

Update on LabXAS activities at TU Berlin

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TU Berlin



motivation
in-situ & operando LabXAS
FAIR rdm
concluding remarks

motivation

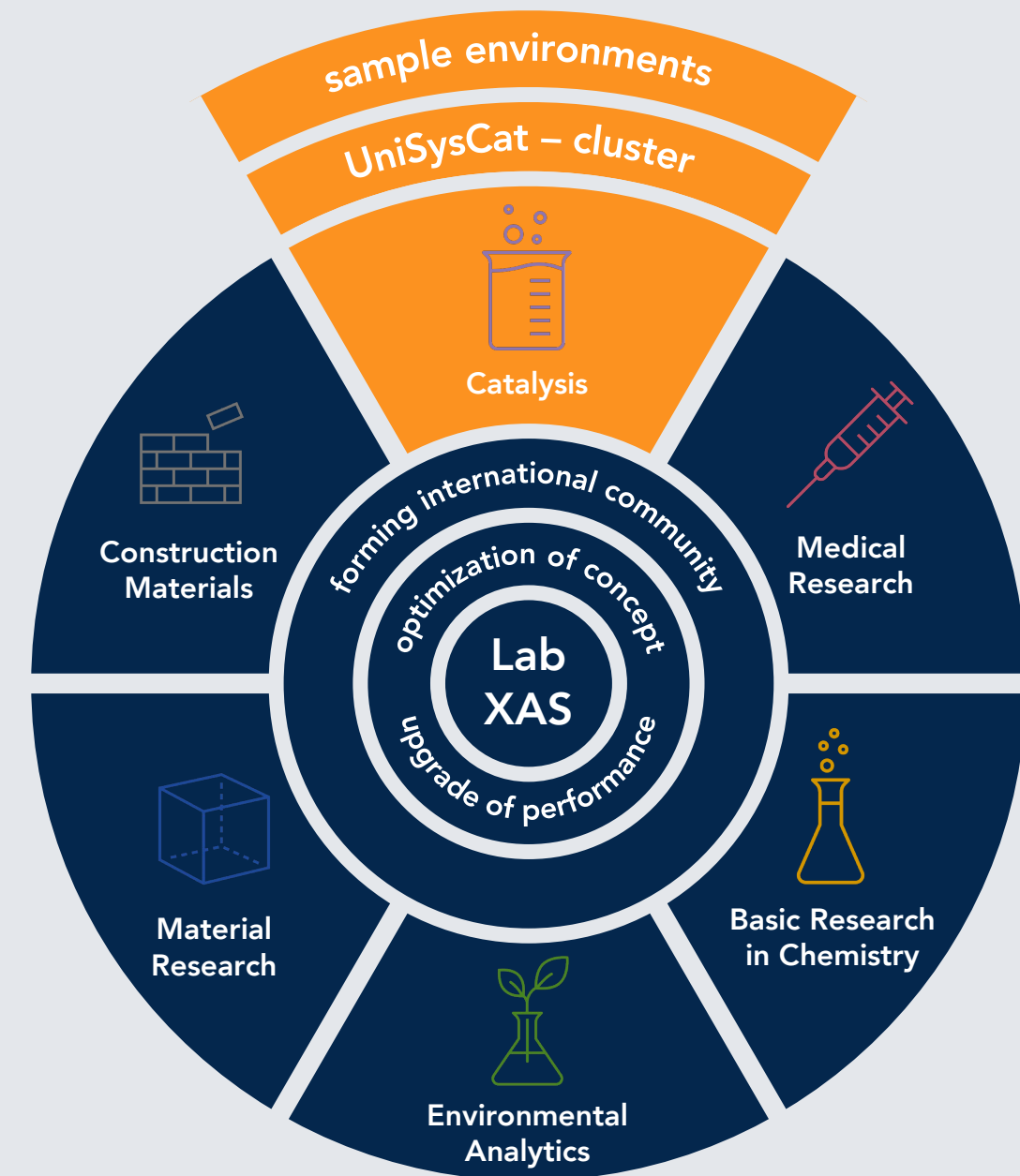
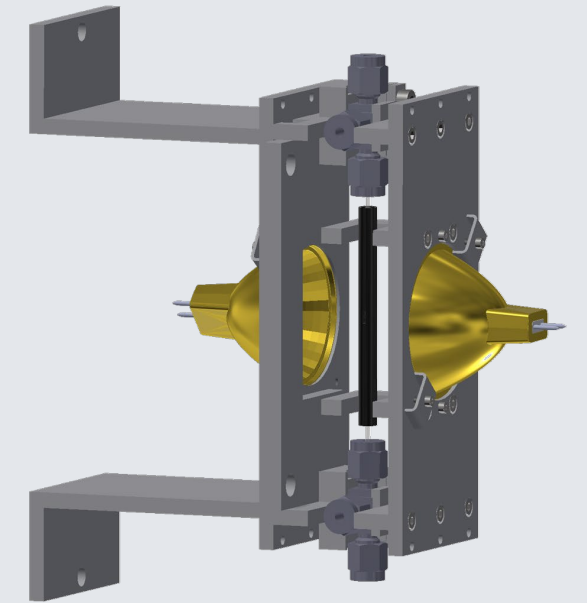
in-situ & operando LabXAS

FAIR rdm

concluding remarks

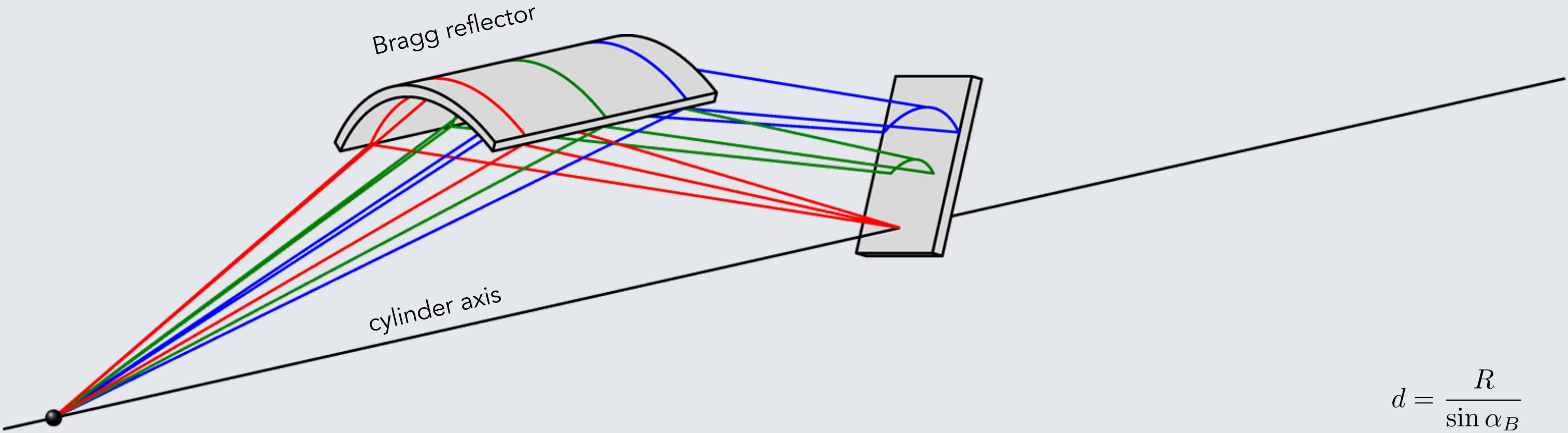
catalysis research with LabXAS

- in-situ and operando experiments
- thermal, long-term stability of spectrometer
- FAIR research data management concept for collaboration with various partners



wavelength dispersive spectrometer with divergent source and position sensitive detector

adapted von Hamos geometry – tilted detector for increased spectral bandwidth & necessary to benefit from mosaic crystals

