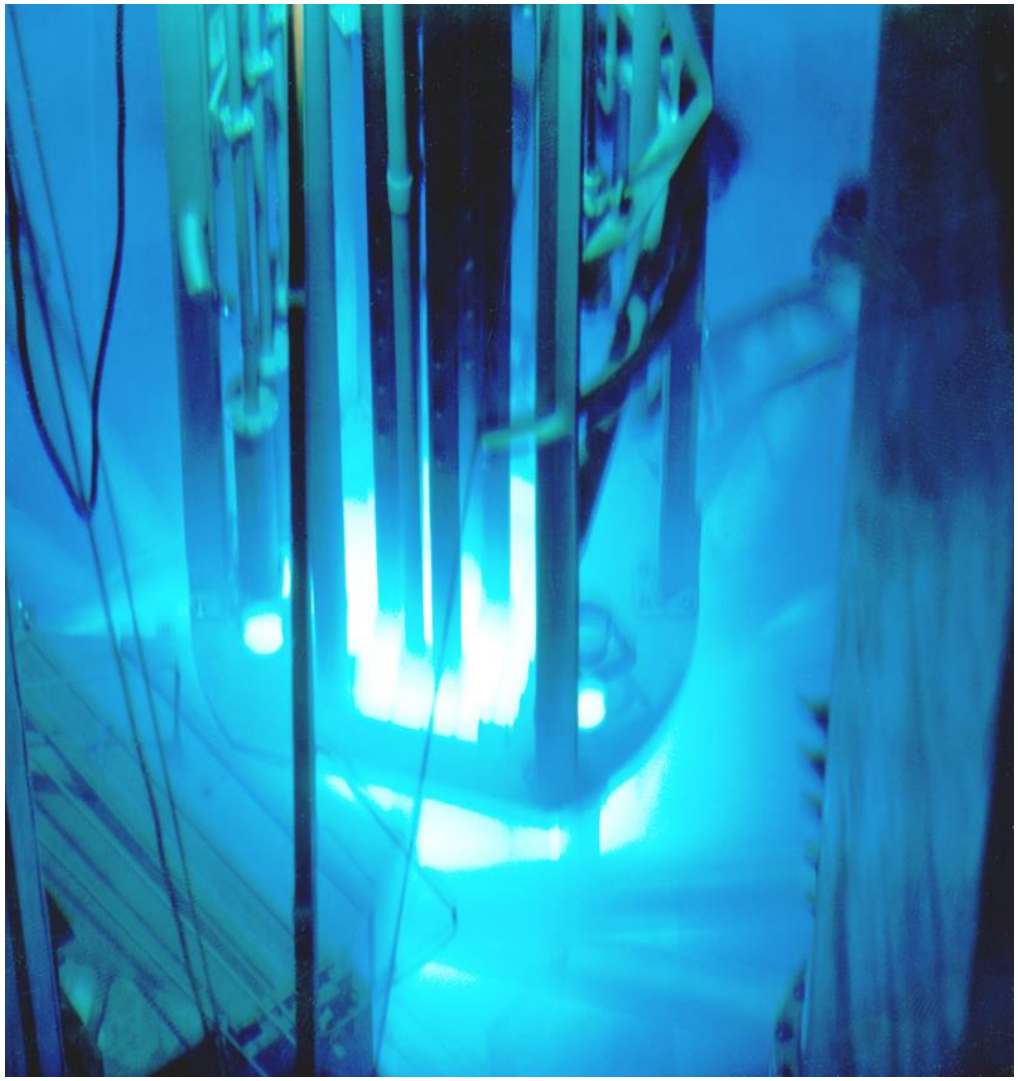
HZB Helmholtz Zentrum Berlin

SwedNESS: Real-Space Neutron Imaging

Energy-selective neutron imaging steady-state sources

Nikolay Kardjilov, Ingo Manke, André Hilger, John Banhart

BER-2 research reactor



Type: open, light-water-moderated swimming pool reactor

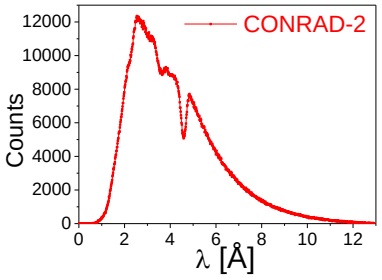
Capacity:
more than 10^{14} neutrons per square centimeter per second in the core 10 megawatts thermal power

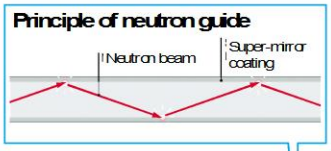
The research reactor BER II is a source of neutron beams for around 10 instruments used for structural and materials research.

CONRAD-2

Cold neutrons

Wavelength range: 1.5 Å – 10 Å



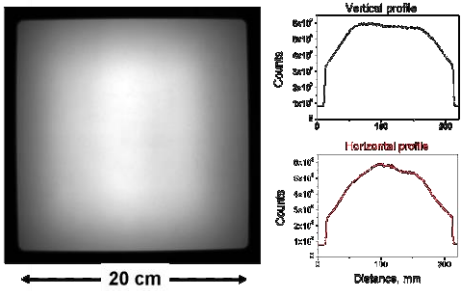


Labs

Micro-CT Lab
3D Data Analytics Lab

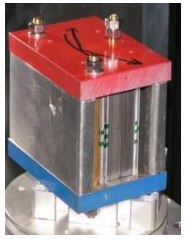
Large beam

Beam size: 20 cm x 20 cm

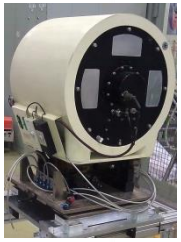


Instrumentation

Neutron polarizers

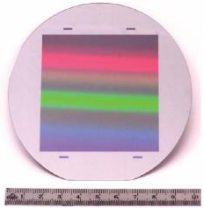


Velocity selector



Double-crystal monochromator

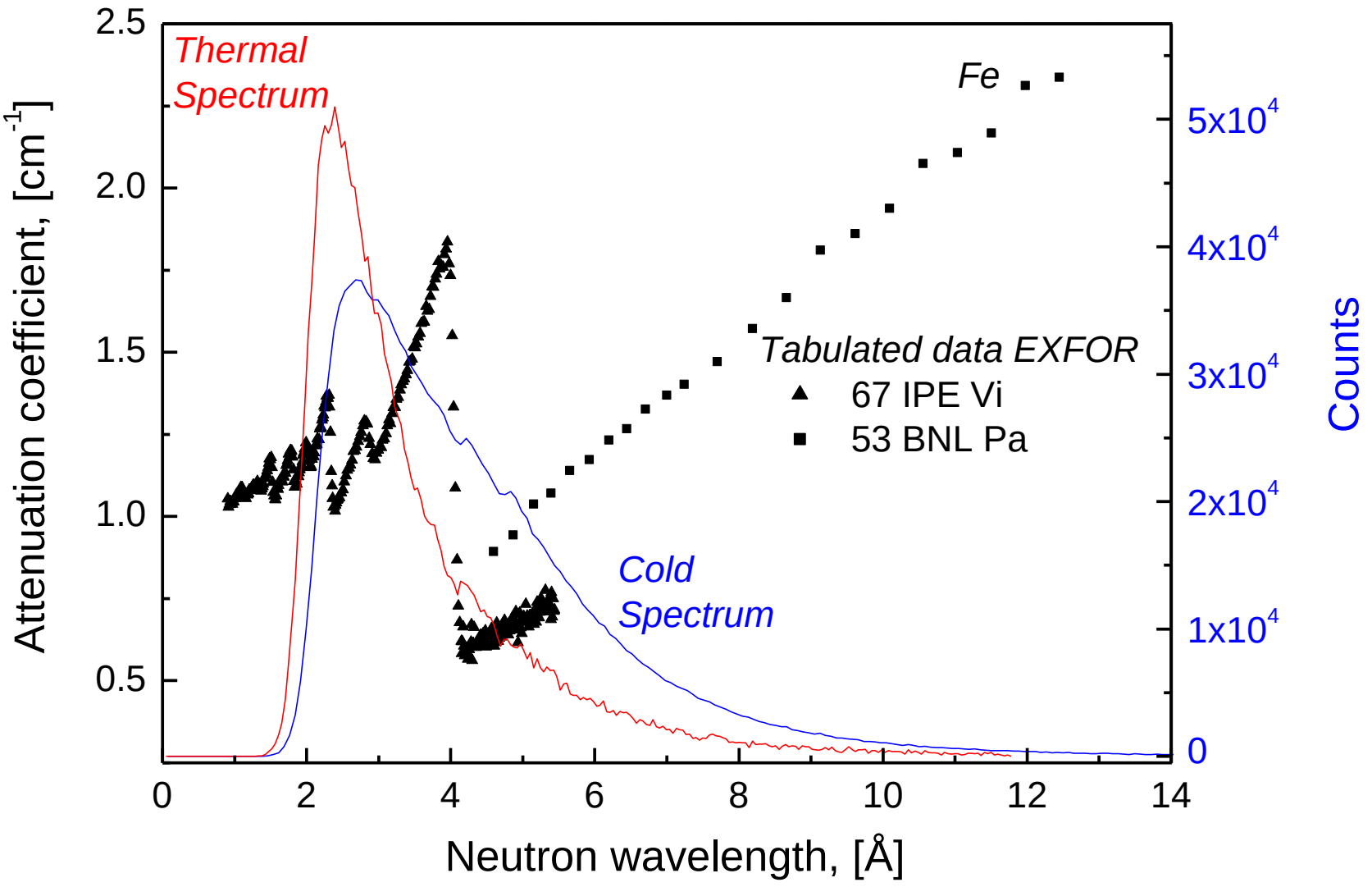
Grating interferometry



High flux

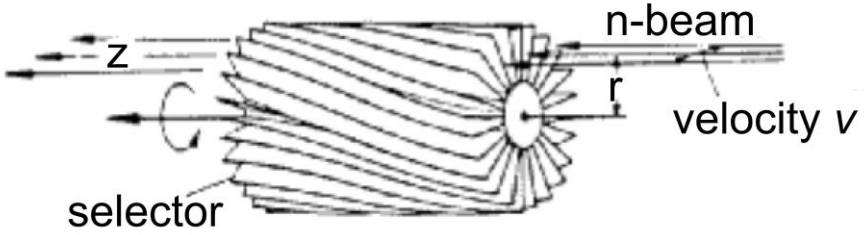
Flux (guide end): 2.7x10^9 n/cm^{2}s

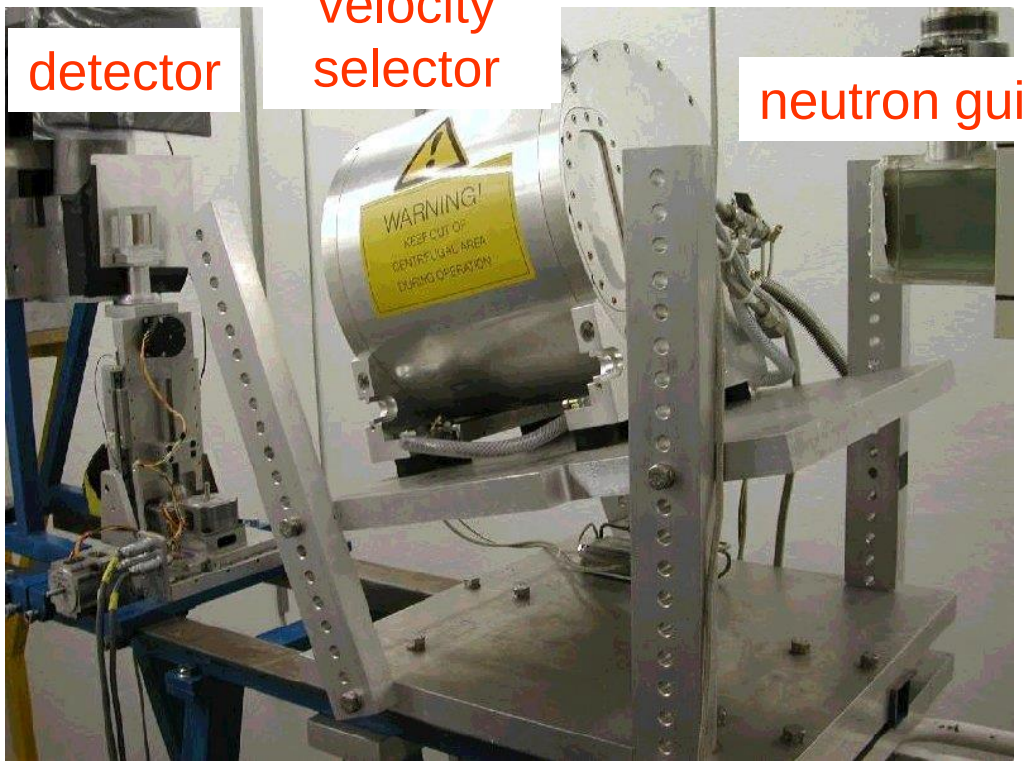
Guide system: super-mirror coated neutron guide (M=3) with a curvature of 750 m and length of 15 m followed by linear guide section (M=2) with a length of 10 m


Thermal
Spectrum
Fe
Tabulated data EXFOR
67 IPE Vi
53 BNL Pa
Cold
Spectrum
Attenuation coefficient, [cm-1]
Counts
Neutron wavelength, [Å]
2.5
2.0
1.5
1.0
0.5
5x10^4
4x10^4
3x10^4
2x10^4
1x10^4
0
0
2
4
6
8
10
12
14

Beam monochromatization

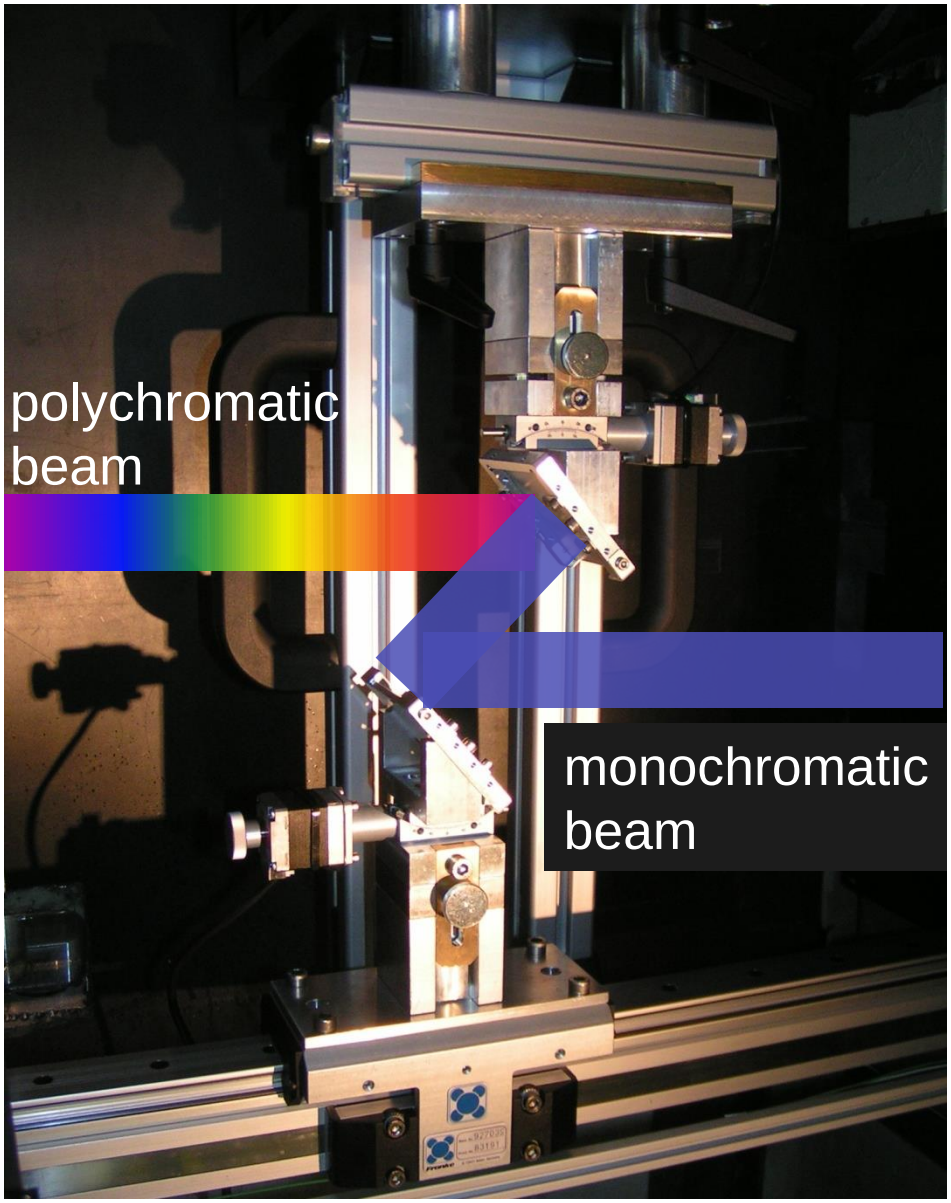
Velocity selector





Resolution: $\Delta\lambda/\lambda$ ~ 15-30 %

Beam monochromatization

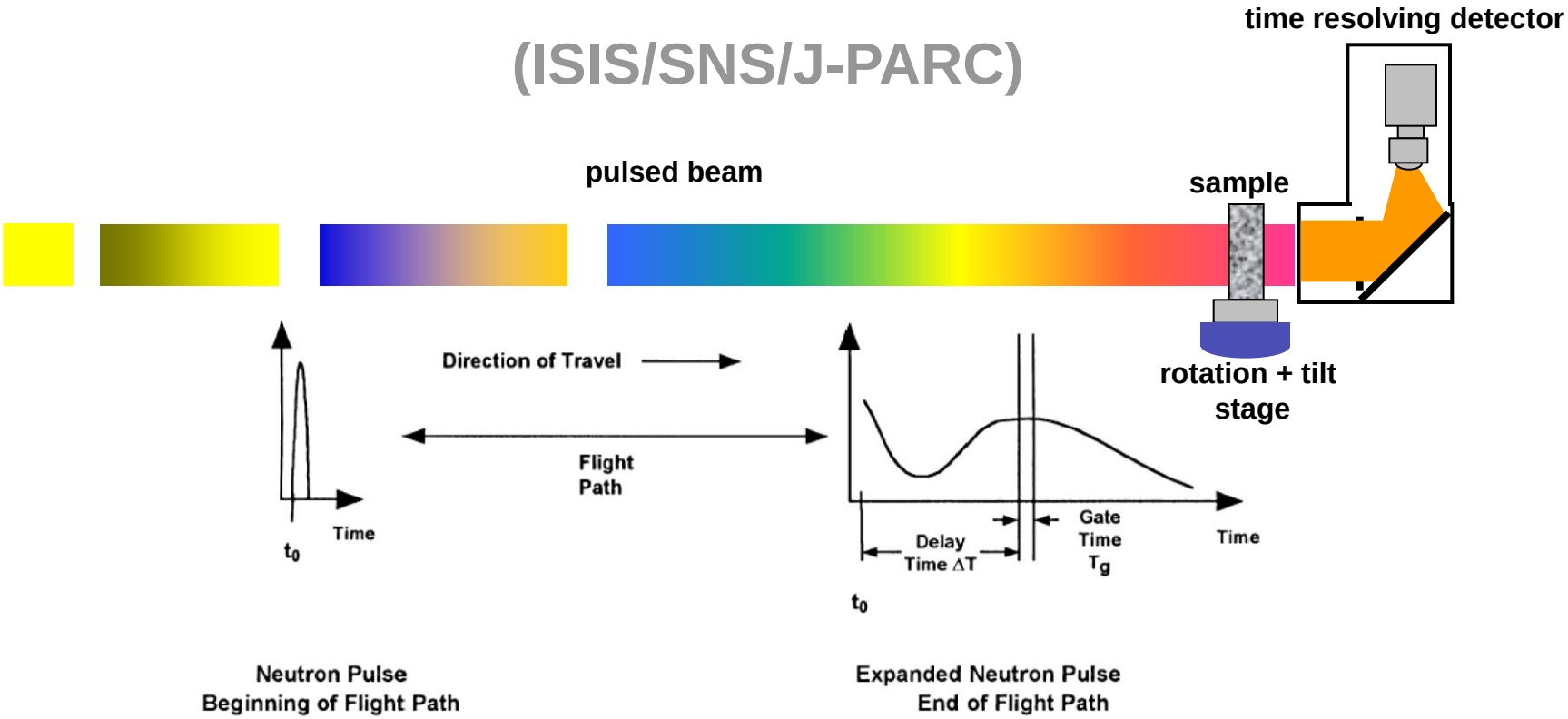


Double crystal monochromator:
PCG crystals (mosaicity of 0.8°-3.5°)

