SAMPLE ENVIRONMENTS AT A SYNCHROTRON

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

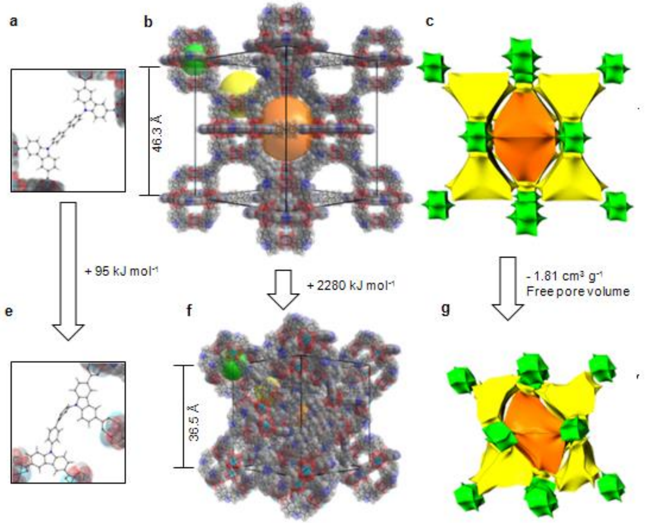
School on Synchrotron Light Sources and their Applications | (SMR 3815), 26 January 2023

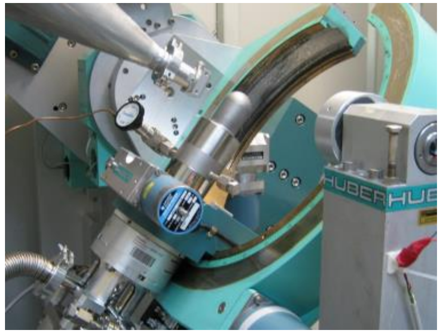
Sample Environment

What is sample environment?

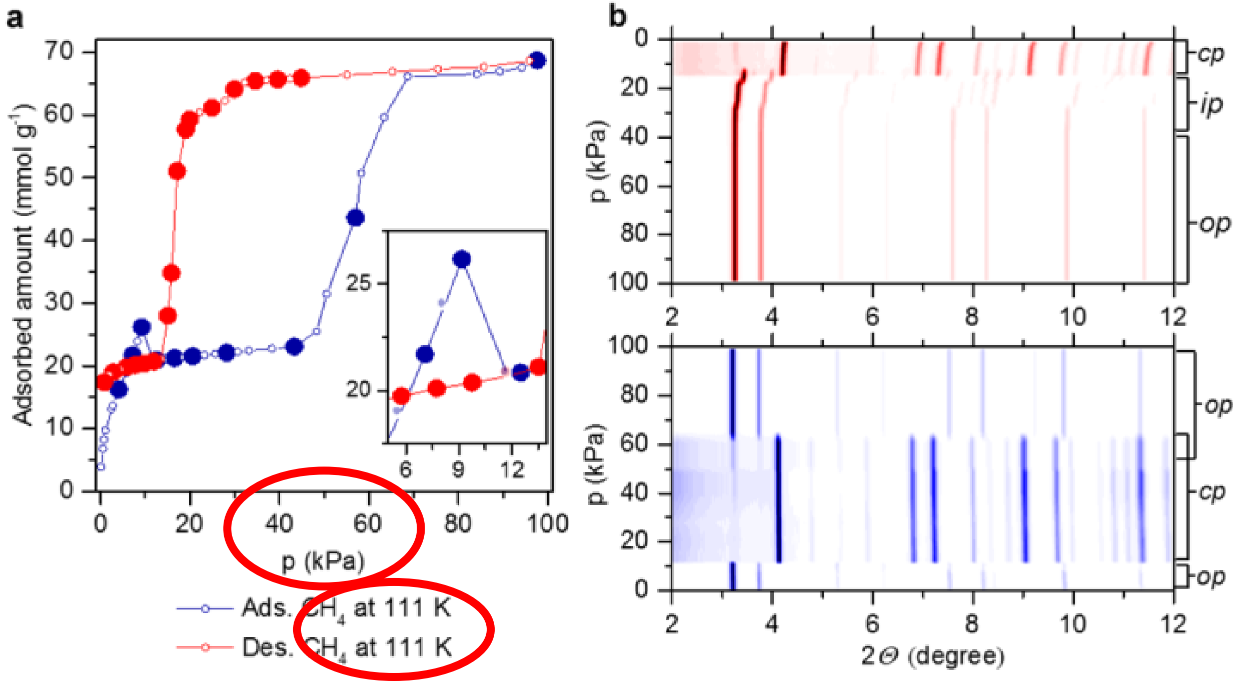
- Physical parameters: temperature, pressure, magnetic field, electric field, …
- In-situ / in-operando experiments: catalysis, energy storage, …
- Sample positioning: nano-positioning, rotation, …
- Sample mounting
- Everything that effects the sample - minus the actual measurement

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)

What is sample environment?
knowledge & people

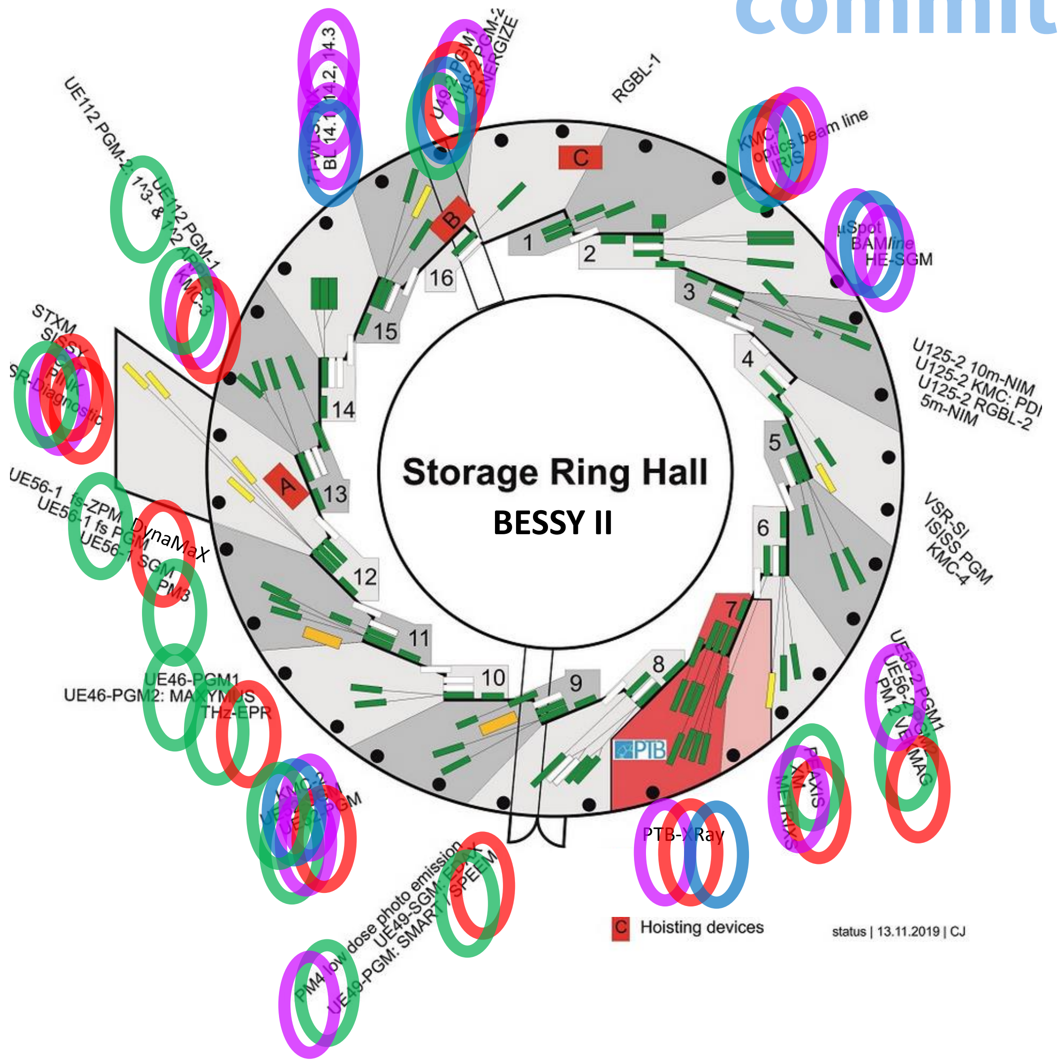
Scientists

Engineers

Technicians

Students

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)