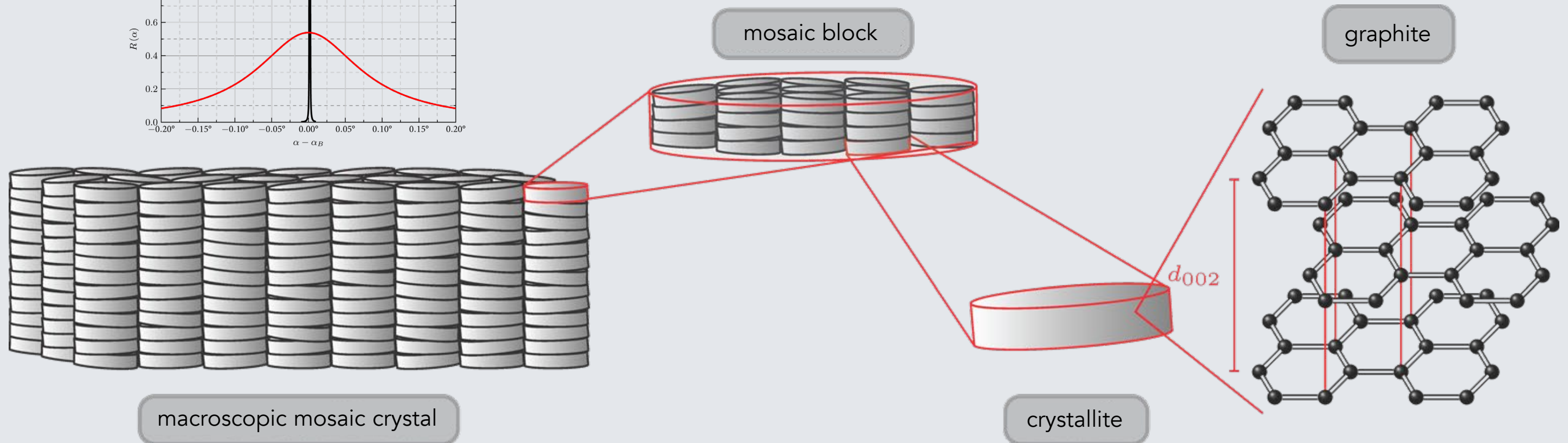
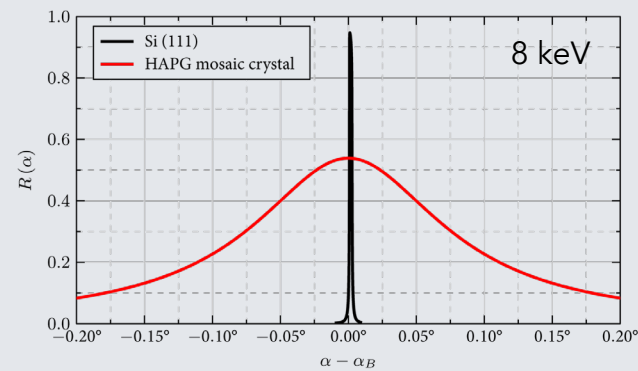


$$d = \frac{R}{\sin \alpha_B}$$

[J. Anal. At. Spectrom., 2015]

XAFS spectroscopy by an X-ray tube based spectrometer using a novel type of HOPG mosaic crystal and optimized image processing

Highly Annealed Pyrolytic Graphite (HAPG) – special mosaic crystal for increased integral reflectivity



[J. Appl. Cryst., 2017]

A new model for the description of X-ray diffraction from mosaic crystals for ray-tracing calculations

Inna Grigorieva
Alexander Antonov



LabXAS at TU Berlin

source

Mo microfocus x-ray tube
optimized to 15 kV

high power at low voltages



optic

HAPG optics optimized to
EXAFS or XANES

tailored to needs



detector

dectris EIGER2R 500K hybrid
cmos 2D pixelated detector

fast readout & large area



setup

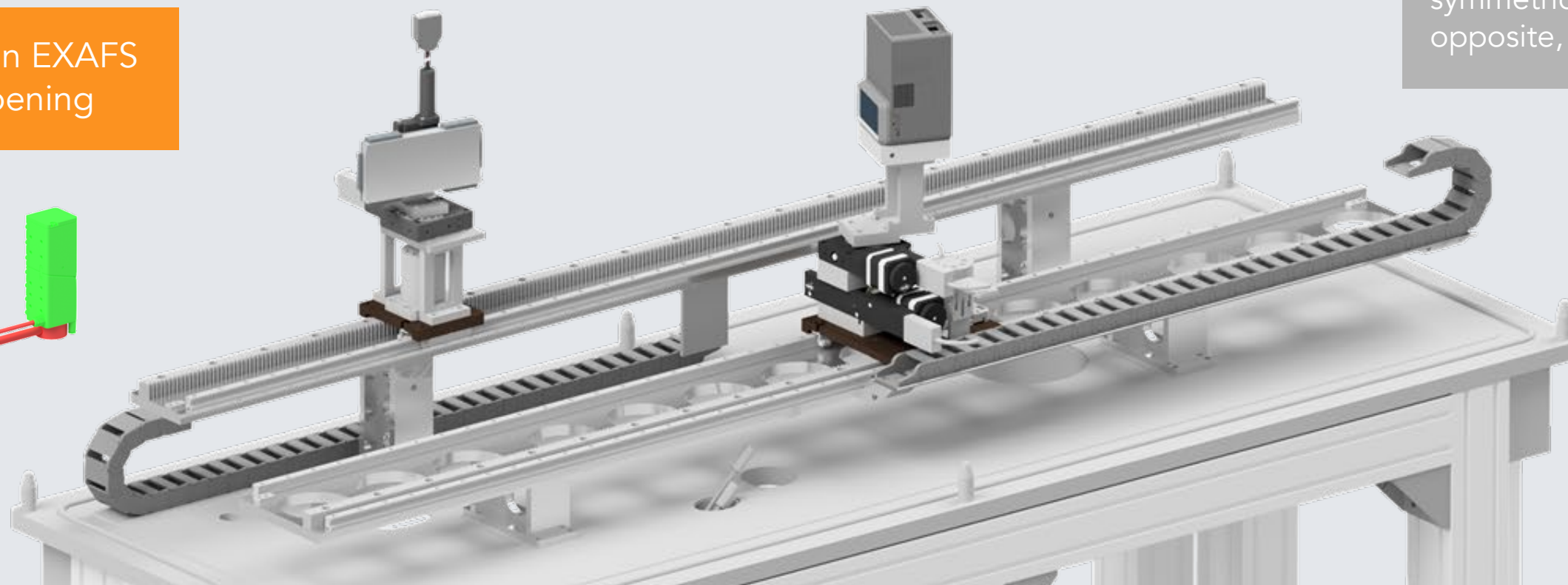
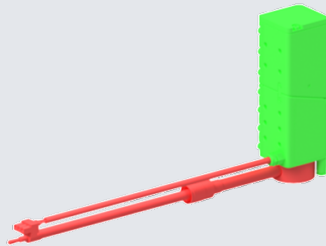
mechanics to change to
requested energy

