Sicherheit in Technik und Chemie

BAM
Bundesanstalt für Materialforschung und -prüfung

01.02.2019

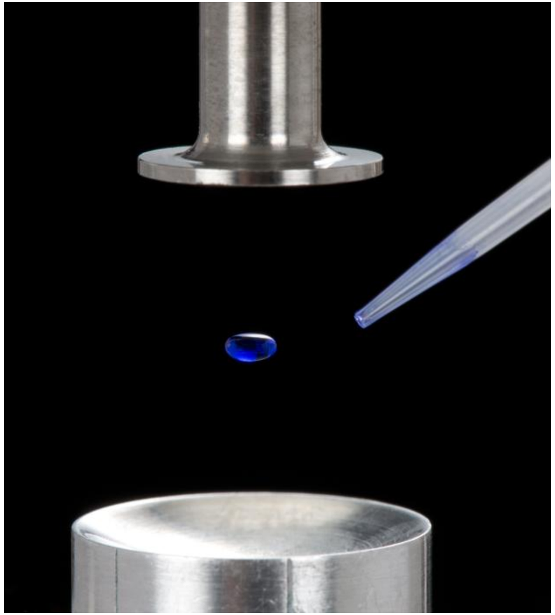
HARD X-RAY SPECTROSCOPY AND IMAGING AT THE BAM*LINE* AND µSPOT BEAMLINES @BESSY II (BERLIN, GERMANY)

Dr. Ana Guilherme Buzanich

Structural Analysis division / Dept. Analytical Chemistry

www.bam.de

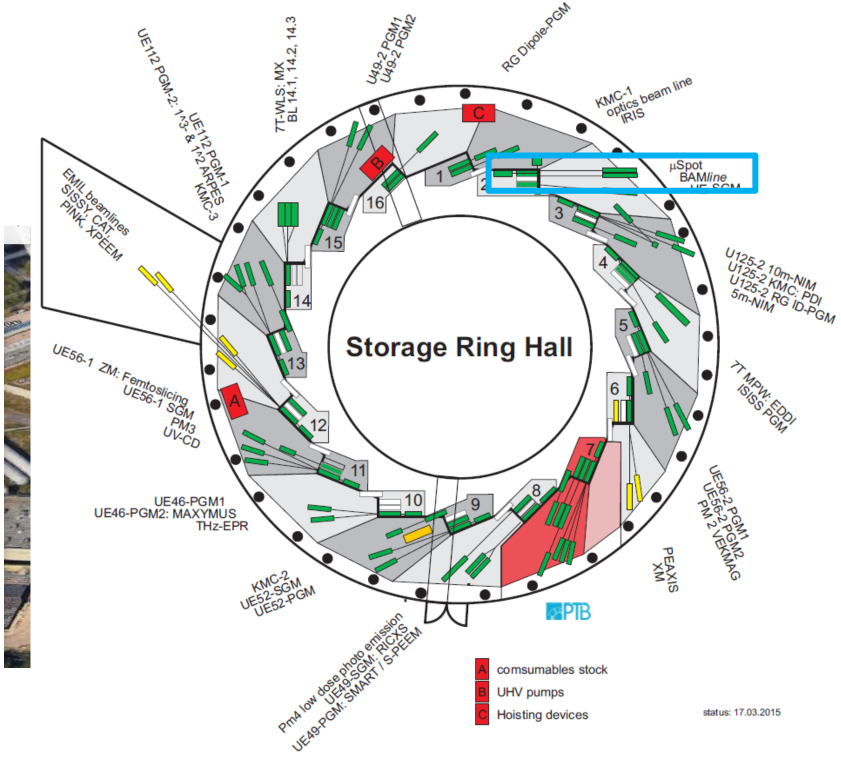
Division Structural Analysis – Department of Analytical Chemistry

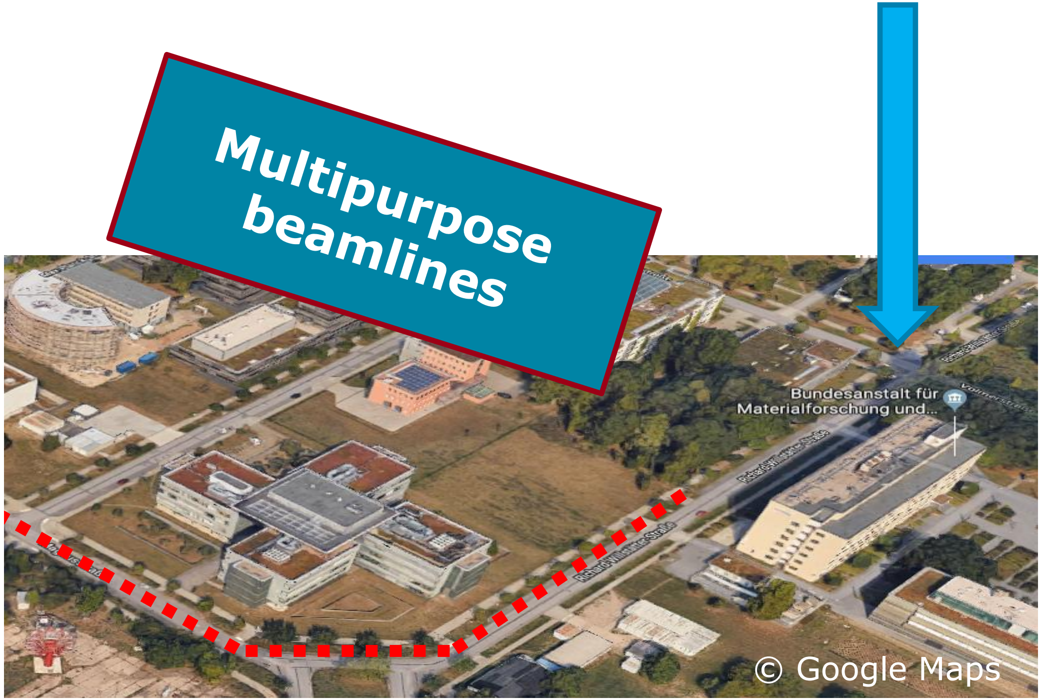


Fields of expertise

- Quantitative analysis of X-ray powder diffraction
- Investigation of materials at different length scales
- Identification and refinement of crystalline structures
- Liquid and solid state NMR
- Synchrotron-based spectroscopies (XRF, XAS) & diffraction (XRD, SAXS, WAXS)
- Combination of several analytical methods: In Situ
- Surface and pore analysis of porous solids and nanomaterials
- Characterization of polymers (size, weight, chemical heterogeneity)

