Non-destructive testing with neutrons: Engineering materials and components revealed

African School of Fundamental Physics and Applications

ASP Online Seminar Series: Photons and Neutrons in the quest to solve societal challenges

Part B: Large Research Infrastructure as tools for innovation

PRESENTED BY ROBIN WORACEK
// INSTRUMENT CLASS COORDINATOR IMAGING & ENGINEERING

ROBIN.WORACEK@ESS.EU

2021-01-26

Non-destructive testing with neutrons: Engineering materials and components revealed

- ❑ Characterization Techniques & Contrast Mechanisms
- ❑ Neutron Methods & Length Scales
- ❑ Applications using Imaging (Attenuation Contrast)
- ❑ Applications using SANS and Diffraction
- ❑ Applications using Imaging (Diffraction and Scattering Contrast)
- ❑ Summary & Outlook

Array of characterization techniques

Traditional lab methods

Optical microscopy

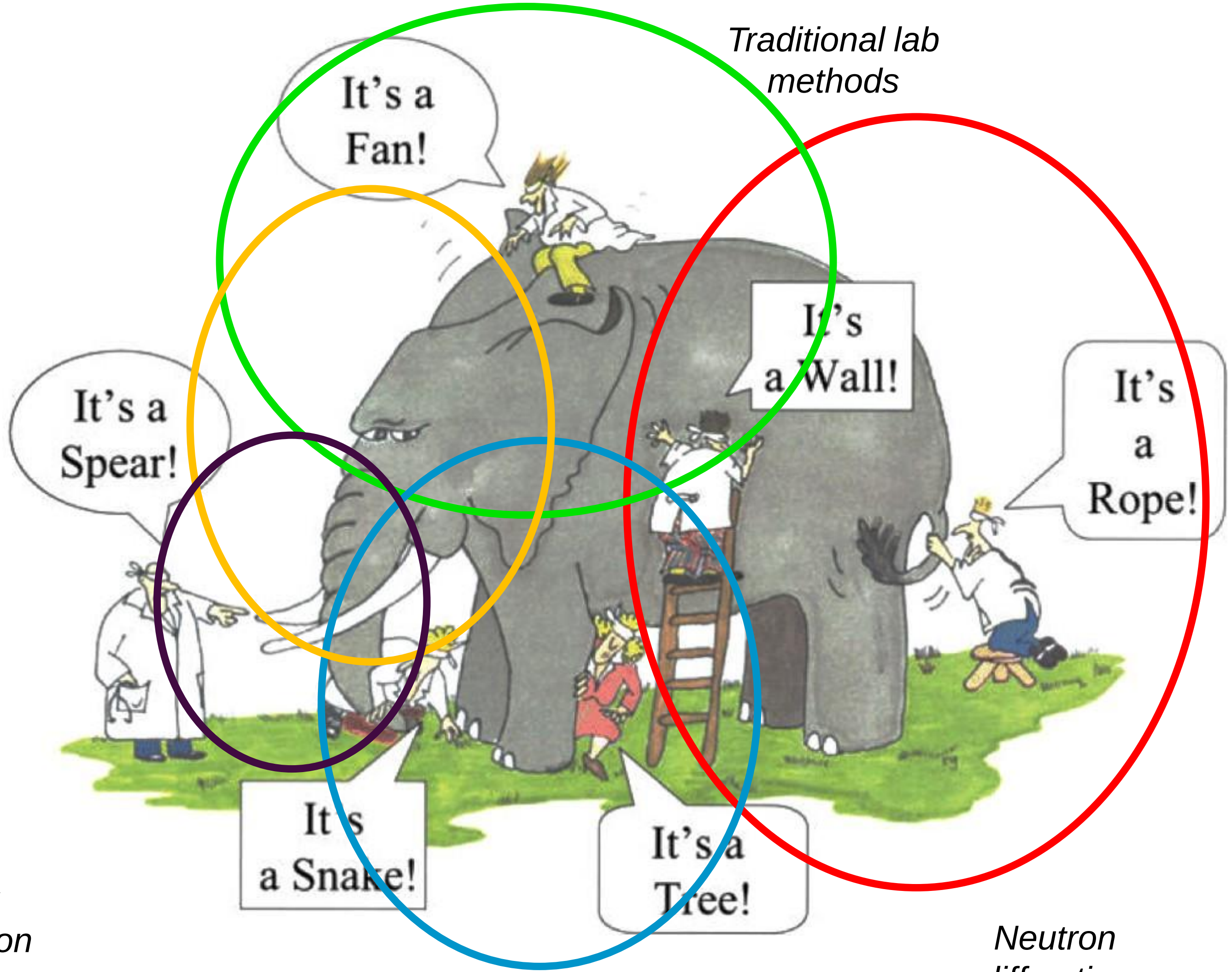
SEM

TEM

Atom probe

SANS/SAXS

X-ray diffraction



The six blind men and the elephant

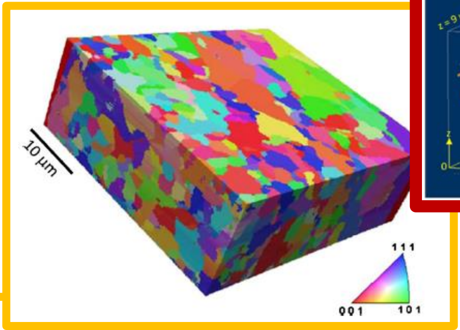
Neutron diffraction

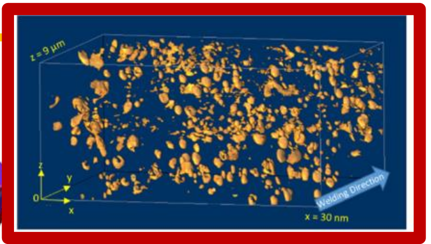
Neutron Imaging

X-ray imaging

....many methods..

Array of characterization techniques: Tomography





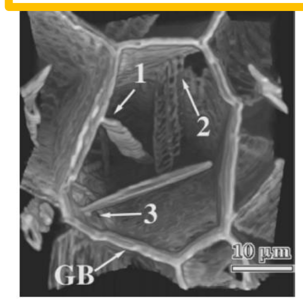
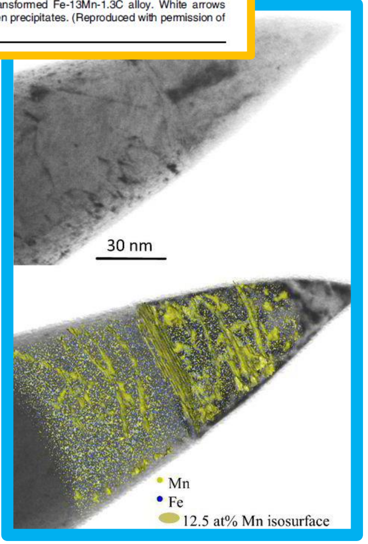


Fig. 2. 3D reconstruction of proeutectoid cementite (white features) in an isothermally transformed Fe-13Mn-1.3C alloy. White arrows point to Widmanstätten precipitates. (Reproduced with permission of Ref. 54).



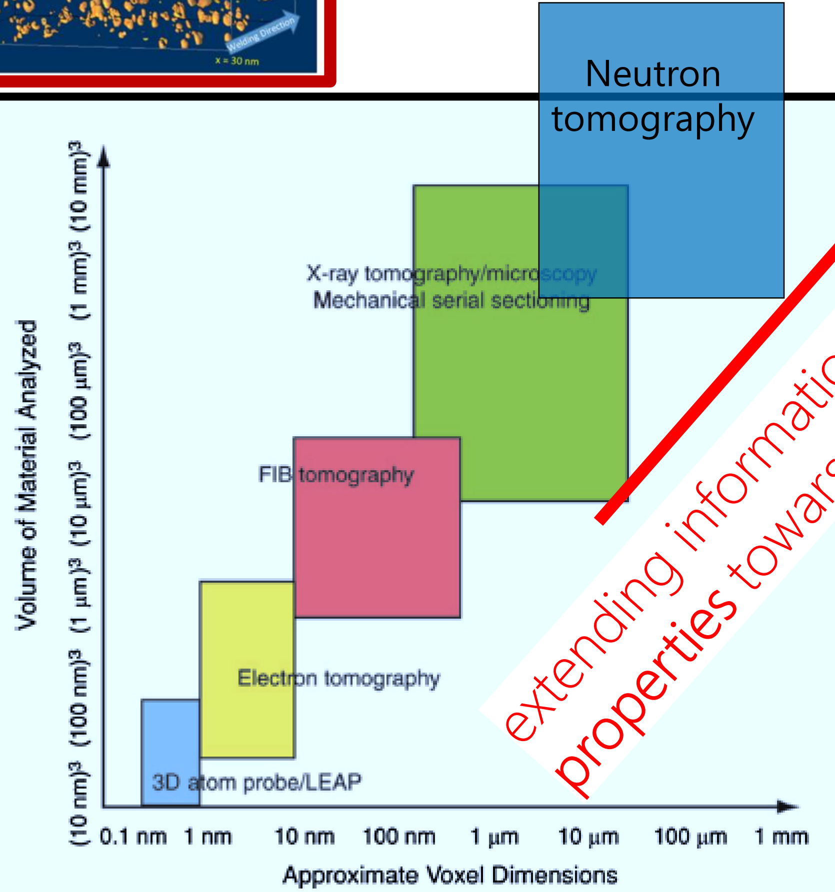
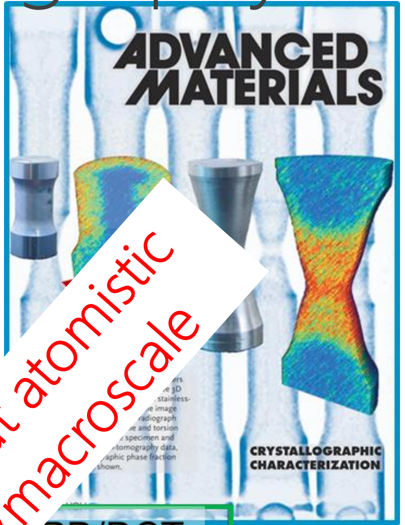
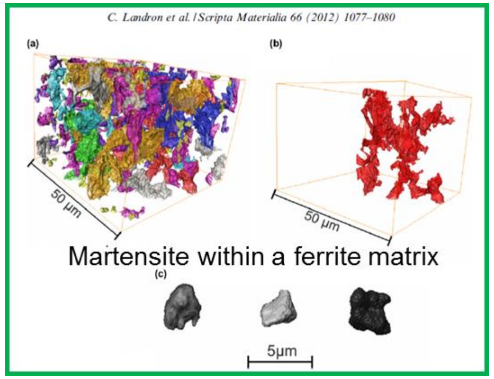


Fig. 3. Graphical representation of the resolution and typical volume analyzed per experiment for modern tomographic characterization methods. (Reproduced with permission of Ref. 59).

extending information about atomistic properties towards meso-/macroscale



XRD/DCT



Martensite within a ferrite matrix

S.N. Monteiro, S. Paciornik. "From Historical Backgrounds to Recent Advances in 3D Characterization of Materials: An Overview." JOM 69.1 (2017): 84-92.

X-rays and neutrons

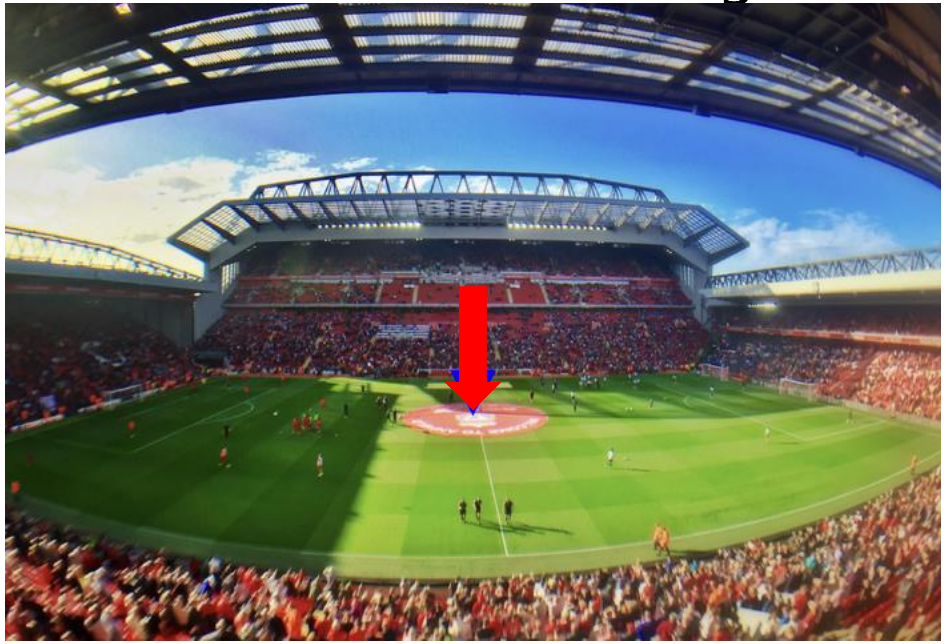
- **Neutrons** interact with the **nuclei** of the atoms: **strong nuclear force**.
- Different to **light** and **X-rays,** which interact with **the electron clouds** surrounding the nuclei: **electromagnetic force**.

- We can imagine the **nucleus** of the size of a **marble**.

Courtesy J. Houston, ESS

X-rays and neutrons

- **Neutrons** interact with the **nuclei** of the atoms: **strong nuclear force**.
- Different to **light** and **X-rays**, which interact with **the electron clouds** surrounding the nuclei: **electromagnetic force**.

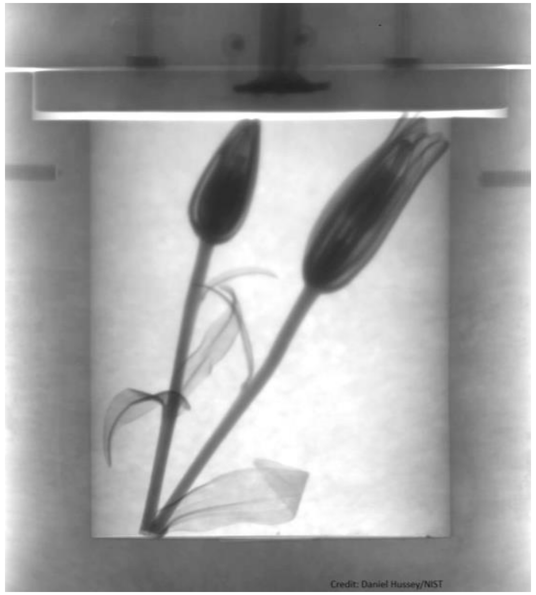


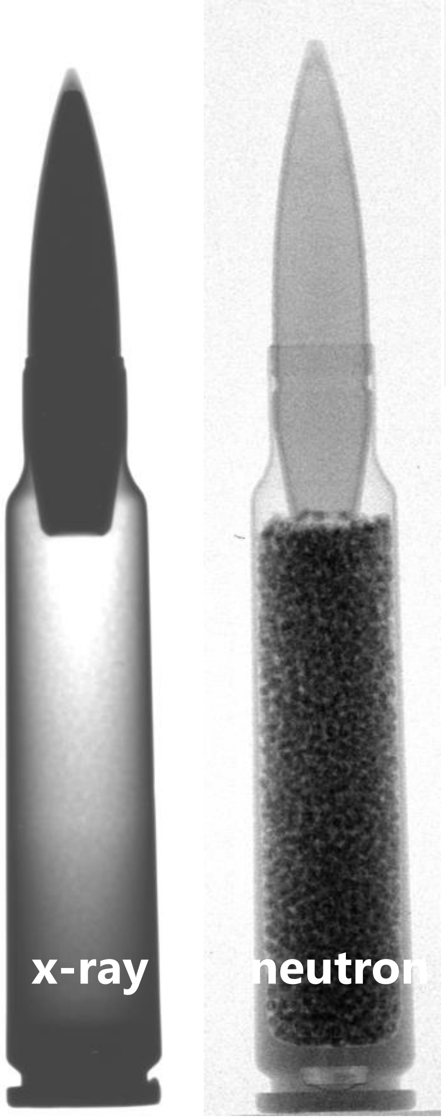
- We can imagine the **nucleus** of the size of a **marble**.
- The **atom** in proportion will be as big as a football **stadium**.
- **Neutrons interact** with the sample **only** when they **hit the nucleus**.

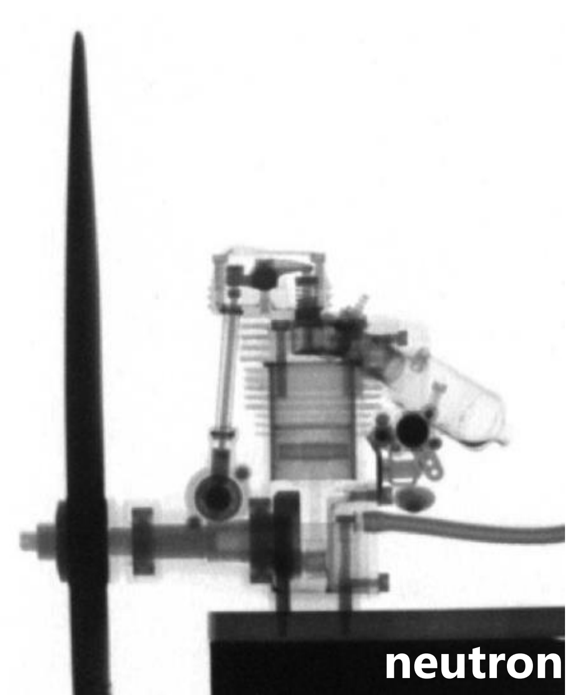
Courtesy J. Houston, ESS

X-rays and neutrons

Neutrons ‘see’ light elements







Courtesy of NIST, PSI

X-rays and neutrons

Attenuation coefficients with X-ray [cm?¹]

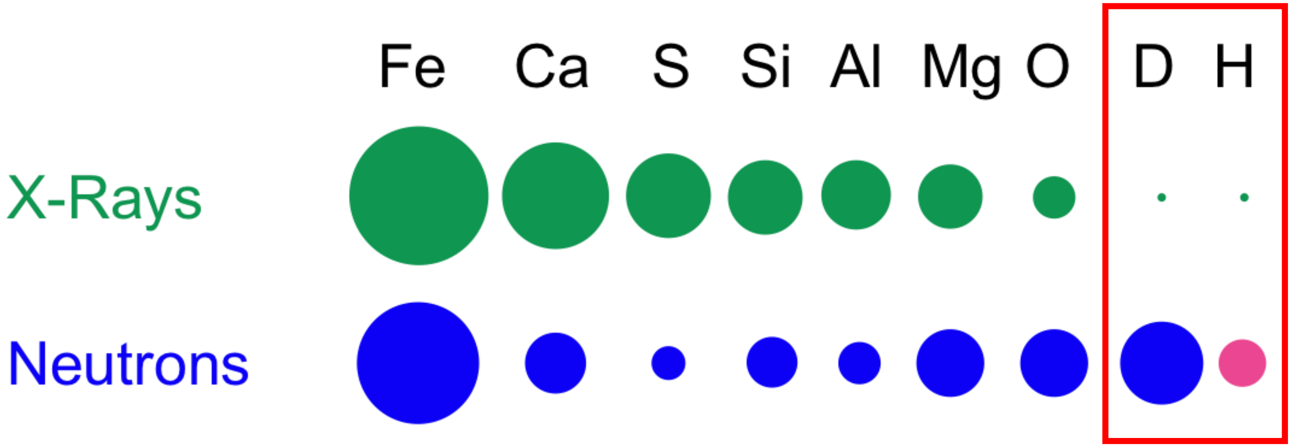
1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 0.02																	He 0.02
Li 0.06	Be 0.22											B 0.28	C 0.27	N 0.11	O 0.16	F 0.14	Ne 0.17
Na 0.13	Mg 0.24											Al 0.38	Si 0.33	P 0.25	S 0.30	Cl 0.23	Ar 0.20
K 0.14	Ca 0.26	Sc 0.48	Ti 0.73	V 1.04	Cr 1.29	Mn 1.32	Fe 1.57	Co 1.78	Ni 1.96	Cu 1.97	Zn 1.64	Ga 1.42	Ge 1.33	As 1.50	Se 1.23	Br 0.90	Kr 0.73
Rb 0.47	Sr 0.86	Y 1.61	Zr 2.47	Nb 3.43	Mo 4.29	Tc 5.06	Ru 5.71	Rh 6.08	Pd 6.13	Ag 5.67	Cd 4.84	In 4.31	Sn 3.98	Sb 4.28	Te 4.06	I 3.45	Xe 2.53
Cs 1.42	Ba 2.73	La 5.04	Hf 19.70	Ta 25.47	W 30.49	Re 34.47	Os 37.92	Ir 39.01	Pt 38.61	Au 35.94	Hg 25.88	Tl 23.23	Pb 22.81	Bi 20.28	Po 20.22	At	Rn 9.77
Fr	Ra 11.80	Ac 24.47	Rf	Ha													
Lanthanides	Ce 5.79	Pr 6.23	Nd 6.46	Pm 7.33	Sm 7.68	Eu 5.66	Gd 8.69	Tb 9.46	Dy 10.17	Ho 10.91	Er 11.70	Tm 12.49	Yb 9.32	Lu 14.07			
*Actinides	Th 28.95	Pa 39.65	U 49.08	Np	Pu	Am	Cm	Bk	Vf	Es	Fm	Md	No	Lr x-ray			

For X-rays = proportional increase!

Attenuation coefficients with neutrons [cm?¹]

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 3.44																	He 0.02
Li 3.30	Be 0.79											B 101.60	C 0.56	N 0.43	O 0.17	F 0.20	Ne 0.10
Na 0.09	Mg 0.15											Al 0.10	Si 0.11	P 0.12	S 0.06	Cl 1.33	Ar 0.03
K 0.06	Ca 0.08	Sc 2.00	Ti 0.60	V 0.72	Cr 0.54	Mn 1.21	Fe 1.19	Co 3.92	Ni 2.05	Cu 1.07	Zn 0.35	Ga 0.49	Ge 0.47	As 0.67	Se 0.73	Br 0.24	Kr 0.61
Rb 0.08	Sr 0.14	Y 0.27	Zr 0.29	Nb 0.40	Mo 0.52	Tc 1.76	Ru 0.58	Rh 10.88	Pd 0.78	Ag 4.04	Cd 115.11	In 7.58	Sn 0.21	Sb 0.30	Te 0.25	I 0.23	Xe 0.43
Cs 0.29	Ba 0.07	La 0.52	Hf 4.99	Ta 1.49	W 1.47	Re 6.85	Os 2.24	Ir 30.46	Pt 1.46	Au 6.23	Hg 16.21	Tl 0.47	Pb 0.38	Bi 0.27	Po	At	Rn
Fr	Ra 0.34	Ac	Rf	Ha													
*Lanthanides	Ce 0.14	Pr 0.41	Nd 1.87	Pm 5.72	Sm 171.47	Eu 94.58	Gd 1479.04	Tb 0.93	Dy 32.42	Ho 2.25	Er 5.48	Tm 3.53	Yb 1.40	Lu 2.75			
**Actinides	Th 0.59	Pa 8.46	U 0.82	Np 9.80	Pu 50.20	Am 2.86	Cm	Bk	Cf	Es	Fm	Md	No	Lr neut.			

For neutrons = completely unsystematic!



...even for different isotopes of the same element!

X-rays and neutrons

Attenuation coefficients with X-ray [cm?¹]

For X-rays = proportional increase!

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 0.02																	He 0.02
Li 0.06	Be 0.22											B 0.28	C 0.27	N 0.11	O 0.16	F 0.14	Ne 0.17
Na 0.13	Mg 0.24											Al 0.38	Si 0.33	P 0.25	S 0.30	Cl 0.23	Ar 0.20
K 0.14	Ca 0.26	Sc 0.48	Ti 0.73	V 1.04	Cr 1.29	Mn 1.32	Fe 1.57	Co 1.78	Ni 1.96	Cu 1.97	Zn 1.64	Ga 1.42	Ge 1.33	As 1.50	Se 1.23	Br 0.90	Kr 0.73
Rb 0.47	Sr 0.86	Y 1.61	Zr 2.47	Nb 3.43	Mo 4.29	Tc 5.06	Ru 5.71	Rh 6.08	Pd 6.13	Ag 5.67	Cd 4.84	In 4.31	Sn 3.98	Sb 4.28	Te 4.06	I 3.45	Xe 2.53
Cs 1.42	Ba 2.73	La 5.04	Hf 19.70	Ta 25.47	W 30.49	Re 34.47	Os 37.92	Ir 39.01	Pt 38.61	Au 35.94	Hg 25.88	Tl 23.23	Pb 22.81	Bi 20.28	Po 20.22	At	Rn 9.77
Fr	Ra 11.80	Ac 24.47	Rf	Ha													

Lanthanides	Ce 5.79	Pr 6.23	Nd 6.46	Pm 7.33	Sm 7.68	Eu 5.66	Gd 8.69	Tb 9.46	Dy 10.17	Ho 10.91	Er 11.70	Tm 12.49	Yb 9.32	Lu 14.07
*Actinides	Th 28.95	Pa 39.65	U 49.08	Np	Pu	Am	Cm	Bk	Vf	Es	Fm	Md	No	Lr x-ray

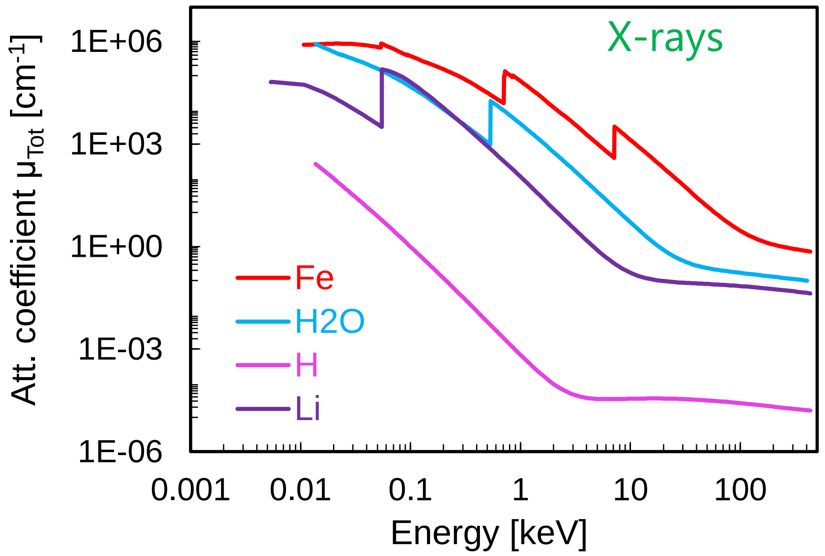
Attenuation coefficients with neutrons [cm?¹]

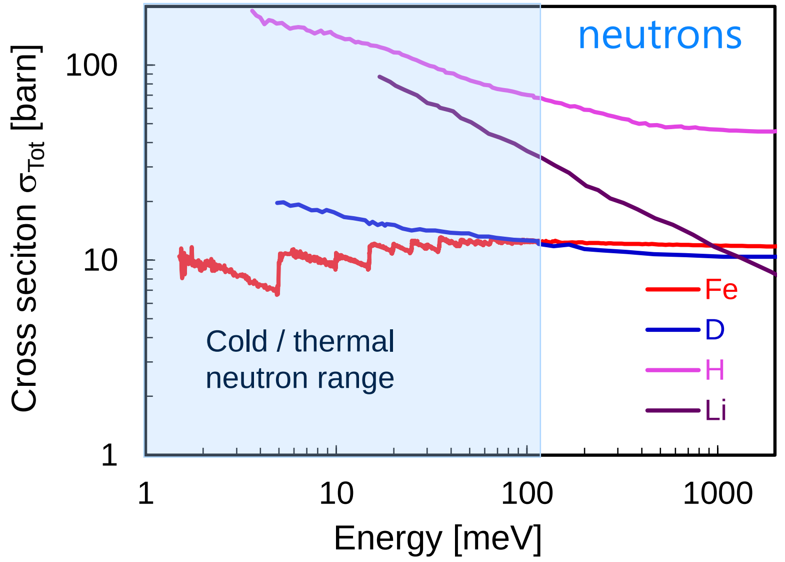
For neutrons = completely unsystematic!

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 3.44																	He 0.02
Li 3.30	Be 0.79											B 101.60	C 0.56	N 0.43	O 0.17	F 0.20	Ne 0.10
Na 0.09	Mg 0.15											Al 0.10	Si 0.11	P 0.12	S 0.06	Cl 1.33	Ar 0.03
K 0.06	Ca 0.08	Sc 2.00	Ti 0.60	V 0.72	Cr 0.54	Mn 1.21	Fe 1.19	Co 3.92	Ni 2.05	Cu 1.07	Zn 0.35	Ga 0.49	Ge 0.47	As 0.67	Se 0.73	Br 0.24	Kr 0.61
Rb 0.08	Sr 0.14	Y 0.27	Zr 0.29	Nb 0.40	Mo 0.52	Tc 1.76	Ru 0.58	Rh 10.88	Pd 0.78	Ag 4.04	Cd 115.11	In 7.58	Sn 0.21	Sb 0.30	Te 0.25	I 0.23	Xe 0.43
Cs 0.29	Ba 0.07	La 0.52	Hf 4.99	Ta 1.49	W 1.47	Re 6.85	Os 2.24	Ir 30.46	Pt 1.46	Au 6.23	Hg 16.21	Tl 0.47	Pb 0.38	Bi 0.27	Po	At	Rn
Fr	Ra 0.34	Ac	Rf	Ha													

*Lanthanides	Ce 0.14	Pr 0.41	Nd 1.87	Pm 5.72	Sm 171.47	Eu 94.58	Gd 1479.04	Tb 0.93	Dy 32.42	Ho 2.25	Er 5.48	Tm 3.53	Yb 1.40	Lu 2.75
**Actinides	Th 0.59	Pa 8.46	U 0.82	Np 9.80	Pu 50.20	Am 2.86	Cm	Bk	Cf	Es	Fm	Md	No	Lr neut.

