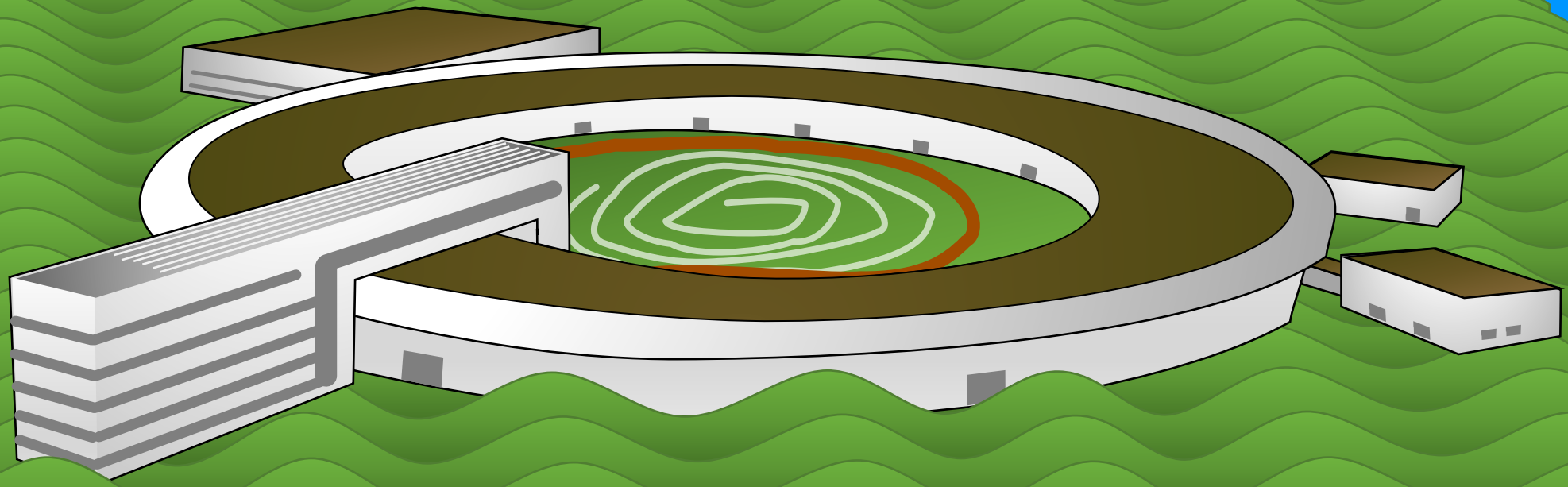
DanMAX – materials science with diffraction and imaging



Ministry of Higher Education and Science

Region Hovedstaden

midt regionmidtjylland

MAX IV

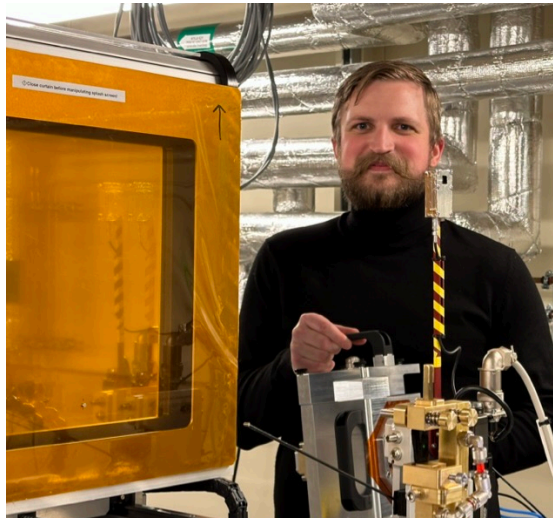
AARHUS UNIVERSITY

DTU

UNIVERSITY OF COPENHAGEN

The DanMAX team

Innokenty Kantor (Imaging)



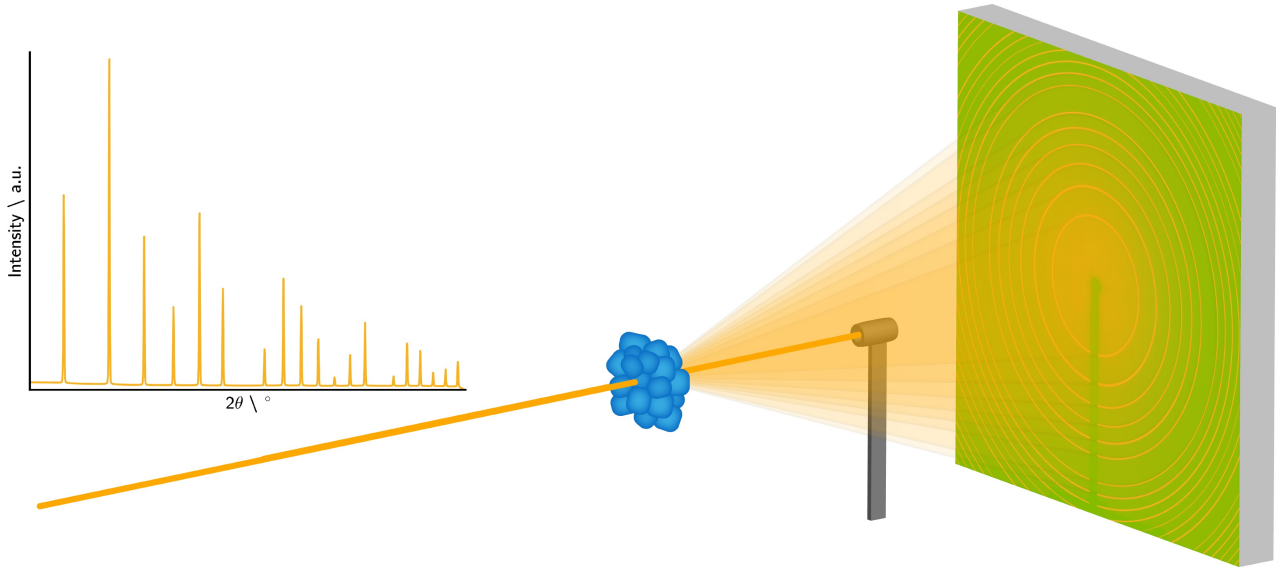
Frederik Gjørup (PD: PXRD)

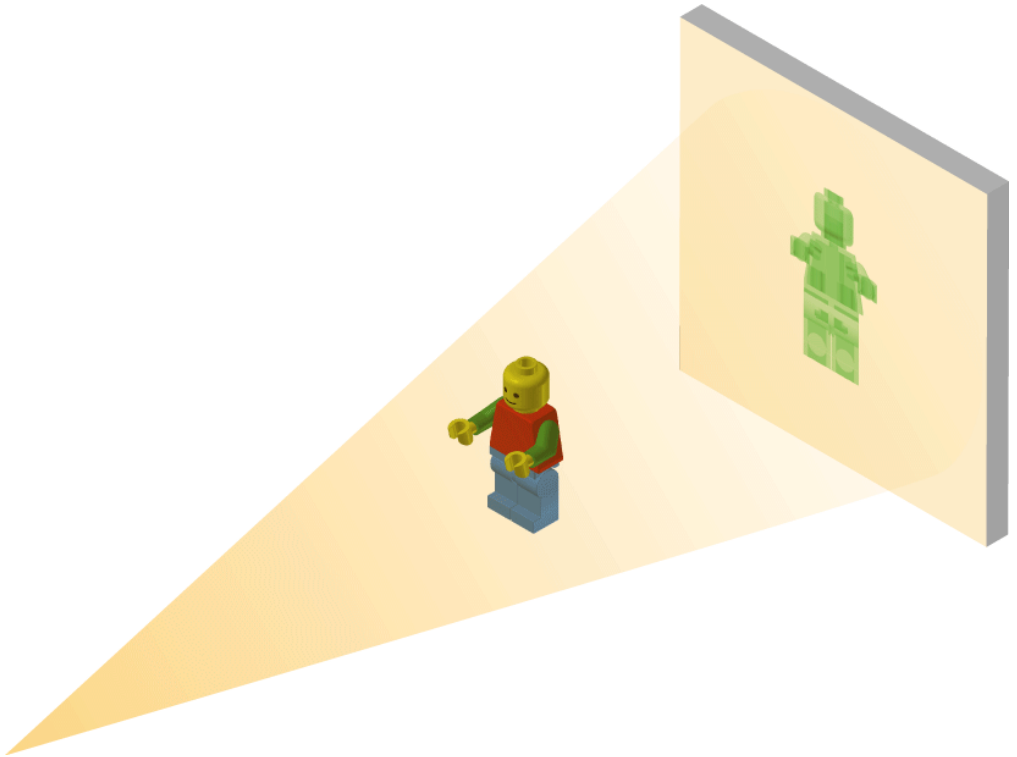
Thorbjørn Christensen
(PD: Imaging)

Lennard Krause (SINCRYS)

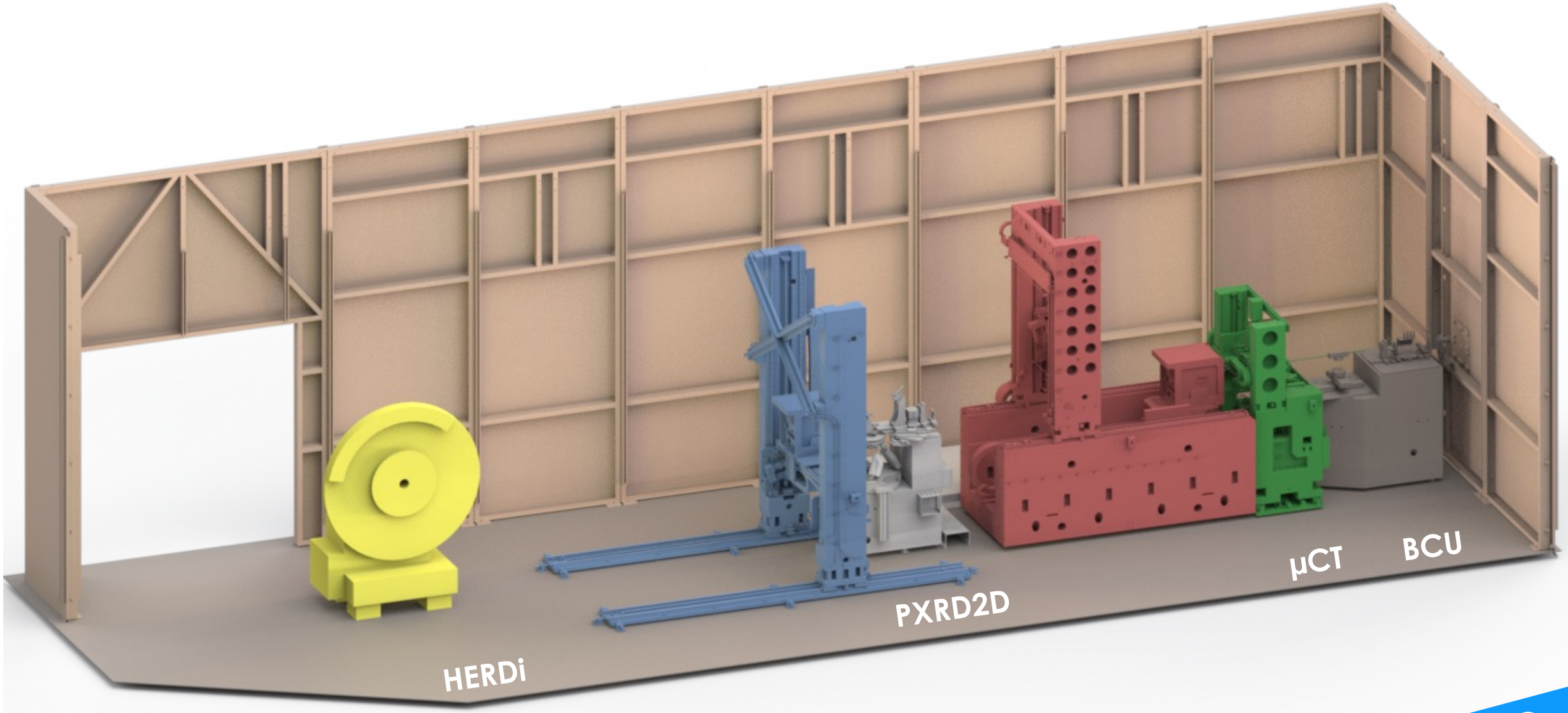
Join our user group!

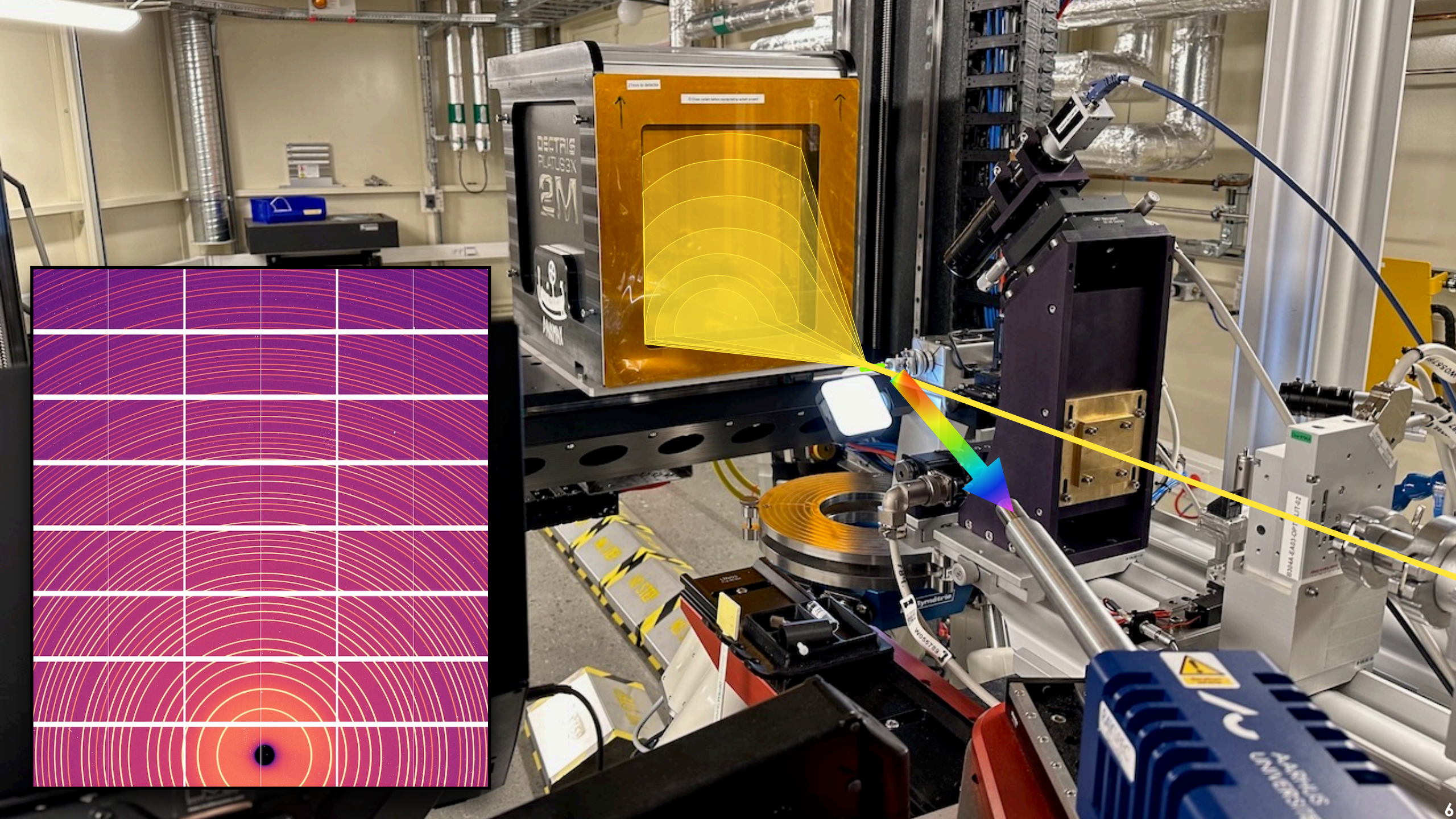
DANMAX





DanMAX: Real materials studied under realistic conditions at realistic time scales

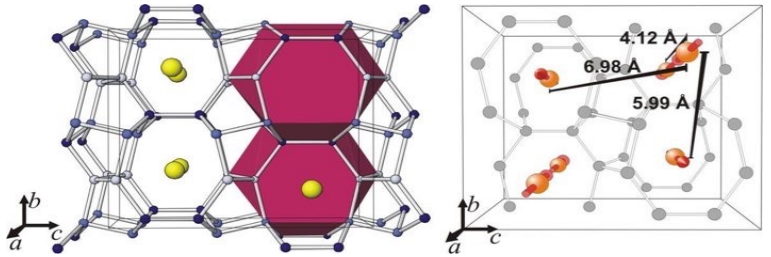

HERDi
PXRD2D
µCT
BCU

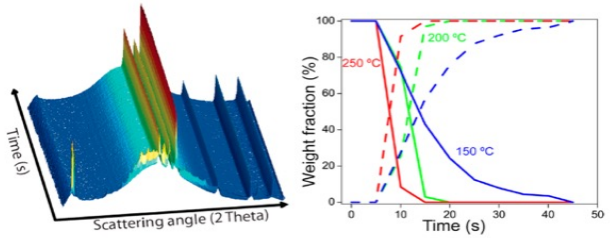

DECTRIS
PILATUS3X
2M

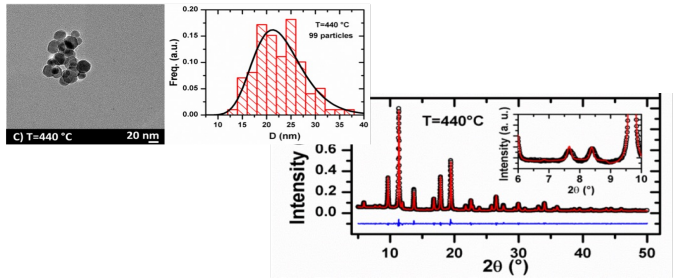
X-ray powder diffraction

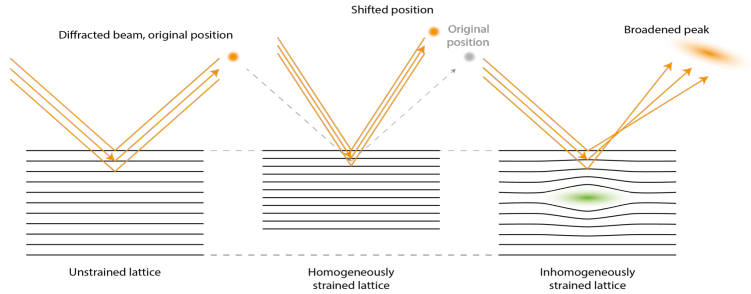
Applications of PXRD:

- Atomic coordinates
- Thermal vibration
- Phase analysis
- Reaction kinetics
- Microstructure:
  - Particle size
  - Grain orientation
  - Stress-stain analysis
- …

