simulation**

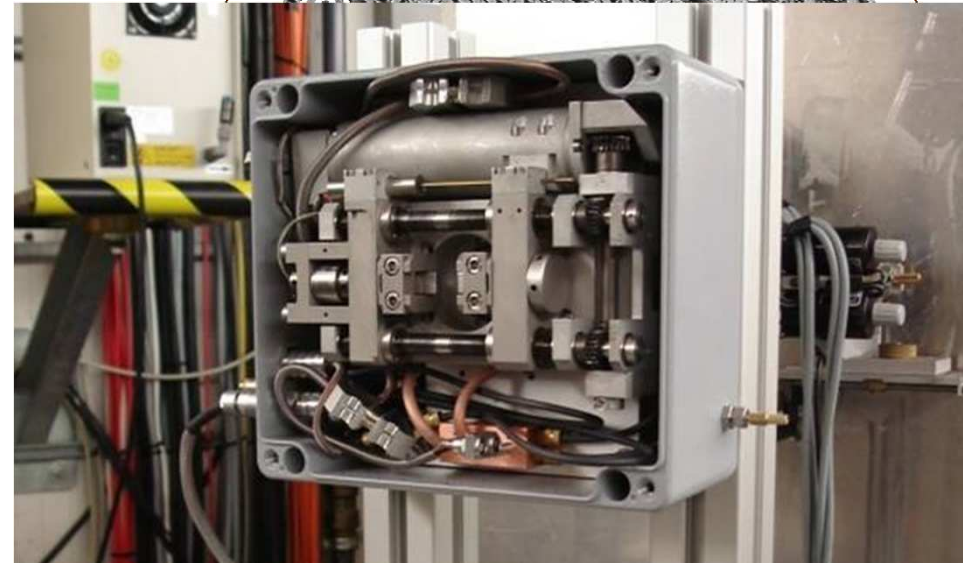
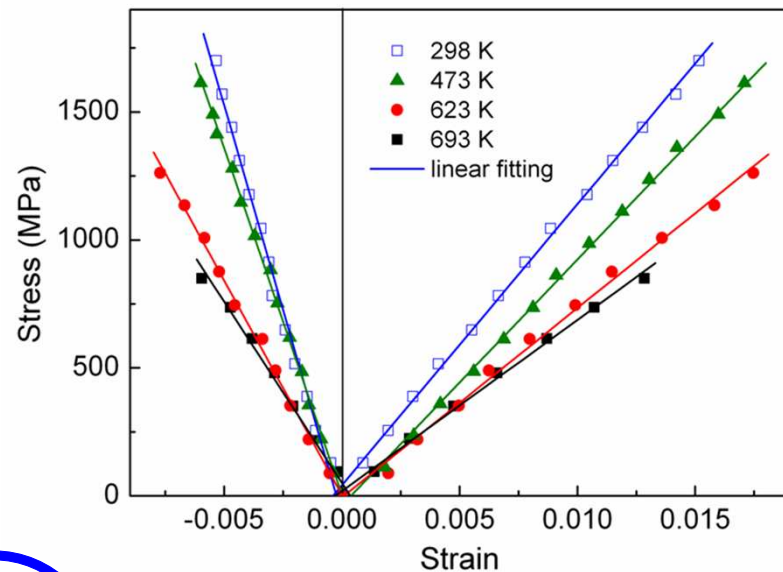
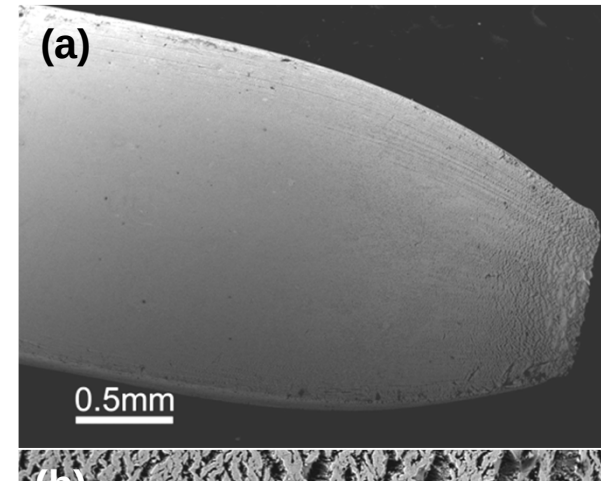
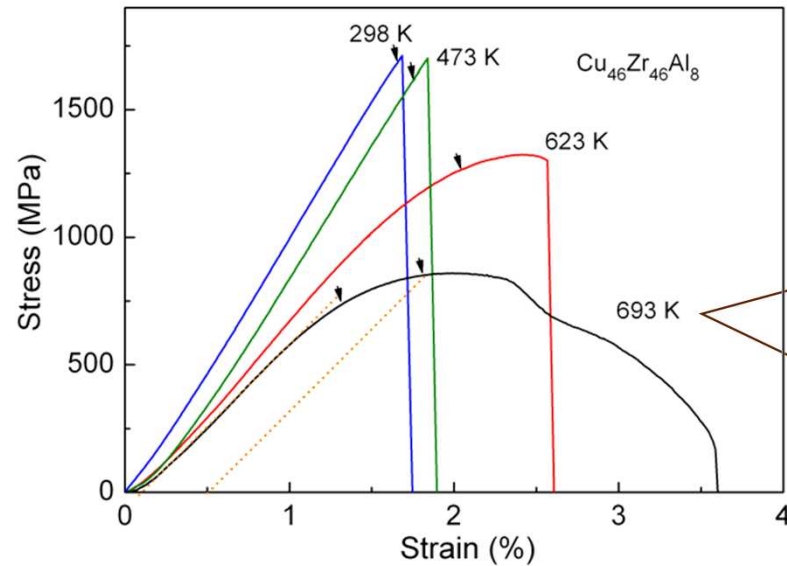
result: **good agreement**