Resolution ($\Delta\lambda/\lambda$): ~ 3% - 10 %

Good resolution
Long exposure times

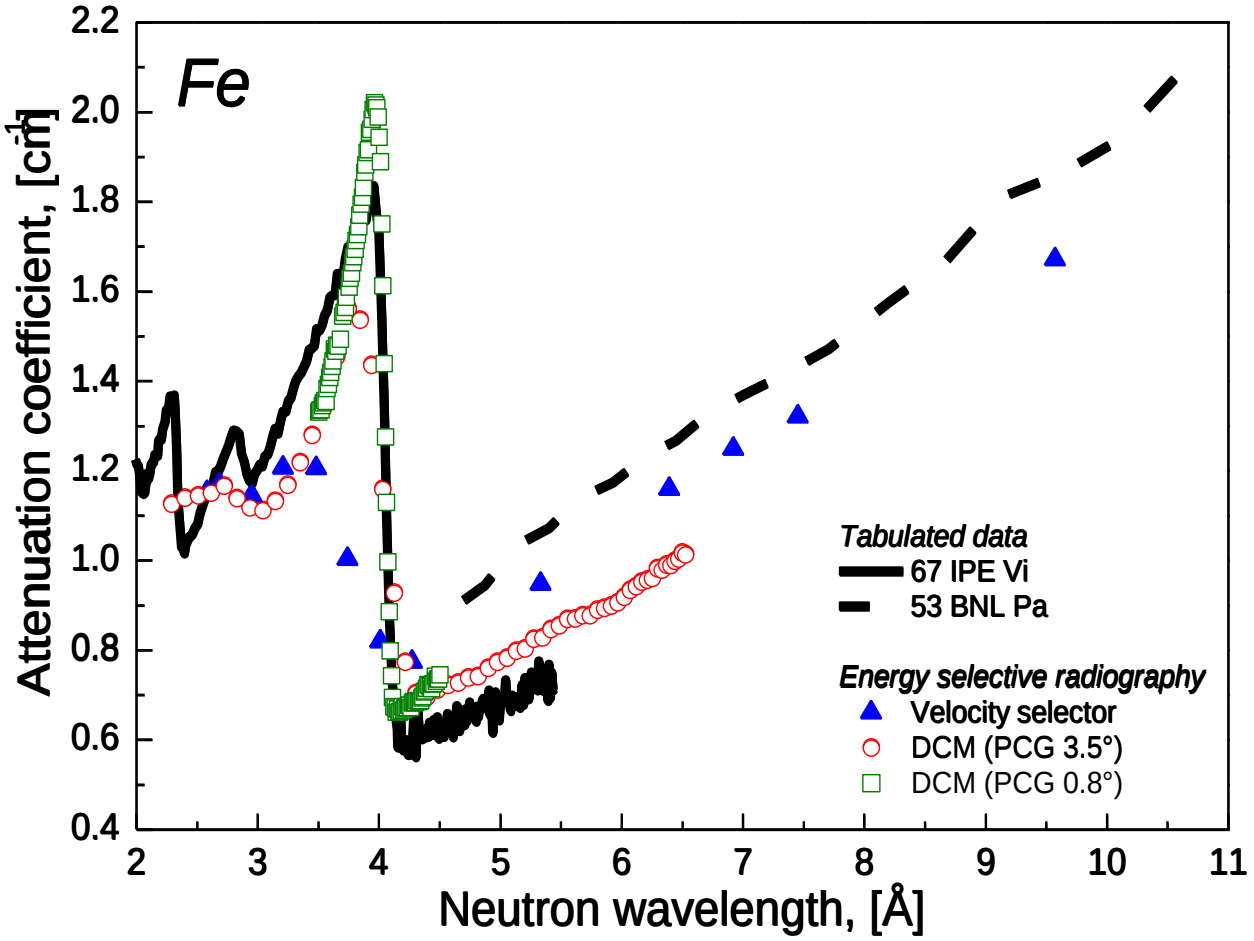
Beam monochromatization



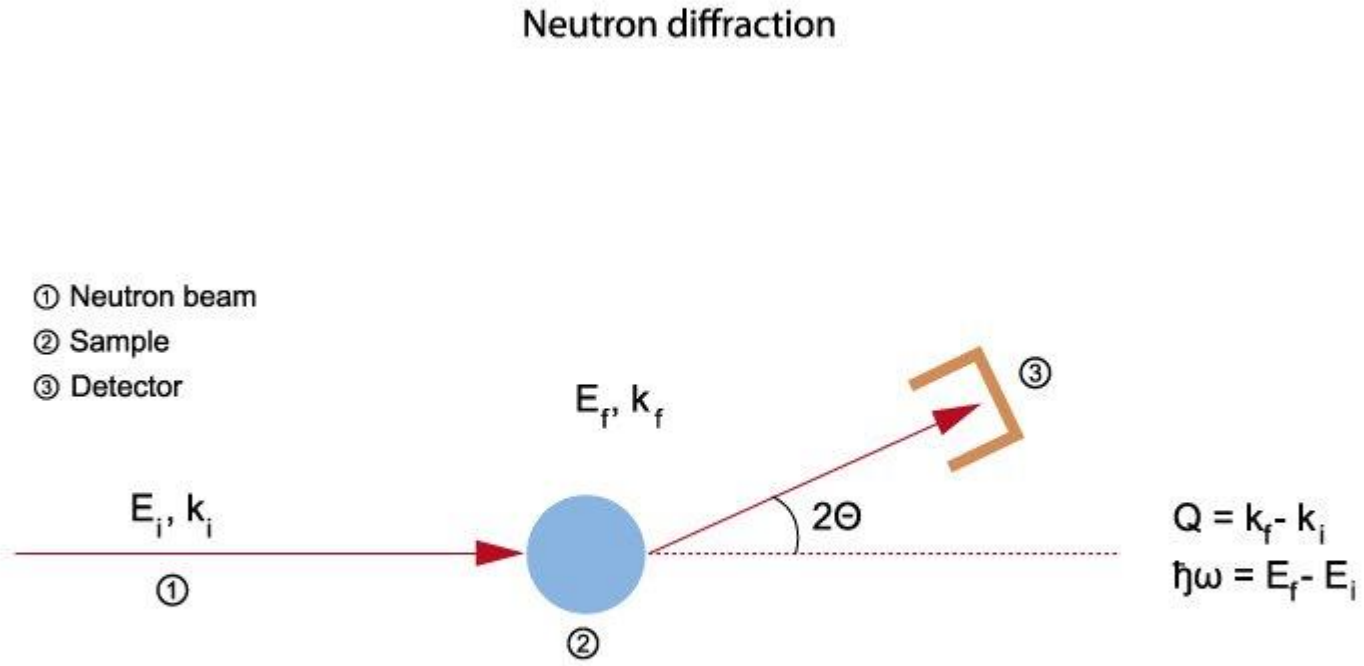
Time of flight

Resolution ($\Delta\lambda/\lambda$): ~ 0.1% - 1 %
Excelent resolution
Extreme long exposure times

Beam monochromatization

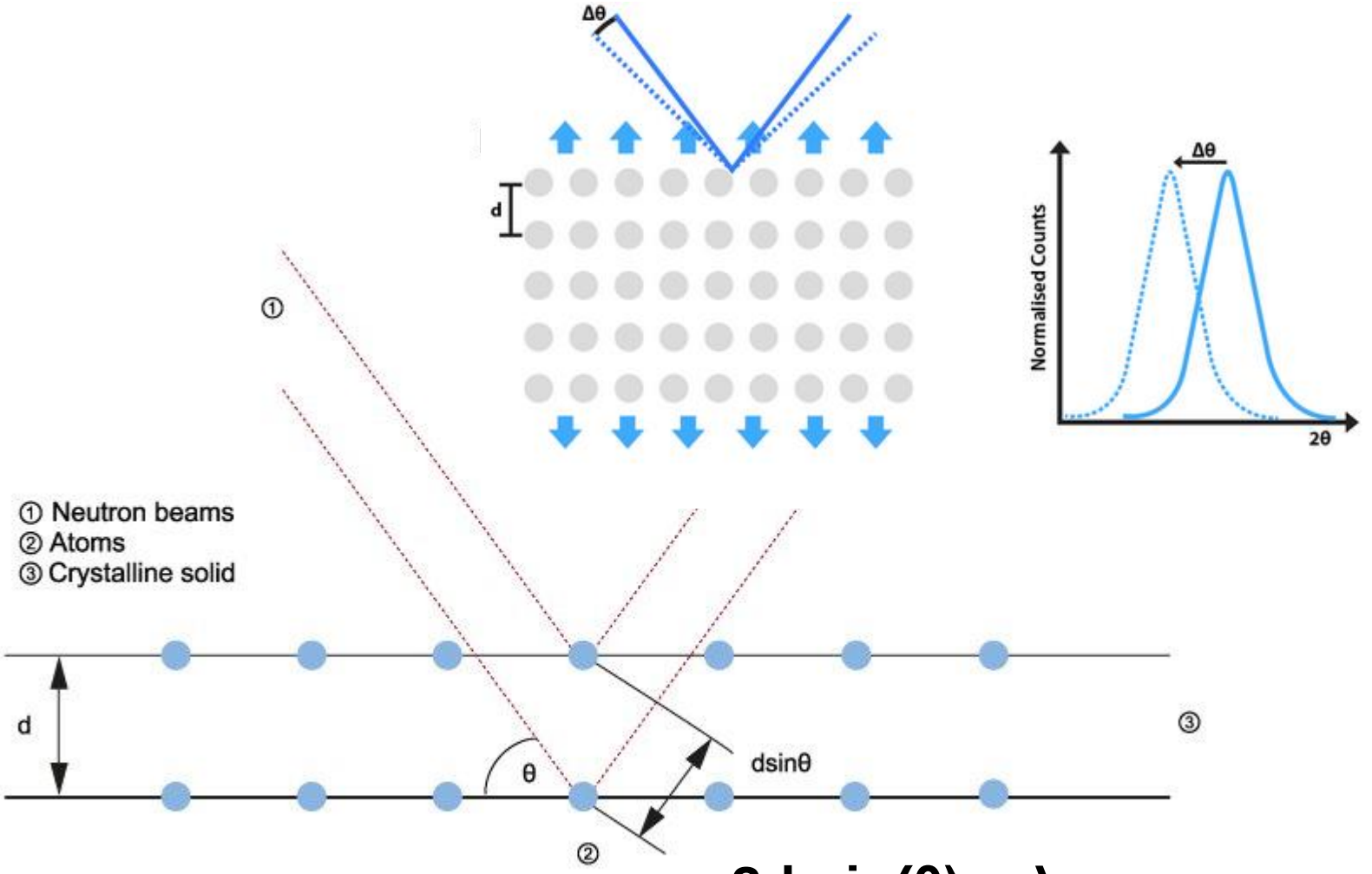


Theory



https://nmi3.eu/neutron-research/techniques-for-/structural-research.html

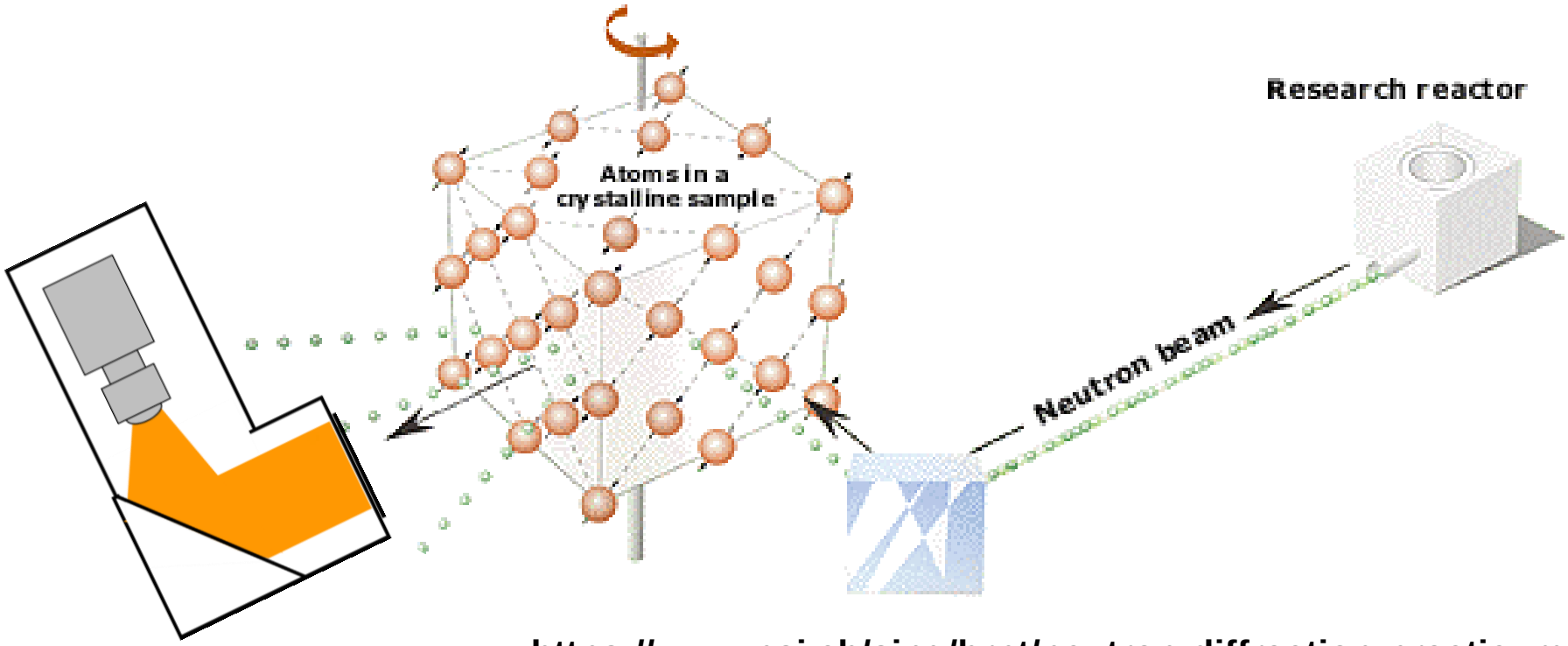
Theory



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

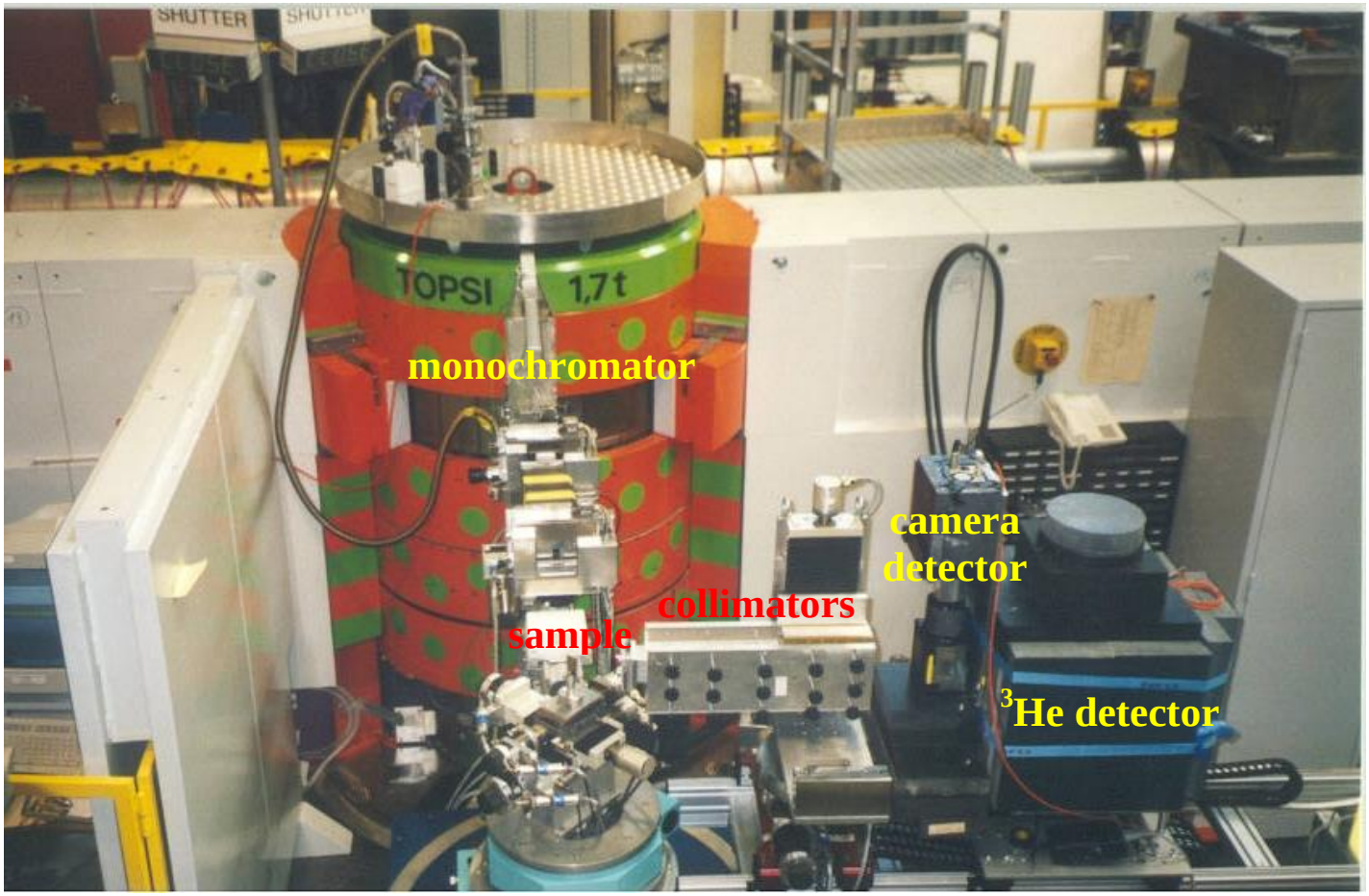
https://nmi3.eu/neutron-research/techniques-for-/structural-research.html