automated image evaluation

sample environment right in
front of X-ray tube

symmetric construction for
opposite, second port

poster on EXAFS
dampening



motivation

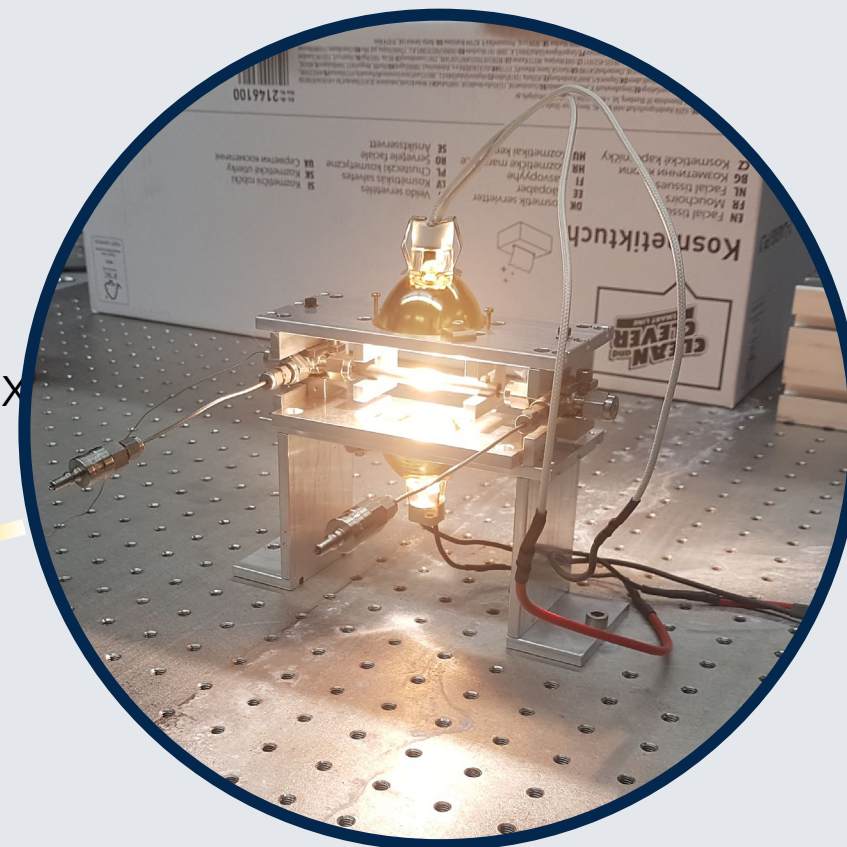
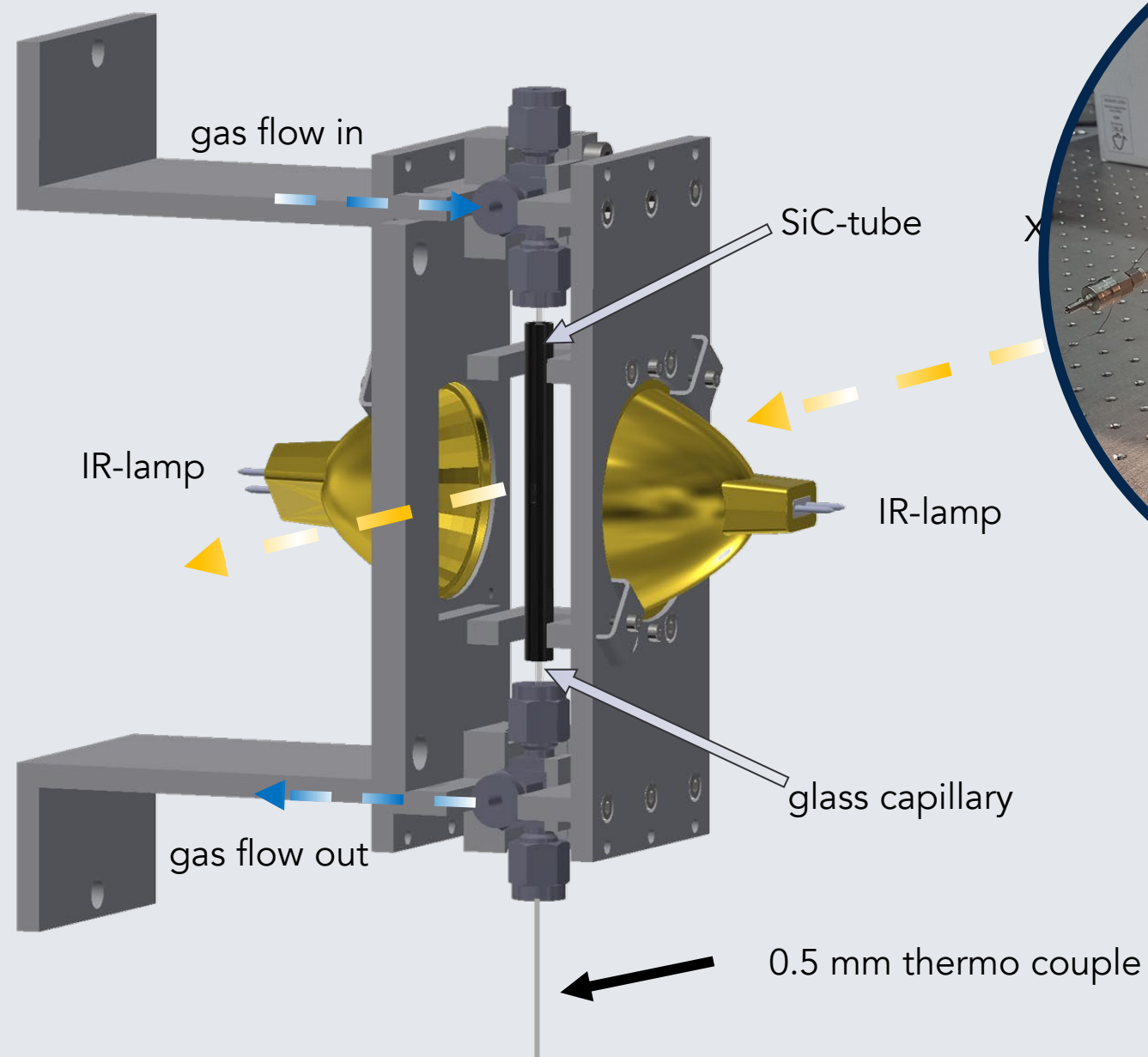
in-situ & operando LabXAS

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concluding remarks

IR tube furnace

- SiC tube with $\varnothing = 6$ mm with two slits for transmission
 - Slit size 1x4 mm (Lab-XAFS)
- Setup/capillary is placed vertical
 - Gas flow in on the top and out on the bottom
 - thermo-couple to track temperature



[Rev. Sci. Instrum., 2017]

Compact low power infrared tube furnace for in situ X-ray powder diffraction

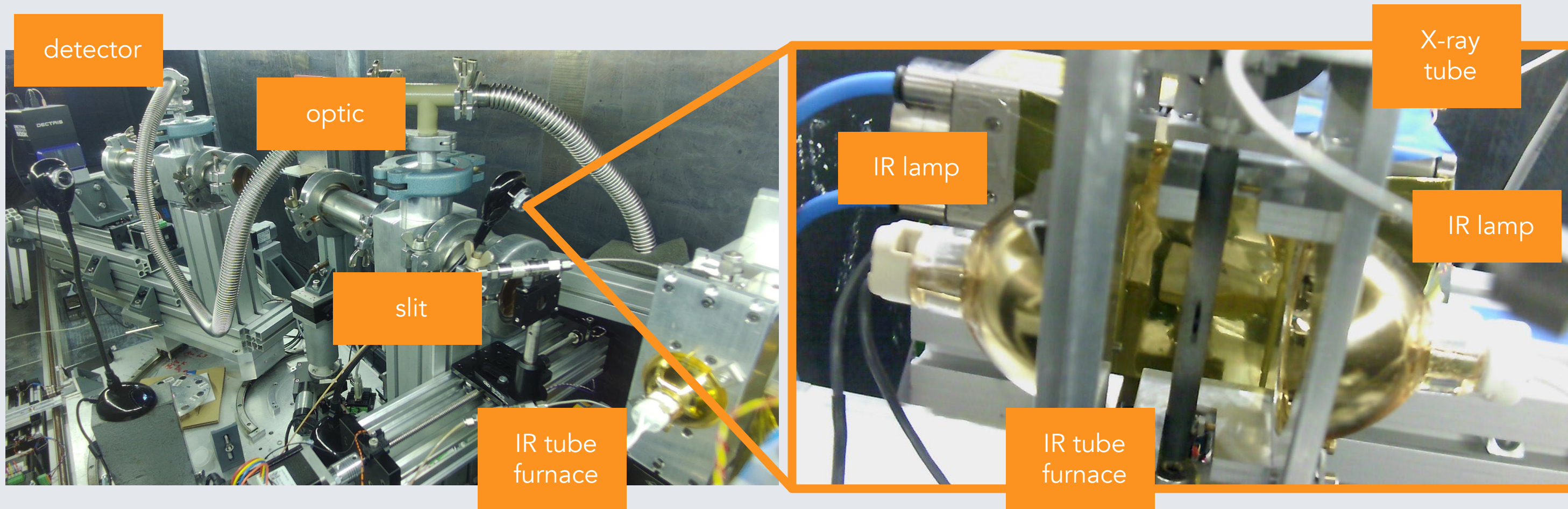
Developed by  Technische Universität Berlin

Chair of Advanced Ceramic Materials

Benjamin Bischoff, Emiliano Dal Molin, Delf Kober, Maged Bekheet, Albert Gili, AG Aleksander Gurlo