The cross section (& attenuation) is energy (wavelength) dependent for most materials!





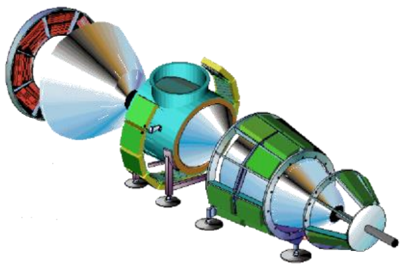
$$\sigma_{\mathrm{tot}}(\lambda) = \sigma_{\mathrm{coh}}(\lambda) + \sigma_{\mathrm{incoh}}(\lambda) + \sigma_{\mathrm{abs}}(\lambda)$$

Non-destructive testing with neutrons: Engineering materials and components revealed

- ❑ Characterization Techniques & Contrast Mechanisms
- ❑ Neutron Methods & Length Scales
- ❑ Applications using Imaging (Attenuation Contrast)
- ❑ Applications using SANS and Diffraction
- ❑ Applications using Imaging (Diffraction and Scattering Contrast)
- ❑ Summary & Outlook

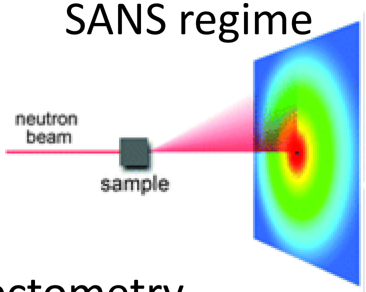
Neutron Methods & Length Scales

- ‘Contrast’ due to small spatial features
- Usually averaged over several mm^3

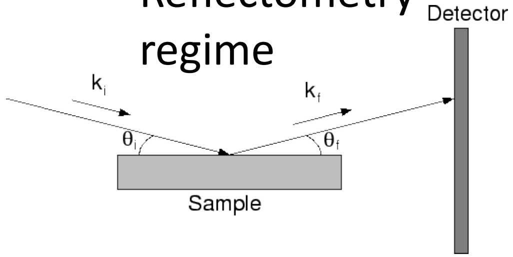


Diffraction regime

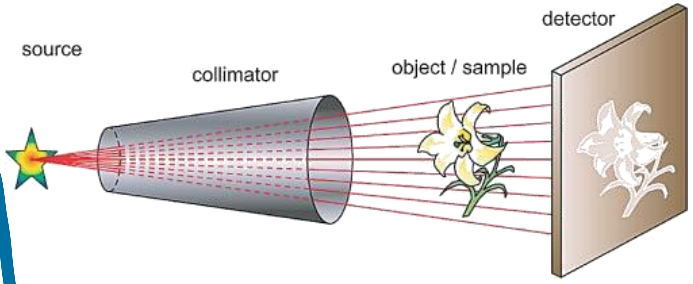
SANS regime



Reflectometry regime

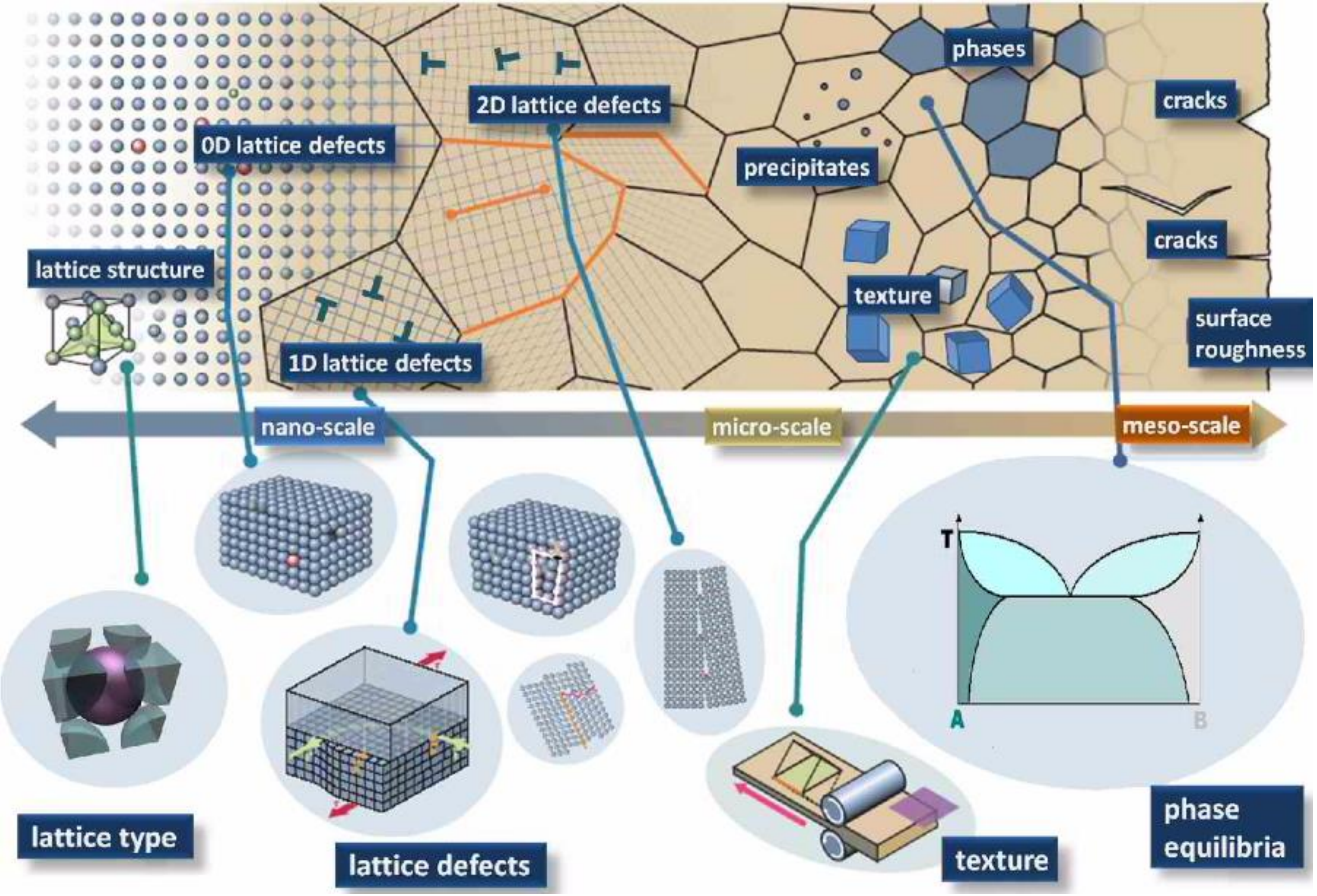


Imaging regime



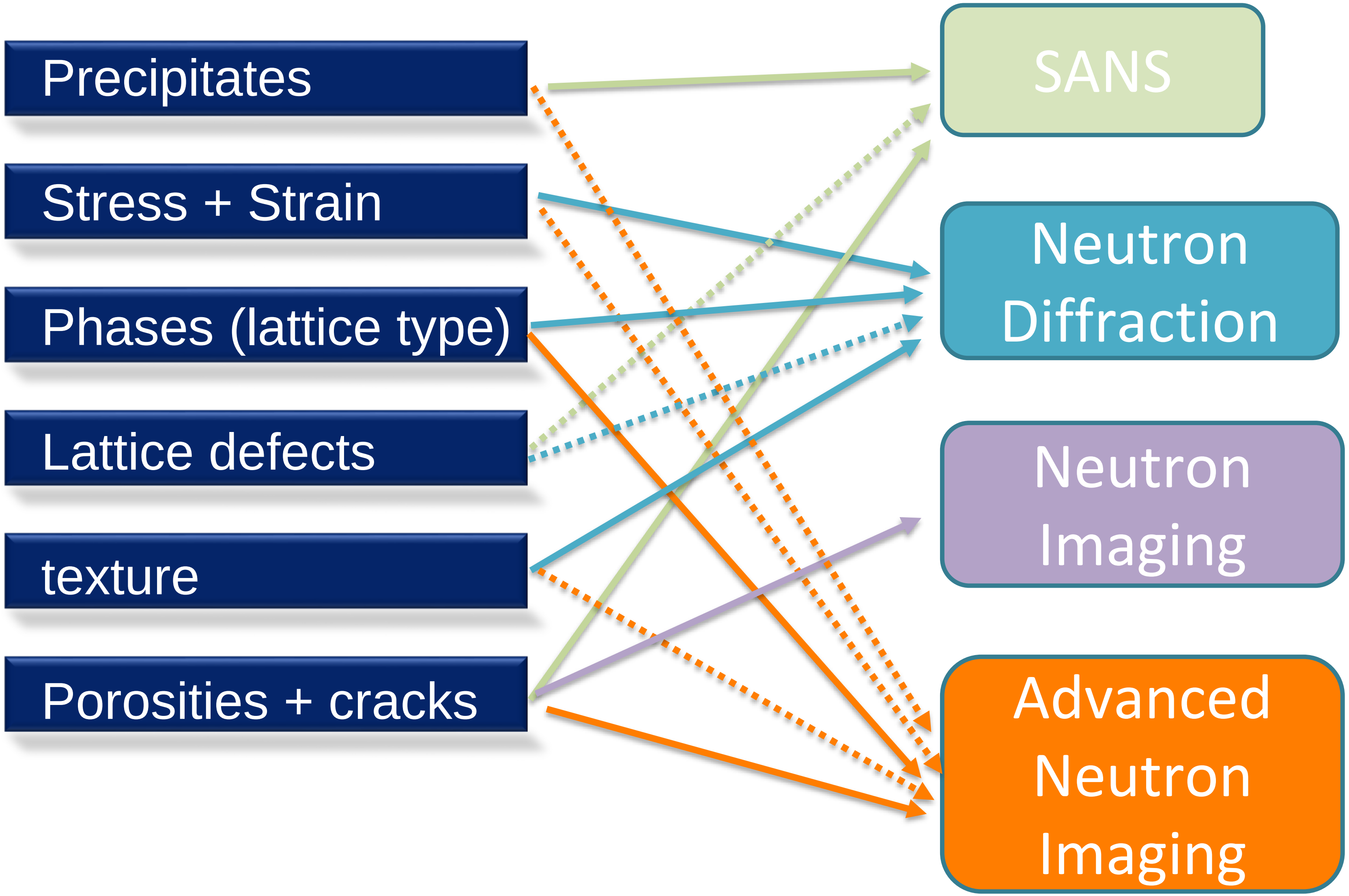
- ‘Contrast’ from larger features resolved in real space with spatial resolution

Neutron Methods & Length Scales



Source: http://www.dierk-raabe.com/

Neutron Methods & Length Scales



Non-destructive testing with neutrons: Engineering materials and components revealed

- ❑ Characterization Techniques & Contrast Mechanisms
- ❑ Neutron Methods & Length Scales
- ❑ Applications using Imaging (Attenuation Contrast)
- ❑ Applications using SANS and Diffraction
- ❑ Applications using Imaging (Diffraction and Scattering Contrast)
- ❑ Summary & Outlook

Neutron Methods & Length Scales

Neutron Imaging

Reflectometry

SANS

Diffraction

Imaging regime

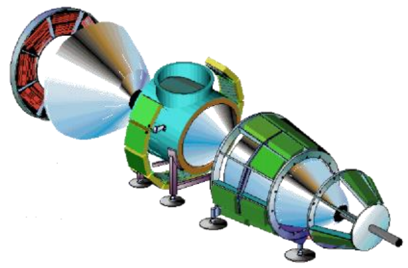
Attenuation

Applications

Hydrogen

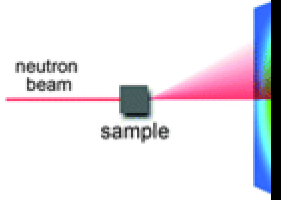
Metals/ Engineering

- 'Contrast' due to small spatial features
- Usually averaged over several mm³

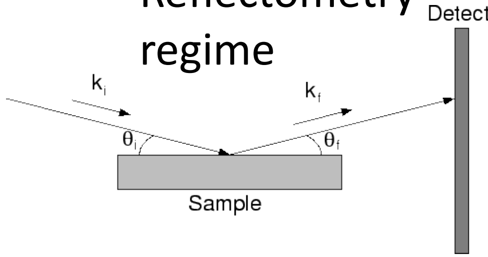


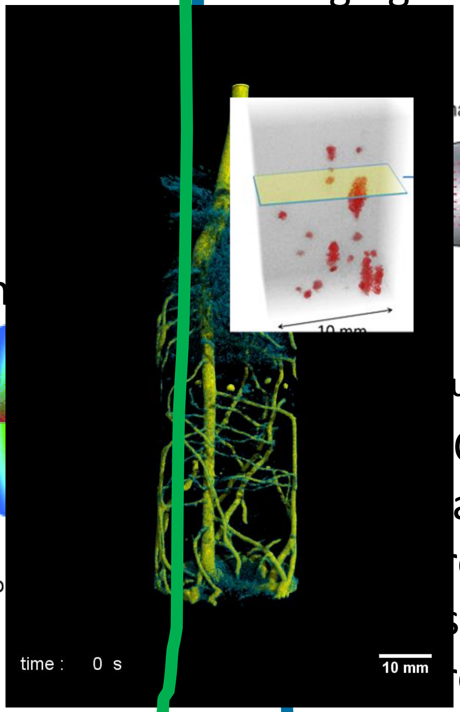
Diffraction regime

SANS regime

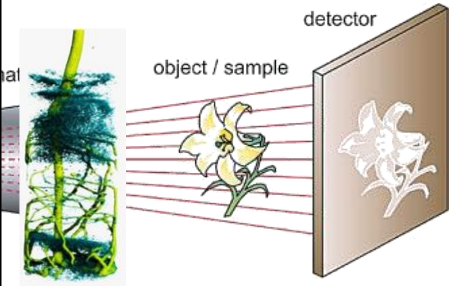


Reflectometry regime





Imaging regime



Water uptake in plants

'Contrast' from larger features resolved in real space with spatial resolution

Å nm µm mm cm

Griesche et al., Acta Materialia 78 (2014)
Tötzke, et al. Scientific Reports, 7(1) (2017)

Neutron Methods & Length Scales

Neutron Imaging

Imaging regime

Attenuation

Applications

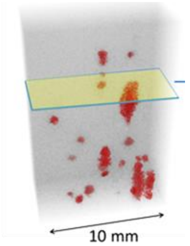
Metals/ Engineering

Hydrogen

Energy Storage

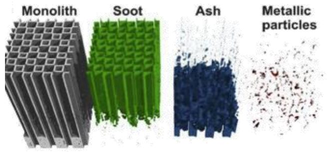
Cultural Heritage

Hydrogen in metals



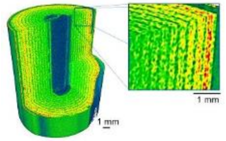
Water uptake in plants

Device inspection: particle filters

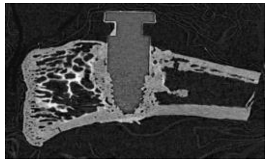


Grünzweig et al., MTZ worldwide 73.4 (2012)

Le Cann et al., Journal of the Mechanical Behavior of Biomedical Materials 75 (2017)



Li transport in batteries



Bone Structures + implants

S Journal of Power Sources

2Griesche et al., Acta Materialia 78 (2014)

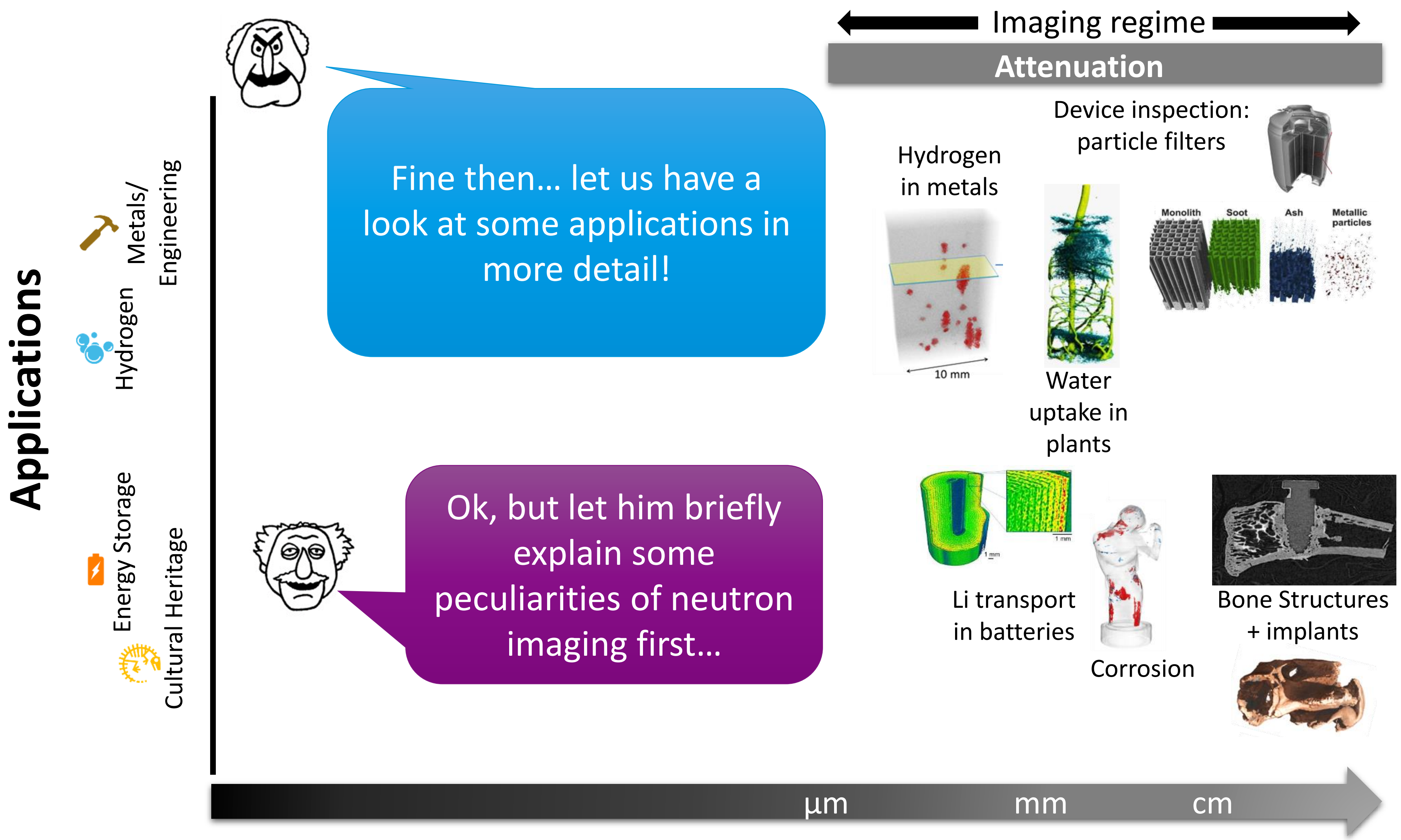
Masalles et al., Physics Procedia 69 (2015)

µm mm cm

Neutron Methods & Length Scales

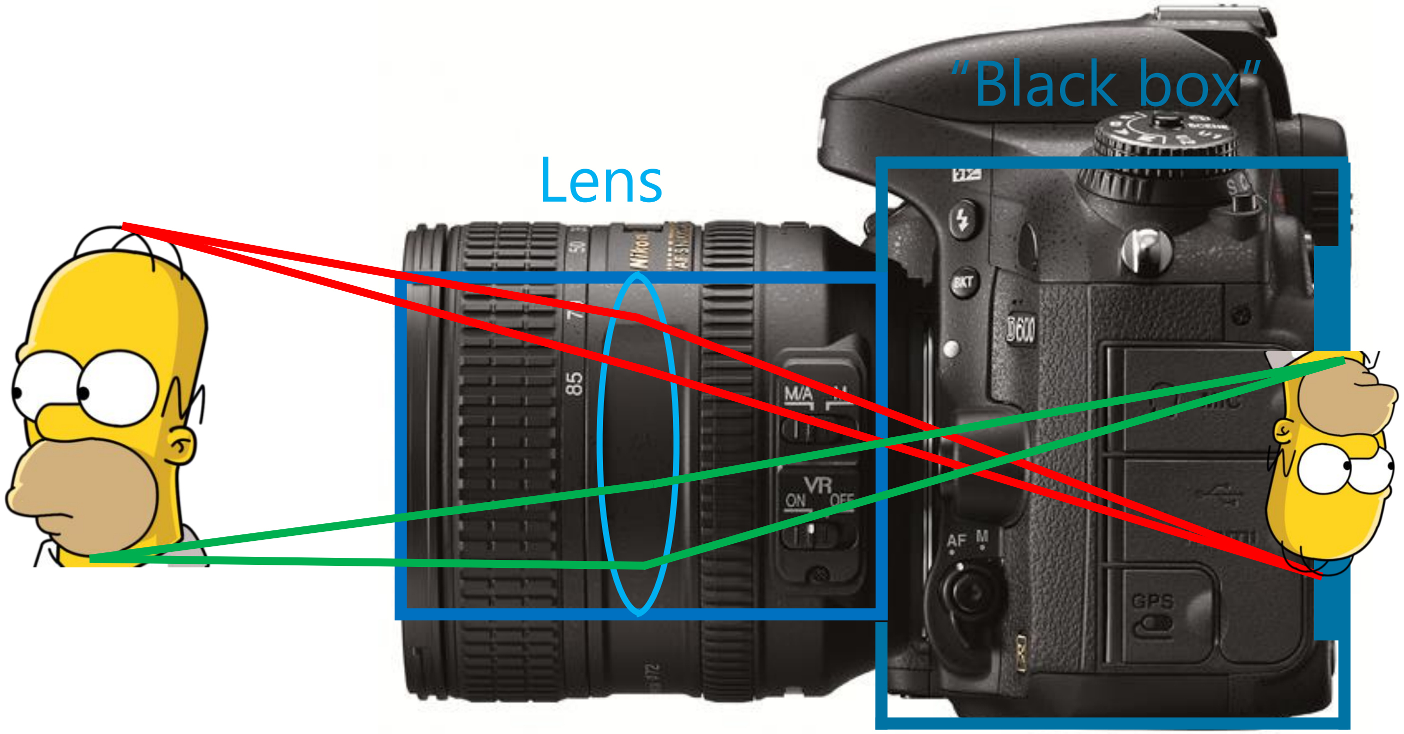
Neutron Imaging

Kardjilov, Manke, Woracek, Banhart,. Advances in neutron imaging. Materials Today 21 (2018)



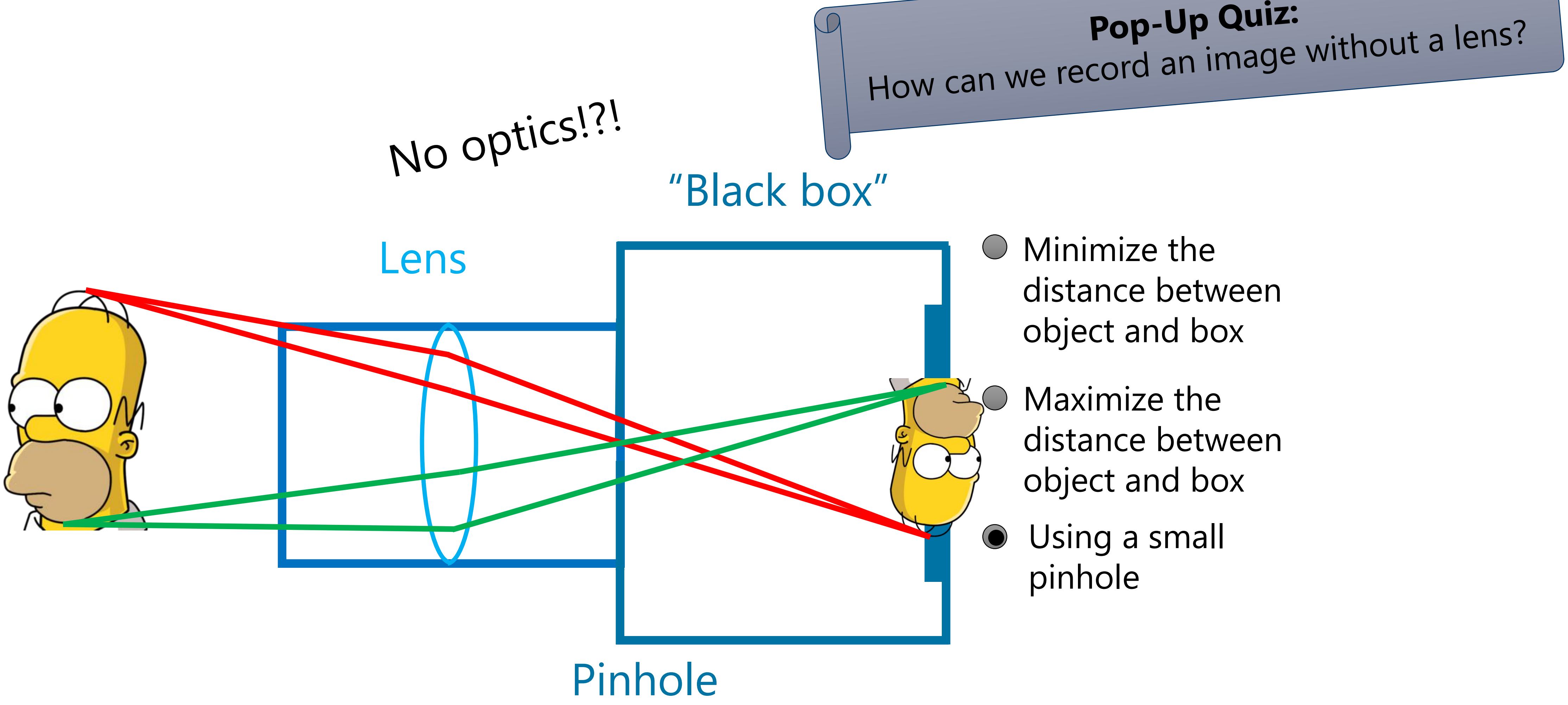
Neutron Imaging

How is an image recorded?



Neutron Imaging

How is an image recorded?

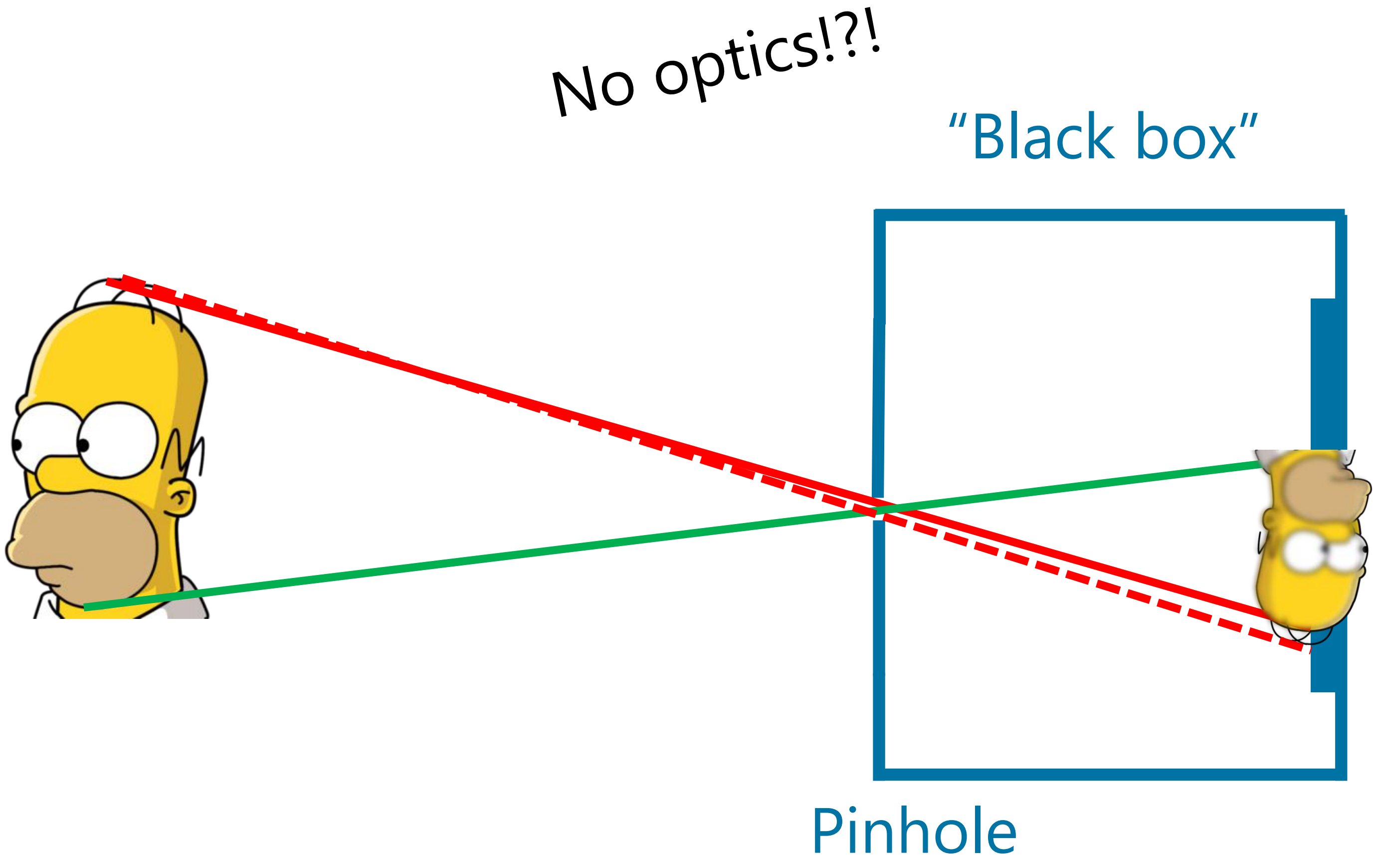


- Minimize the distance between object and box
- Maximize the distance between object and box
- Using a small pinhole

Neutron Imaging

How is an image recorded?

