

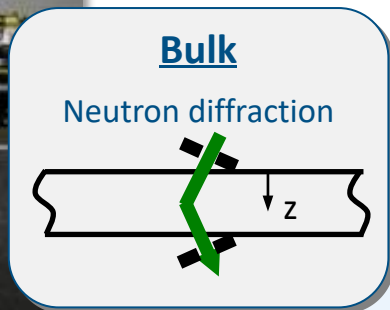
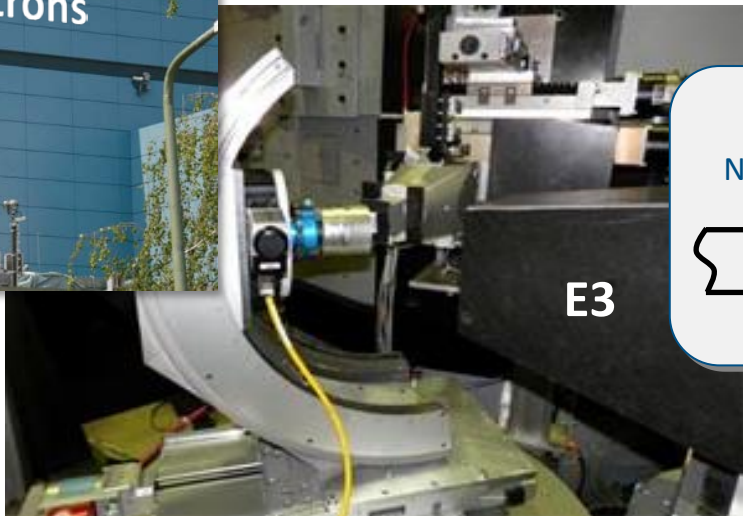
# Operating Liquid MetalJet X-ray Sources for Materials Research

Mirko Boin, Roland Mainz, Manuela Klaus, Daniel Apel, Paul Kamm, Robert Wimpory, Francisco Garcia-Moreno, Guido Wagener, Christoph Genzel

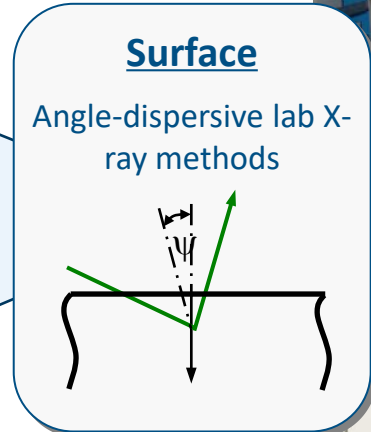
FLS 2023 Workshop, Aug 27 - Sept 1 2023, Lucerne



# Microstructure & Residual Stress Analysis @ HZB



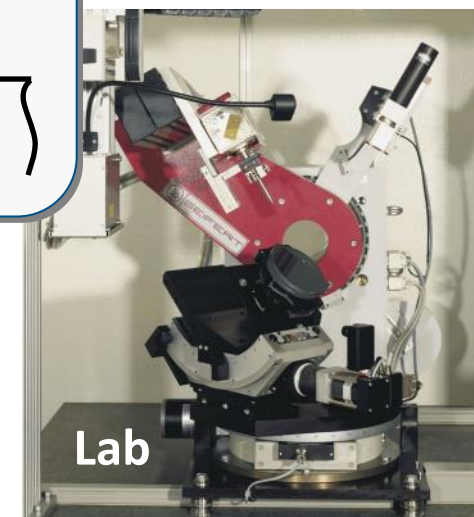
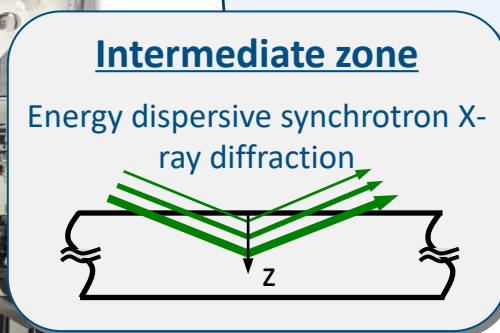
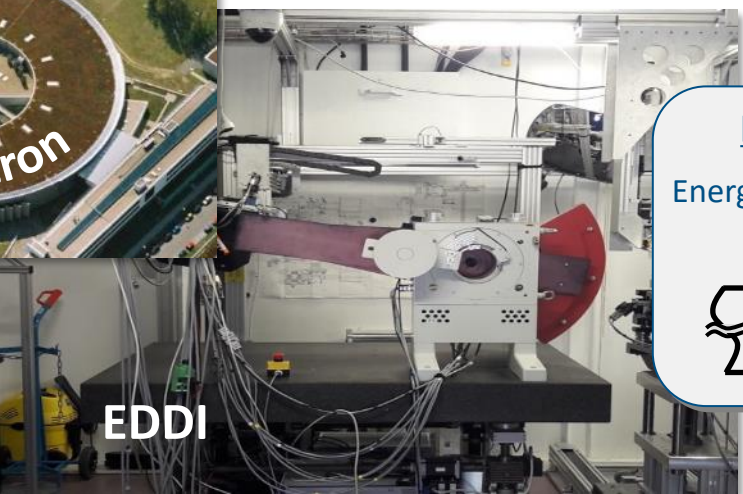
Student internships  
Promoting young scientist  
**In-house research**  
Method development  
Academic qualification



**Materials research utilizing diffraction & imaging methods**

Academic training  
**User service**  
External academic researchers

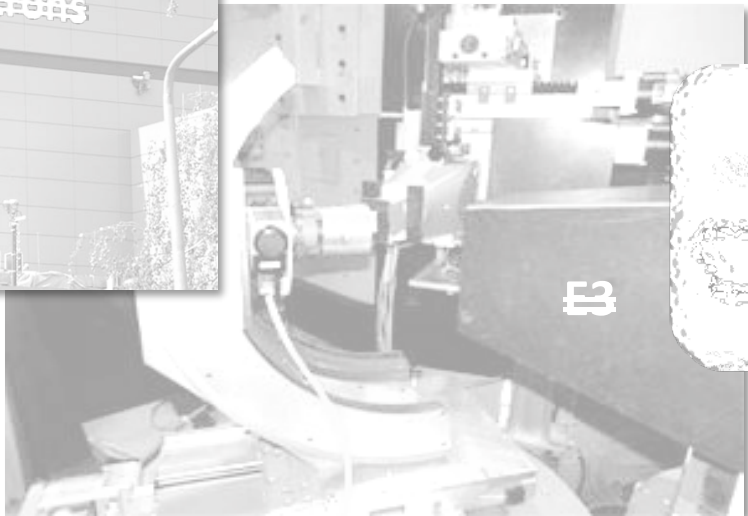
Long-term cooperations  
Technology transfer  
**Industrial research**  
Contract measurements



Lab



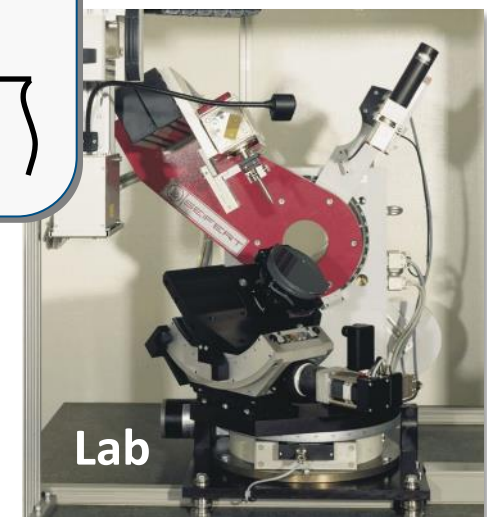
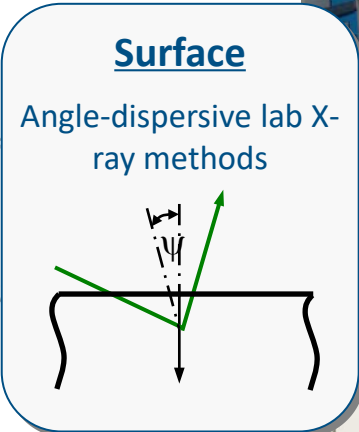
# Microstructure & Residual Stress Analysis @ HZB



Student internships  
 Promoting young scientist  
**In-house research**  
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 Academic qualification

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 External academic researchers

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 Technology transfer  
**Industrial research**  
 Contract measurements



# Prof. Hans M. Hertz & Excillum

- Applied Physics at **KTH Royal Institute of Technology, Stockholm**
- Proof of principle** with liquid SnPb alloy as anode

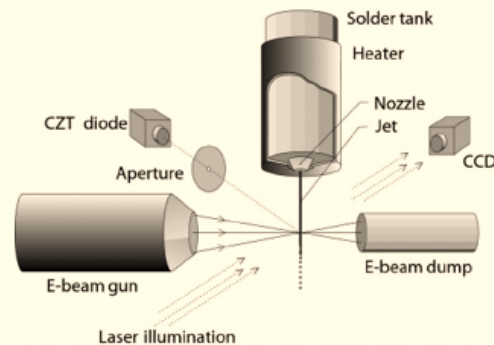
## Liquid-metal-jet anode electron-impact X-ray source