Neutron diffraction instrument



https://www.psi.ch/sinq/hrpt/neutron-diffraction-practicum

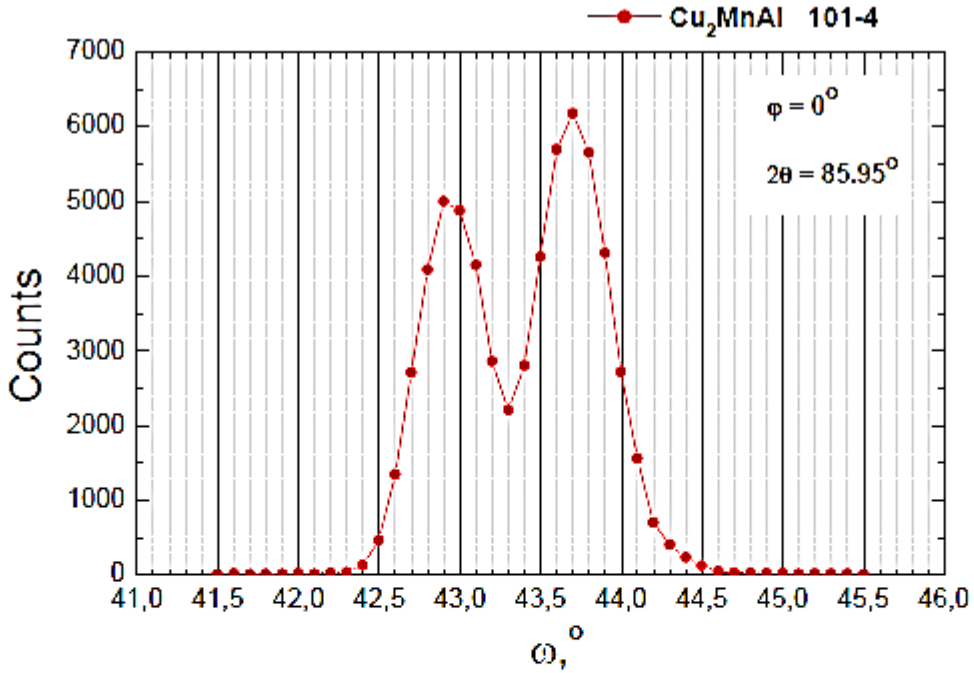
Topography

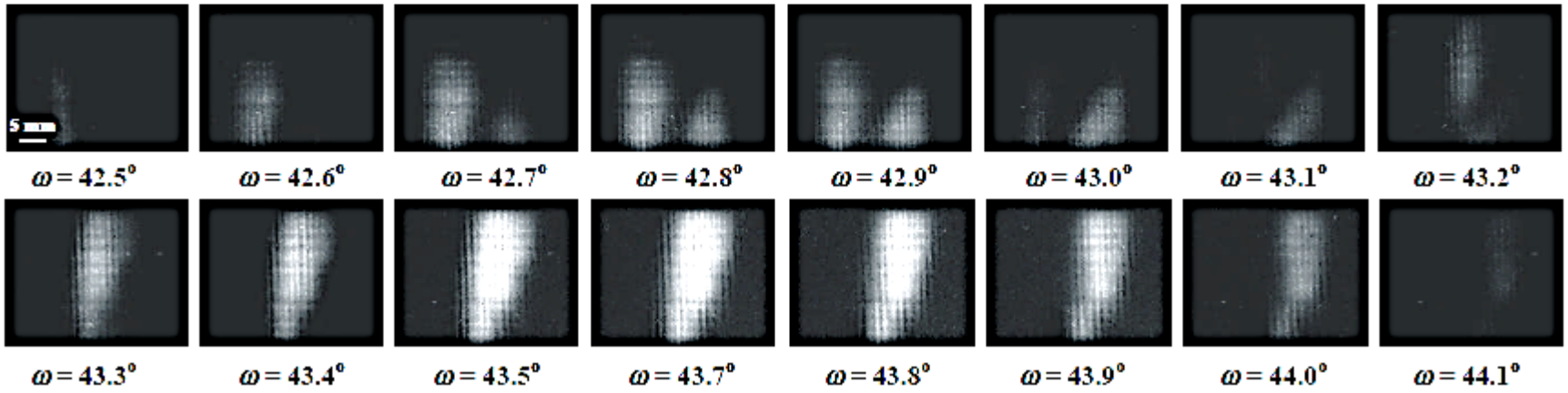


https://mediatum.ub.tum.de/doc/602975/document.pdf

Topography

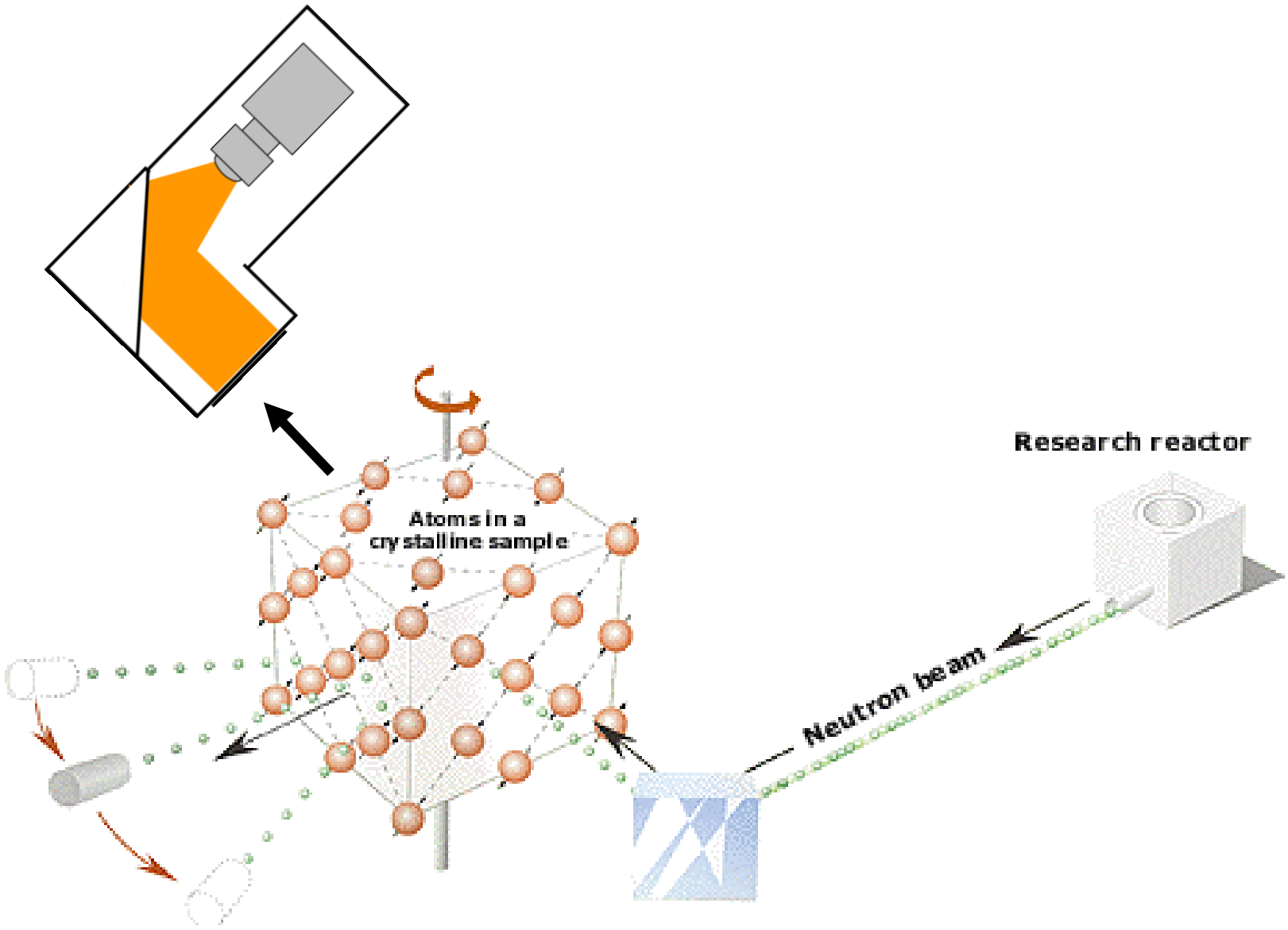
Figure 4.8 c:
TOPSI Diffractometer, PSI
$\lambda = 4.74$ Å
Sample: Cu_2MnAl Crystal with two domains
Date: 26.06.2001





https://mediatum.ub.tum.de/doc/602975/document.pdf

Diffraction based imaging



https://www.psi.ch/sinq/hrpt/neutron-diffraction-practicum

Beam monochromatisation

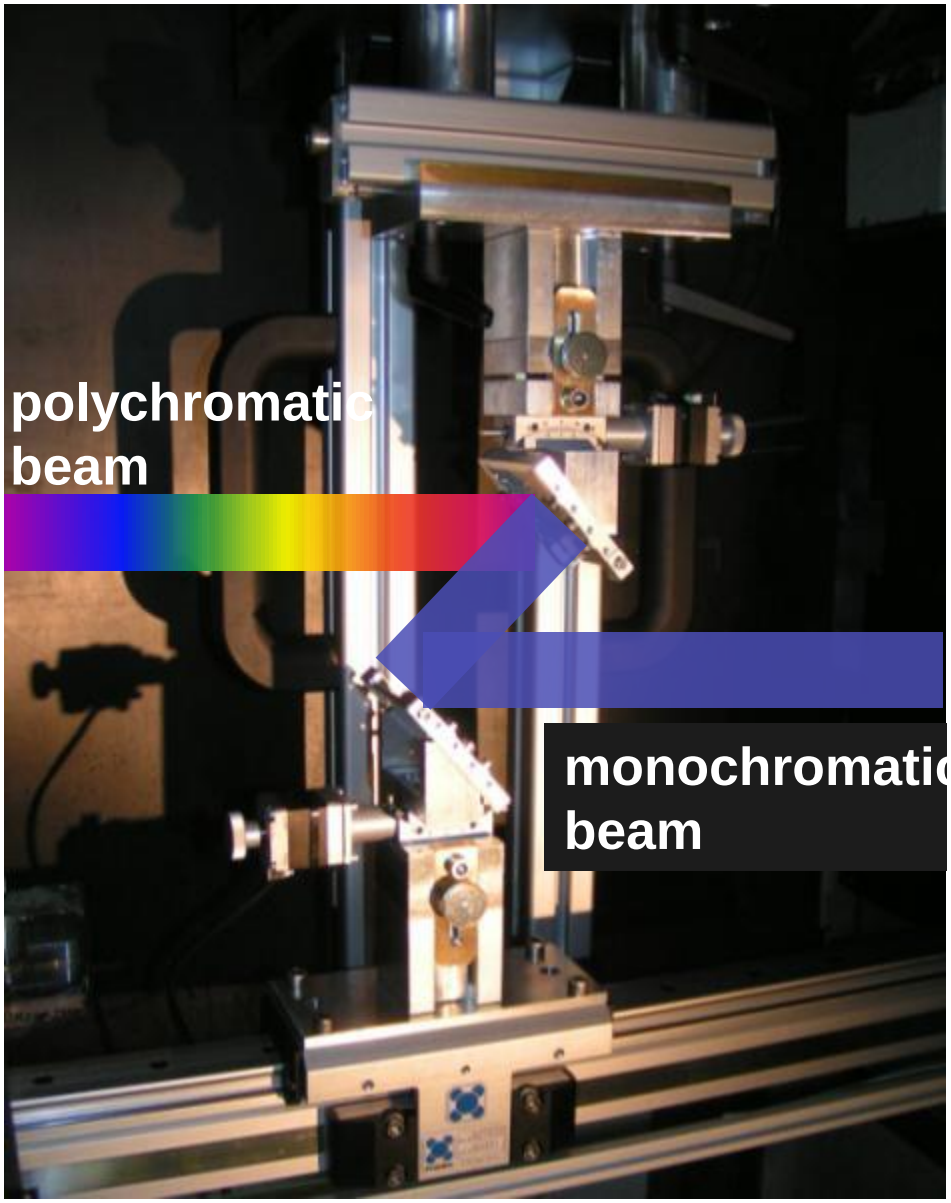
Double crystal monochromator: PCG crystals (mosaicity of 0.8°)

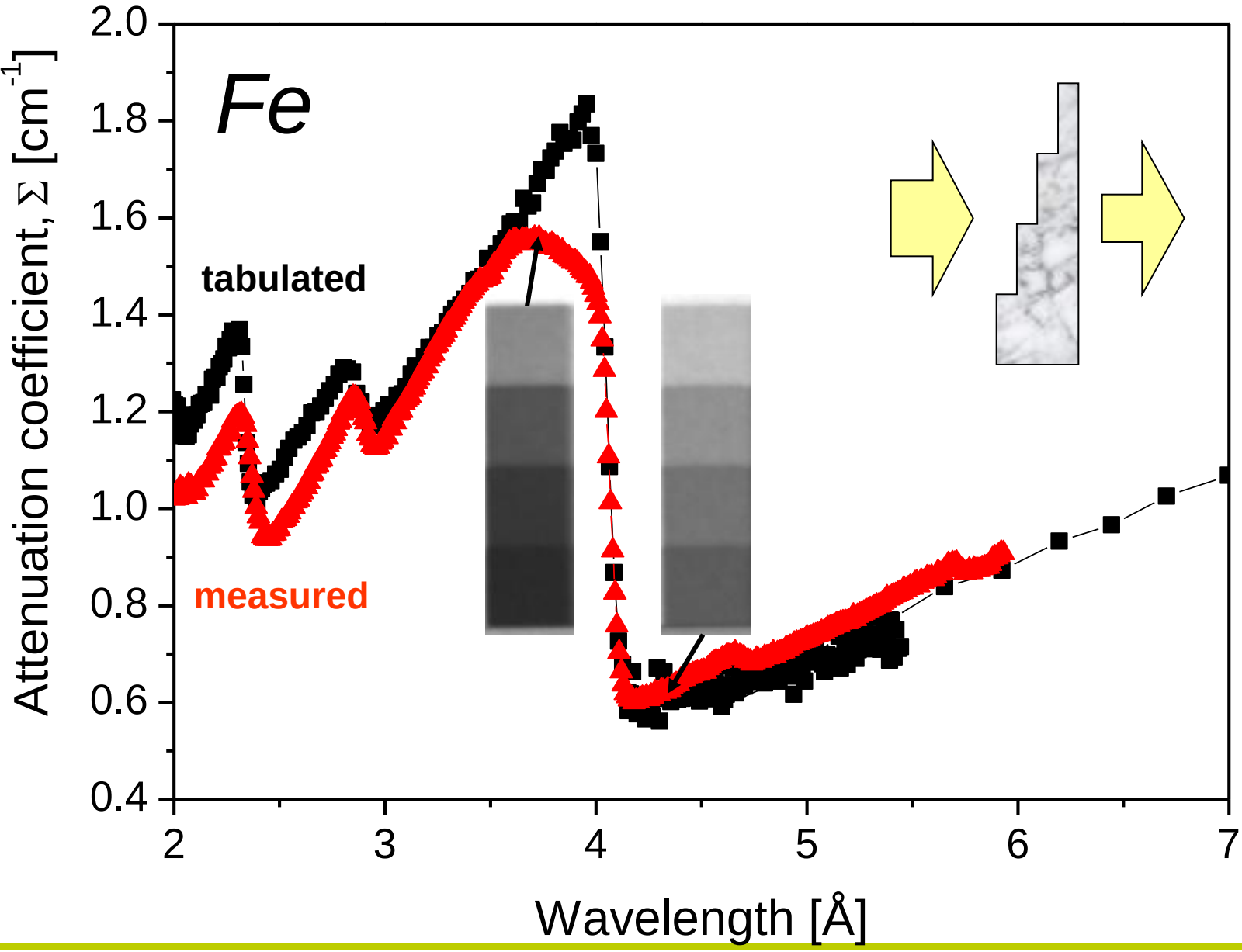
Range: 2.0 – 6.5 Å

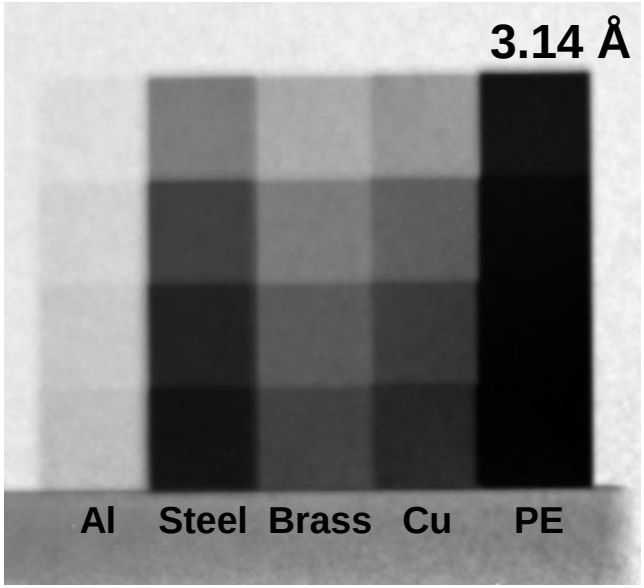
Resolution ($\Delta\lambda/\lambda$): ~ 3%

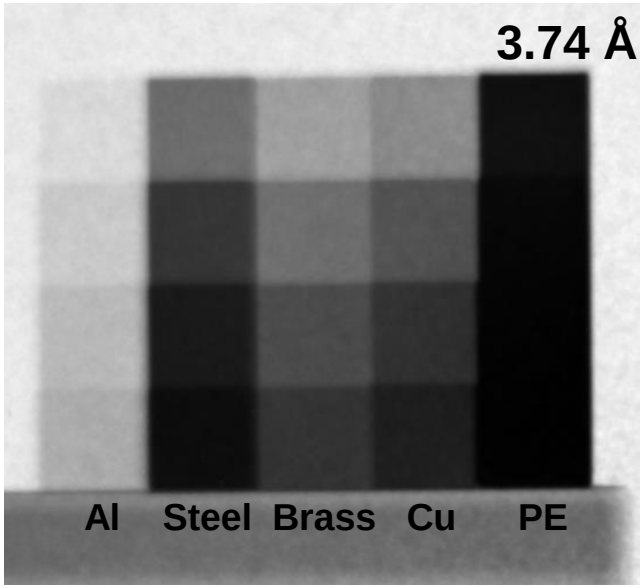
Neutron flux: ~ 4×10^5 n/cm^{2}s (at λ=3.0 Å)

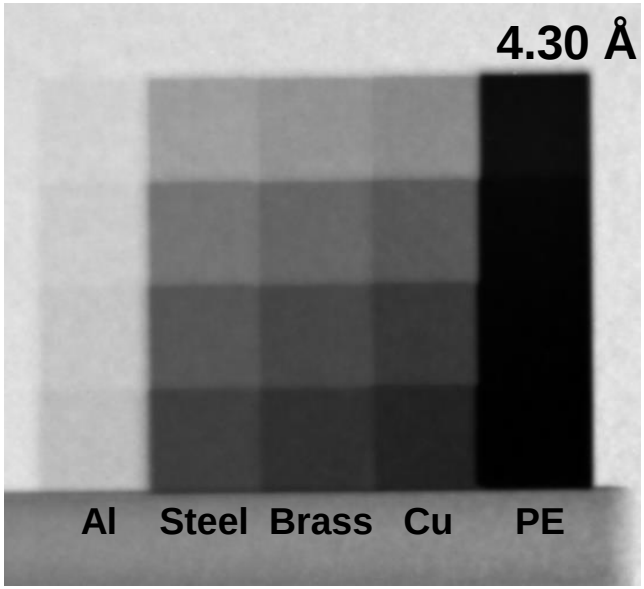
Beam size: 5 x 20 cm^2

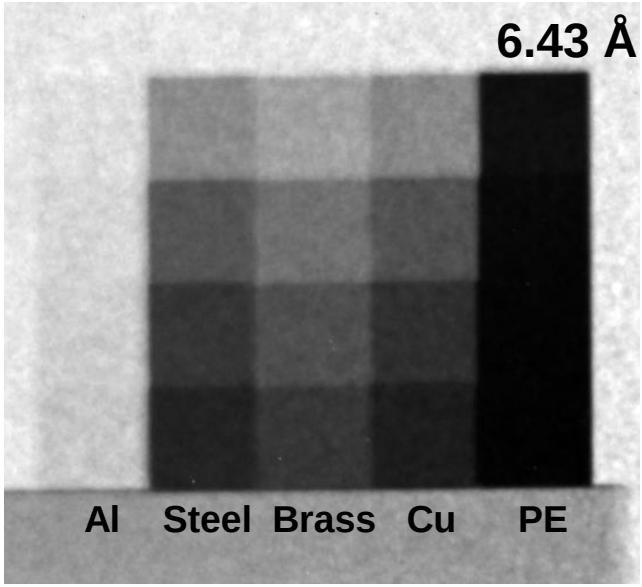


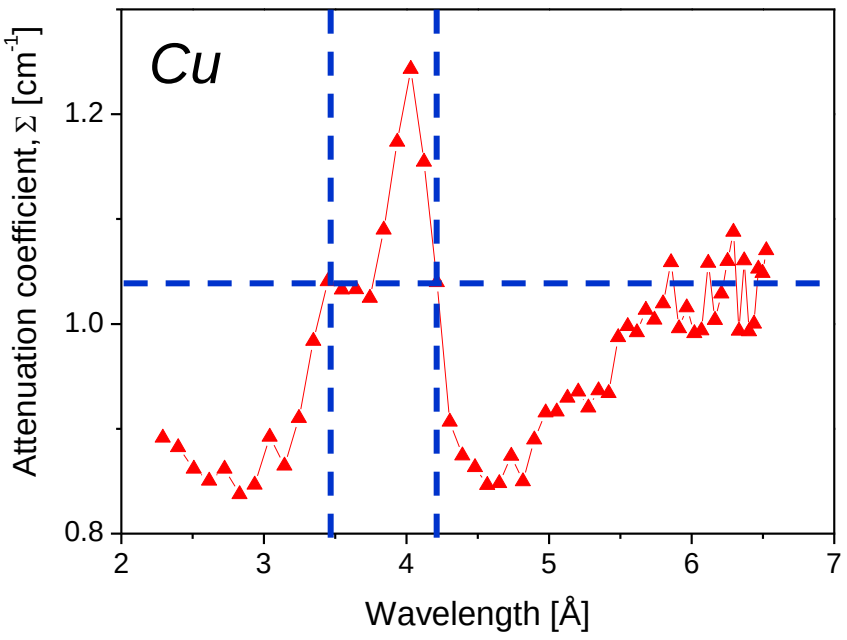

Fe
tabulated
measured
Attenuation coefficient, Σ [cm-1]
Wavelength [Å]
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
2
3
4
5
6
7