Transmission



Neutron Imaging

How is a Transmission image recorded?

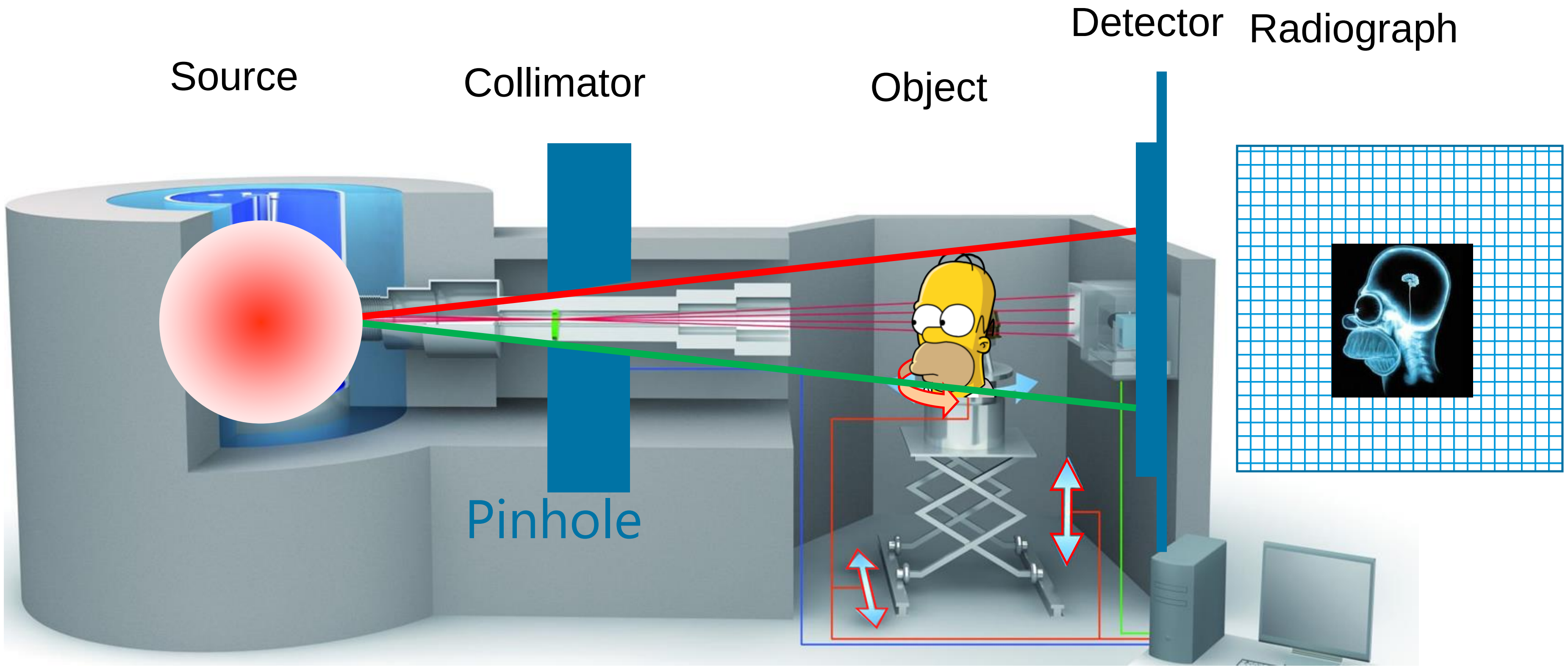
Incident beam

Transmitted beam

Radiograph

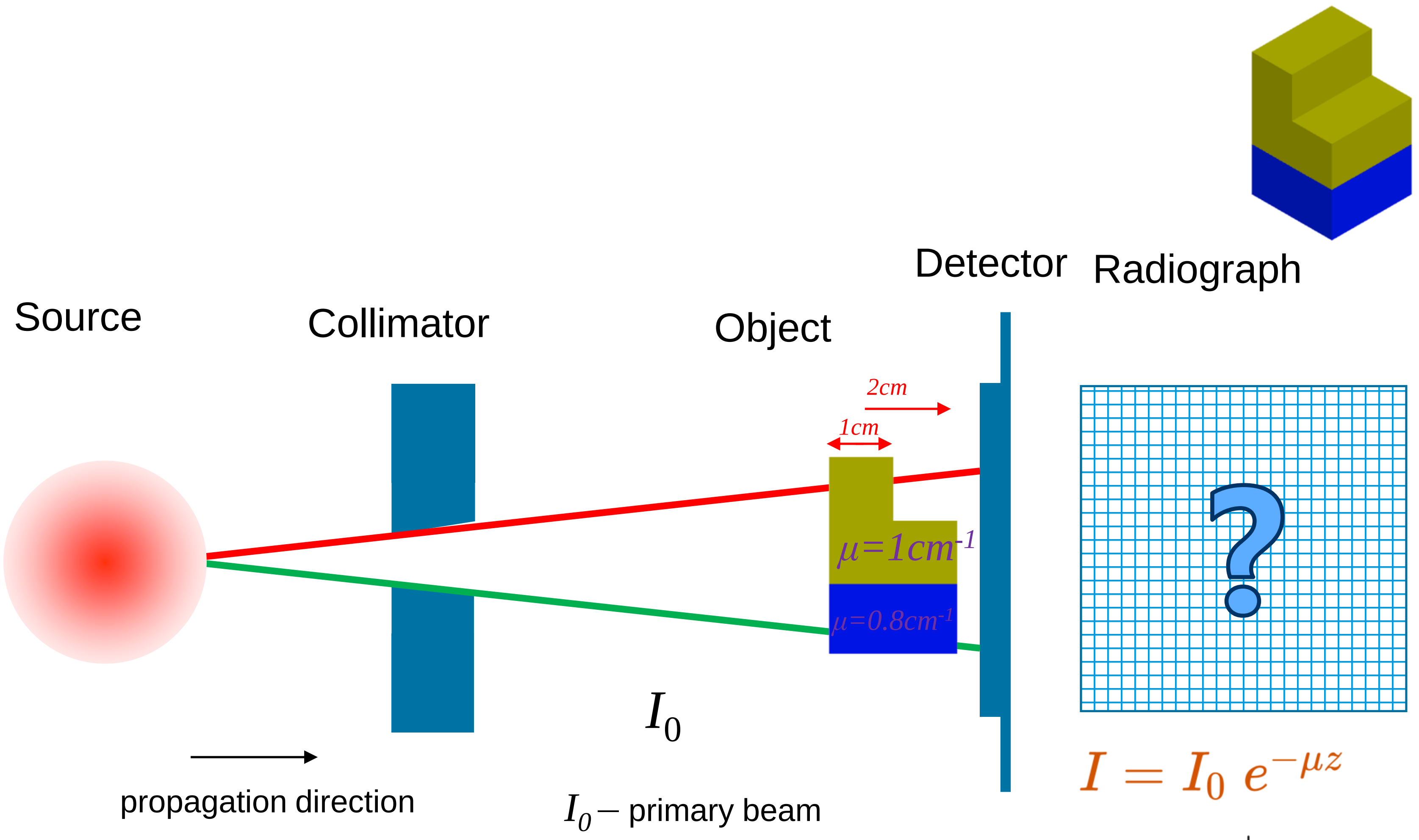
Neutron Imaging

The neutron imaging setup



Neutron Imaging

Neutron attenuation



$$I = I_0 \, e^{-\mu z}$$

$$\mu = \mu_{\mathrm{abs}} + \mu_{\mathrm{scatt}}$$

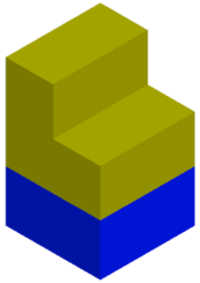
attenuation coefficient (cm^{-1})

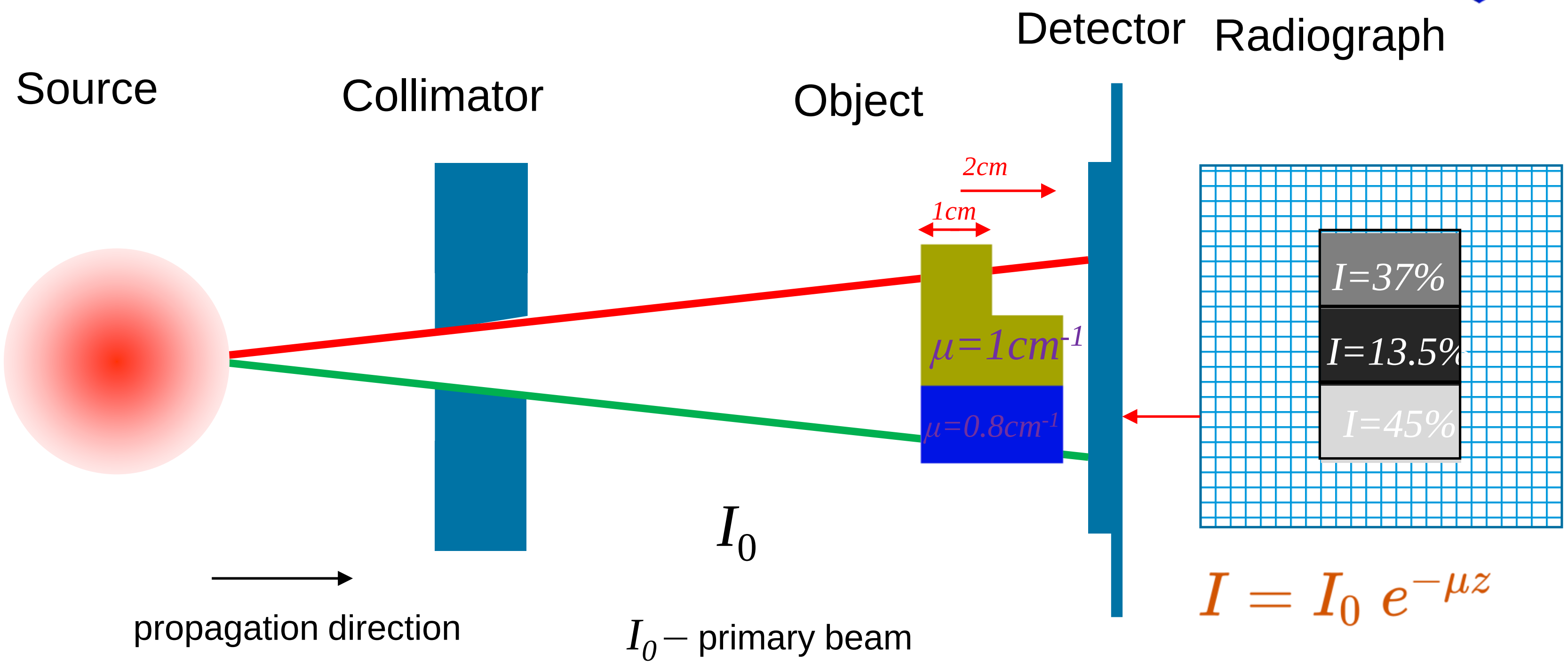
sample thickness (cm)

Neutron Imaging

Neutron attenuation

Fine... show us some more applications then...





$$I = I_0\, e^{-\mu z}$$

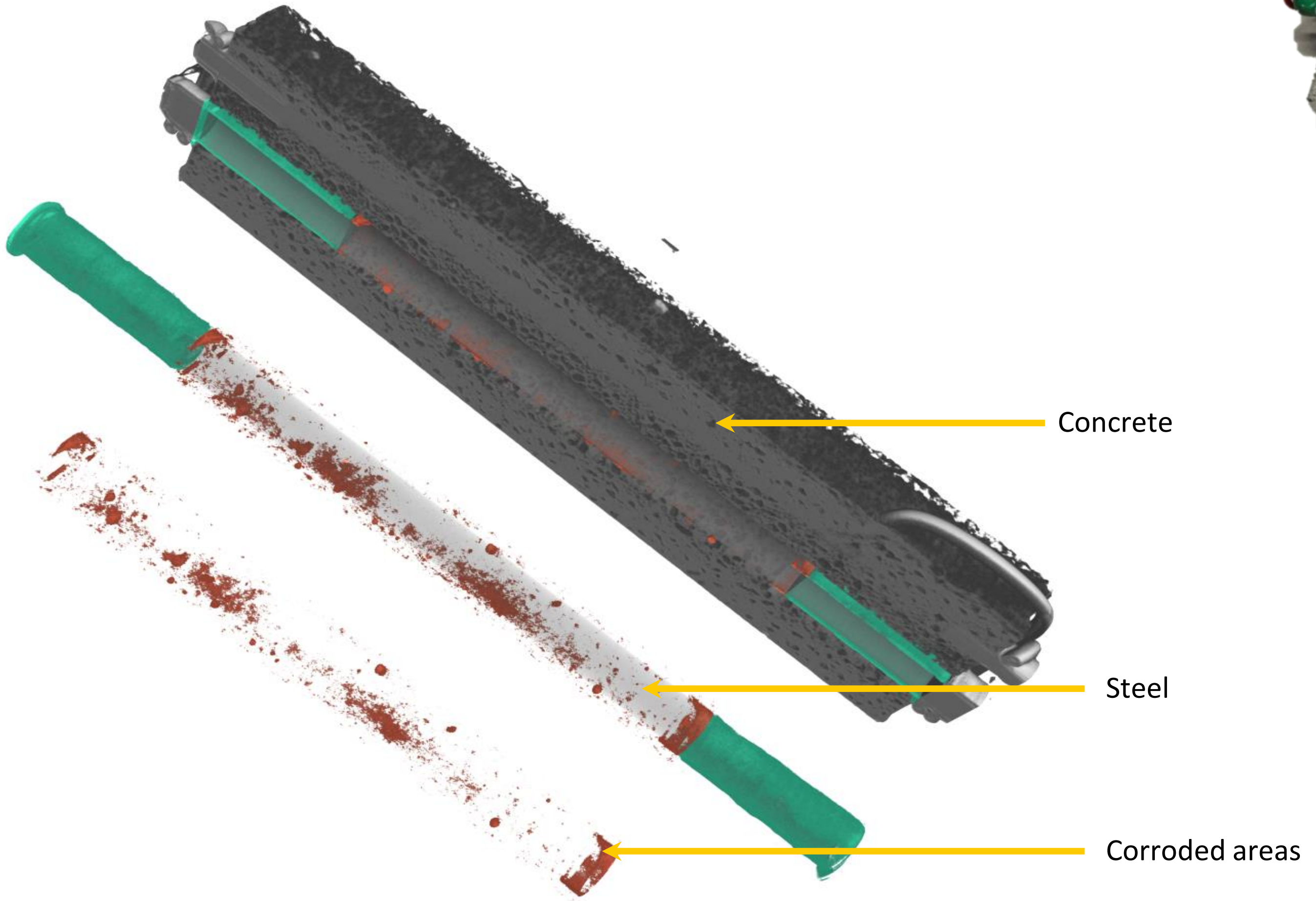
$$\mu = \mu_{\mathrm{abs}} + \mu_{\mathrm{scatt}}$$

attenuation coefficient (cm^{-1})

sample thickness (cm)

Applications: Civil Engineering

Corrosion of steel inside concrete

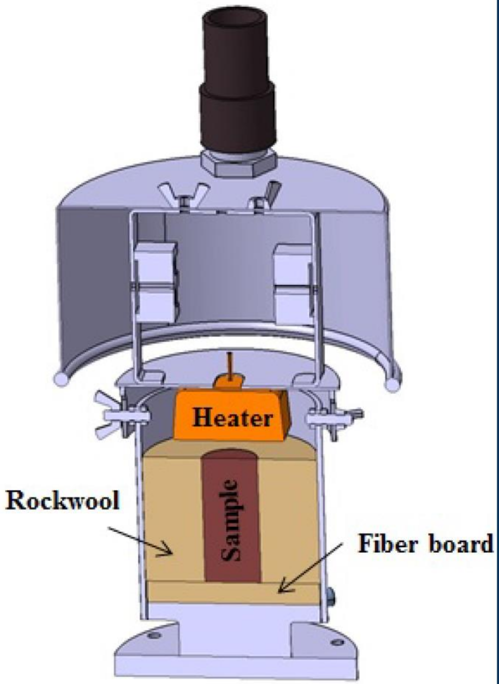


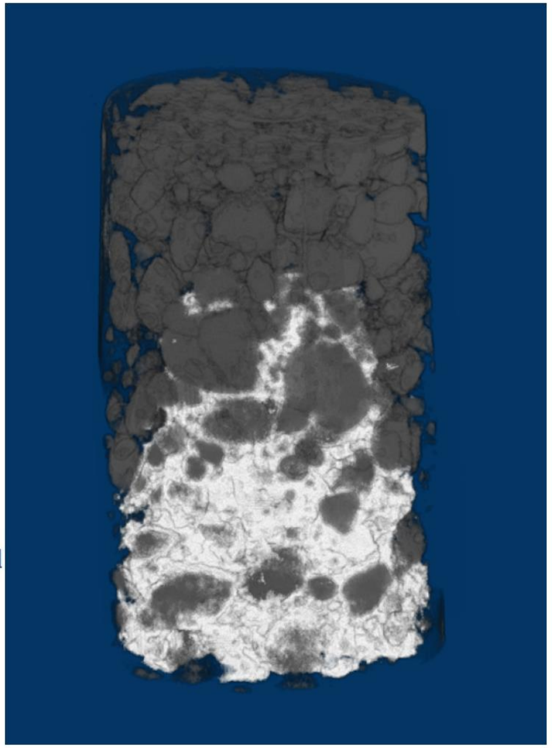
Visualized 3D-evaluation of Neutron-CT

Courtesy E. Lehmann (PSI; collaboration with P. Chang, Y. Wang, China)

Applications: Civil Engineering

Moisture migration in concrete at high temperature



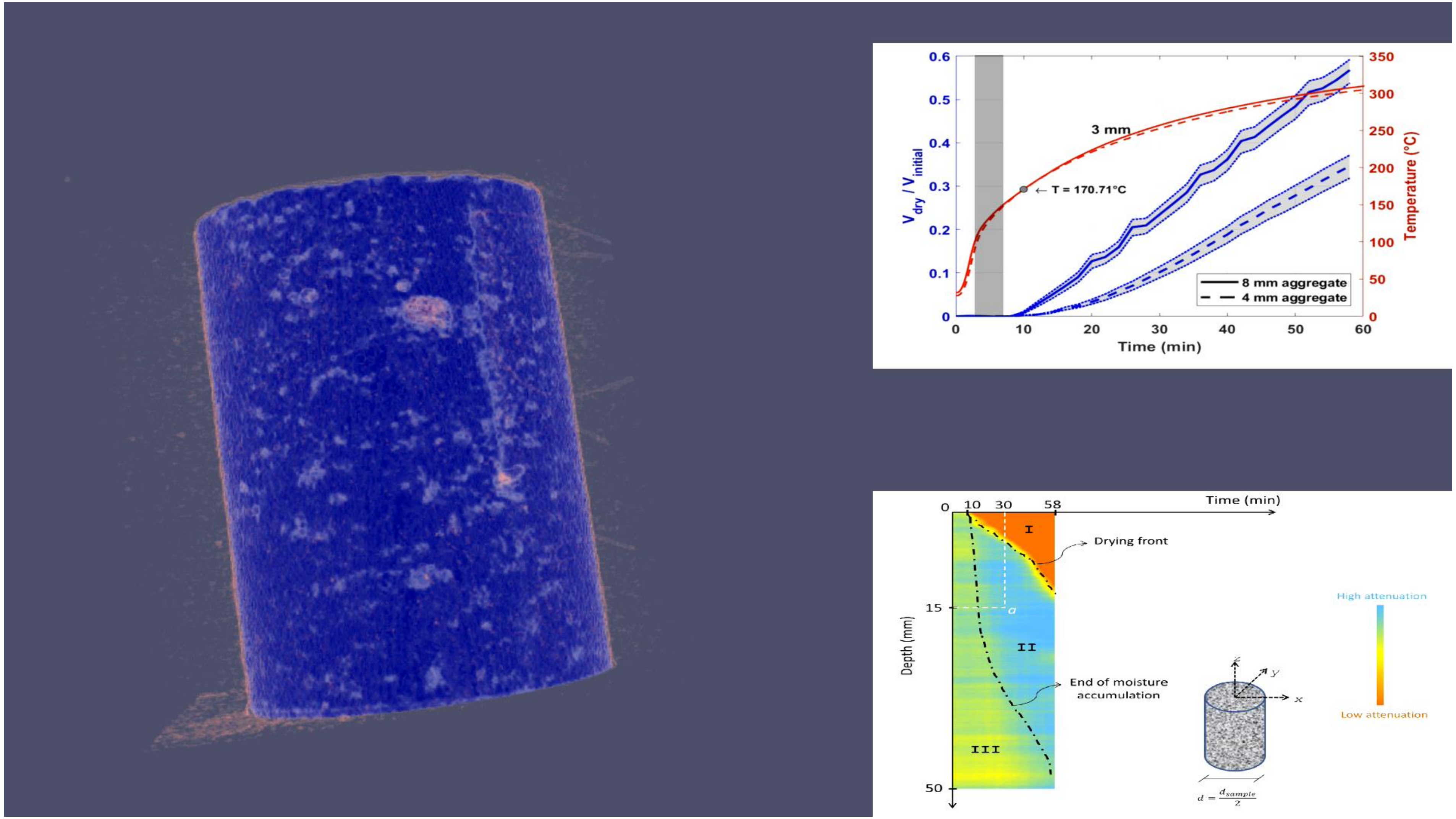


Courtesy A. Tengattinia (ILL)

Dauti et al., Cement and Concrete Research 111 (2018)

Applications: Civil Engineering

Moisture migration in concrete at high temperature



Courtesy A. Tengattinia (ILL)

Dauti et al., Cement and Concrete Research 111 (2018)

Applications: Automotive

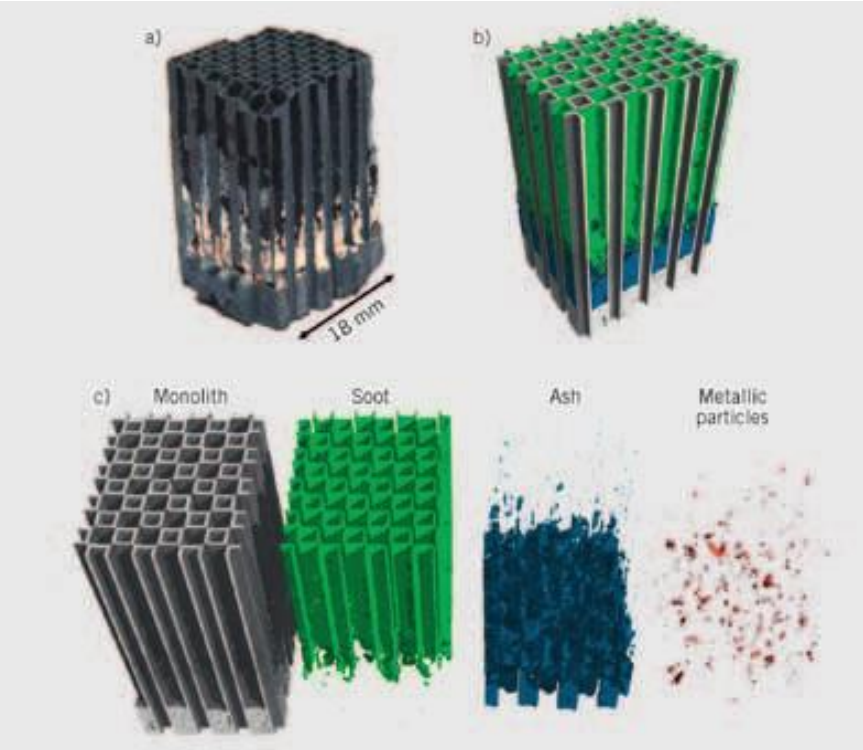
Diesel particulate filters

AUTHORS

DR. DIPL.-PHYS.
CHRISTIAN GRÜNZWEIG

VISUALISING THE SOOT AND ASH DISTRIBUTION IN DIESEL PARTICULATE FILTERS USING NEUTRON IMAGING

Neutron tomography is presently the only possibility to obtain information about



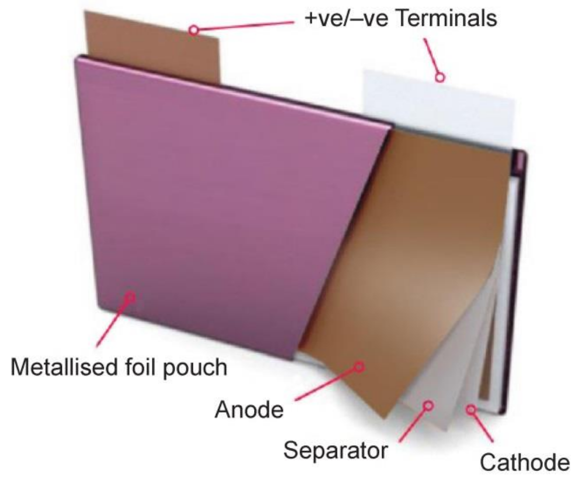
Courtesy C. Gruenzweig (PSI)

Applications: Energy

In-situ filling of Li-ion Pouch Batteries

Pouch Batteries

- High potential for electro mobility and stationary energy storage
- Electrolyte filling is a key process in cell production
- So far only limited knowledge about the process
- Phenomenological: pressure cycles to optimize wetting with electrolyte



Why Neutron Imaging?