BAM ⇔ BESSY-II close connection



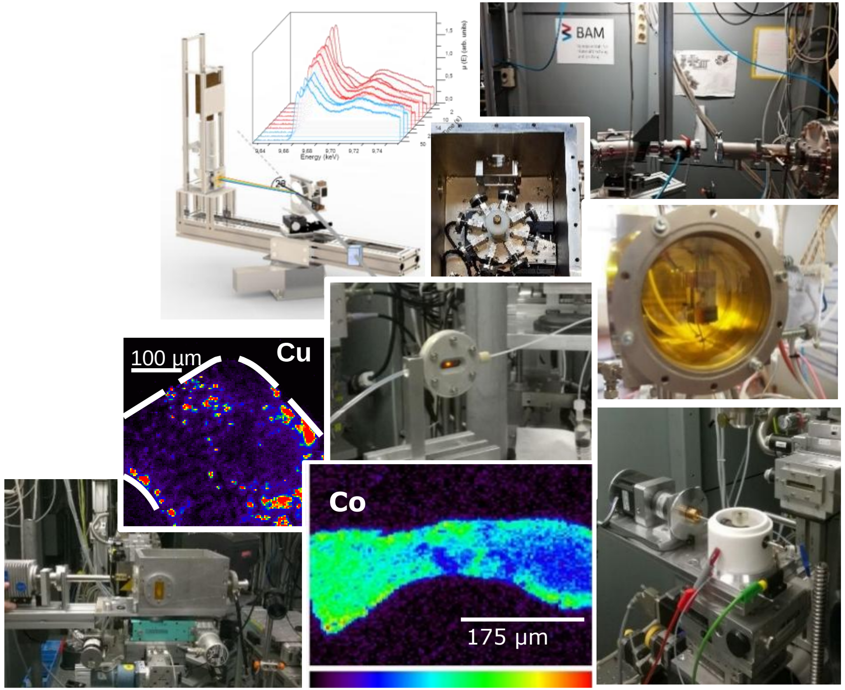


Outline

Analytical Methods

Applications
&
Collaboration Partners

Future Perspectives



BAM

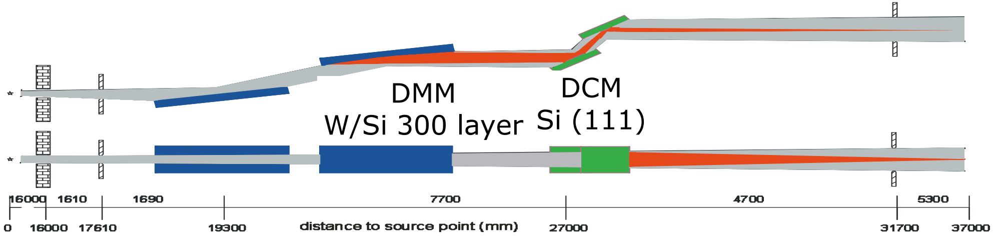
BAM*line* & µSpot

Analytical Methods

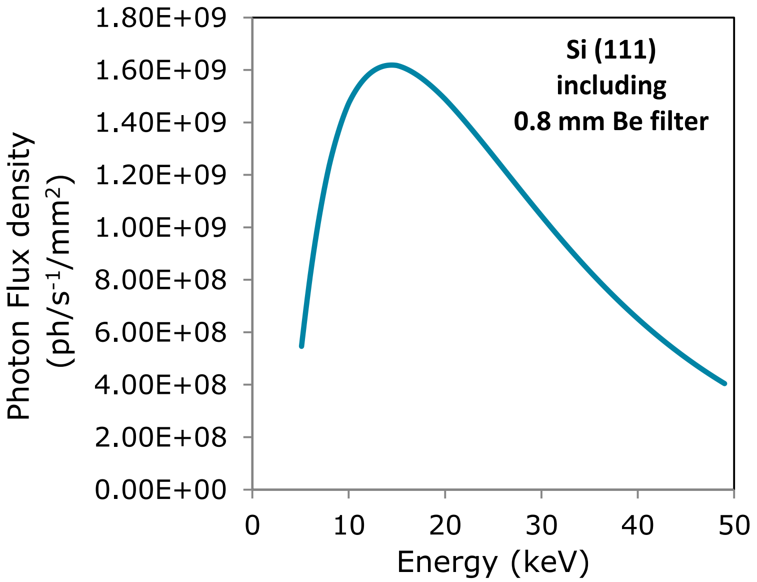
Applications & Collaboration Partners

Future Perspectives

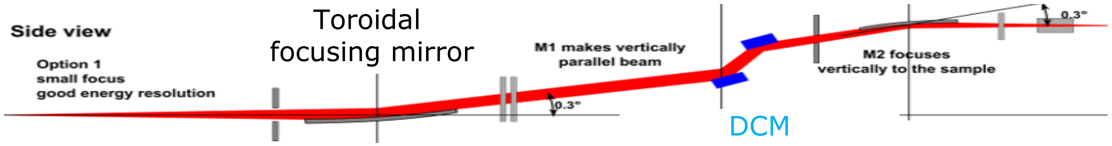
Analytical methods – BAM*line*



Analytical Methods		Information
DCM 7-30 keV (10^9 ph/s)	XAS - XANES - EXAFS	Chemical speciation: -Oxidation state - Local coordination geometry
DMM 4-50 keV (10^{11} ph/s)	XRF -(µ-)EDXRF - WDXRF	Elemental composition

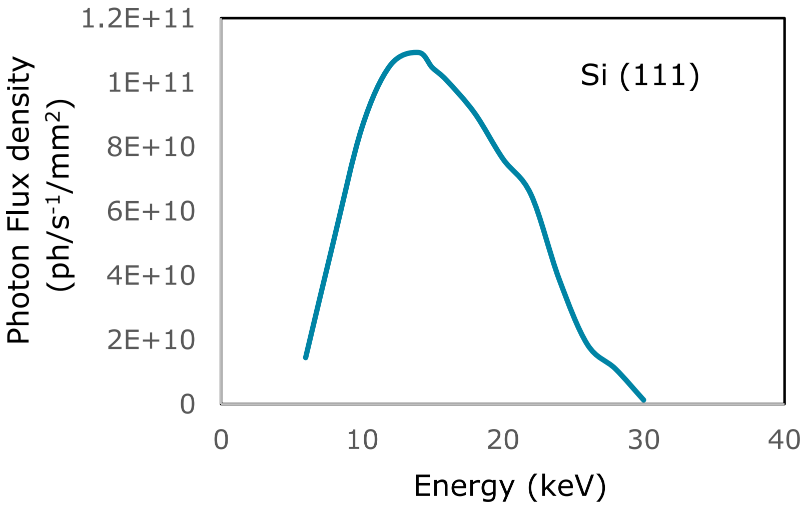


Analytical methods – µSpot beamline

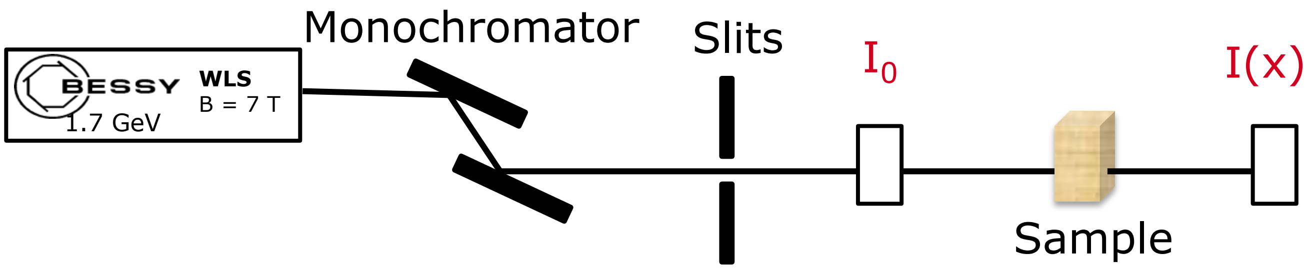


Analytical Methods		Information
DCM 5-20 keV (10^{11} p/s)	XAS	Chemical speciation - Oxidation state
	XRD	Crystal structure
	SAXS/WAXS	Size, shape, polydispersity
DMM 5-20 keV (10^{13} p/s)	XRF	Elemental composition
	Raman spectroscopy	Molecular structure