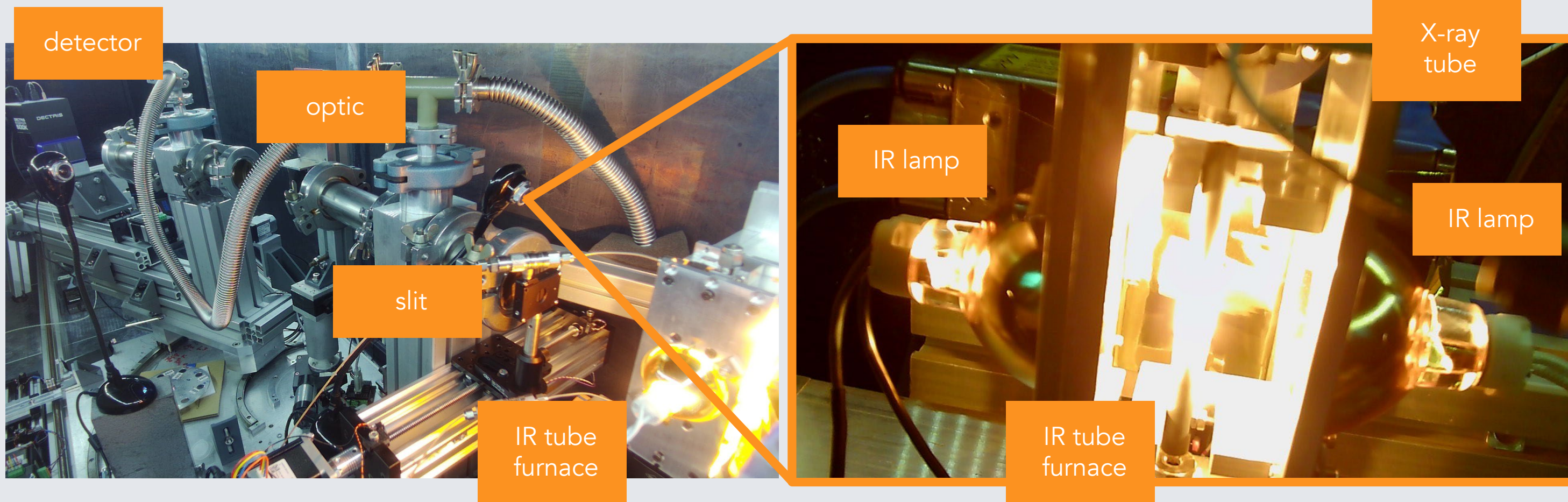
in-situ & operando LabXAS

room
temperature



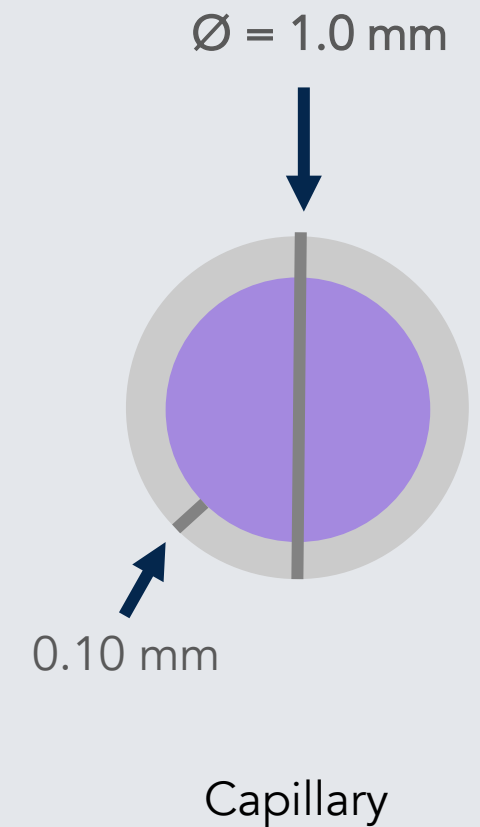
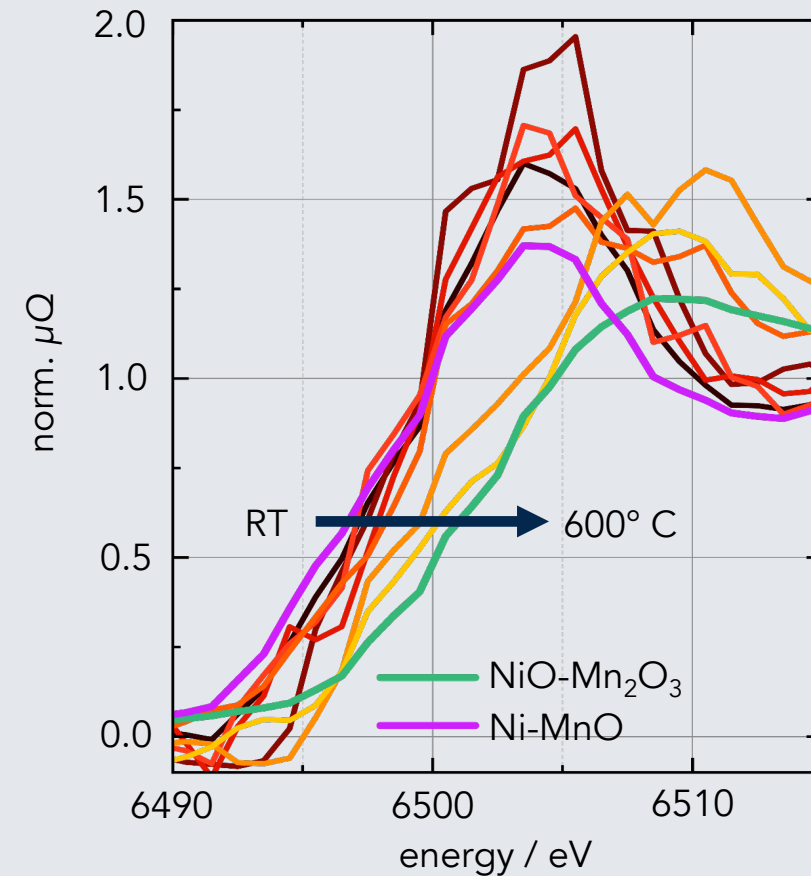
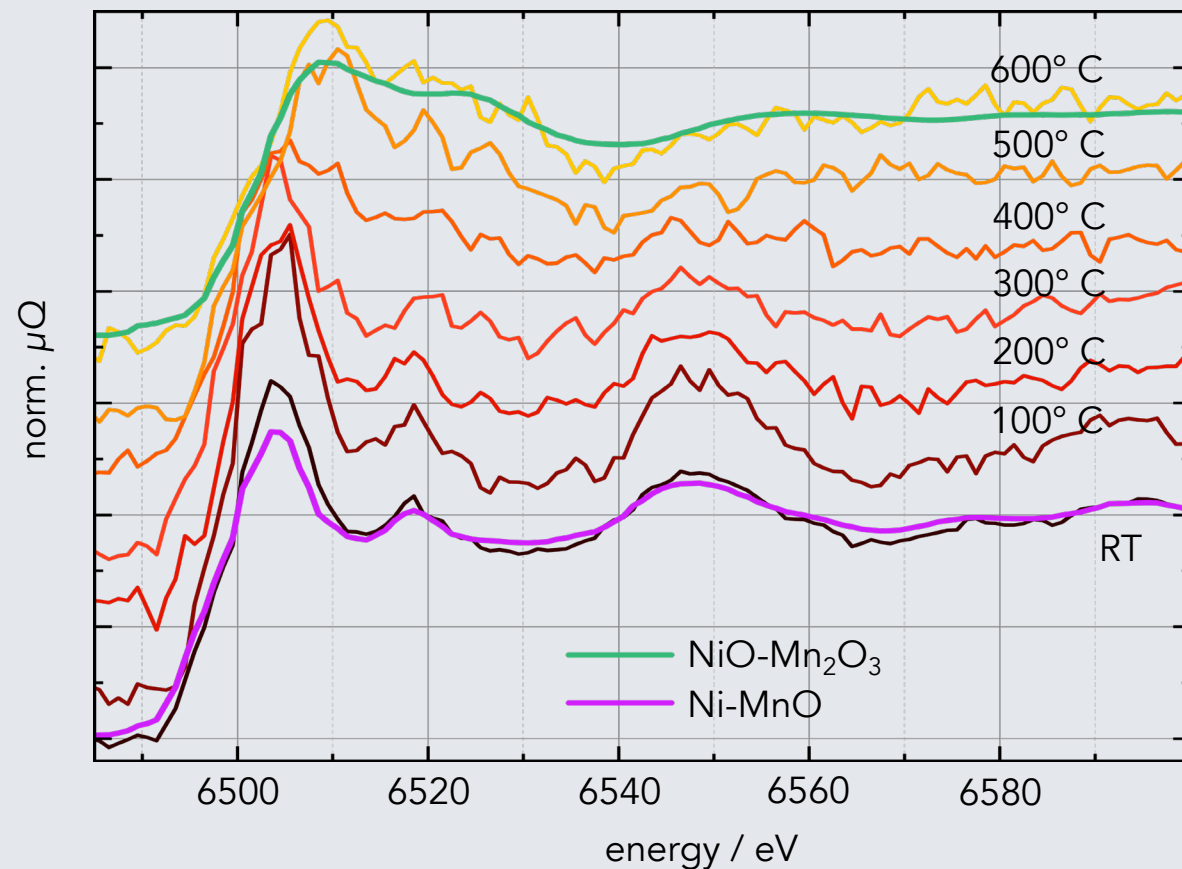
in-situ & operando LabXAS

350°C



Oxidation of MnO to Mn₂O₃

- 5% Ni on MnO catalyst measured at the Mn-K edge to investigate the oxidation during heating under air flow
- sample was diluted with boron nitride in the ratio of 1:8 → ~ 8 wt% to 10 wt% Mn in the capillary
- oxidation of MnO to Mn₂O₃ is visible while heating the sample up to 600°C
- measurement time: 15 min per spectrum, references has been measured longer