- Cell housing optically intransparent
- Other approaches not successful
- Neutrons offer high contrast due to H-content in electrolyte

Goals

- In-situ visualization of the wetting process
- Study and optimize influence of process parameters

Courtesy M . Schulz (TUM) T. Knoche, V. Zinth, M. Schulz, J. Schnell, R. Gilles, G. Reinhart, J. Power Sources 331, (2016) 267

Applications: Energy

In-situ filling of Li-ion Pouch Batteries

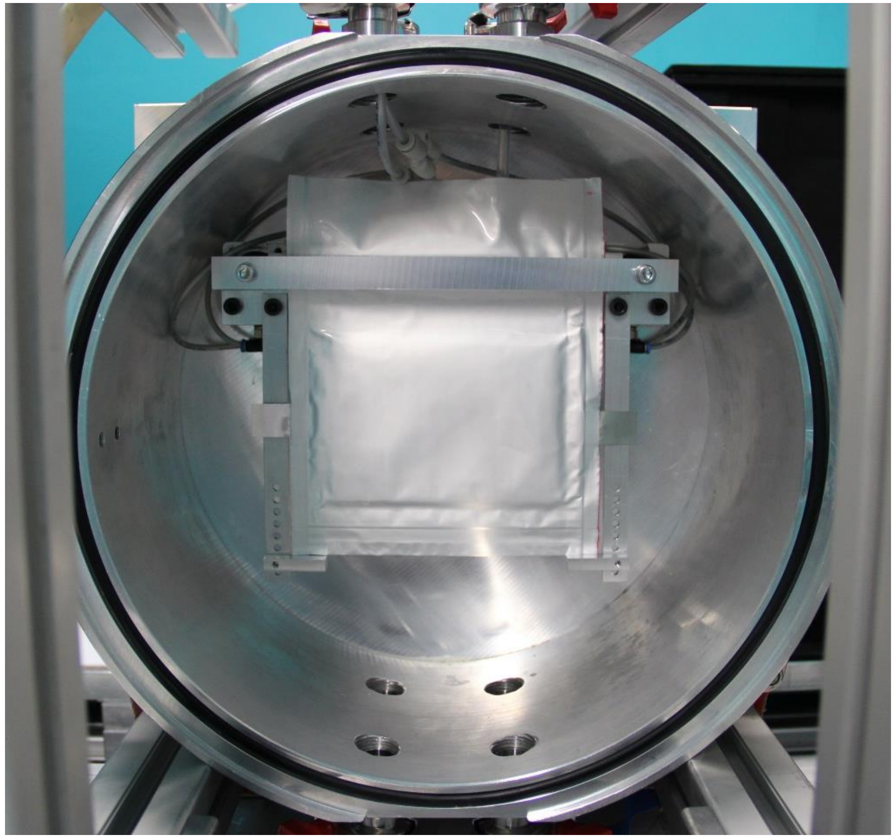
MLZ Heinz Maier-Leibnitz Zentrum
FRM II Forschungs-Neutronenquelle Heinz Maier-Leibnitz
iwb Institut für Werkzeugmaschinen und Betriebswissenschaften
ess

Setup with cell

Materials

Cell
- 5 Anodes,
- 4 Cathodes,
- z-folded
- ExZellTUM-format

Elektrolyte
- EC:EMC 3:7
- No $LiPF_6$,
- No VC



Courtesy M . Schulz (TUM)

T. Knoche, V. Zinth, M. Schulz, J. Schnell, R. Gilles, G. Reinhart, J. Power Sources 331, (2016) 267

Applications: Energy

MLZ Heinz Maier-Leibnitz Zentrum
FRM II Forschungs-Neutronenquelle Heinz Maier-Leibnitz
iwb Institut für Werkzeugmaschinen und Betriebswissenschaften
ess

In-situ filling of Li-ion Pouch Batteries

Negative example of filling process

Courtesy M . Schulz (TUM)

T. Knoche, V. Zinth, M. Schulz, J. Schnell, R. Gilles, G. Reinhart, J. Power Sources 331, (2016) 267

Applications: Energy

Institut für Werkzeugmaschinen
und Betriebswissenschaften

In-situ filling of Li-ion Pouch Batteries

Positive example of filling process

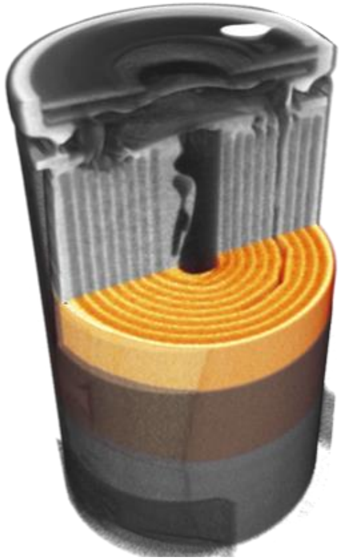
Courtesy M . Schulz (TUM)

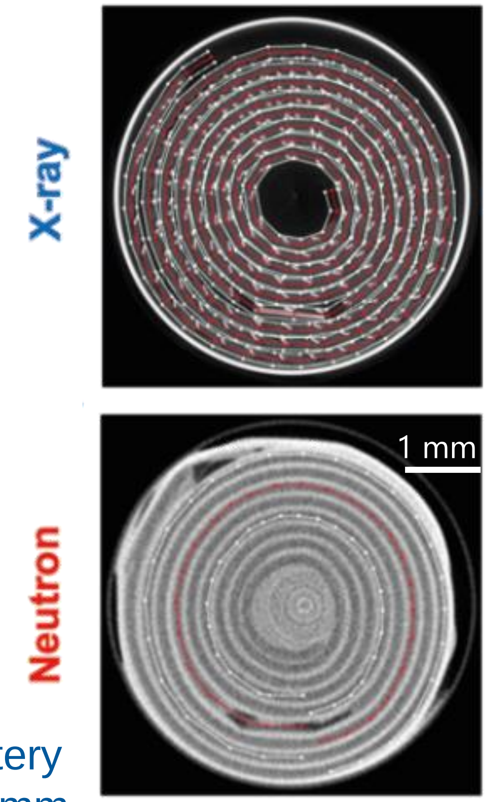
T. Knoche, V. Zinth, M. Schulz, J. Schnell, R. Gilles, G. Reinhart, J. Power Sources 331, (2016) 267

Applications: Energy

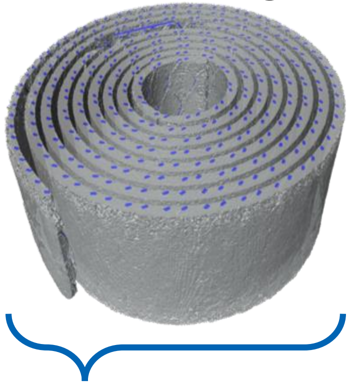
Combined X-ray and neutron imaging of a Li-Ion battery

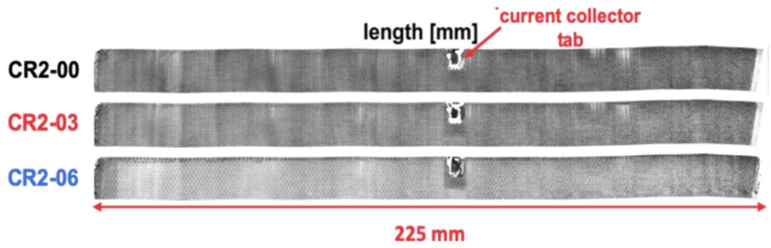
How to characterize lithium diffusion in batteries?





Virtual Unrolling





3D reconstruction of CR2 battery (Li_xMnO_2) with diameter of 26 mm.
(neutron tomography: pixel size: 13 µm, 600 projections /360°, time: 8 h)

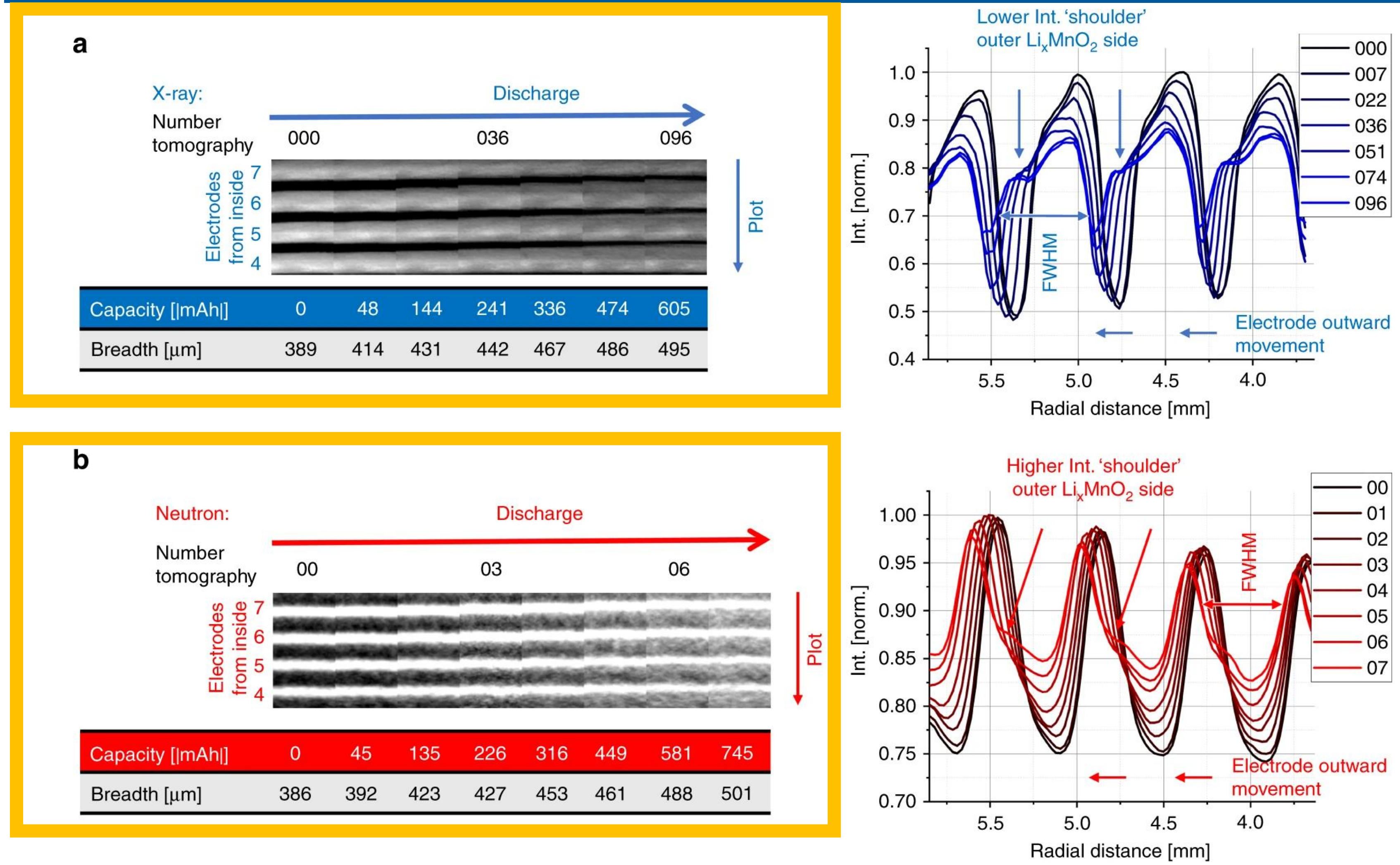
- ➔ **Analysis of the dual-mode tomography data**
- ➡ Temporally and spatially resolved tracking of lithium intercalation.

R. Ziesche *et al*., Nature Communications 11, **(2020)**

Applications: Energy

Combined X-ray and neutron imaging of a Li-Ion battery

How to characterize lithium diffusion in batteries?



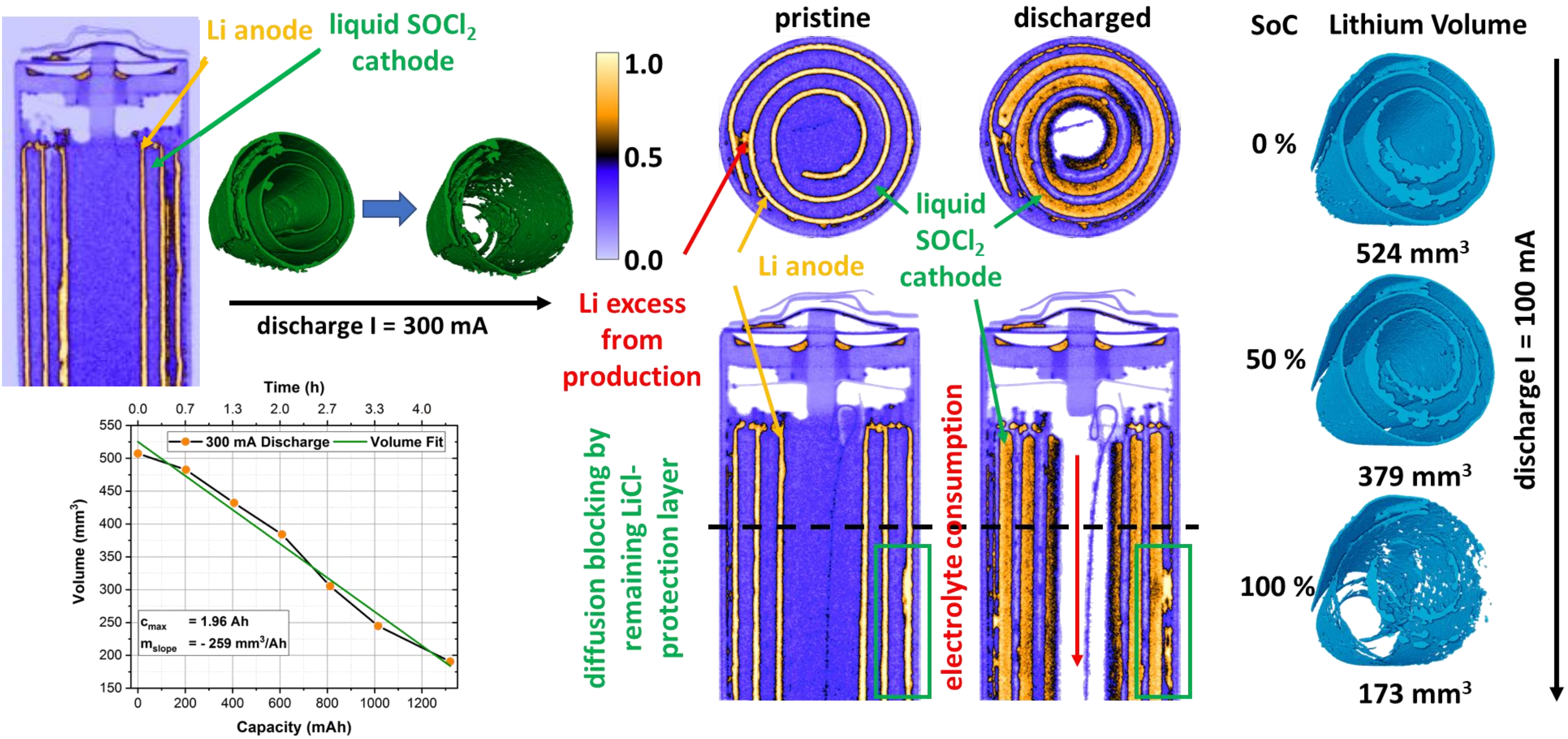
R. Ziesche *et al*., Nature Communications 11, **(2020)**

Applications: Energy

Neutron Imaging: Attenuation Contrast

How to characterize lithium diffusion in batteries?

4D Study of $SOCl_2$ Battery **(pixel size: 8 µm, time step: 7.5 min)**

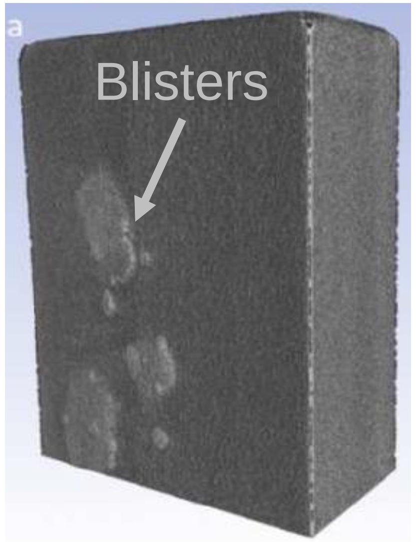


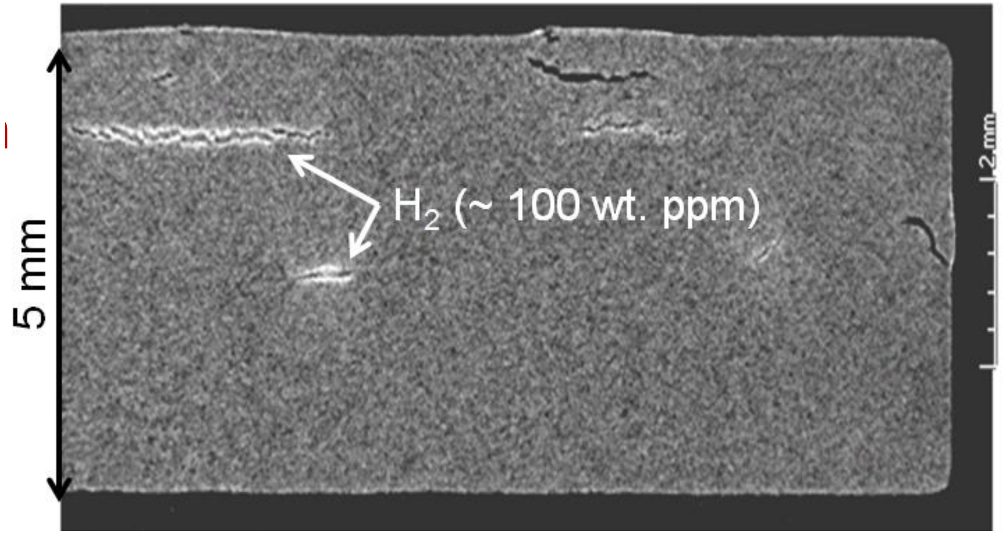
R. Ziesche *et al*., Journal of The Electrochemical Society 167, **(2020)**

Applications: Metallography

Hydrogen blistering + embrittlement in metals

Hydrogen charged ARMCO (technical iron) sample (Electrochemically loaded)





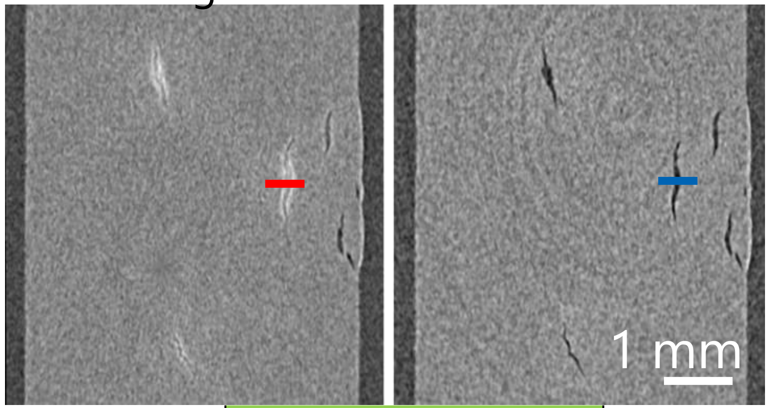
Resolution: 15 µm (pixel size: 6.5 µm), FOV: 13 x 13 mm^2, 600 proj. x 60 s

A. Griesche et al., Acta Materialia 78 **(2014)**

Applications: Metallography

Hydrogen blistering + embrittlement in metals

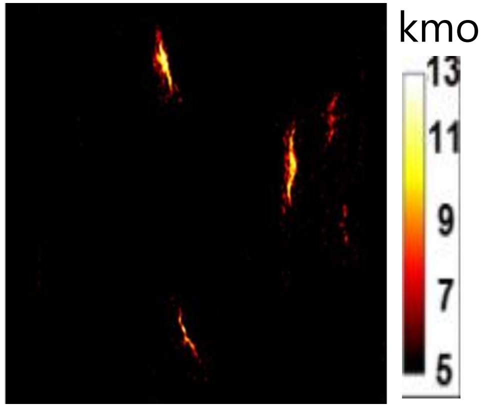
Charged Annealed



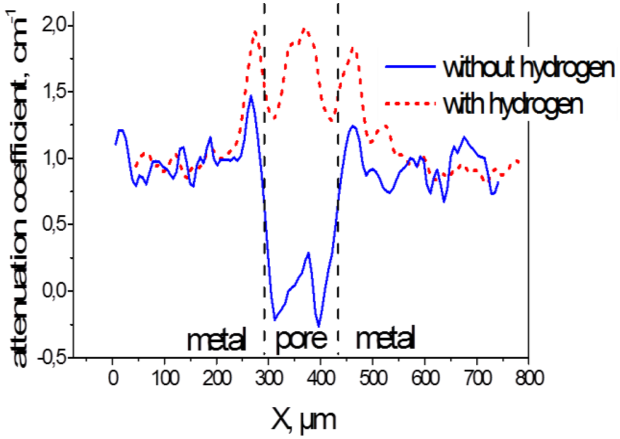
Volume registration

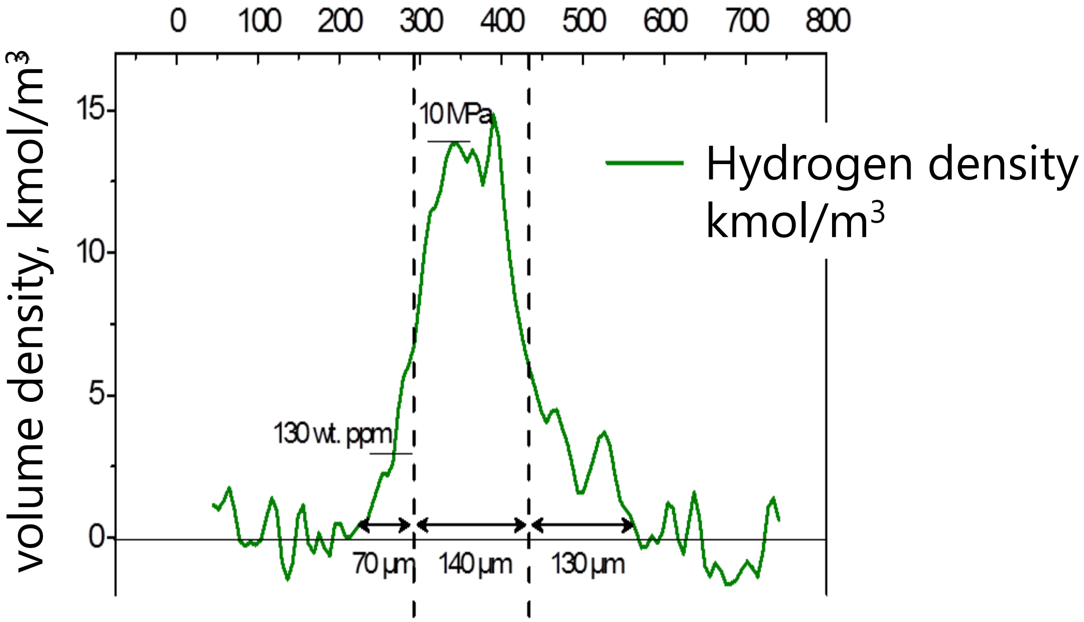
Quantification

kmol/m^3



$$\rho(mol/m^3) = \frac{\Sigma * at.wt}{M\sigma_{H,total}\,0.6023}$$

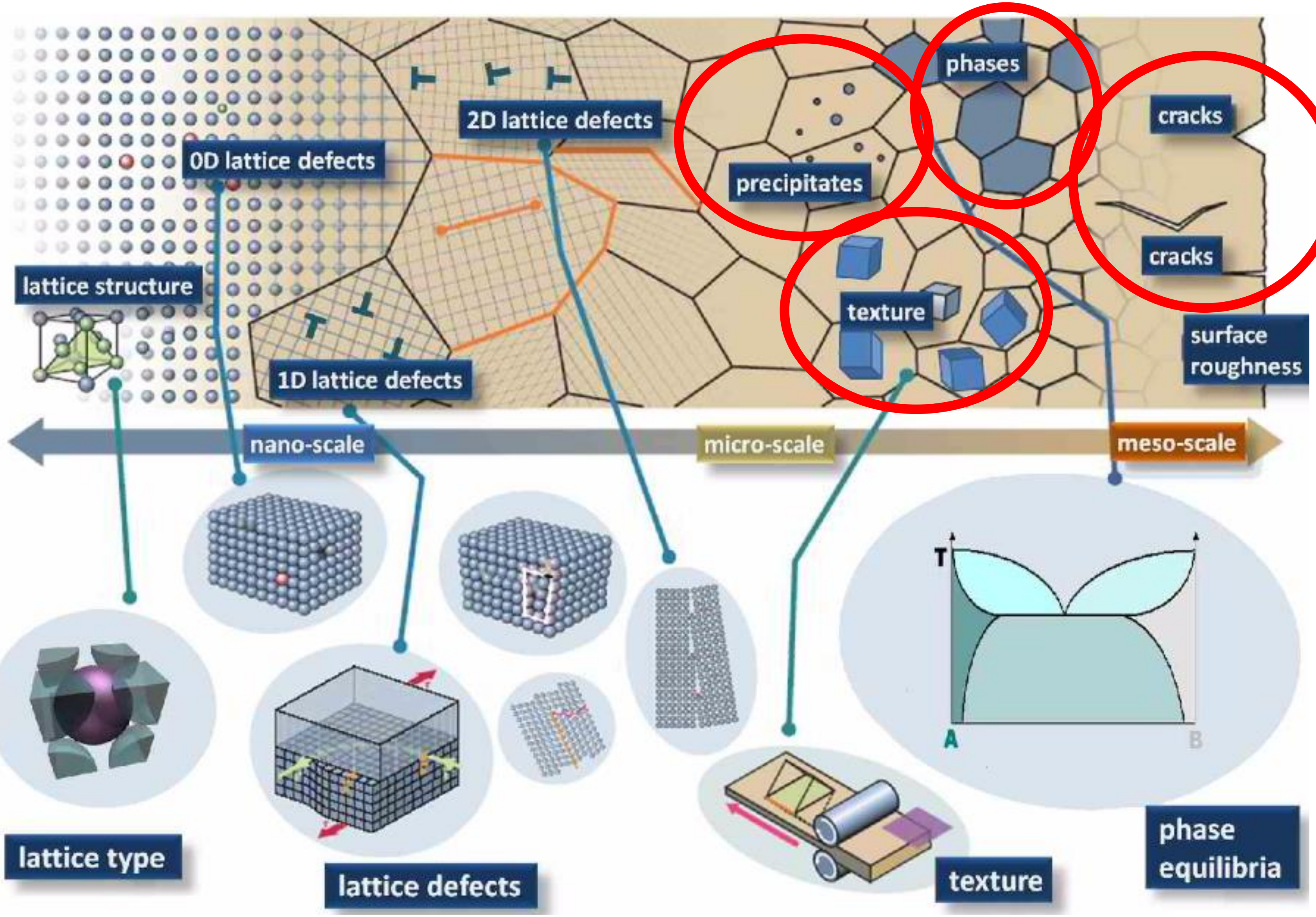




A. Griesche et al., Acta Materialia 78 **(2014)**

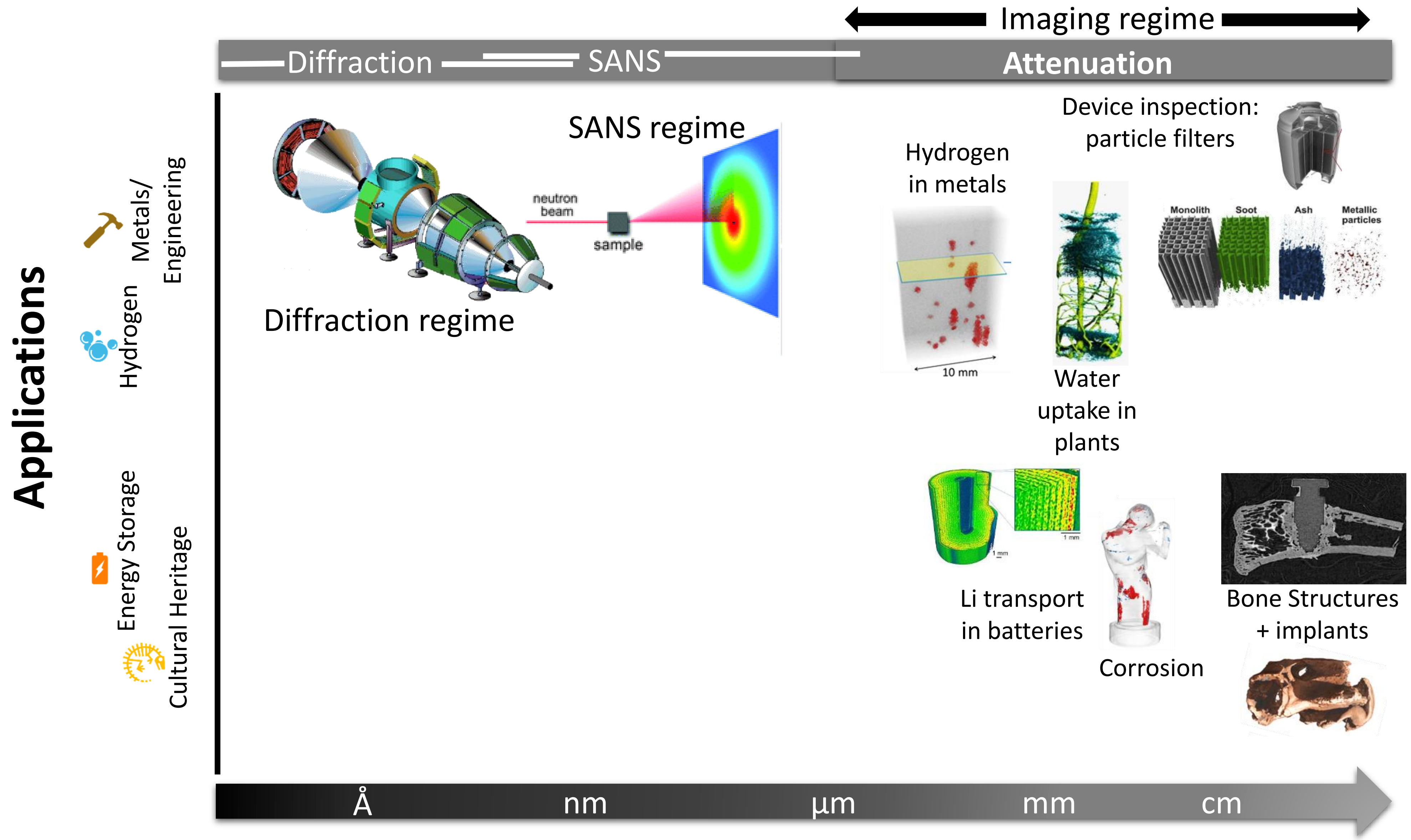
Applications: Metallography

Crystalline materials and properties



Source: http://www.dierk-raabe.com/

Neutron Methods & Length Scales



Non-destructive testing with neutrons: Engineering materials and components revealed

Applications: Metallography

Phase Separation in Fe-Cr

TEM

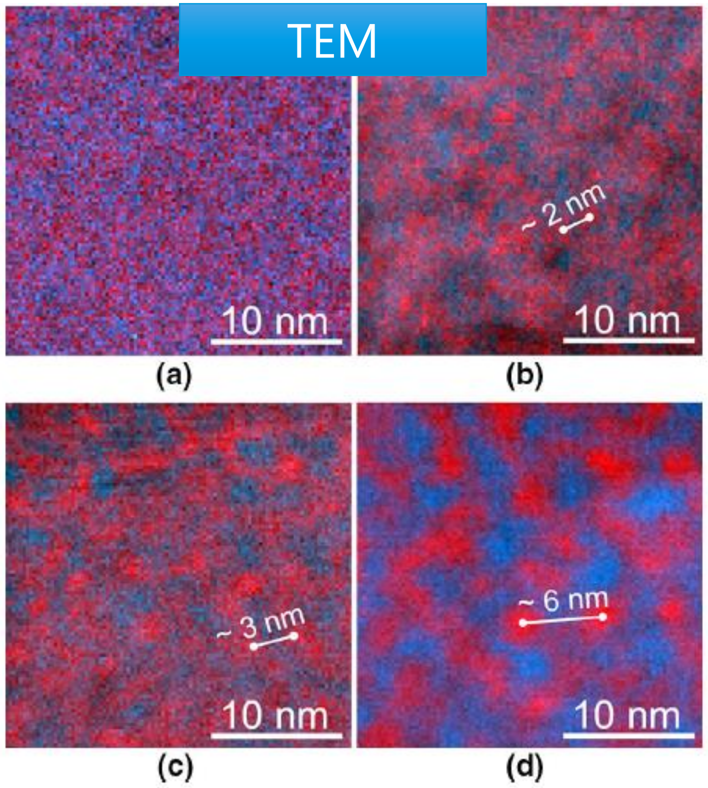
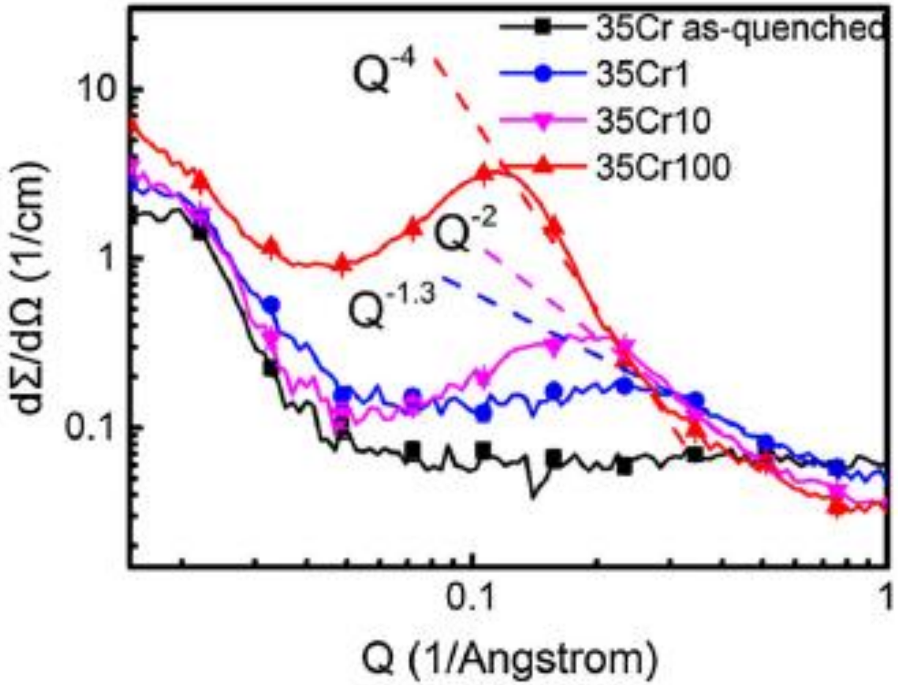


Fig. 5—Analytical TEM composite elemental maps (EELS) of multi-linear least squares (MLLS) fitting coefficients for the Cr-signal (red) and Fe-signal (blue) for alloy 35Cr aged at 773 K (500 °C) for different times[17]: (*a*) 0 h, (*b*) 1 h, (*c*) 10 h, and (*d*) 100 h. The estimated wavelength is schematically marked on the figures (Color figure online).