Max. Spotsize: 300 µm x 2 mm

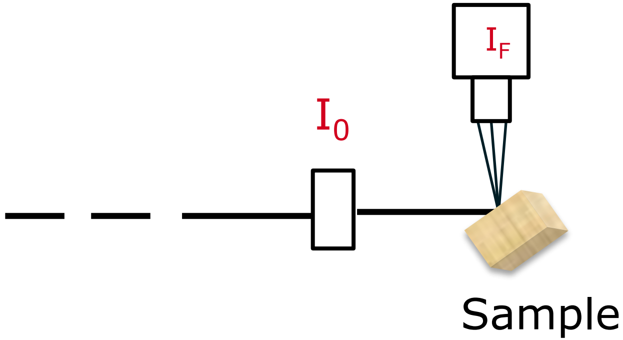


XAS *modus operandi* – standard



Absorption / Transmission

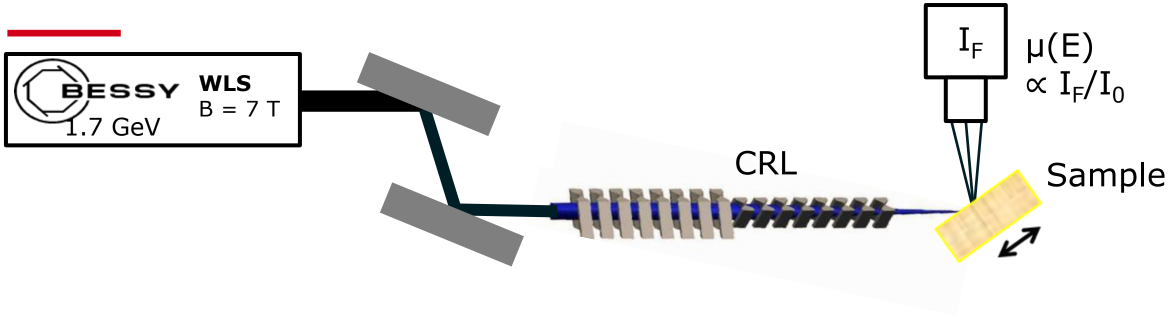
$$I(x) = I_0 e^{-\mu(E)x}$$



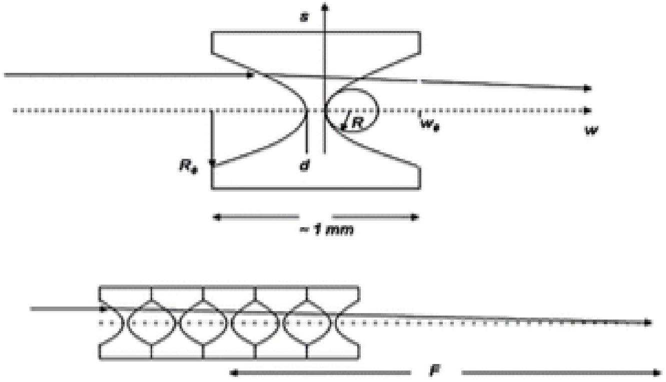
Fluorescence

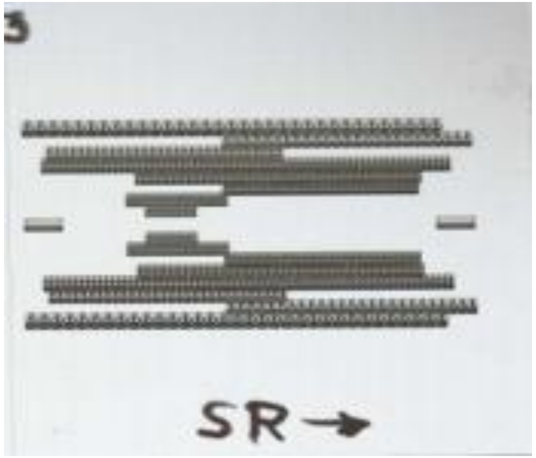
$$\mu(E) \propto I_F/I_0$$

XAS *modus operandi* – micro-XAS



Compound refractive lens (CRL)

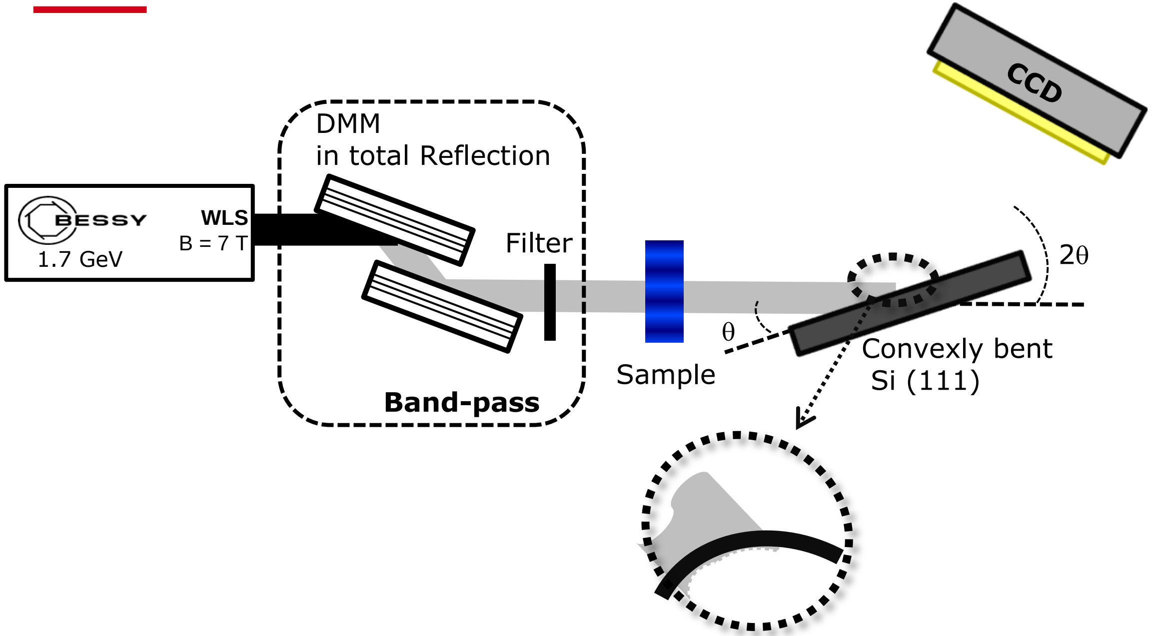




- ✓ narrow bandwidth (~100 eV)
- ✓ workable focal lengths (350 mm)
- ✓ focusing regions from 100 nm