53.6 keV, 1 x 1 mm², MAR345

tensile stresses
in the weld,
balanced by
compressive
stresses near
the sample
edges



P07: Deformation of CuZrAl bulk metallic glass

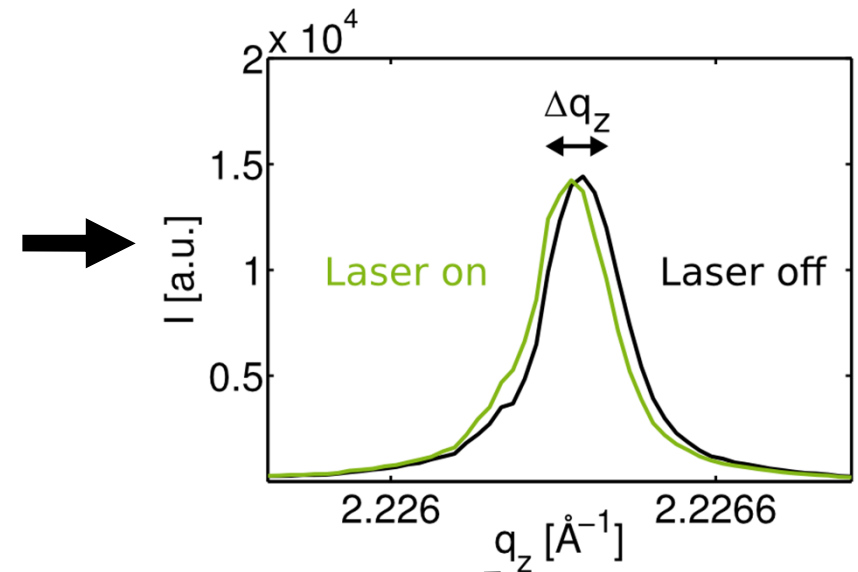
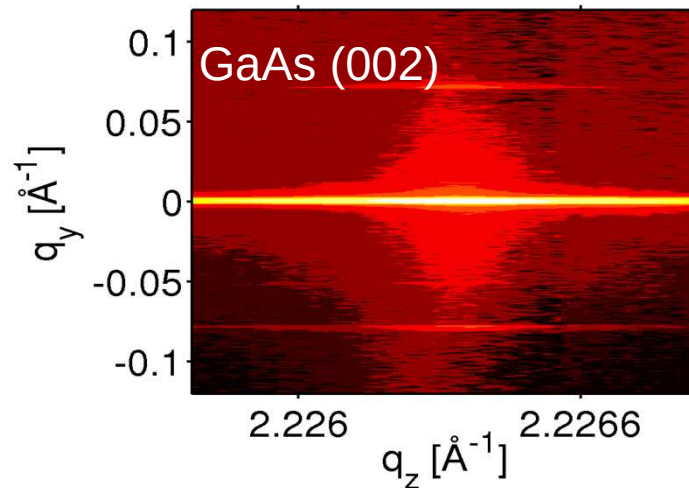
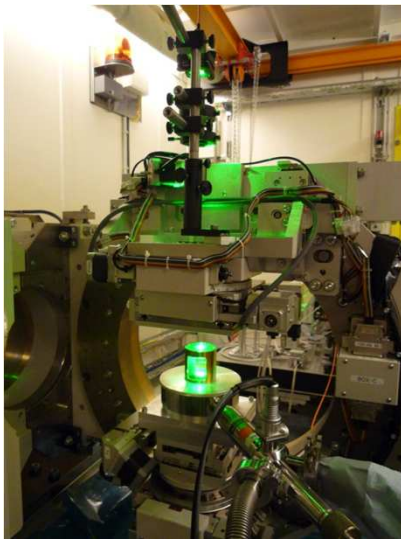