SANS



-> Fit model: derive the particle size values

-> SANS enables in-situ studies

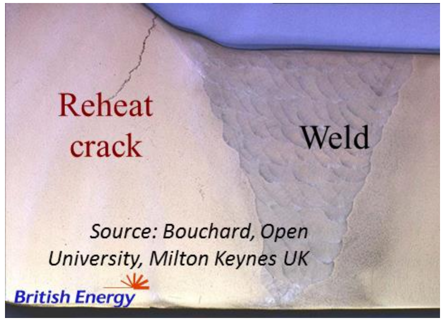
Xu, Xin, et al. Metallurgical and Materials Transactions A 47.12 (2016): 5942-5952.

Applications: Metallography

Stress/Strain analysis by Diffraction

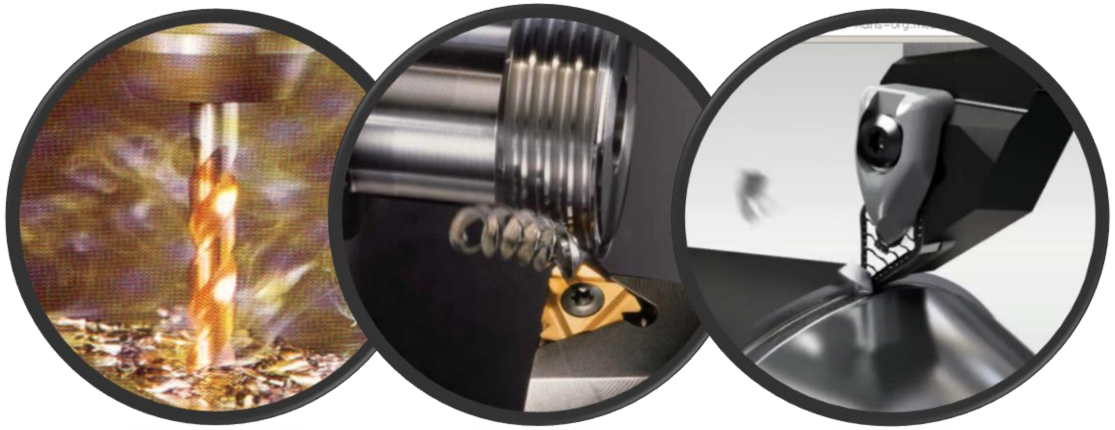
Residual Stress: Introduced during manufacture and/or during use by e.g. mechanical forming processes, welding and heat treatments. Residual stresses are present in virtually every solid material or component.

Tensile stresses (especially near surface) can aid the onset of cracking which can cause premature failure.



RS superpose external load stresses under service conditions.

In general, compressive stresses at a surface are beneficial and enhance resistance to failure.



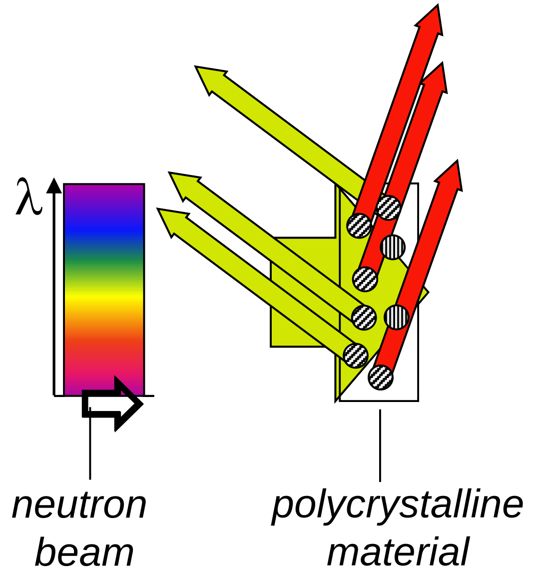
Courtesy M. Boin, HZB

Applications: Metallography

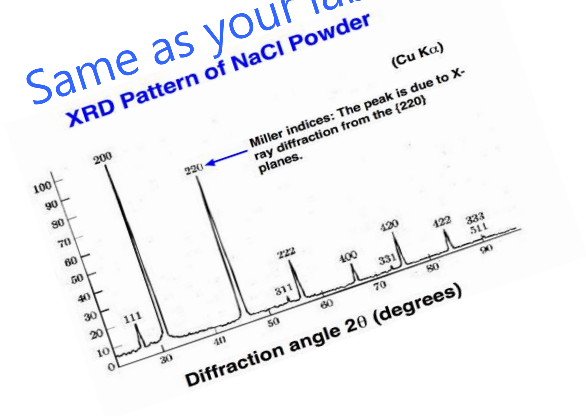
Stress/Strain analysis by Diffraction

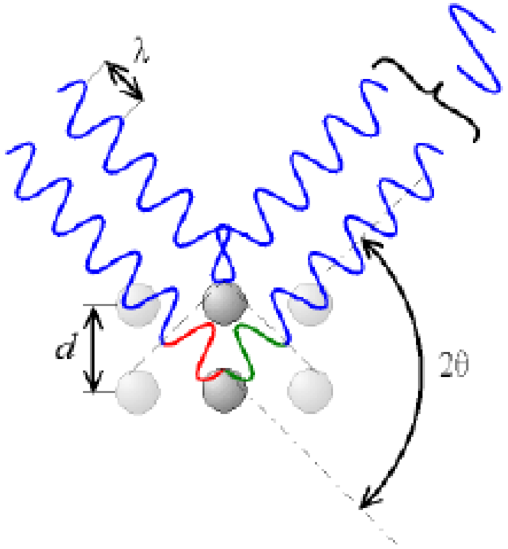
Constructive interference only when:

Bragg's law: $\lambda = 2d_{hkl}\sin\theta^{B}$

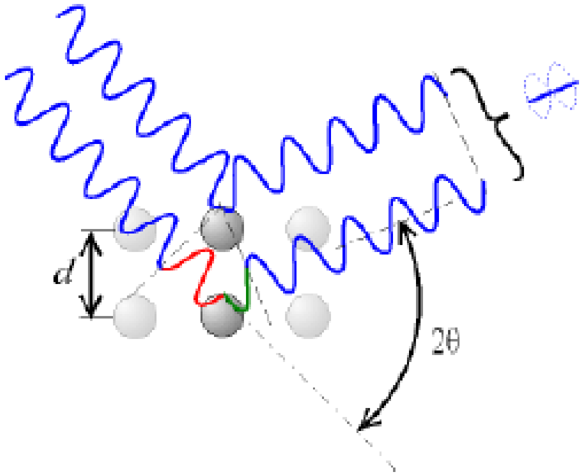


Same as your lab X-rays…





"peaks" at well defined 2θ angles indicate "d-spacing"

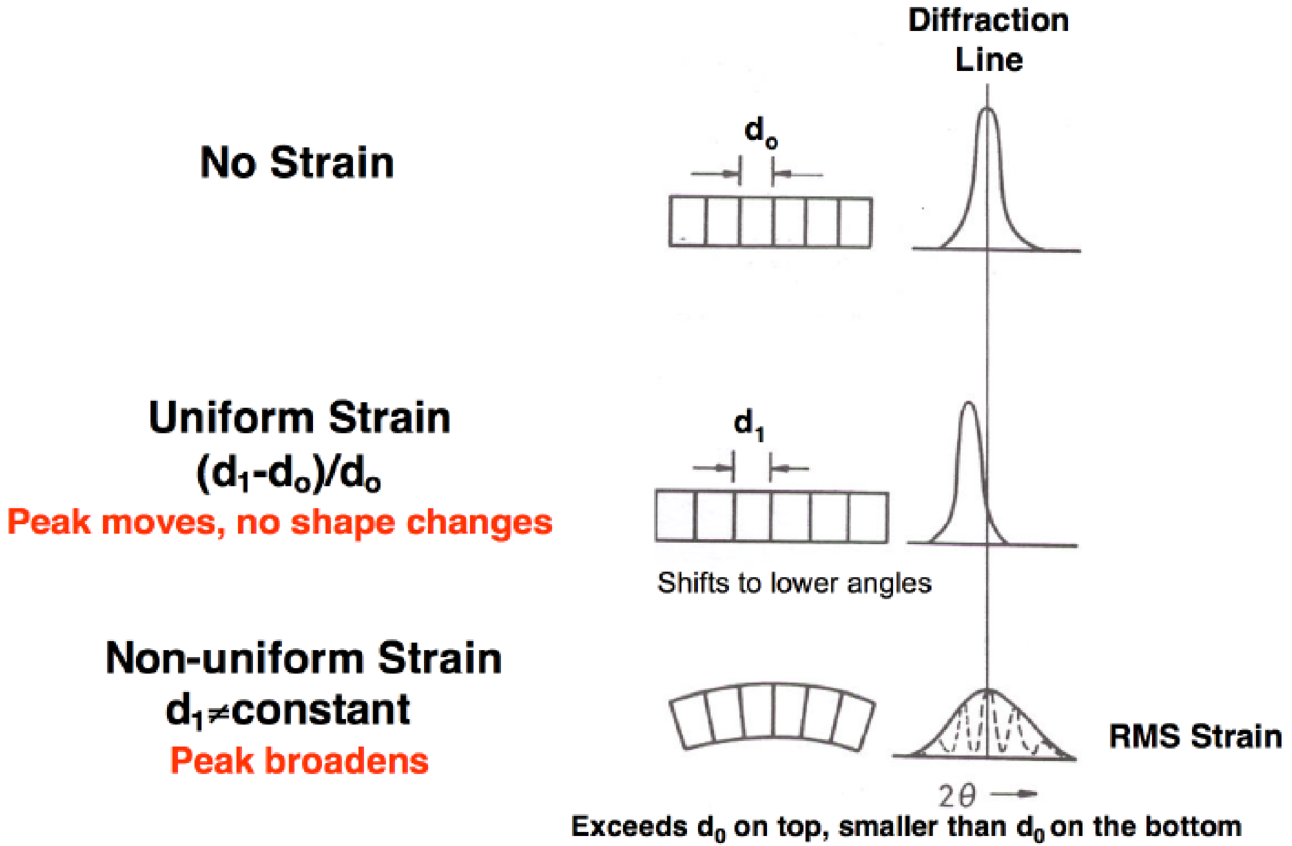


no peaks in other directions

Applications: Metallography

Stress/Strain analysis by Diffraction

Strain due to any mechanical, chemical or thermal process. e.g. loading, machining, plating and welding induces variations in d-spacing and thus 2-theta



P Moeck, Portland State University

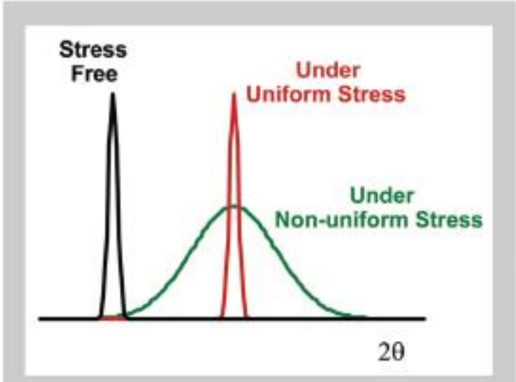


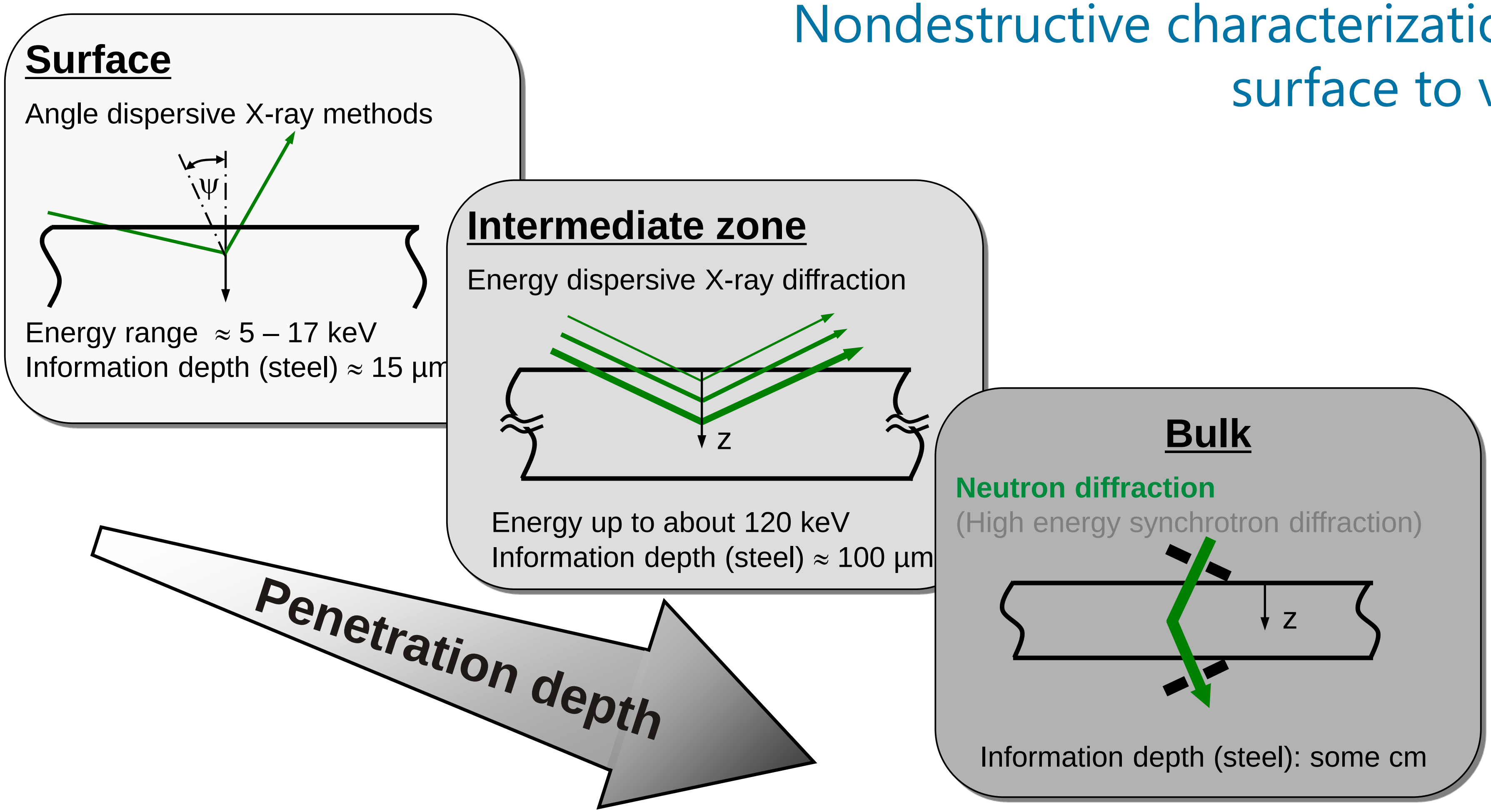
Figure 2. The principle of strain measurements by neutron diffraction.

$$\varepsilon = \frac{d - d_0}{d_0} = \frac{\sin\theta_0}{\sin\theta} - 1$$

Applications: Metallography

Stress/Strain analysis by Diffraction

Nondestructive characterization from surface to volume!

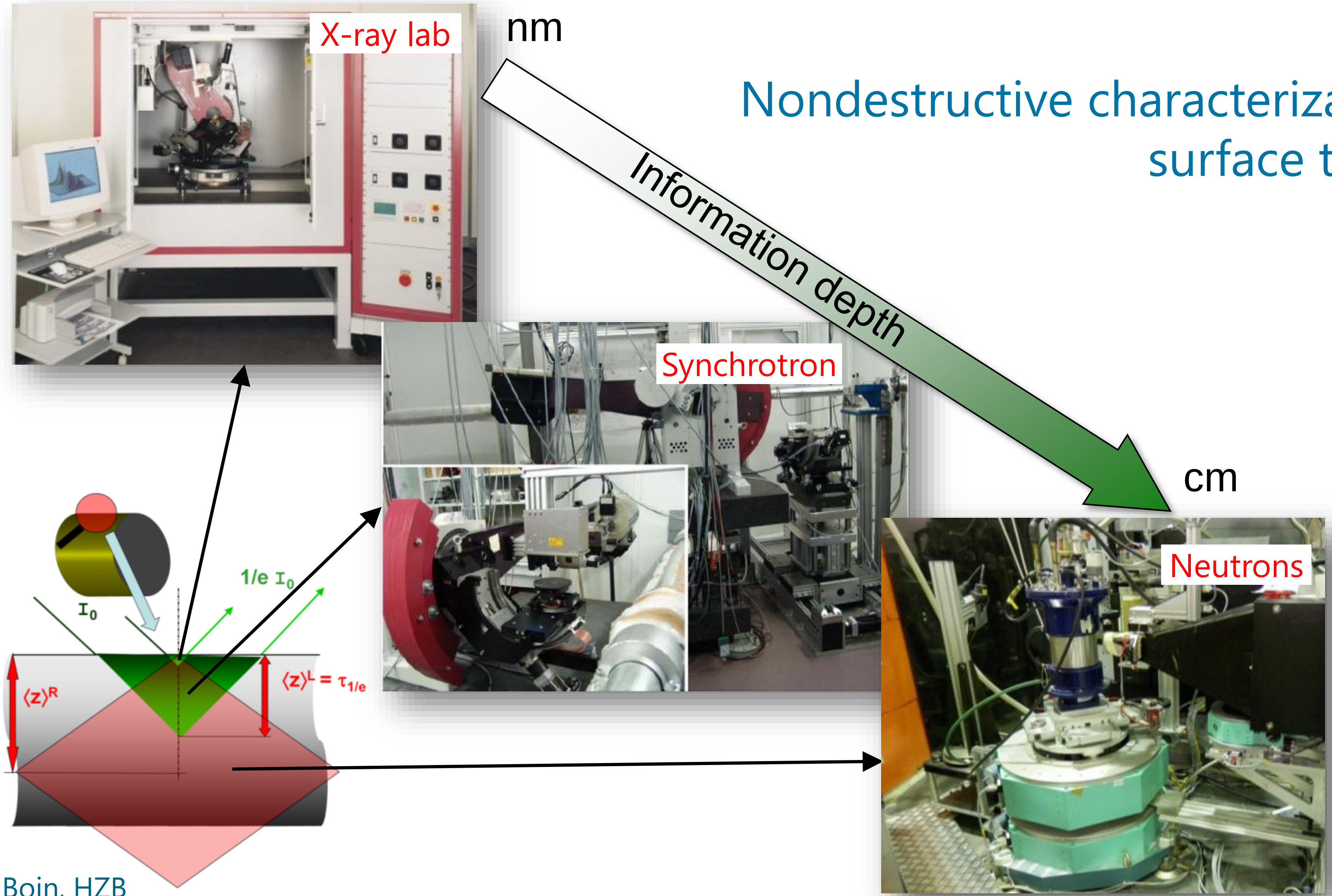


Courtesy M. Boin, HZB

Applications: Metallography

Stress/Strain analysis by Diffraction

Nondestructive characterization from surface to volume!



Courtesy M. Boin, HZB

Applications: Metallography

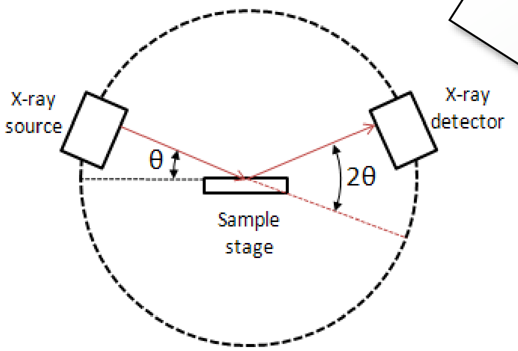
Stress/Strain analysis by Diffraction

X-ray lab

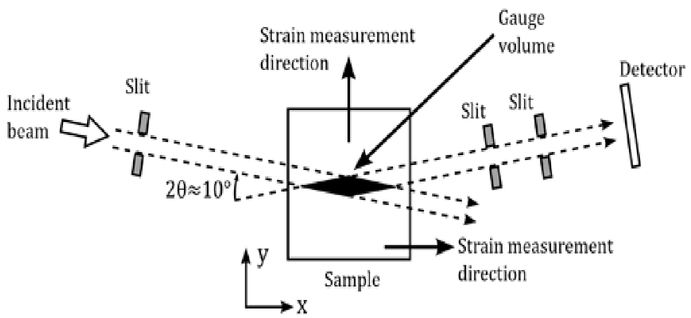
nm

Nondestructive caracterization from surface to volume!

Information depth

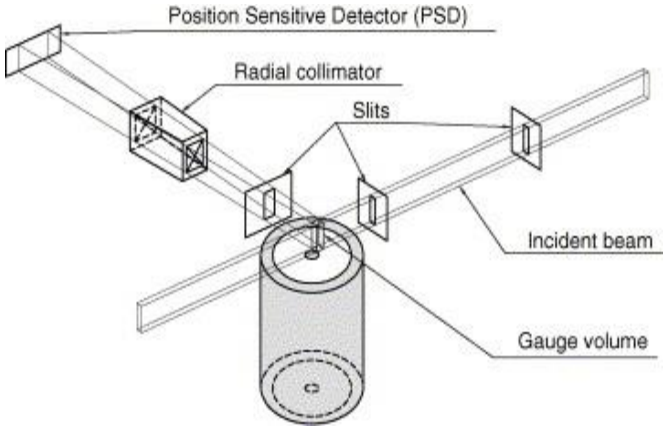


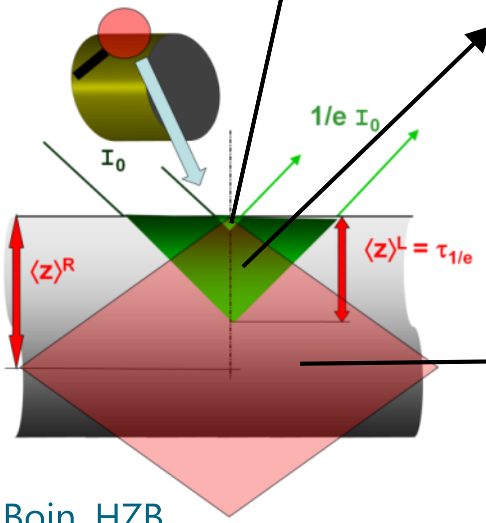
Synchrotron



cm

Neutrons

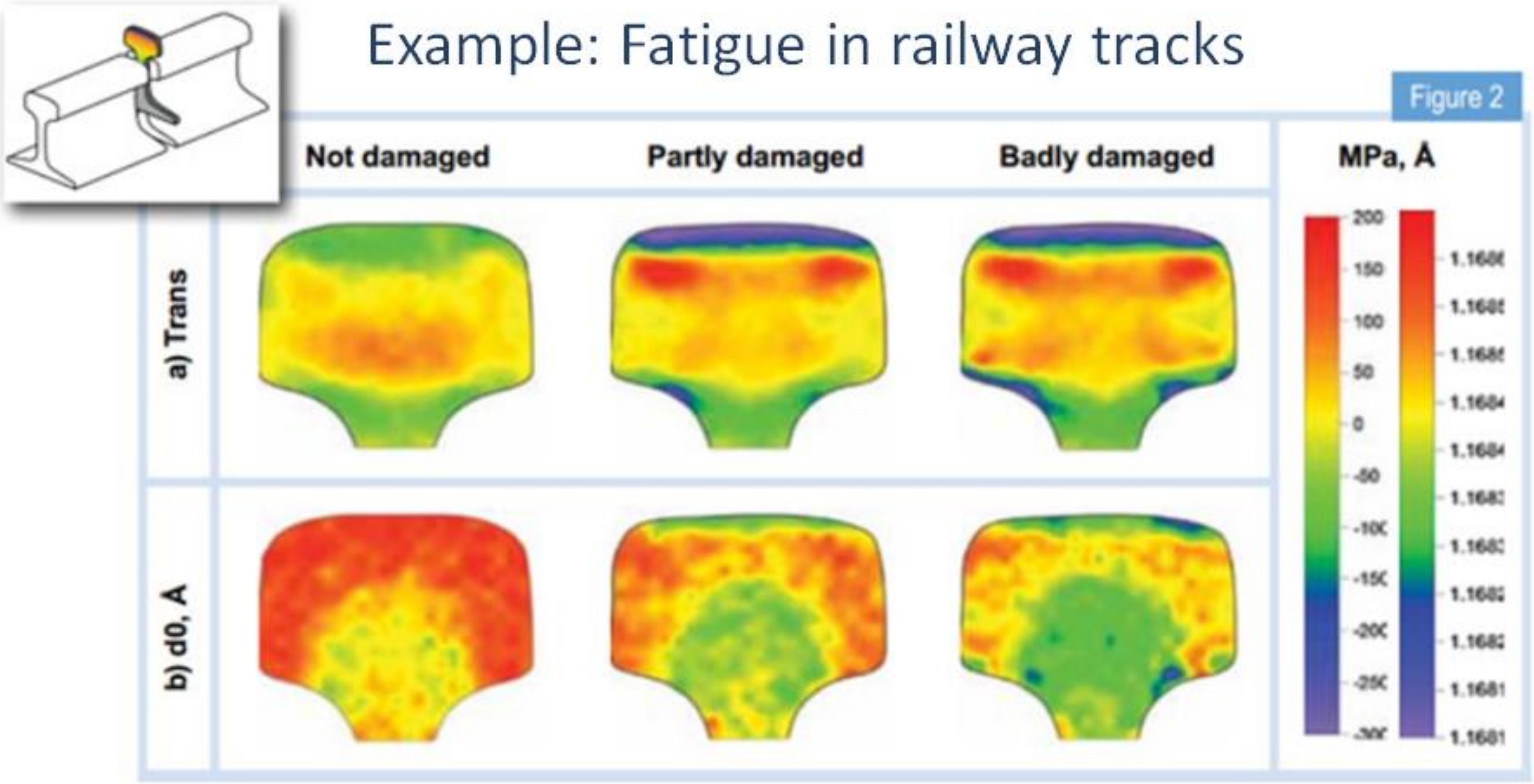




Courtesy M. Boin, HZB

Applications: Metallography

Stress/Strain analysis by Diffraction

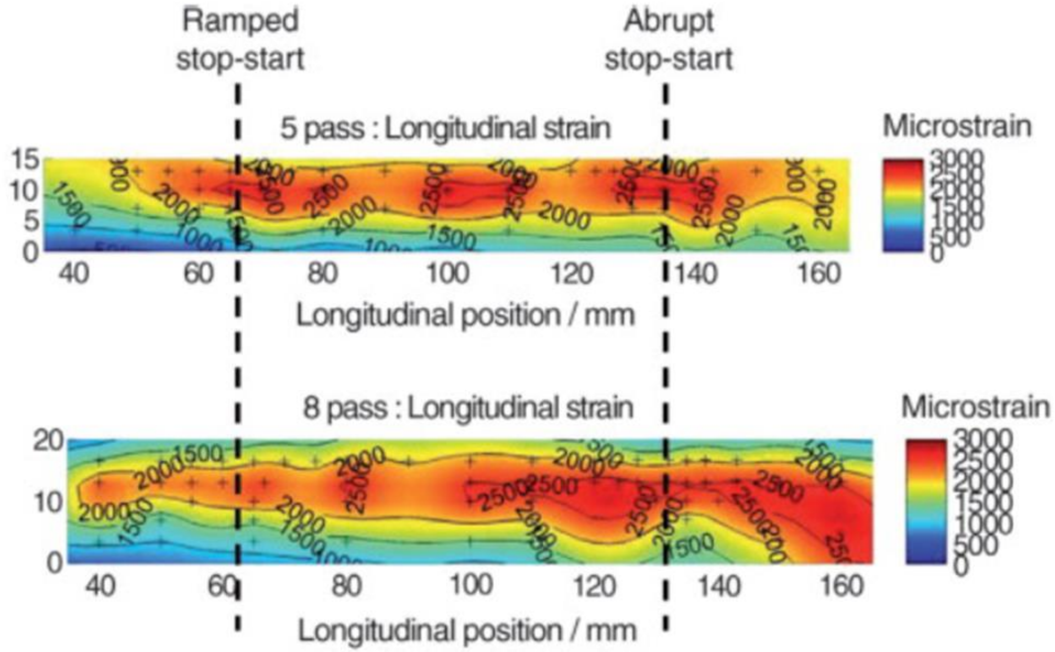


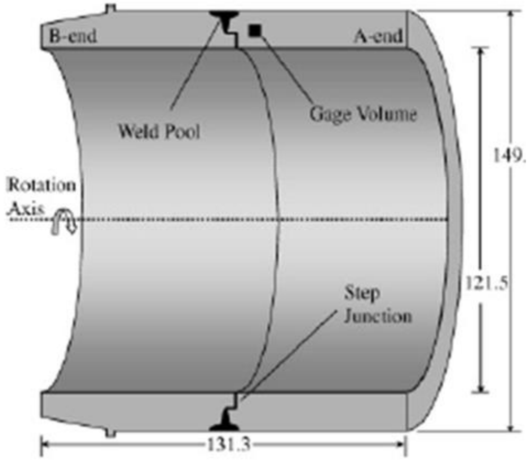
Jun et al., The Journal of Strain Analysis for Engineering Design 44.7 (2009)