G. Buzanich et al. **J. Anal. At. Spectrom.** (2012) 27, 1803.

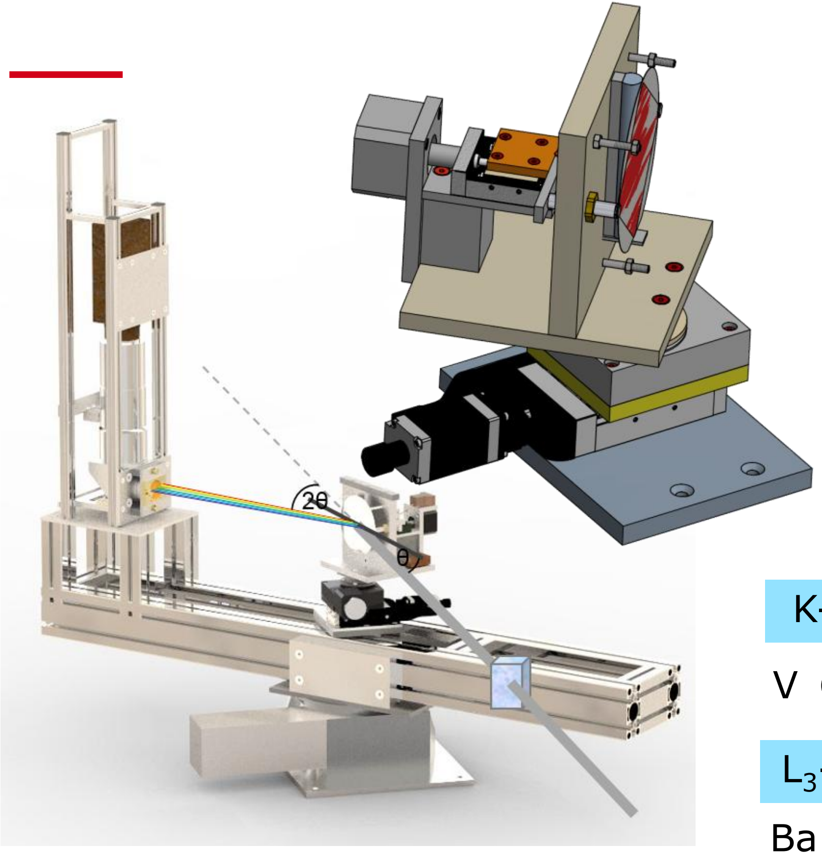
XAS *modus operandi* – dispersive XAS (S^2XAFS)

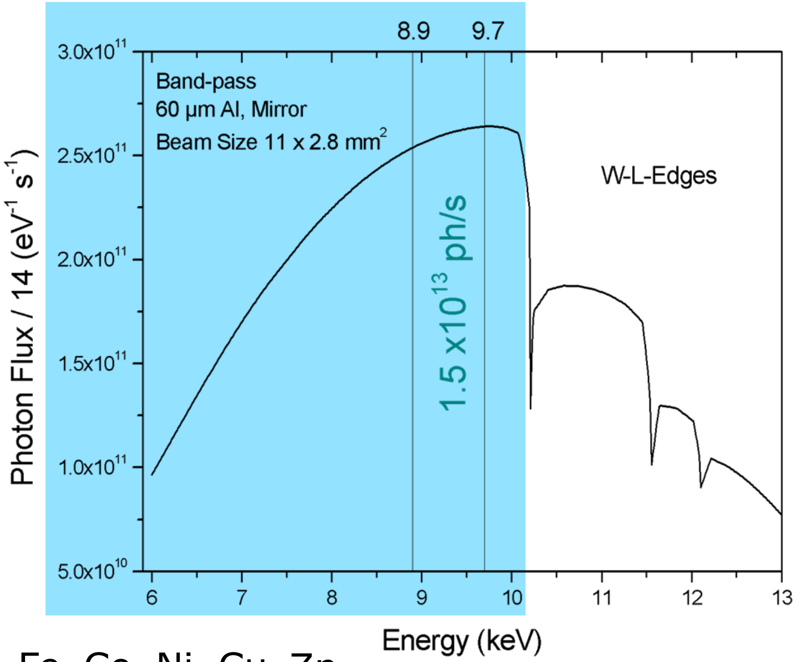


- ✓ Simple
- ✓ Scanningless
- ✓ Time & spatial resolution
- ✓ no scattering contribution
- ✓ Time-resolution ≥ 1 s
- ✓ Spatial-resolution: ≥ 20 μm
- ✓ Beam size up to 20 x 8 mm^2

A. Guilherme Buzanich, M. Radtke, U. Reinholz, H. Riesemeier, F. Emmerling.
J. Synchrotron Rad. (2016), 23, 769-776.

XAS *modus operandi* – dispersive XAS (S^2XAFS)





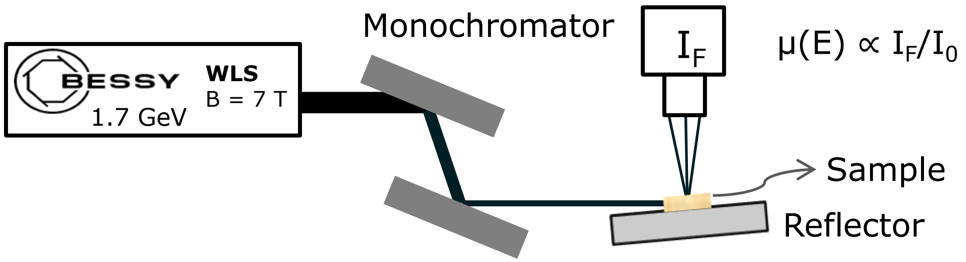
K-lines

V Cr Mn Fe Co Ni Cu Zn

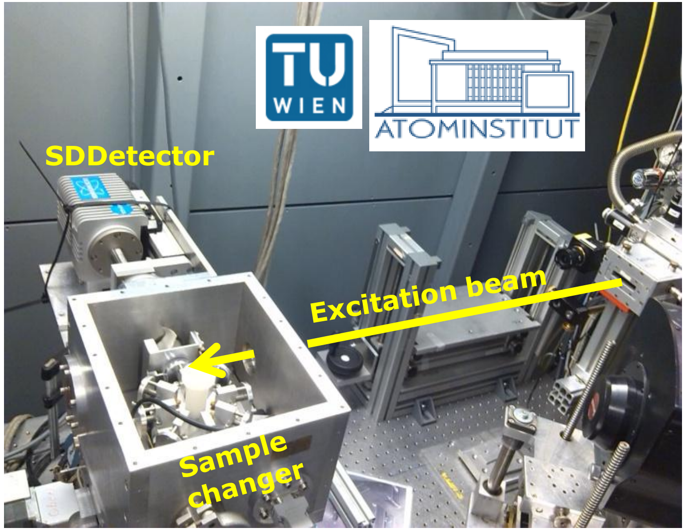
L_3-lines

Ba La Ce Pr Nd Pm Sm Eu Gd Tb

XAS *modus operandi* – total reflection XAS (TXRF-XAS)