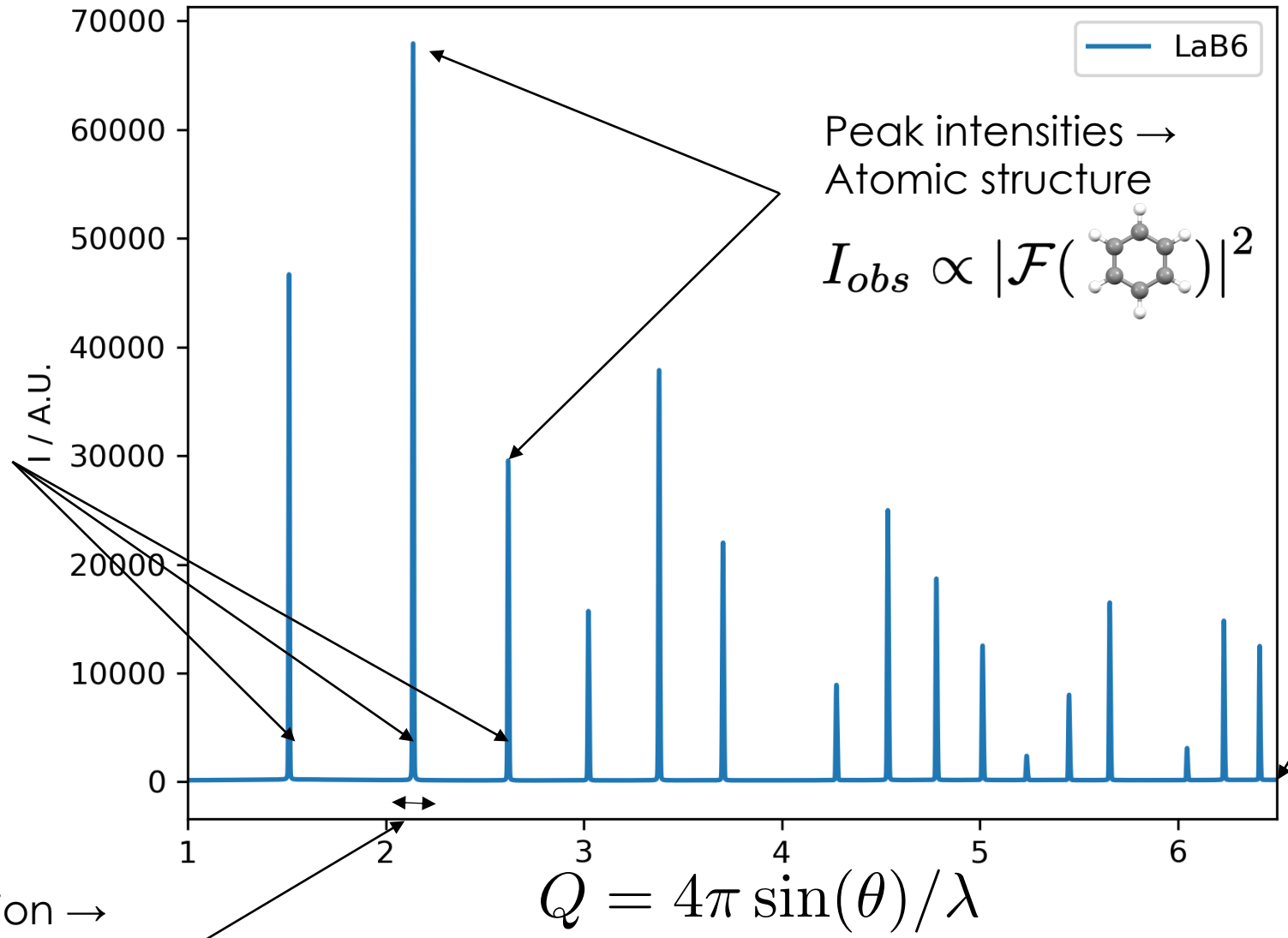








PXRD data



Peak intensities →
Atomic structure

$I_{obs} \propto |\mathcal{F}($ $)|^2$

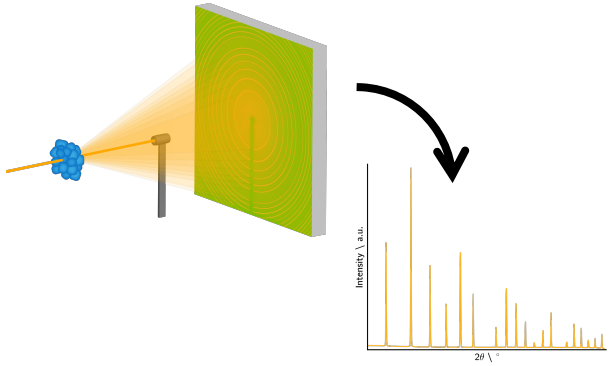
Peak position →
Sample: unit cell

$\lambda = 2d \sin(\theta)$

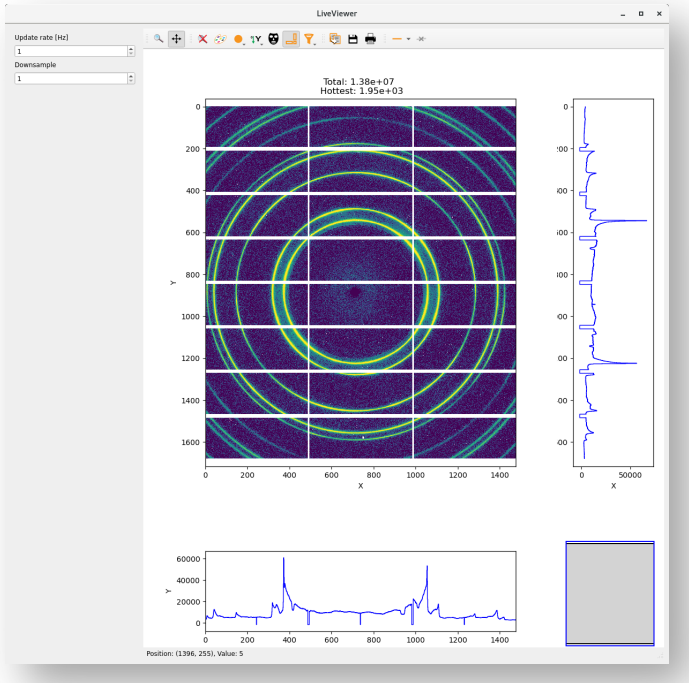
$Q_{\max}$ resolution→
Smallest features that can be resolved

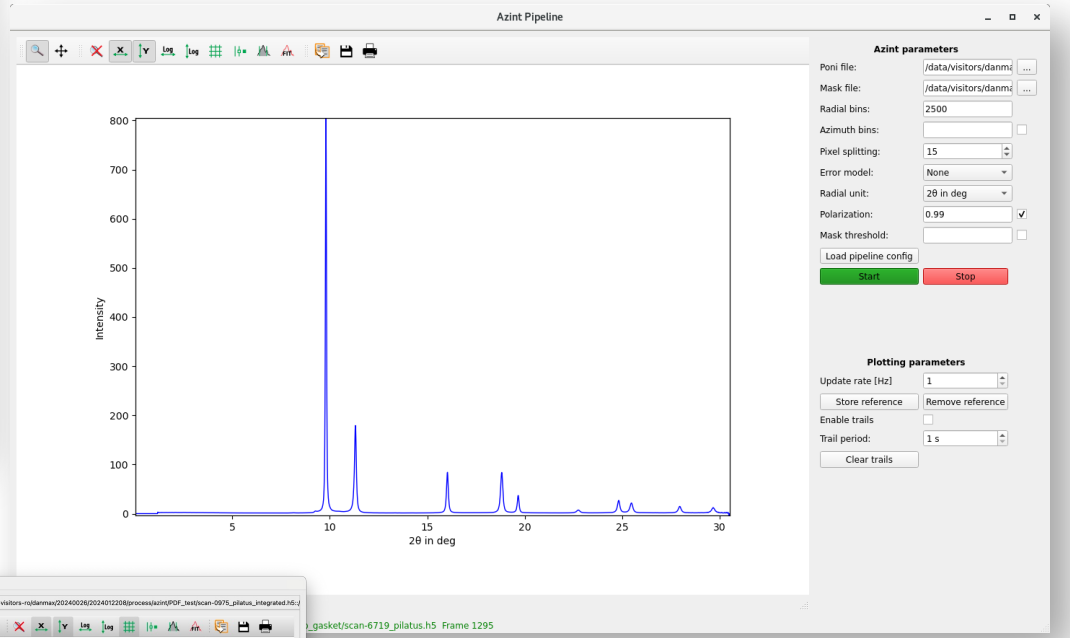
Angular resolution →
Instrument
Sample: microstrucutre

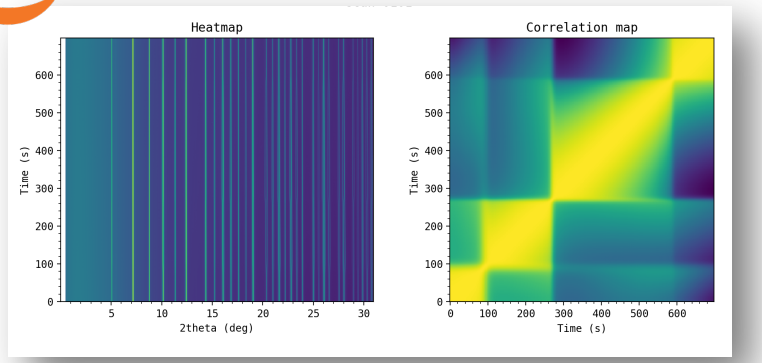
Live azimuthal integration - and quick analysis

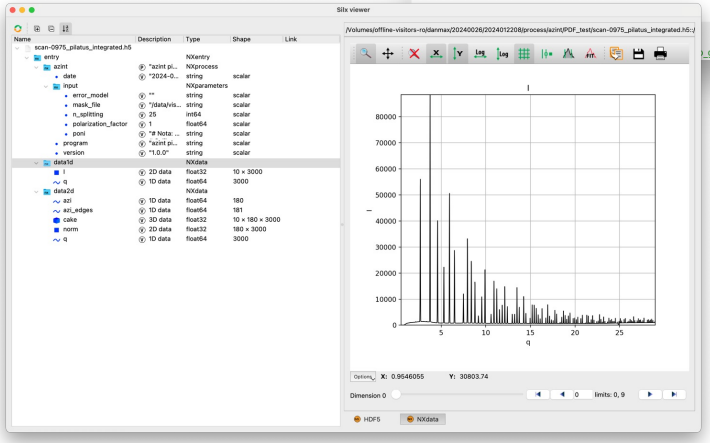


Live integration (> kHz) with live update using the MATRFAIA* algorithm





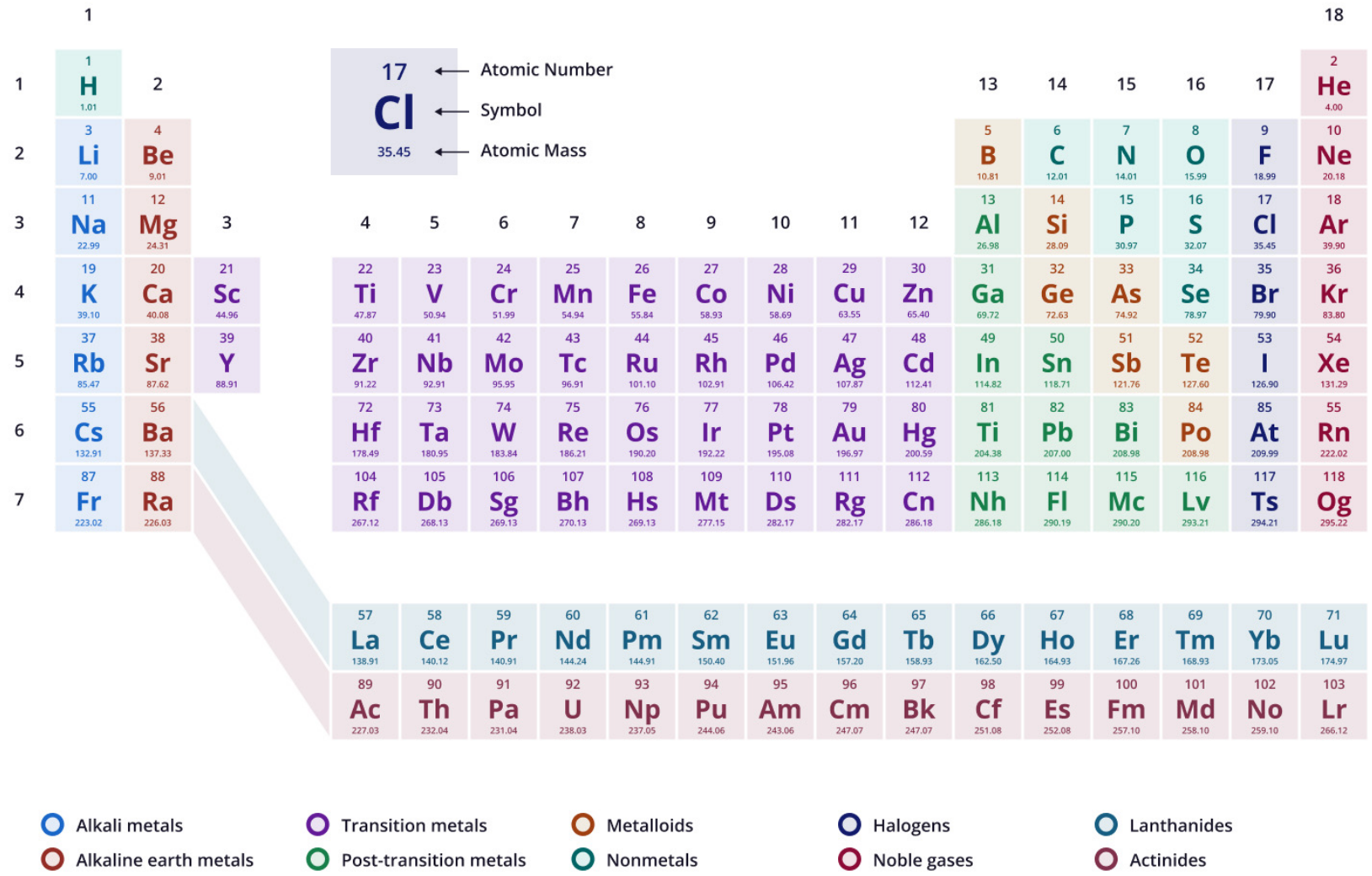




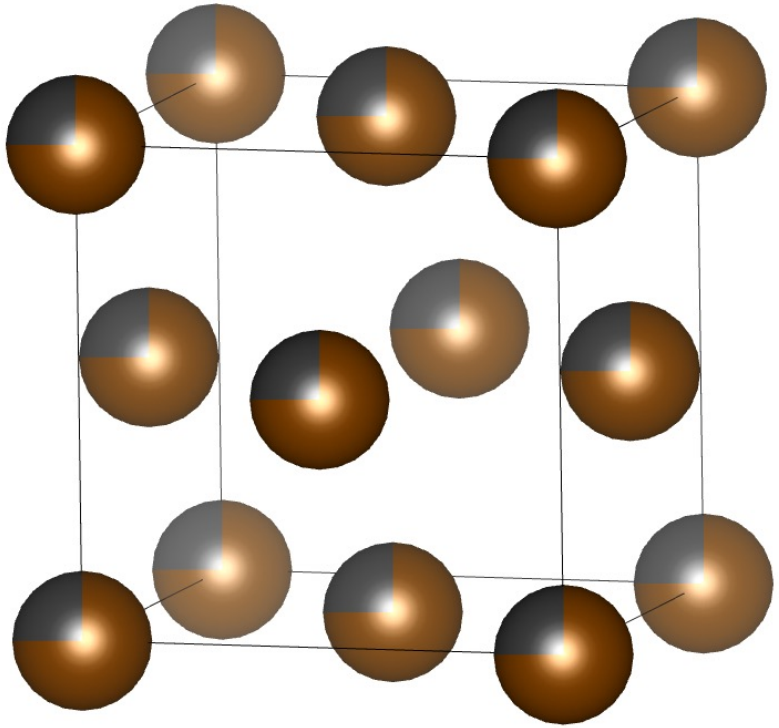
*A. Jensen *et al.*, J. Synchrotron Rad., 2022, **29**, 1420-1428

in situ Annealing of Brass

Brass – CuZn

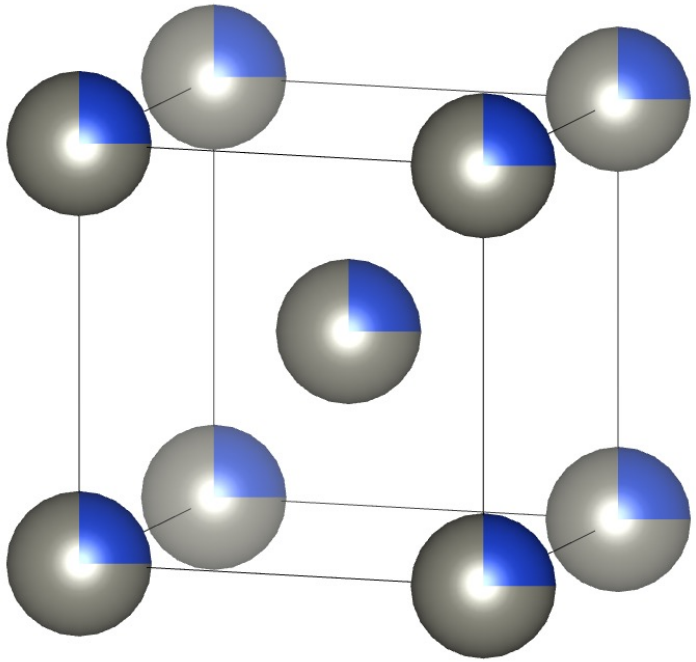

17
Cl
35.45
Atomic Number
Symbol
Atomic Mass
Alkali metals
Alkaline earth metals
Transition metals
Post-transition metals
Metalloids
Nonmetals
Halogens
Noble gases
Lanthanides
Actinides