(2003)

O. [Hemberg](#); M. [Otendal](#); H. M. Hertz

*Appl. Phys. Lett.* 83(7), 1483-1485

doi: [10.1063/1.1602157](https://doi.org/10.1063/1.1602157)



great-great-nephew  
of Heinrich R. Hertz



grand-son of  
Gustav L. Hertz



# excillum

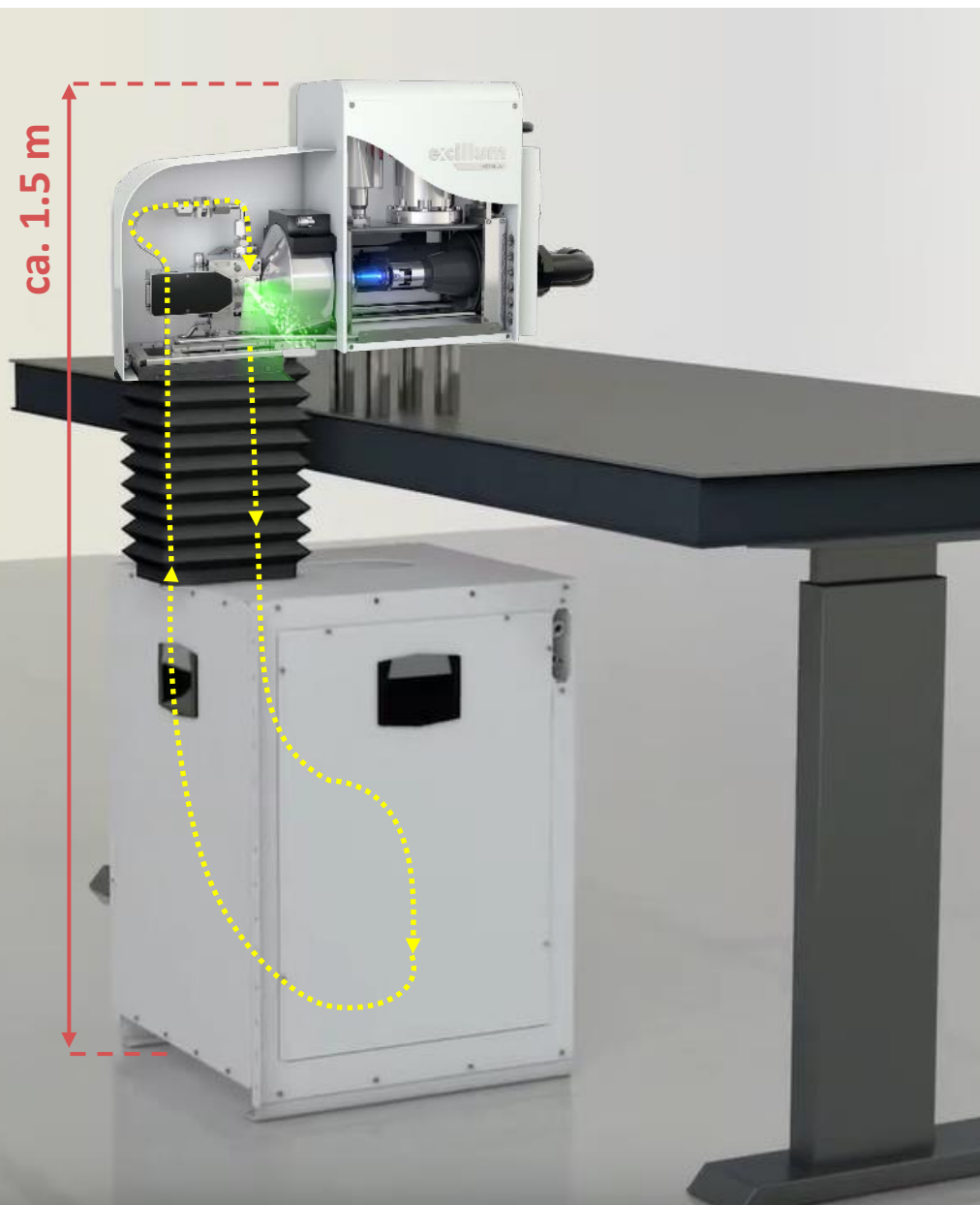
- Co-founder of **Excillum** (2007)

Anode alloys: Ga [47-95 wt-%], In [5-37 wt-%], Sn [rest]

MetalJet

# Installation, Operation & Maintenance

# Installation



## X-ray head

cathode chamber & jet chamber  
under high vacuum ( $\sim 10^{-7}$  mbar)



## 19" rack components:

- System controller
- HV controller + generator
- Water chiller

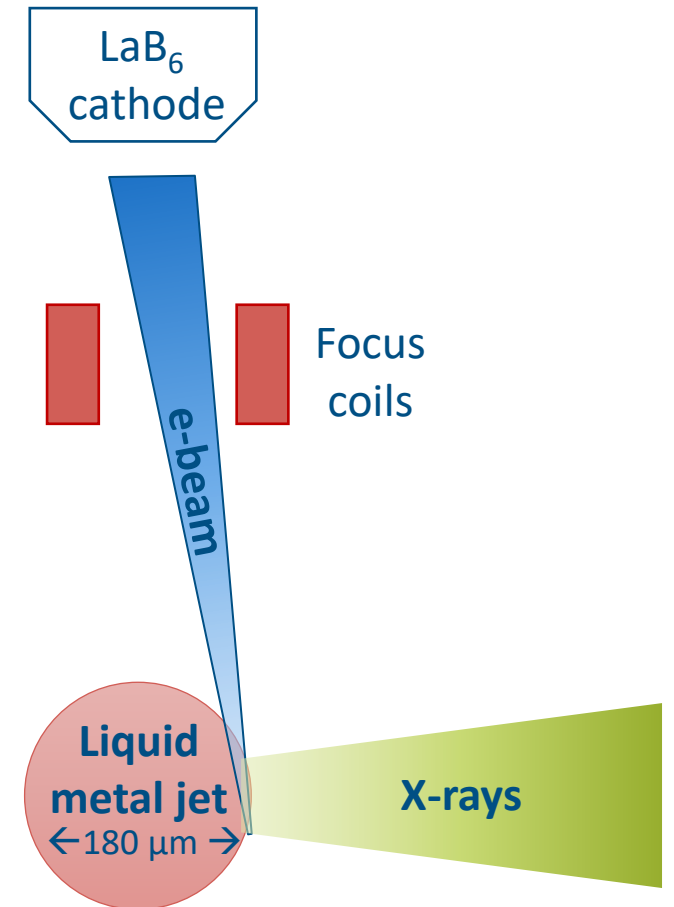
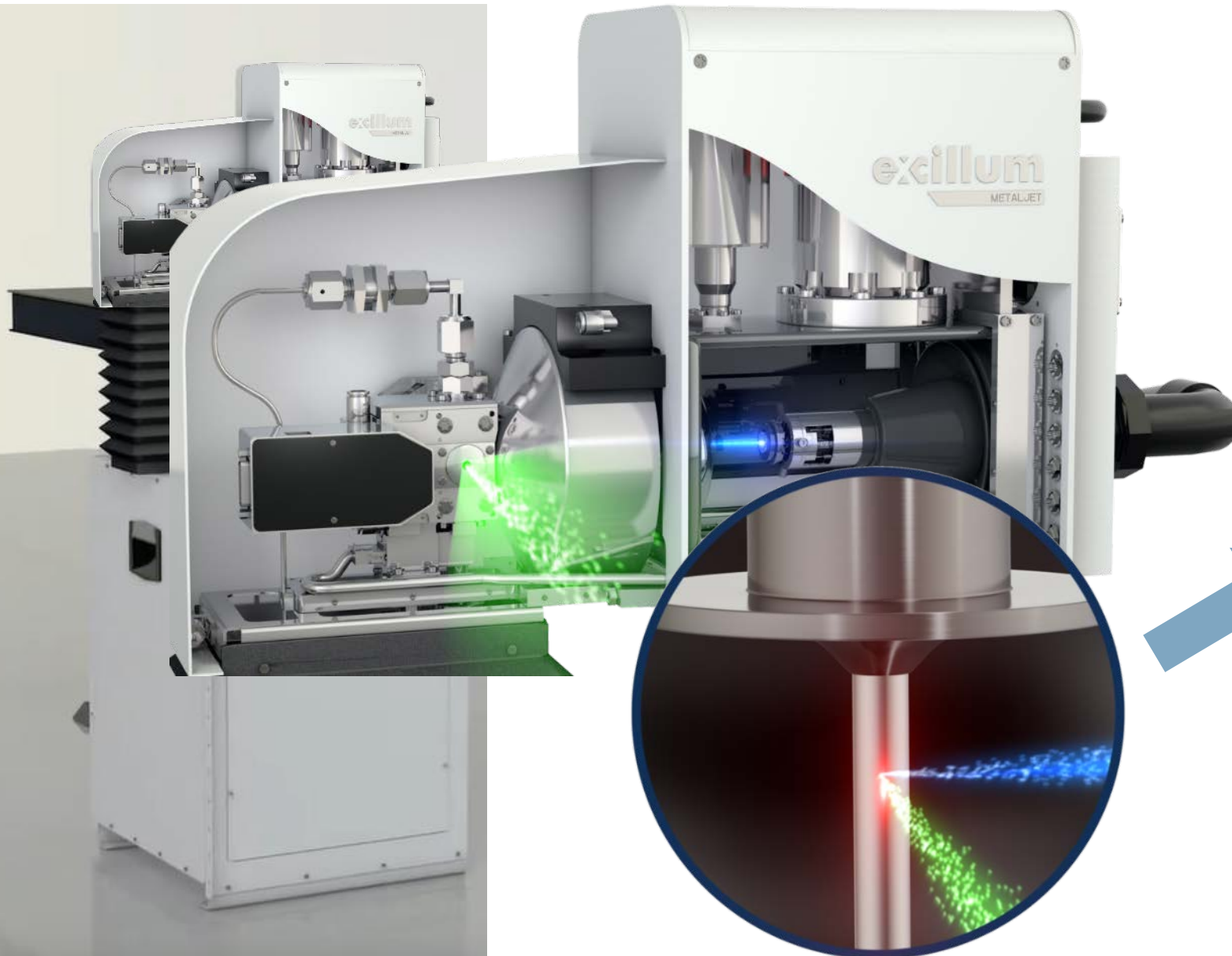
## Pump box