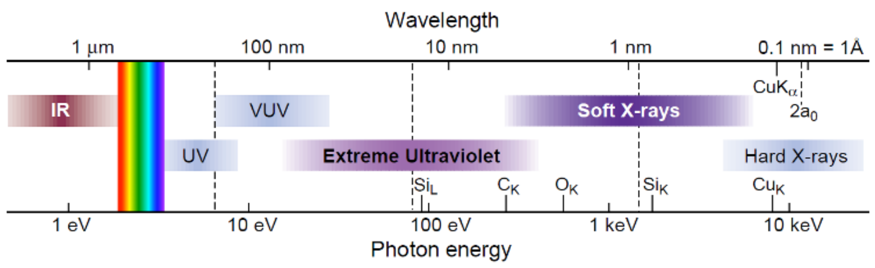
Overview

- What is sample environment?
- General challenges
- Sample environment for T, B, p
- In-situ, in-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

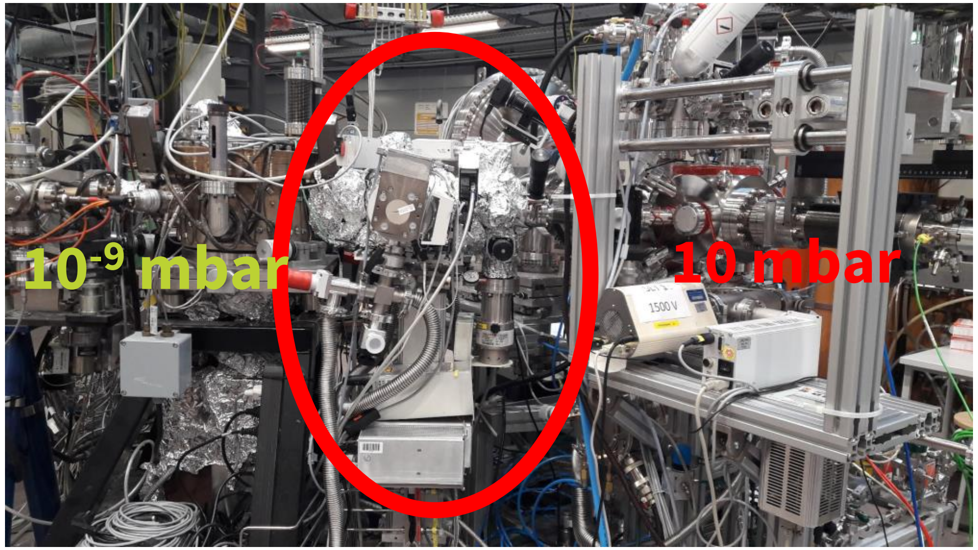


Experiments

- Spectroscopy (detection of e$^-$, hν, ions): UHV necessary
- X-ray Microscopy, soft matter, biological tissue: UHV necessary
- Resonant, non-resonant Scattering (photon detection): UHV necessary
- X-ray-diffraction (in air)

