SAMPLE ENVIRONMENTS AT A SYNCHROTRON

K. Kiefer (HZB), S. Carlson (MAXIV), M. Bartkowiak (PSI)

School on Synchrotron Light Sources and their Applications | (SMR 4051), 16 January 2025
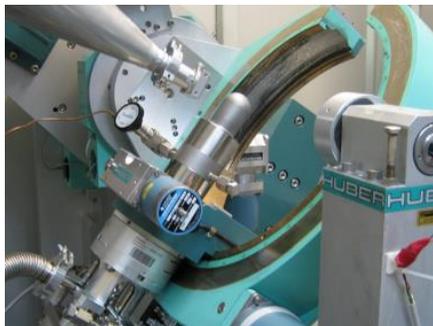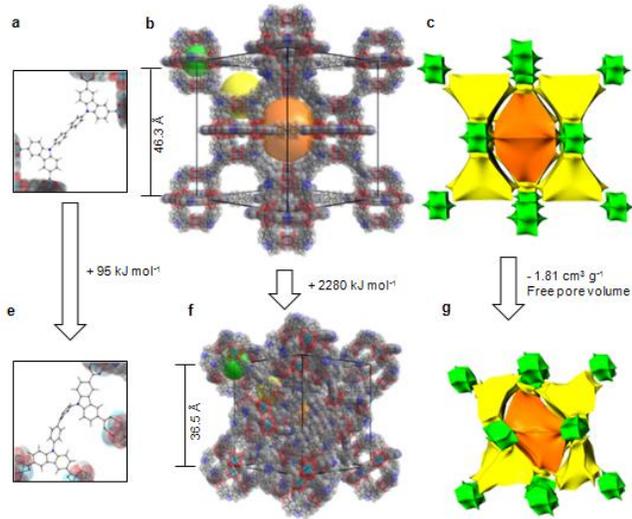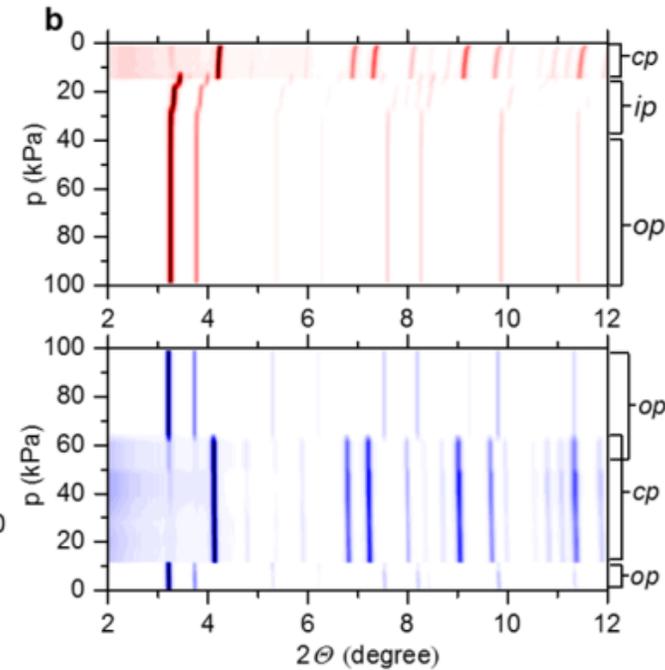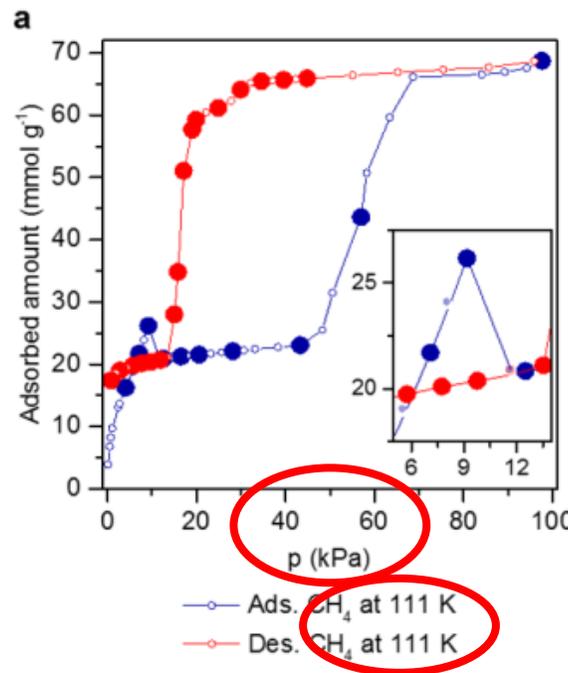
Sample Environment

WHAT IS SAMPLE ENVIRONMENT?

- Physical parameters: temperature, pressure, magnetic field, electric field, …

- In-situ / operando experiments: catalysis, energy storage, …

- Sample positioning: nano-positioning, rotation, …

- Sample mounting

- Everything that effects the sample - minus the actual measurement

MOTIVATION: PRESSURE AMPLIFYING FRAMEWORK WITH NEGATIVE GASADSORPTION TRANSITIONS

In-situ gas adsorption in combination with WAXS at beamline KMC-2

S. Krause et al. Nature 532, 348 (2016)

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WHAT IS SAMPLE ENVIRONMENT?
KNOWLEDGE & PEOPLE

Scientists

Engineers

Technicians

Students

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WHAT IS SAMPLE ENVIRONMENT?
COMMITMENT!

Technical developments

Optimization and repair (of existing SE equipment)

Development (of novel SE)

Consulting (of new beamline projects)

User service

Provision and setup (of modular and complex SE)

OVERVIEW

- What is sample environment?