- ✓ 2-fold increased intensity
- ✓ Trace element speciation in ng to pg range



NIST water 1640

Amount of Ni (pg)	LOD Sy-TXRF (pg)	LOD conventional -TXRF (pg)
10	0.06	< DL
100	0.07	8

U. E. A. Fittschen, A. Guilherme et al. **J. Synchrotron Rad.** (2016), 23, 820-824.

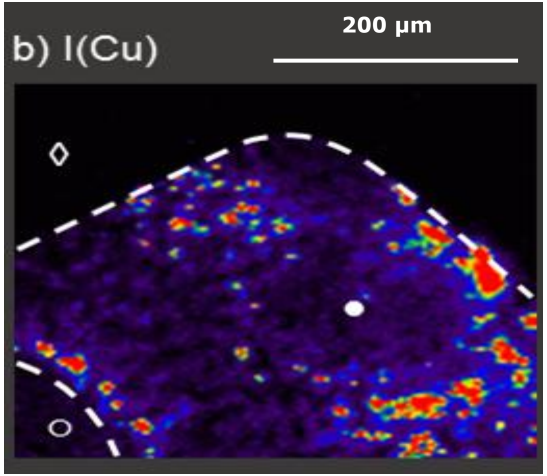
BAM*line* & µSpot

Analytical Methods

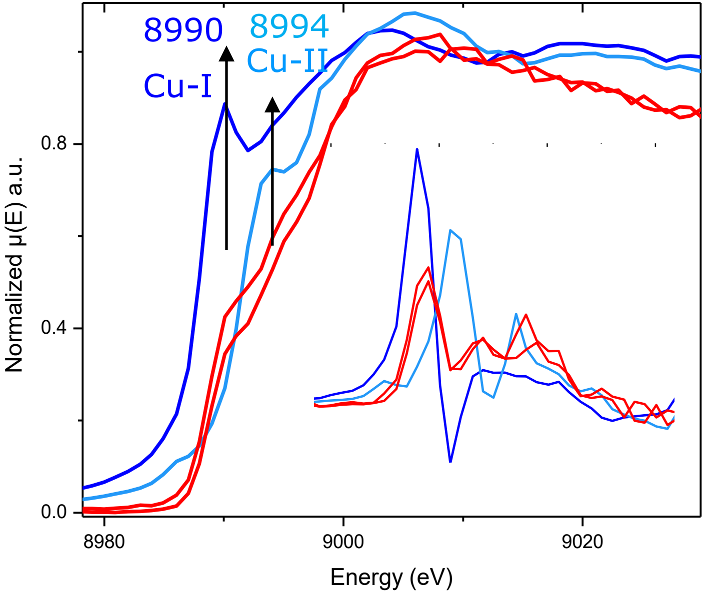
Applications & Collaboration Partners

Future Perspectives

micro-XAS – Morbus Wilson´s Disease: Cu speciation in liver



Cu distribution
on a liver specimen



Cu K-edge XANES on two areas

1 µm spot size
Cellular resolution

Partners

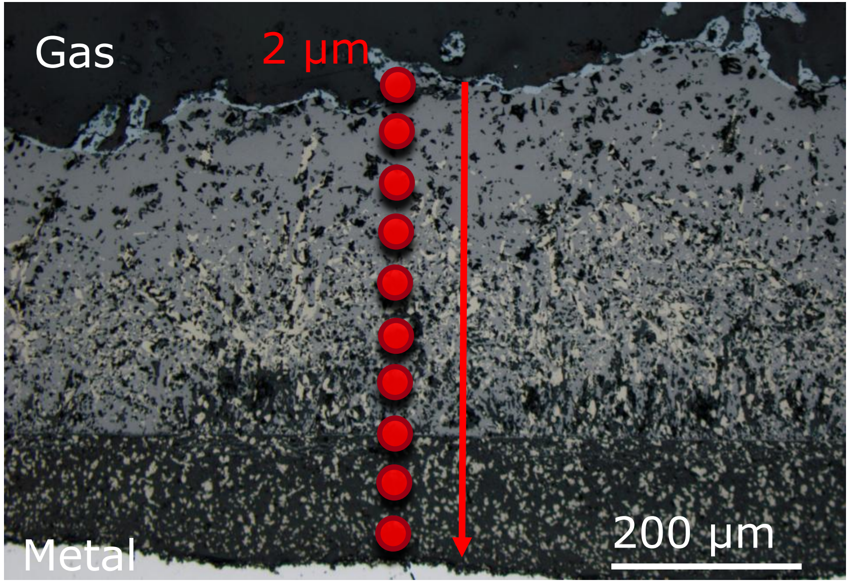
WWU MÜNSTER
Institute for Inorganic and Analytical Chemistry

O. Hachmöller, A. Guilherme Buzanich, et al. **Metallomics** (2016) 8, 648.

micro-XAS – identification of corrosion phases in steel

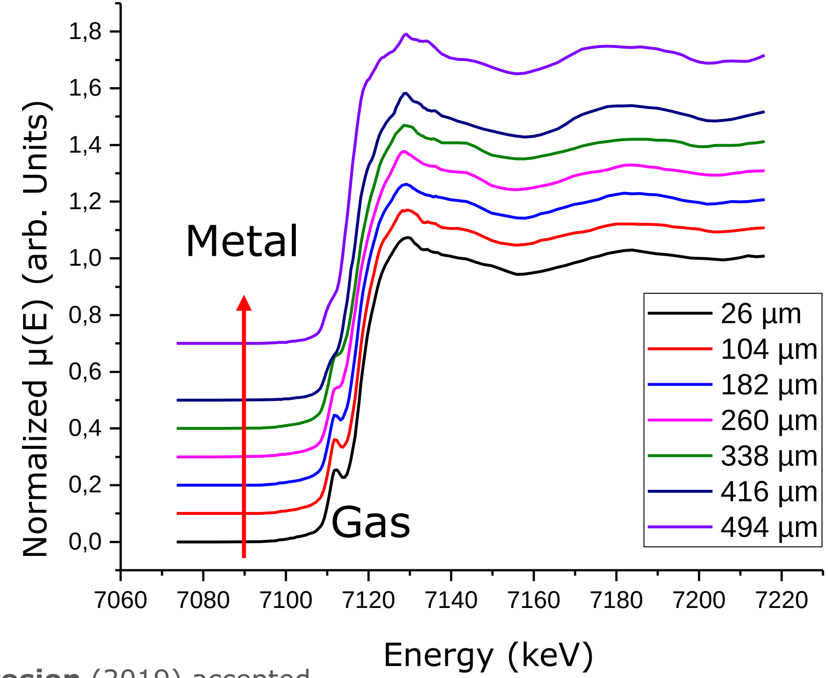
Ferritic steel with 2% Cr content

Fe2Cr (250h @ 650°C)