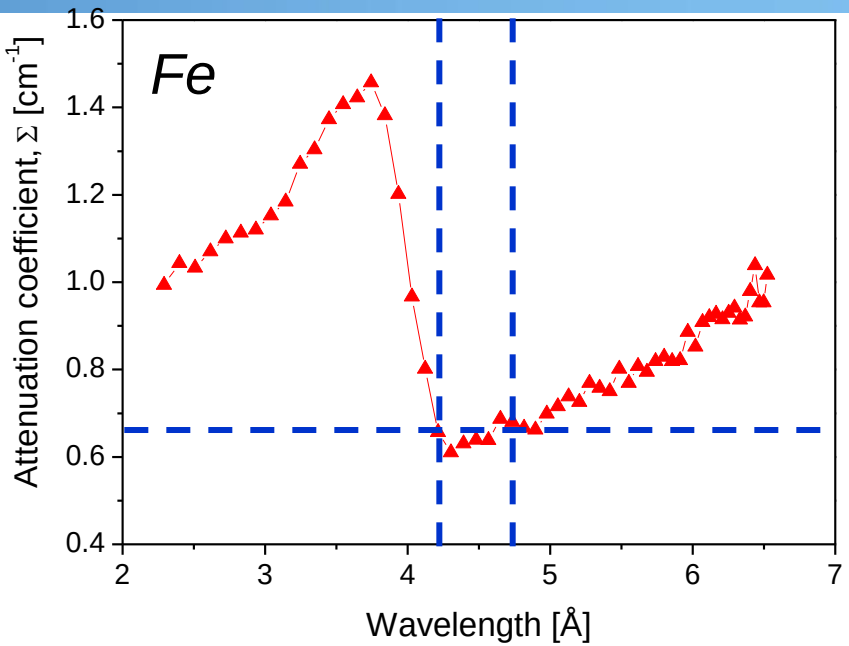

3.14 Å
Al
Steel
Brass
Cu
PE

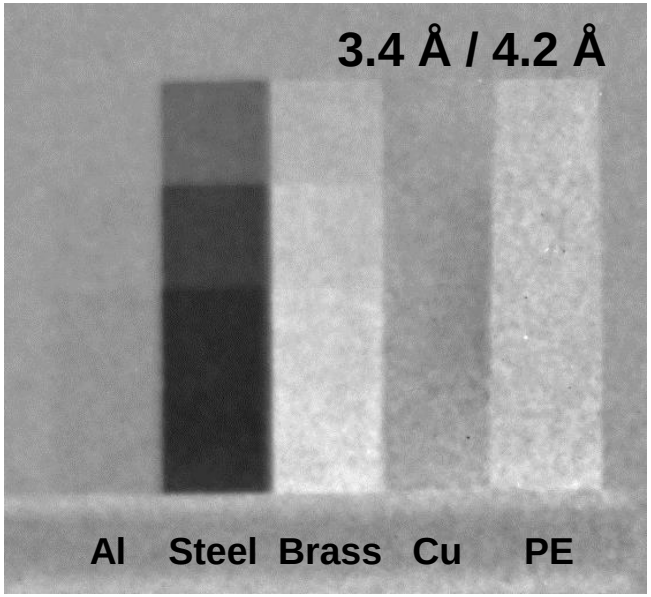

3.74 Å
Al
Steel
Brass
Cu
PE

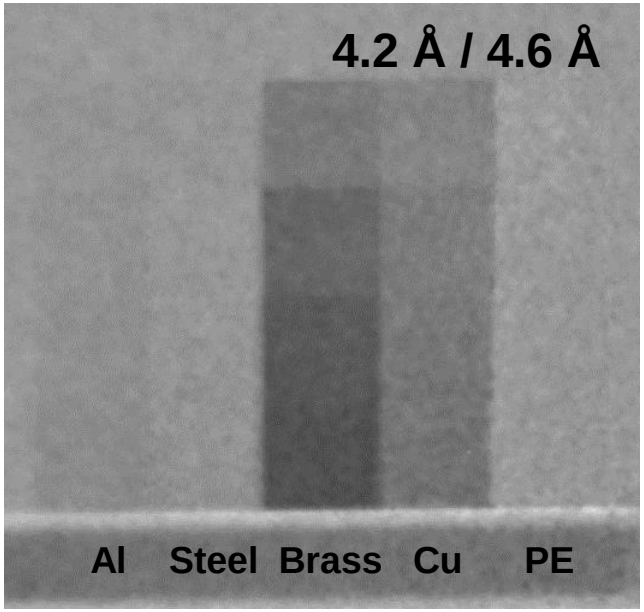

4.30 Å
Al
Steel
Brass
Cu
PE


6.43 Å
Al
Steel
Brass
Cu
PE

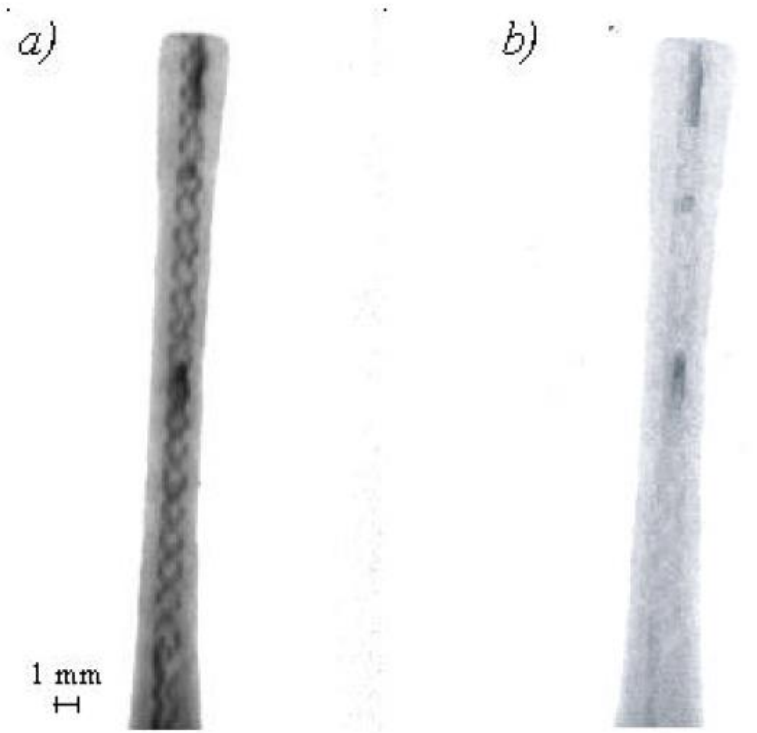

Cu
Attenuation coefficient, Σ [cm-1]
Wavelength [Å]
0.8
1.0
1.2
2
3
4
5
6
7

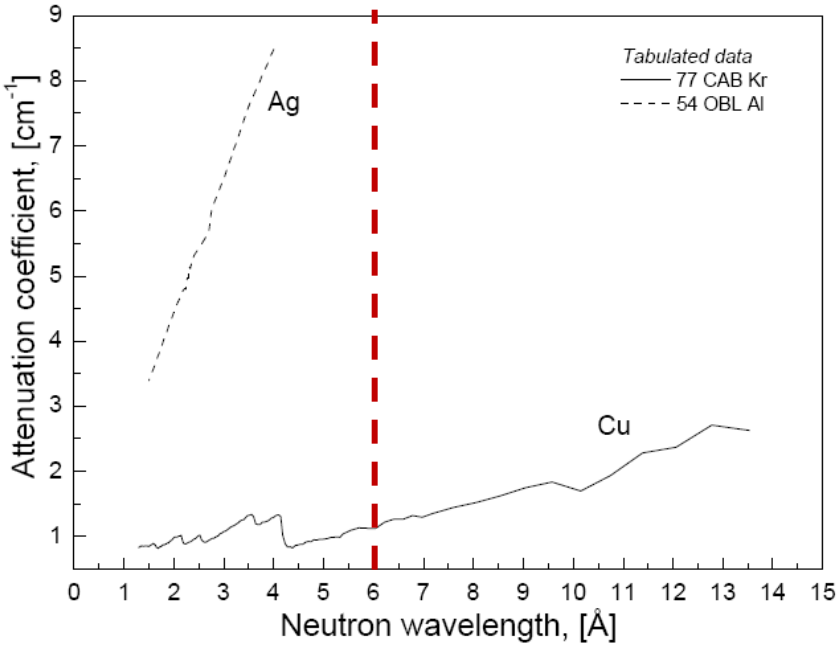

Fe
Attenuation coefficient, Σ [cm-1]
Wavelength [Å]
0.4
0.6
0.8
1.0
1.2
1.4
1.6
2
3
4
5
6
7


3.4 Å / 4.2 Å
Al
Steel
Brass
Cu
PE


4.2 Å / 4.6 Å
Al
Steel
Brass
Cu
PE

Contrast enhancement (Velocity selector)



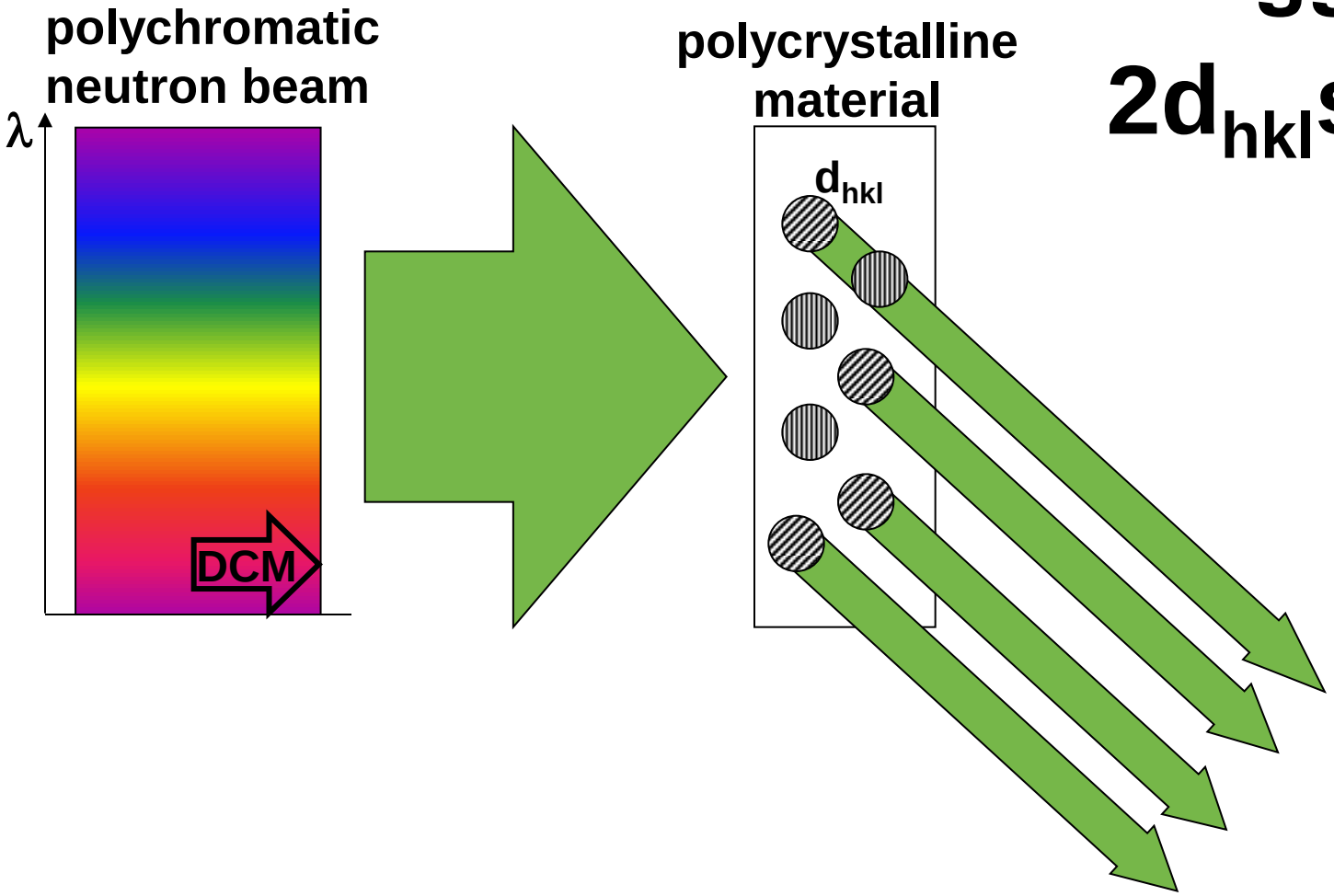


Radiographs of an ancient roman brooch taken with a) energy selective neutron radiography – 6 Å and b) standard thermal neutron radiography technique – NEUTRA (Courtesy of the Museum Aventicum, Switzerland).

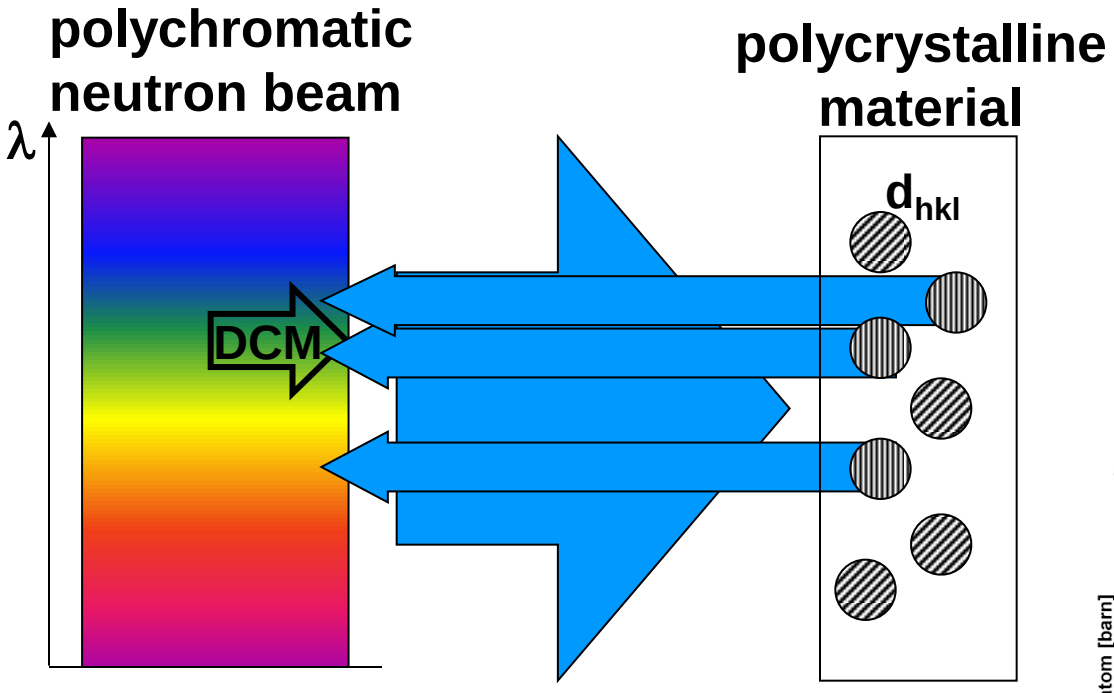
Coherent scattering – Bragg edges

Bragg's law

$$2d_{hkl}\sin\theta=\lambda$$



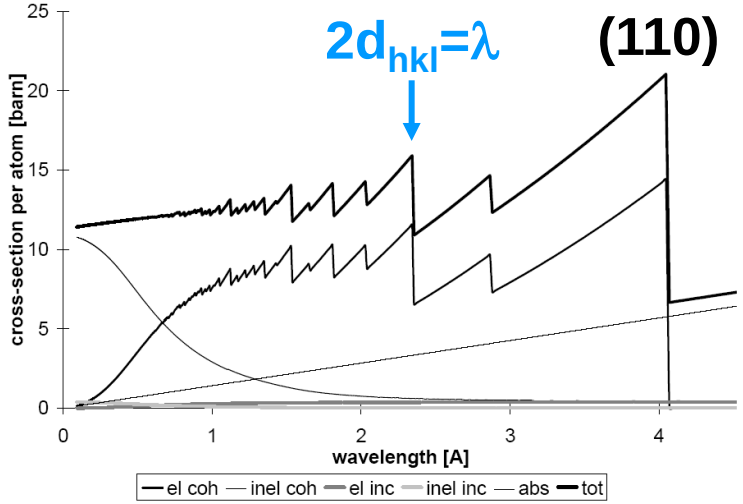
Coherent scattering – Bragg edges



Bragg's law

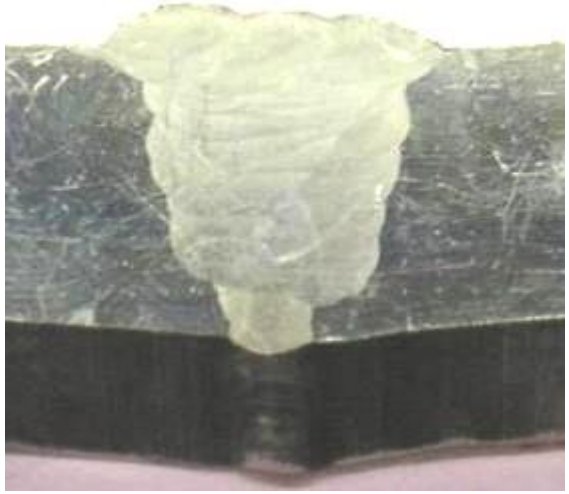
$$2d_{hkl}\sin 90° = \lambda$$

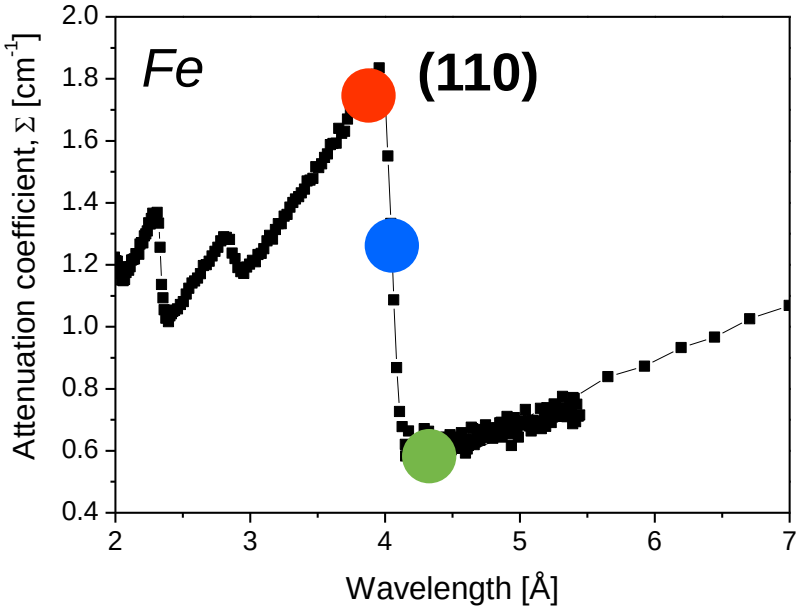
Cross-sections of iron per ator



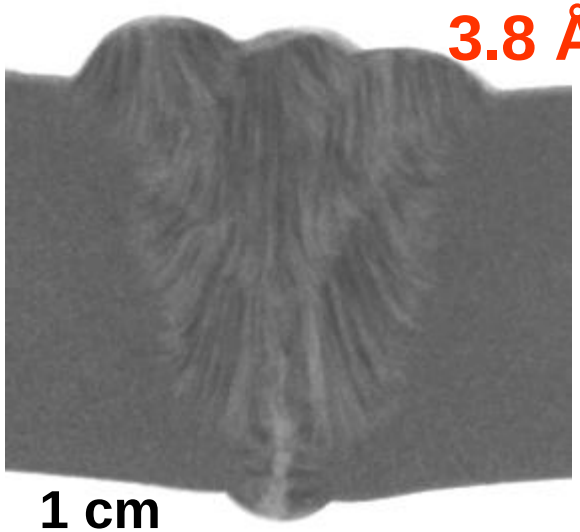
Energy-selective radiography

Welded joint (photo)