FCC



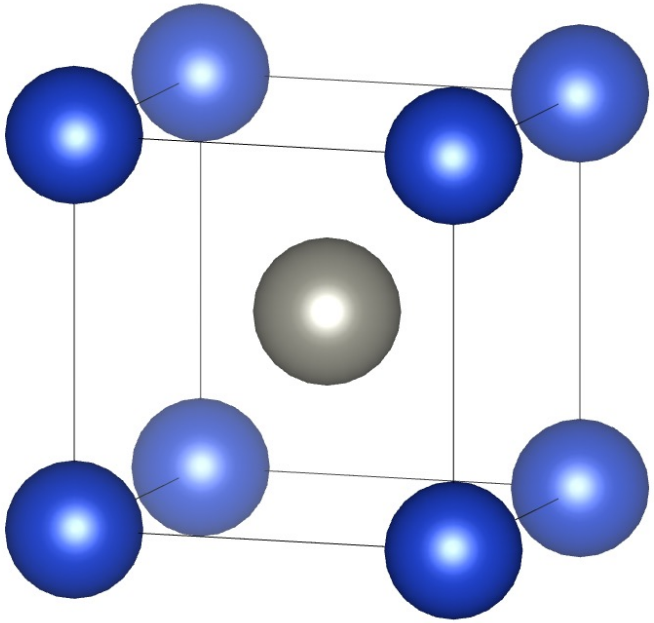
α-Brass

BCC

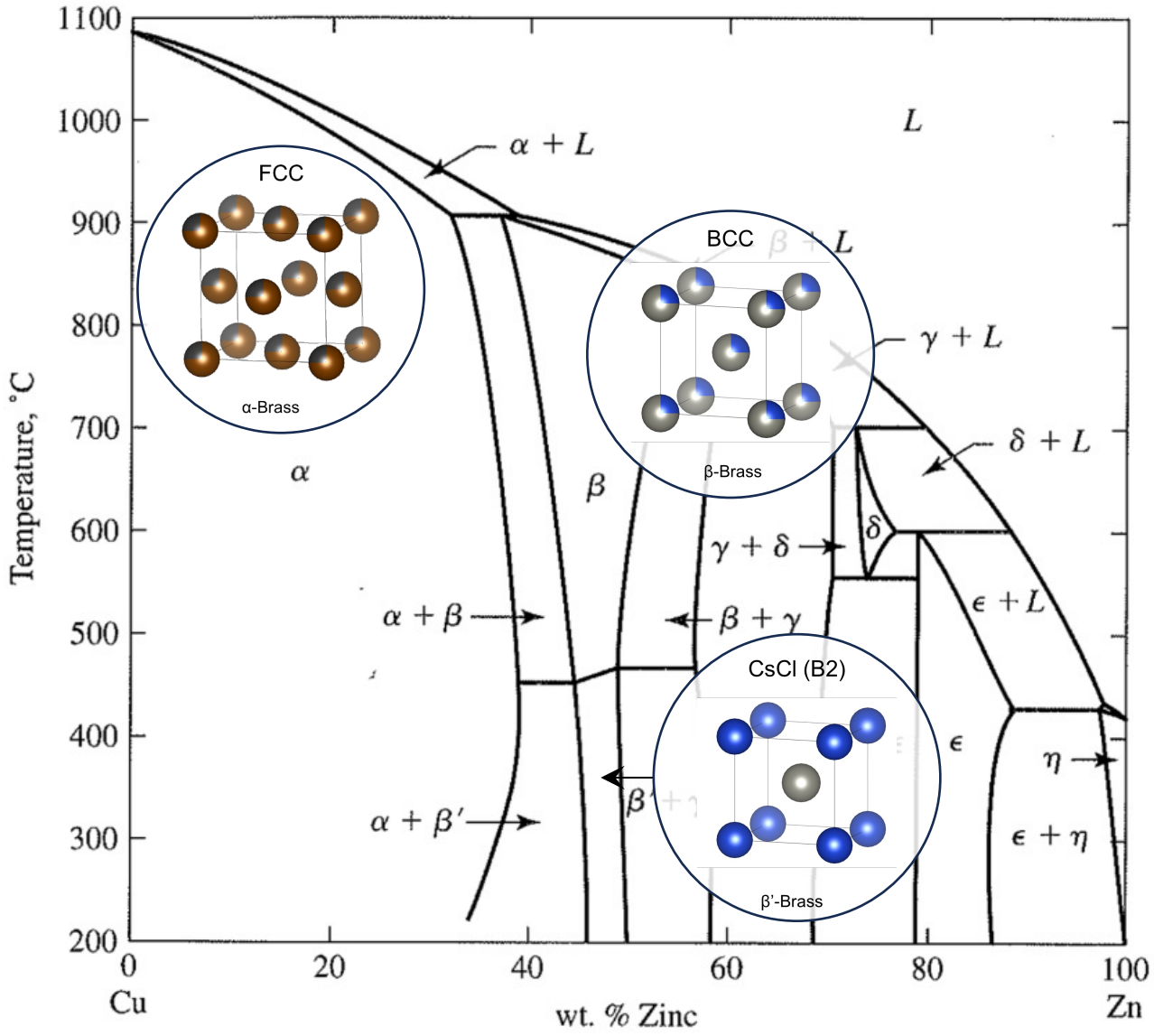


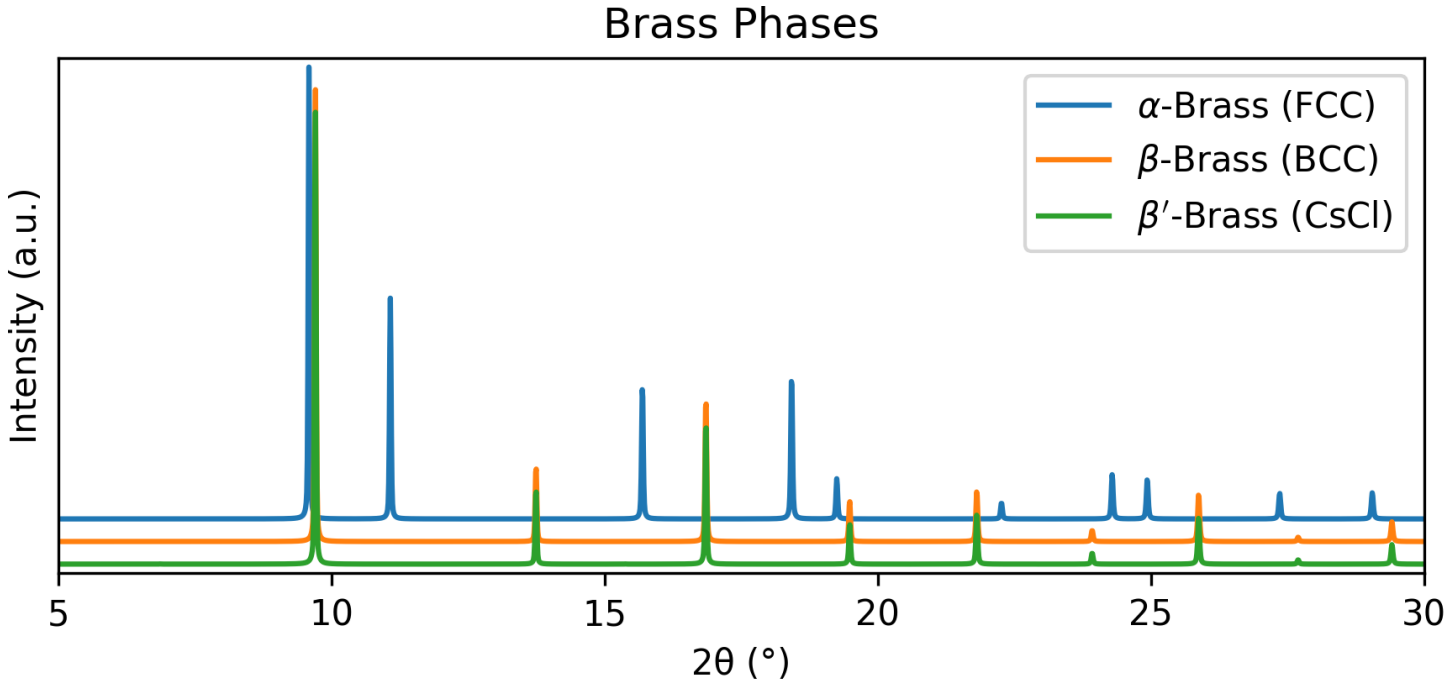
β-Brass

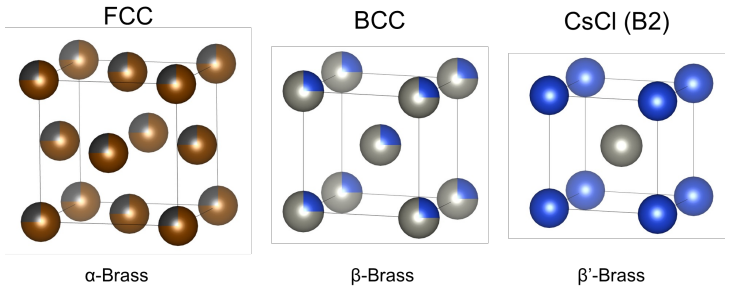
CsCl (B2)

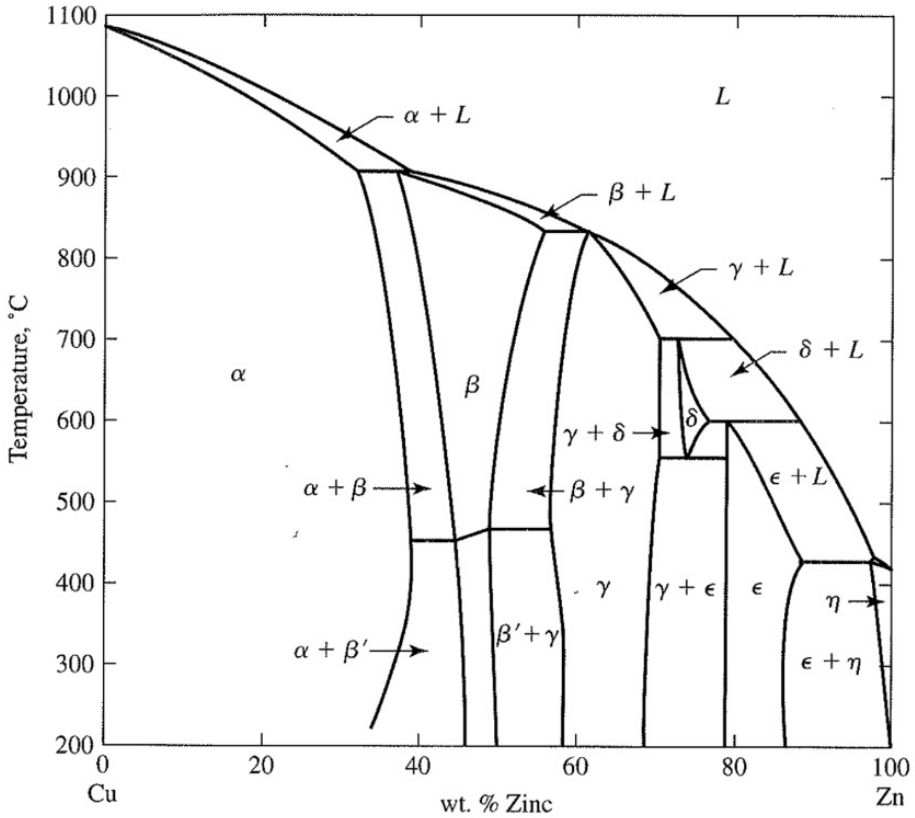


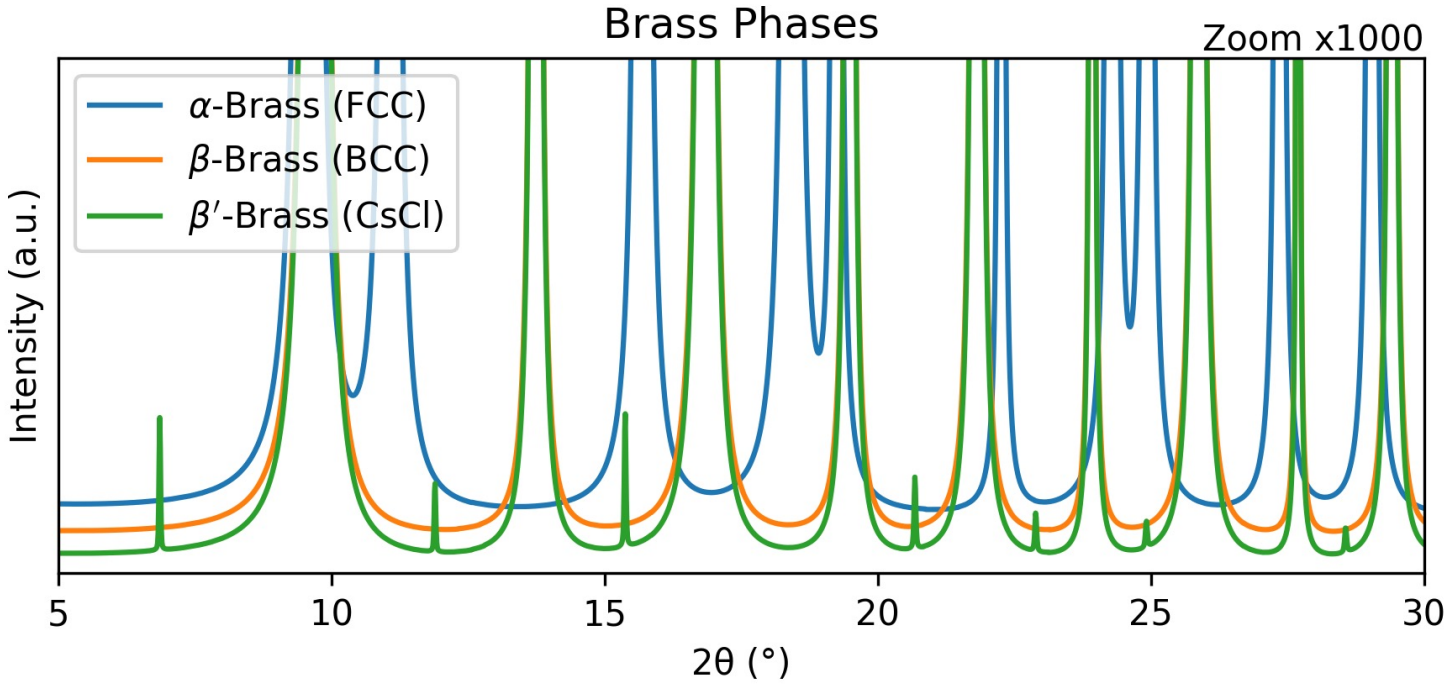
β’-Brass

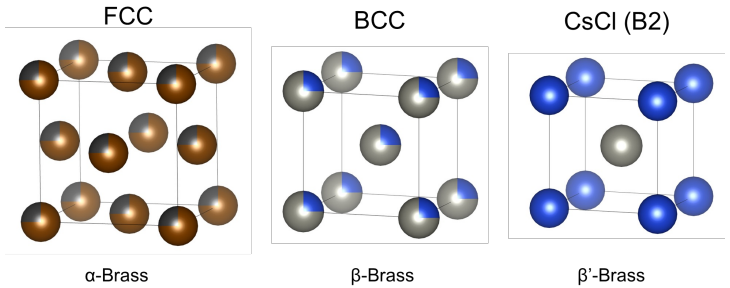

1100
1000
900
800
700
600
500
400
300
200
Temperature, °C
0
20
40
60
80
100
Cu
wt. % Zinc
Zn
L
α + L
γ + L
δ + L
ϵ + L
α
β
γ + δ
α + β
β + γ
α + β′
β′ + η
ϵ
η
ϵ + η
δ
FCC
α-Brass
BCC
β-Brass
CsCl (B2)
β'-Brass

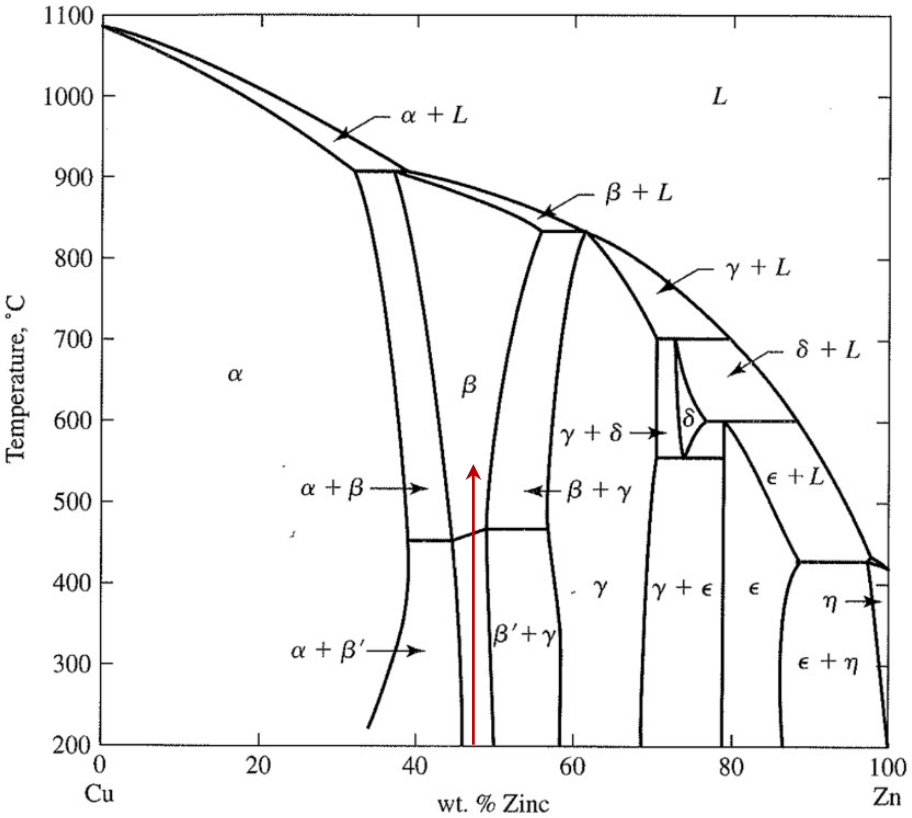

Brass Phases
α-Brass (FCC)
β-Brass (BCC)
β′-Brass (CsCl)
Intensity (a.u.)
5
10
15
20
25
30
2θ (°)

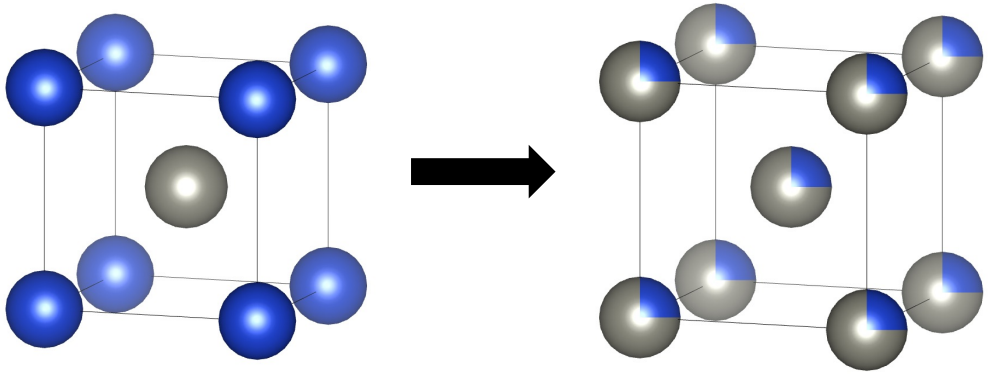

FCC
BCC
CsCl (B2)
α-Brass
β-Brass
β'-Brass


1100
1000
900
800
700
600
500
400
300
200
Temperature, °C
α + L
L
β + L
γ + L
δ + L
α
β
γ + δ
δ'
α + β
β + γ
ϵ + L
γ
γ + ϵ
ϵ
η
α + β′
β′ + γ
ϵ + η
0
20
40
60
80
100
Cu
wt. % Zinc
Zn



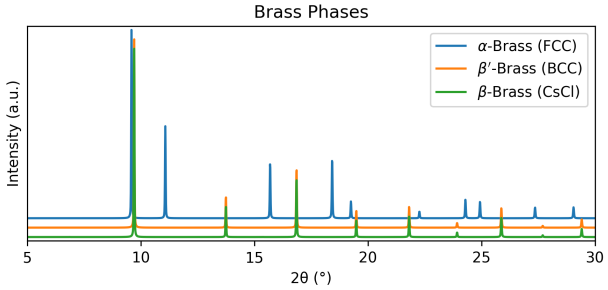


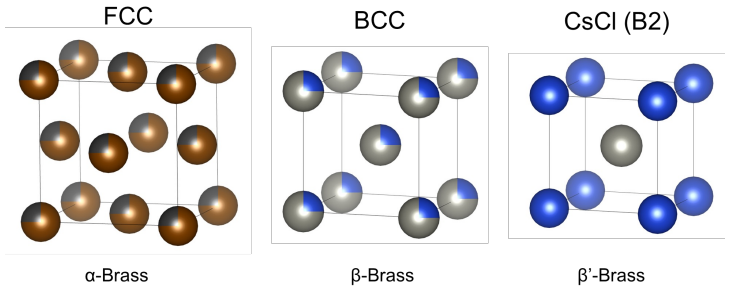


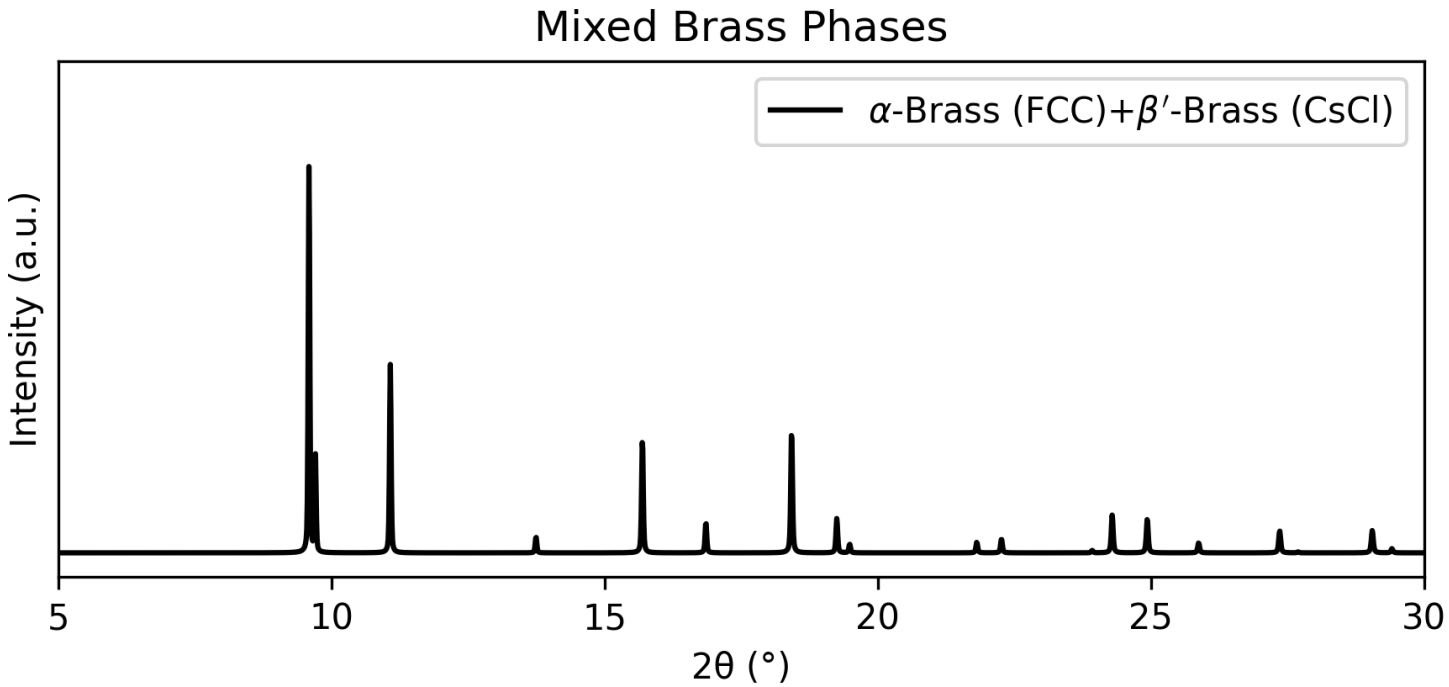


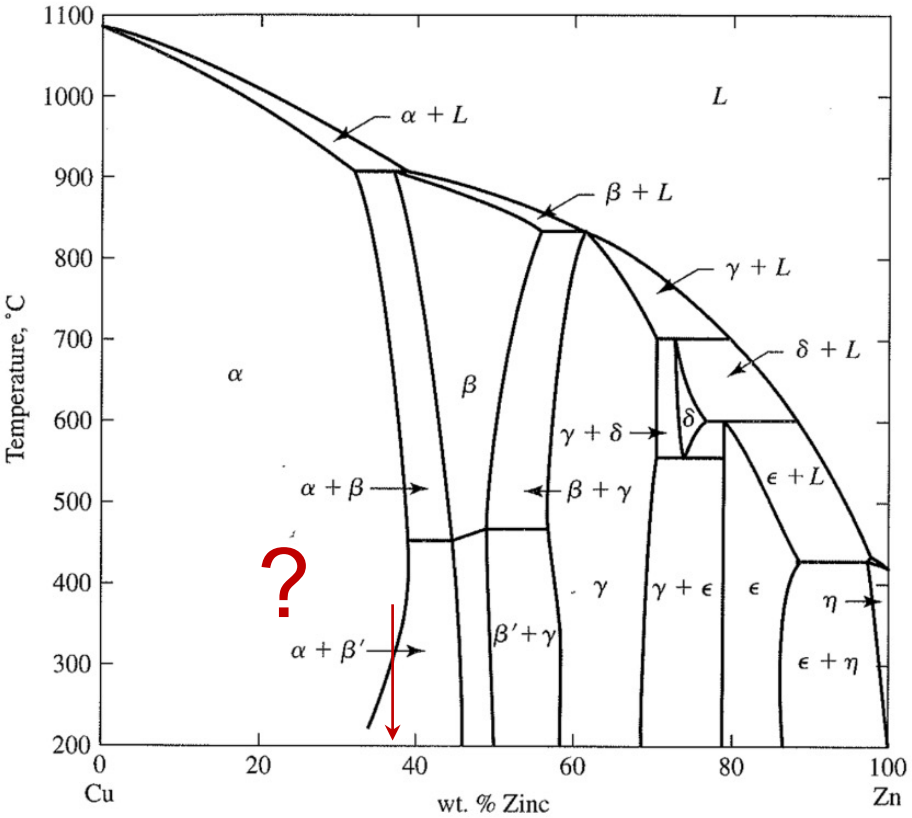
β'-Brass

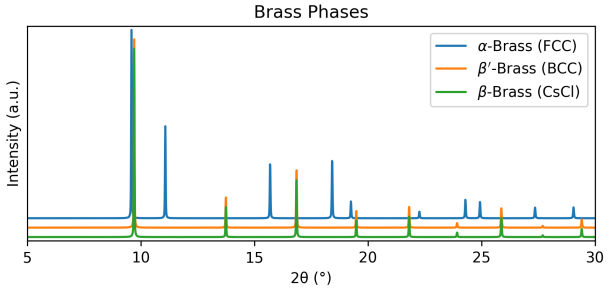
β-Brass


Brass Phases
α-Brass (FCC)
β′-Brass (BCC)
β-Brass (CsCl)
Intensity (a.u.)
5
10
15
20
25
30
2θ (°)