Matter to be explored

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

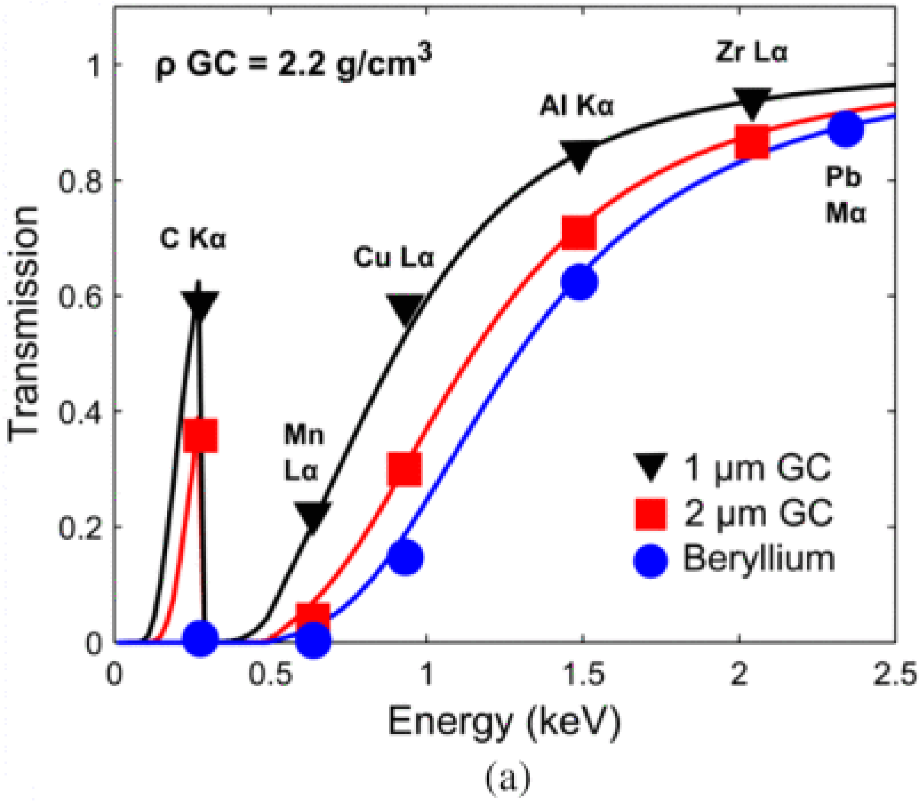


Differential pumping stage, UE56, HZB / FHI

General challenges: windows

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

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

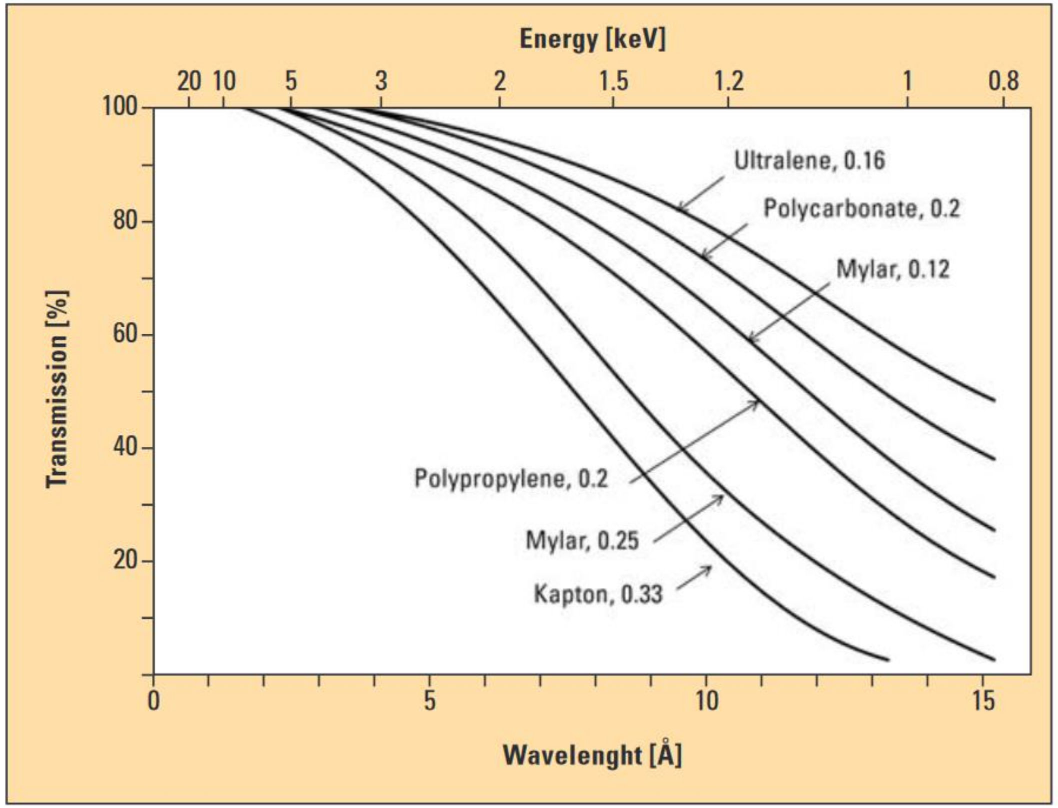
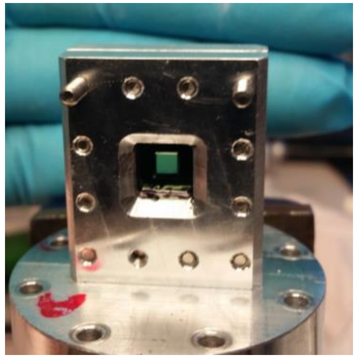


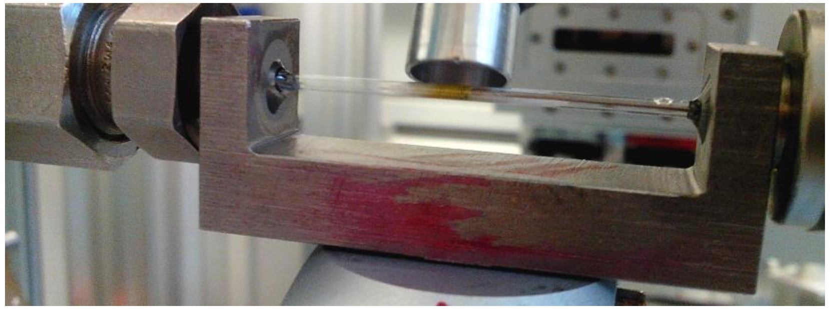
Figure 2: Transmission capacity of various polymer films as a function of the wave-length of the incident X-ray radiation. Lighter elements exhibit a higher wavelength and a lower energy. They are found at the right-hand side of the graphic.

Shimadzu News 1/2012

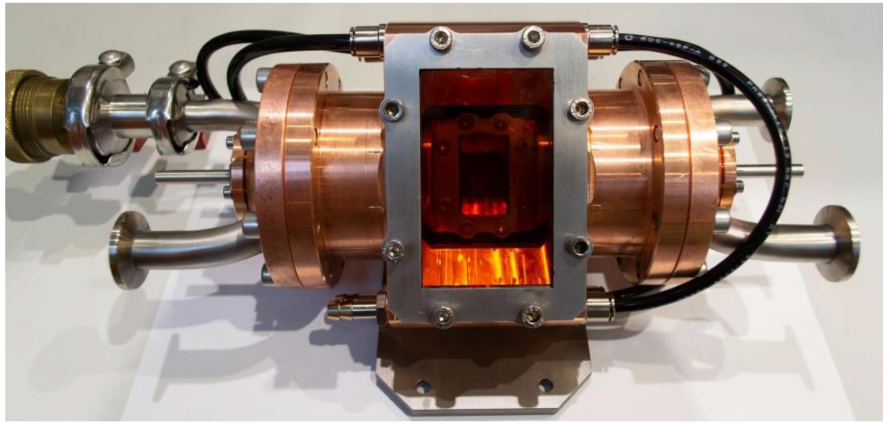
General challenges: windows



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



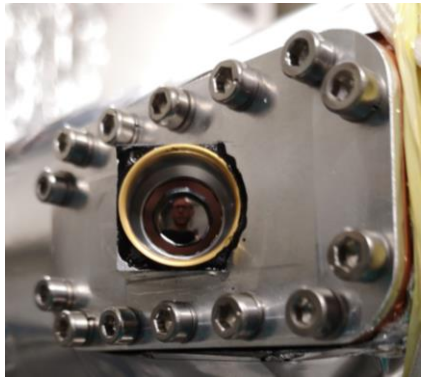
Quartz tubes: cryo- and hot air jets, KMC-2, HZB



Kapton: vapor chamber, HZB

Kapton: miniature cryostat, HZB

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

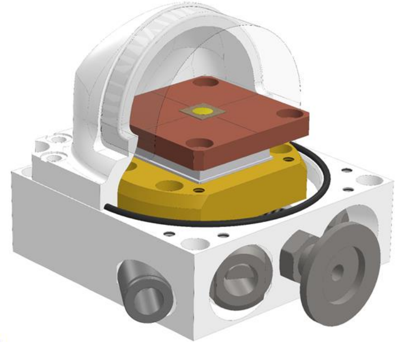


Graphenic carbon window for XES at PINK-beamline, HZB

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

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)

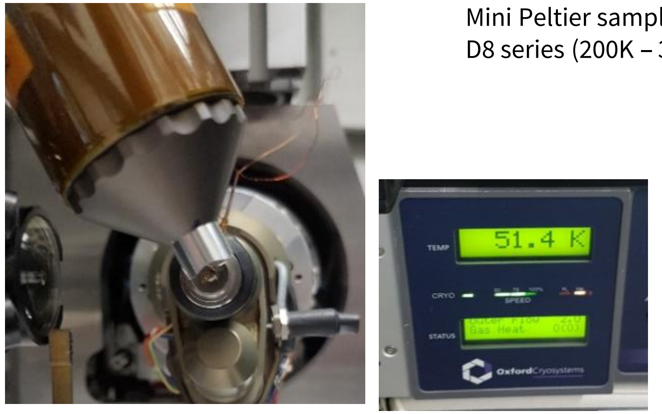


Mini Peltier sample stage for Bruker D8 series (200K – 320K)

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)

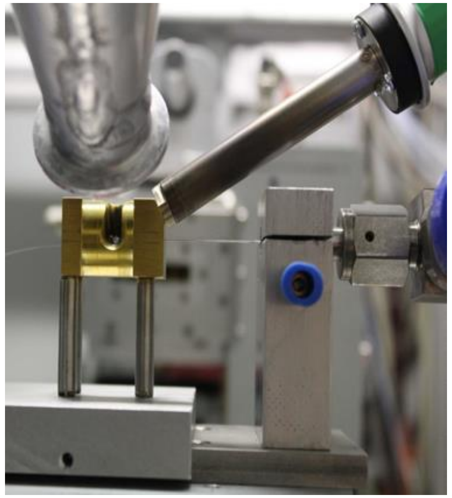
Sample Environment for T, B, p: temperature