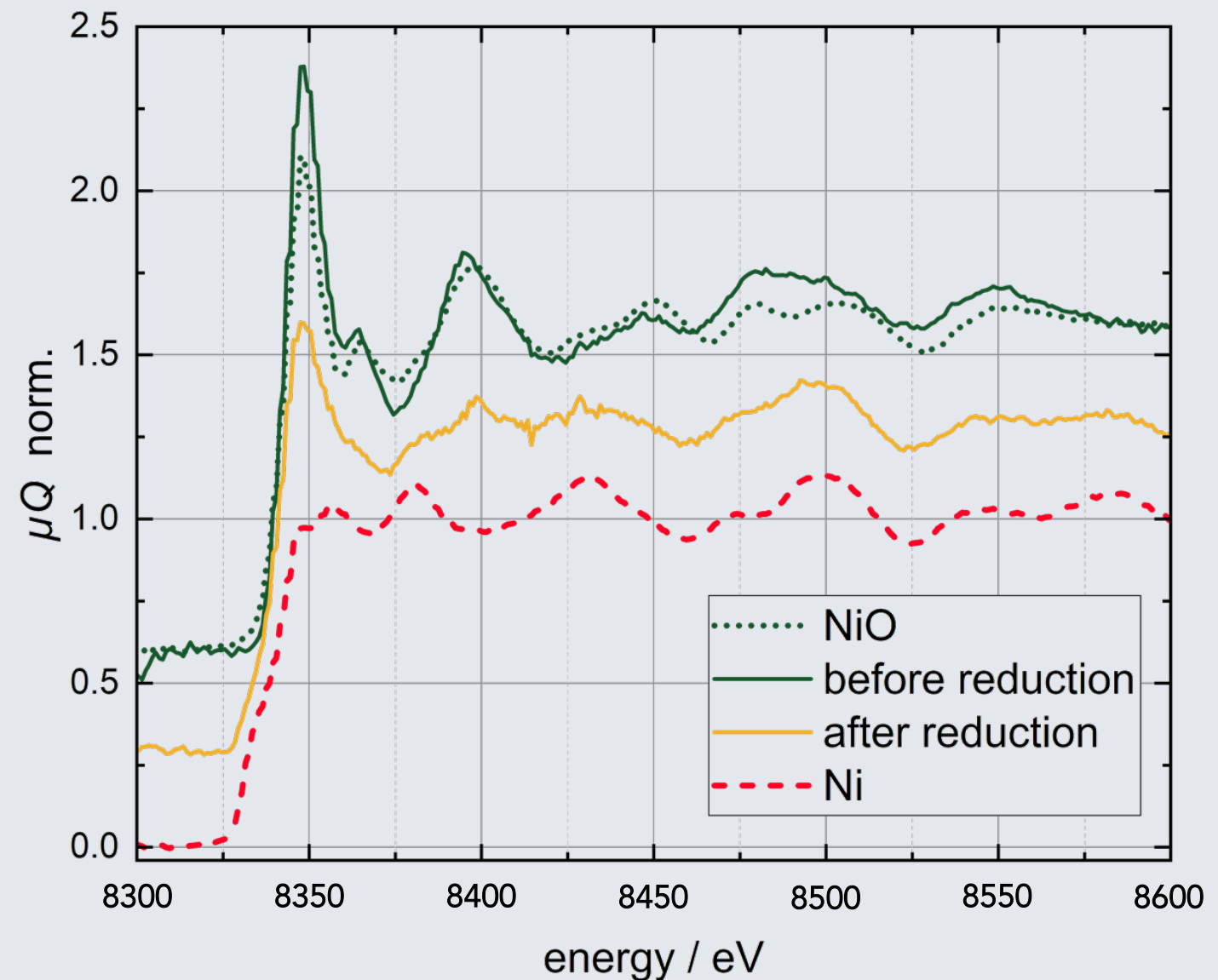


catalyst synthesized and reduced as in
[ACS Catal., 2018]

Surface Carbon as a Reactive Intermediate in Dry Reforming of Methane to Syngas on a 5% Ni/MnO Catalyst

Operando LabXAS

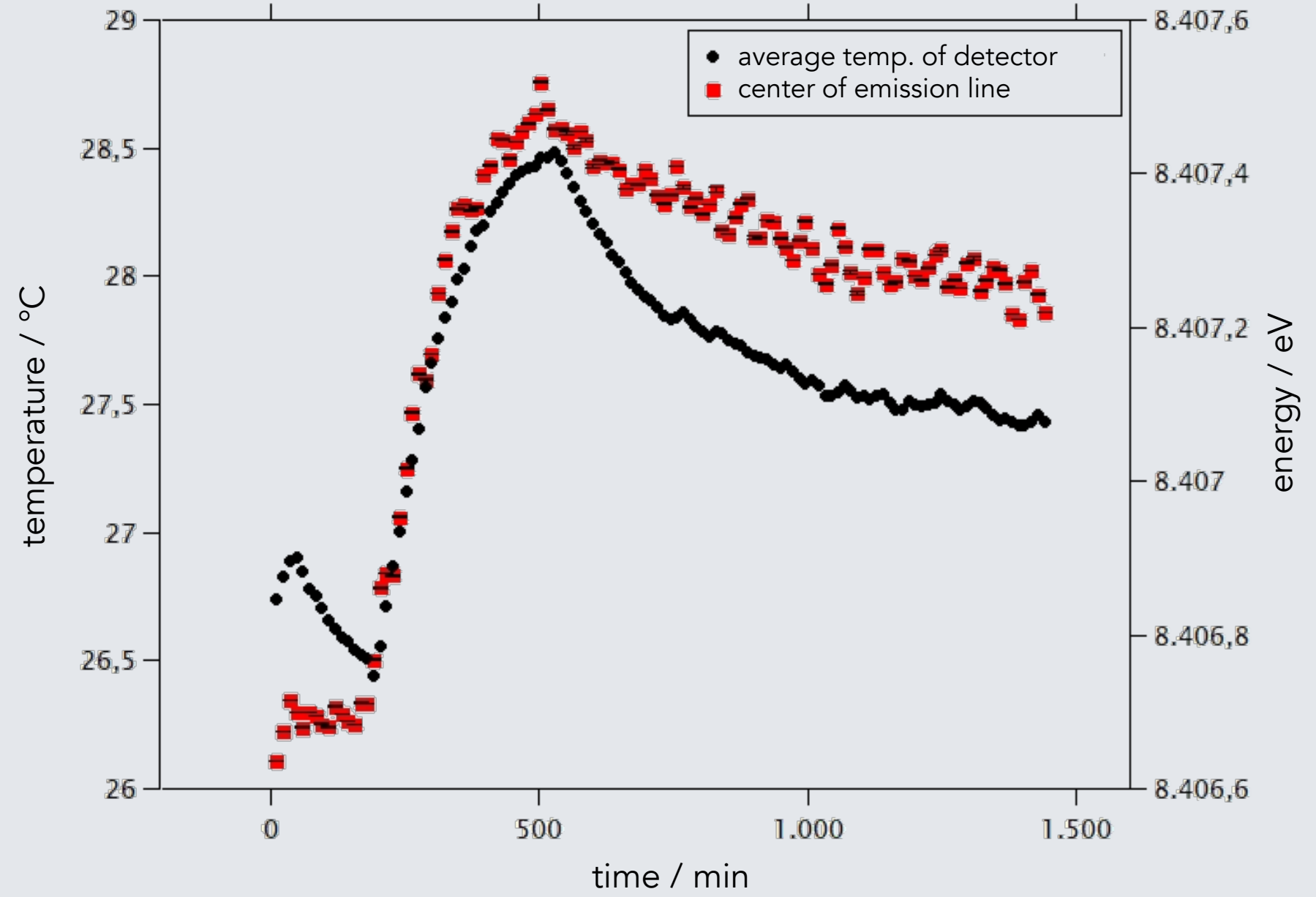
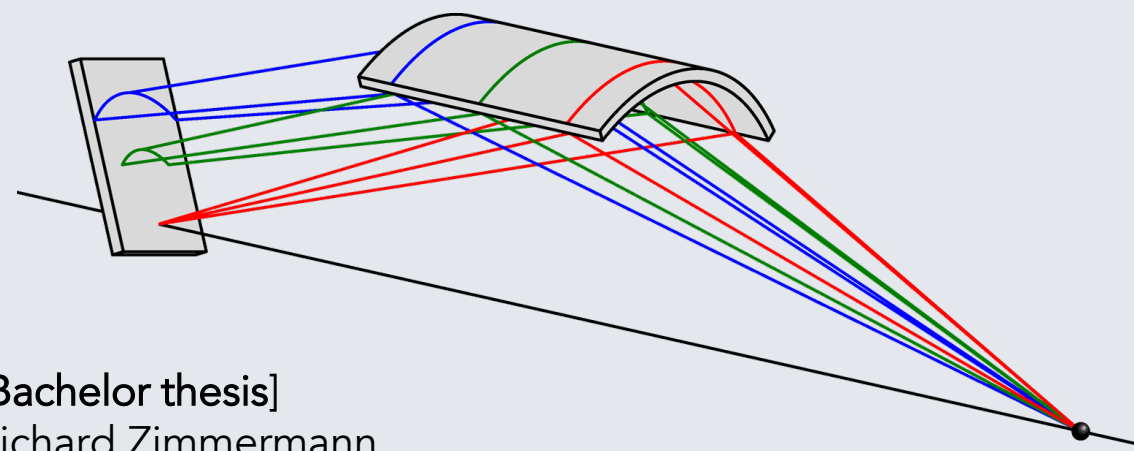
- NiO (20 wt% Ni) on silica
- activation @ 600°C with 24 mln/min 5% H₂ in Ar for 60 min
- No CH₄ or CO detection before activation (gas chromatograph)
- catalytic reaction @ 350°C with H₂ to CO₂ ratio of 1:4
- after activation ~ 50 % Ni⁰
- spectrum every 5 min



temperature stability

stability of spectrometer while heating

- track diverse temperatures inside and outside of spectrometer
- determine spectral shifts on absorption edge and/or emission line(s)
- still work in progress
- images every ten minutes, here without heating cell
- strong energy shift