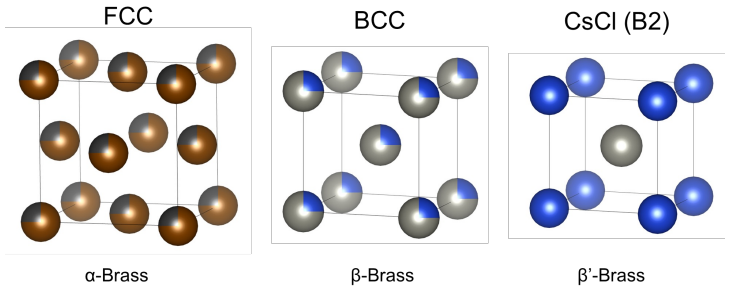

FCC
BCC
CsCl (B2)
α-Brass
β-Brass
β'-Brass

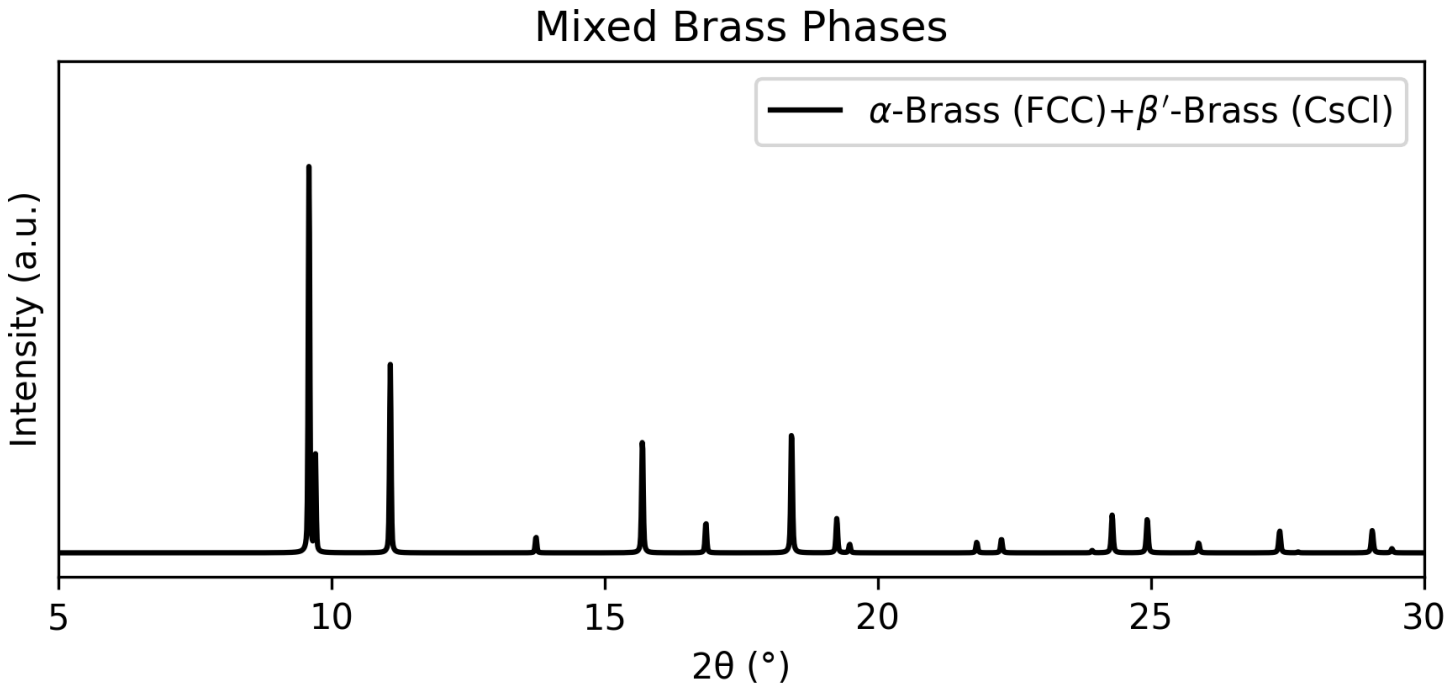

Mixed Brass Phases
α-Brass (FCC)+β′-Brass (CsCl)
Intensity (a.u.)
5
10
15
20
25
30
2θ (°)

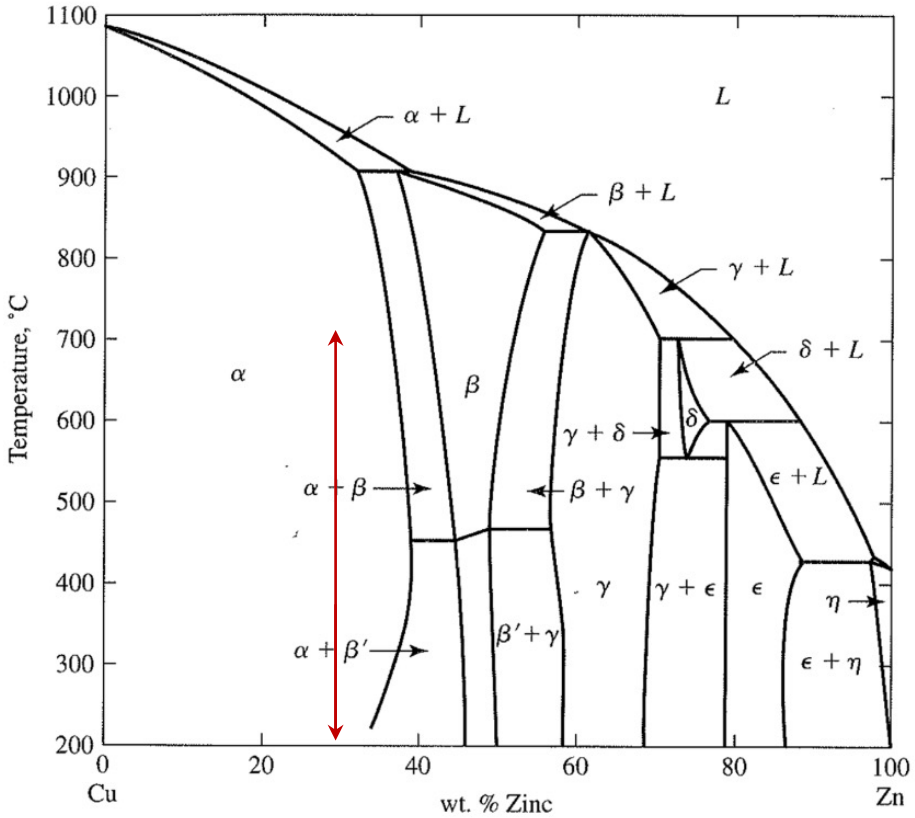

Temperature, °C
1100
1000
900
800
700
600
500
400
300
200
0
20
40
60
80
100
Cu
Zn
wt. % Zinc
α + L
L
β + L
γ + L
δ + L
α
β
γ + δ
δ'
α + β
β + γ
ϵ + L
?
γ
γ + ϵ
ϵ
η
α + β′
β′ + γ
ϵ + η


Brass Phases
α-Brass (FCC)
β′-Brass (BCC)
β-Brass (CsCl)
Intensity (a.u.)
5
10
15
20
25
30
2θ (°)

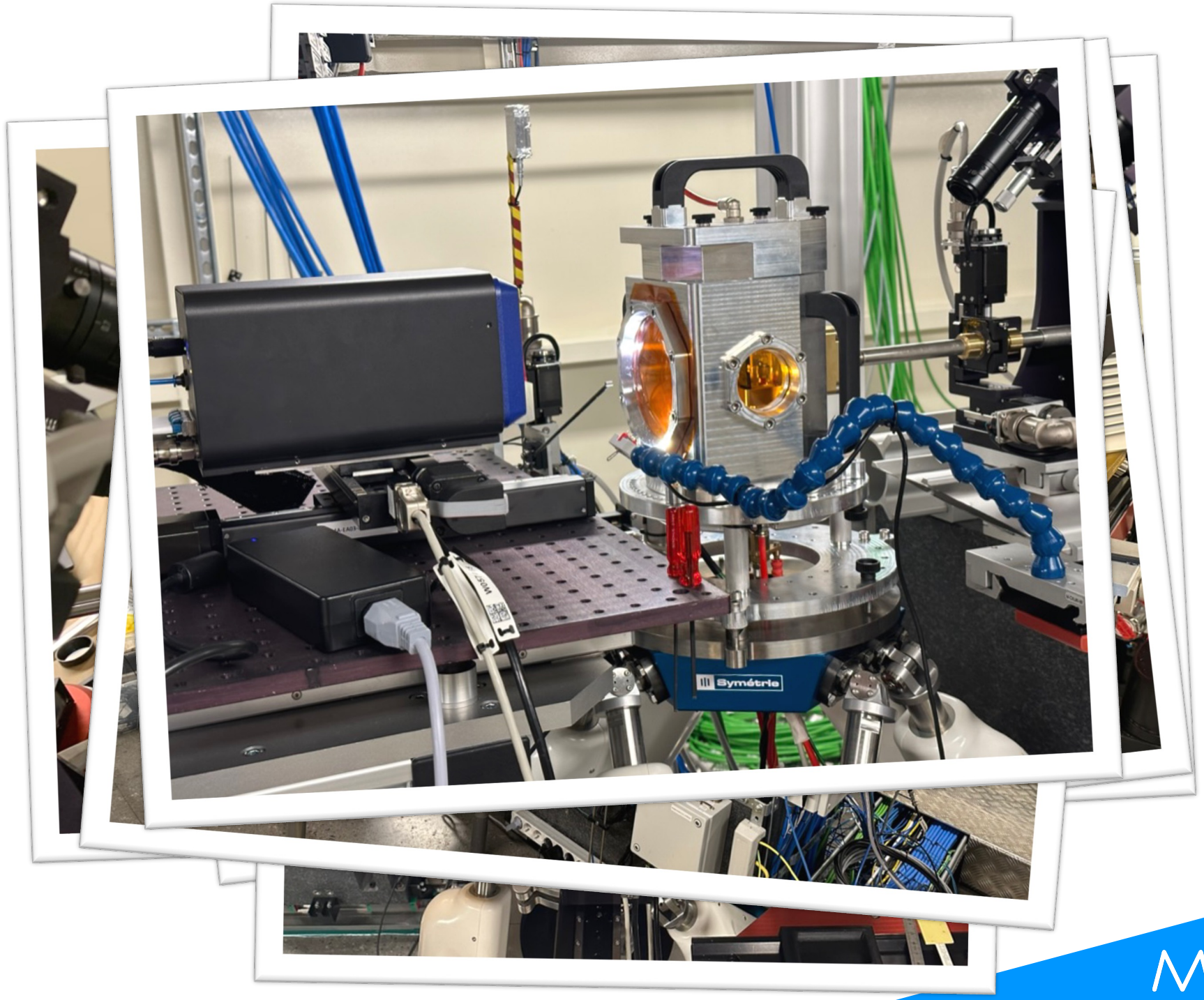
Annealing !


FCC
BCC
CsCl (B2)
α-Brass
β-Brass
β'-Brass

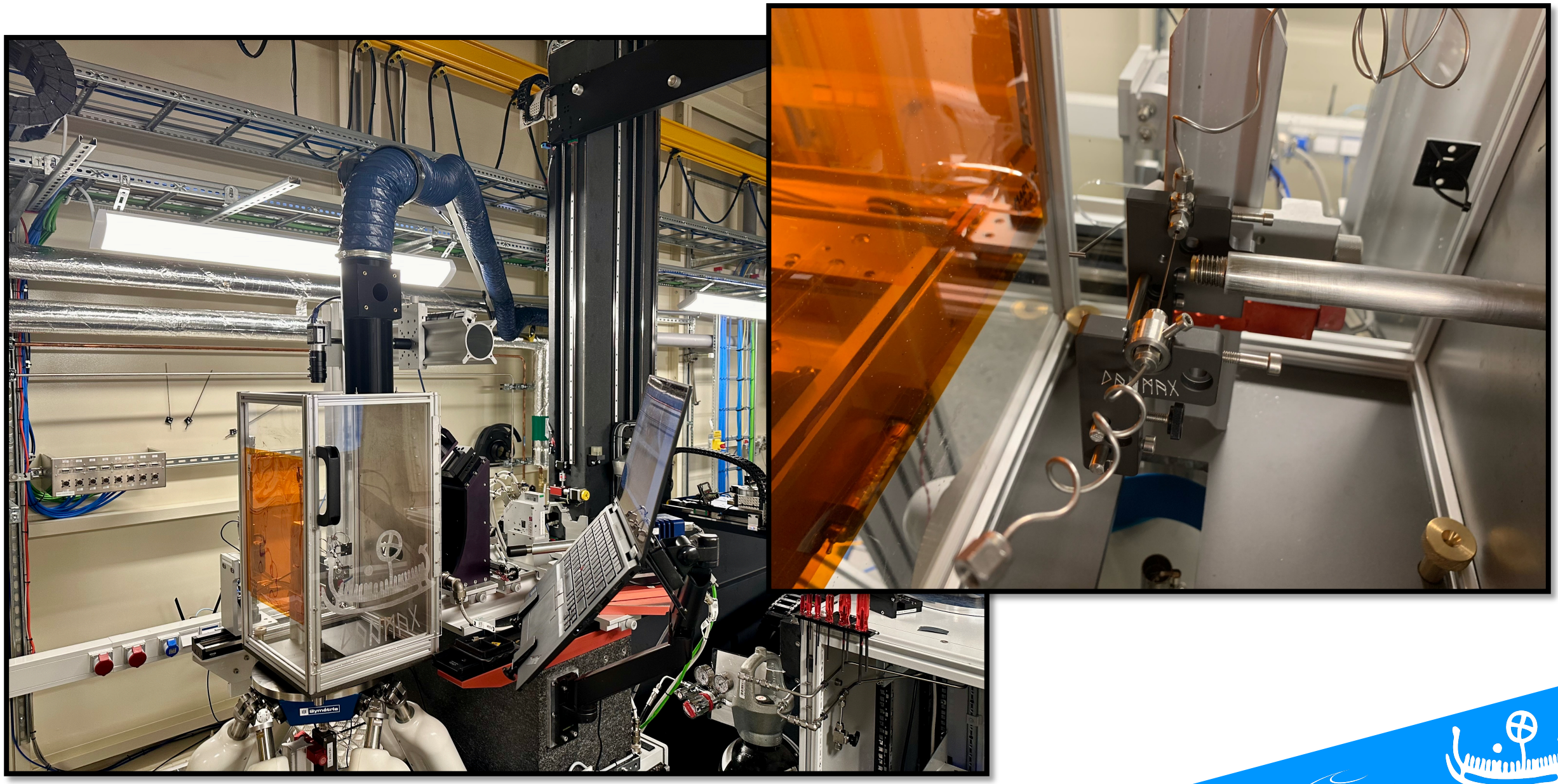

Mixed Brass Phases
α-Brass (FCC)+β′-Brass (CsCl)
Intensity (a.u.)
5
10
15
20
25
30
2θ (°)


Temperature, °C
1100
1000
900
800
700
600
500
400
300
200
0
20
40
60
80
100
Cu
Zn
wt. % Zinc
α + L
L
β + L
γ + L
δ + L
α
β
γ + δ
δ'
α + β
β + γ
ϵ + L
γ
γ + ϵ
ϵ
η
α + β′
β′ + γ
ϵ + η

Other experiments at DanMAX

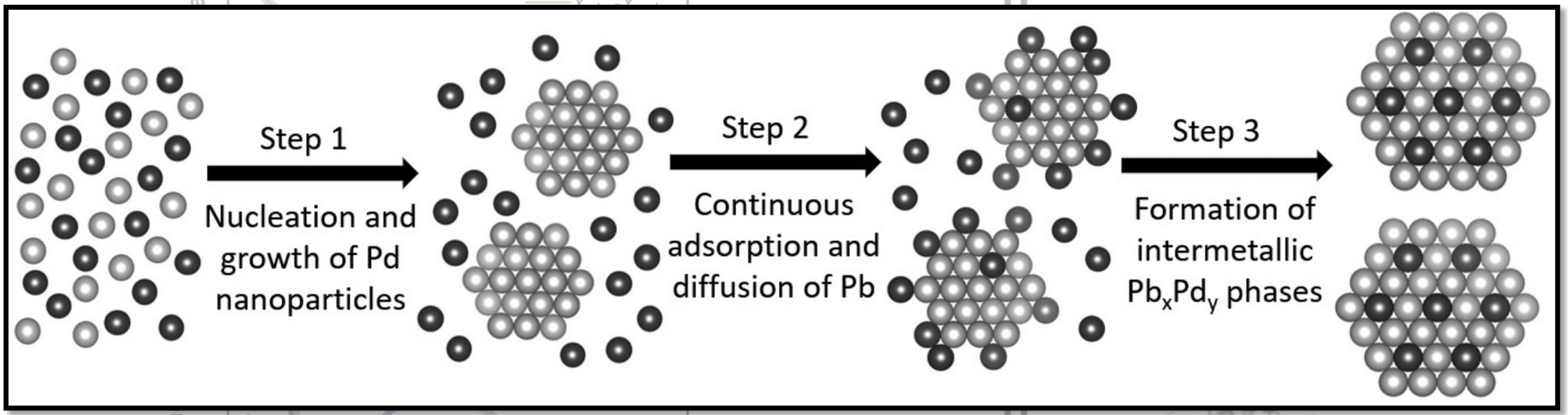

Symétrie

Solvothermal reactor



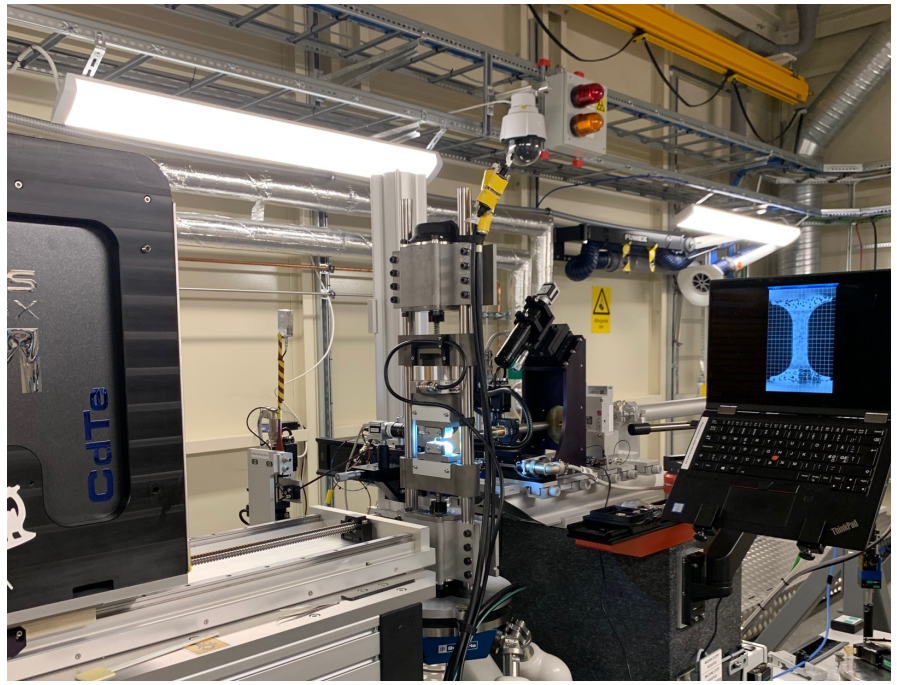
M. Roelsgaard *et al.*, Journal of Applied Crystallography. 2023, **56**, 581-588