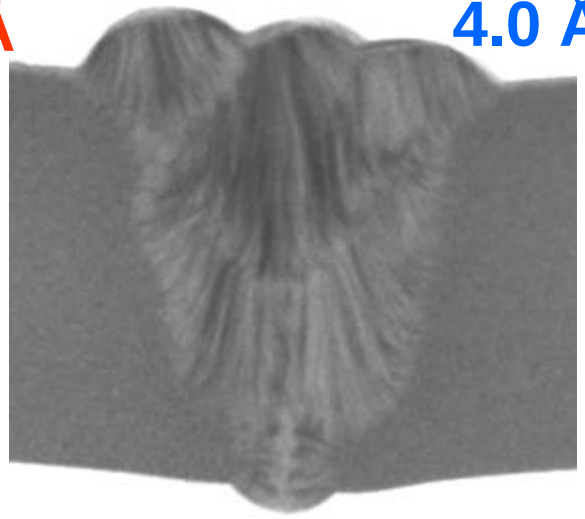




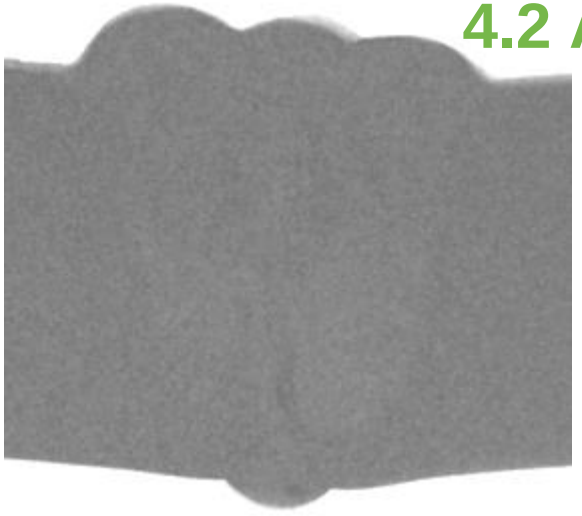
3.8 Å



4.0 Å

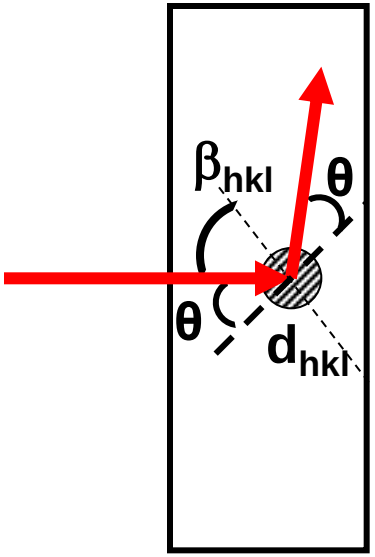


4.2 Å

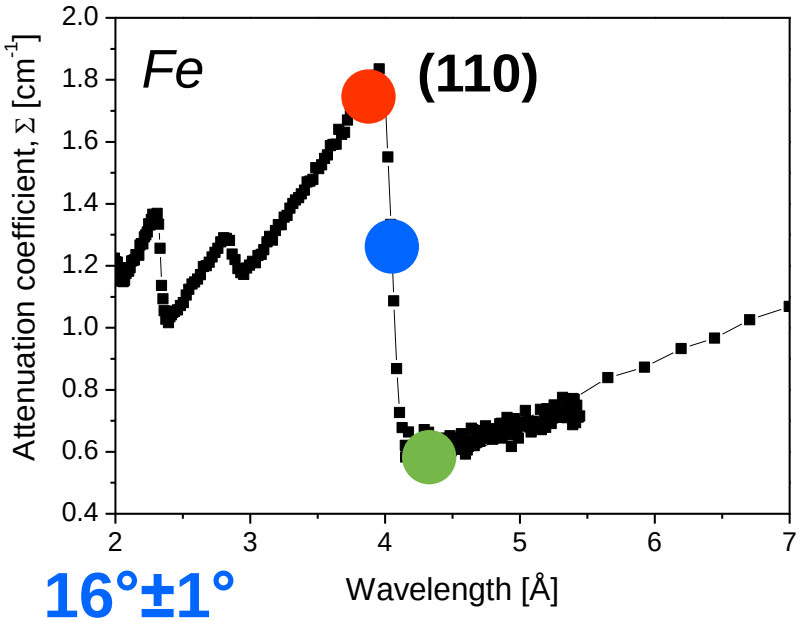


1 cm

Energy-selective radiography



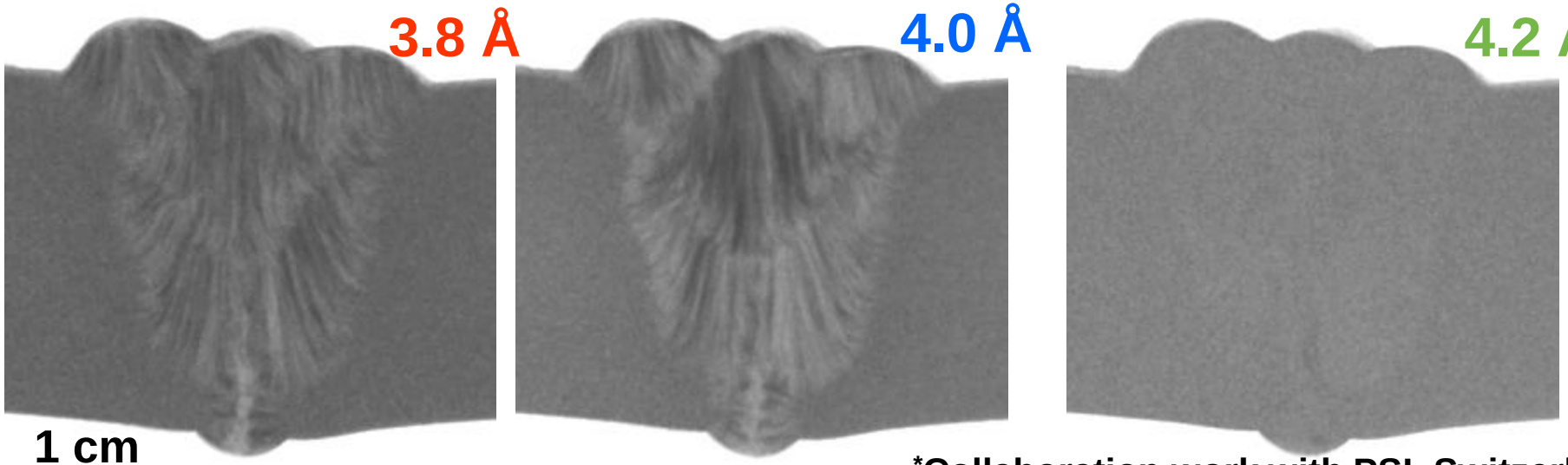
$$\beta_{hkl} = \frac{\pi}{2} - \arcsin\left(\frac{\lambda}{2d_{hkl}}\right)$$



24°±1°
3.8 Å

16°±1°
4.0 Å

4.2 Å

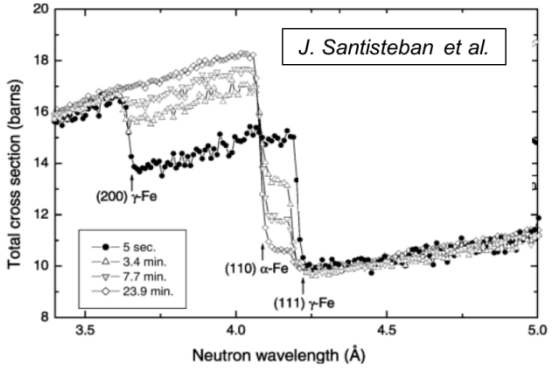


*Collaboration work with PSI, Switzerlan

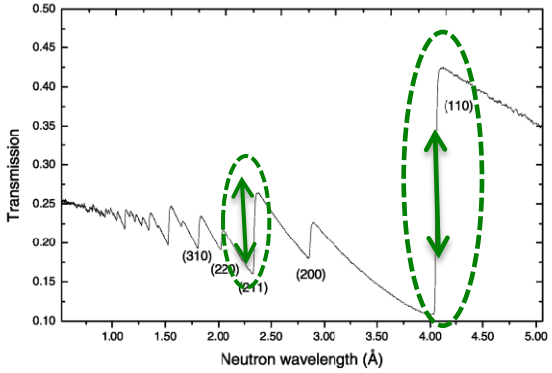
Neutron "Bragg Edge" Imaging: Applications

- **Record transmitted neutron beam energy resolved (TOF, tunable monochromator)**
- ***hkl* lattice spacing is probed in the direction of incoming beam and is "averaged" through thickness**

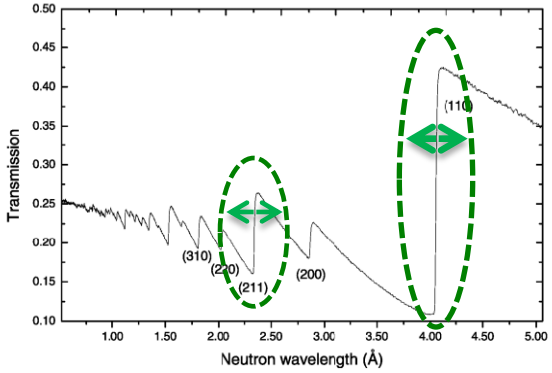
- **Phase**



- **Texture**



- **Strain**



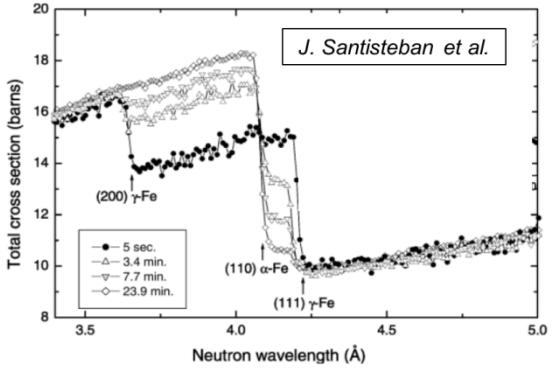
Selected References:

- **S. Vogel, A Rietveld-Approach for the Analysis of Neutron Time-of-Flight Transmission Data, Uni Kiel, Kiel, (2000)**
- **A. Steuwer, P.J. Withers, J.R. Santisteban, L. Edwards, Using pulsed neutron transmission for crystalline phase imaging and analysis, Journal of Applied Physics, 97 (2005)**
- ***A. Steuwer, J.R. Santisteban, P.J. Withers, L. Edwards, Pattern decomposition and quantitative-phase analysis in pulsed neutron transmission, Physica B: Condensed Matter, 350 (2004)***

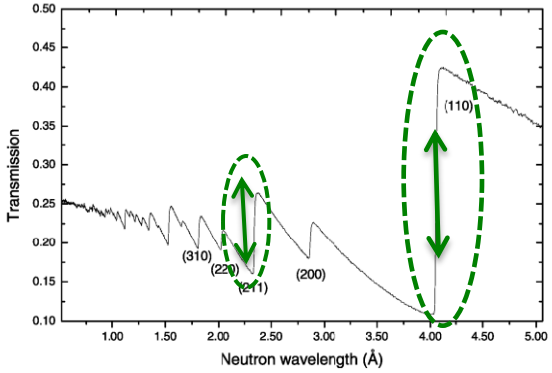
Neutron "Bragg Edge" Imaging: Applications

- **Record transmitted neutron beam energy resolved (TOF, tunable monochromator)**
- ***hkl* lattice spacing is probed in the direction of incoming beam and is "averaged" through thickness**

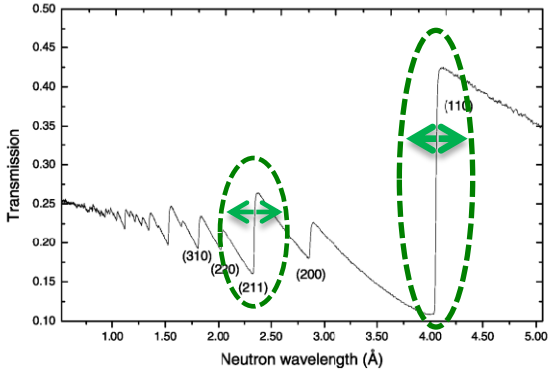
- **Phase**



- **Texture**



- **Strain**



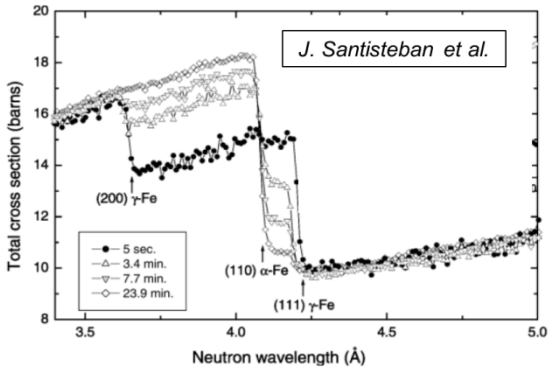
Selected References:

- A. Steuwer, P.J. Withers, J.R. Santisteban, L. Edwards, G. Bruno, M.E. Fitzpatrick, M.R. Daymond, M.W. Johnson, D. Wang, Bragg Edge Determination for Accurate Lattice Parameter and Elastic Strain Measurement, physica status solidi (a), 185 (2001)
- J.R. Santisteban, L. Edwards, M.E. Fitzpatrick, A. Steuwer, P.J. Withers, M.R. Daymond, M.W. Johnson, N. Rhodes, E.M. Schooneveld, Strain imaging by Bragg edge neutron transmission, NIMA, 481 (2002)
- R. Woracek, D. Penumadu, N. Kardjilov, A. Hilger, M. Strobl, R.C. Wimpory, I. Manke, J. Banhart, Neutron Bragg-edge-imaging for strain mapping under in situ tensile loading, Journal of Applied Physics, 109 (2011)

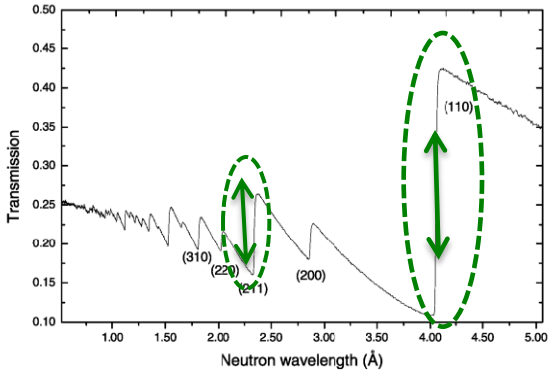
Neutron "Bragg Edge" Imaging: Applications

- **Record transmitted neutron beam energy resolved (TOF, tunable monochromator)**
- ***hkl* lattice spacing is probed in the direction of incoming beam and is "averaged" through thickness**

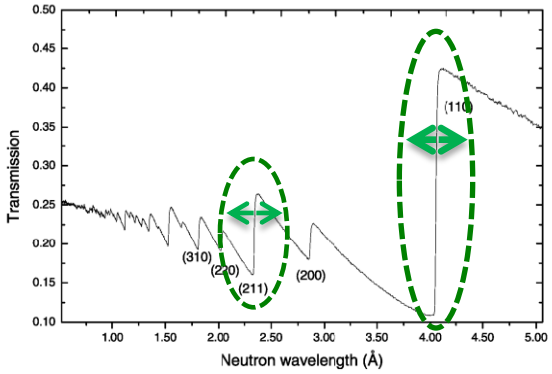
- **Phase**



- **Texture**



- **Strain**



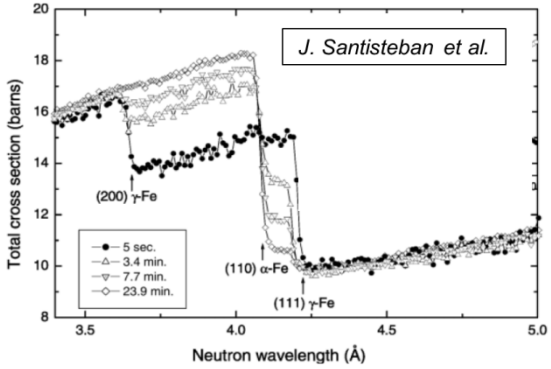
Selected References:

- *J.R. Santisteban, L. Edwards, V. Stelmukh, Characterization of textured materials by TOF transmission, Physica B: Condensed Matter, 385-386, (2006)*
- *J.R. Santisteban, M.A. Vicente-Alvarez, P. Vizcaino, A.D. Banchik, S.C. Vogel, A.S. Tremsin, J.V. Vallerga, J.B. McPhate, E. Lehmann, W. Kockelmann, Texture imaging of zirconium based components by total neutron cross-section experiments, Journal of Nuclear Materials, (2011).*

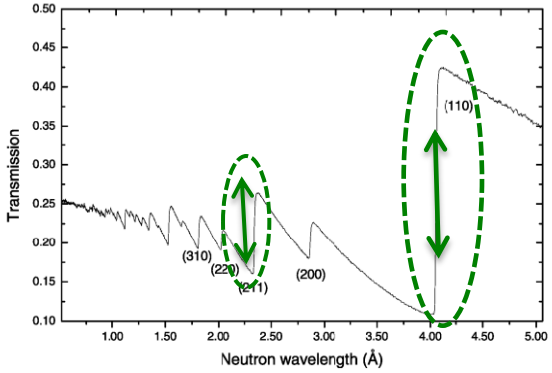
Neutron "Bragg Edge" Imaging: Applications

- **Record transmitted neutron beam energy resolved (TOF, tunable monochromator)**
- ***hkl* lattice spacing is probed in the direction of incoming beam and is "averaged" through thickness**

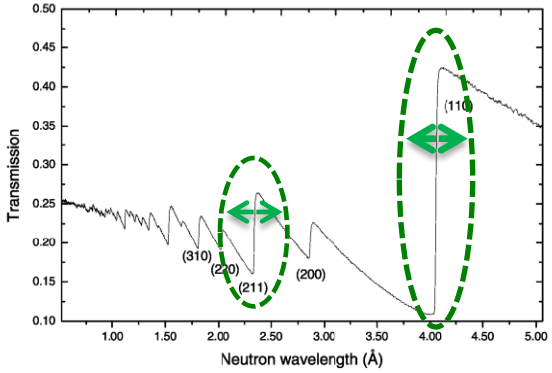
- **Phase**



- **Texture**



- **Strain**

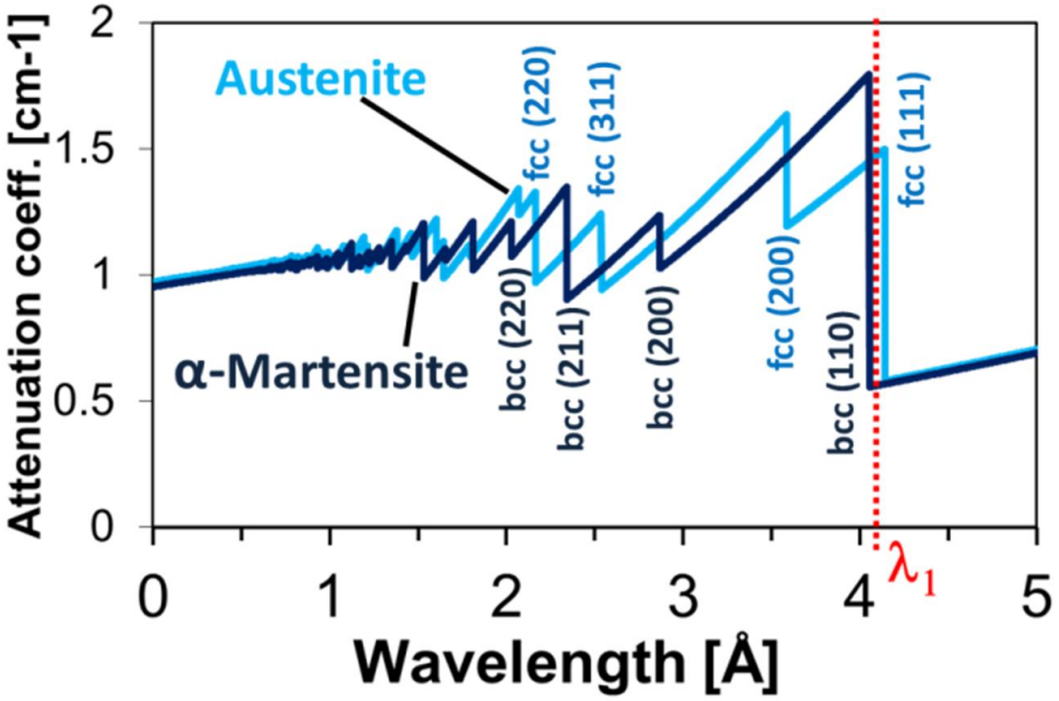


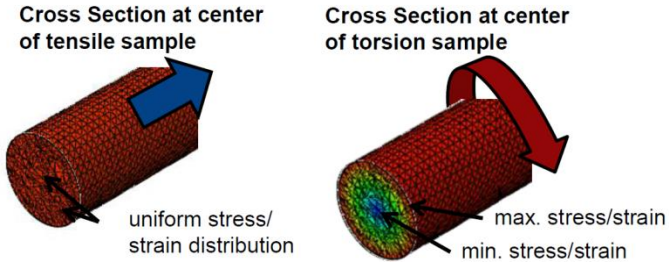
- Strain resolution of ~ 100με ($\Delta d_{hkl}/d_{hkl} \sim 1 \cdot 10^{-4}$) desirable for engineering applications *(E.g.: for iron this corresponds to bragg peak shift of: $2 \cdot \Delta d_{110} \sim 4 \cdot 10^{-4}$ Å)*