[Bachelor thesis]
Richard Zimmermann

motivation

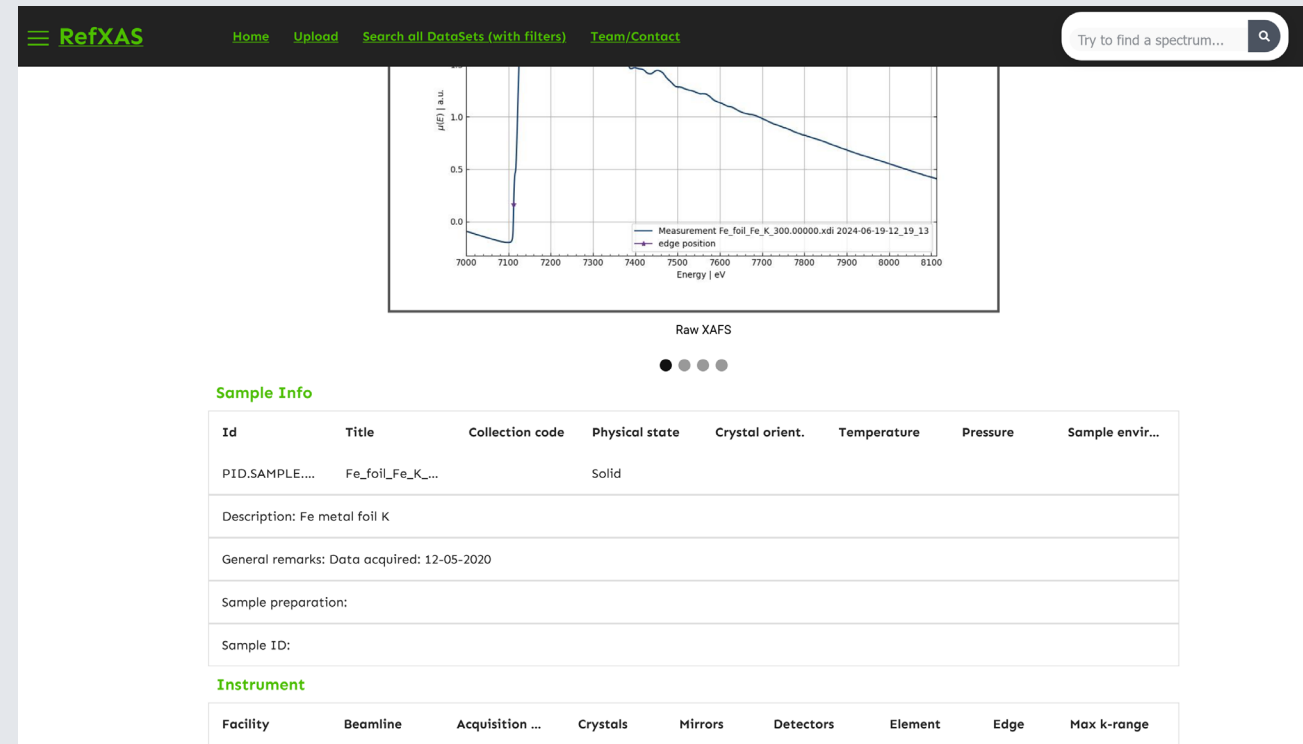
in-situ & operando LabXAS

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concluding remarks



poster on
RefXAS database



RefXAS database

- quality control for curated database
- first for reference materials
- involvement in new NeXuS XAS definition
- definition of metadata fields

Abhijeet Gaur, Dmitry Doronkin, Jan-Dierk Grunwaldt
 Karlsruhe Institute of Technology (KIT)
 Sebastian Paripisa, Dirk Lützenkirchen-Hecht
 Bergische Universität Wuppertal
 Edmund Welter
 Deutsches Elektronen-Synchrotron (DESY)

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sample preparation

- sample entry
- generate barcode

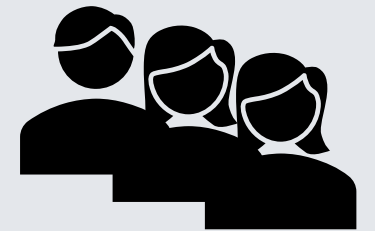
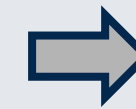
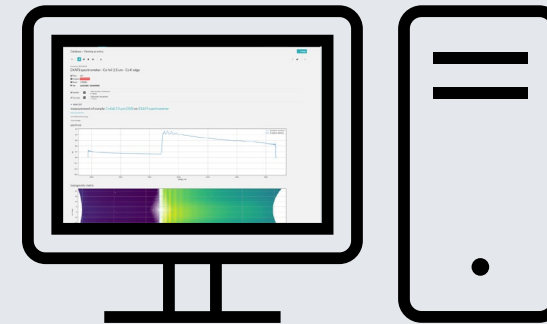
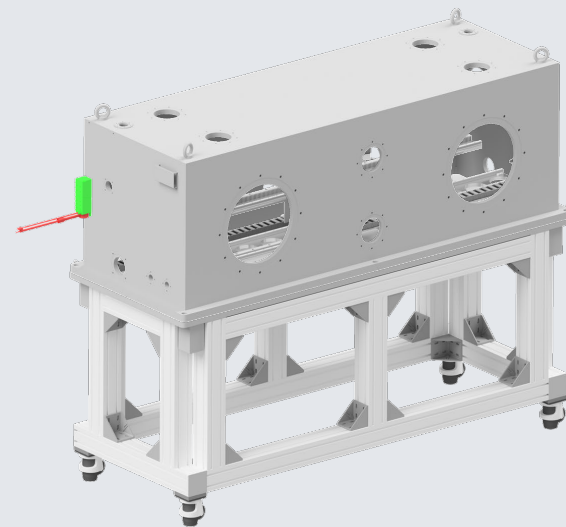
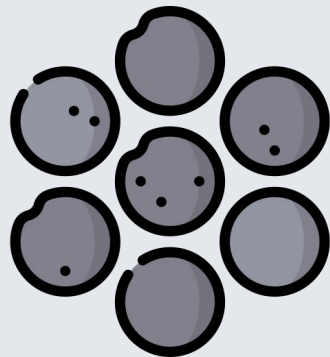
at spectrometers

- scan of sample barcode
- get sampleID
- generate entry for measurement from data