jet pump (190 bar) + vacuum rough pump

**all components: single phase 230 V  $\sim$  16 A  
no need for power current or central water supply**



# Operation



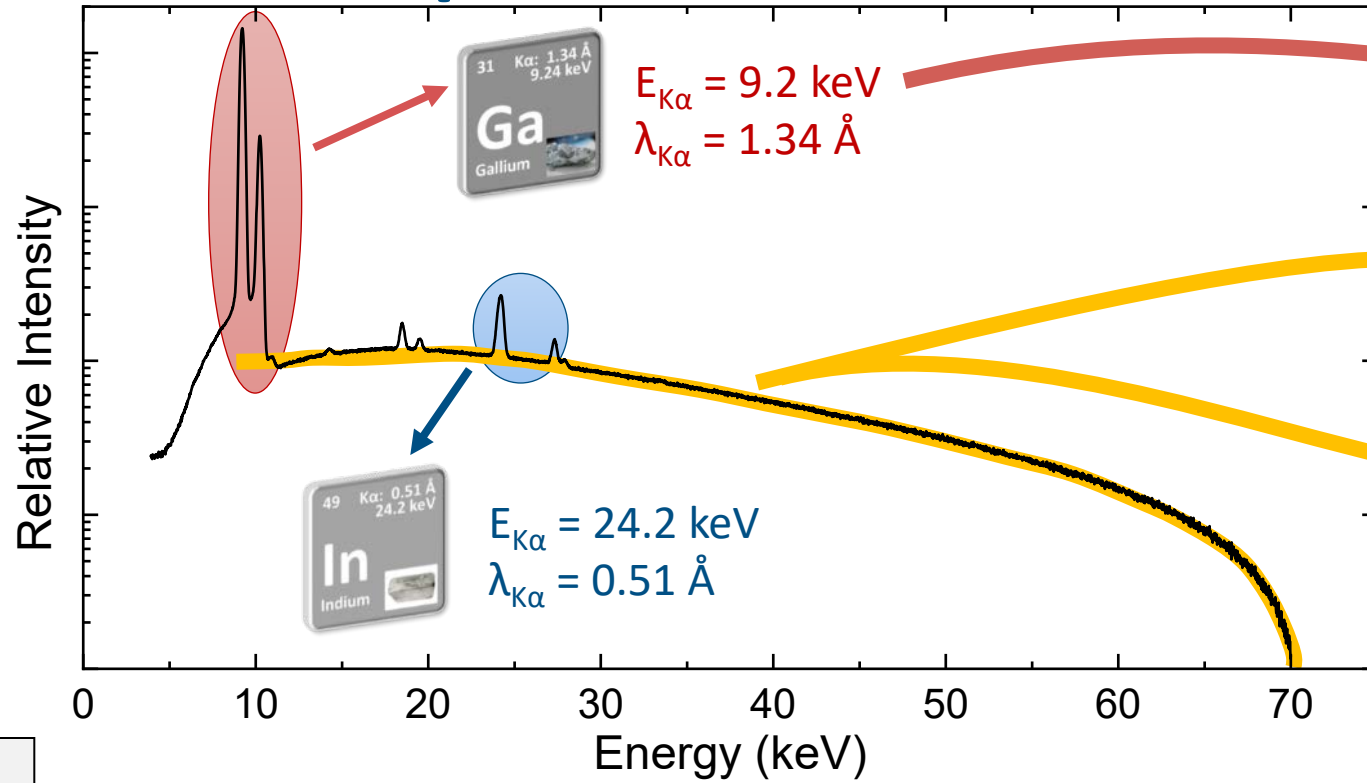
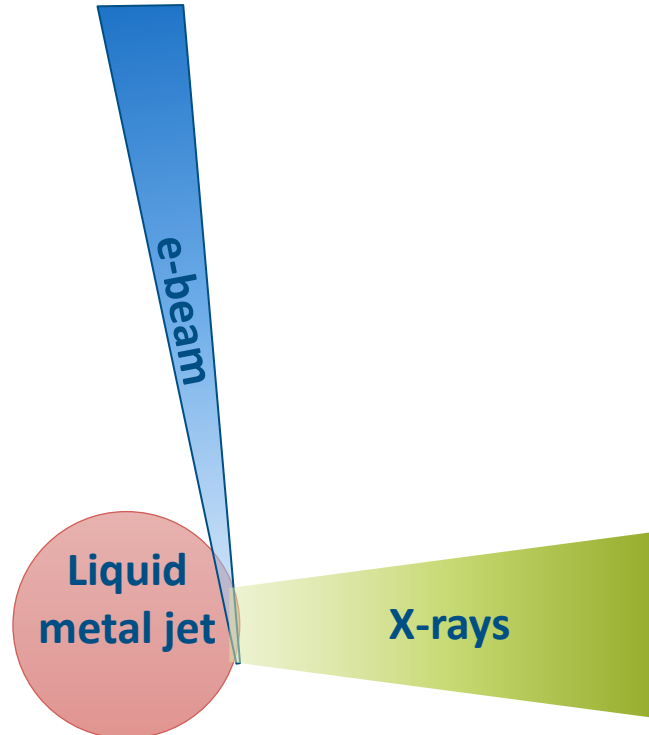
**Min. projected spot size:**

5 μm × 5 μm

**Line focus @ 250 W emission:**

20 μm × 80 μm

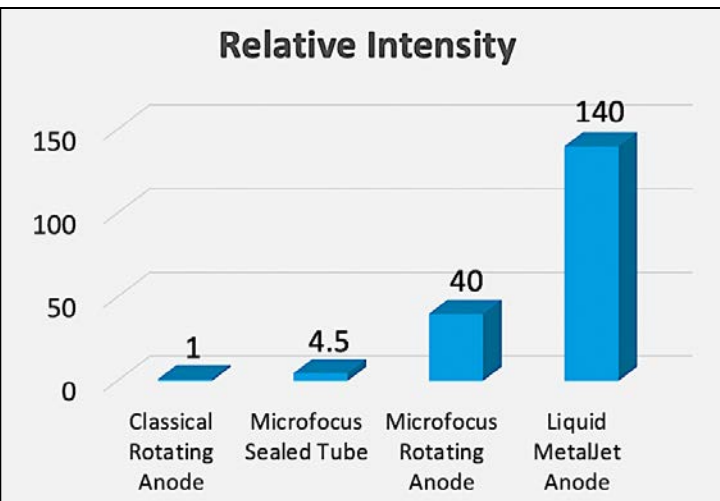
# Operation



Angle-dispersive diffraction

White-beam radiography/tomography

Energy-dispersive diffraction

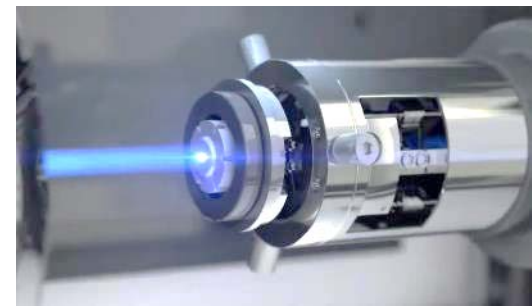


Photon flux [/s/sr/line] at 70 kV/200 W		
Line	Excillum	measured by PTB & HZB
GaKa 9.2 keV	$\sim 5 \cdot 10^{12}$	$6 \cdot 10^{12}$
InKa 24.2 keV	$\sim 4 \cdot 10^{11}$	$4 \cdot 10^{11}$

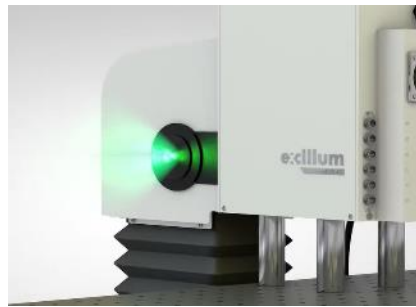
M. Wansleben et al. (2019)  
*J. Analytical Atomic Spectrometry* 34(7) 1497-1502  
 doi: 10.1039/C9JA00127A