X.D. Wang et al. 2010



P08: Laser-induced anisotropic strain in optically excited InAs/GaAs quantum dots

S. Tiemeyer, M. Bombeck, M. Paulus, C. Sternemann, M. Bayer, M. Tolan



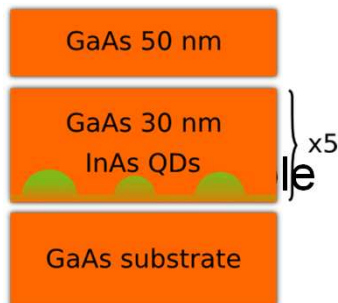
Known: Modification of density of state due to confinement (quantum dot)

Unknown: Modification due to additional heavy excitement with electrical field by LASER

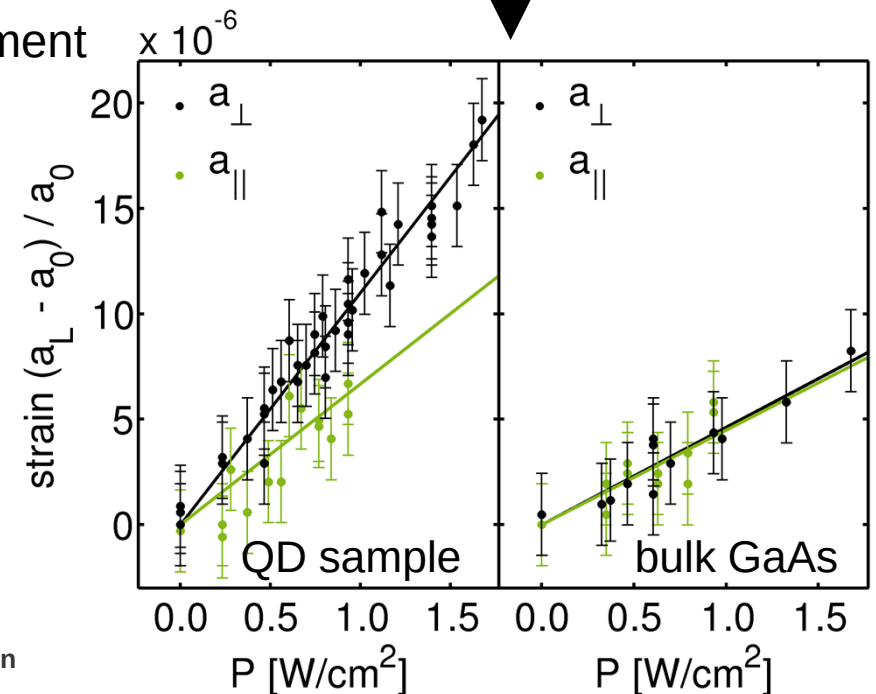
High-resolution x-ray diffraction at P08, PETRA III