- General challenges

- Sample environment for T, B, p

- In-situ, operando & other complex things

- Sample positioning, mounting & automation

- Sample environment at MAX IV (S. Carlson, MAX IV)

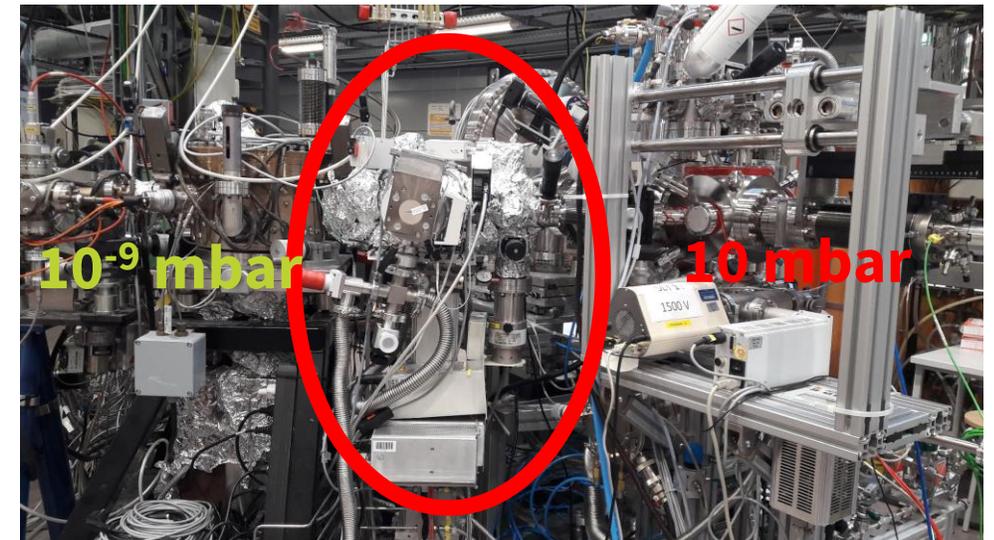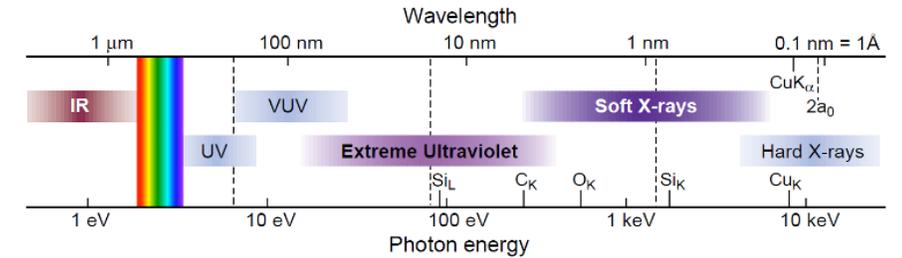- The International Society for Sample Environment (M. Bartkowiak, PSI)

GENERAL CHALLENGES:
VACUUM & SAMPLE

Experiments

- Spectroscopy (detection of e^-, $h\nu$, ions): <u>UHV necessary</u>
- X-ray Microscopy, soft matter, biological tissue: <u>UHV necessary</u>
- Resonant, non-resonant Scattering (photon detection): <u>UHV necessary</u>
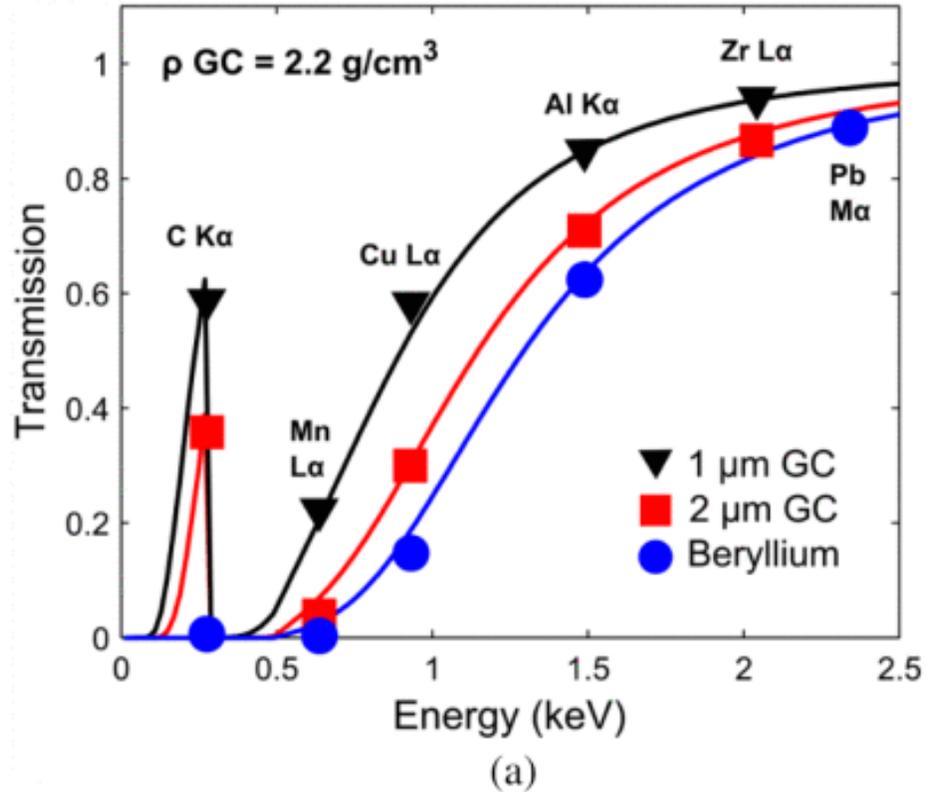- X-ray-diffraction (in air)

Matter to be explored

- Solids, Crystals
- Atoms, molecules, clusters
- Nanoparticles
- Liquids
- Surfaces

Differential pumping stage, UE56, HZB / FHI

GENERAL CHALLENGES:
WINDOWS

	Beryllium	Polymers (Kapton)	SiN	Graphenic Carbon	Quartz	Diamond	Aluminum
scattering properties							
Transparency (E)	+	++	++	++	+	++	+ >10 keV
coherent (Bragg) scattering	-	++	-	-	++	-	-
physical properties							
mechanical strength	+	-	+/-	+/-	+	++	++
chemical resistivity	?	?			+		
thermal properties	conducting	insulating			insulating		conducting
construction suitability							
machinability	--	+	-	-	-	-	++
welding/soldering/ possibilities	+	-	-	-	-	-	+
health aspects							
toxicity	--						
other							
price	-	++	-	-	++	--	++
Typical Applications	"high" pressure windows					highest pressure windows	high pressure windows, low temperatures

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GENERAL CHALLENGES:
TRANSMISSION OF WINDOW MATERIALS

S. Huebner, et al. (2015). High Performance X-Ray Transmission Windows Based on Graphenic Carbon. Nuclear Science, IEEE Transactions on. 62. 10.1109/TNS.2015.2396116.