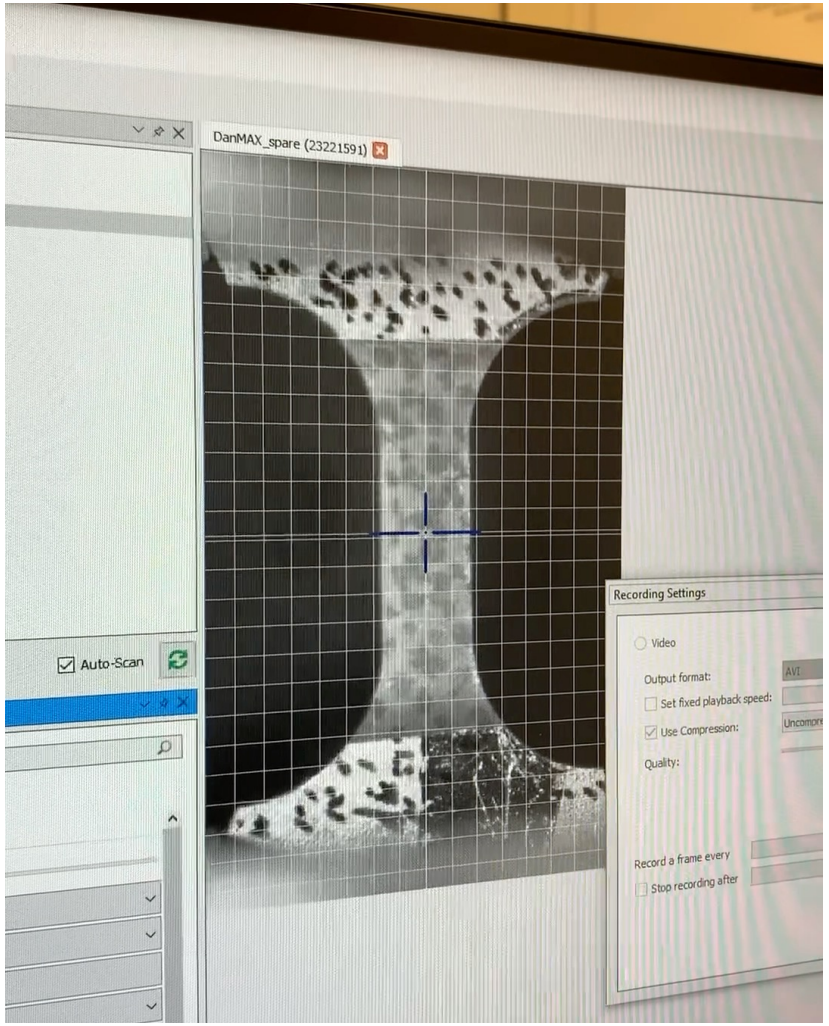
Investigating the formation of PbPd

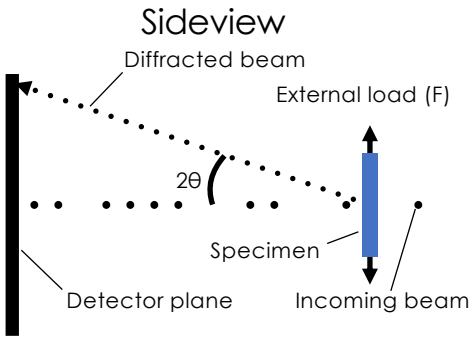


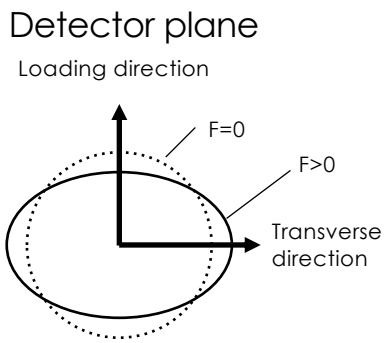
A. B. Borup *et al.*, Nanoscale. 2023, **15**, 18481

Transformation induced plasticity (TRIP) steel



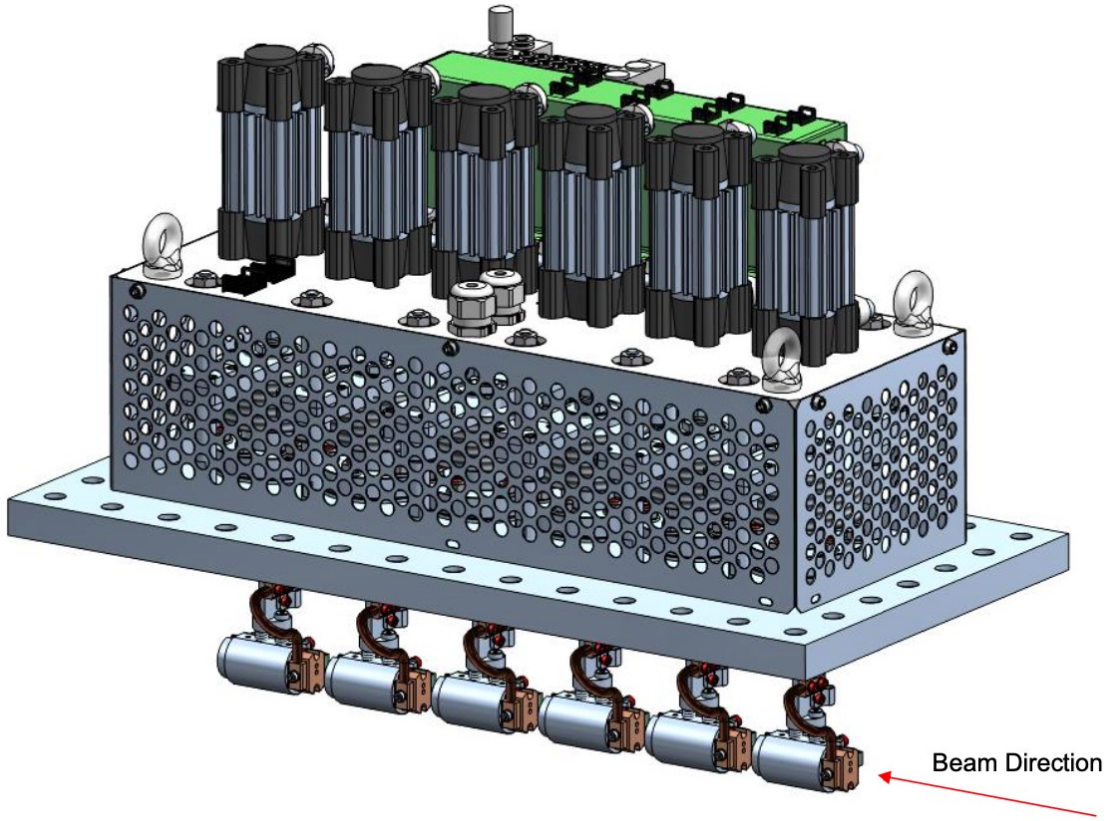


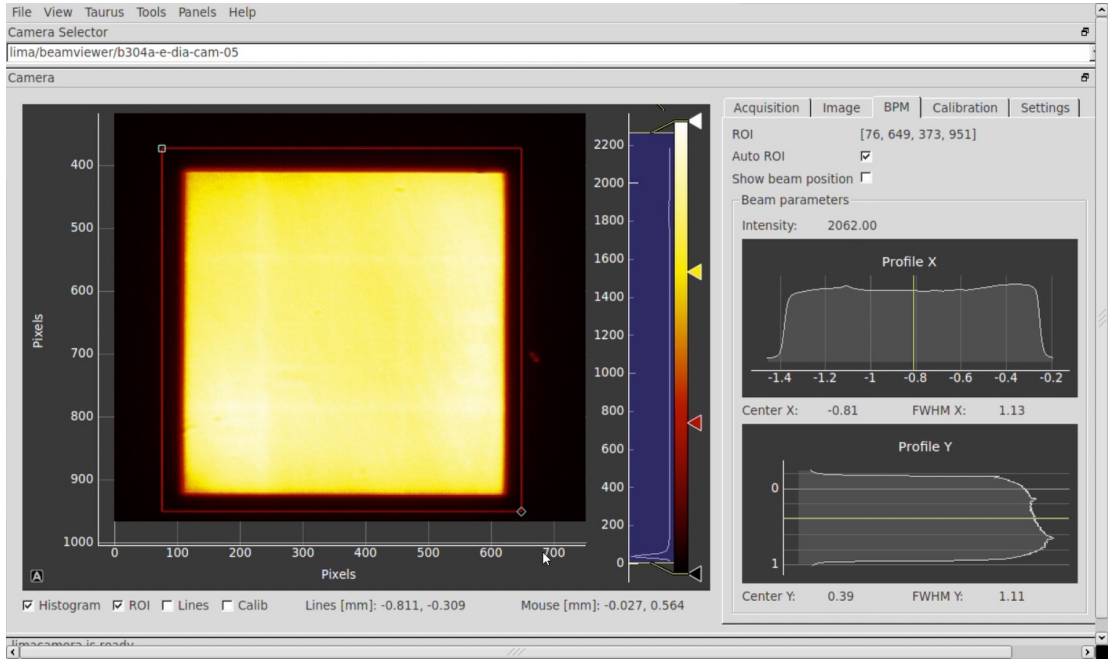




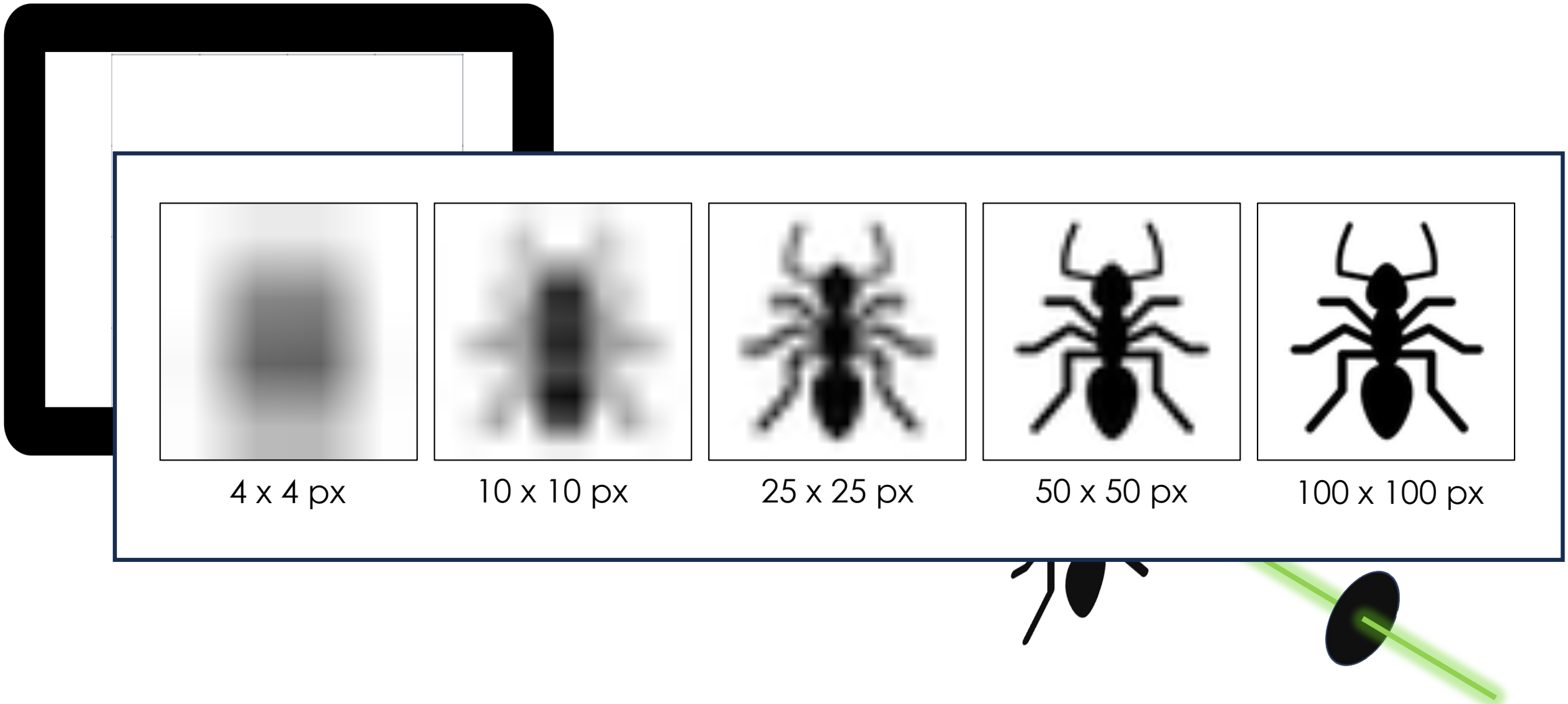
M. Isakov *et al.*, Metall. Mater. Trans. A, 2023, **54**, 1320-1331

Beam focusing - imaging using µXRD and µXRF

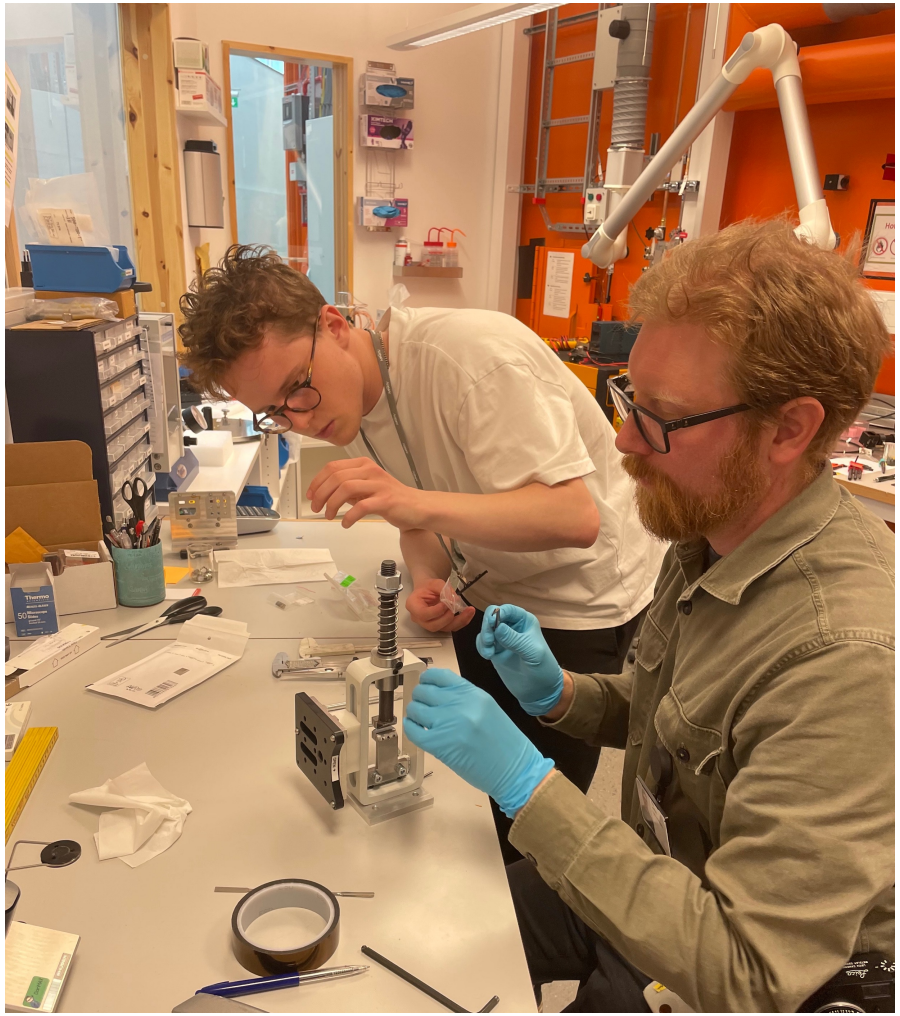


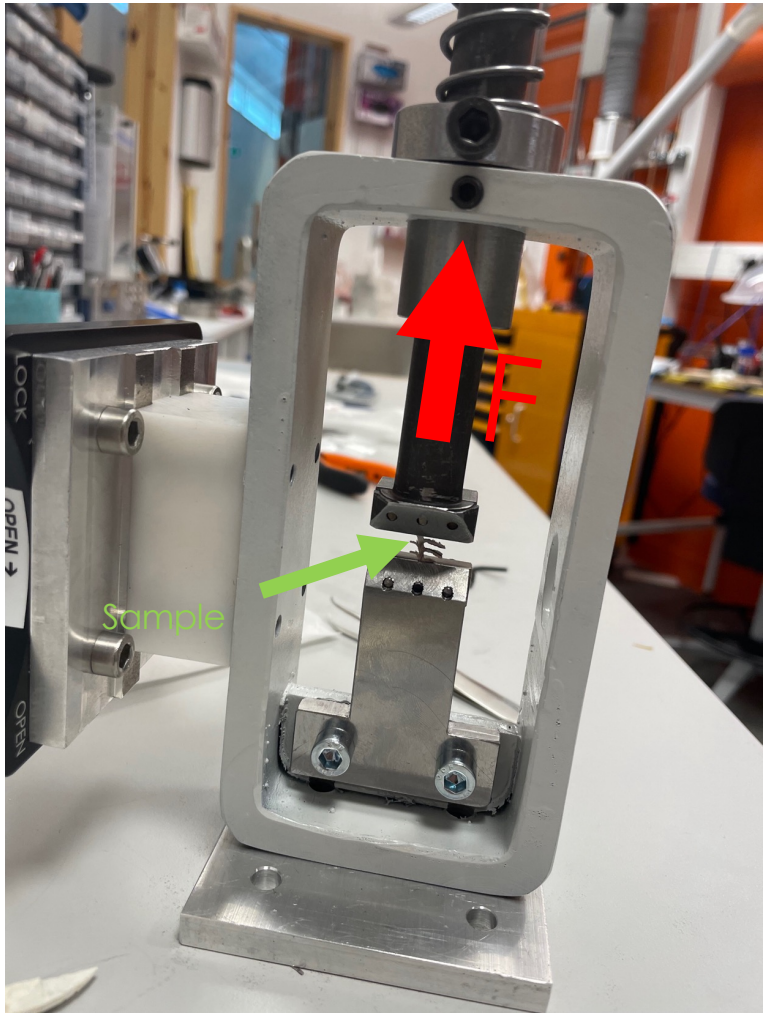


µXRD (and µXRF) mapping



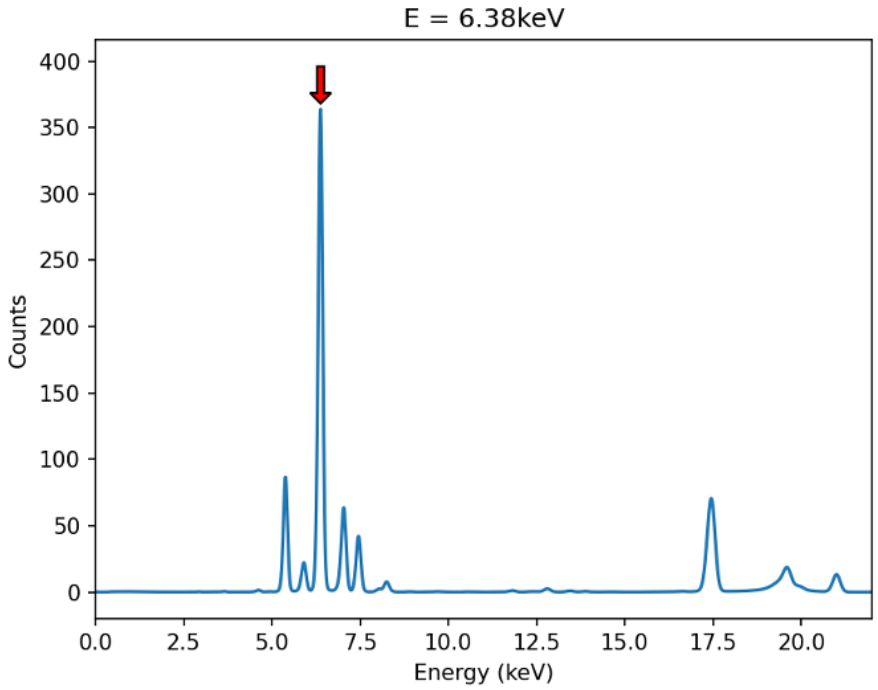
Heat exchanger braze joint (under tension)

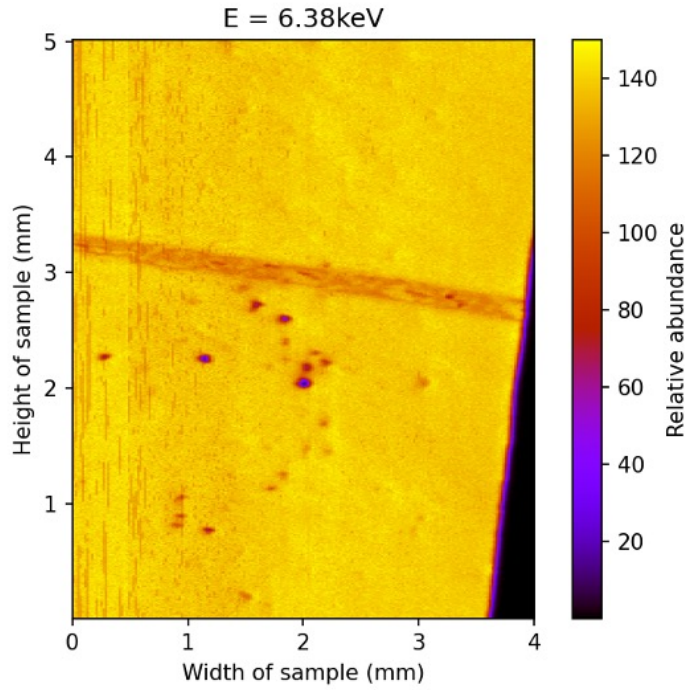




PI: F. Lenrick, Lund U.