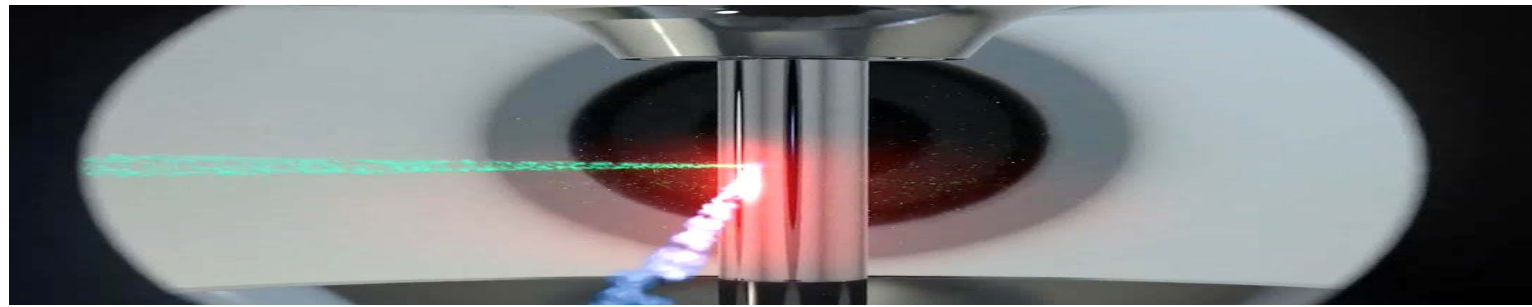
# Maintenance



LaB<sub>6</sub> cathode



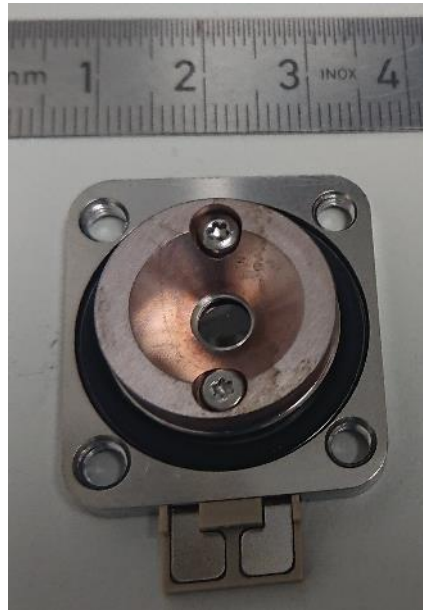
Exit window



Particle filter

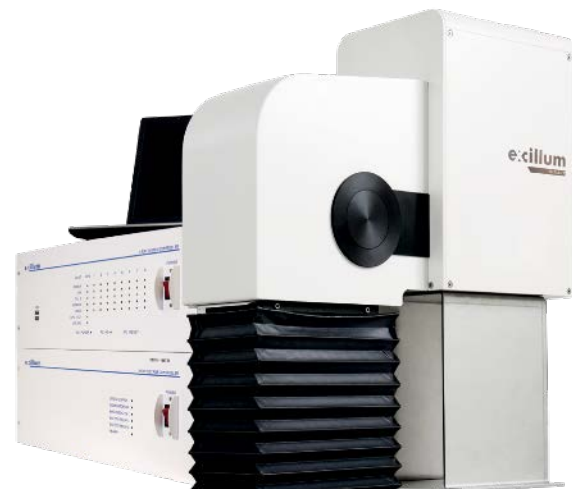
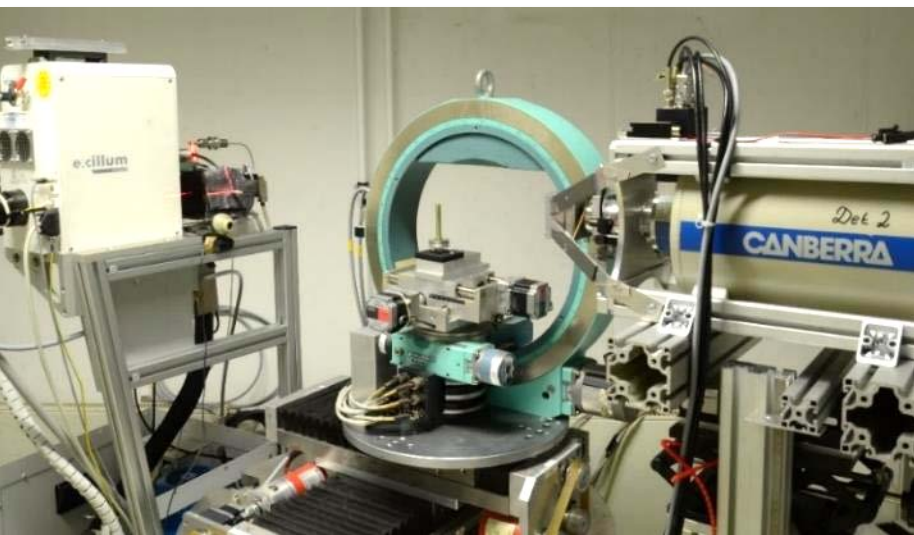
Nozzle

Target alloy

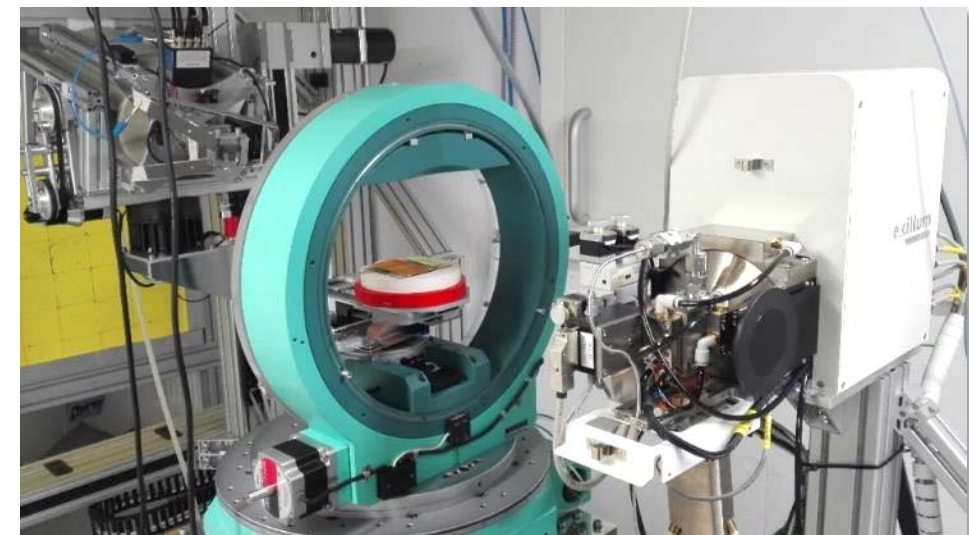


+ jet pump & vacuum pump parts (gaskets, oil, valves), water cooling...

# MetalJet D2 @ HZB



70/160 kV max. 250 W

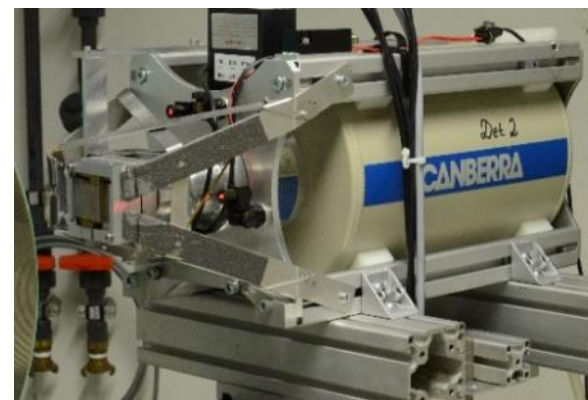
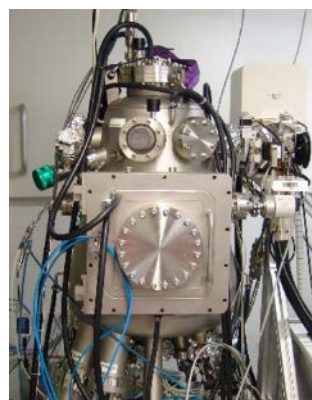
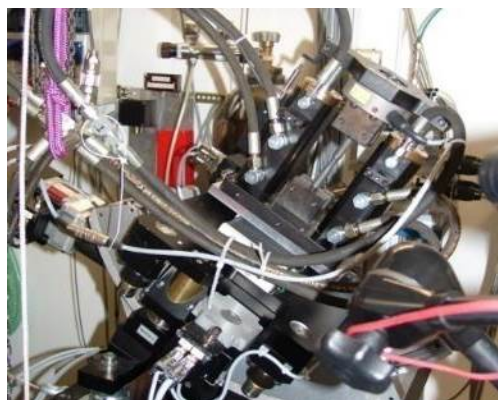
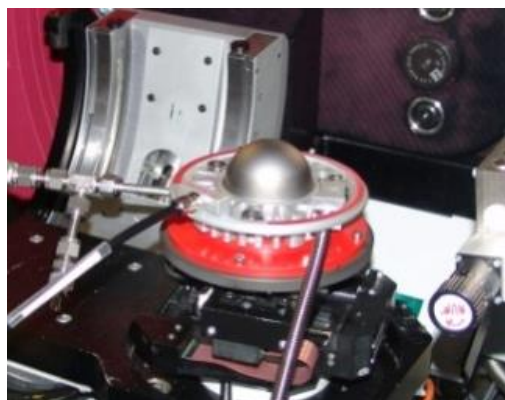