- 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)

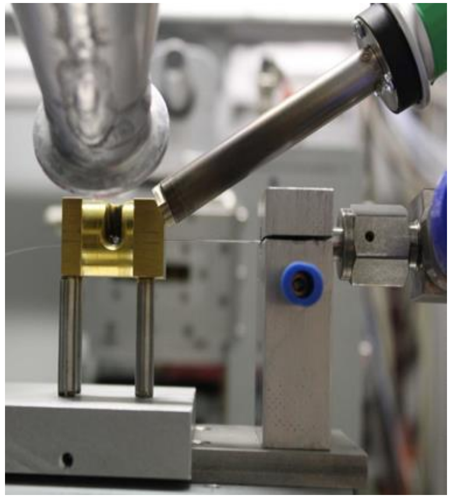
See as well (alternative design): Rev. Sci. Instrum. **91**, 065109 (2020); https://doi.org/10.1063/1.5141139



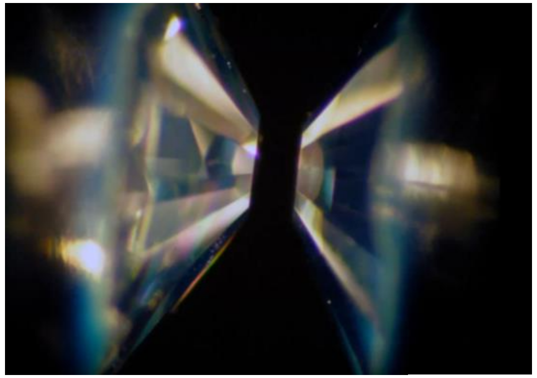
Hot-air-jet at KMC-2 beamline of BESSY-II
200-800 K

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)

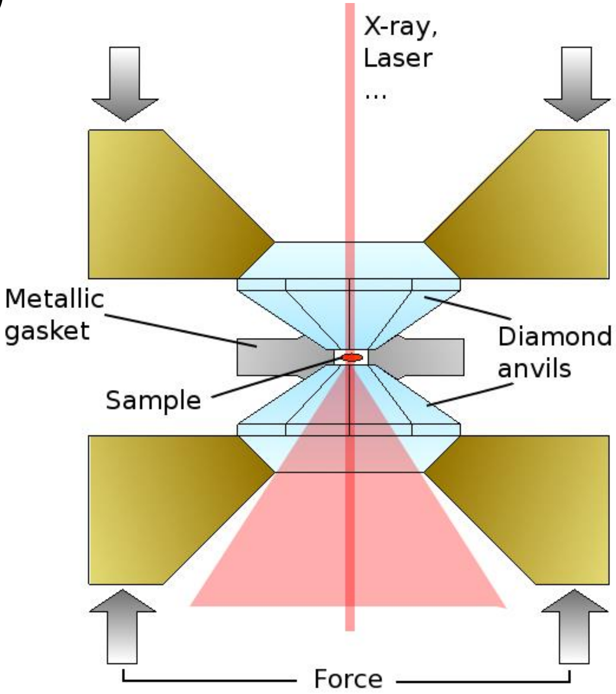


Gas adsorption in glass capillaries
up to 100 bar, 200 - 800 K
at KMC-2 beamline (BESSY-II)



Diamond anvils (D. Haase, MAX IV)

MAX IV



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

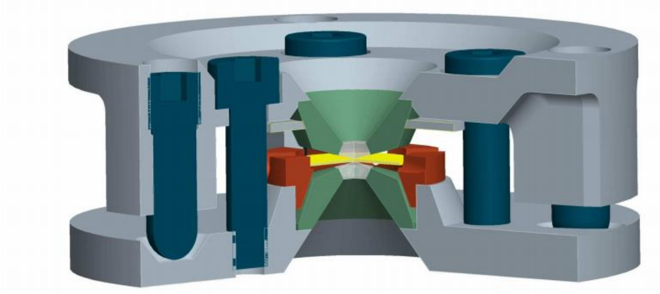


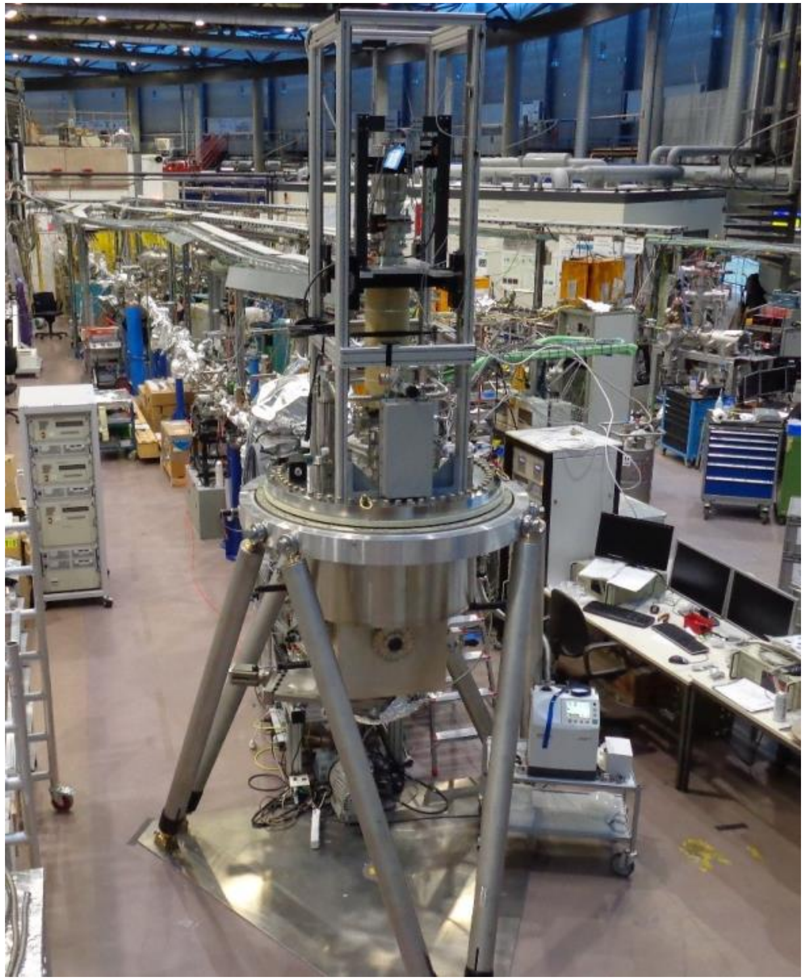
Figure 2: Cross section of Diacell® Bragg XVue.