office

- quick access to spectrum and other important metadata
- sharing with partners

ELN

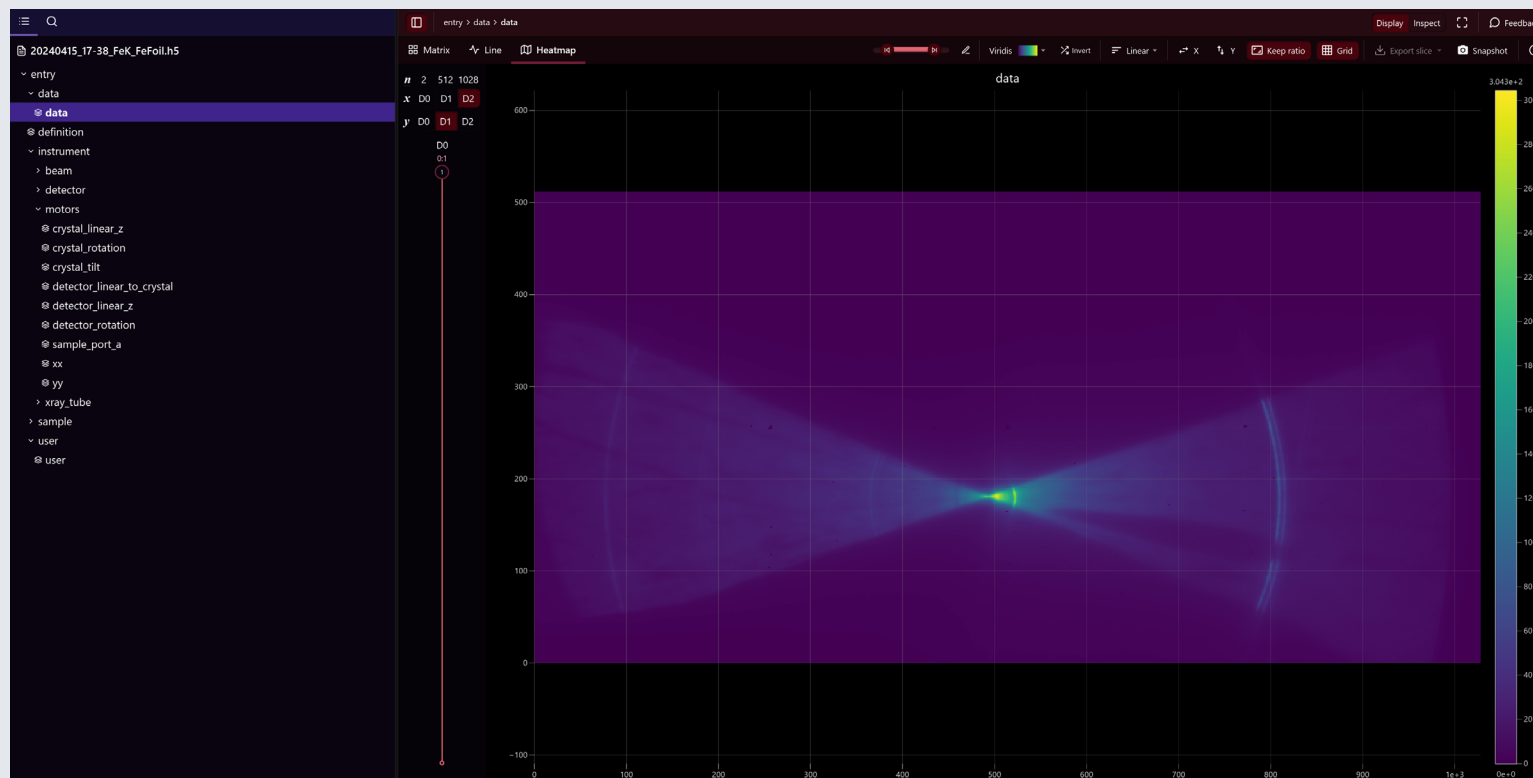


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data saved in *.h5 format
 for easy access spectrum also saved as two column ascii file
 log files for various machine parameters

Name	Date modified	Type	Size
20240807_17-49_FeK_FeFoil_delta_0-005_02_configuration.dat	07/08/2024 18:46	DAT File	1 KB
20240807_17-49_FeK_FeFoil_delta_0-005_03_spectraIntensity.dat	07/08/2024 18:46	DAT File	245 KB
20240807_17-49_FeK_FeFoil_delta_0-005_04_spectraAbsorption.dat	07/08/2024 18:46	DAT File	176 KB
20240807_17-49_FeK_FeFoil_delta_0-005_05_spectraAbsorptionStd.dat	07/08/2024 18:46	DAT File	228 KB
20240807_17-49_FeK_FeFoil_delta_0-005_06_InhomogeneityMatrix.dat	07/08/2024 18:46	DAT File	21.600 KB

Name	Date modified	Type	Size
detector	21/08/2024 00:00	File folder	
vacuum	21/08/2024 00:00	File folder	
xraytube_porta	21/08/2024 00:00	File folder	
20230918_LabEXAFS.log	18/09/2023 17:50	Text Document	1 KB
20230919_LabEXAFS.log	19/09/2023 18:19	Text Document	39 KB
20230920_LabEXAFS.log	20/09/2023 17:15	Text Document	28 KB
20230921_LabEXAFS.log	21/09/2023 15:38	Text Document	22 KB
20230925_LabEXAFS.log	25/09/2023 23:59	Text Document	17 KB
20230926_LabEXAFS.log	26/09/2023 23:50	Text Document	26 KB
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20230928_LabEXAFS.log	28/09/2023 14:03	Text Document	4 KB



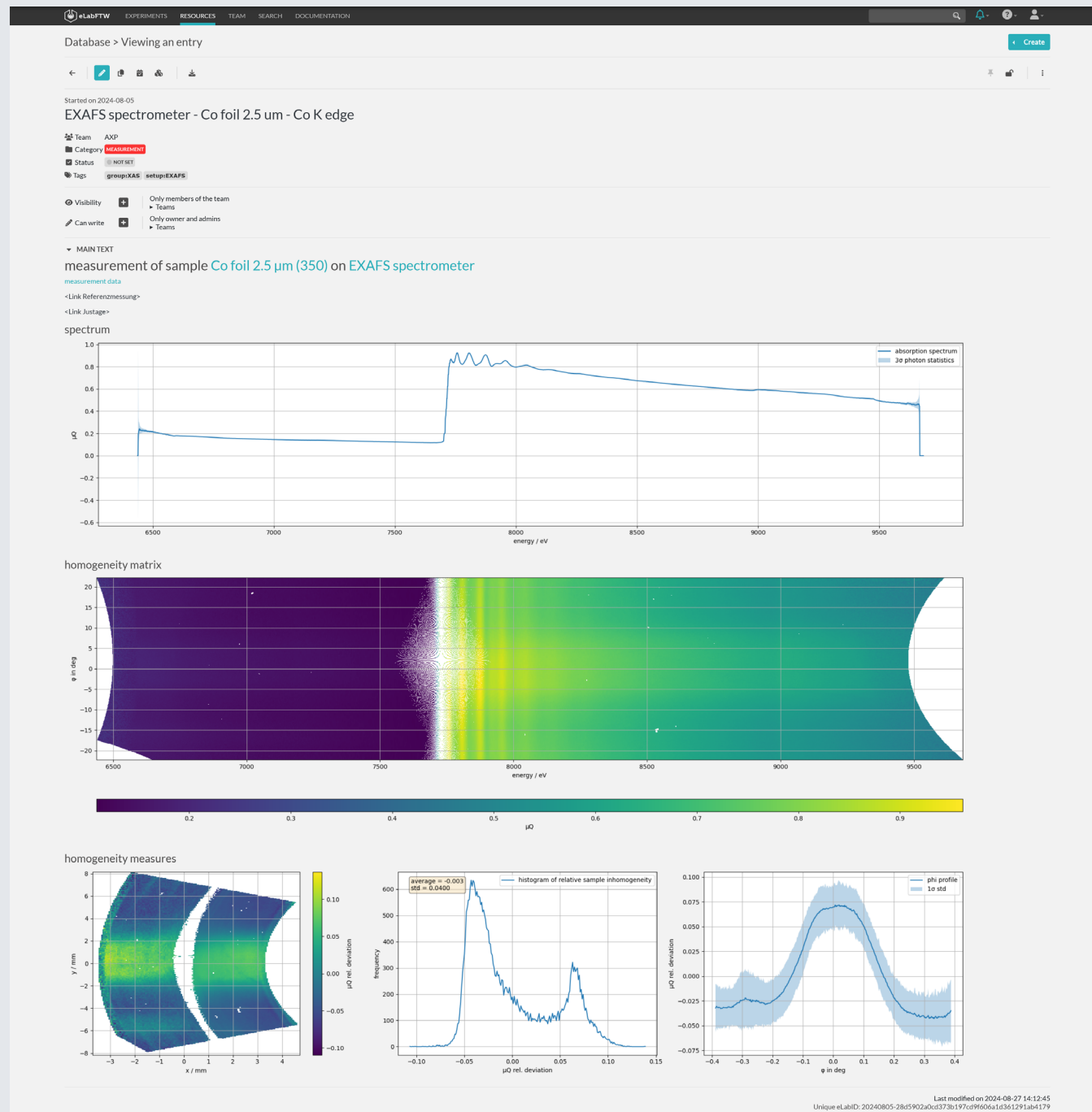
data structure

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20240819_LabEXAFS_xraytube_porta.log	19/08/2024 23:59	Text Document	507 KB
20240818_LabEXAFS_xraytube_porta.log	18/08/2024 23:59	Text Document	507 KB
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20240816_LabEXAFS_xraytube_porta.log	16/08/2024 23:59	Text Document	507 KB
20240815_LabEXAFS_xraytube_porta.log	15/08/2024 23:59	Text Document	507 KB
20240814_LabEXAFS_xraytube_porta.log	14/08/2024 23:59	Text Document	507 KB

log files

time	HV_kv_set	HV_kv_get	I_ma_set	I_ma_get
00:00:03	5.00	5.66	0.30	0.32
00:00:08	5.00	5.71	0.30	0.32
00:00:13	5.00	5.66	0.30	0.32
00:00:18	5.00	5.76	0.30	0.32
00:00:23	5.00	5.66	0.30	0.32
00:00:28	5.00	5.76	0.30	0.32
00:00:33	5.00	5.66	0.30	0.32
00:00:38	5.00	5.71	0.30	0.32
00:00:43	5.00	5.86	0.30	0.32
00:00:48	5.00	5.76	0.30	0.32
00:00:53	5.00	5.76	0.30	0.32
00:00:58	5.00	5.66	0.30	0.32
00:01:03	5.00	5.62	0.30	0.32
00:01:08	5.00	5.76	0.30	0.32
00:01:13	5.00	5.81	0.30	0.32
00:01:18	5.00	5.71	0.30	0.32
00:01:23	5.00	5.66	0.30	0.32
00:01:28	5.00	5.76	0.30	0.32
00:01:33	5.00	5.66	0.30	0.31
00:01:38	5.00	5.81	0.30	0.31
00:01:43	5.00	5.71	0.30	0.32
00:01:48	5.00	5.71	0.30	0.31
00:01:53	5.00	5.71	0.30	0.32