furnace

load frame

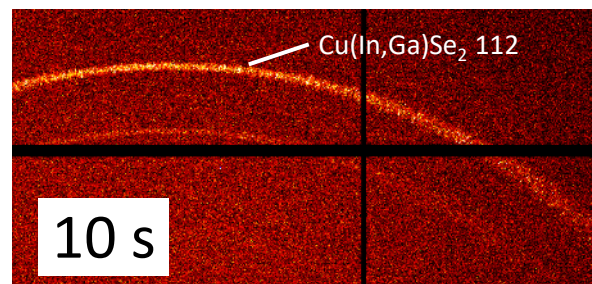
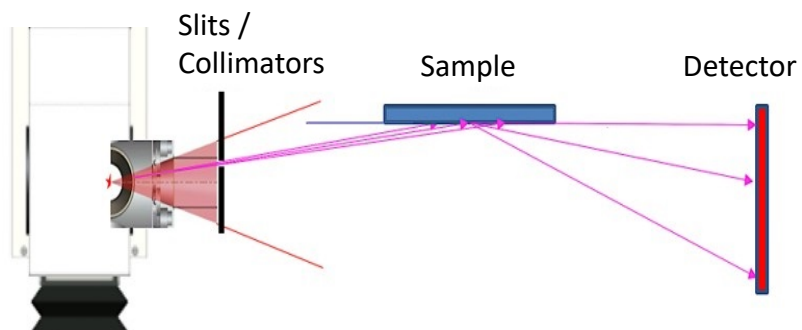
PVD

detectors...

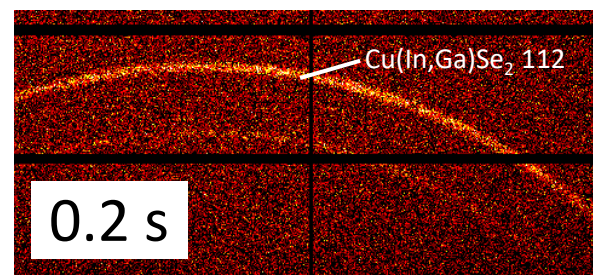
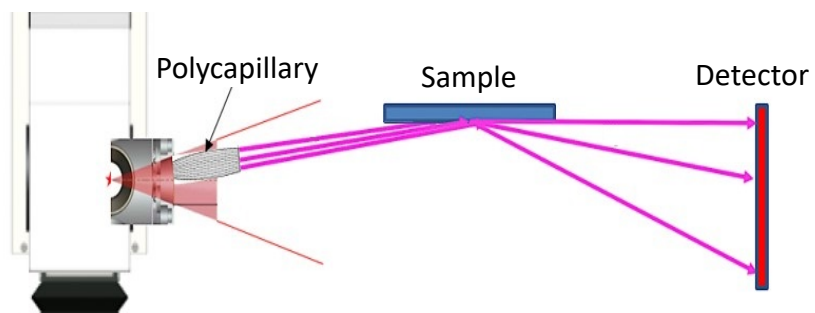
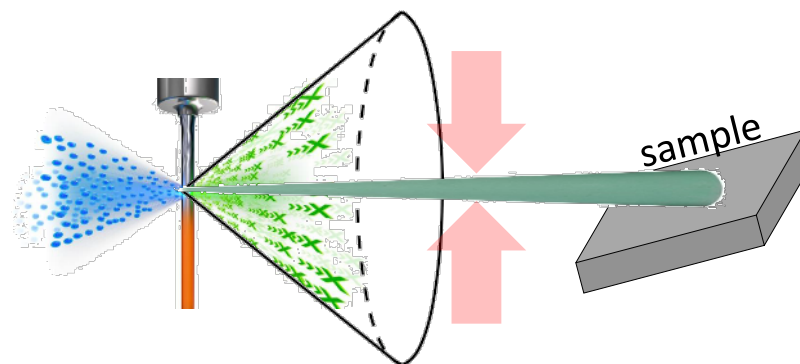




# Primary optics

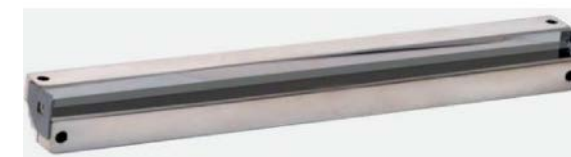
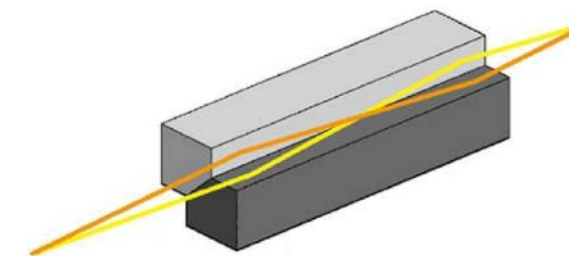