Applications: Metallography

Stress/Strain analysis by Diffraction

Example:
Residual stress around welds
& changing welding parameters





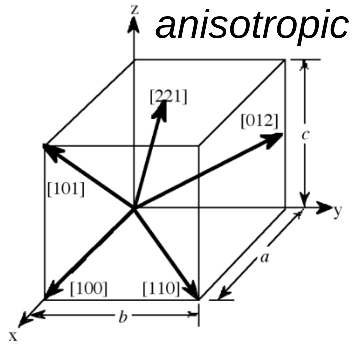
Applications: Metallography

Example: Applied stress

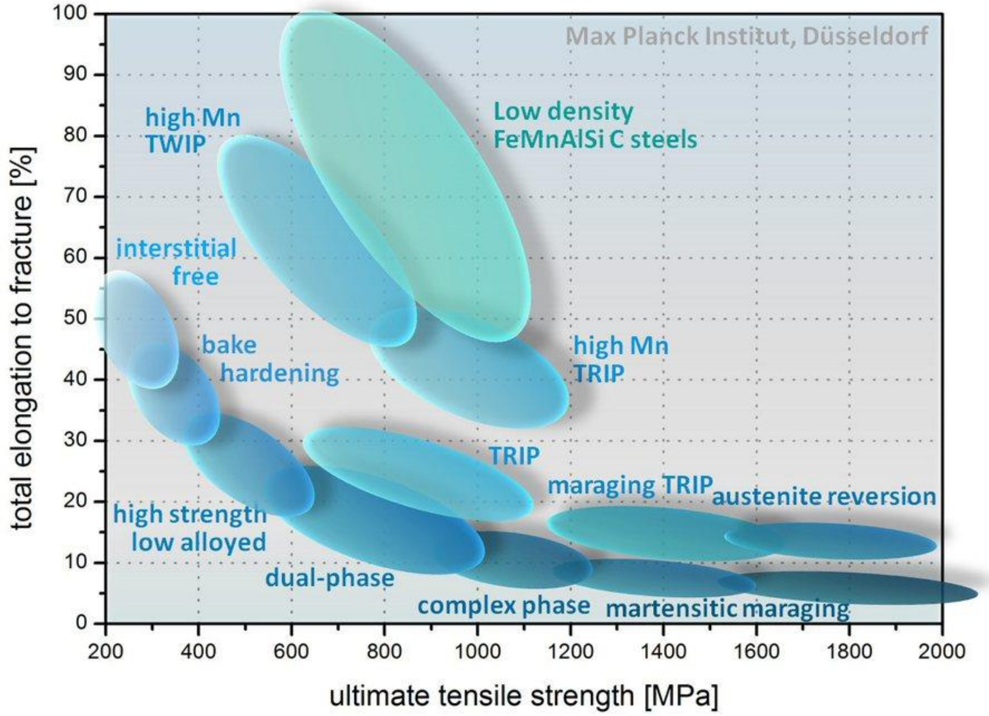
Neutron diffraction used extensively to:

- study lattice spacing changes under uni-axial deformation (tension/compression)
- determine lattice specific elastic constants (E_{hkl}, ν_{hkl})
- validate and develop deformation models

Stiffness generally anisotropic



-> High performance alloys are multi-phase and complex

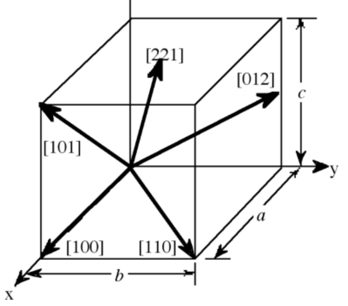


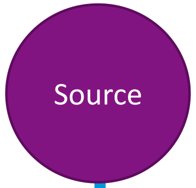
Applications: Metallography

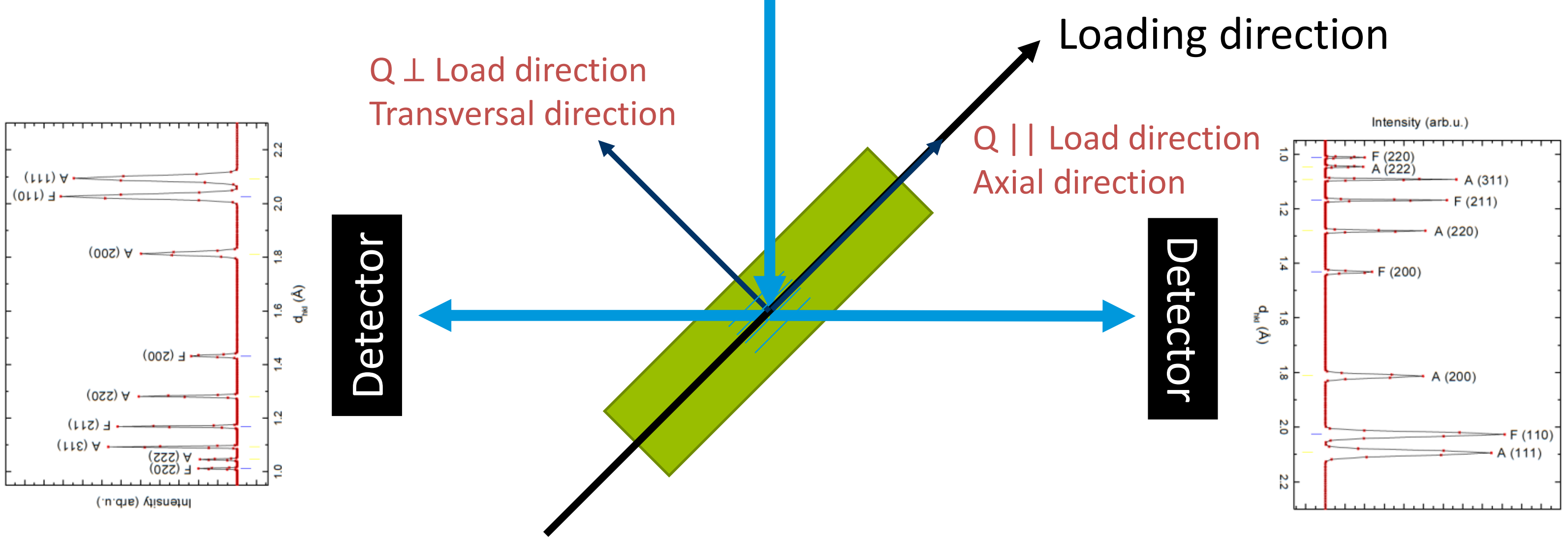
Stress/Strain analysis by Diffraction

- Special arrangement for Time of Flight
- Incident beam and 2 detectors at ±90°
- **Example tensile test:** Load axis along 45° with respect to the incident beam
- Measurement of two perpendicular components for multiple reflections at once
- Axial direction: Q || load axis
- Transversal direction: Q ⊥ load axis

Stiffness generally anisotropic





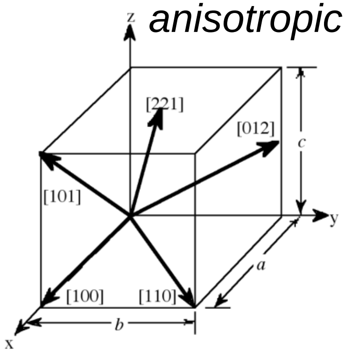


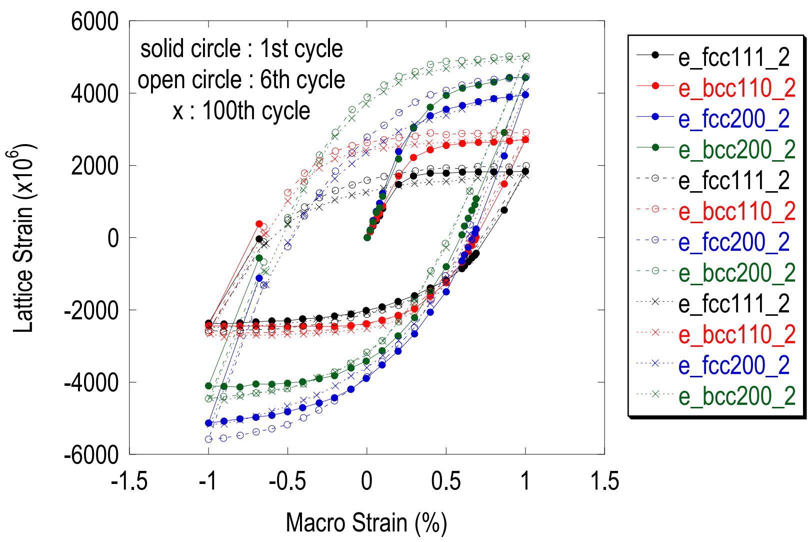
Applications: Metallography

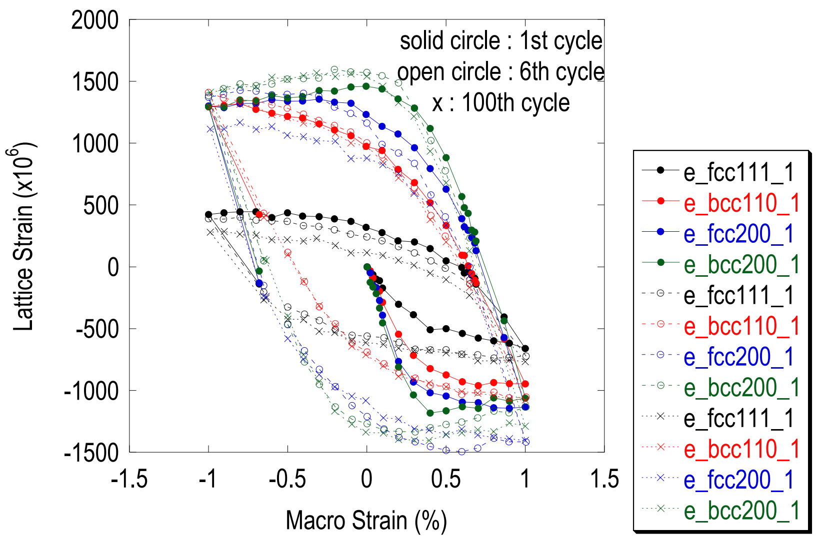
Stress/Strain analysis by Diffraction

- Special arrangement for Time of Flight
- Incident beam and 2 detectors at ±90°
- **Example tensile test:** Load axis along 45° with respect to the incident beam
- Measurement of two perpendicular components for multiple reflections at once
- Axial direction: Q | | load axis
- Transversal direction: Q ⊥ load axis

Stiffness generally anisotropic





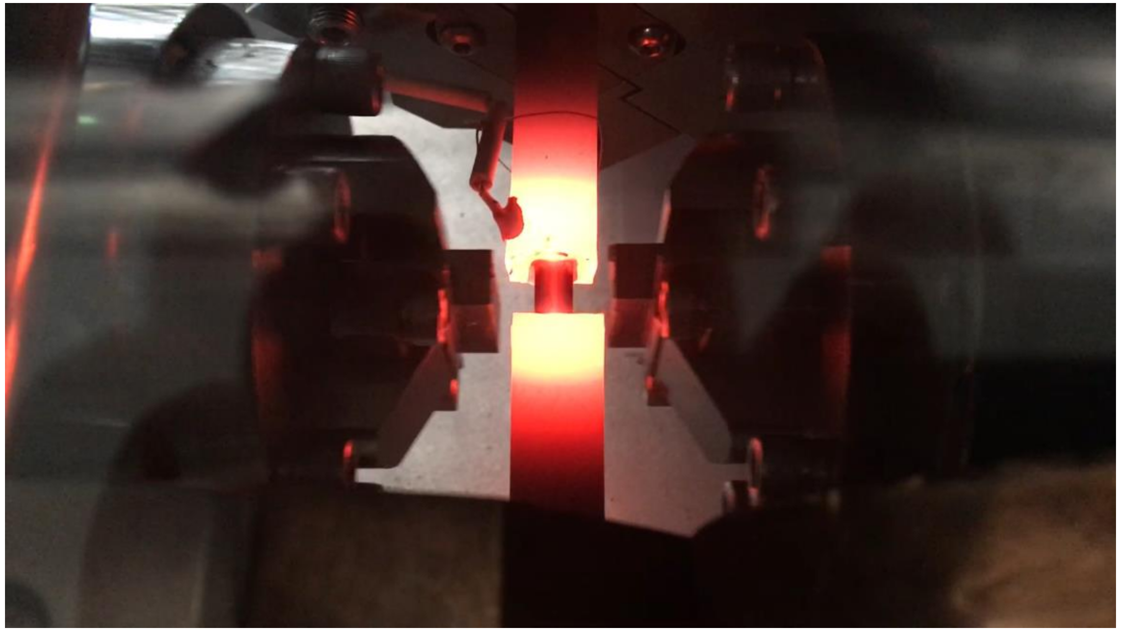


Applications: Metallography

Stress/Strain analysis by Diffraction

Completion option on the ESS Engineering diffractometer BEER:

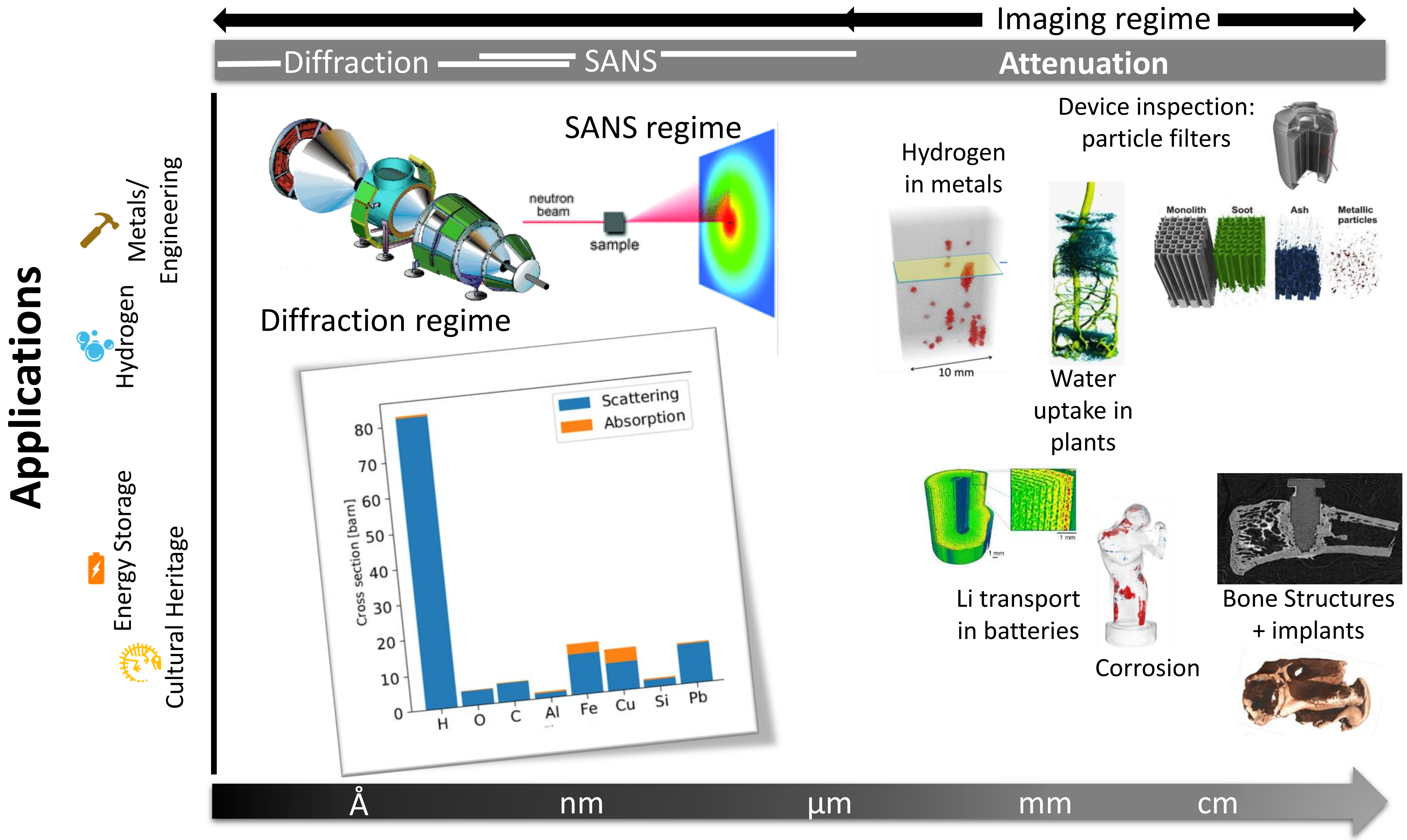
Thermal and mechanical processes simulator using GLEEBLE system on beamline



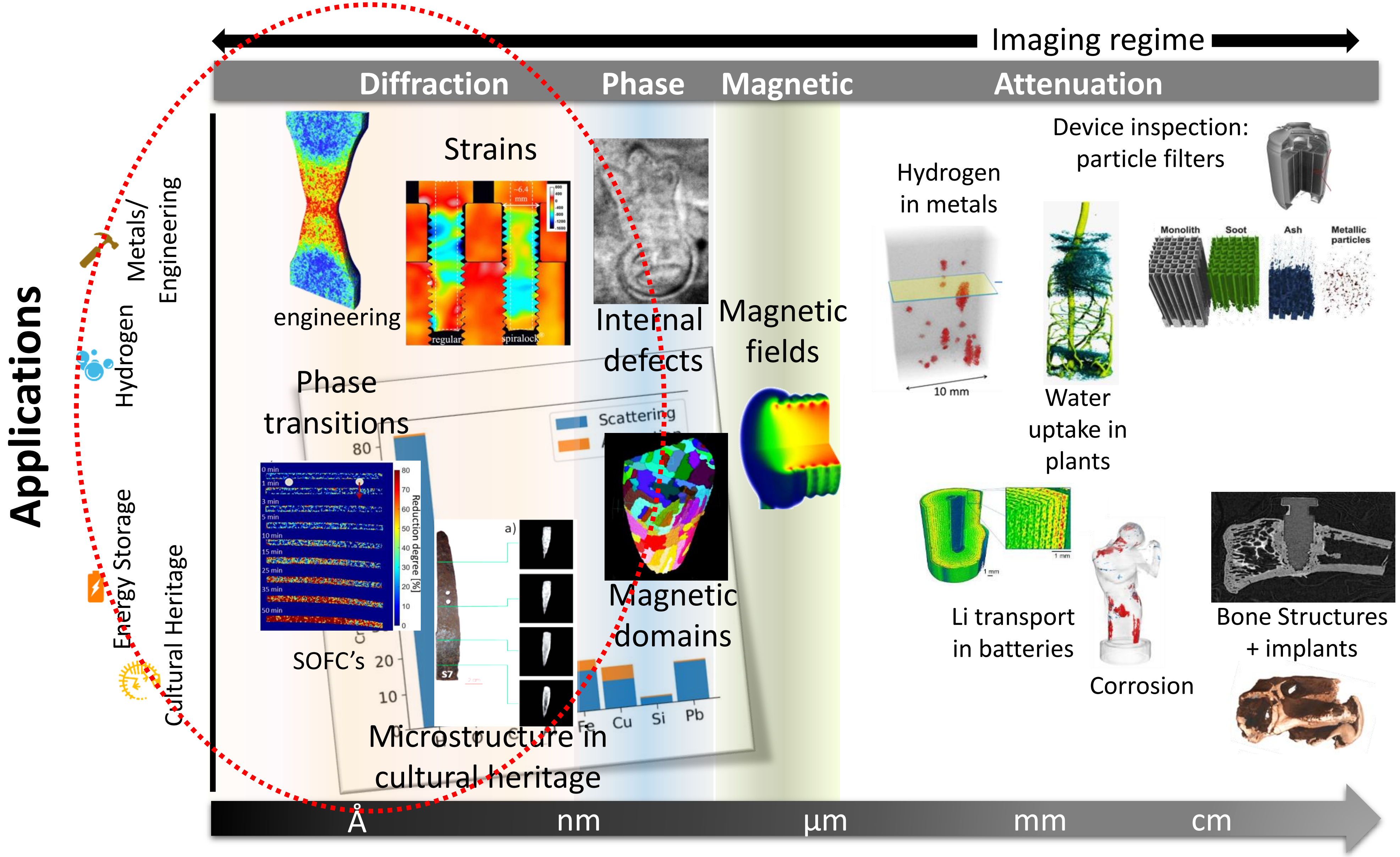
Non-destructive testing with neutrons: Engineering materials and components revealed

Neutron Methods & Length Scales

Kardjilov, Manke, Woracek, Banhart,. Advances in neutron imaging. Materials Today 21 (2018)



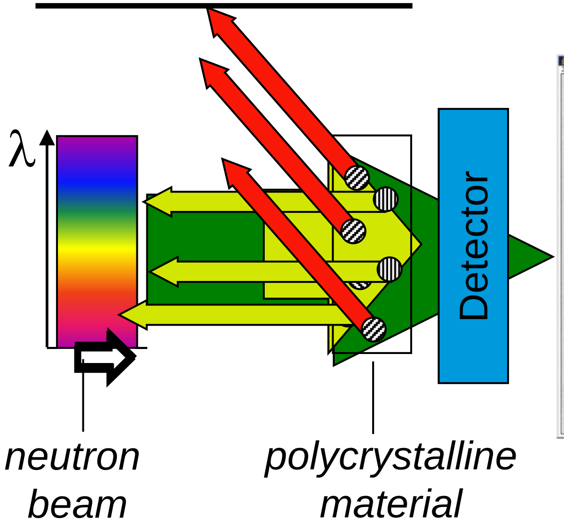
Neutron Methods & Length Scales

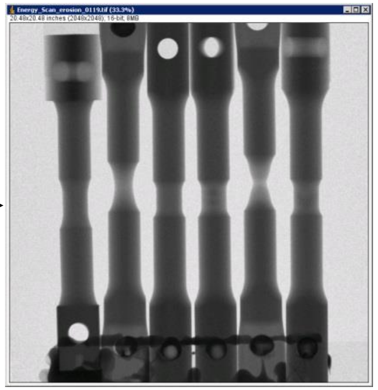


Neutron Imaging: Diffraction Contrast

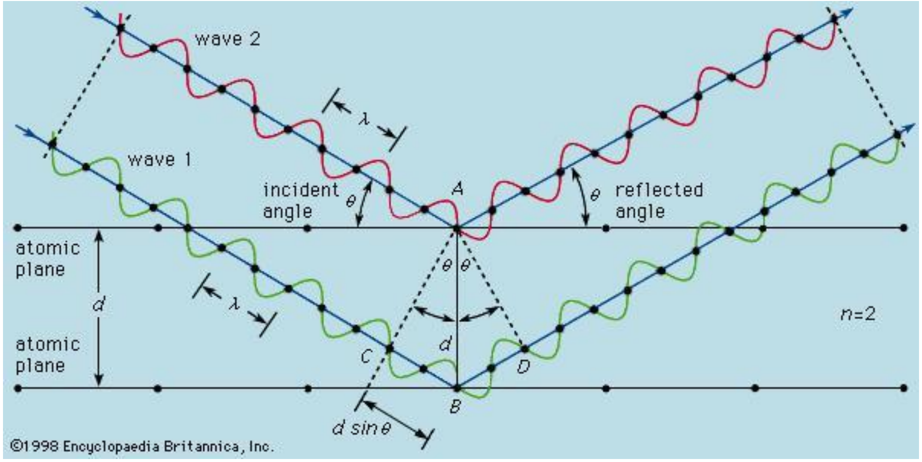
- Coh. elastic scattering $\sigma_T(\lambda) = \sigma_{el.coh.}(\lambda) + \sigma_{el.inc.}(\lambda) + \sigma_{inel.coh.}(\lambda) + \sigma_{inel.incoh.}(\lambda) + \sigma_{abs}(\lambda)$
- *hkl* spacing probed in beam direction (“averaged” through thickness)

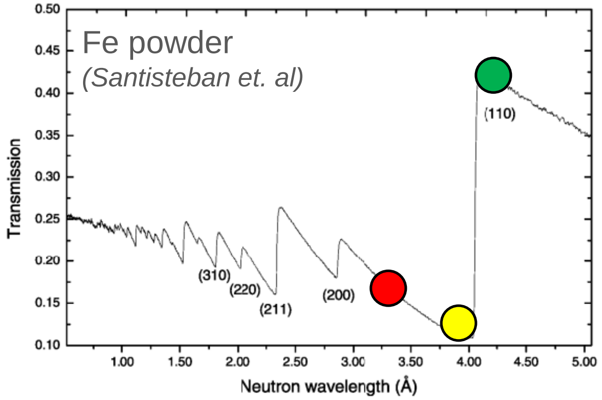
<u>Monochromatic</u>





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





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

$2d_{hkl}\sin 90°=\lambda$

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

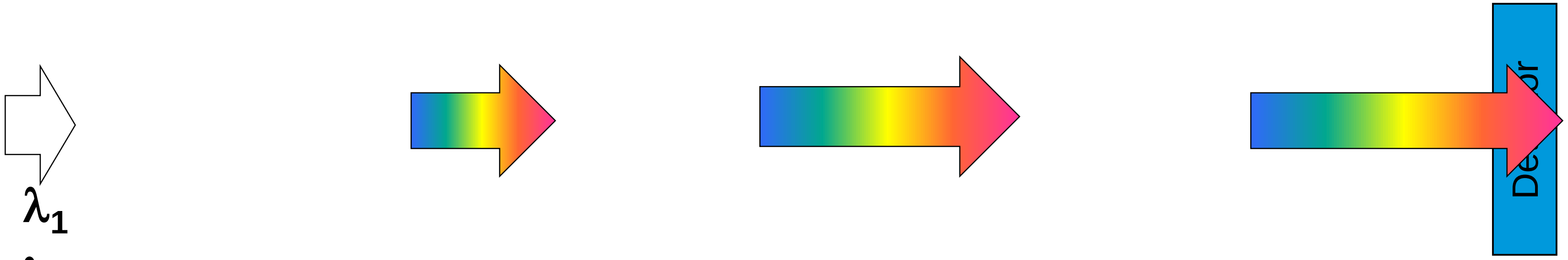
Neutron Imaging: Diffraction Contrast

- Coh. elastic scattering $\sigma_T(\lambda) = \sigma_{el.coh.}(\lambda) + \sigma_{el.inc.}(\lambda) + \sigma_{inel.coh.}(\lambda) + \sigma_{inel.incoh.}(\lambda) + \sigma_{abs}(\lambda)$
- *hkl* spacing probed in beam direction ("averaged" through thickness)