FAIR rdm



EXTRA FIELDS

GENERAL

name
long, installed name - use title for short name

contact

keywords
space separated list of keywords

MEASUREMENT

sample
Co foil 2.5 μm - exafs reference

setup
EXAFS spectrometer

creator
Christopher Schlesiger

date creation
2024-08-05

storage location
<https://axpsyn.axp.tu-berlin.de>

date and time of measurement
2024-08-04T11:00

length of measurement (t0 + isample)
1695 s + 2435 s

high voltage, current, power
14.8 kV, 1.53 mA, 22.64 W

delta_rel
0.005

focus energy / eV
7729

optic
EXAFS optic

log files location
<https://axpsyn.axp.tu-berlin.de>

LINKED RESOURCES

- Co foil 2.5 μm - exafs reference
- EXAFS spectrometer

ATTACHED FILES

- EXAFS-spectrum_Co-foil.dat 90.08 KiB - 2024-08-05 14:50:23
- homogeneity_measures.png 200.85 KiB - 2024-08-05 14:31:20
- homogeneity_matrix.png 544.16 KiB - 2024-08-05 14:06:29
- spectrum.png 43.81 KiB - 2024-08-05 14:08:18

COMMENTS

Add a comment

Powered by LabFTW 5.0.0
Made with Fly Outboard

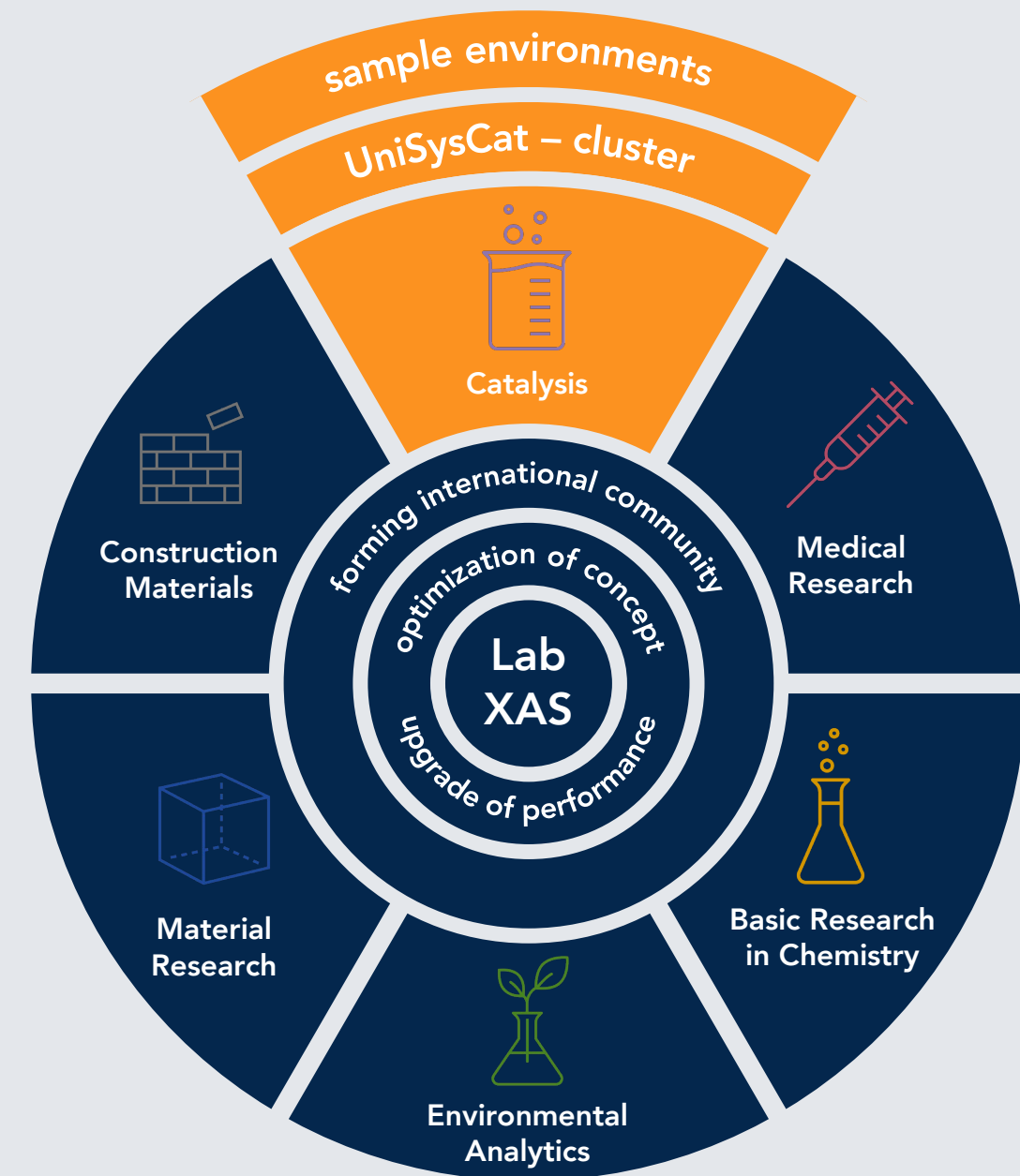
ELN integration for easy access and sharing with overview on data and meta data

motivation

in-situ & operando LabXAS

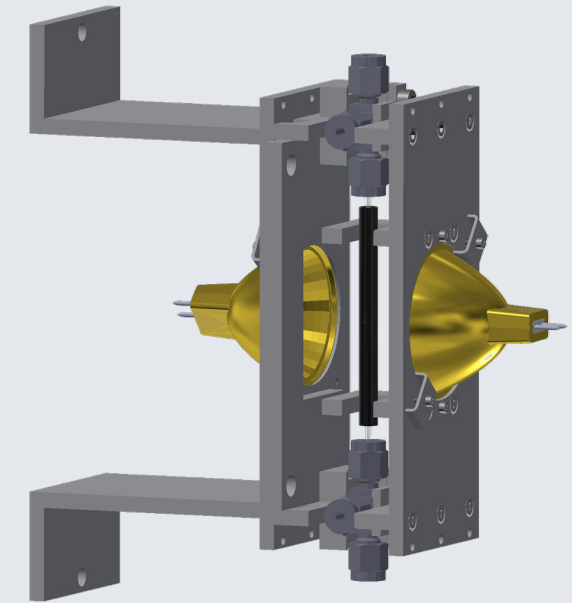
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concluding remarks

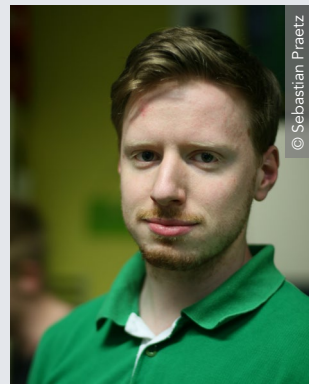


catalysis research with LabXAS

- in-situ and operando experiments
- thermal, long-term stability of spectrometer
- FAIR research data management concept for collaboration with various partners



acknowledgement



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Ha Vu Le
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Rafael Lima Oliveira



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Maurice Nohl
Julien Olck
Tobias Rattmann
Juan Mateo Rodriguez
Johannes Stark
Felix Steinbach

Holger Stiel
Julian Verhey
Maria Würth
Richard Zimmermann
all members of our research group

thank you
for your
attention

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Carsten Walter



Annette Trunschke
Maria Dimitrakopoulou

Ferdi Hellweger
Lukas Massa
Luisa Rabe



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