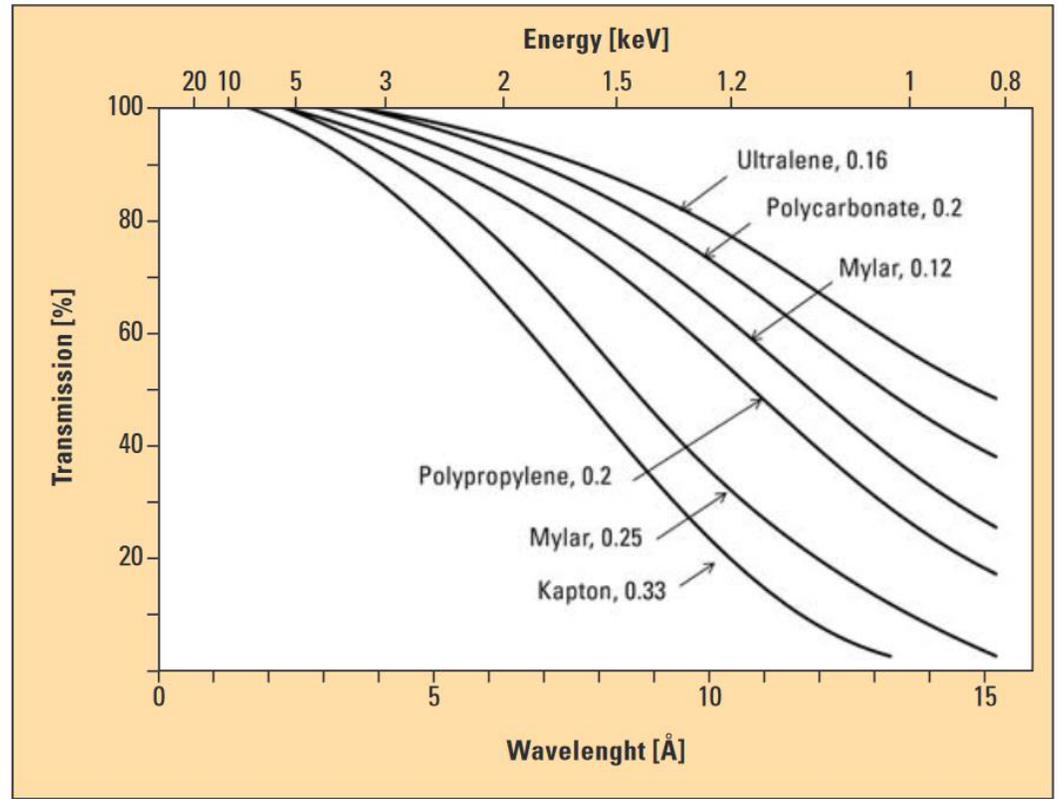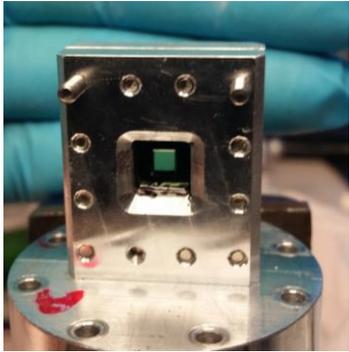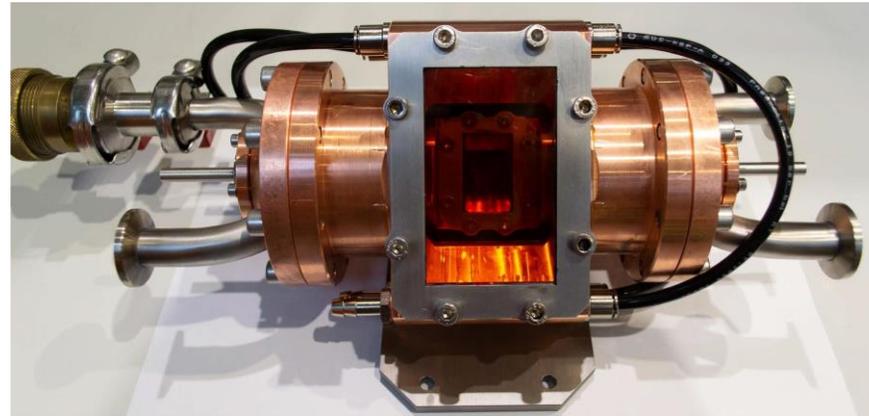N. Grimm (HZB) cleaning up the mess from a busted Berillium window

Figure 2: Transmission capacity of various polymer films as a function of the wavelength of the incident X-ray radiation. Lighter elements exhibit a higher wavelength and a lower energy. They are found at the right-hand side of the graphic.

Shimadzu News 1/2012

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GENERAL CHALLENGES:
WINDOWS

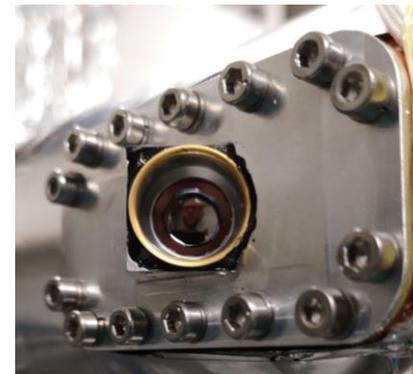SiN – membrane (200nm) in gas adsorption cell for ASAXS and XAS, HZB
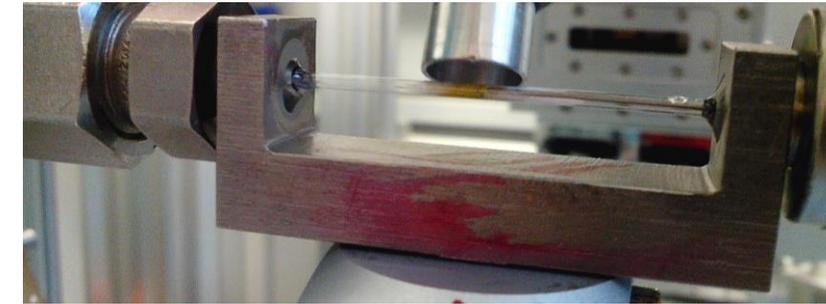