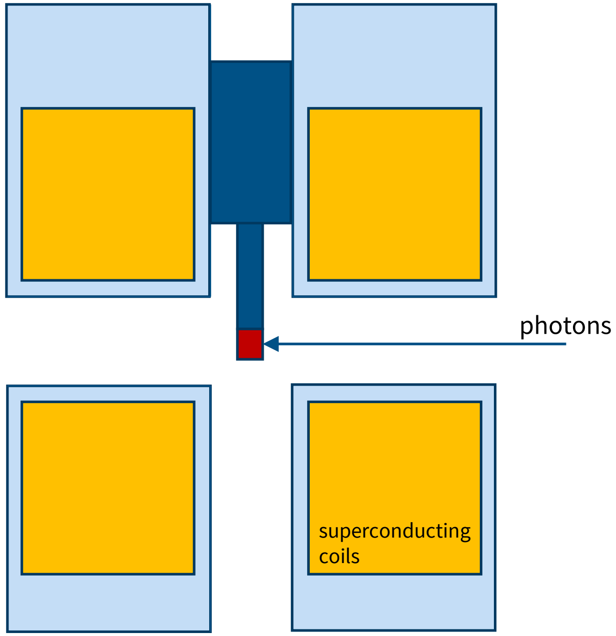
DAC with side access for XRD (Illustration from Almax easylab technical notes for Plate DAC)