Nd:YAG laser 532 nm, max. 1 W

helium flow cryostat T = 100 K



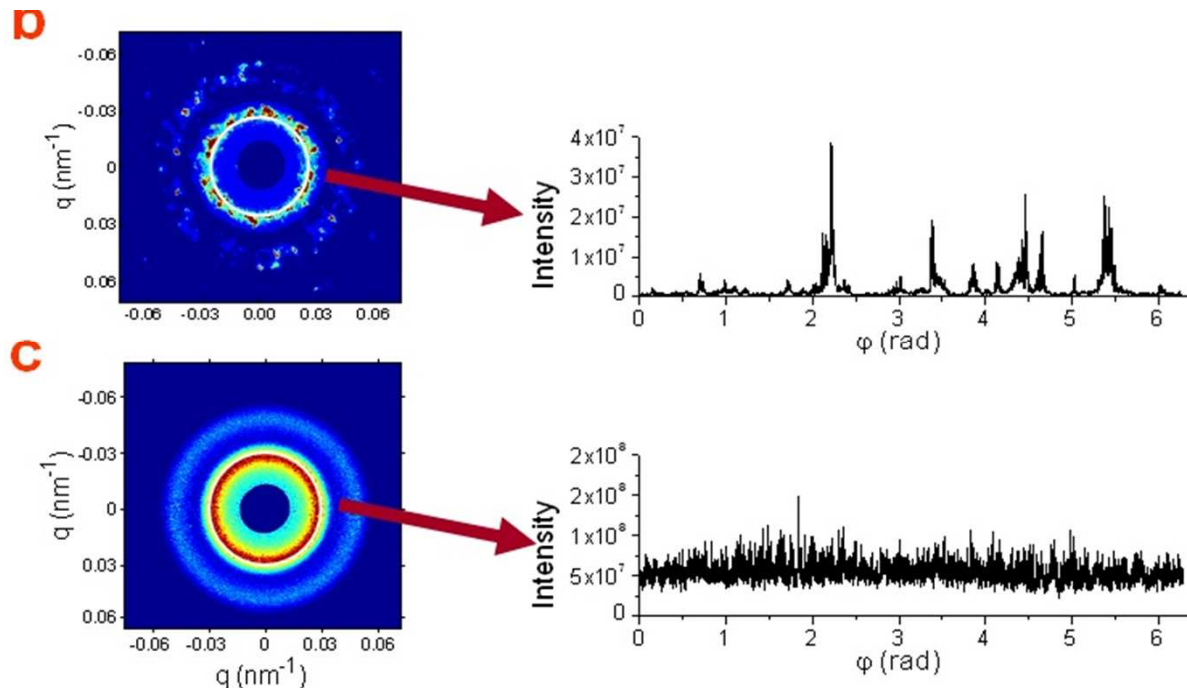
Upcoming experiment will clarify the origin of the anisotropic strain



P10: Coherence beamline

- **Coherent flux** is given by the brilliance
- Coherence **length** by the source size and the length of the beamline
- Dedicated set-up for XPCS and coherent imaging
- Vertical SAXS set-up with Rheometer in plate-plate geometry