## Polycapillary optics



## Montel optics

- e.g. monochromatic Ga  $K\alpha$  radiation
- available at HZB since 2021

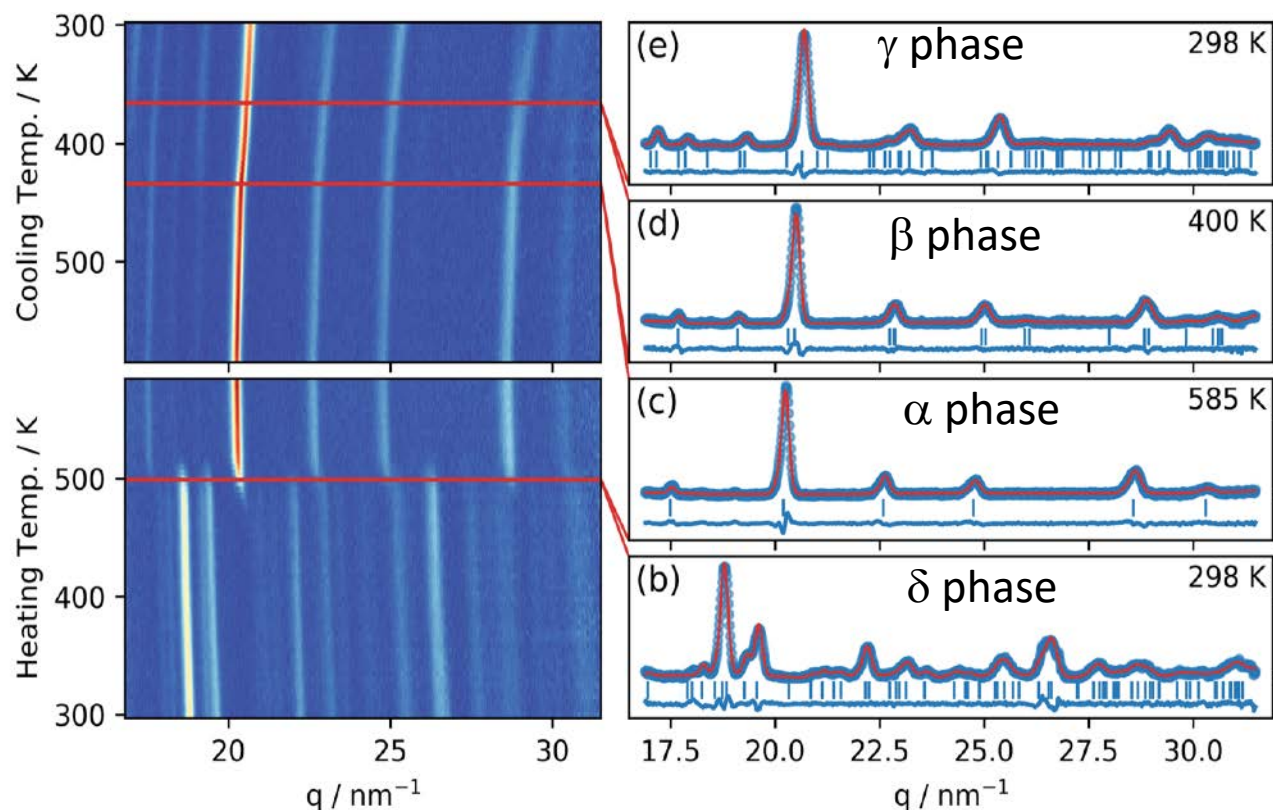


**MetalJet**

# **Application examples**

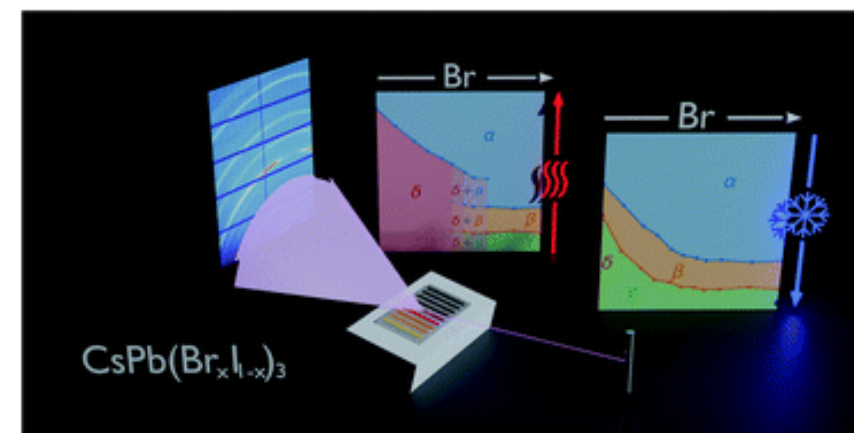
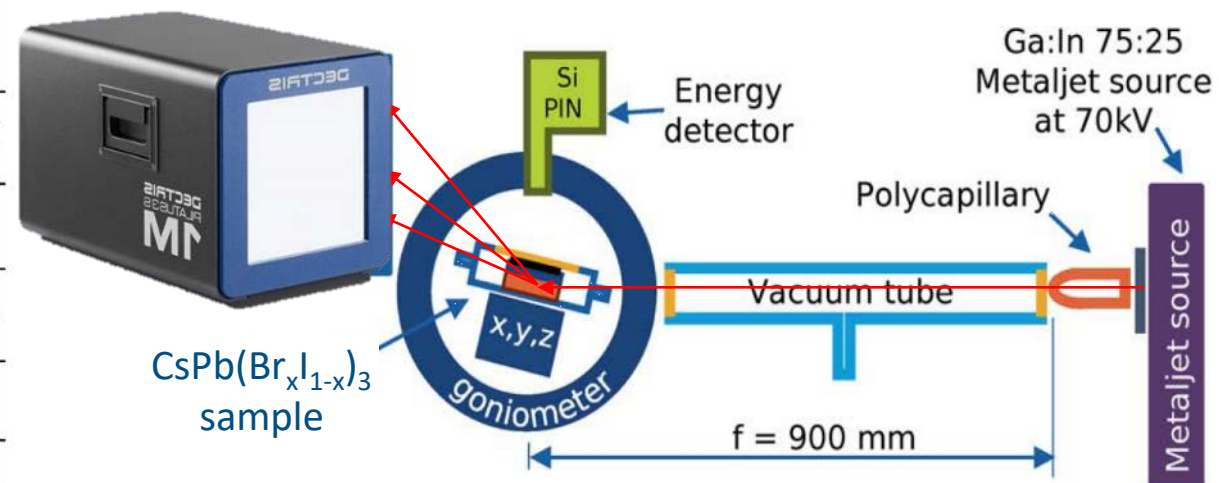


# Phase-transitions in PV materials



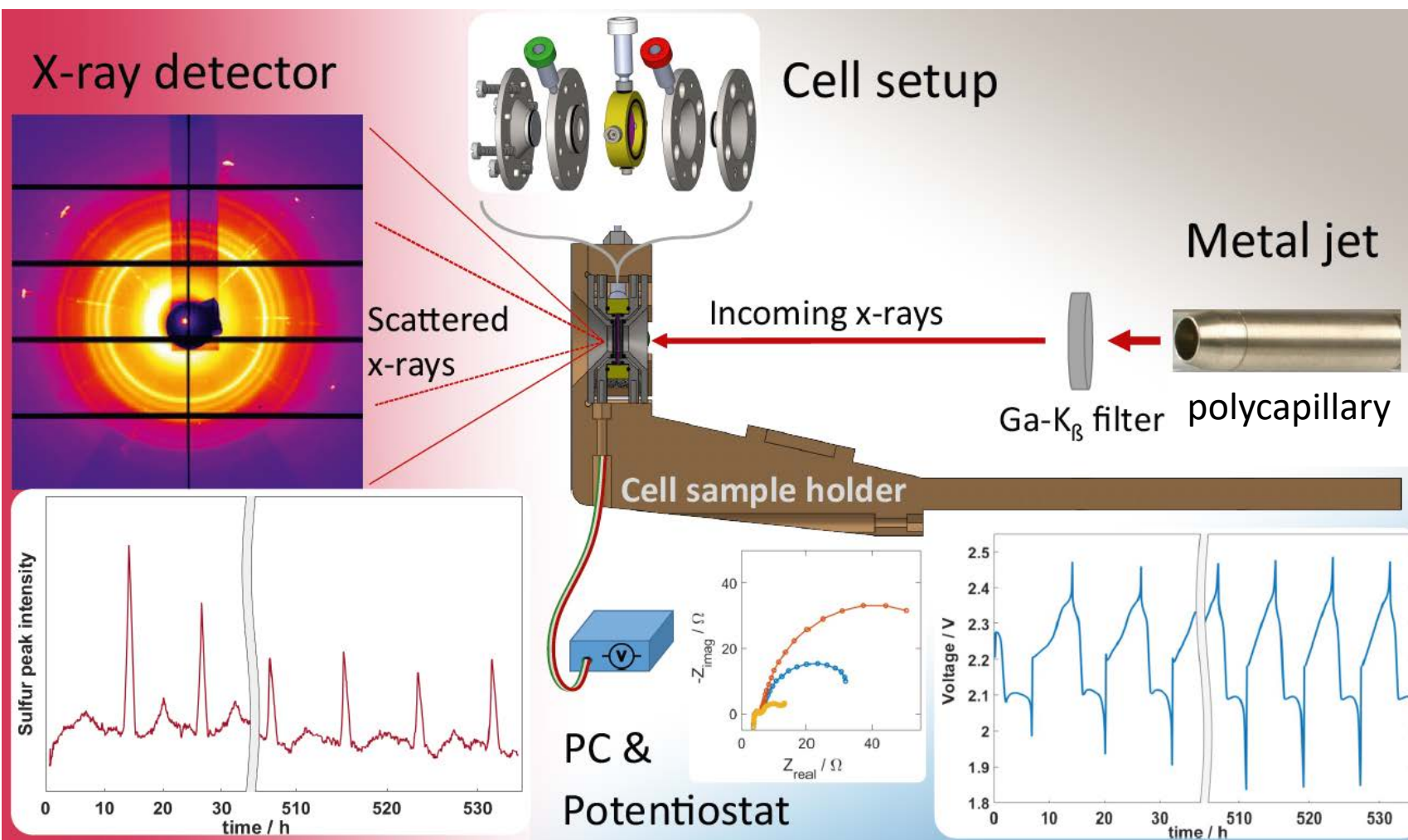
Integrated diffraction pattern as a function of temperature for  $\text{CsPb}(\text{Br}_x\text{I}_{1-x})_3$  with  $x = 0.23$

**Time resolution down to 0.2 s possible!**



H. Näsström, P. Becker, J. Márquez, O. Shargaieva, R. Mainz, E. Unger and T. Unold. *J. Mater. Chem. A* (2020), doi: 10.1039/D0TA08067E