Kapton: miniature cryostat, HZB

Kapton: vapor chamber, HZB

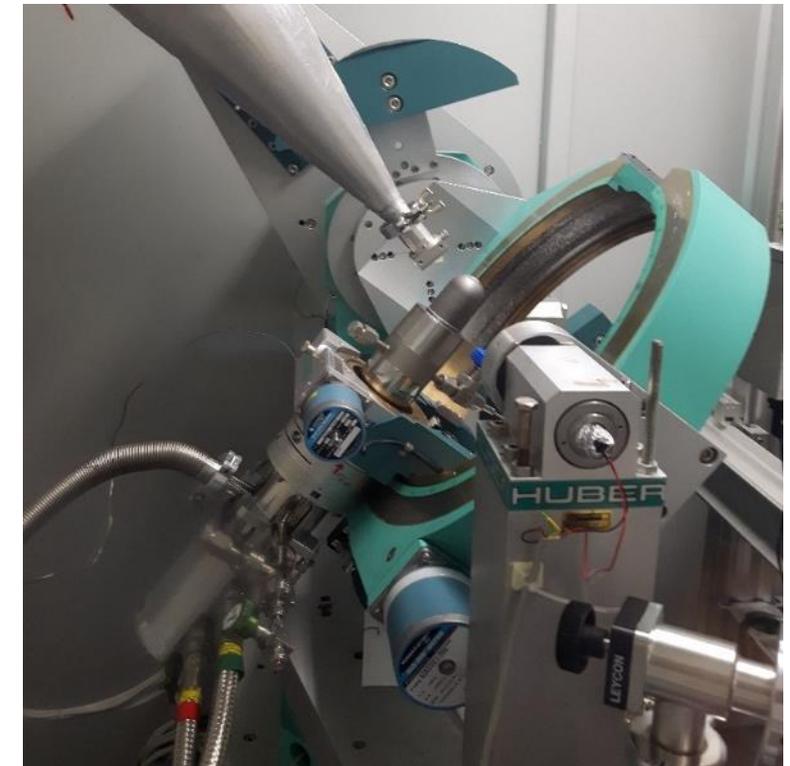

Ultralene membrane (4µ) for Omicron compatible liquid sample cells, KMC-1, HZB

Graphenic carbon window for XES at PINK-beamline, HZB

Quartz tubes: cryo- and hot air jets, KMC-2, HZB

Berillium: gas adsorption & cryostat, KMC-2, HZB

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SAMPLE ENVIRONMENT FOR T, B, P:
TEMPERATURE

- Low temperatures (closed cycle, Helium, Nitrogen, flow-cryostats, bath-cryostats, Peltier,…)

- Very low temperatures (^{3}He, ^{3}He/^{4}He dilution)

Mini Peltier sample stage for Bruker D8 series (200K – 320K)
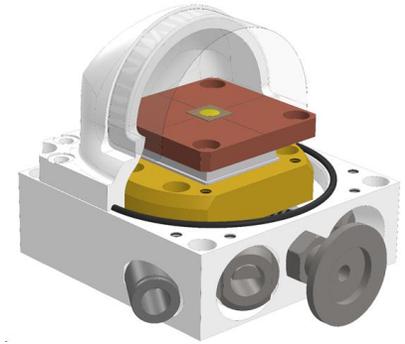

Miniature Stirling cooler (35-320 K)

Gifford McMahon cooler (4 -600 K)

Automatic He-transfer for 7T horizontal cryomagnet: High Field Diffractometer UE46_PGM-1, HZB

N-HeliX, OxfordCryosystems (T < 60 K)

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SAMPLE ENVIRONMENT FOR T, B, P:
TEMPERATURE

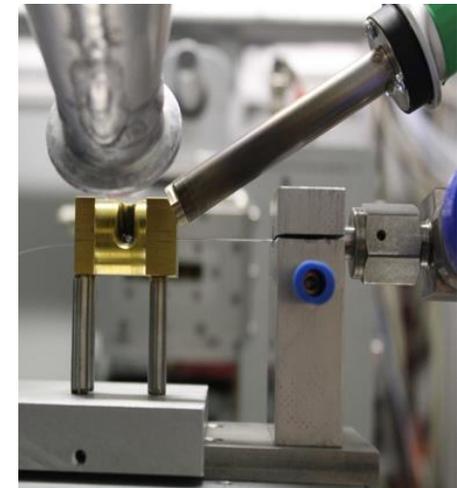- High temperatures (resistive, radiation, levitation, hot air blower, Peltier,…)

KMC-2 at BESSY-II: custom-build Beryllium dome furnace with double sided boralectric® heater reaches temperatures up to 1000 °C in vacuum (< 10-9 mbar)

Halogen-bulb **Mirror Furnace**. Sample contained in Pt capillary heated to ~1500°C (Andrew N. Fitch, ID22, ESRF)
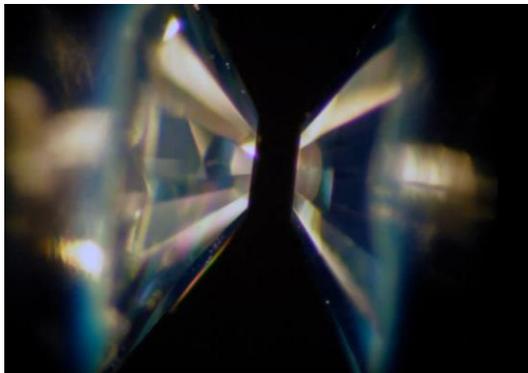
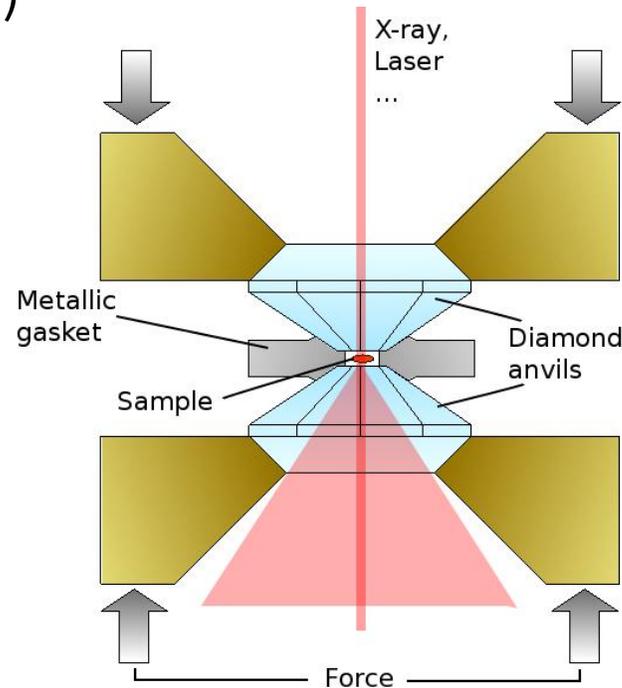See as well (alternative design): Rev. Sci. Instrum. **91**, 065109 (2020); https://doi.org/10.1063/1.5141139