XRF maps - ROI on raw spectra





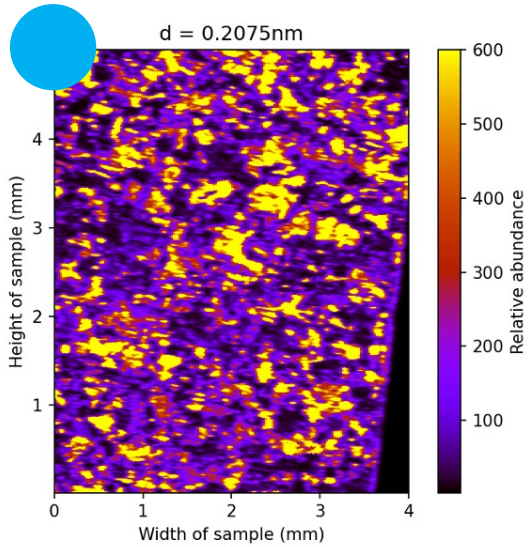
Select energy ROI ➡️ Sum intensity in ROI for each point ➡️ Plot map

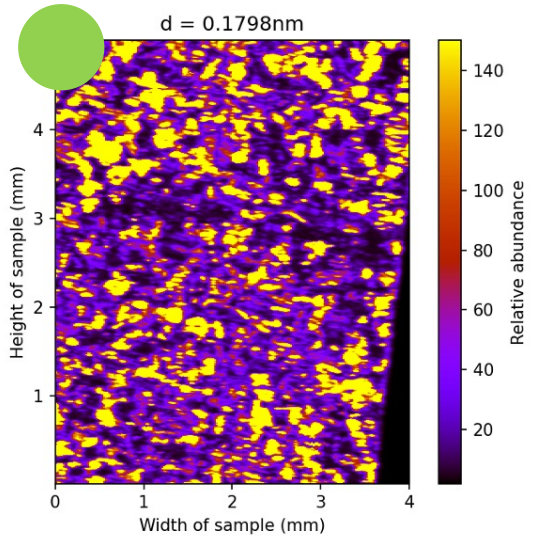
Pixel size:
10 µm x 20 µm
(V x H)

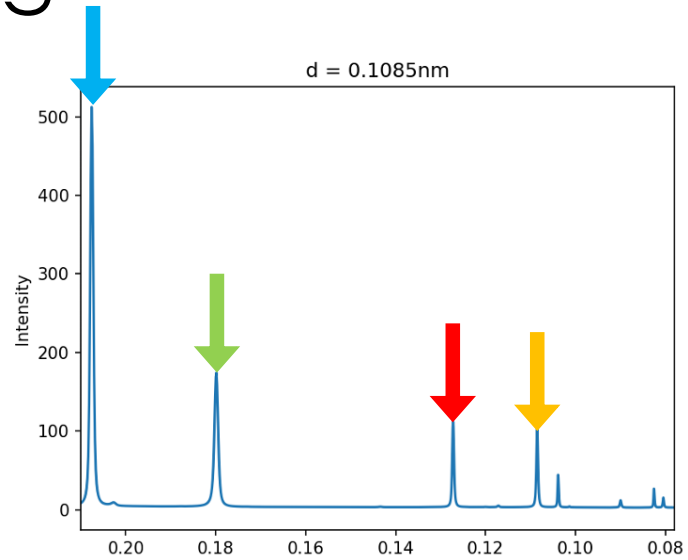
Scan time
1:20 h
100701 points

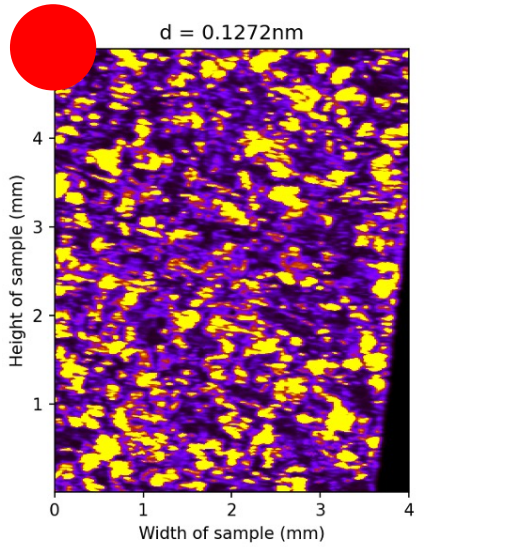
PI: F. Lenrick, Lund U.

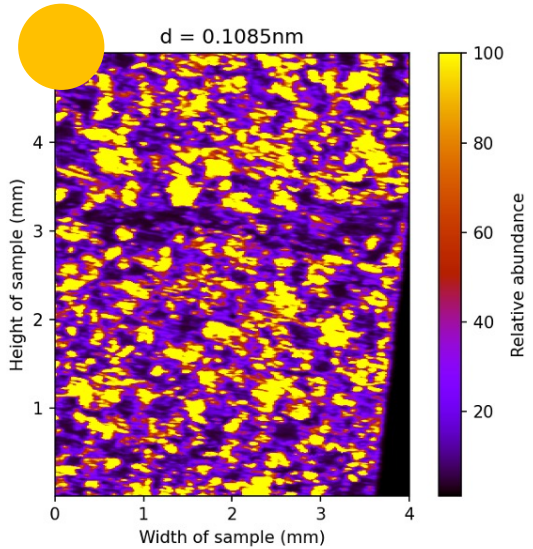
XRD - Far from the braze zone - large grains

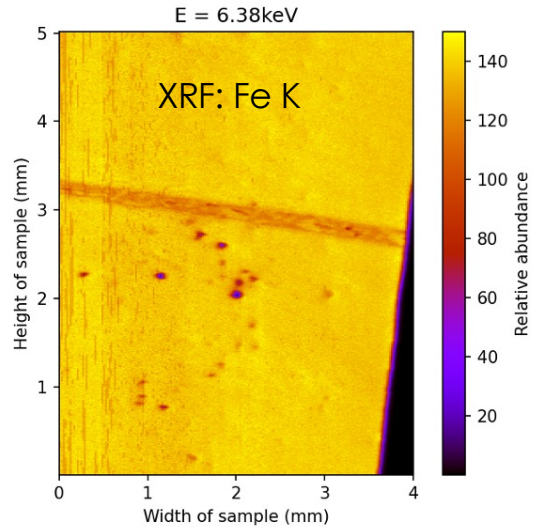










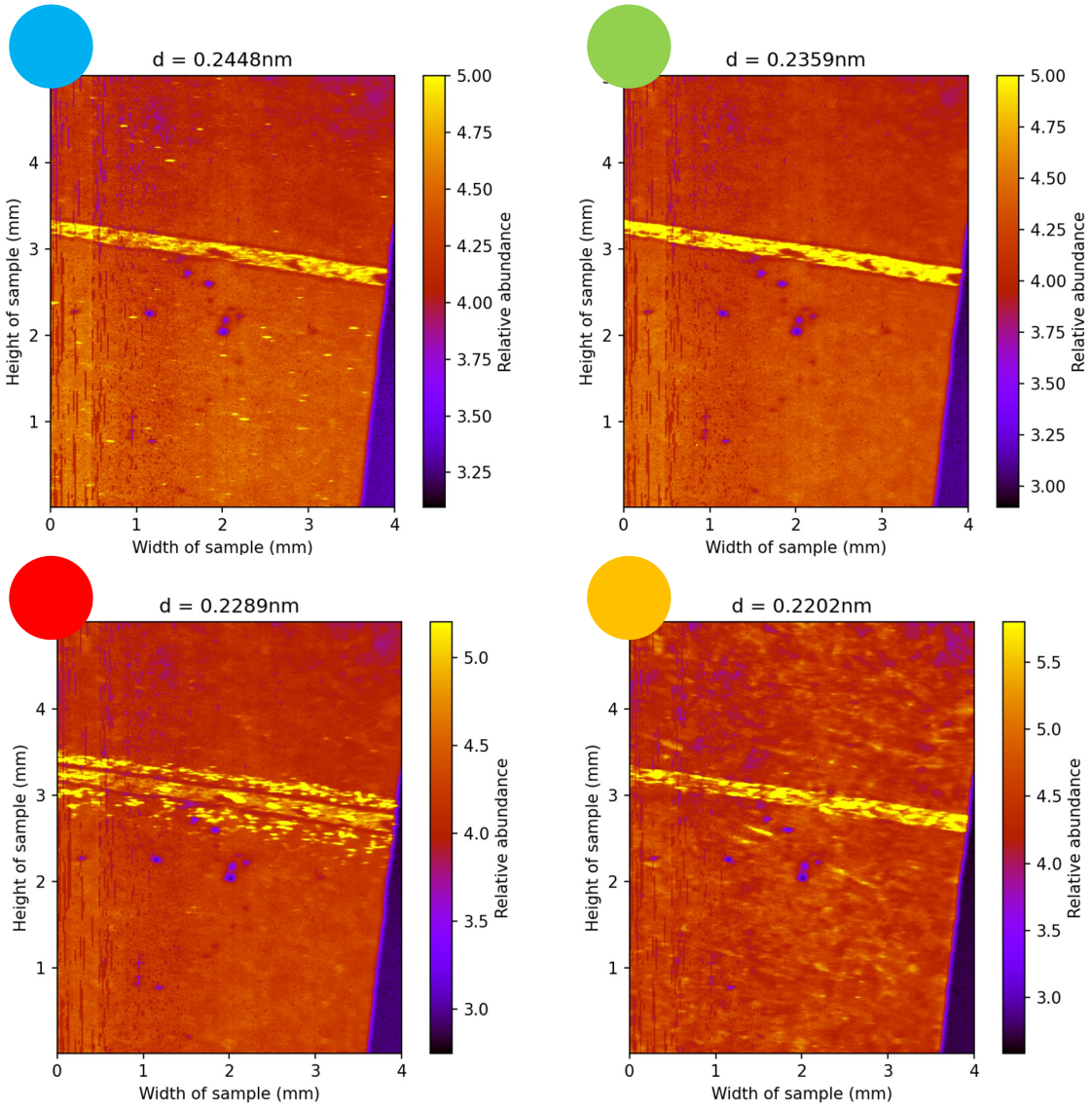


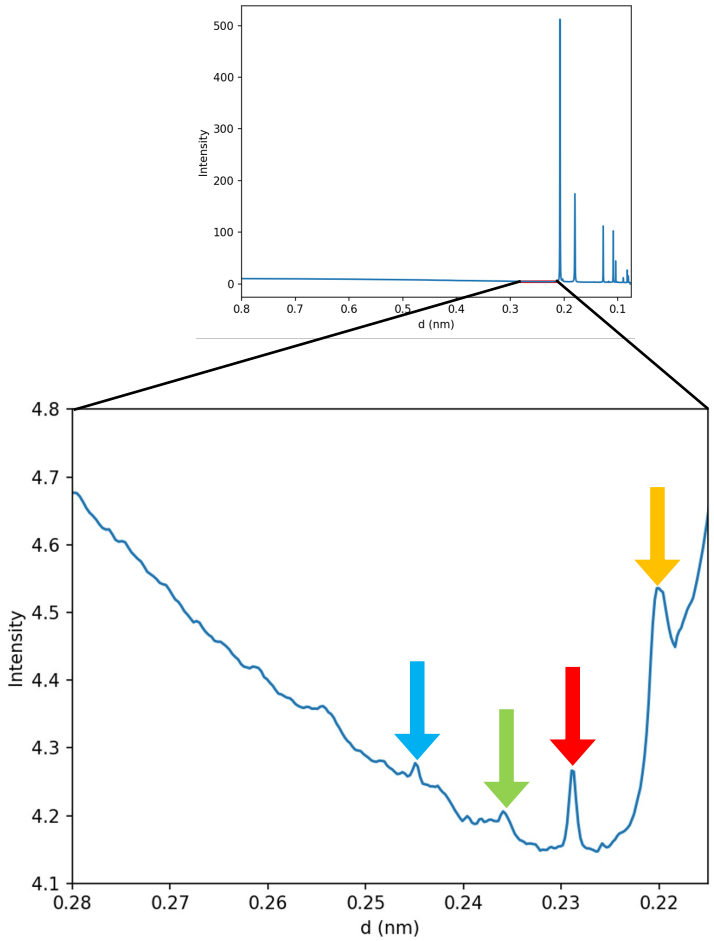
Pixel size:
10 µm x 20 µm
(V x H)

Scan time
1:20 h
100701 points

PI: F. Lenrick, Lund U.

XRD - Close to the braze zone



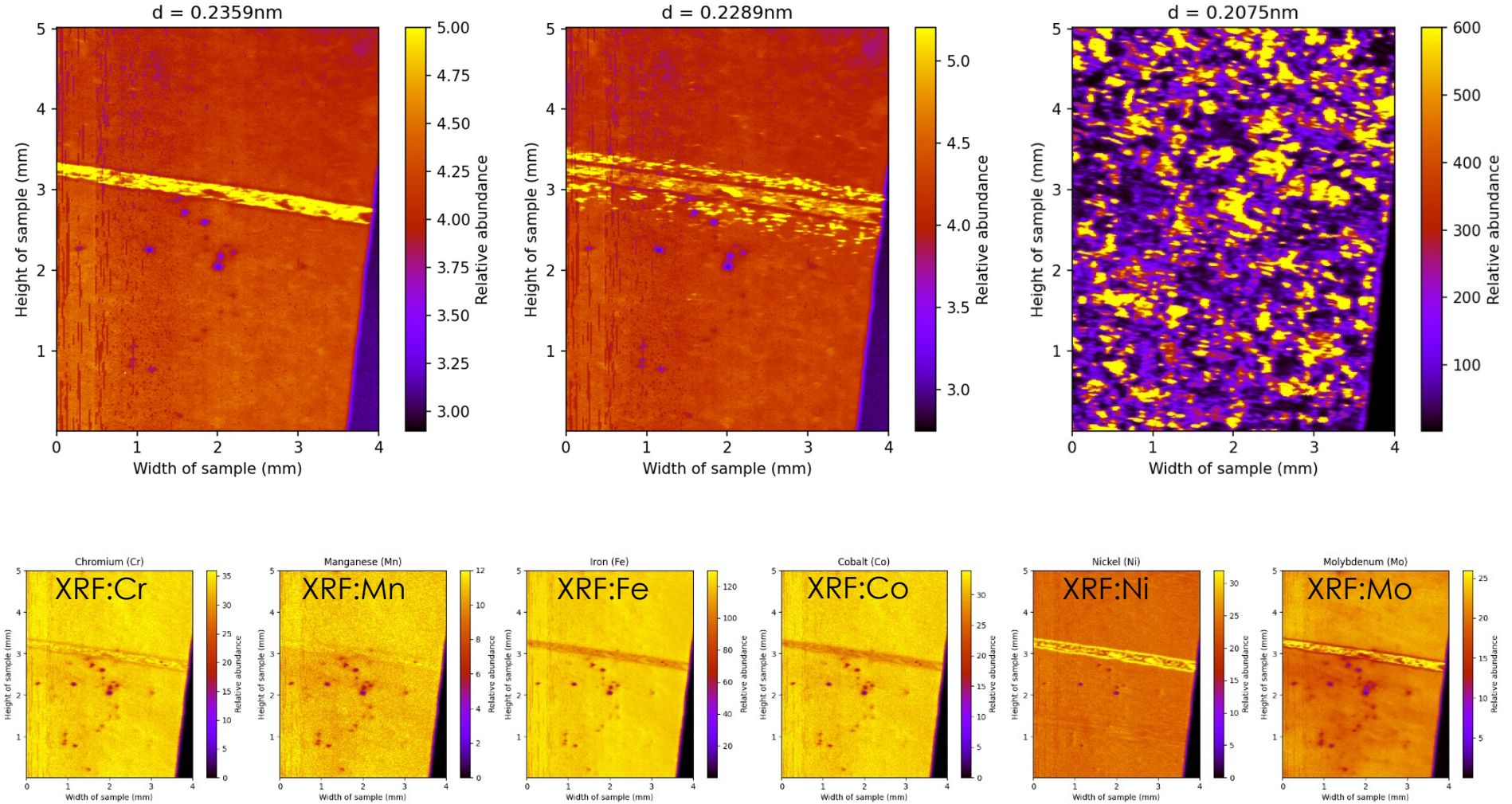


Pixel size:
10 µm x 20 µm
(V x H)

Scan time
1:20 h
100701 points

PI: F. Lenrick, Lund U.

Combining XRD and XRF



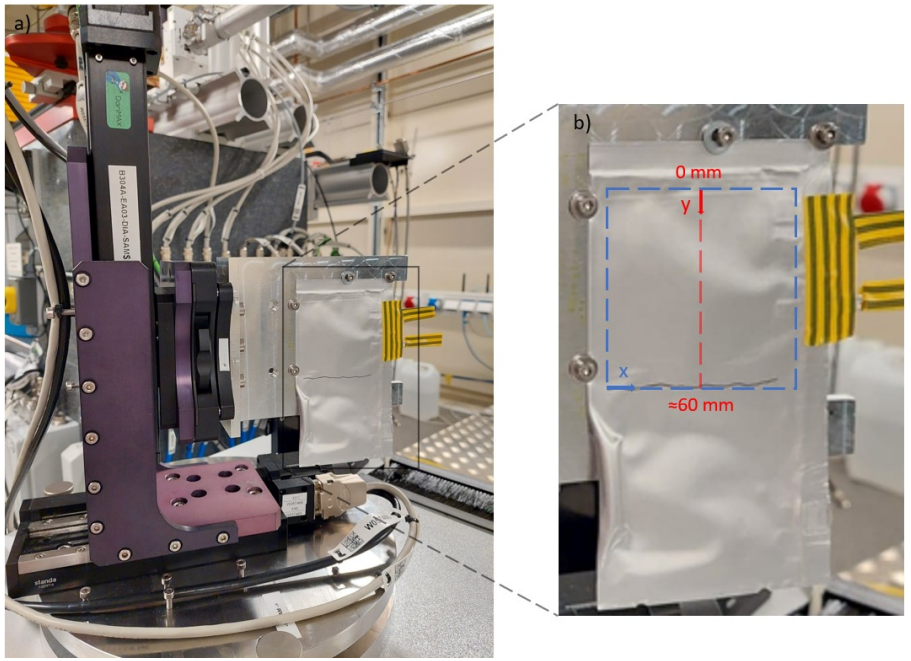
Pixel size:
10 µm x 20 µm
(V x H)

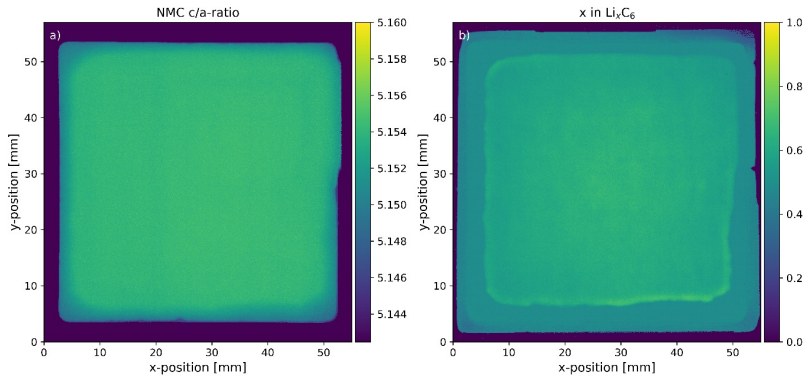
Scan time
1:20 h
100701 points

PI: F. Lenrick, Lund U.