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

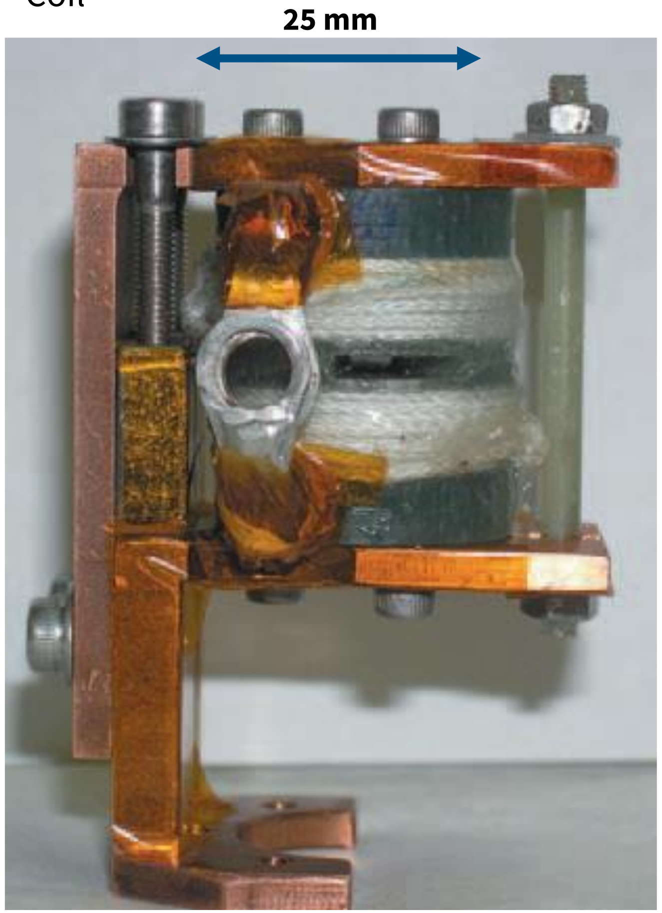




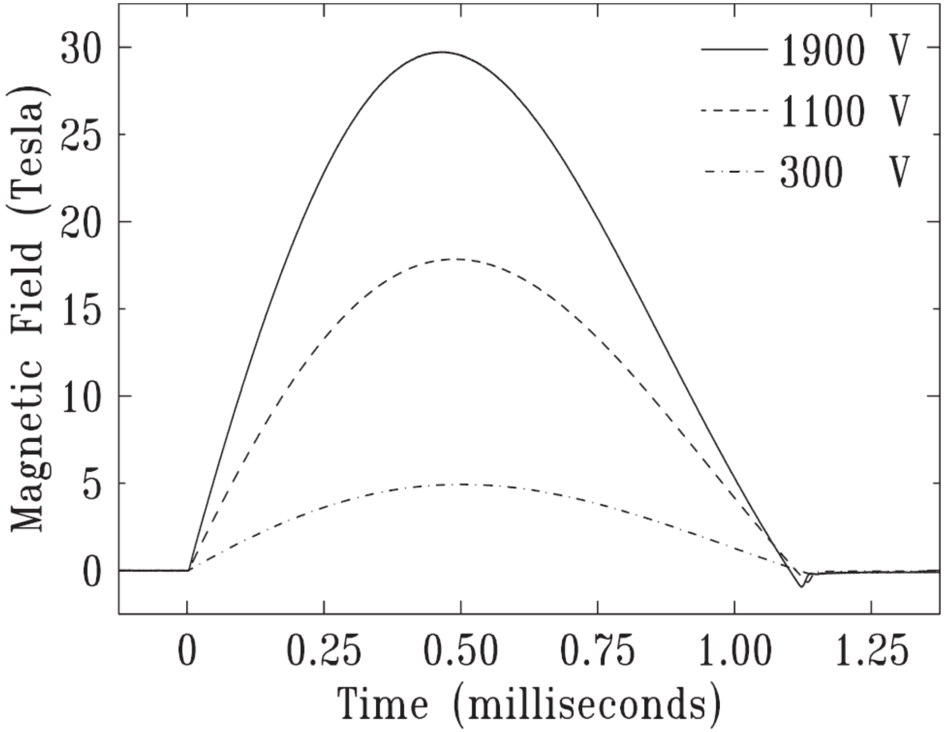
Split pair magnet

Sample Environment for T, B, p: pulsed magnetic fields

Coil

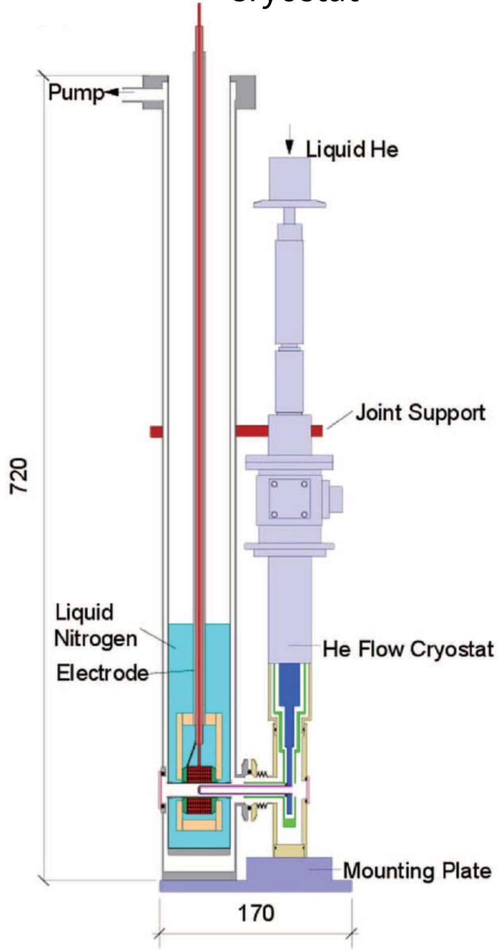


Field profile



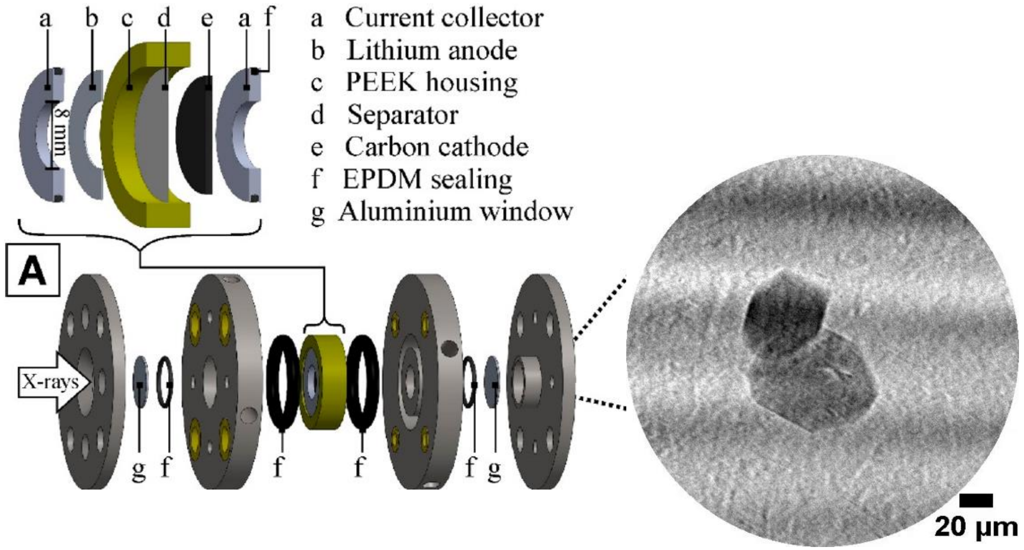
Z. Islam, J. Ruff, Hiroyuki Nojiri, et al., Rev. Sci. Instrum. ***80****, 113902 (2009); https://doi.org/10.1063/1.3251273*
G. T. Noe, H. Nojiri, et al., Rev. Sci. Instrum. ***84****, 123906 (2013); https://doi.org/10.1063/1.4850675*

Cryostat



In-situ, in-operando & other complex things

Battery research



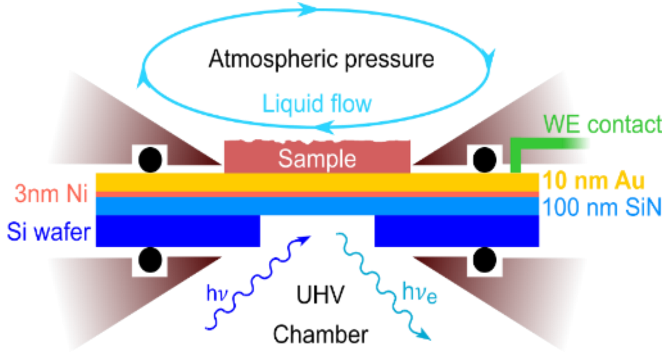
In-operando cells for battery research / X-ray Imaging

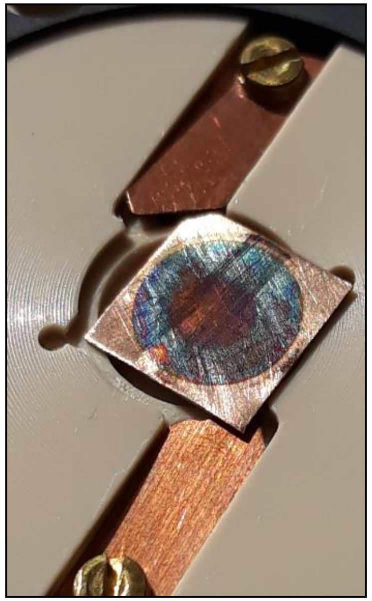
S8 formation in lithium/sulfur batteries during cyclic voltammetry

*S. Risse et al., J. Phys. Chem. Lett. 11 (**2020**) 5674–5679*

Video: https://doi.org/10.1021/acs.jpclett.0c01284

Electro chemical 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, in-operando & other complex things: liquids

Liquid jets et al.