Time of flight

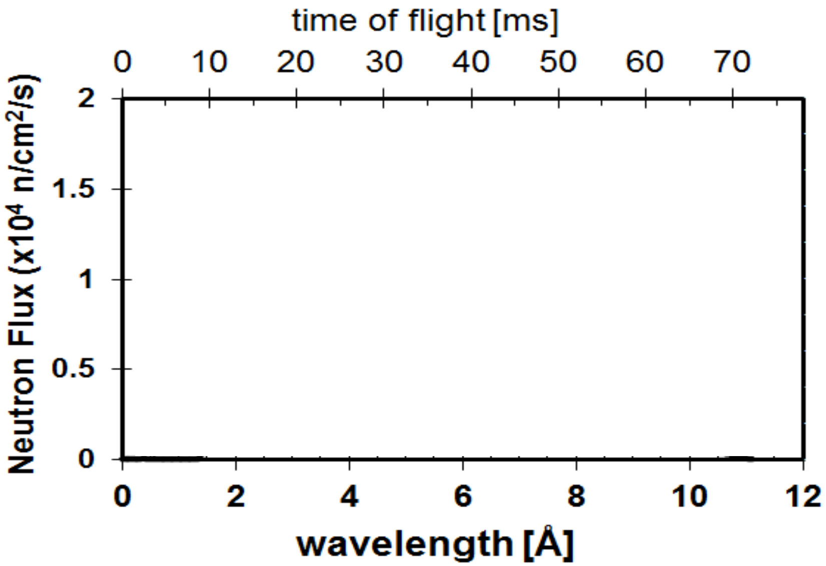
Source

Detector



λ_1

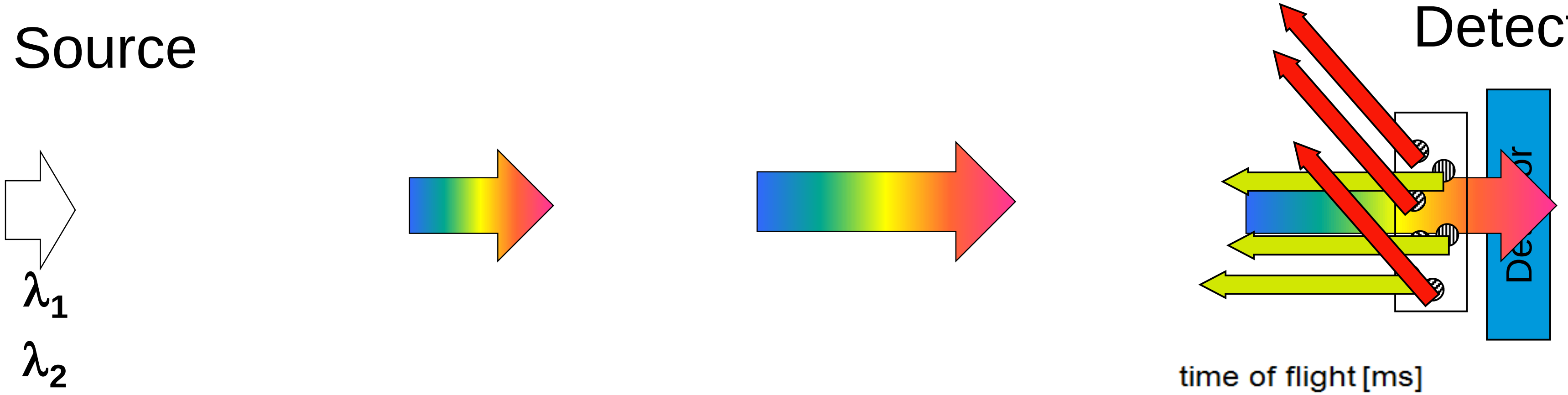
λ_2

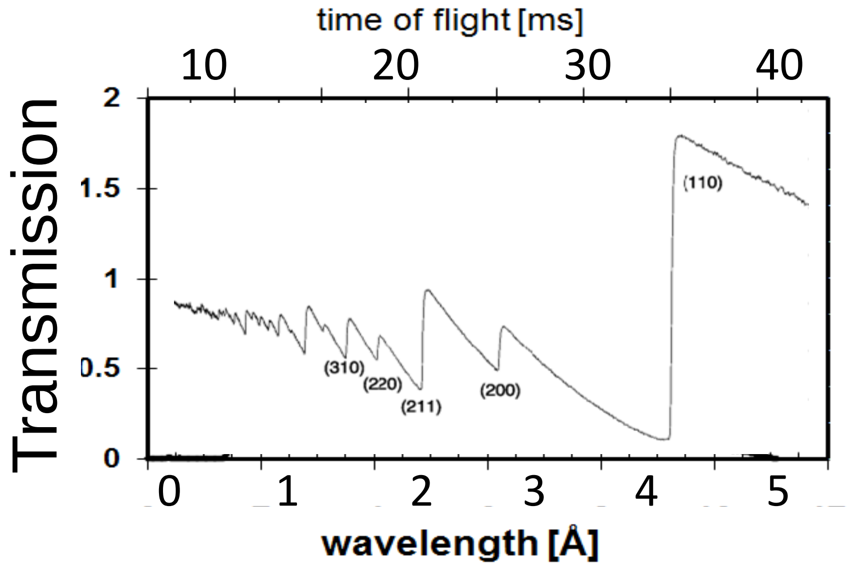


Neutron Imaging: Diffraction Contrast

- Coh. elastic scattering $\sigma_T(\lambda) = \sigma_{el.coh.}(\lambda) + \sigma_{el.inc.}(\lambda) + \sigma_{inel.coh.}(\lambda) + \sigma_{inel.incoh.}(\lambda) + \sigma_{abs}(\lambda)$
- *hkl* spacing probed in beam direction (“averaged” through thickness)

Time of flight



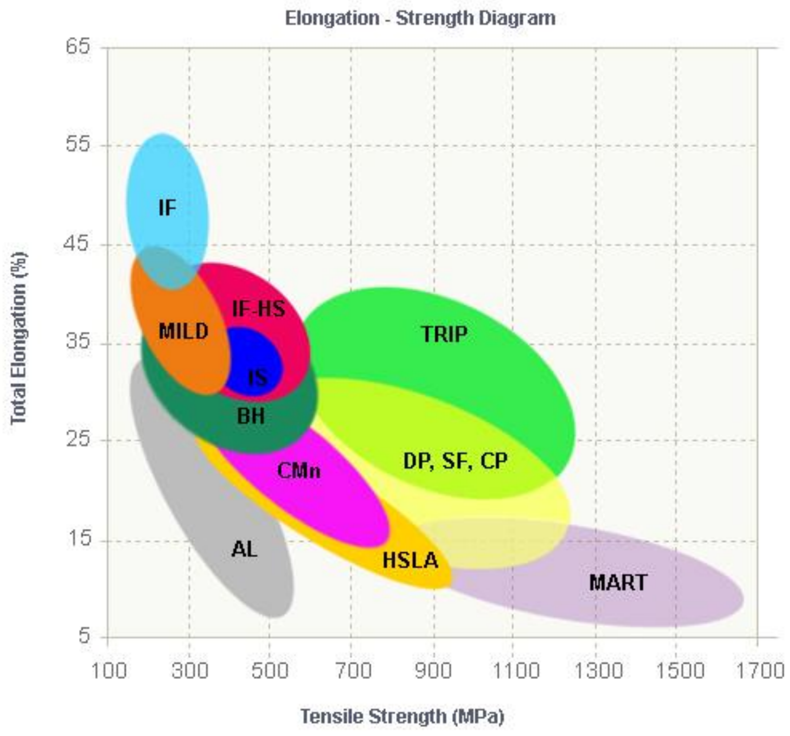


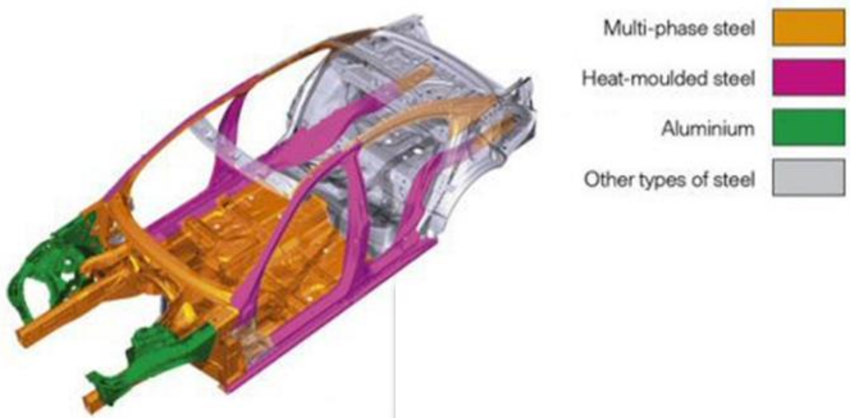
Applications: Metallography

Crystalline phase fractions in 3D

- Multiphase steels posses a good combination of strength and elongation.
- TRIP steel: FCC Austenite transforms to HCP and BCC Martensites under strain.

Formability Chart: Material Based on Strength and Elongation

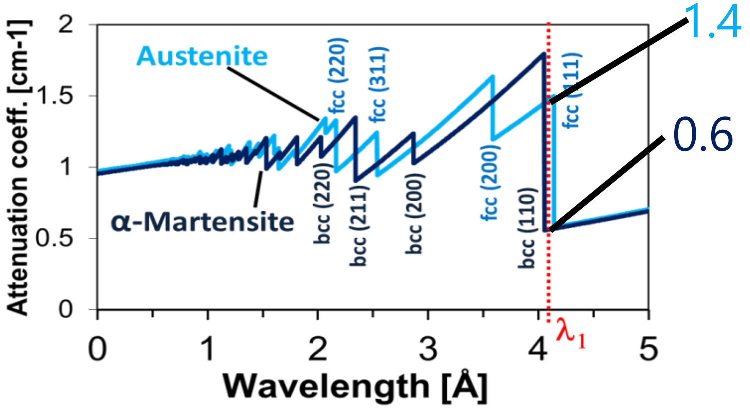


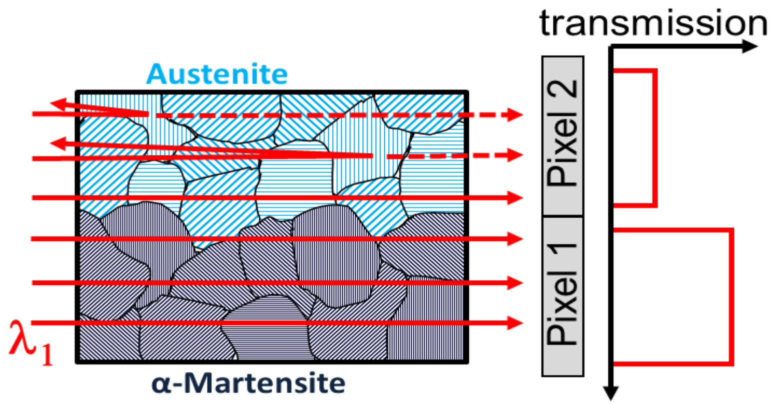


Applications: Metallography

Crystalline phase fractions in 3D

- Multiphase steels posses a good combination of strength and elongation.
- TRIP steel: FCC Austenite transforms to HCP and BCC Martensites under strain.
- Phases can be well separated by diffraction contrast in transmission (Bragg edges).
- Challenge: Many crystalline properties are directional dependent (*tensorial*) → tomographic reconstruction challenging.
- Non-tensorial properties such as phase fractions can be reconstructed for non-textured samples.

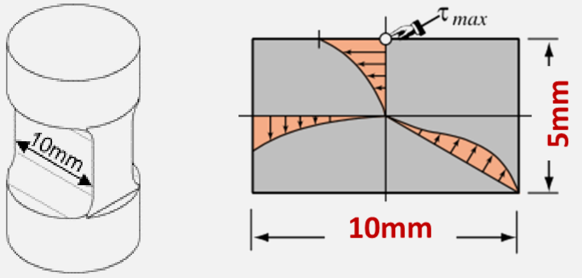


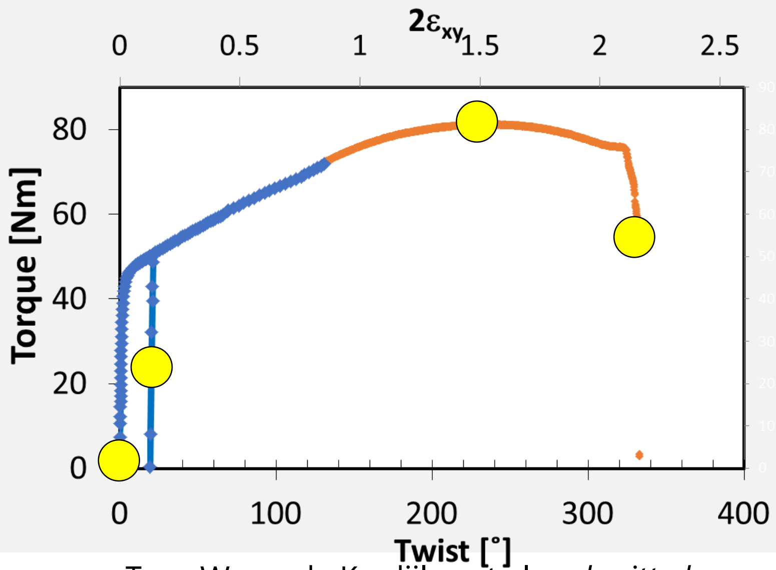


Applications: Metallography

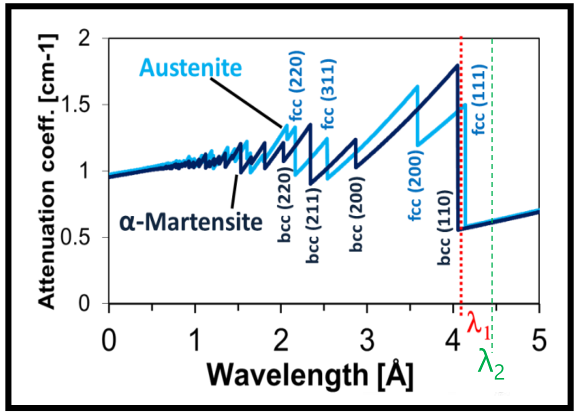
Crystalline phase fractions in 3D

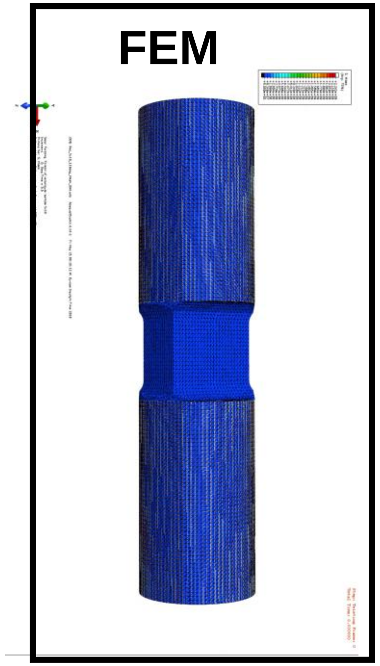
- Torsion of rectangular cross-section (304L)

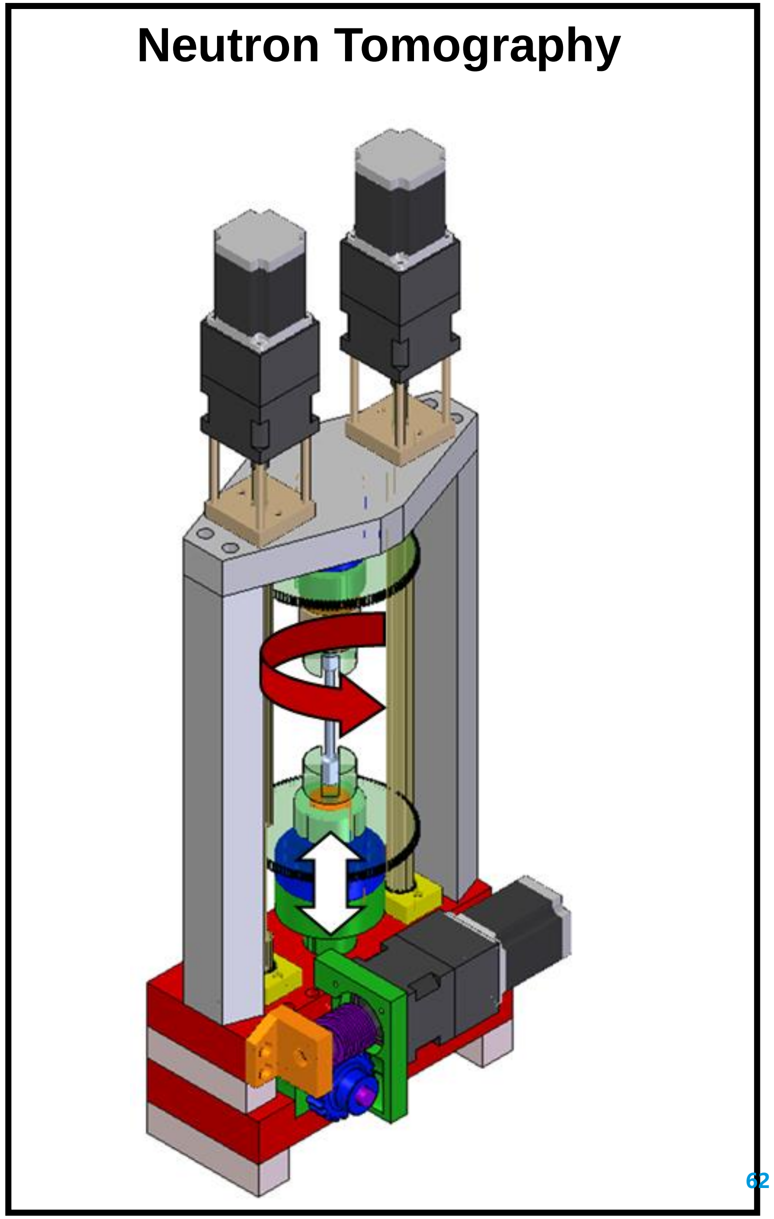




Tran, Woracek, Kardjilov et al., *submitted*



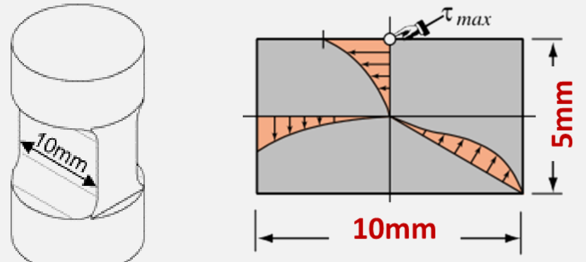


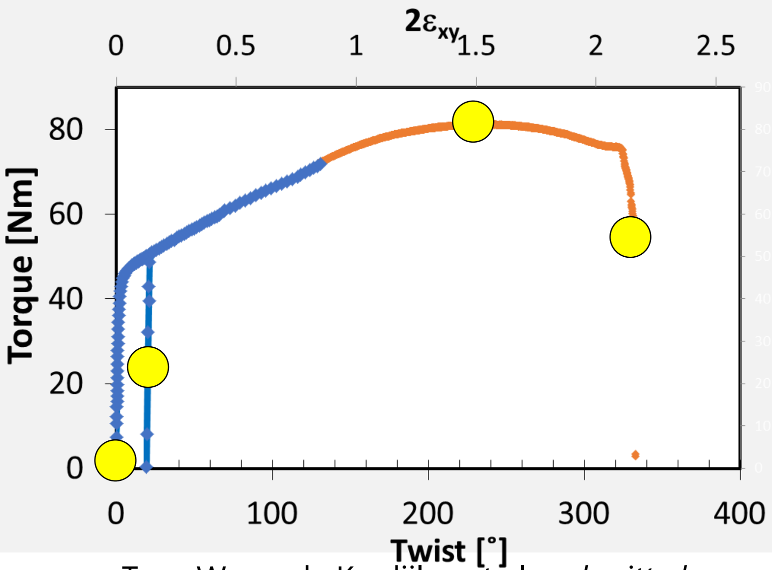


Applications: Metallography

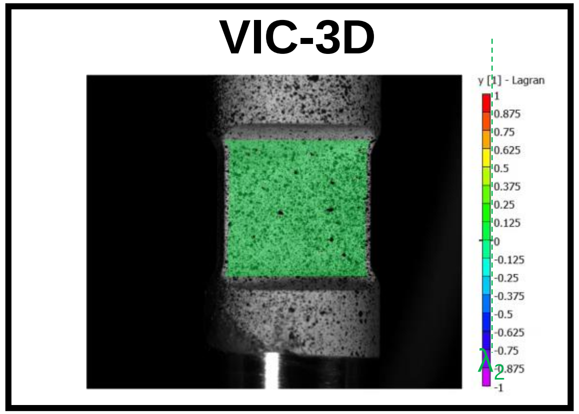
Crystalline phase fractions in 3D

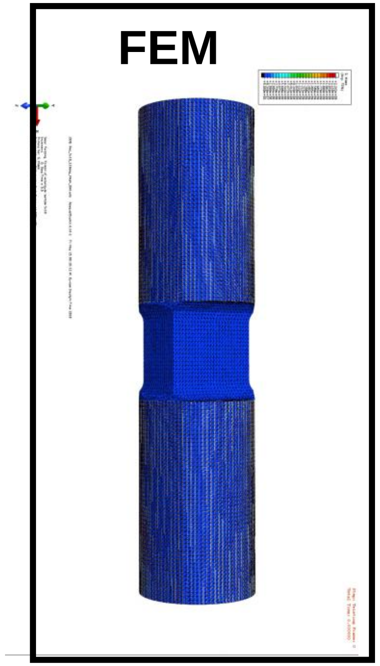
- Torsion of rectangular cross-section (304L)

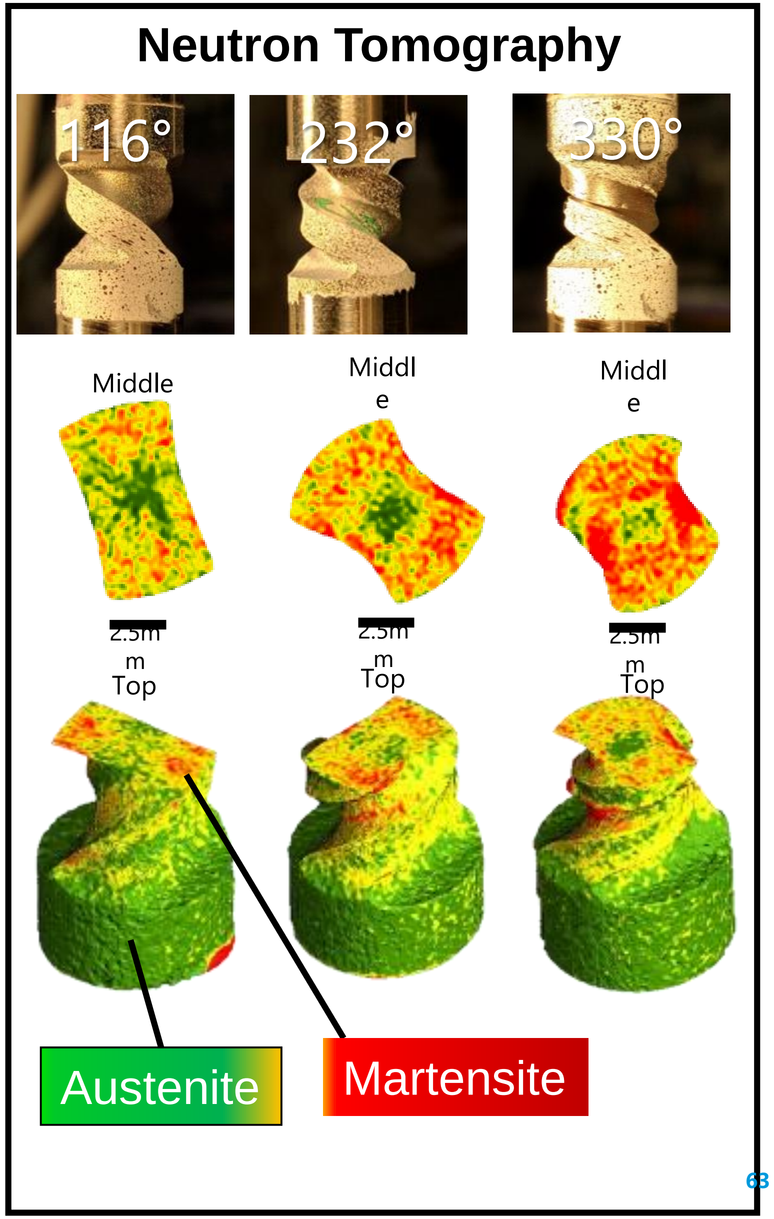




Tran, Woracek, Kardjilov et al., *submitted*

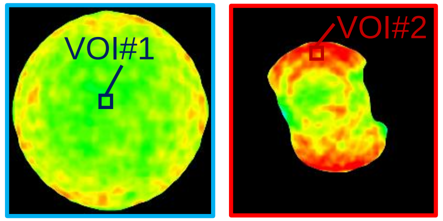




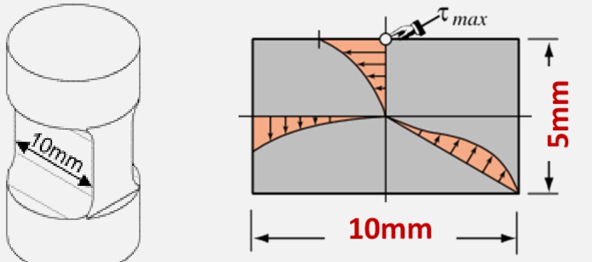


Applications: Metallography

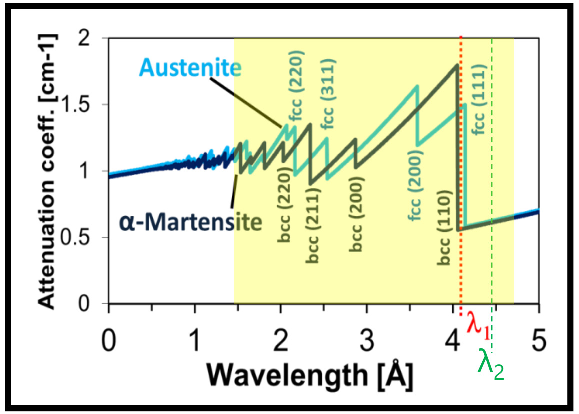
Crystalline phase fractions in 3D

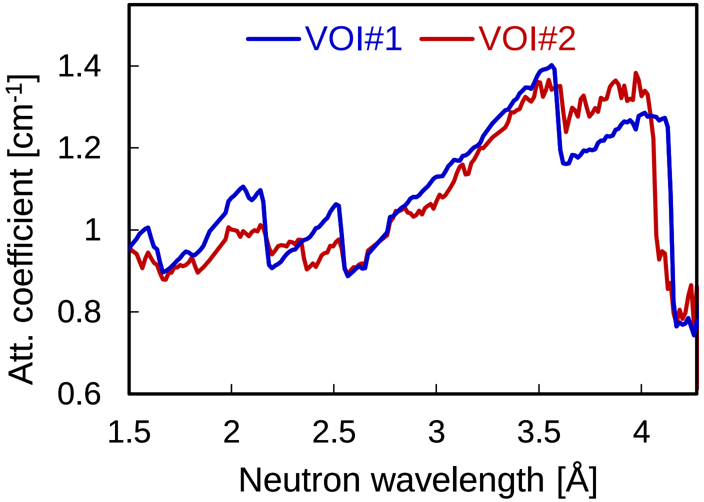


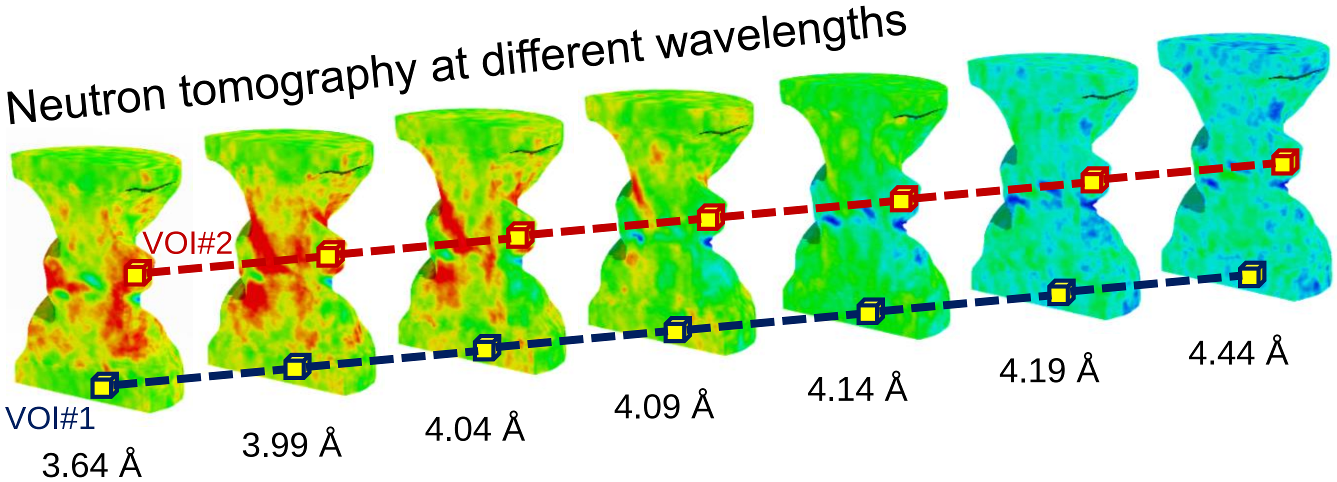
- Torsion of rectangular cross-section (304L)



- In past: Phase evaluation based on tomographic reconstruction before/after Bragg edge
- Recently: Established spectral neutron tomography (naturally suited for ToF)