Evaluating homogeneity during fast cycling

- Single layer Li-ion pouch cell made by KIT
- Cells are initially very homogeneous as confirmed by 2D µXRD mapping
- A line scan was used as a proxy for homogeneity during charging at 1C and 3C
  - 100 µm spatial resolution, 25 Hz data collection, approx. 30 s time resolution (one line of 600 points)

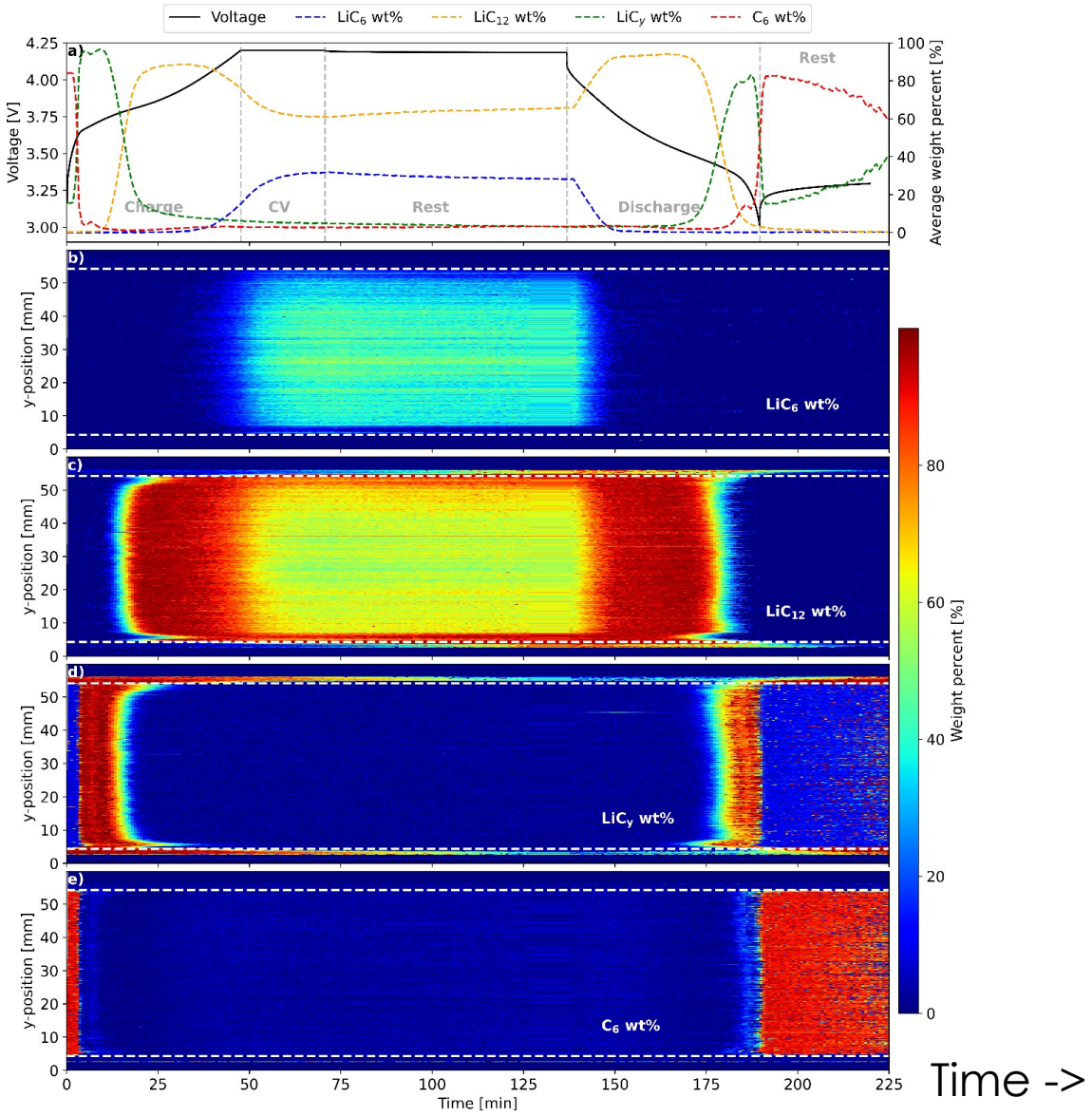




NMC c/a-ratio x in Li_xC_6

D. R. Sørensen *et al.*,: In Operando Evaluation of Heterogeneity Development in Fast-Cycled Single-Layer Pouch Cells. Chem. Mater. *Accepted*

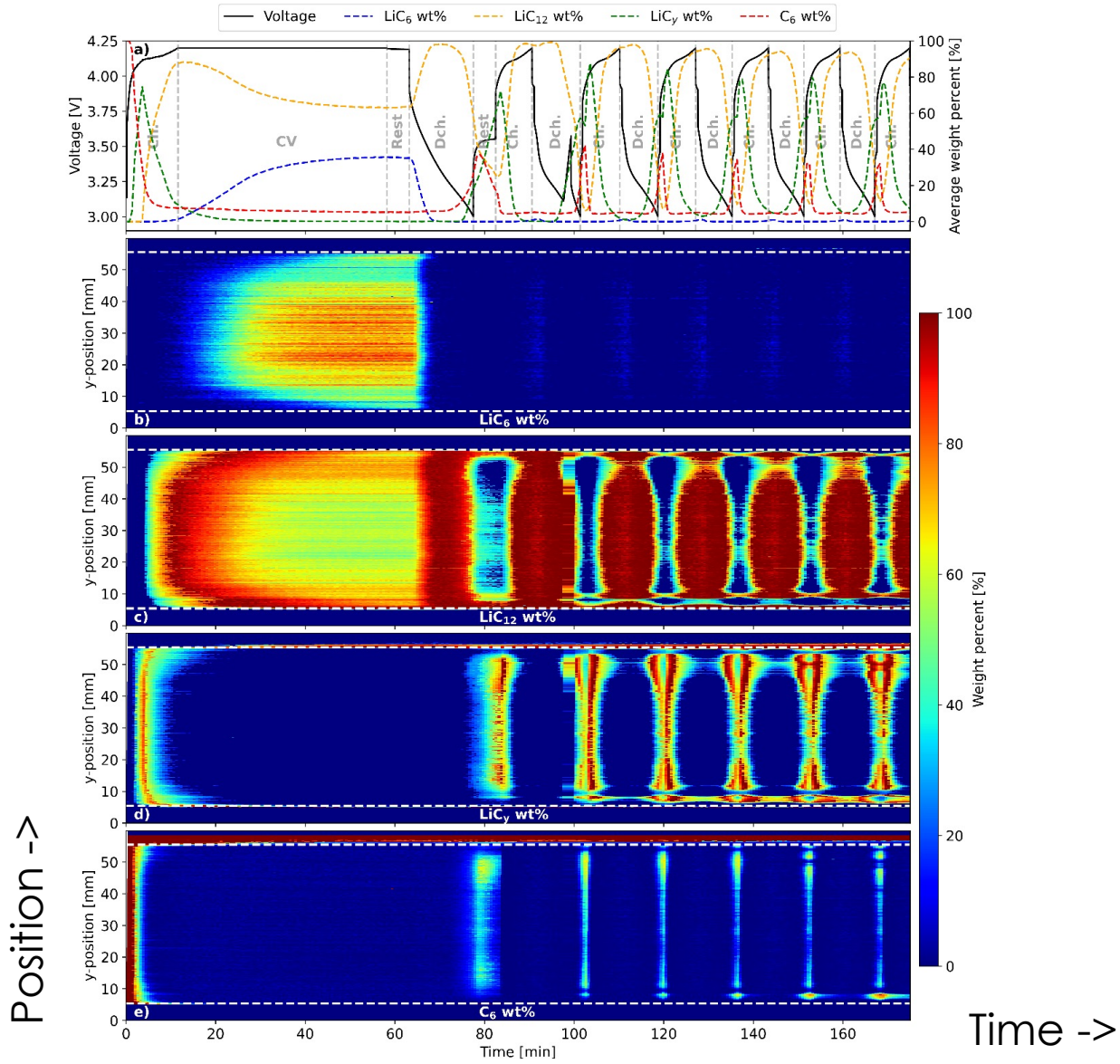
Evaluating homogeneity during fast cycling



- A single charge cycle at 1C reveals expected lithiation behavior:
  - $C_6 \rightarrow LiC_y \rightarrow LiC_{12} \rightarrow LiC_{12} + LiC_6$
- The charging and discharging are quite homogeneous, but the edges lag behind the center of the cell.

D. R. Sørensen *et al.*,: In Operando Evaluation of Heterogeneity Development in Fast-Cycled Single-Layer Pouch Cells. Chem. Mater. *Accepted*

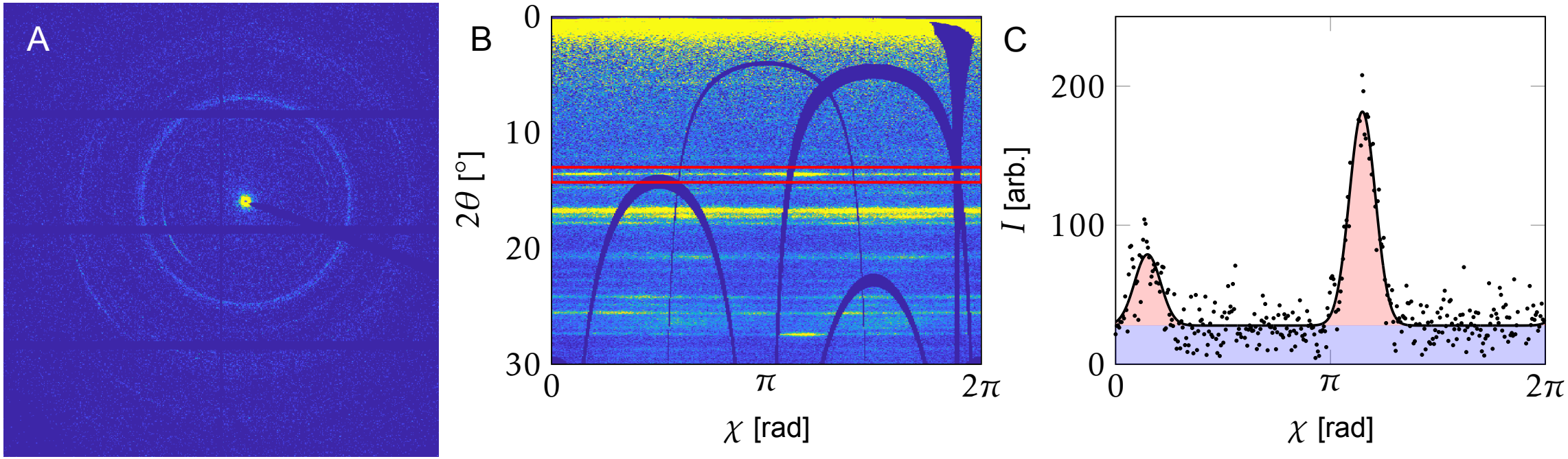
Evaluating homogeneity during fast cycling



- Repeated charge cycles at 3C reveal:
- The induced inhomogeneity is higher in the anode than in the cathode
- The charging is less homogeneous
- At high discharge rates, LiC_{12} is also converted to C_6 directly, not only via LiC_y as seen for lower discharge rates.

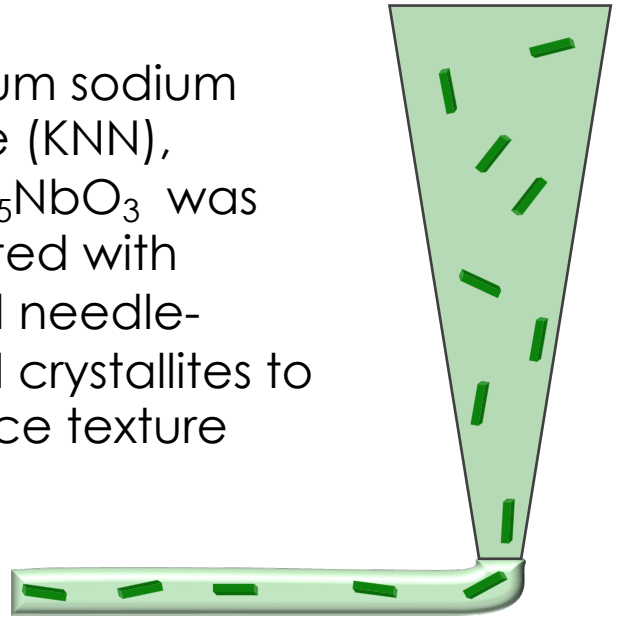
D. R. Sørensen *et al.*,: In Operando Evaluation of Heterogeneity Development in Fast-Cycled Single-Layer Pouch Cells. Chem. Mater. *Accepted*

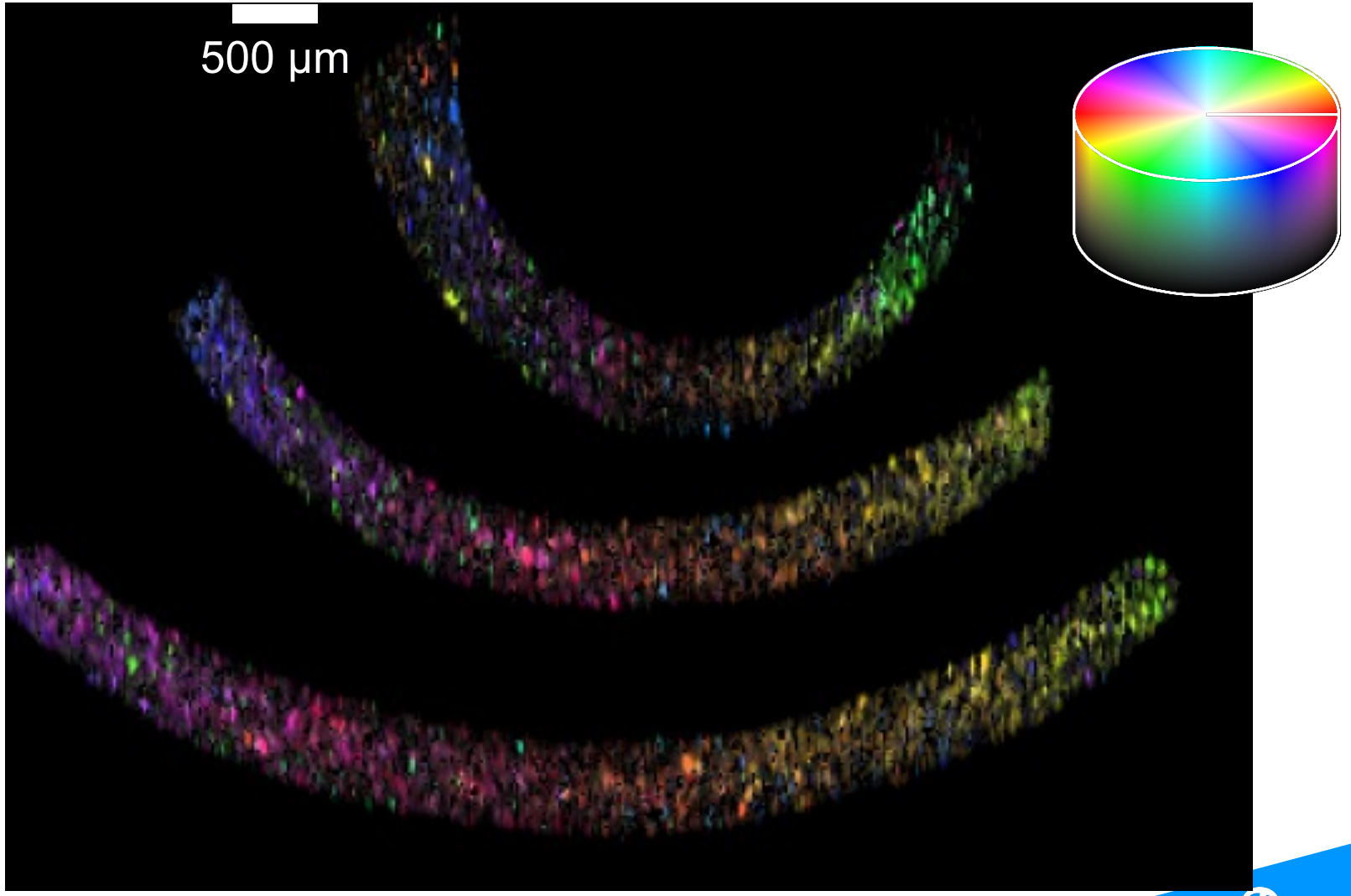
Mapping of crystallographic texture



3D-printing with template crystallites

Potassium sodium niobate (KNN), $K_{0.5}Na_{0.5}NbO_3$ was 3D printed with aligned needle-shaped crystallites to introduce texture