Flat jet, HZB

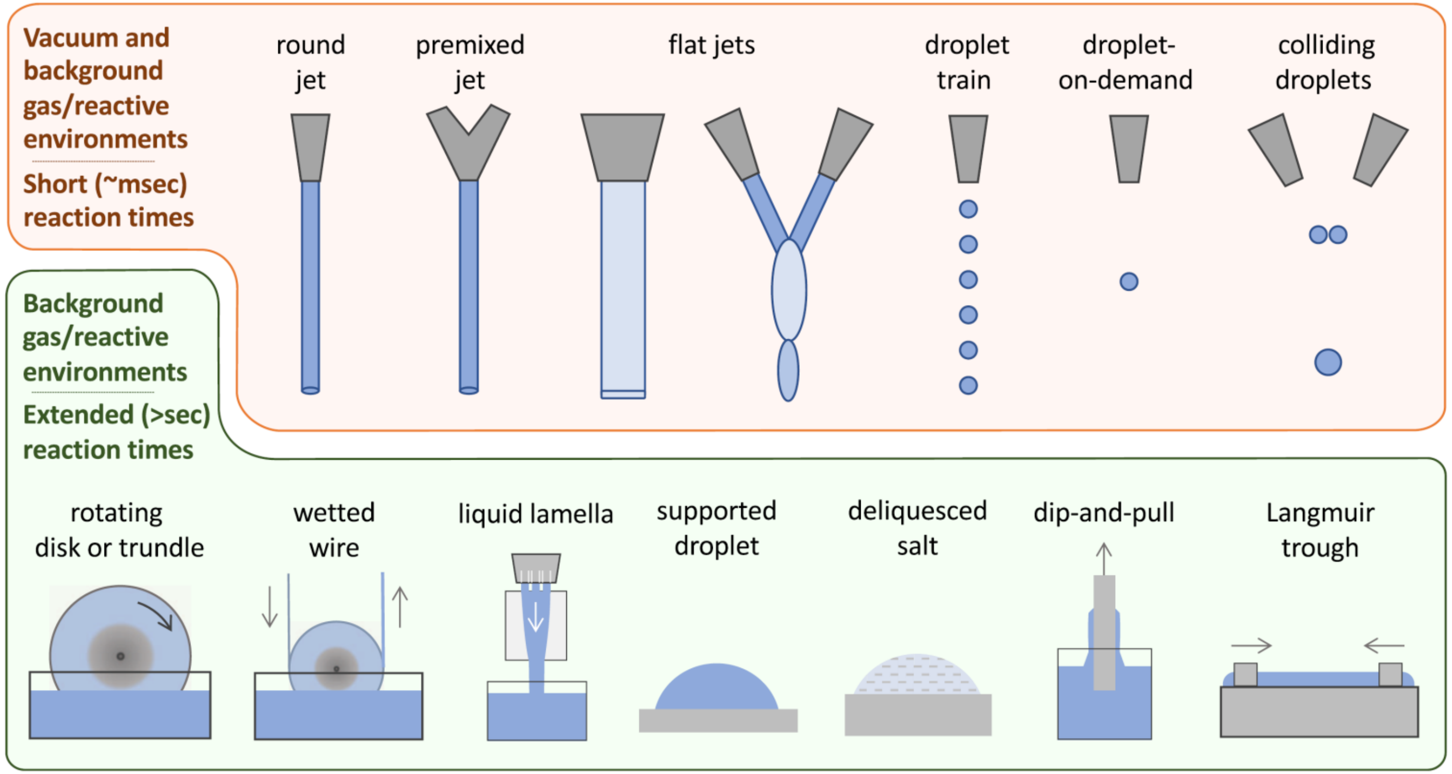
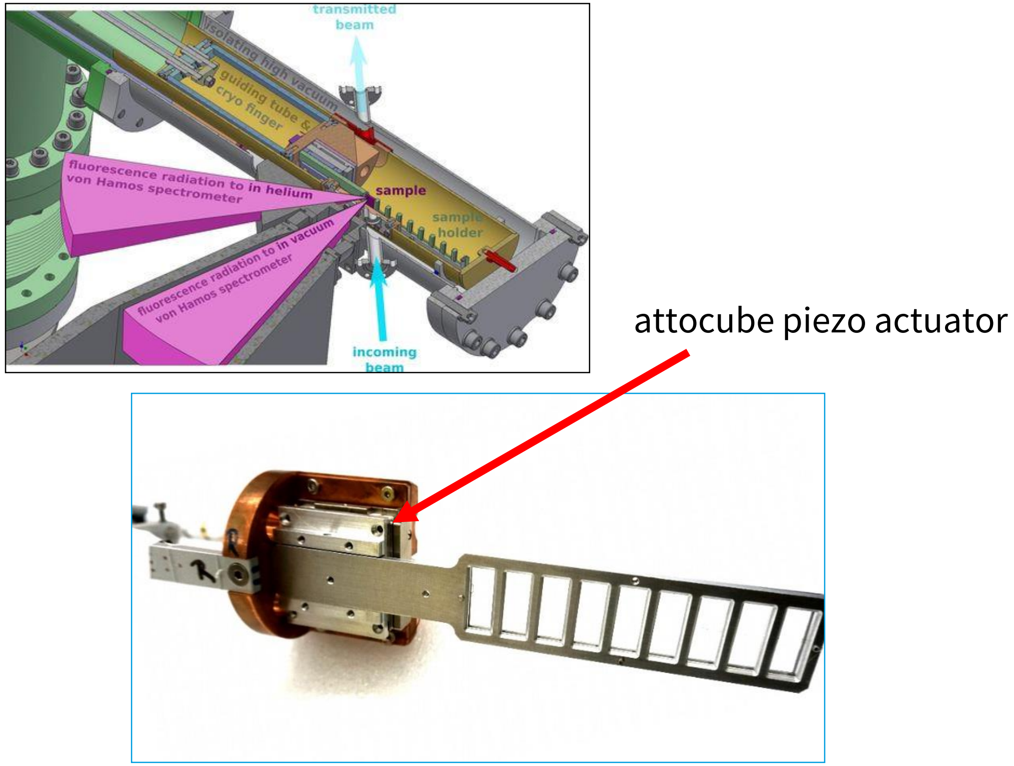
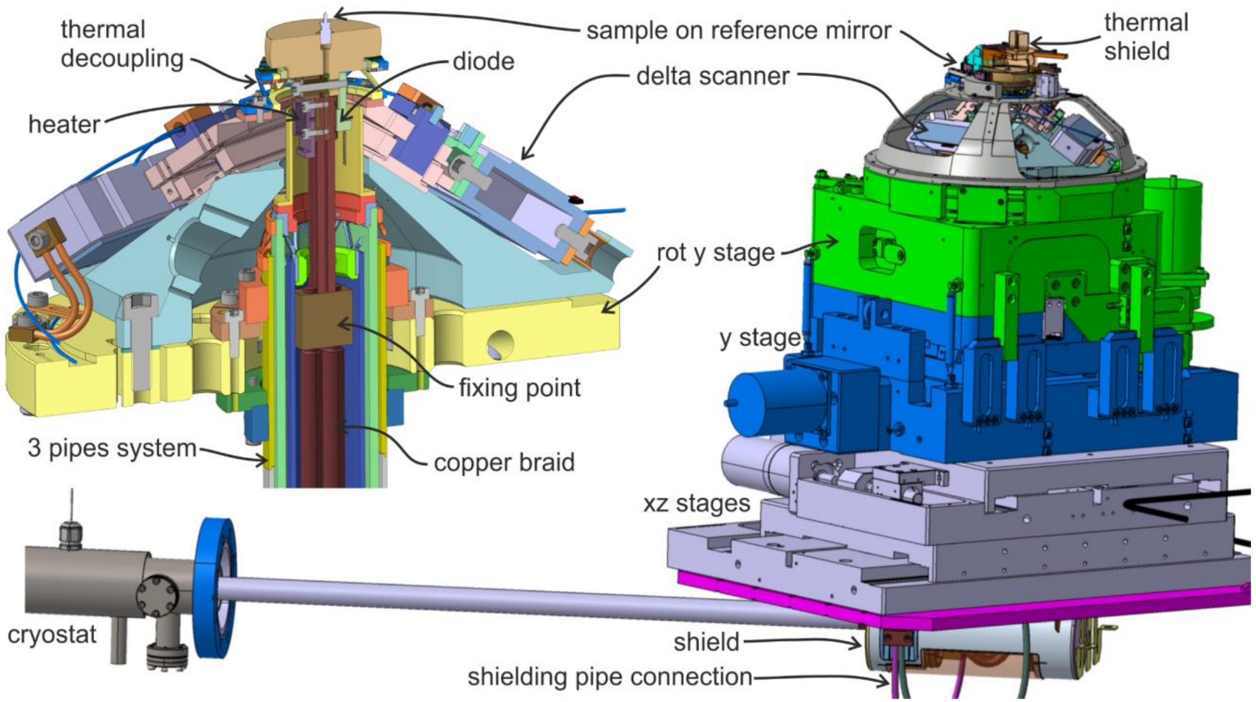


FIG. 4. Preparation of liquid–vapor interfaces inside vacuum chambers. The upper row shows methods based on fast moving jets or droplets that have a very brief interaction time with the surrounding environment before being measured. These methods can be used in a vacuum environment since the liquid reservoir 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.

R. Dupuy, et al., J. Chem. Phys. ***154****, 060901 (2021); doi: 10.1063/5.0036178*

Sample positioning





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 PSI

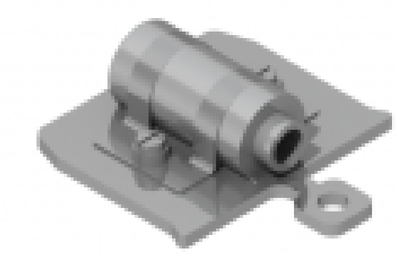
Sample mounting: standardized flag style sample plates

Omicron

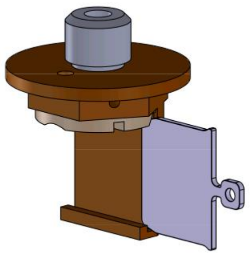
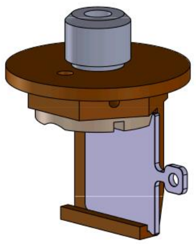
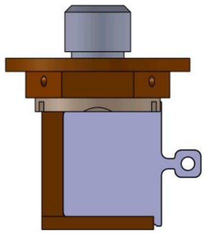


www.scientaomicron.com

Ferrovac



www.ferrovac.com

www.esrf.eu

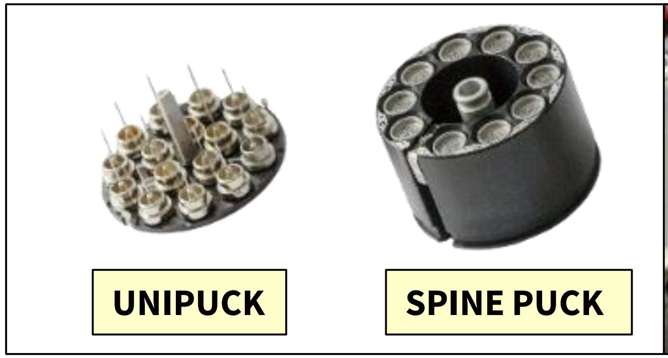
CoESCA station, T. Leitner, HZB:
put sample into preparation chamber