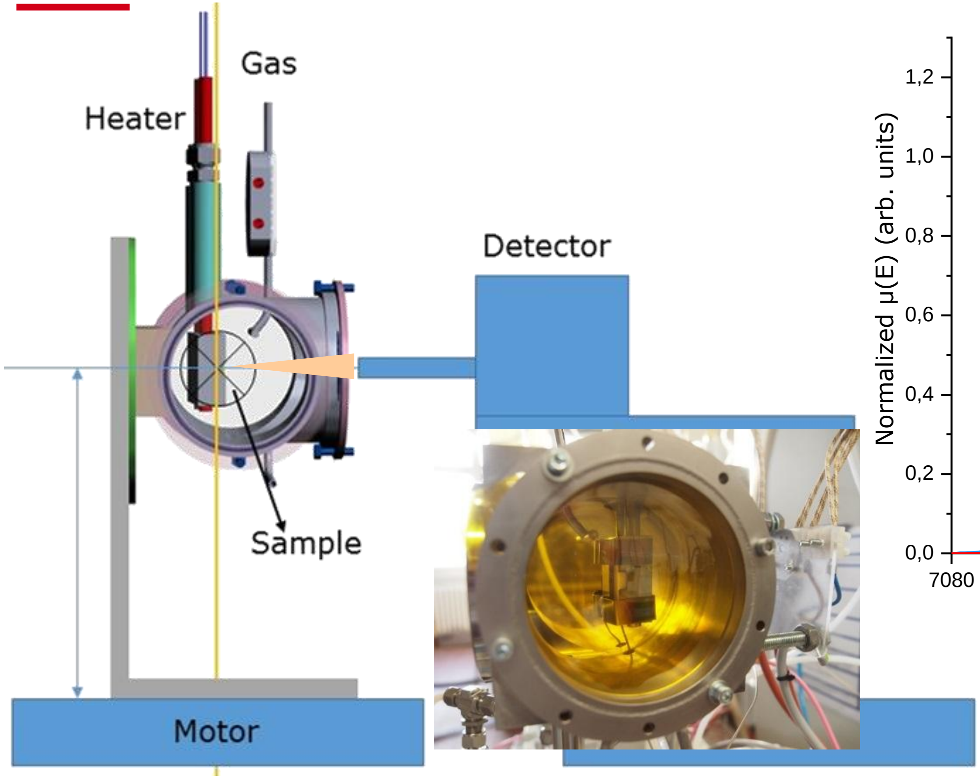
ex situ XANES @ Fe K-edge

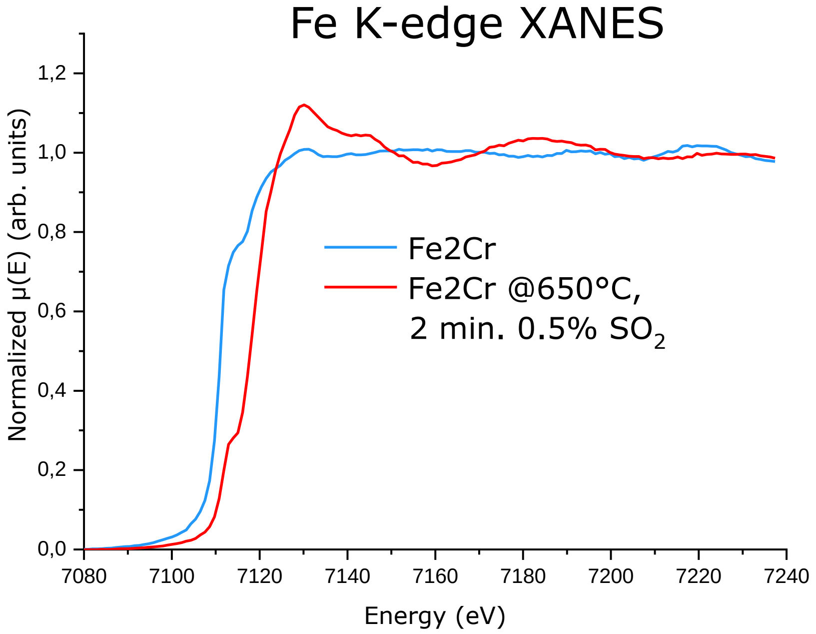
2 µm spotsize



K. Nützmann, A. Guilherme Buzanich, et al. **Materials and Corrosion** (2019) accepted.

micro-XAS: Understanding the corrosion of Fe2Cr alloys *in situ*

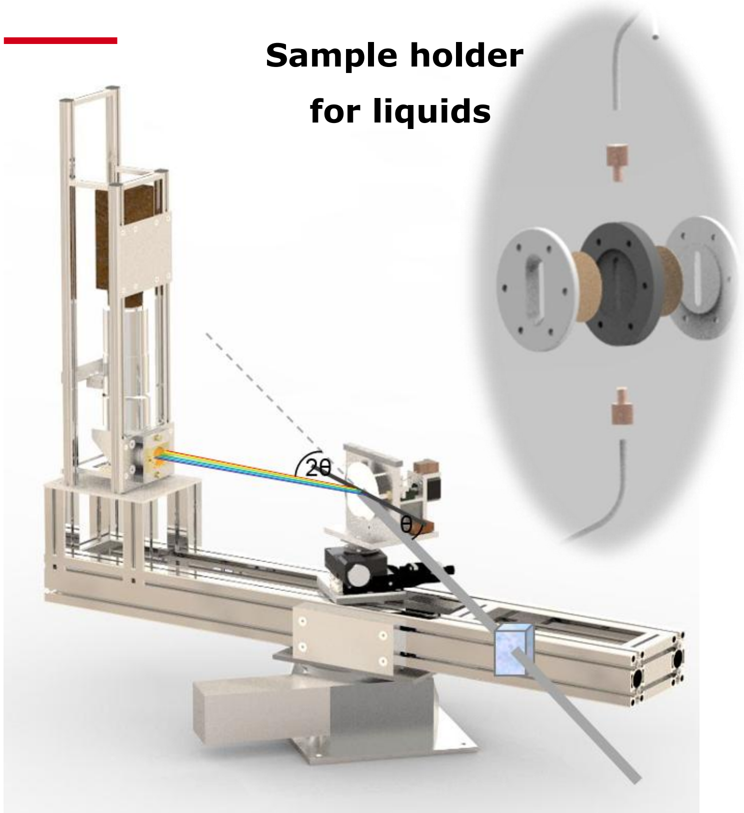




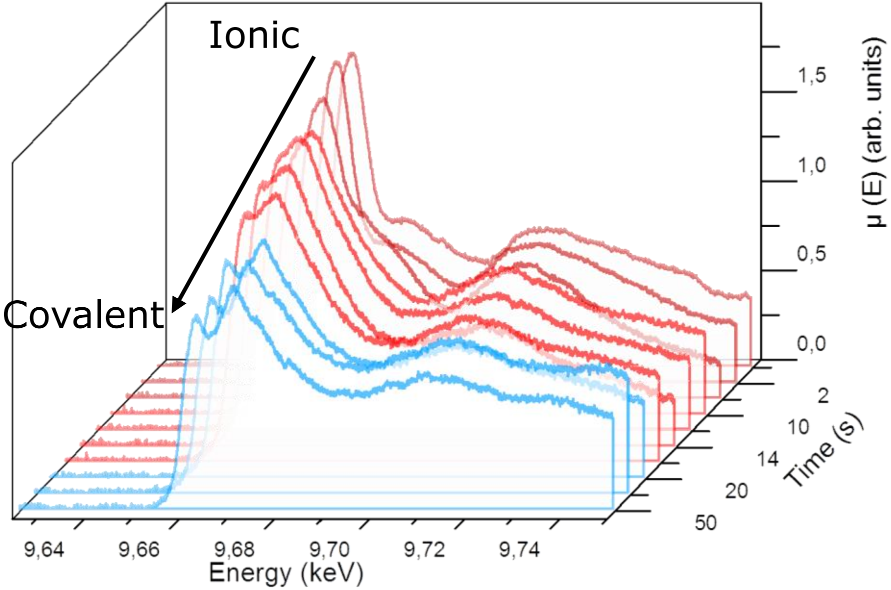
Fe_3O_4 is formed under extreme conditions – inverse spinel

Dispersive XAS (S^2XAFS) – In Situ Crystallization of ZIF-8