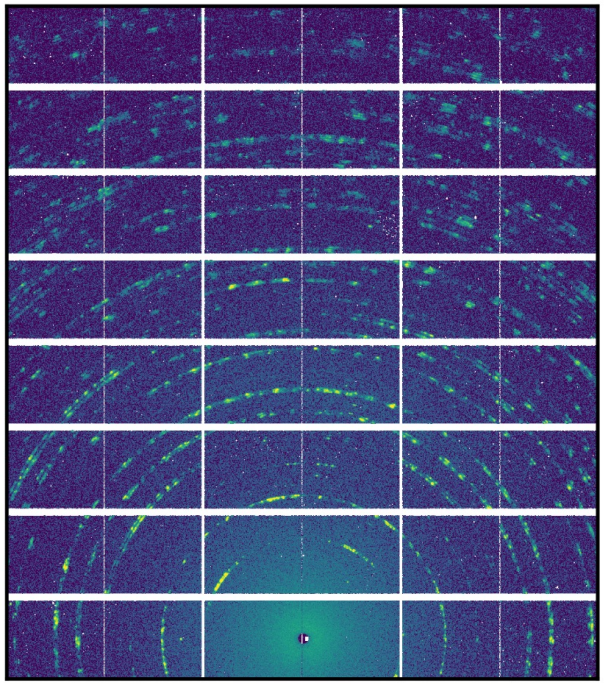


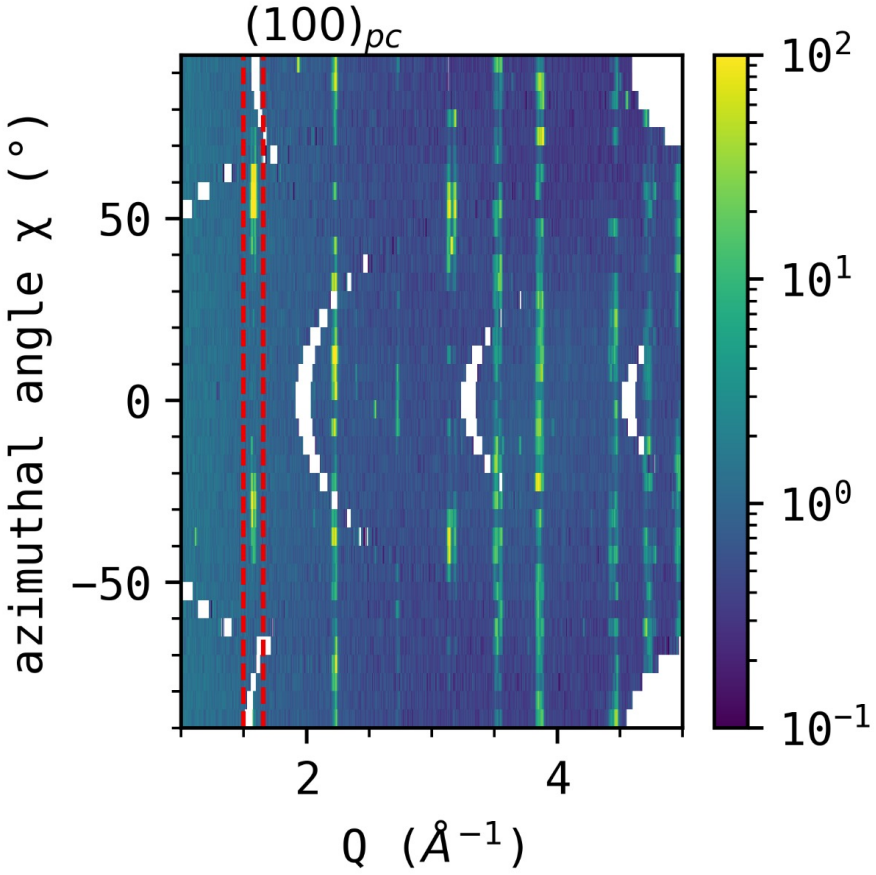


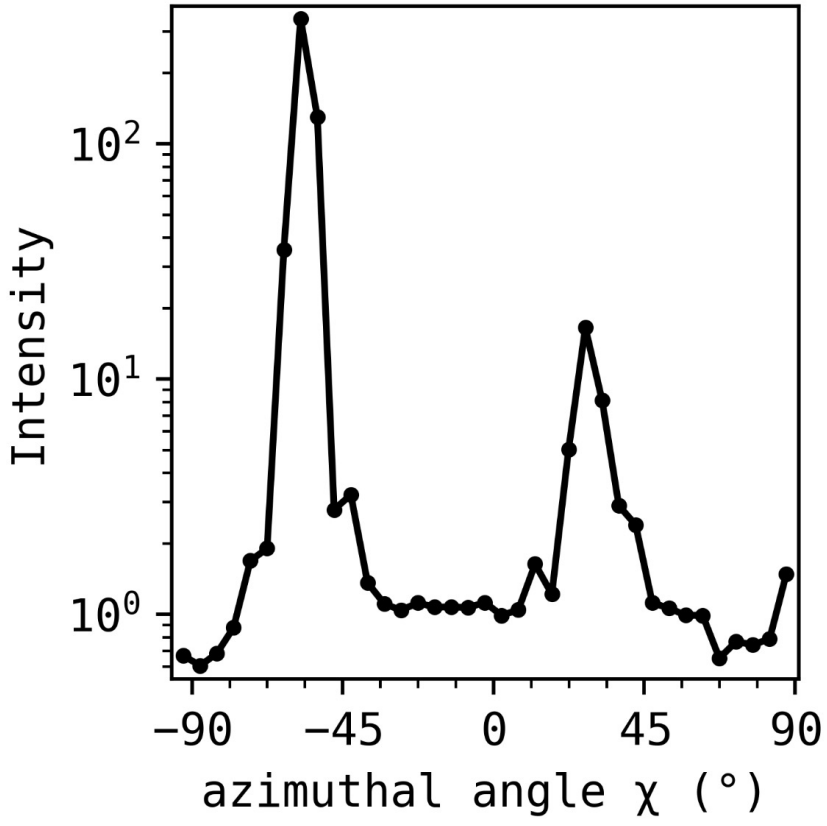
Frederik H. Gjørup

Collaboration w. Astri Haugen

3D-printing with template crystallites



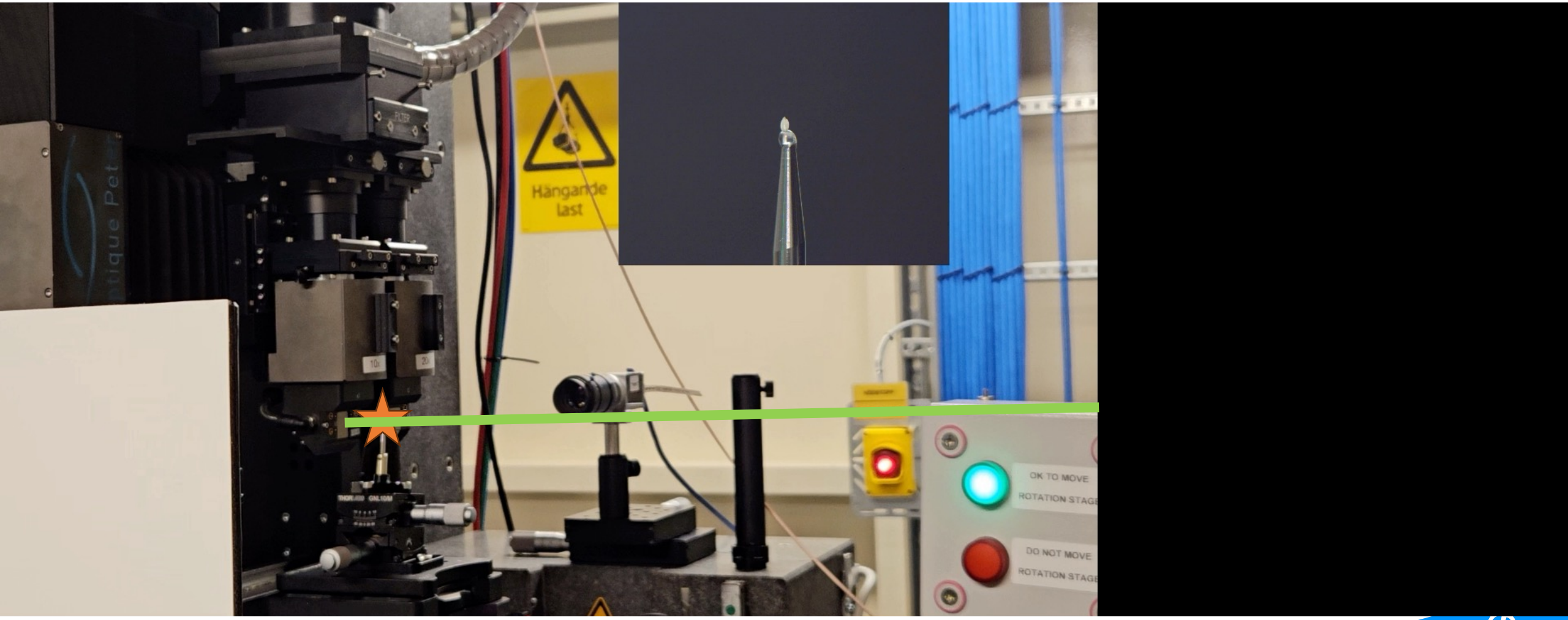




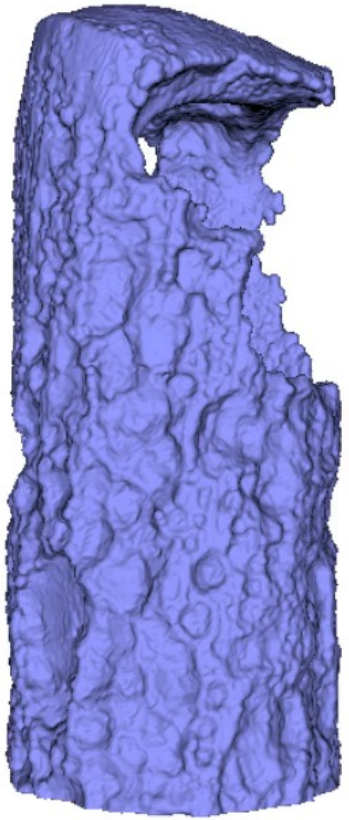
Large crystallites → Spotty diffraction data
Azimuthally binned data still show texture

Frederik H. Gjørup

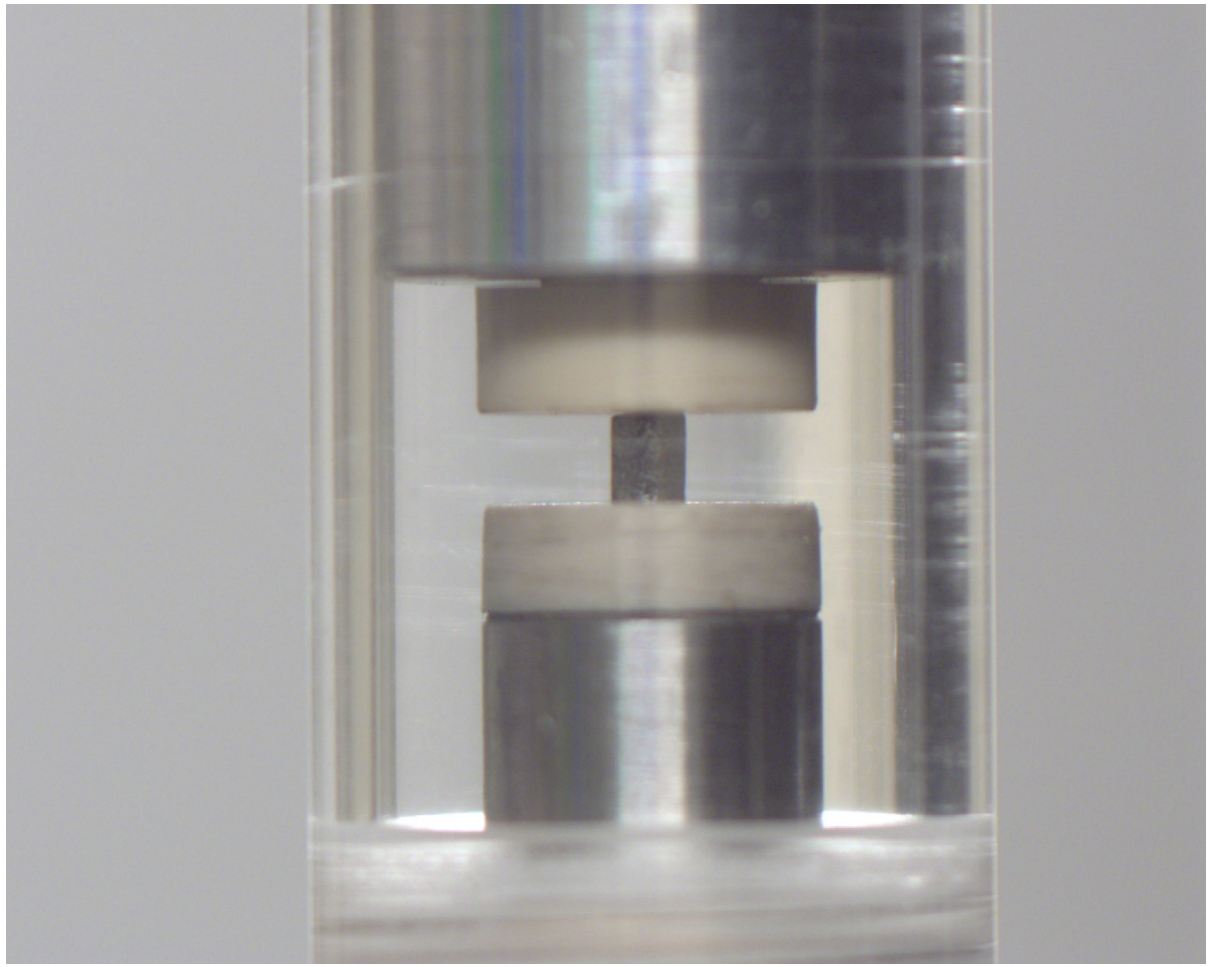
Tomographic imaging

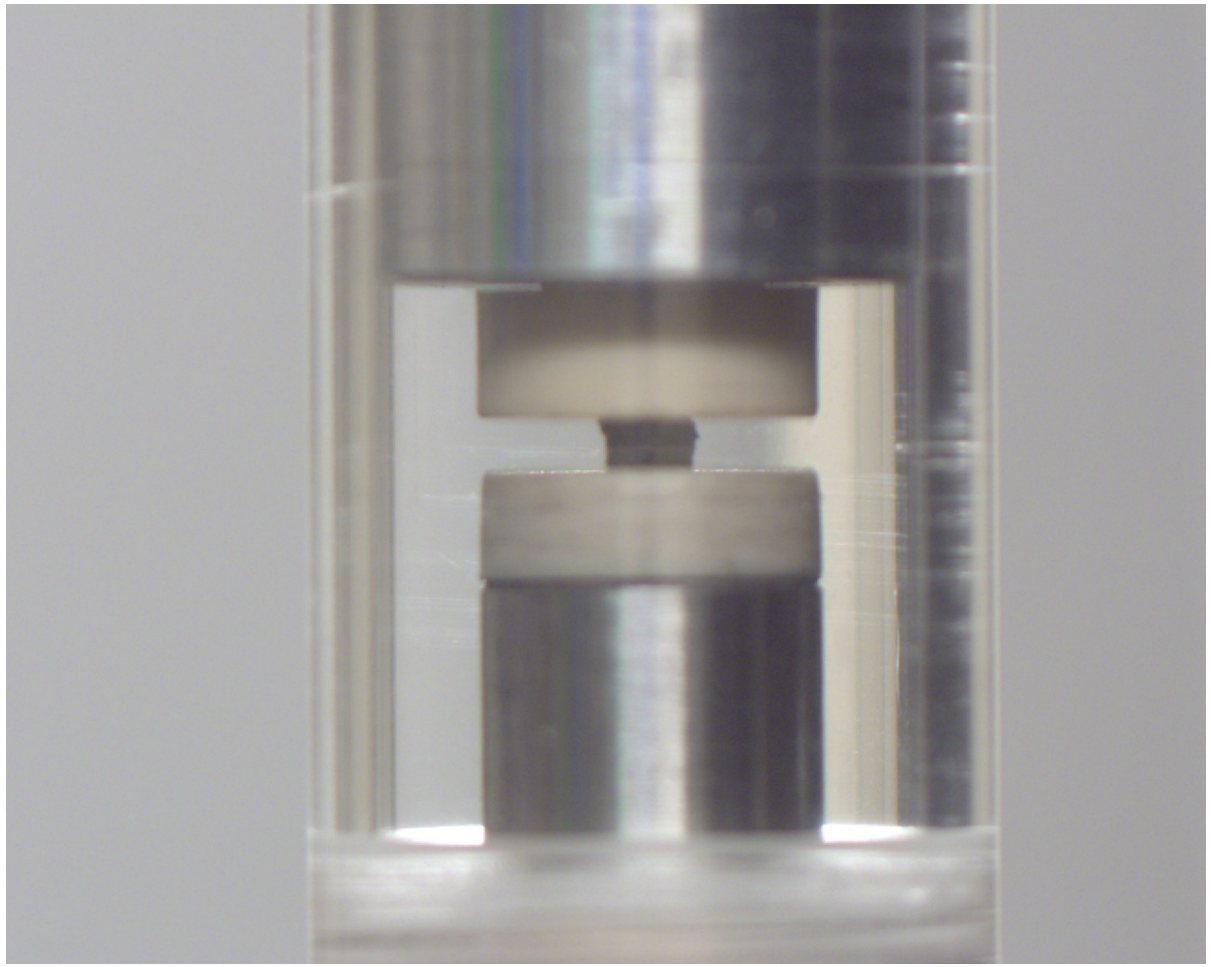


µCT of a Thermocouple



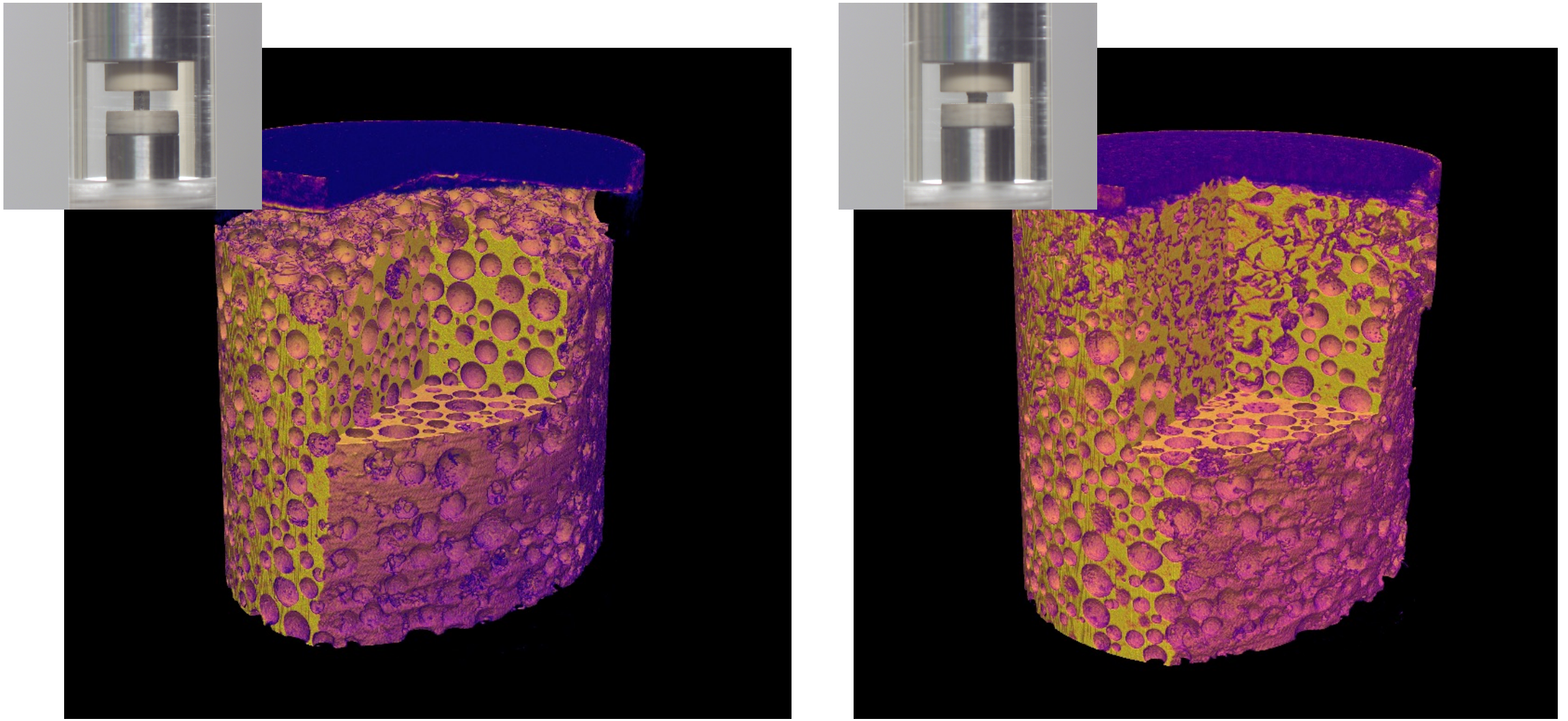
µCT of closed-cell Al-Si foam during deformation





PI: J. Hektor, Malmö U.

µCT of closed-cell Al-Si foam during deformation



PI: J. Hektor, Malmö U.

Your experiment today

Experimental flow chart

Callibration of heat gun
Already performed

→

Mounting of LaB6
At the instrument

→

LaB6 PXRD collection
Collect data – control PC

↓

Calibration of instrument geometry
Software - control PC

→

Start integration pipeline
Software - control PC

→

Mounting of brass sample
At the instrument

↓

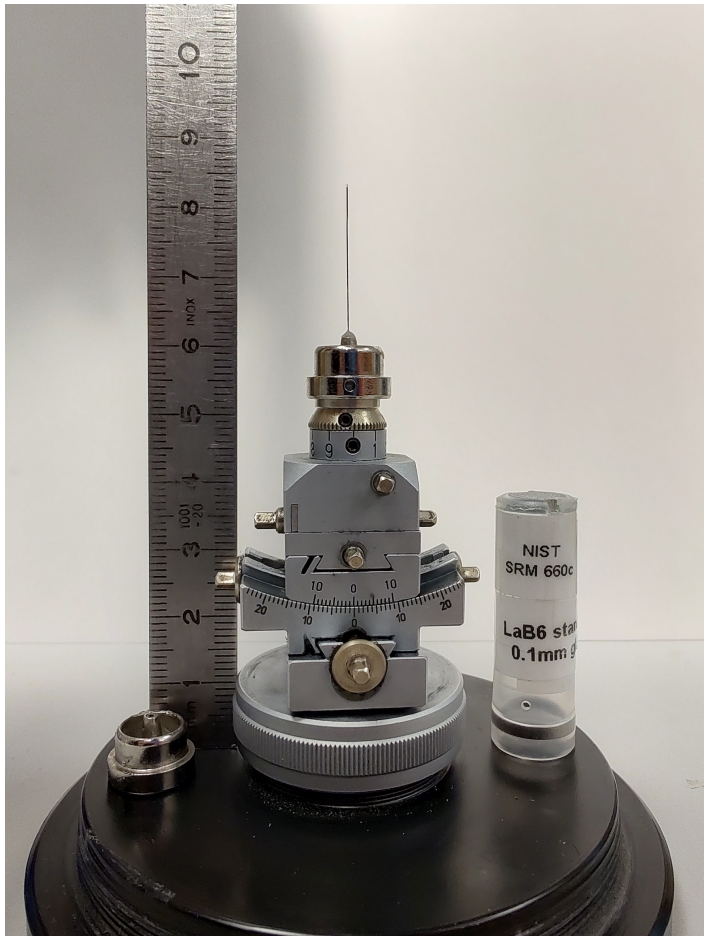
RT PXRD collection
Collect data – control PC

→

in situ PXRD collection
Collect data – control PC

→

Analysis of in situ PXRD data
Software - control PC



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Ministry of Higher Education and Science

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