Hot-air-jet at KMC-2 beamline of BESSY-II 200-800 K

SAMPLE ENVIRONMENT FOR T, B, P:
PRESSURE

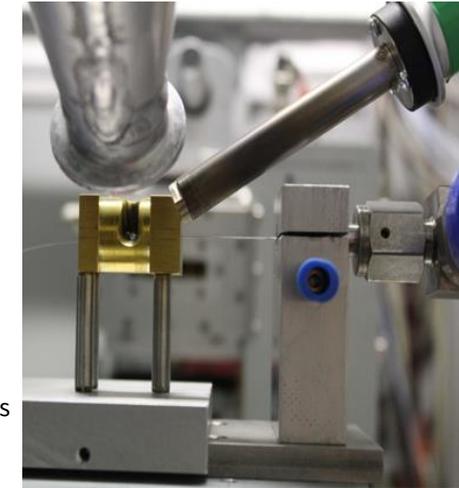- Clamped cells (dimaond anvil cells, some 100 GPa)

- Gas pressure (some 100 bar)

- Near ambient pressure

  (some 10 mbar)

Gas adsorption in glass capillaries
up to 100 bar, 200 - 800 K
at KMC-2 beamline (BESSY-II)

Diamond anvils (D. Haase, MAX IV)

Sketch of a diamond anvil cell
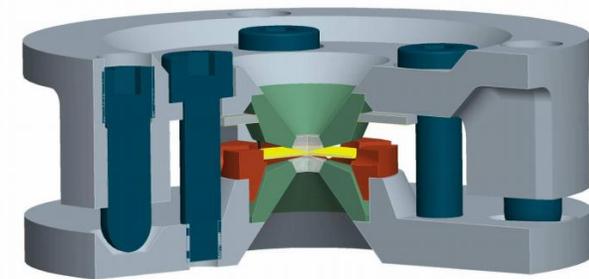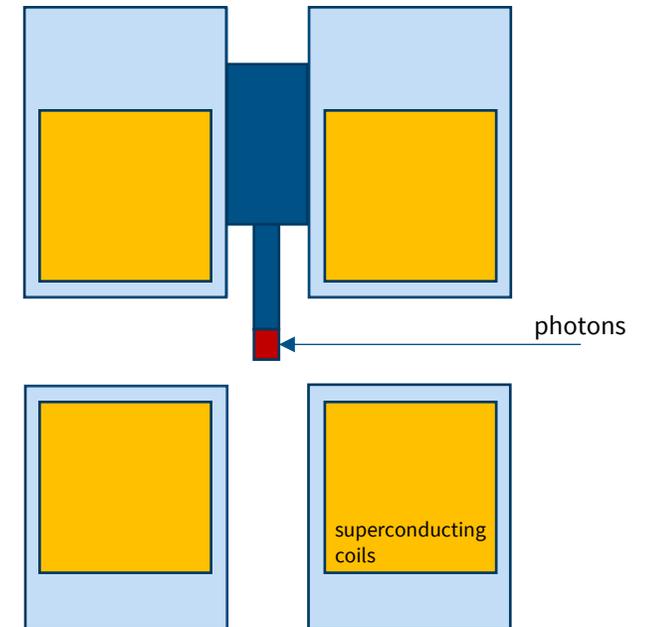Illustration by Sebastian Merkel, Lille

Figure 2: Cross section of Diacell® Bragg XVue.

DAC with side access for XRD (Illustration from Almax
easylab technical notes for Plate DAC)
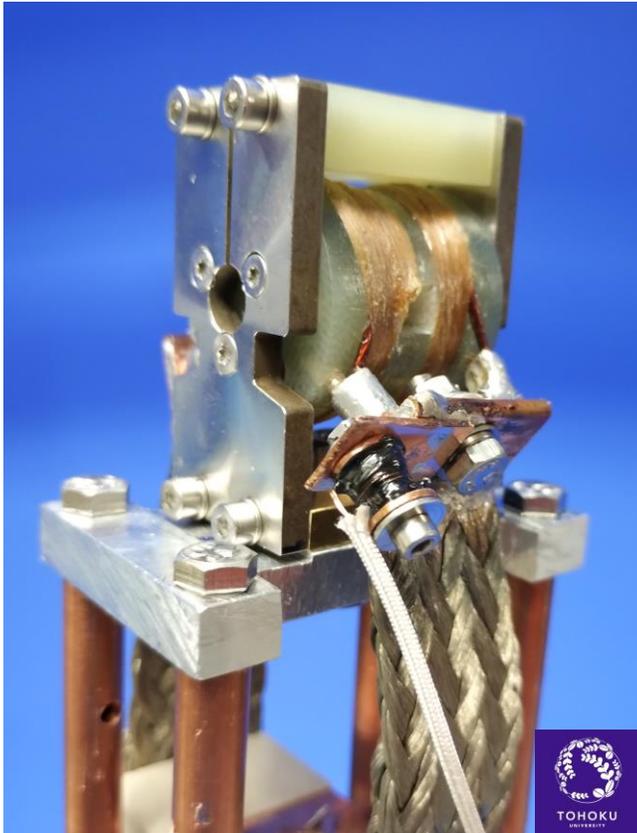
SAMPLE ENVIRONMENT FOR T, B, P:
MAGNETIC FIELD

Vekmag at BESSY-II:

- 9T – 2T – 1T vector magnet
- different temperature inserts
- UHV sample space
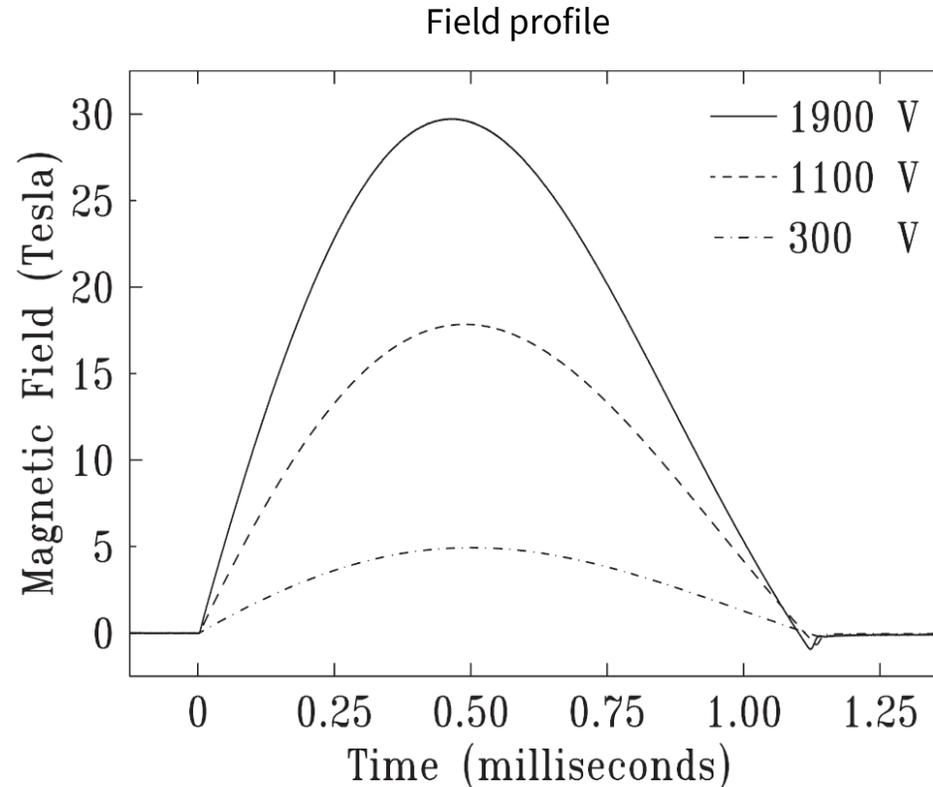- liquid Helium bath
- recondensing

photons

superconducting coils

Split pair magnet

SAMPLE ENVIRONMENT FOR T, B, P:
PULSED MAGNETIC FIELDS

Coil

30 Tesla split-coil magnet (Hiroyuki Nojiri)

Field profile

Cryostat

Z. Islam, J. Ruff, Hiroyuki Nojiri, et al., Rev. Sci. Instrum. **80**, 113902 (2009); https://doi.org/10.1063/1.3251273
G. T. Noe, H. Nojiri, et al., Rev. Sci. Instrum. **84**, 123906 (2013); https://doi.org/10.1063/1.4850675

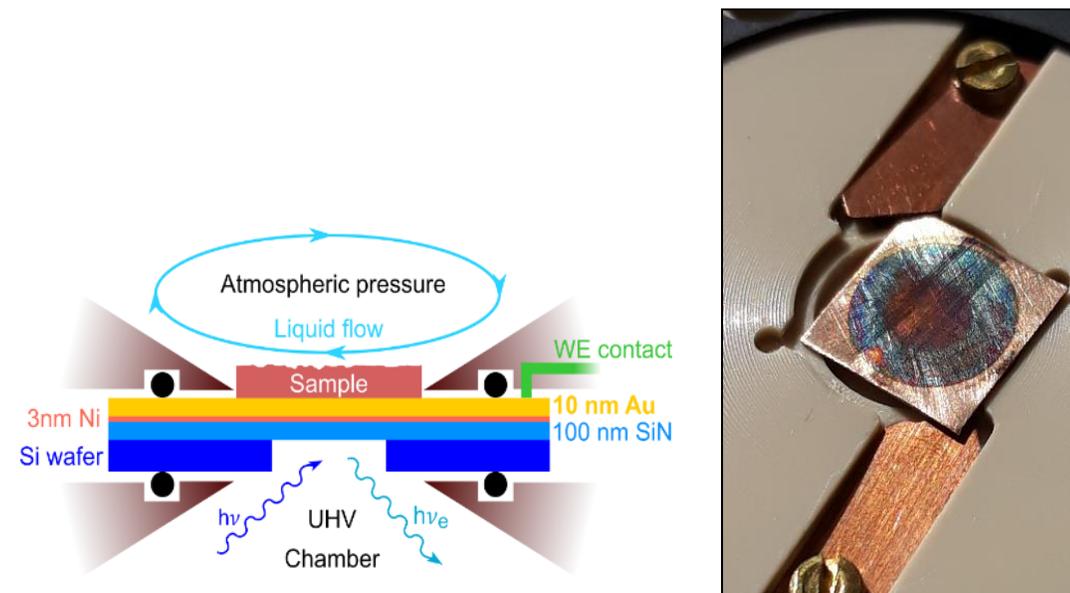# IN-SITU, OPERANDO & OTHER COMPLEX THINGS

BATTERY RESEARCH

In-operando cells for battery research / X-ray Imaging

S8 formation in lithium/sulfur batteries during cyclic voltammetry

*S. Risse et al., J. Phys. Chem. Lett. 11 (**2020**) 5674−5679*
Video: https://doi.org/10.1021/acs.jpclett.0c01284
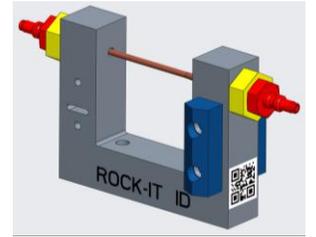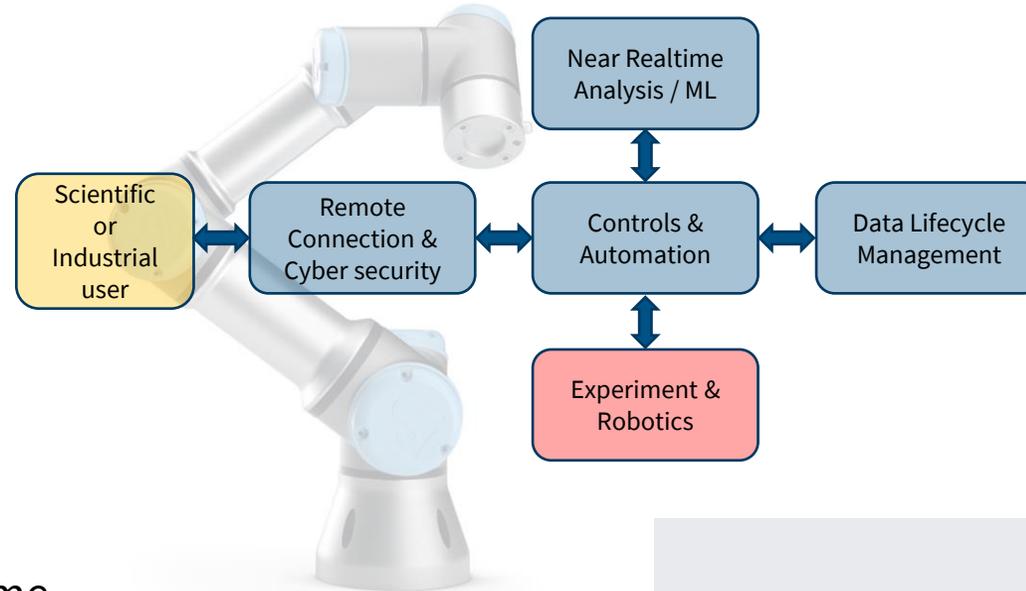
ELECTROCHEMICAL REACTIONS