Sample holder for liquids

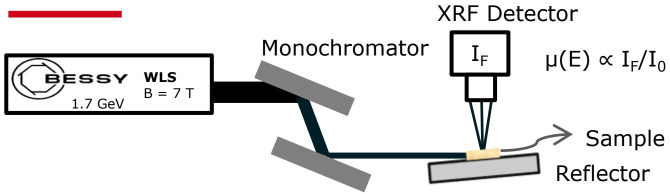


XANES Zn K-edge
1 second time resolution



A. Kulow et al. **J. Anal. At. Spectrom.** (2019) 34, 239-246.

TXRF-XANES – speciation of traces in ng range



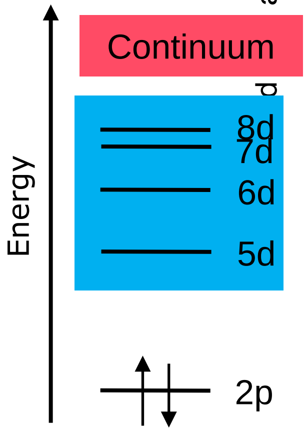
Partners

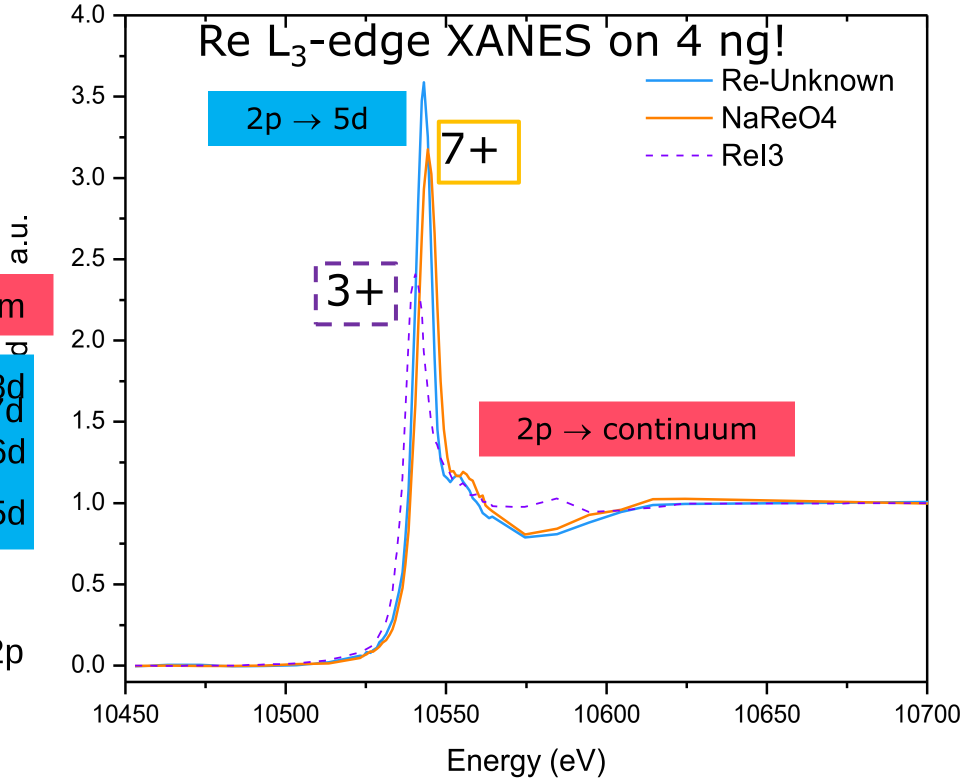
TU WIEN

ATOMINSTITUT

TU Clausthal

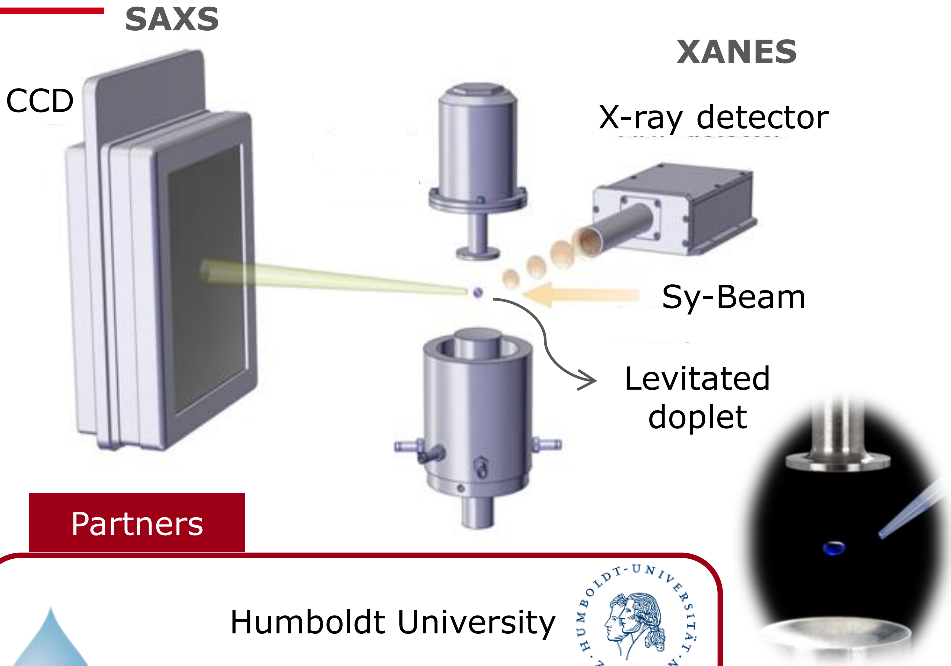
Material Analysis
and Functional Solid Matter