# Long-term in-operando studies of LiS batteries



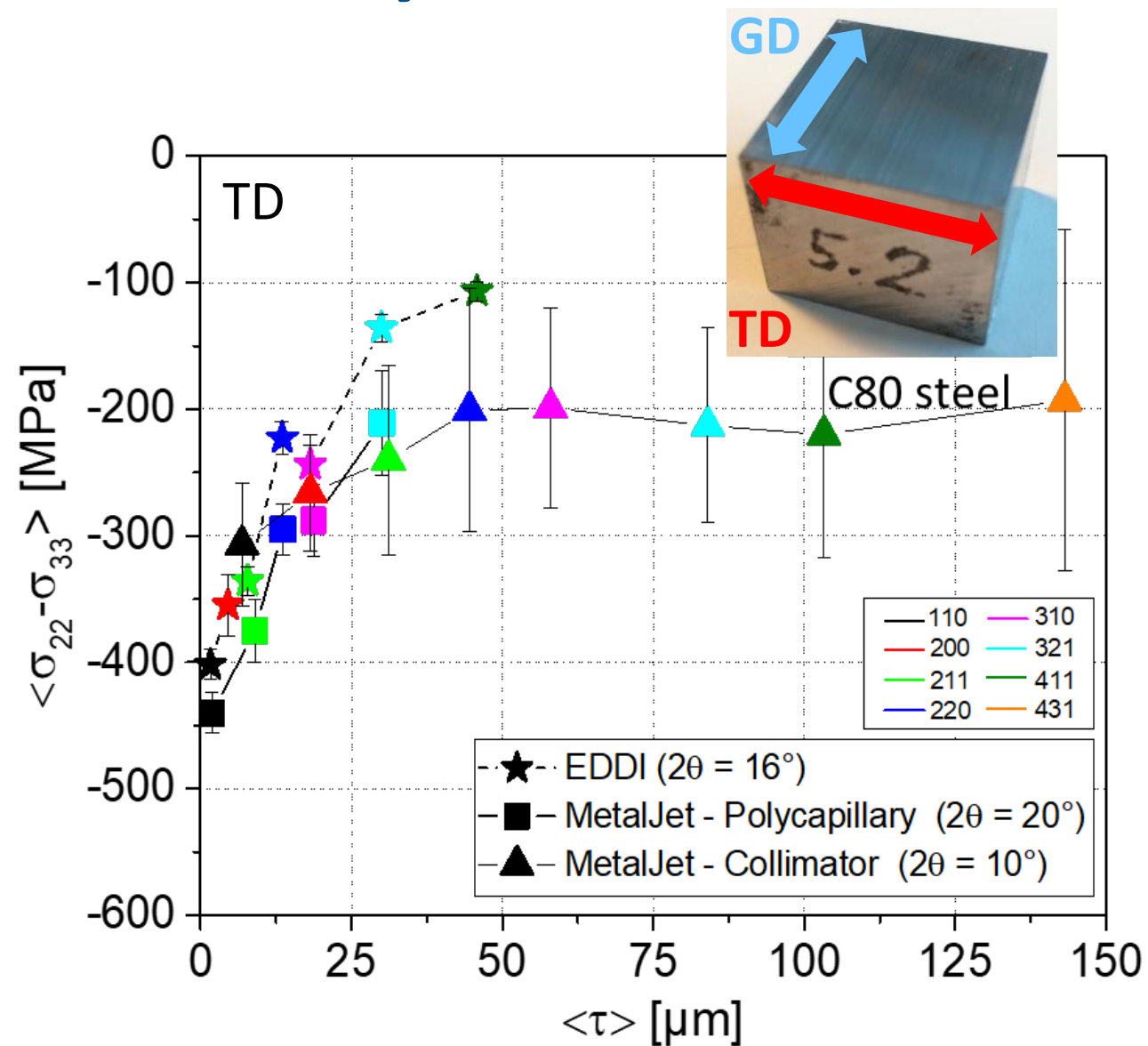
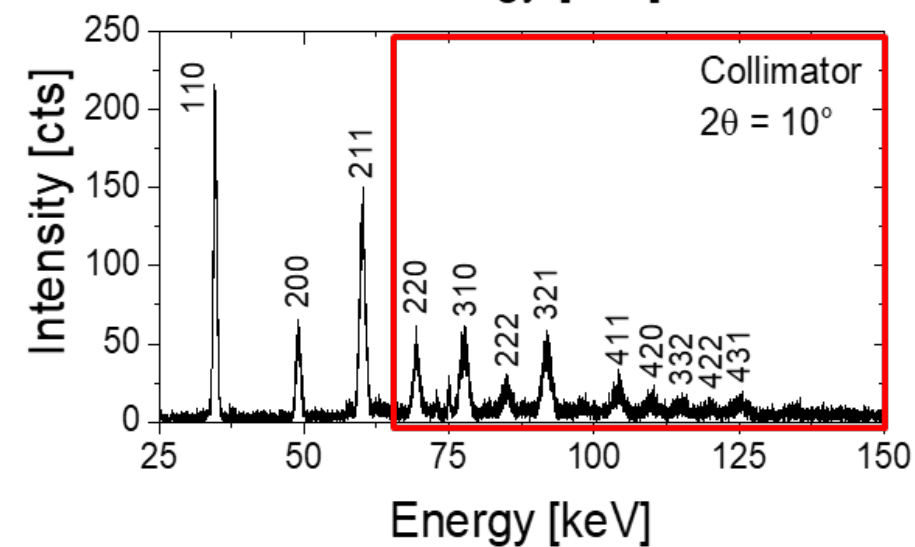
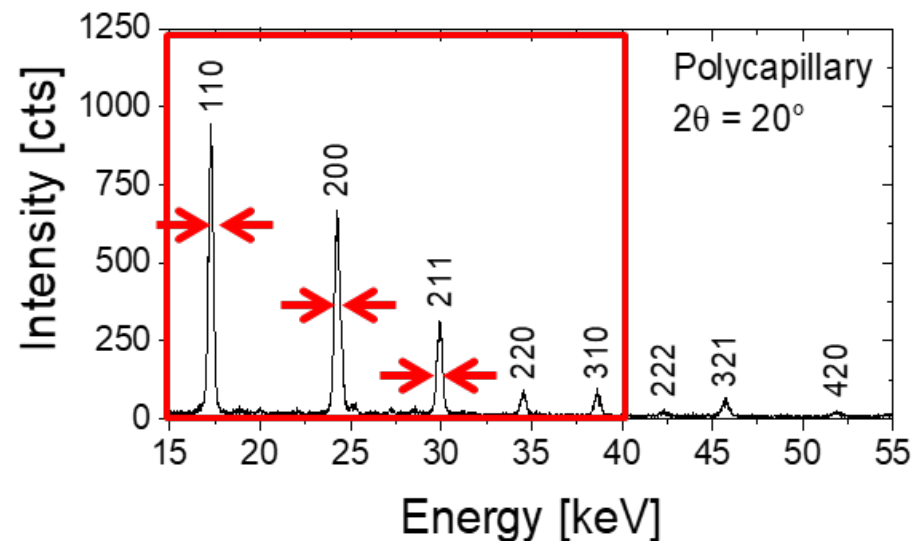
- cathode: sulfur loaded CNTs
- anode: lithium
- discharge: forming of  $\text{Li}_2\text{S}_2$
- charging: decomposing to  $\text{Li}_2\text{S} + \text{S}$

**more than 3 weeks  
non-stop  
measurement**

- sulfur diffraction line intensities
- domain/crystallite size

# Residual stress analysis

MetalJet D2 @ 160 kV + Ge Detector





# White-beam Imaging

- MetalJet D2 (70/160 kV)
- Full cone beam & full energy spectrum
- Detector choice depends on sample attenuation

**AM Structure**

**Tomography**

**Metaljet + CMOS**

**W-Microfocus + Flatpanel**

**Polymer foams**  
C. Jiménez et al., (2015)

**Dectris Pilatus**  
Si photon-counting

**Advacam Medipix**  
CdTe photon-counting

**Hamamatsu flat panel**  
CsI scintillator plate

$E \leq 30 \text{ keV}$

$E \approx 30 \text{ keV}$

$E > 30 \text{ keV}$

Sample

Sample

Sample

10 mm

5 s

0.5 s

5 s

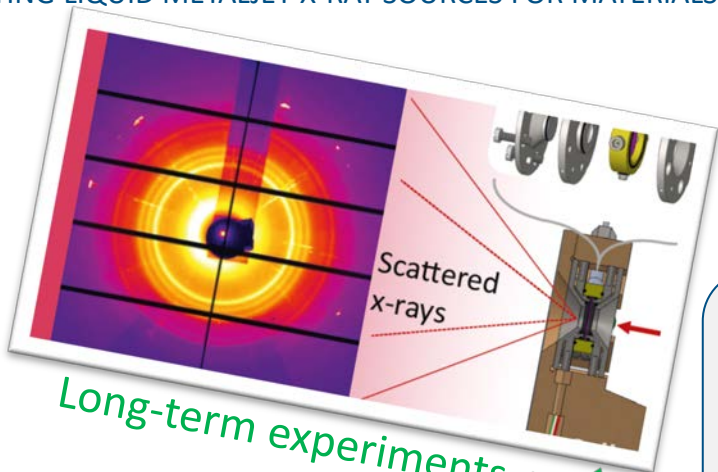
0.5 s



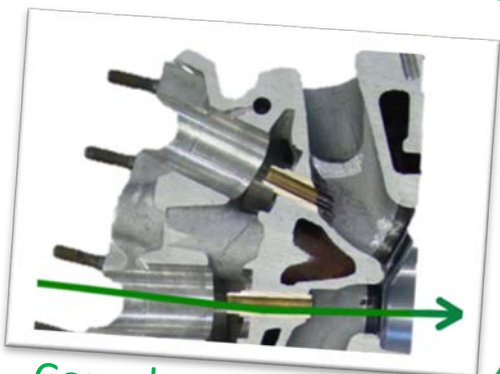
# Conclusion, Review & Outlook

# Conclusion

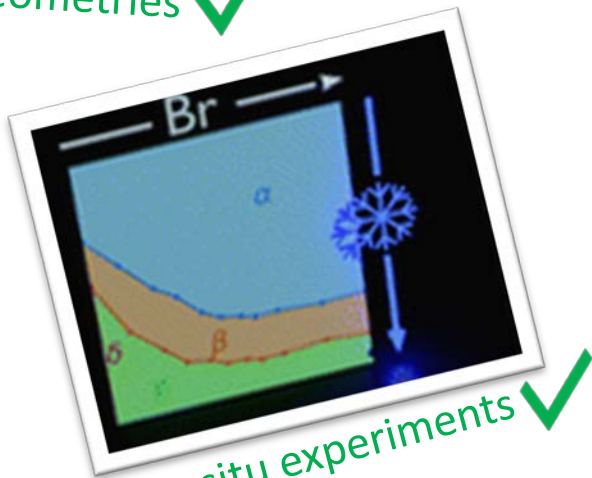
**Materials research utilizing diffraction & imaging methods**