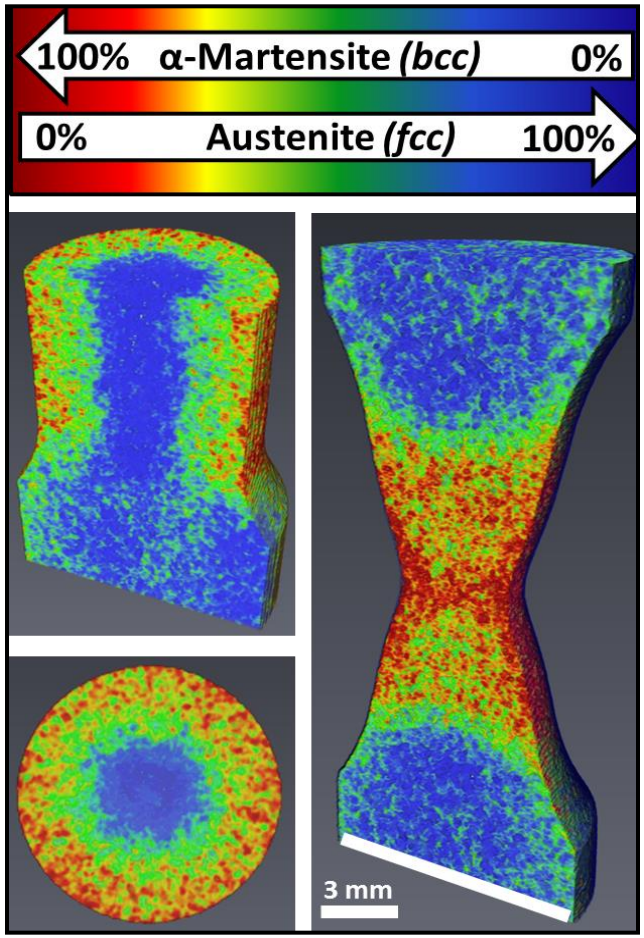
Diffraction Contrast

University of Tennessee Knoxville

3D Phase mapping in metals



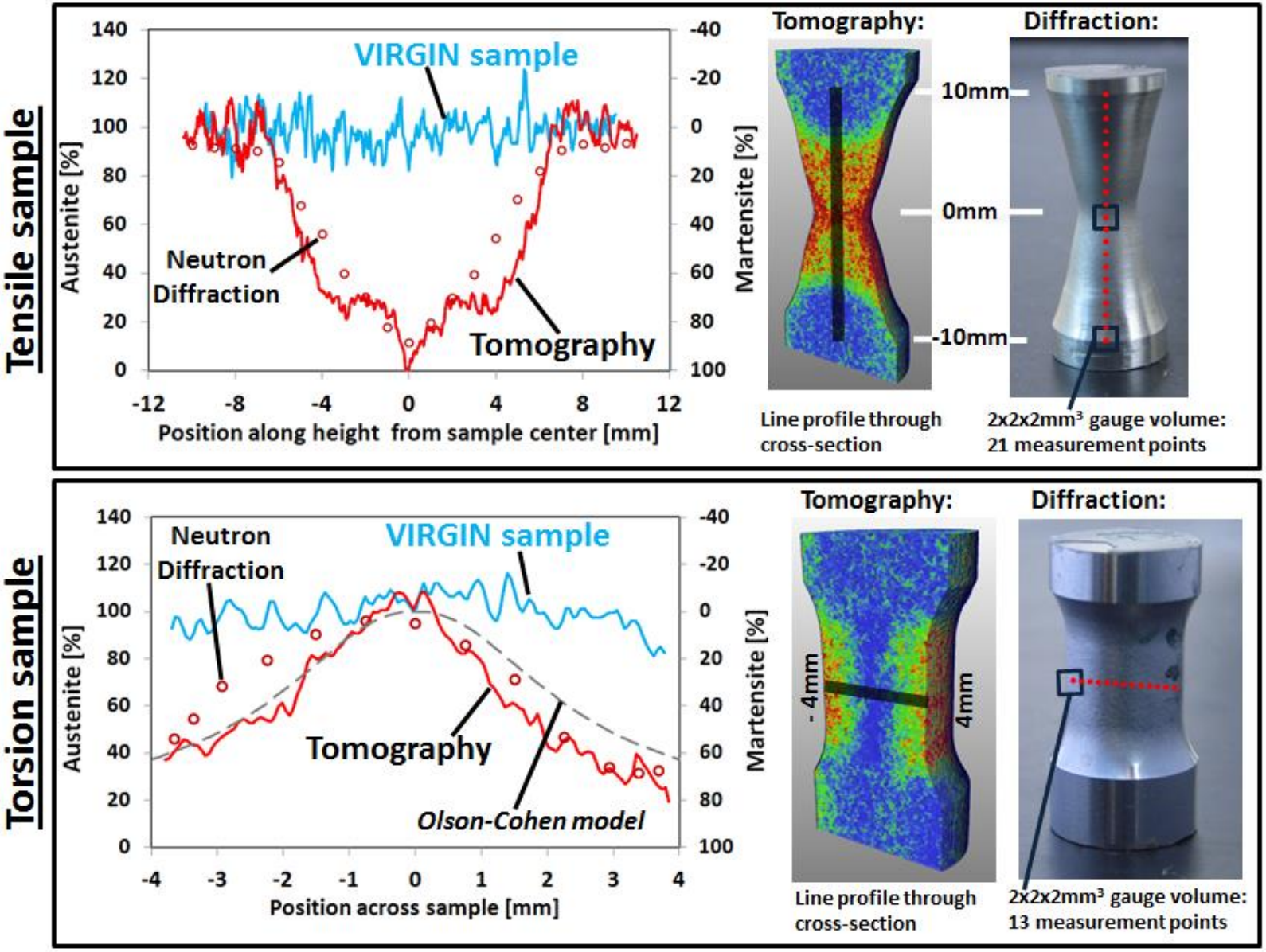




Energy-selective neutron tomography of TRIP-steel

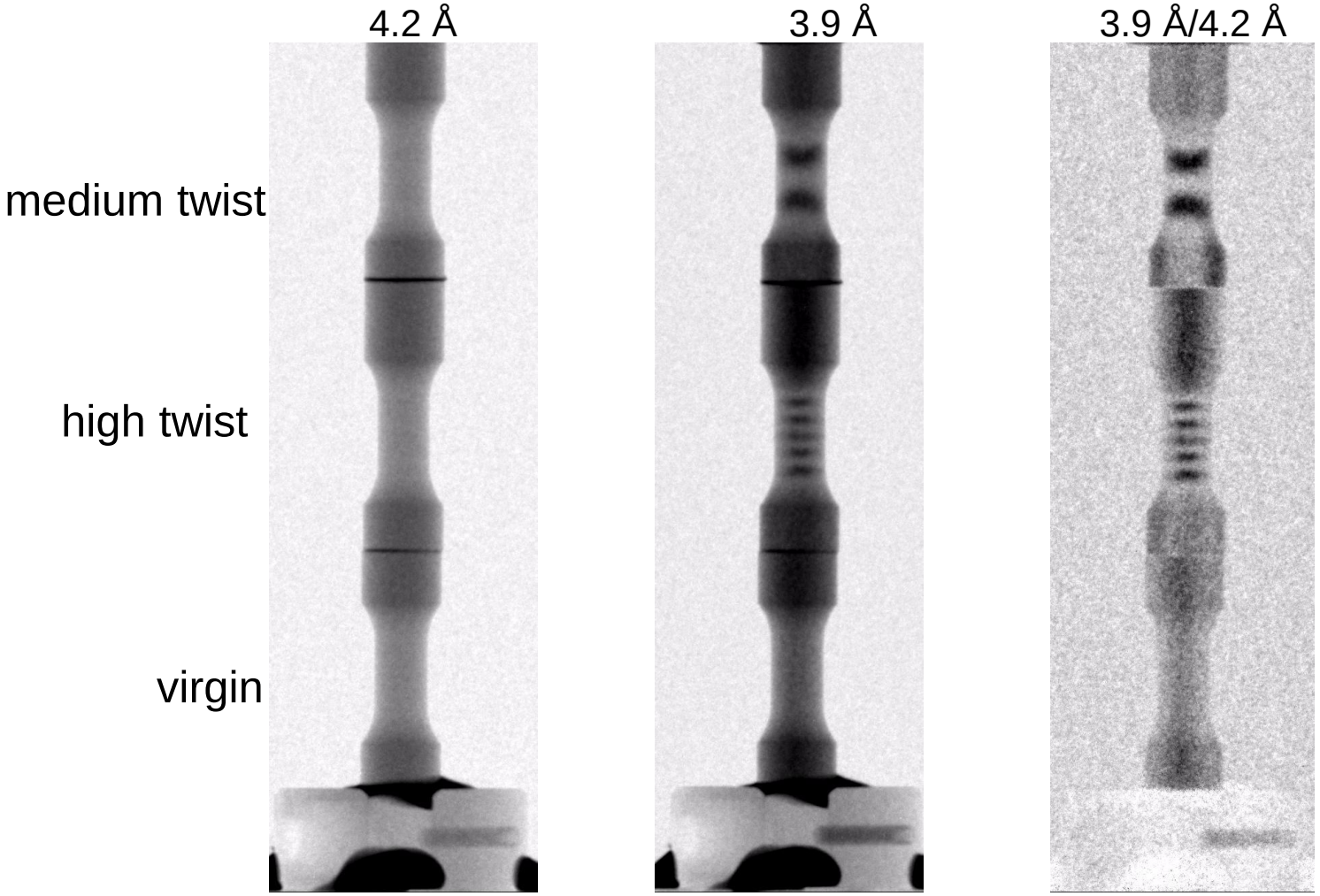
R. Woracek et al., **Advanced Materials** 26 (2014)

Diffraction Contrast



Diffraction Contrast

- Different set of torsion samples: Strong texture
- Difficult to study phase transformation in such samples for both imaging & diffraction methods (dominated by texture effect)



Diffraction Contrast

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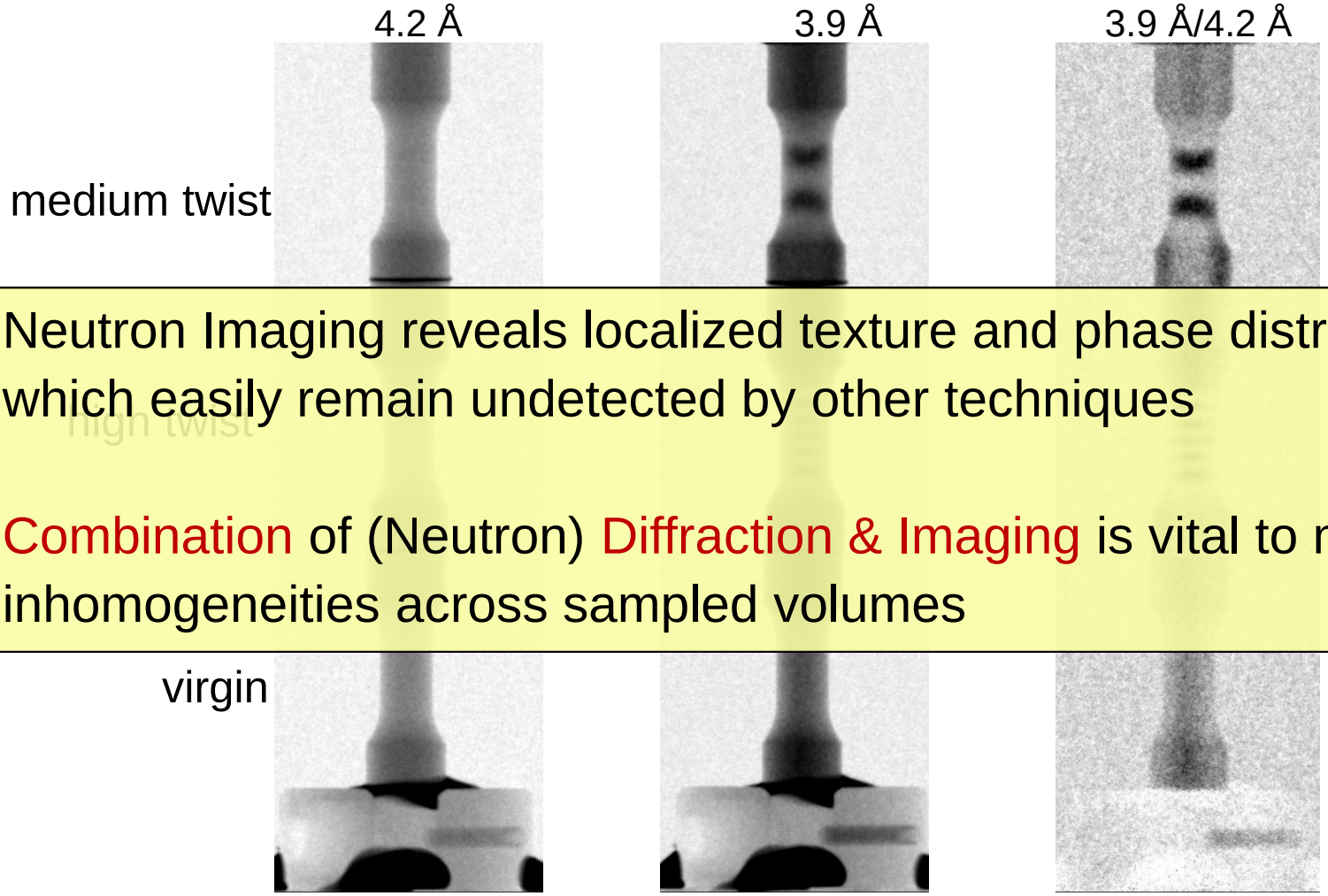


- Neutron Imaging reveals localized texture and phase distribution which easily remain undetected by other techniques
- Combination of (Neutron) Diffraction & Imaging is vital to monitor for inhomogeneities across sampled volumes

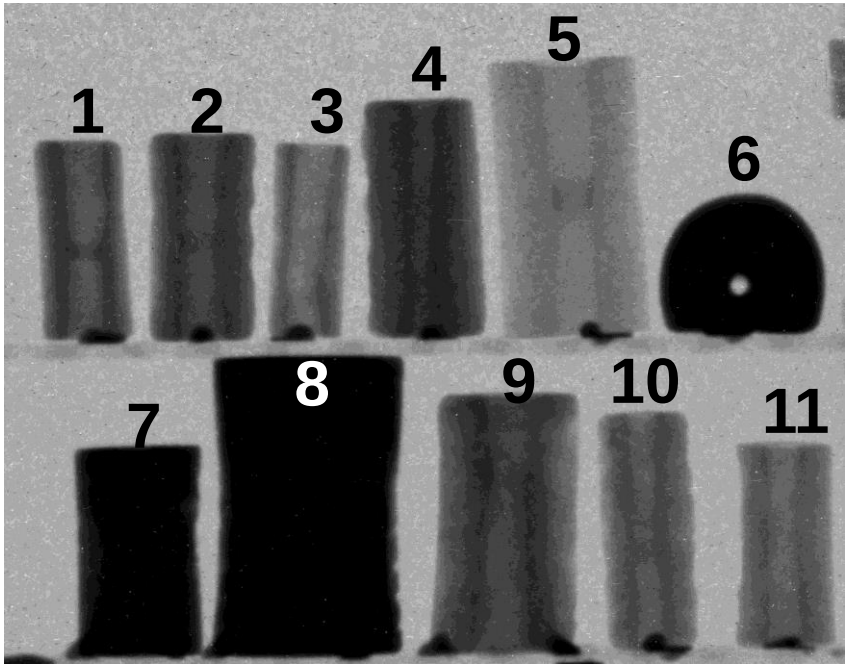
Diffraction Contrast

Mesopotamian Seals (from c. 2000 - 1600 BC)

(Theo Krispijn, *NINO Institute*, Dirk Visser, *Delft University of Techn*ology, Holland)

Photo

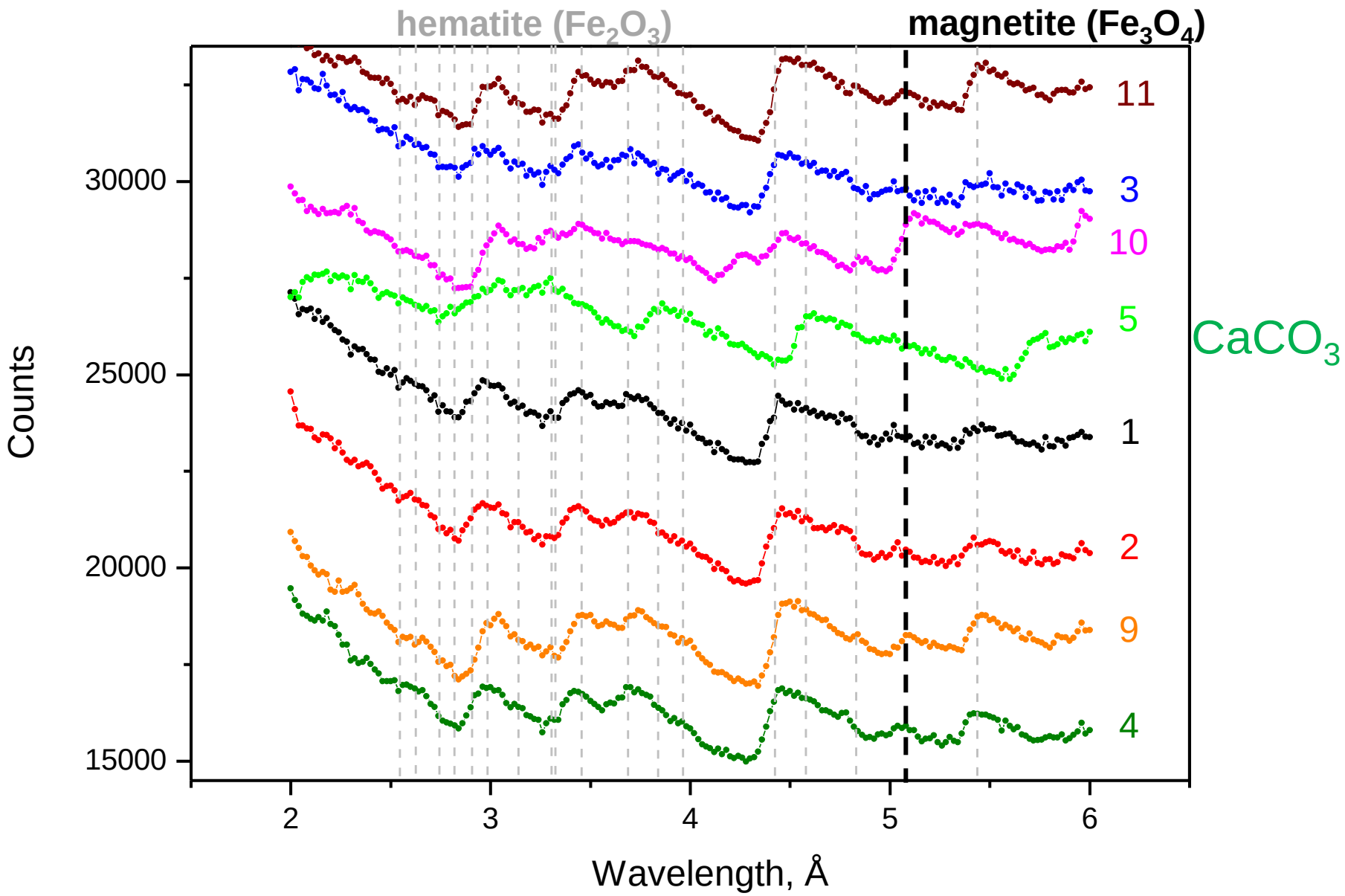
Neutron radiography



1 cm

The De Liagre-Böhl collection of the Dutch Institute for the Near East (NINO) houses about 150 seals, 13 of which were visually identified as hematite. The seals were acquired in Iraq at the beginning of the last century.