Sample mounting: standardized sample holders for MX

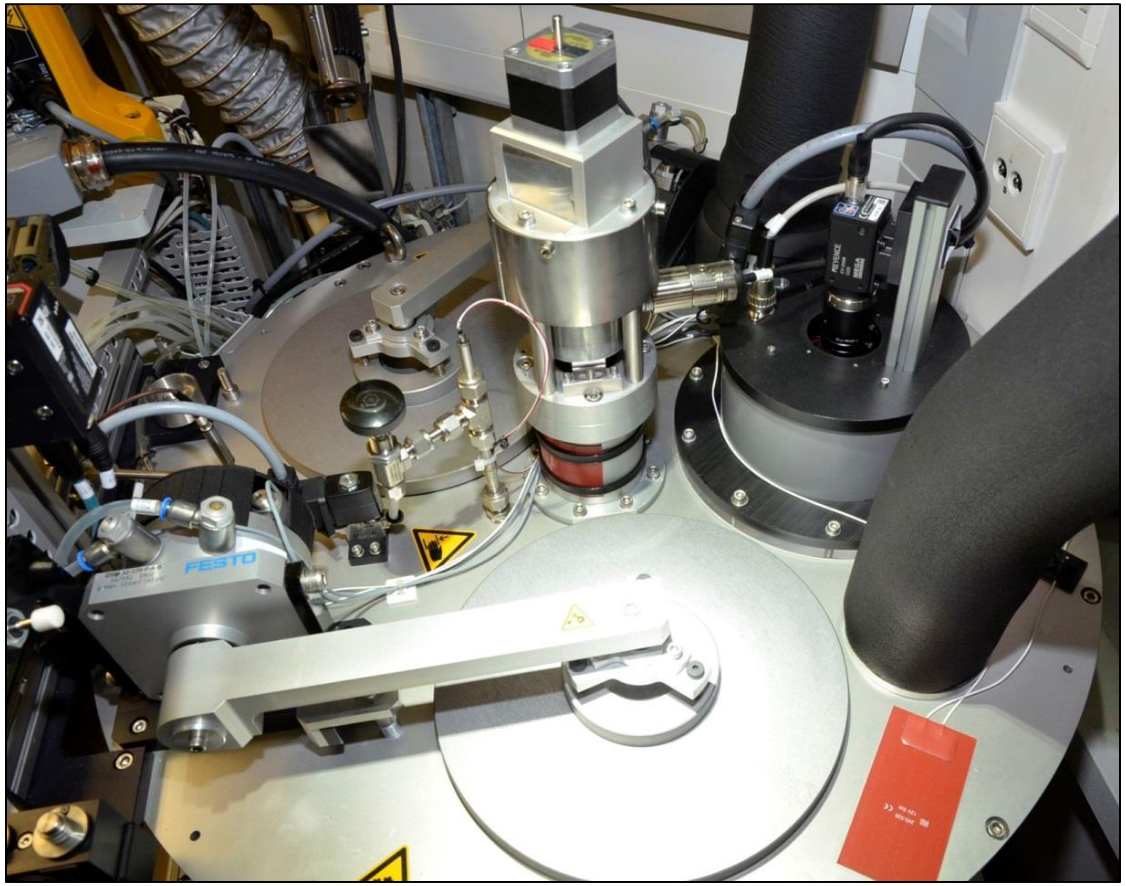
Macromolecular Crystallography

Example:

Macromolecular crystallography beamline BL14.2 (HZB)



G-rob sample changer



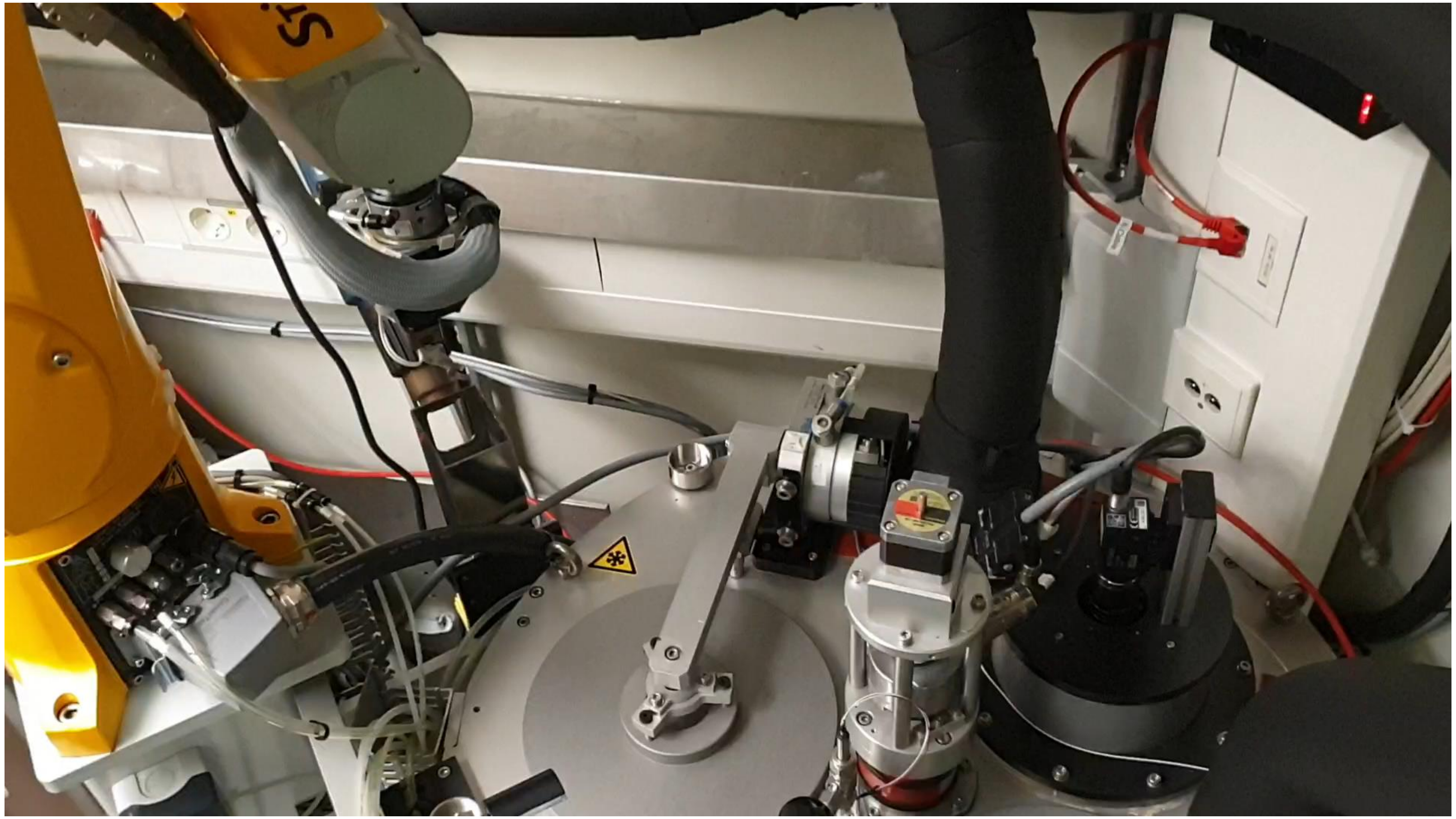
U. Mueller et al. (2012). J. Synchrotron Rad. 19, 442-449
U. Mueller et al. (2016). Eur. Phys. J. Plus 130, 141-150

Sample mounting: standardized sample holders for MX

Macromolecular Crystallography

Example:

Macromolecular crystallography beamline BL14.2 (HZB)



Video by C. Feiler (HZB)

G-rob sample changer

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