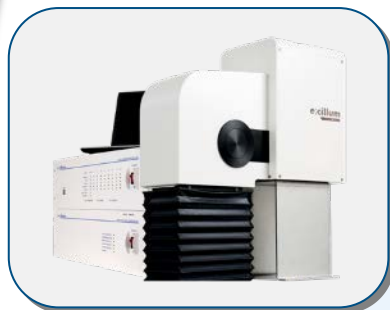
Long-term experiments ✓



Complex geometries ✓

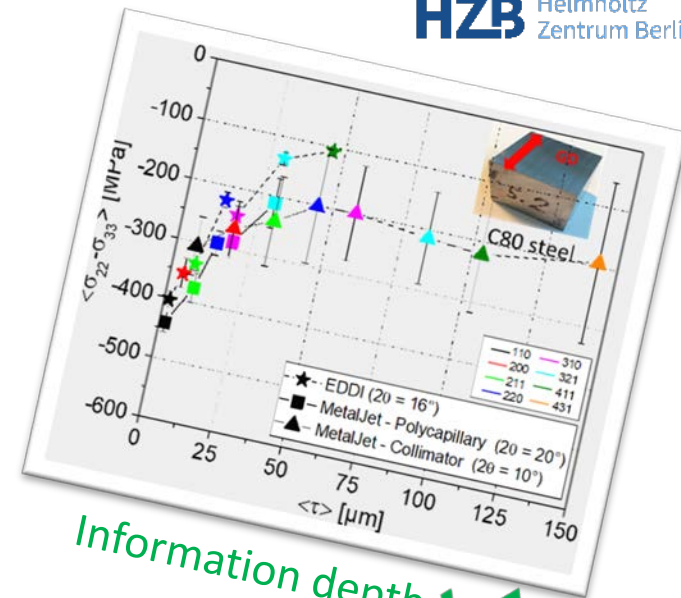


In-situ experiments ✓

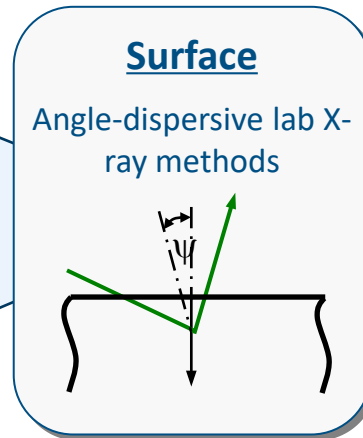


Student internships  
Promoting young scientist  
**In-house research**  
Method development  
Academic qualification

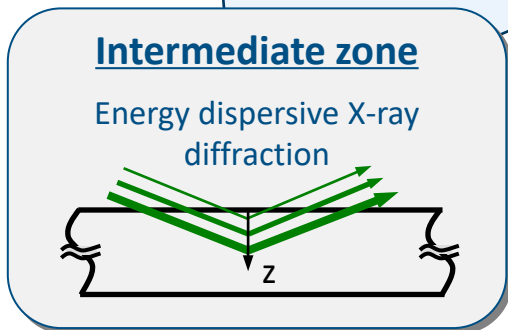
Academic training  
**User service**  
External academic researchers



Information depth ✓



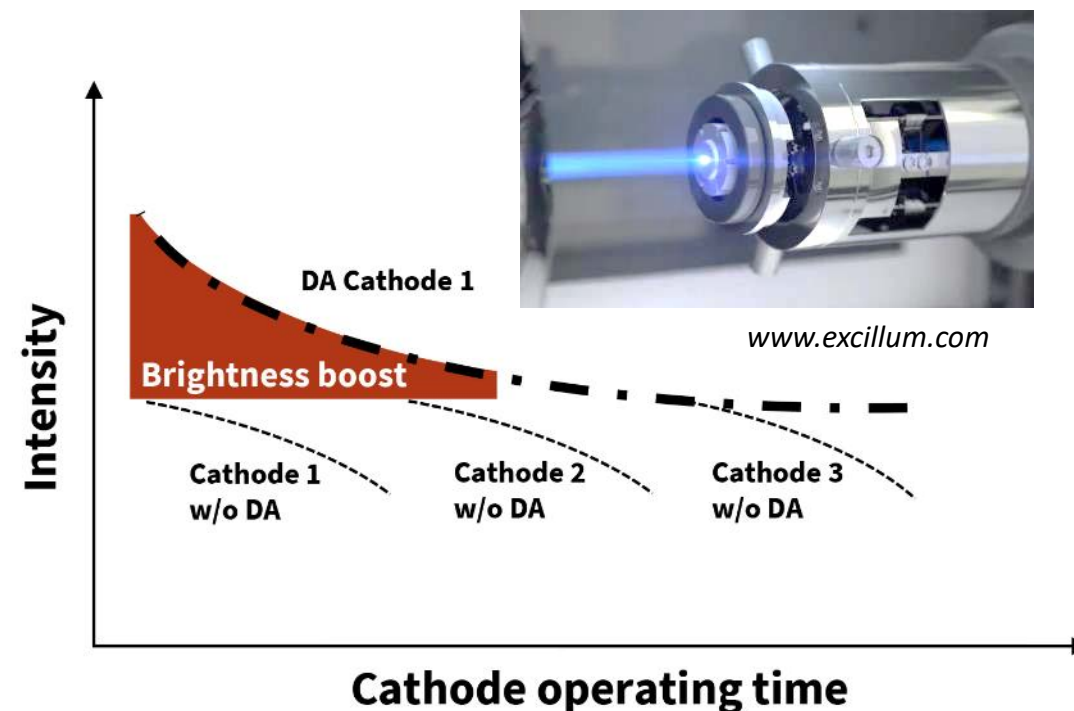
Fast imaging ✓



Long-term cooperations  
Technology transfer  
**Industrial research**  
Contract measurements

# Review On Performance & Limitations

- 2003** SnPb alloy, 100 W, 50 kV, 150  $\mu\text{m}$  focus
- 2007** Ga-based alloys (liquid at room temperature)  
MetalJet D1, 70kV, 100 W / 200 W
- 2011** MetalJet D2
- 2015** 200 W  $\rightarrow$  250 W
- 2019** Dynamic Adaptation Upgrade  
Extending cathode lifetime  
24/7 operation



**Theory:** Higher jet heat-load capacity & boiling point of Ga  $>2000$   $^{\circ}\text{C}$

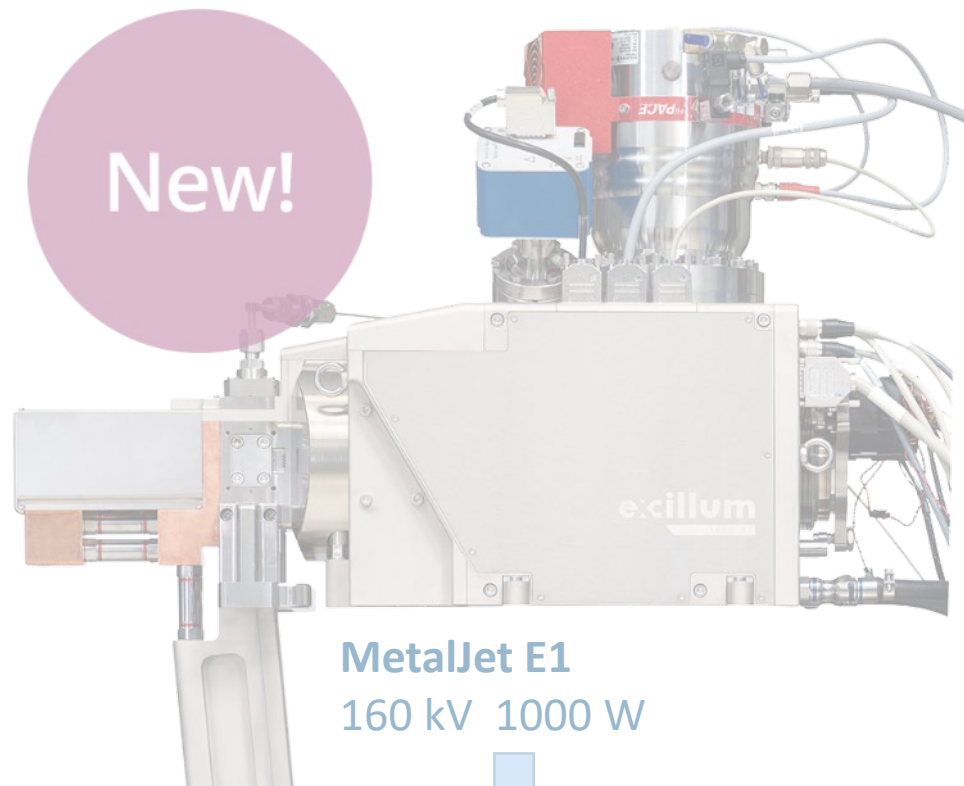
**Practice:** Evaporation effects  $\rightarrow$  possible vacuum issues

More emission power  $\leftrightarrow$  bigger focus spot (under same distance)

Higher brightness  $\rightarrow$  **Compact device**  $\leftarrow$  careful balance of vacuum/temperature/jet

# Outlook

... at HZB in 2023



**MetalJet E1**  
160 kV 1000 W



Expected intensity gain: 4x

# Thank you very much for your attention!

Do not hesitate to contact us:  
[boin@helmholtz-berlin.de](mailto:boin@helmholtz-berlin.de)