In operando electrochemical flow-through cell for XES / XAS (OÆSiS in EMIL@BESSYII)

- Electrochemistry with 3-electrode design
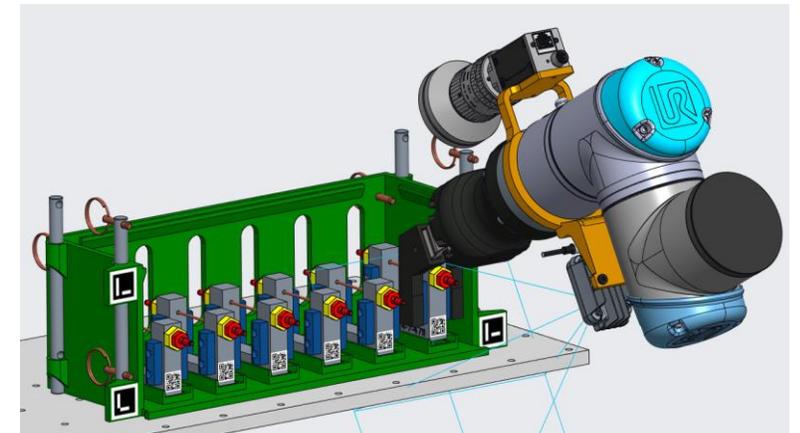- Vacuum and liquid separated by 100 nm SiN membrane

D. Wallacher, R. Garcia Diez, M. Bär et al.

IN-SITU, OPERANDO & OTHER COMPLEX THINGS:
CATALYSIS

- Fully automised operando catalytic experime
- Standardised sample holder
- Standardised gas supply and analysis layout
- Standard software interfaces for interchangeable equipment
- Safe remote access
- AI controlled experiments
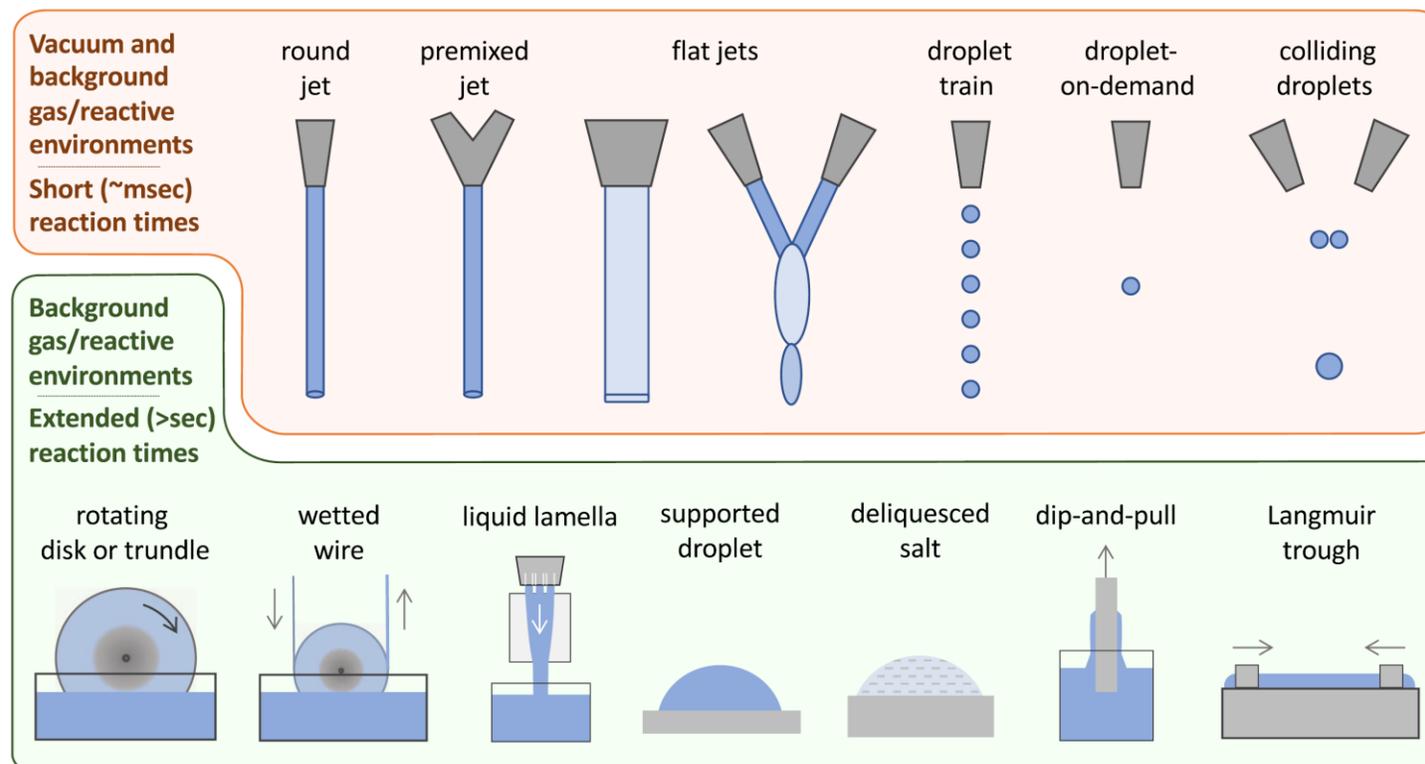
IN-SITU, OPERANDO & OTHER COMPLEX THINGS: LIQUIDS
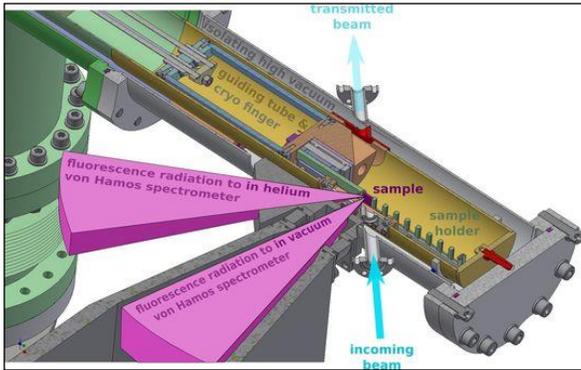
Liquid jets et al.