Glass transition of hard sphere colloidal systems



P10 Coherence beamline

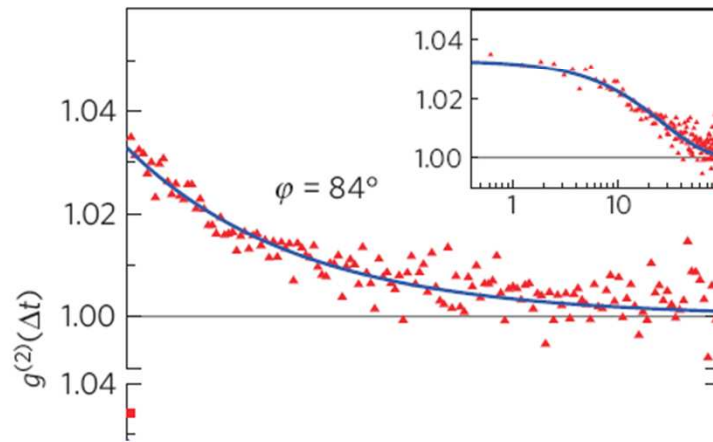
nature
materials

LETTERS

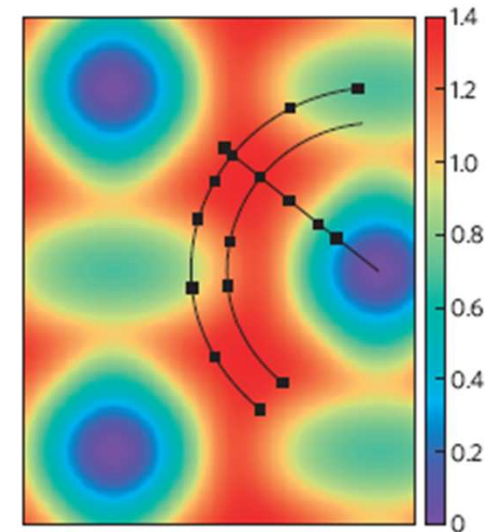
PUBLISHED ONLINE: 26 JULY 2009 | DOI:10.1038/NMAT2506

Atomic diffusion studied with coherent X-rays

Michael Leitner^{1*}, Bogdan Sepiol¹, Lorenz-Mathias Stadler², Bastian Pfau³ and Gero Vogl¹



Data
from
ESRF



$$g^{(2)}(\mathbf{q}, \Delta t) = \frac{\langle I(\mathbf{q}, \cdot) I(\mathbf{q}, \cdot + \Delta t) \rangle}{\langle I(\mathbf{q}, \cdot) \rangle^2}$$

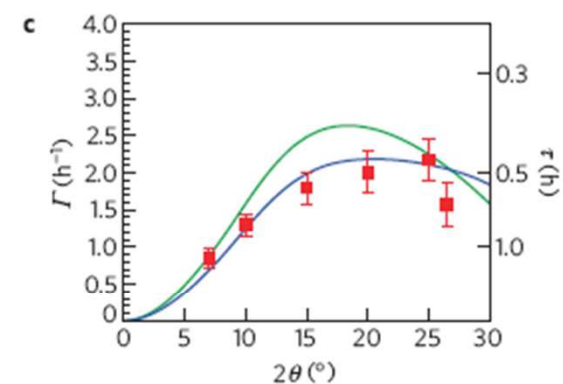
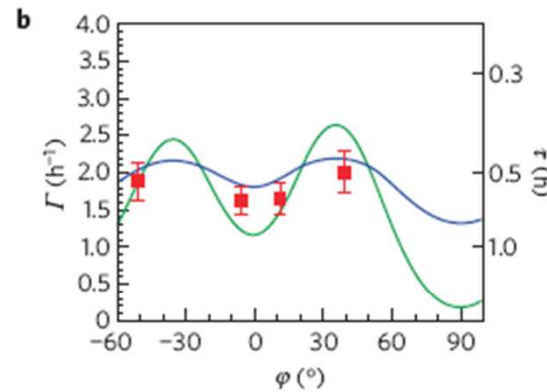
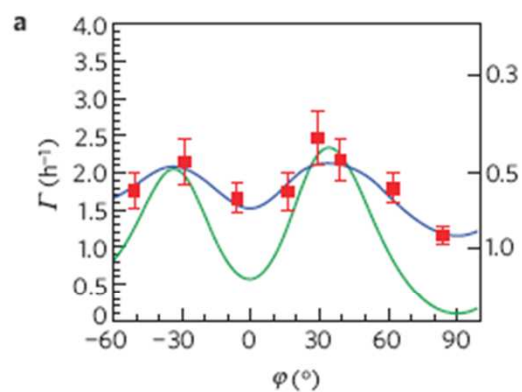