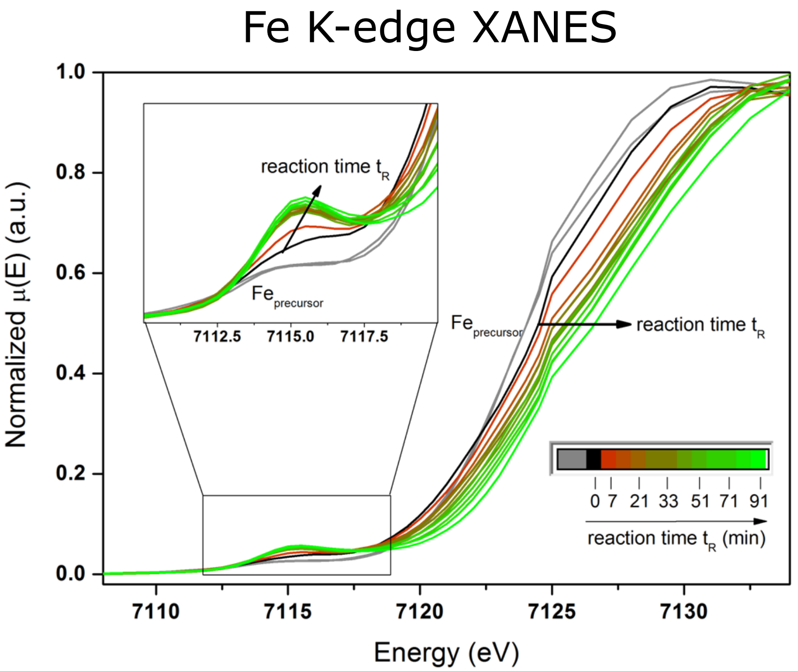




U. E. A. Fittschen, A. Guilherme, et al. **J. Synchrotron Rad.** (2016), 23, 820-824.

XAS + Scattering (SAXS) – *in situ* formation of Fe nanoparticles





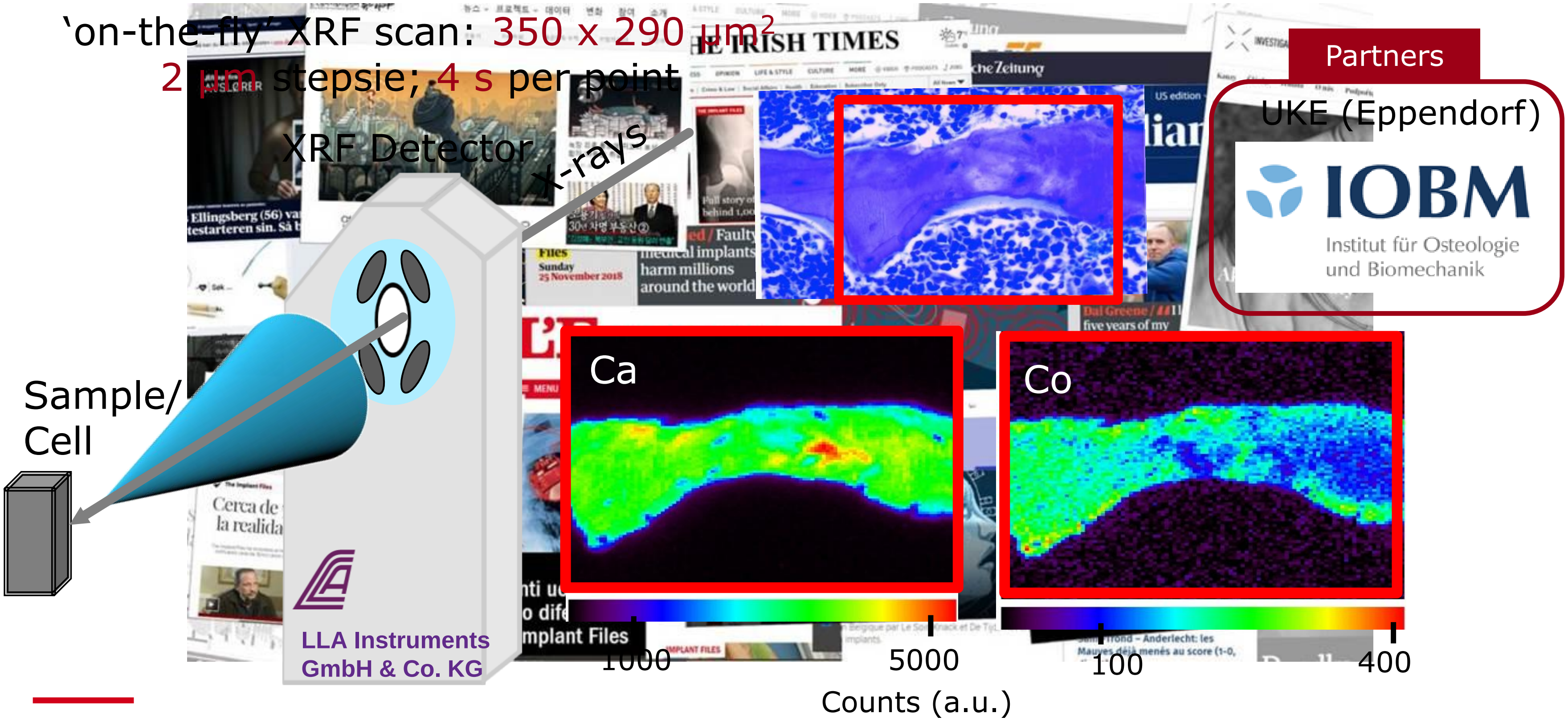
A. Kabelitz, et al. **CrystEngComm.** (2015) 17, 8463-8470

Partners

Humboldt University

CRC 1109 Understanding of Metal Oxide/Water Systems at the Molecular Scale: Structural Evolution, Interfaces, and Dissolution

XRF – Co distribution in bone by implants



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Future Perspectives

The future of our hard X-ray beamlines for material characterization

Automation	Reproducibility	Flexibility
• Control of adjusment parameters • Faster sample adjustment • High-throughput experiments	• Ensure equal measuring conditions • Reliable data	• Different technical specifications • Cover more fields of applications

Thank you for your attention !

synchrotronanalytik@bam.de

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