Flat jet, HZB

FIG. 4. Preparation of liquid–vapor interfaces inside vacuum chambers. The upper row shows methods based on fast moving jets or droplets that have a very brief interaction time with the surrounding environment before being measured. These methods can be used in a vacuum environment since the liquid reservoir is outside of the vacuum chamber. The bottom row shows preparation methods where the bulk reservoir of the liquid is inside the vacuum chamber and the measurements thus are done in the presence of the equilibrium vapor pressure. These investigations permit longer interaction times between gases and the liquid.

SAMPLE POSITIONING

attocube piezo actuator

PINK: Cryostat for X-Ray Emission Spectroscopy @ EMIL (HZB)

Temperature 20K .. 300 K

S. DeBeer, S. Peredkov, R. Ringel, K. Martin, L. Drescher,
D. Wallacher, R. Grüneberger, B. Klemke, H. Thiel

Nanopositioning: OMNY at PSI

Tomography nano cryo endstation

Temperature 90K (LN2), 10 K (LHe)

M. Holler, Jörg Raabe, et al., PSI
*Review of Scientific Instruments **89**, 043706 (2018); https://doi.org/10.1063/1.5020247*

PAUL SCHERRER INSTITUT

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SAMPLE MOUNTING:
STANDARDIZED FLAG STYLE SAMPLE PLATES

Omicron

www.scientaomicron.com

Ferrovac

www.ferrovac.com

www.esrf.eu

CoESCA station, T. Leitner, HZB:
put sample into preparation chamber

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SAMPLE MOUNTING:
STANDARDIZED SAMPLE HOLDERS FOR MX

Macromolecular Crystallography

Example:

Macromolecular crystallography beamline BL14.2 (HZB)

UNIPUCK **SPINE PUCK**

G-rob sample changer

U. Mueller et al. (2012). J. Synchrotron Rad. 19, 442-449
U. Mueller et al. (2016). Eur. Phys. J. Plus 130, 141-150

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SAMPLE MOUNTING:
STANDARDIZED SAMPLE HOLDERS FOR MX

Macromolecular Crystallography

Example:

Macromolecular crystallography beamline BL14.2 (HZB)

G-rob sample changer

Video by C. Feiler (HZB)

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STANDARDISATION:
THE SAMPLE ENVIRONMEMT COMMUNICATION PROTOCOL

Sample Environment Communication Protocol
simple, inclusive, self explaining
provides metadata

High **abstraction** of the functionality

Agnostic to specific control system

Complete metadata provision

Machine readable information for plug&play

International standard

STANDARDISATION:
THE SAMPLE ENVIRONMEMT COMMUNICATION PROTOCOL

Sample Environment Communication Protocol
simple, inclusive, self explaining
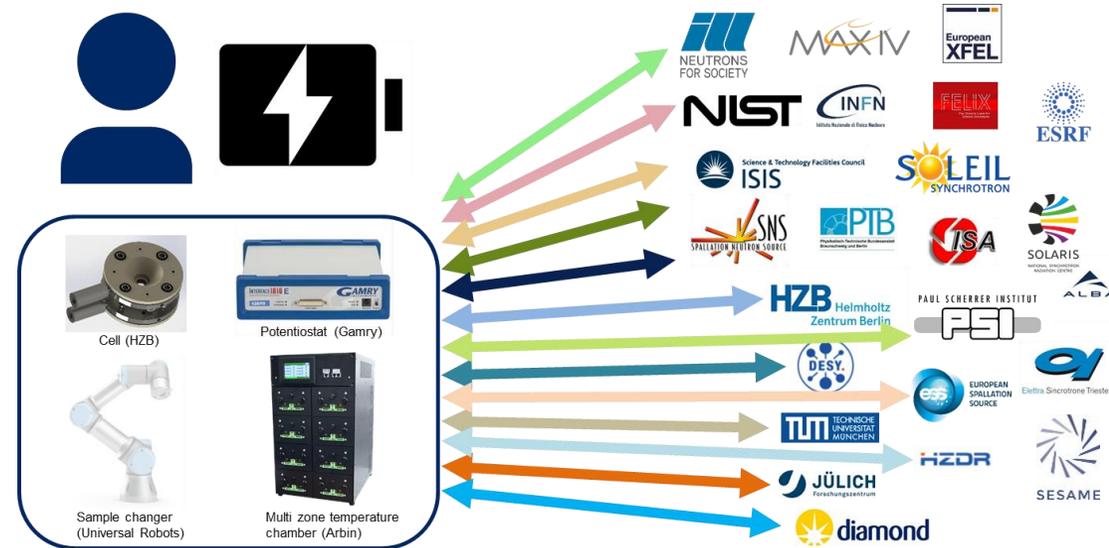**provides metadata**

High **abstraction** of the functionality

Agnostic to specific control system

Complete metadata provision

Machine readable information for plug&play

International standard

STANDARDISATION:
THE SAMPLE ENVIRONMEMT COMMUNICATION PROTOCOL

Sample Environment Communication Protocol
simple, inclusive, self explaining
provides metadata

Benefits:

- **Faster integration** of sample environment
- **Complete** data & metadata sets
- Faster **analysis**
- **More efficient** experiments

CONCLUSION (FIRST PART)

- Sample environment is crucial

- It is getting more complex

- Some things you just buy, others have to be customized or developed from scratch

- Standardisation and collaboration helps

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