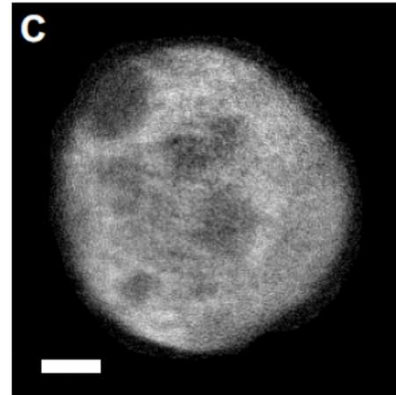
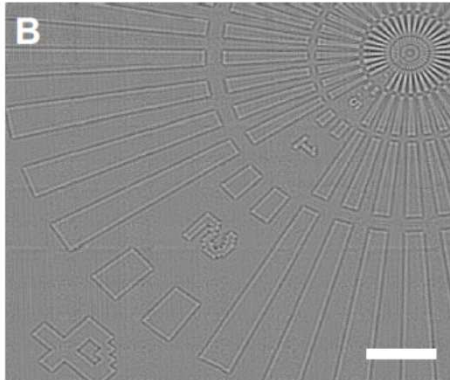
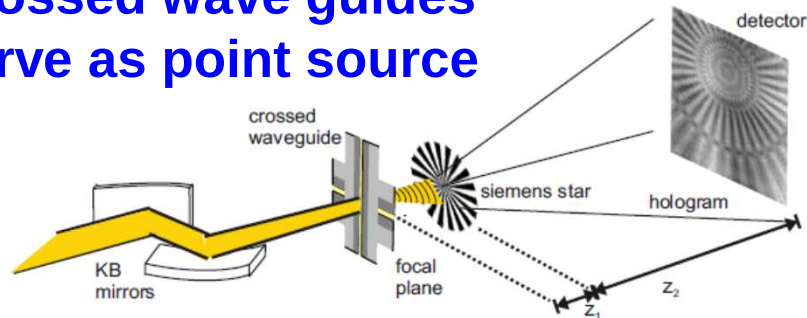
Figure 1
in ref.

fixed scattering angle $2\theta = 25^\circ$ and varying azimuthal angles $\varphi = 84^\circ$ and $\varphi = 39^\circ$ (for definition of the angles see Fig. 3) with fitted exponential decays. Although the modulus of the scattering vector is the same, the fitted correlation times (52 ± 5 min for $\varphi = 84^\circ$ and 28 ± 4 min for $\varphi = 39^\circ$, respectively) differ by a factor of two. The inset shows the measurement with $\varphi = 84^\circ$ plotted on a logarithmic timescale.

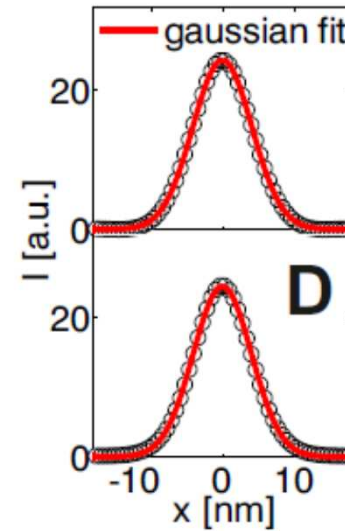
$\tau_0(T)$: time between (successful) jumps
denominator: contains jump mechanism