Diffraction Contrast



Diffraction Contrast

Seal Nr. DLB 67 (De Liagre-Böhl collection)

31 wt. % hematite (Fe_2O_3)

69 wt. % magnetite (Fe_3O_4)

1 cm

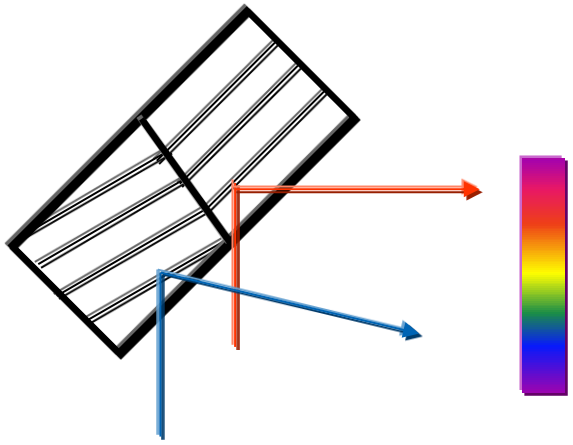
Wavelength gradient

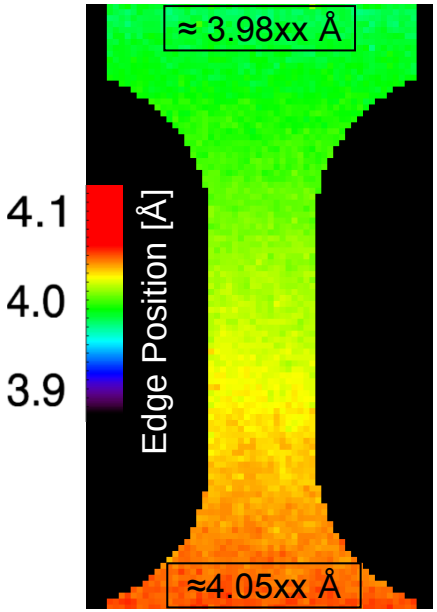
Advantages:

- good energy resolution (1 – 10 %)
- constant neutron flux
- no need for vacuum and cooling instalations

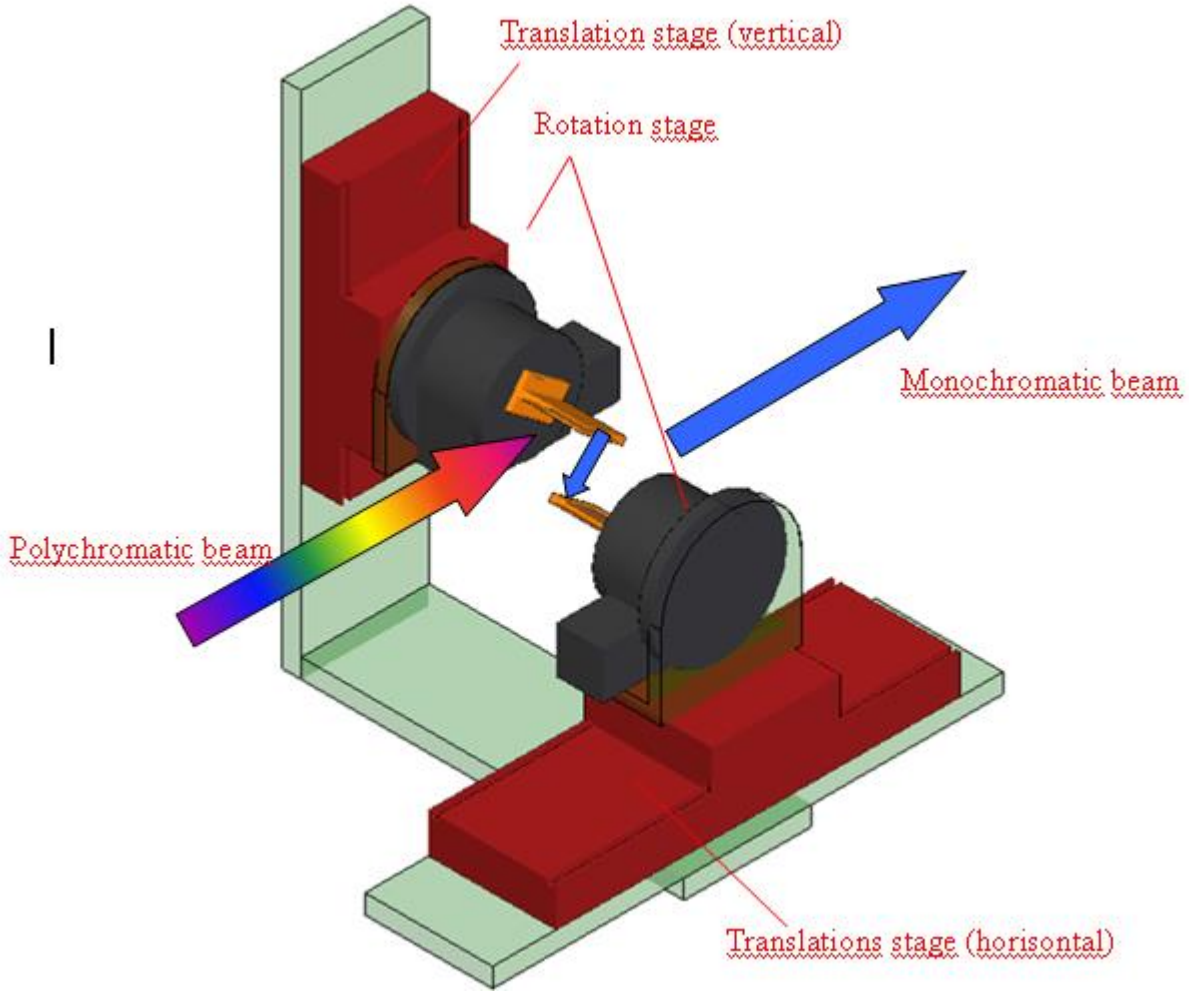
Disadvantages:

- mechanical instability
- wavelength gradient

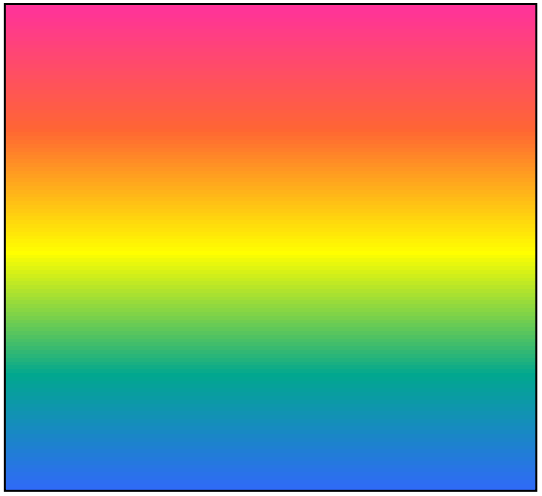




Wavelength gradient

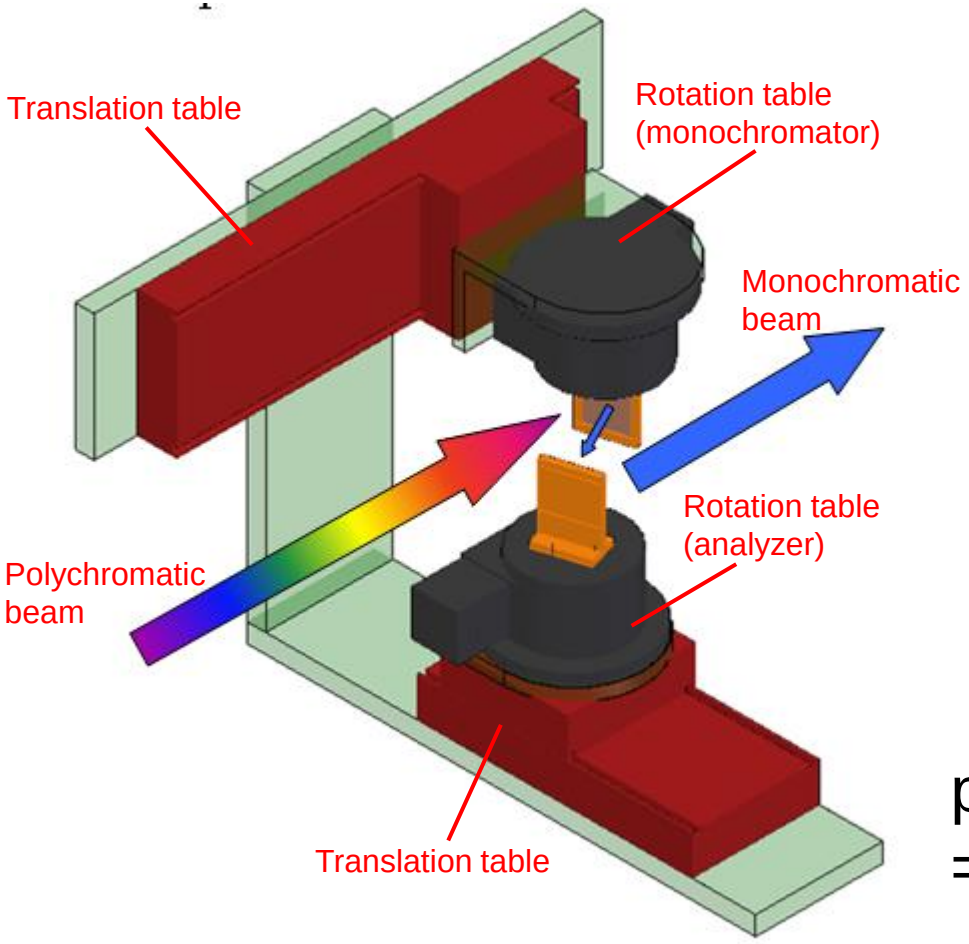


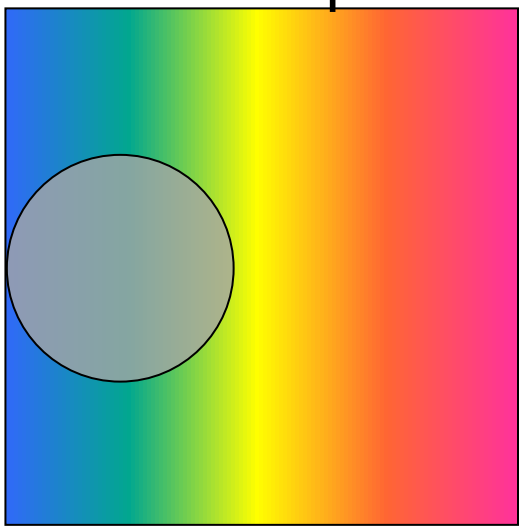
Detector plane



Wavelength gradient: ~ 0.006Å/cm

Wavelength gradient





pixel size: 100 µm = 0.001 cm
=> 0.00006Å / pixel

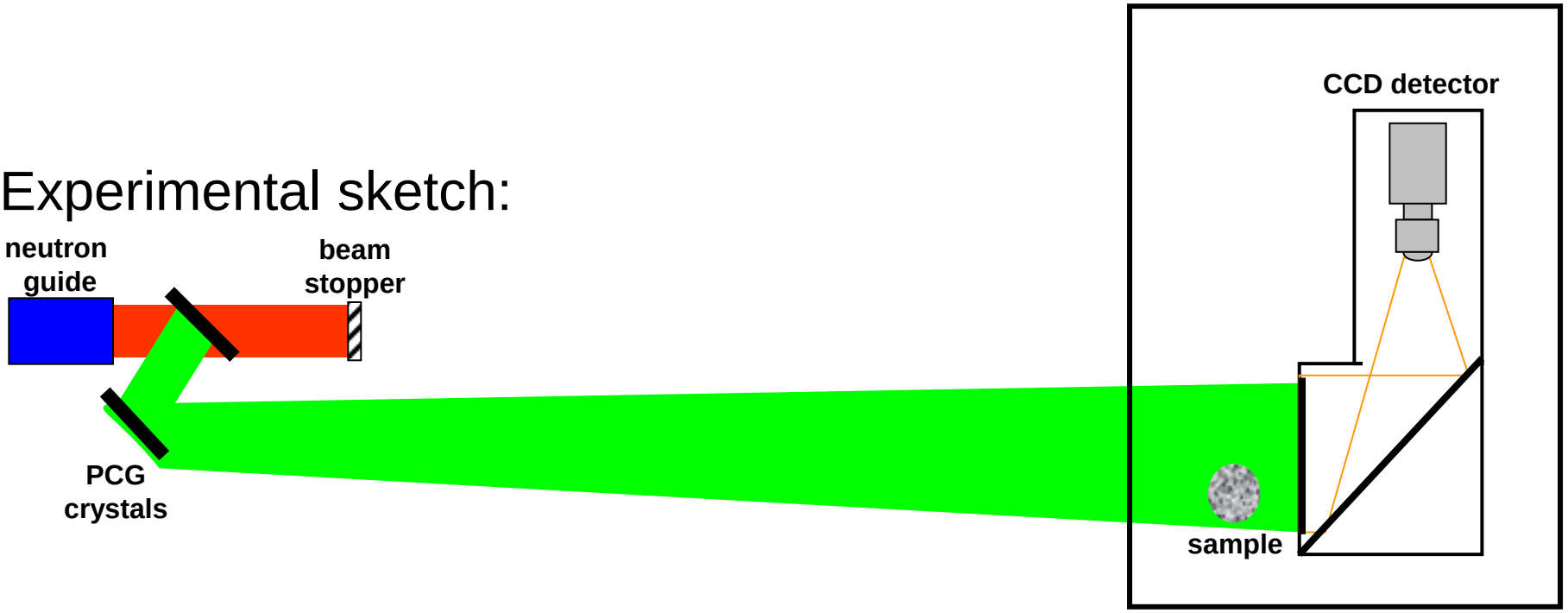
Wavelength gradient: ~ 0.006Å/cm

Wavelength gradient

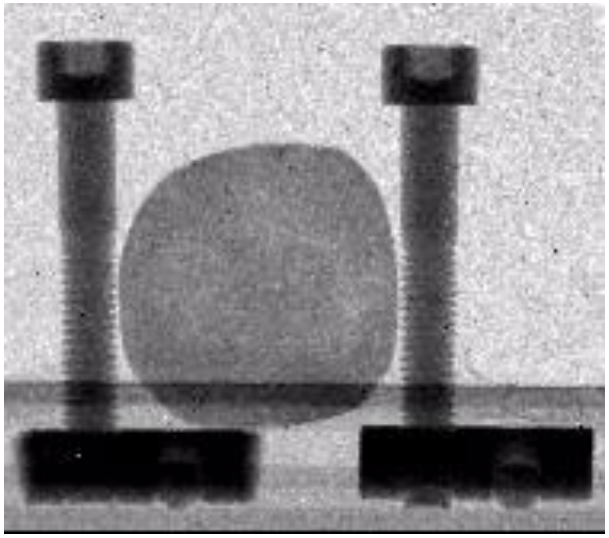
Maximal translation path: 300 mm
Variable scan speed: 0.01 – 200 mm/s
Position accuracy: ± 0.02 / 300 mm

Maximal sample width: 200 mm

Experimental sketch:



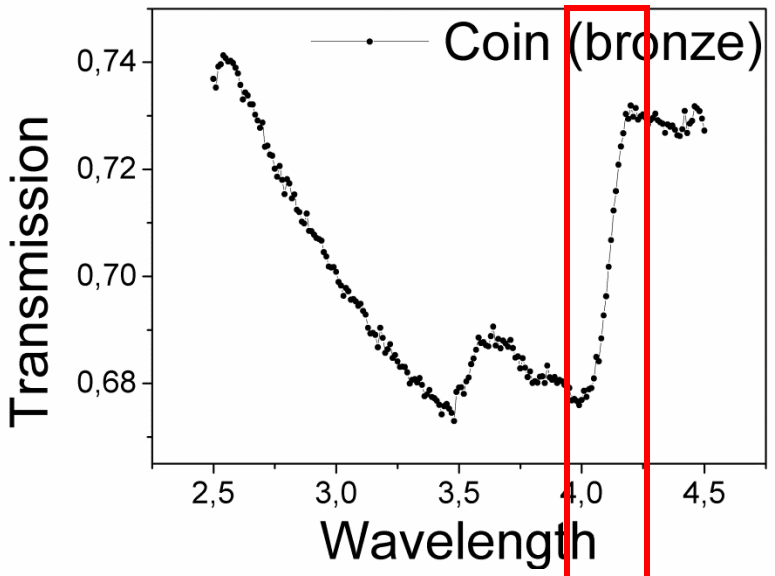
Wavelength gradient

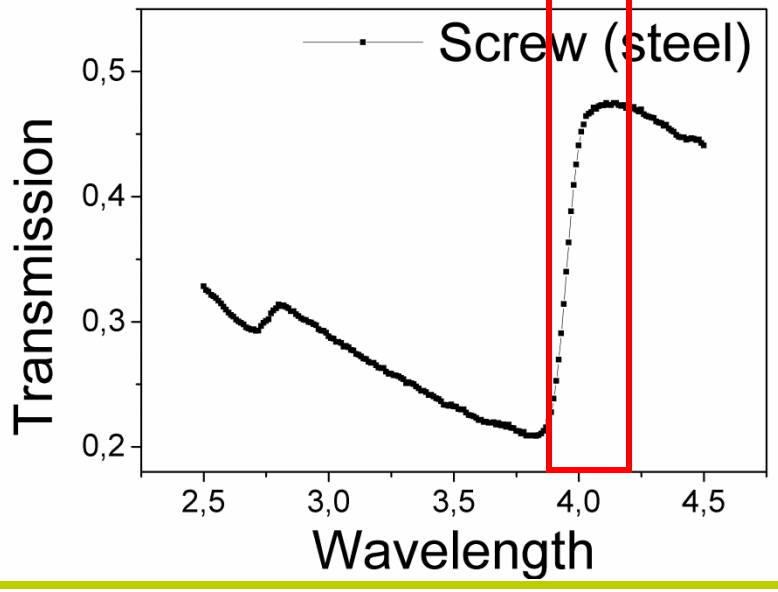


Wavelength scan:
2.5 Å – 4.5 Å, step 0.01 Å

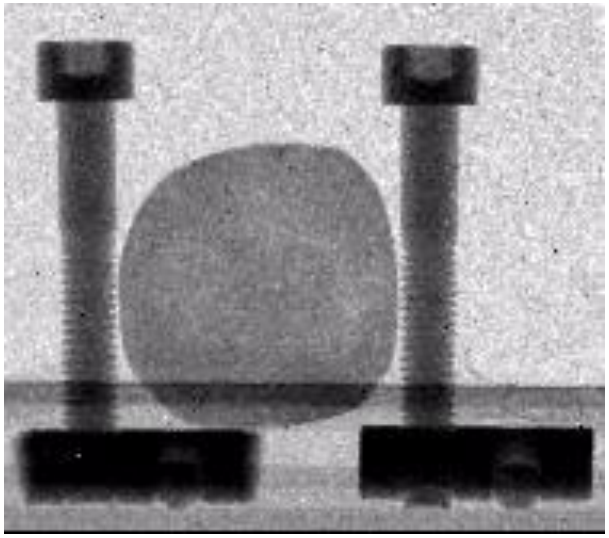
Exposure time:
200 s / image

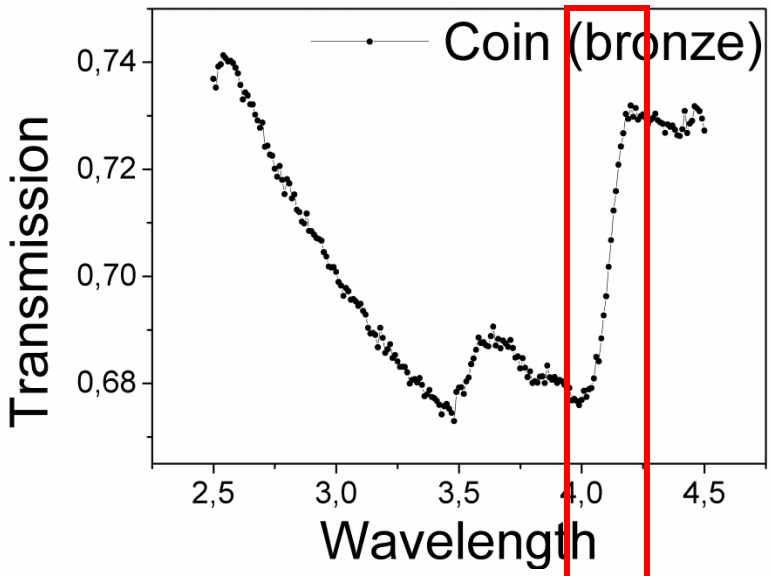
Total: ~ 23 h





Wavelength gradient





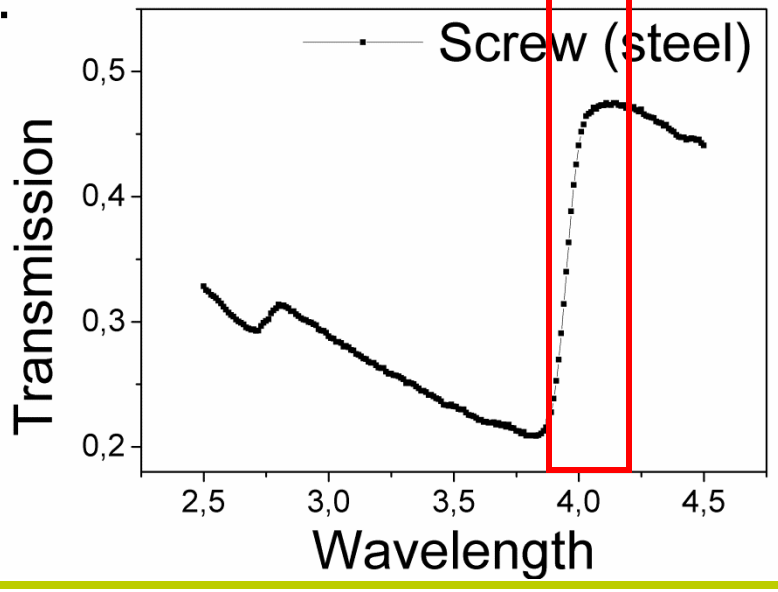
Wavelength + Translation scan:

3.90 Å: 0-100 mm, step 2 mm

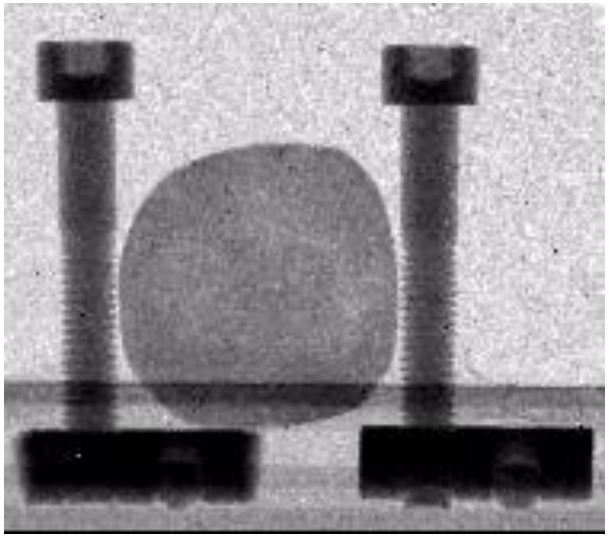
3.95 Å: 0-100 mm, step 2 mm

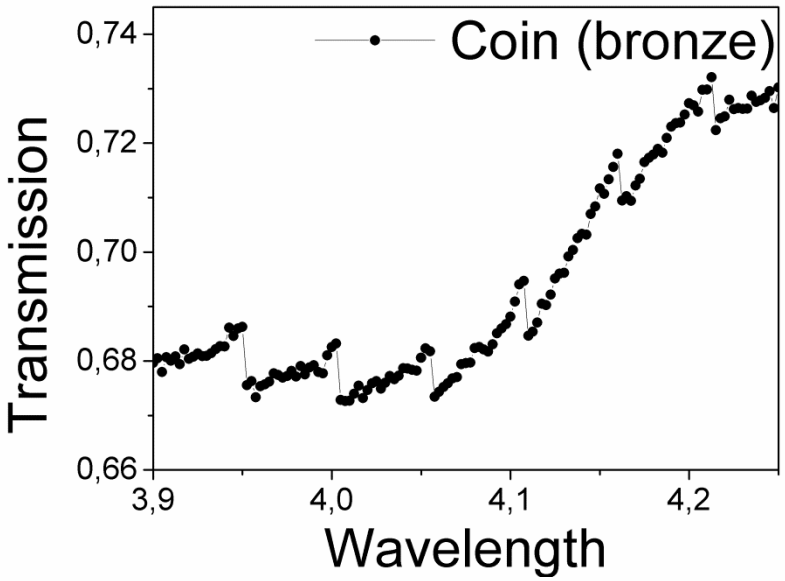
…

4.25 Å: 0-100 mm, step 2 mm



Wavelength gradient





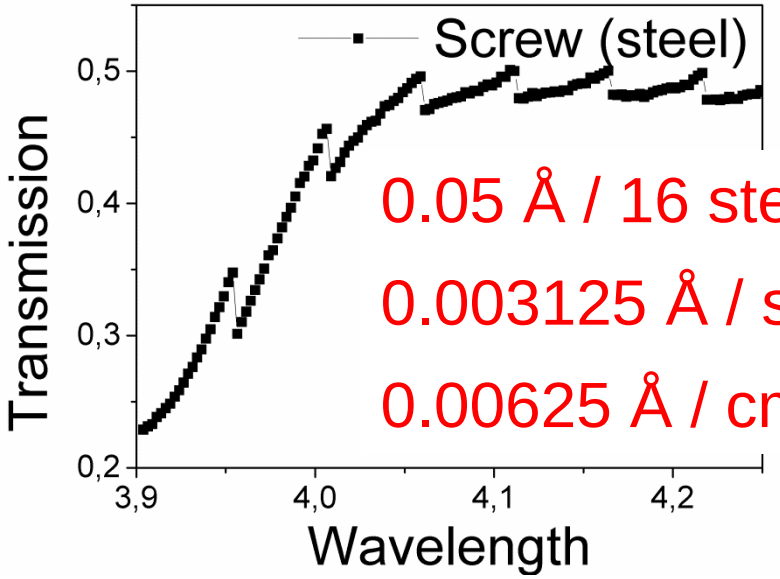
Wavelength + Translation scan:

3.90 Å: 0-100 mm, step 5 mm

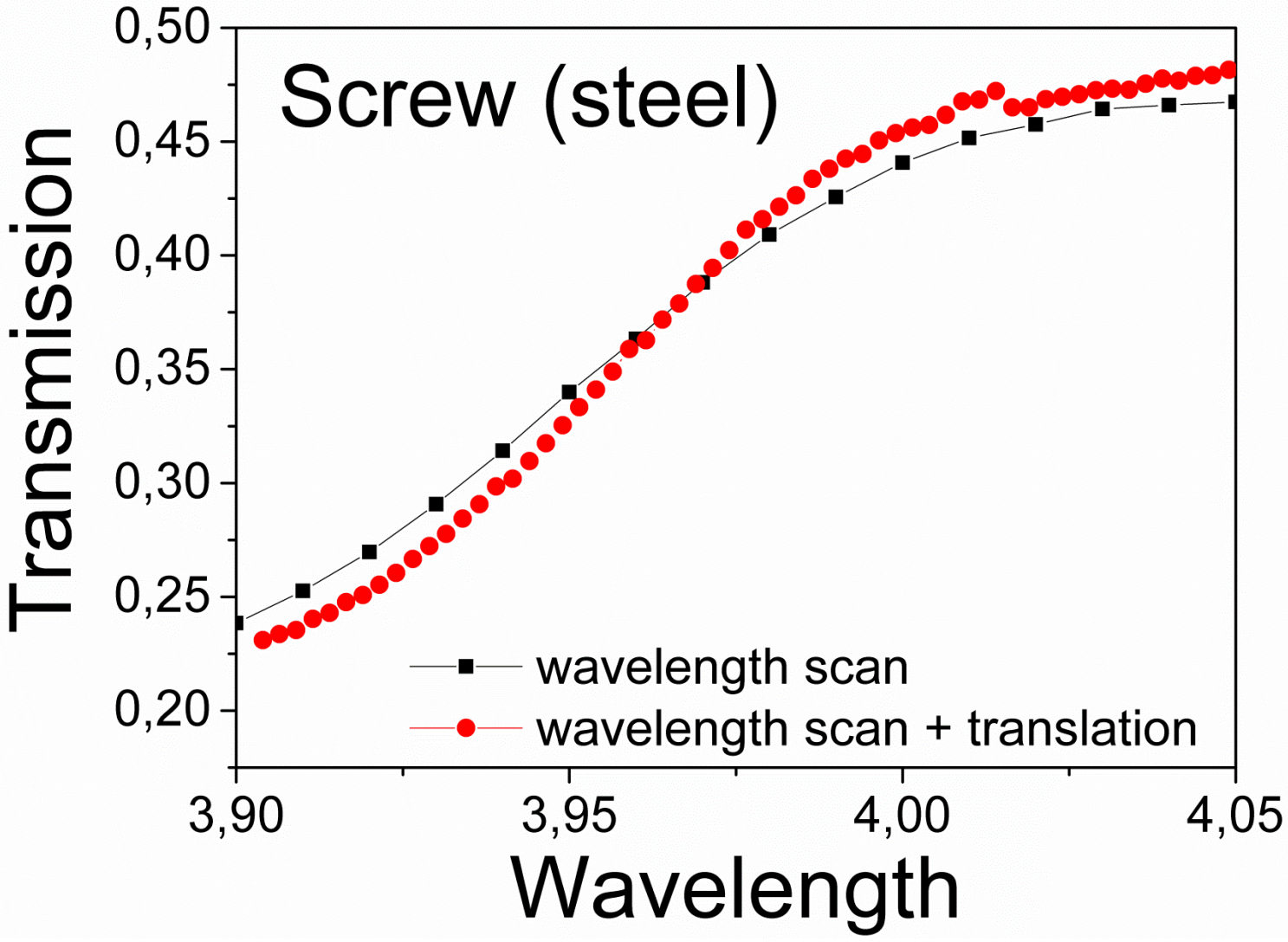
3.95 Å: 0-100 mm, step 5 mm

…

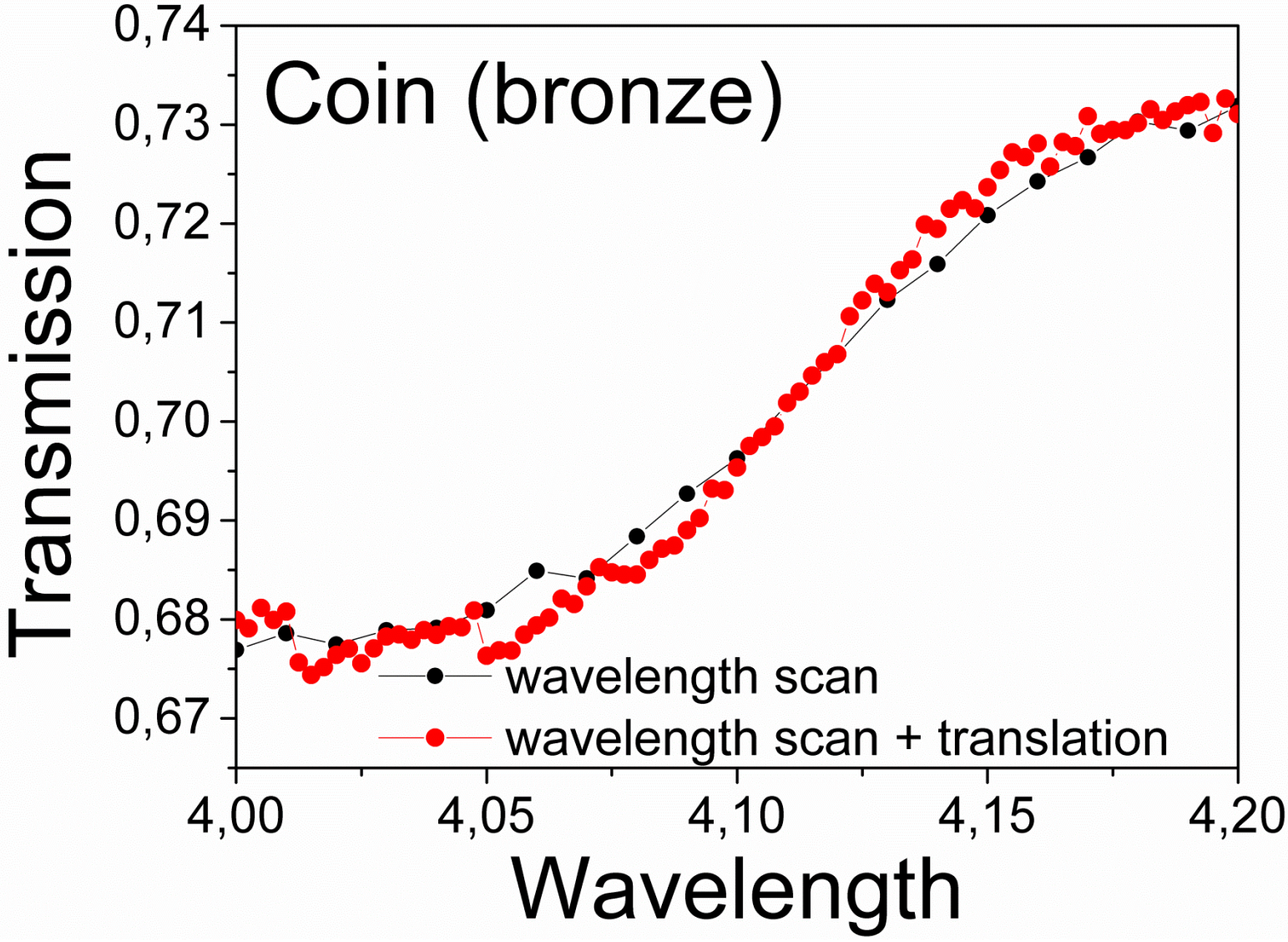
4.25 Å: 0-100 mm, step 5 mm



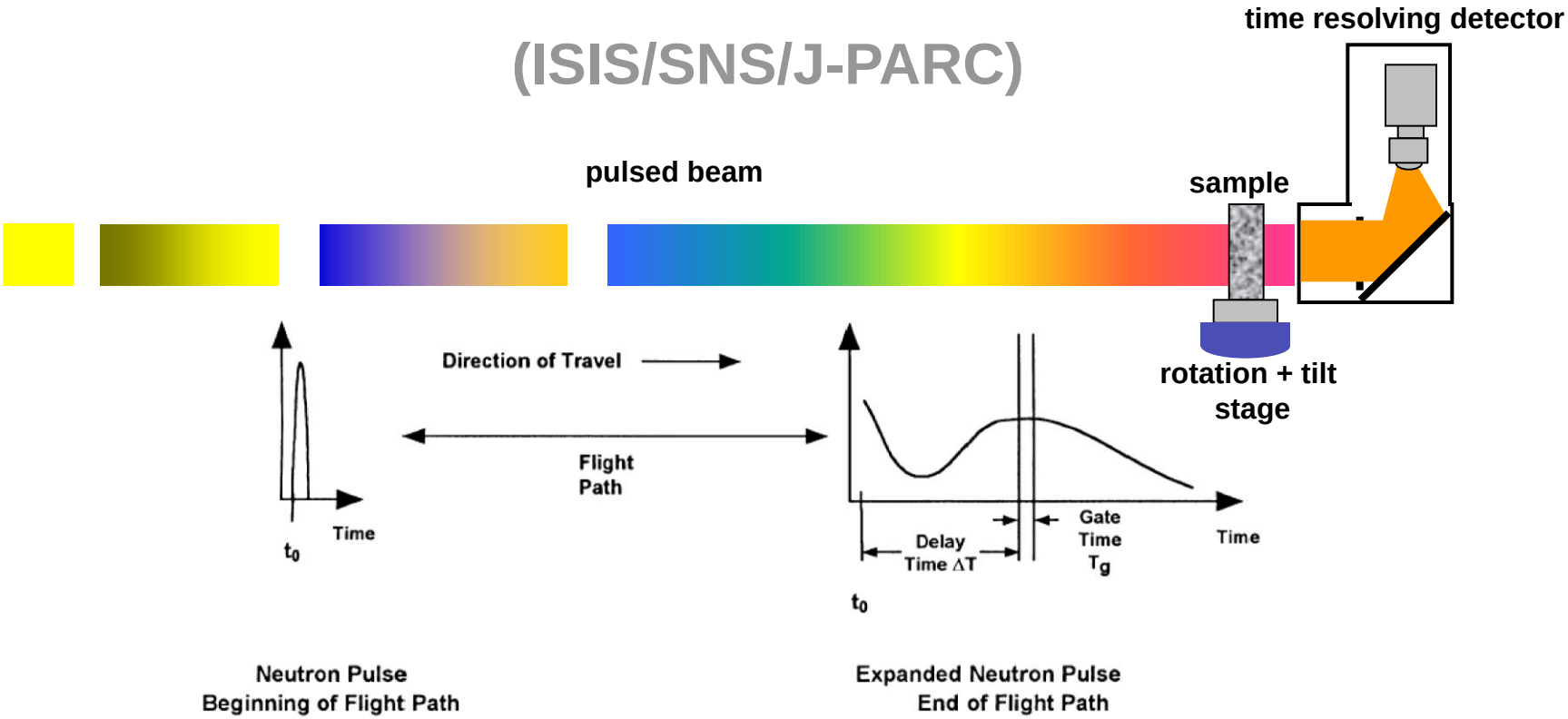
Wavelength gradient



Wavelength gradient

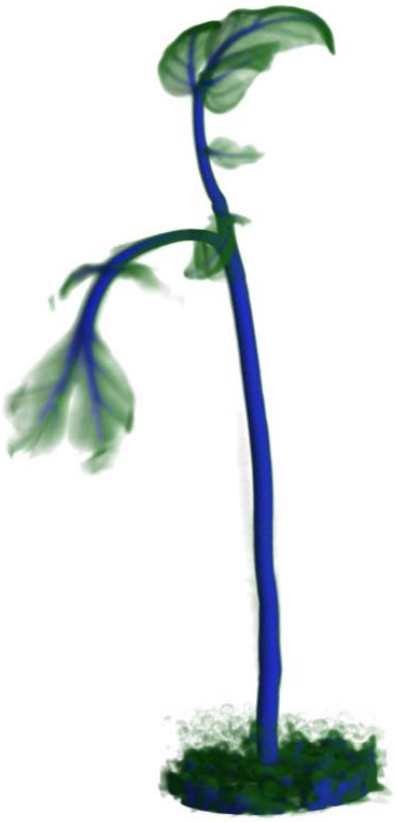


Beam monochromatization



Time of flight

Resolution ($\Delta\lambda/\lambda$): ~ 0.1% - 1 %
Excelent resolution
Extreme long exposure times



Thank you !