Imaging with a 10 nm beam at P10

Crossed wave guides
serve as point source



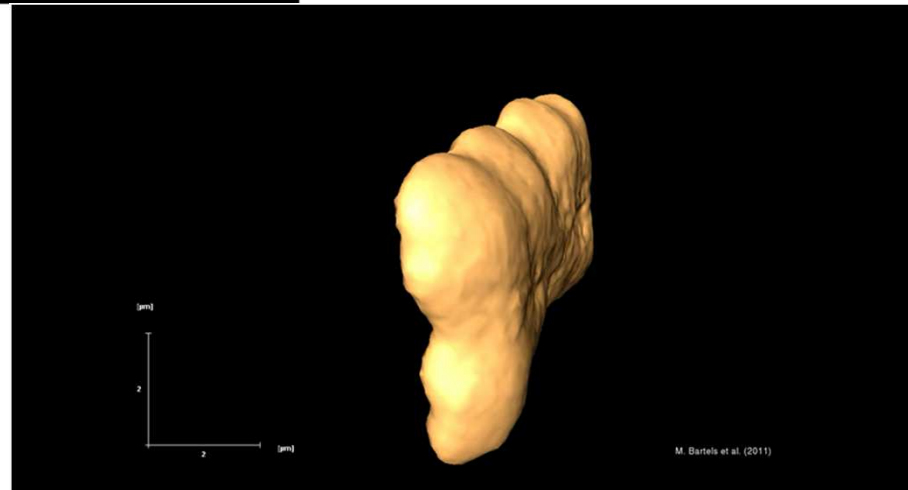
Beam profile
reconstruction



M. Bartels, T. Salditt, M.
Sprung et al: Göttingen
end-station

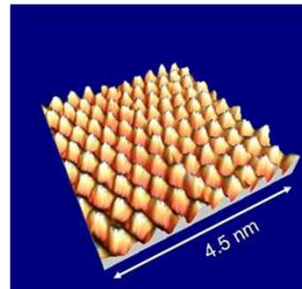
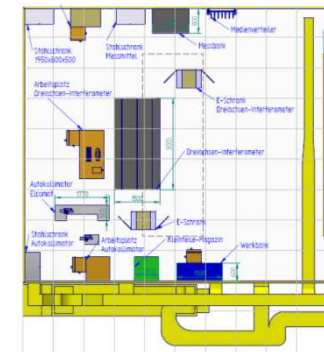
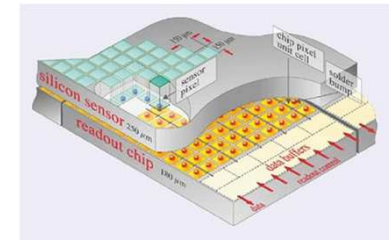
*Reconstructed image of
d.radiodurans using the
10nm beam as point
source.*

Resolution ca 70 nm



Auxiliary labs

- > **Detector pool**
- > **Sample environment pool**
- > **Chemistry lab**
- > **Clean-room facilities**
- > **Metrology lab**
- > **AFM**
- > **High pressure lab**
- > **Laser labs (class 4)**
- > **Wet-labs (with EMBL)**



New beamlines in hall North 2014+

- o **P61 (damping wigglers)**

- P61.1 - fixed-energy branch for PDF structure analysis**

- P61.2 - high-energy engineering materials science station
(Helmholtz-Center Geesthacht, HZG)
- extreme conditions station (large volume press)**

- o **P62 (mini-undulator)**

- X-ray micro fluorescence spectroscopy**

- o **P63 (mini-undulator)**

- small-angle X-ray scattering**

- o **P64 (2m undulator)**

- X-ray absorption spectroscopy: time-resolved and bio-XAFS**

- o **P65 (mini-undulator)**

- X-ray absorption spectroscopy**



New beamlines in hall East 2014+

- o **P21 (4m undulator & opt. wiggler)**
 - High-energy X-ray material science
(Swedish-German collaboration)
- o **P22 (2m undulator)**
 - "nano spectroscopy" working title
(Indian-German collaboration)
- o **P23 (2m undulator)**
 - "nano diffraction" working title
(Russian-German collaboration)
- o **P24 (mini-undulator)**
 - chemical crystallography
- o **P25 (mini-undulator)**
 - P25.1** - education, training, test beamline
 - P25.2** - fixed energy side station



Summary

- > PETRA has reached all design goals within the first year of operation**
 - 1.0 nm rad emittance and reliable top-up operation
 - Stability in the range of nm and nrad
 - Close to perfect undulators, no mutual influence
- > Measured beam parameters are largely consistent with simulated values**
 - High quality source and optics
 - Very efficient focussing
 - We are well ahead of our roadmap to achieve small, brilliant X-ray beams
- > All PETRA beamlines open for user applications – regular or friendly users**



**Thank you for your
attention**