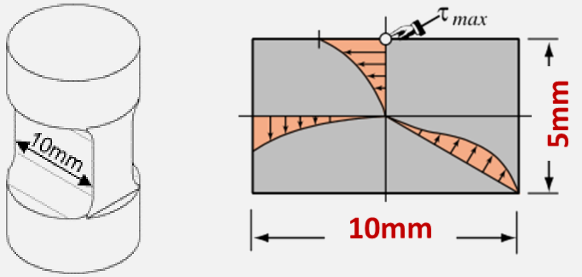


Tran, Woracek, Kardjilov et al., *Materials Today Advances, accepted (2021)*

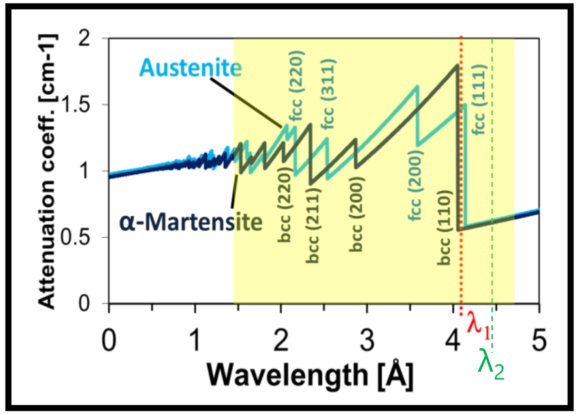
Applications: Metallography

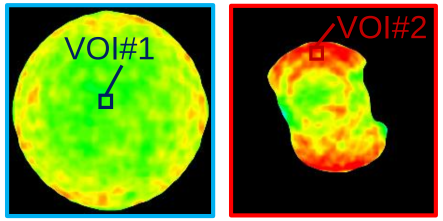
Crystalline phase fractions in 3D

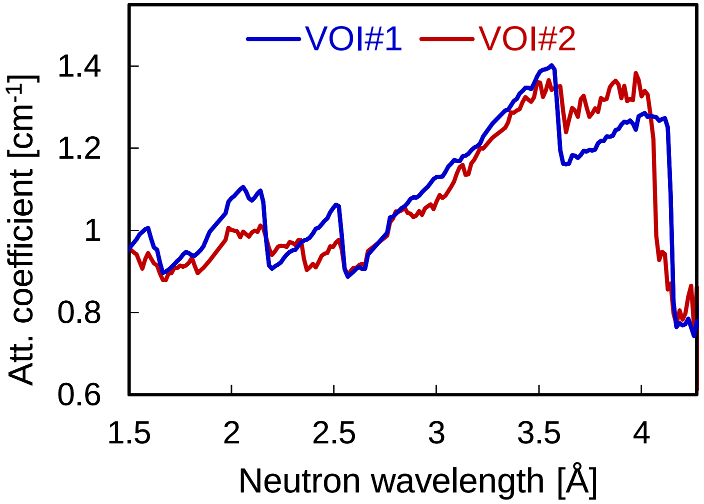
- Torsion of rectangular cross-section (304L)



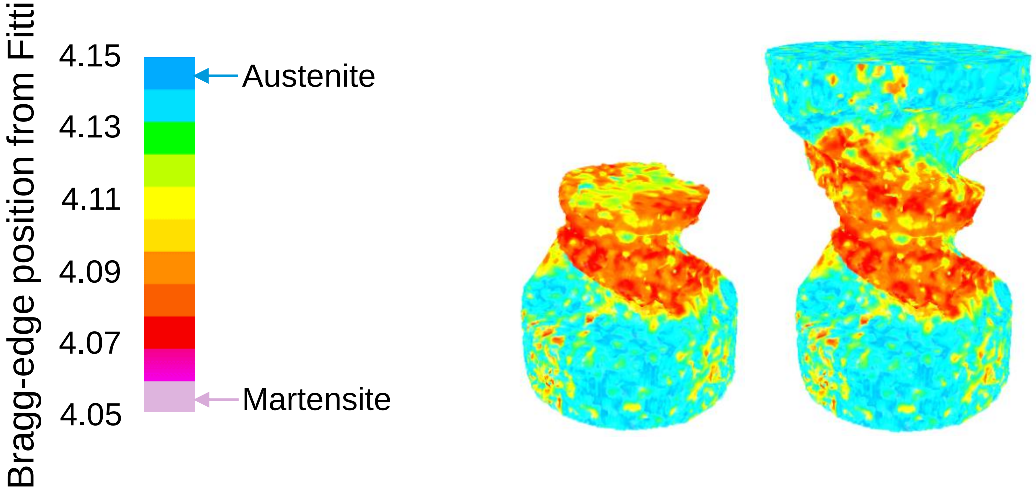
- In past: Phase evaluation based on tomographic reconstruction before/after Bragg edge
- Recently: Established spectral neutron tomography (naturally suited for ToF)







→ Improved quantification



Tran, Woracek, Kardjilov et al., *Materials Today Advances, accepted (2021)*

Applications: Metallography

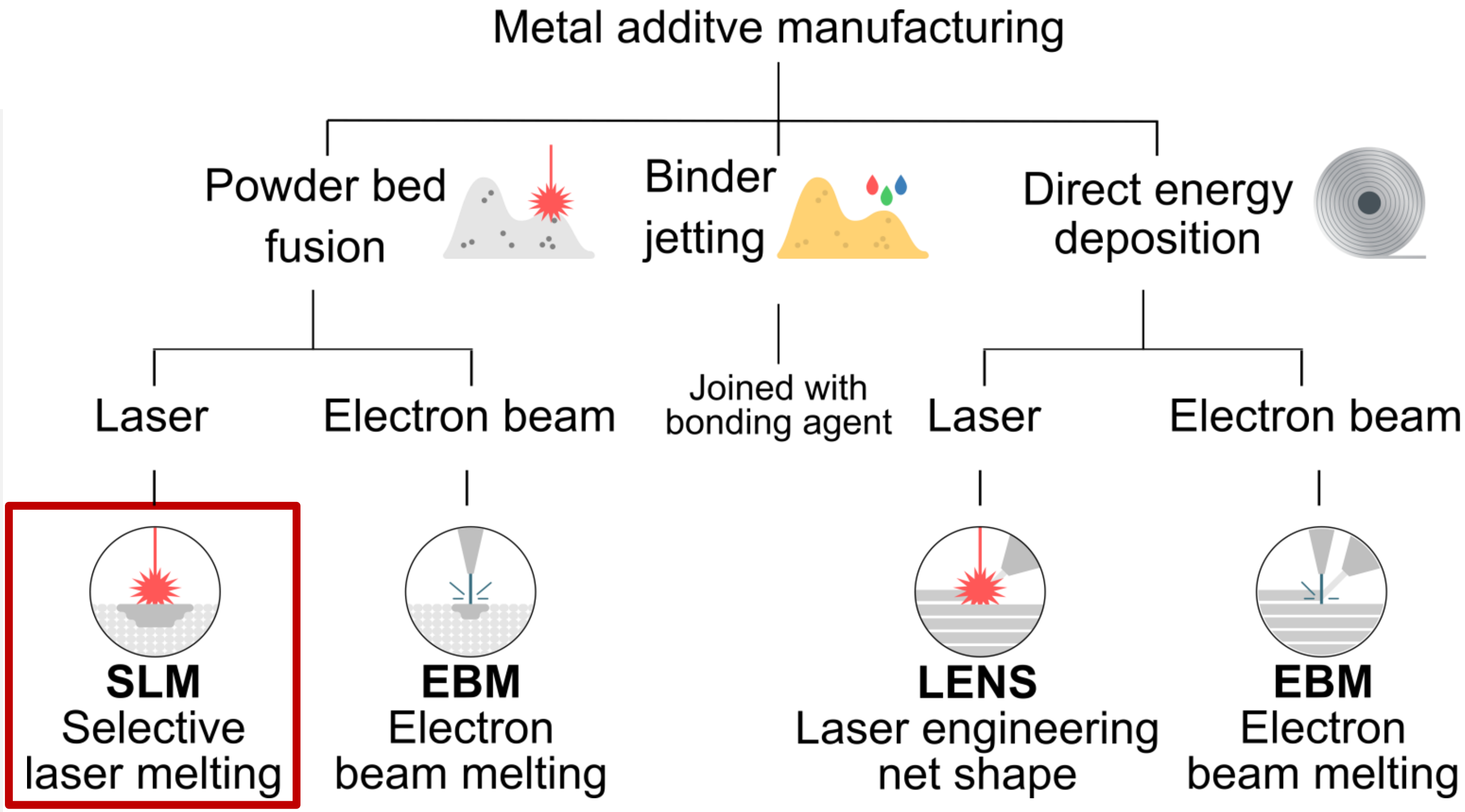
316L stainless steel produced by Selective Laser Melting (SLM)

Additive Manufacturing:

fabrication methods based on layer-by-layer approach using a digital model

→ Complex designs

Selective laser melting is the most industrially matured technique



Applications: Metallography

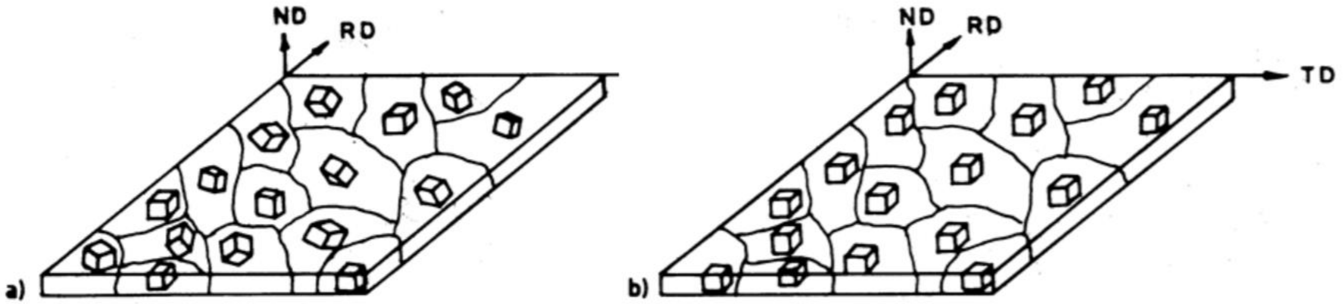
316L stainless steel produced by Selective Laser Melting (SLM)

Additive Manufacturing:

fabrication methods based on layer-by-layer approach using a digital model

→ Complex designs

Selective laser melting is the most industrially matured technique



A texture-less sheet (Random)

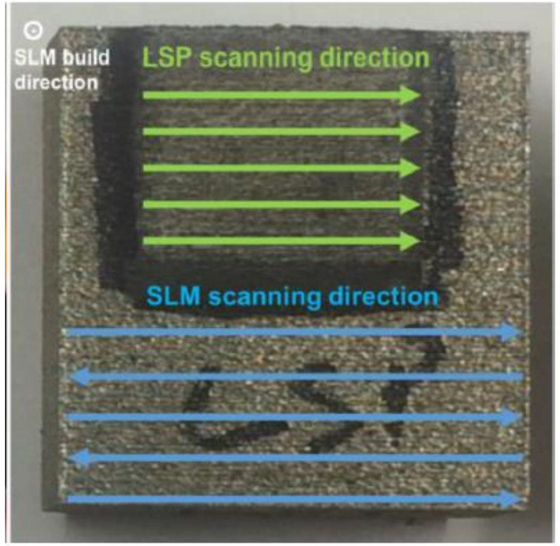
A fully textured sheet (Strong texture)

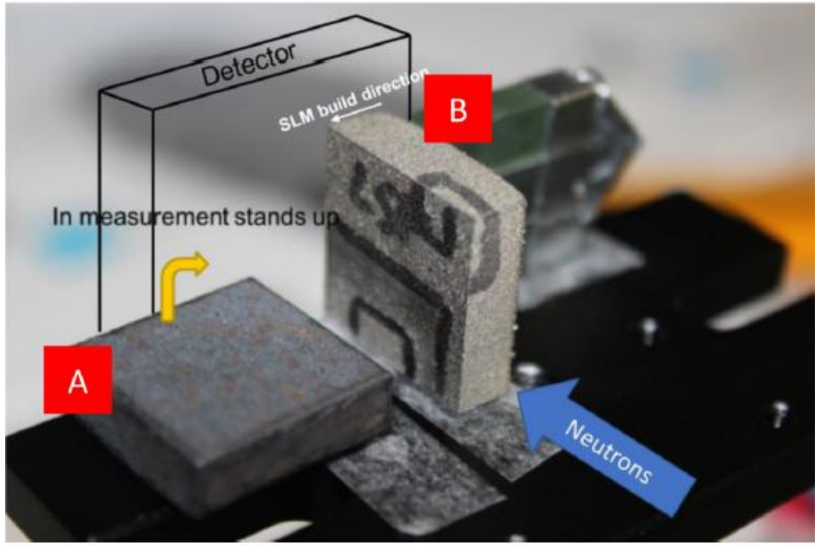
Applications: Metallography

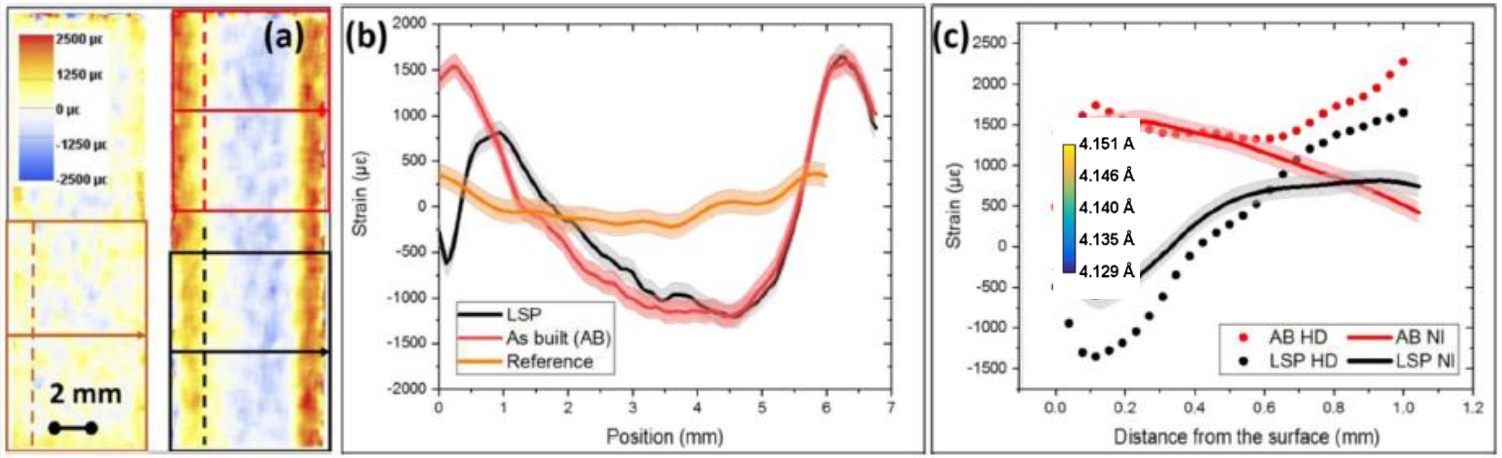
Residual stress and effect of LSP in SLM 316L stainless steel

- Selective laser melting
  - Heating and cooling cycles can induce severe residual stress (RS).
  - Laser shock peening (LSP) can be used to create beneficial compressive RS locally.

→ Diffraction Contrast for full field RS images







M. Morgano et al., Additive Manufacturing 34 (2020)

Applications: Metallography

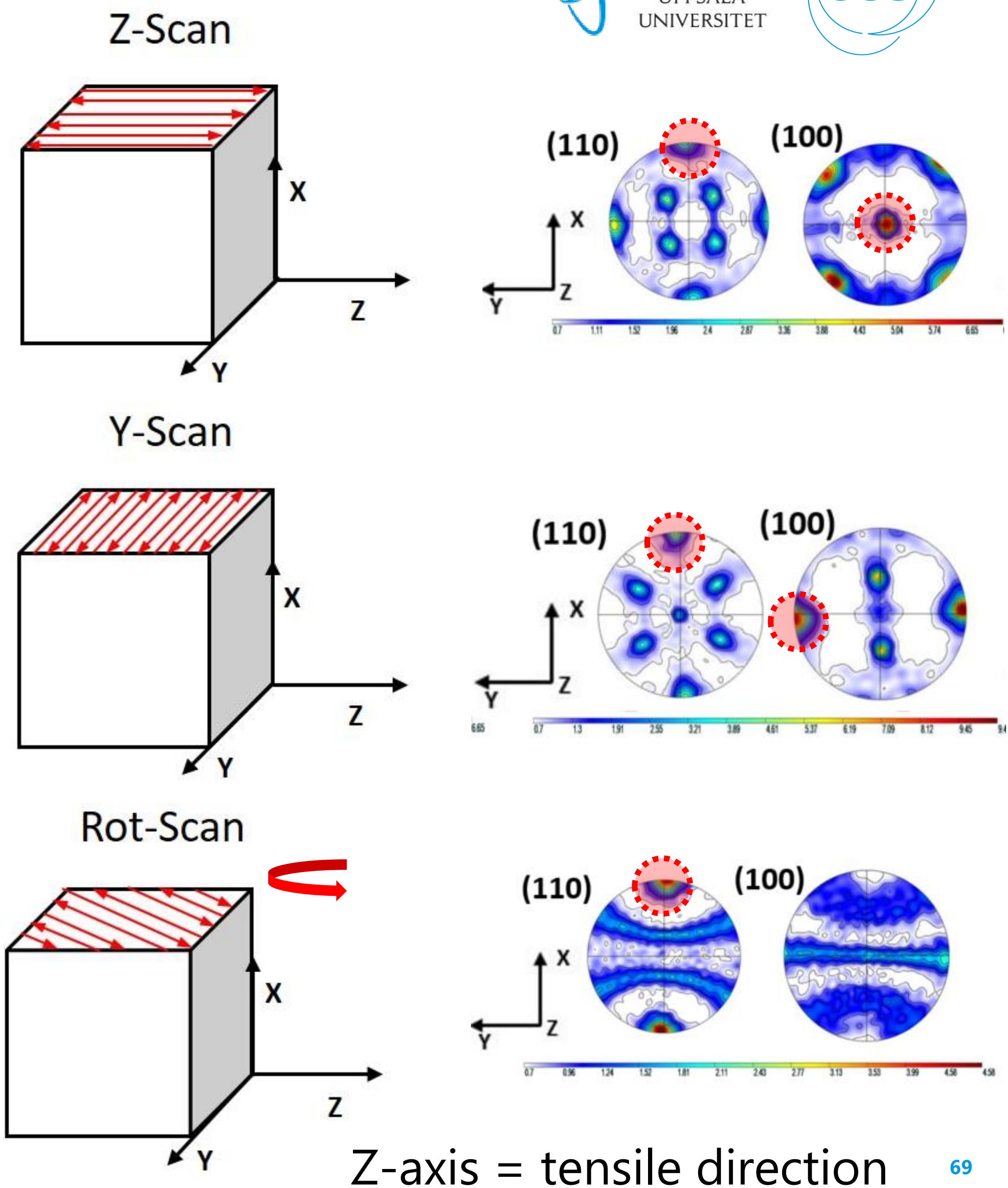
Texture in SLM 316L stainless steel

3 scan strategies

- Z-scan
- Y-scan
- 67°rotation

→ Crystallographic texture can be controlled by movement of laser

- Strong 〈100〉 // laser direction
- Strong 〈110〉 // building direction (x-axis)
- Rotation → fiber texture



Z-axis = tensile direction

J.J. Marattukalam et al., Mater. Des. 193 (2020).

Applications: Metallography

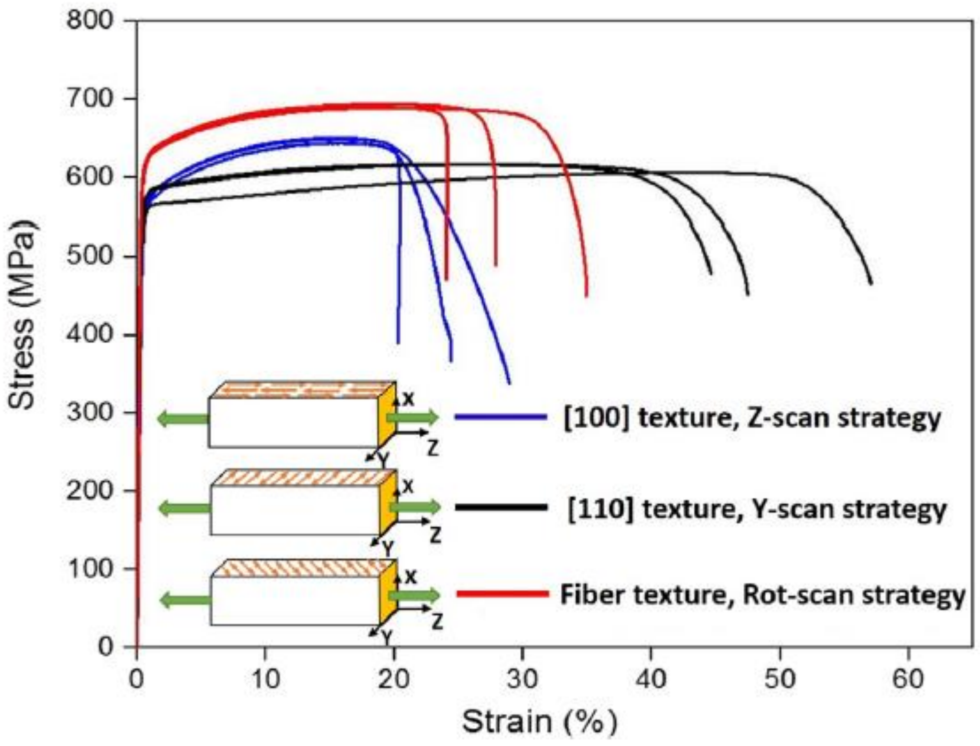
Texture and Mechanical Properties in SLM 316L stainless steel

3 scan strategies

- Z-scan
- Y-scan
- 67°rotation

Mechanical properties

- Hardness, E-modulus, yield strength and elongation influenced by microstructure and texture.
- Large ductility difference



→ Mechanical properties can be tuned with SLM printing parameters

Z-axis = tensile direction

J.J. Marattukalam et al., Mater. Des. 193 (2020).

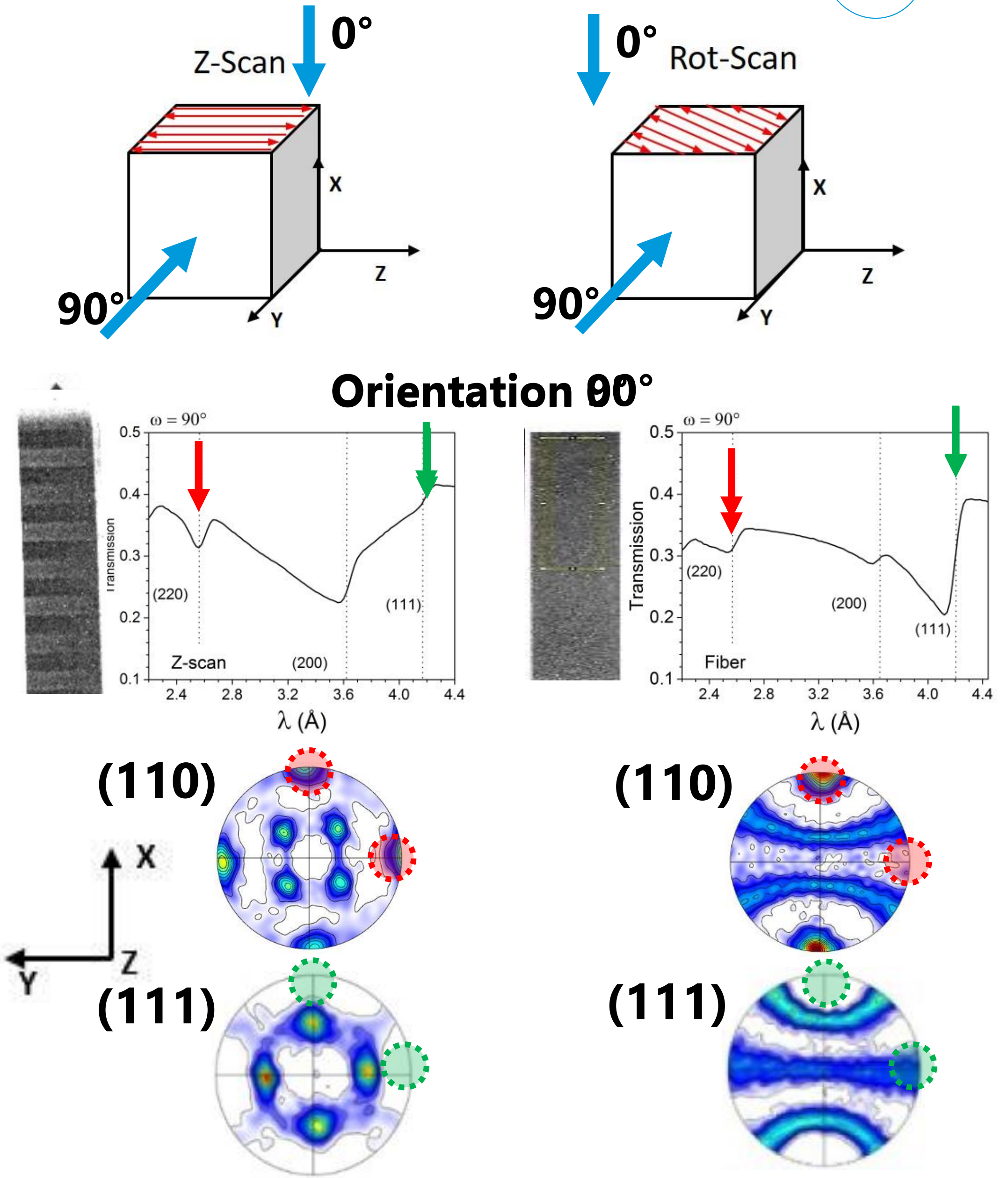
Applications: Metallography

Texture in SLM 316L stainless steel

Neutron Imaging: Wavelength scans at two sample orientations

- Bragg-edges at 0° orientation:
  - (220): strong for Z- and Rot-Scan
  - (111): weak for Z- and absent Rot-Scan
- Bragg-edges at 90° orientation:
  - (220): strong for Z- and weaker for Rot-Scan
  - (111): weak for Z- and strong for Rot-Scan

→ Imaging is consistent with neutron pole figures



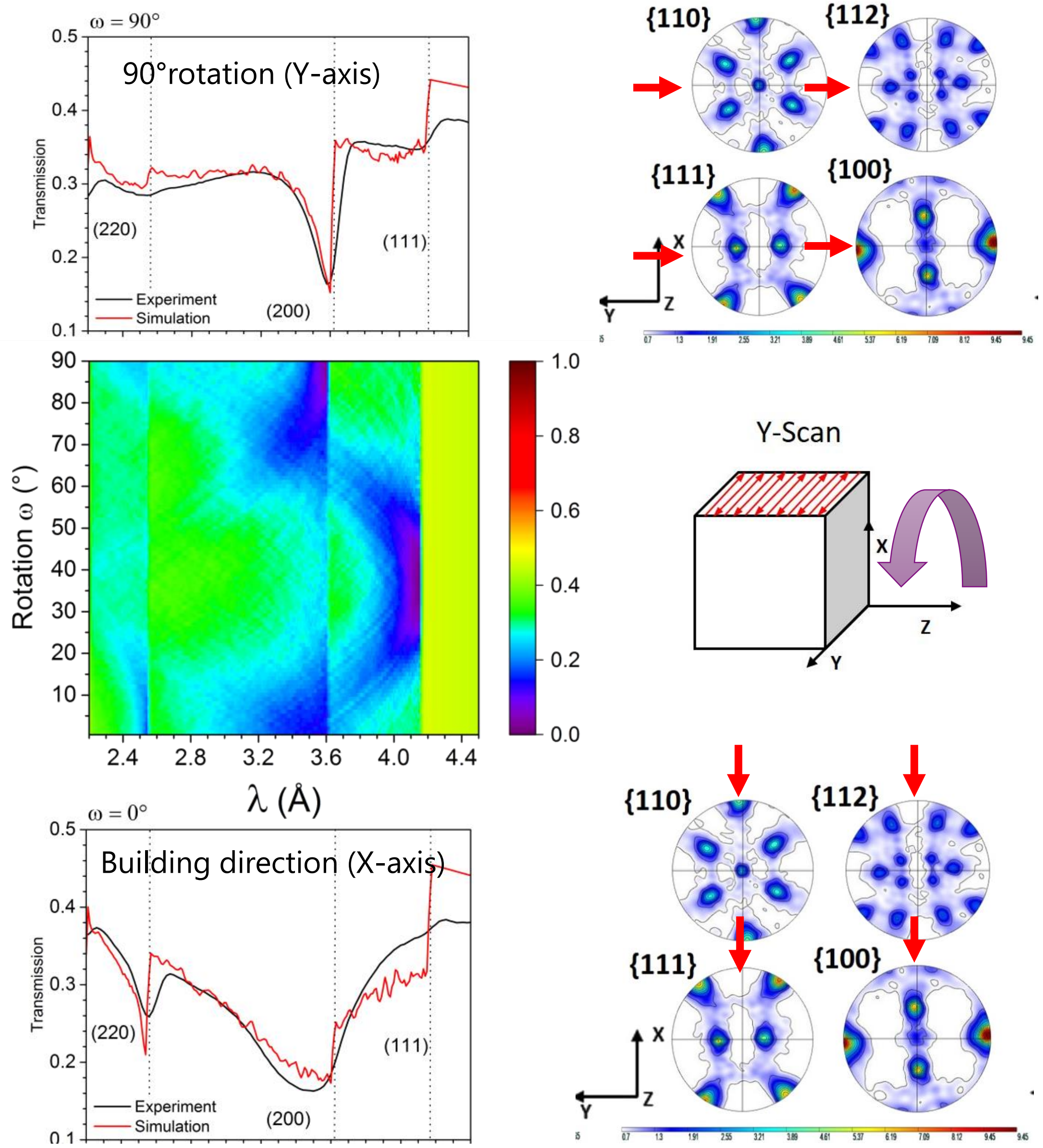
Applications: Metallography

Texture in SLM 316L stainless steel: Transmission simulations

Neutron transmission simulation (Sinpol) was used

- Neutron pole figures used as input
- Obtained simulated transmission map
- Y-Scan sample well described by ‘Goss’ {011}<100> texture

→ Good agreement between experiment and simulation



Dessieux, L. L., et al. "**Neutron transmission simulation of texture in polycrystalline materials**." Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms 459 (2019): 166-178.

Applications: Metallography

Localized Texture in SLM 316L stainless steel

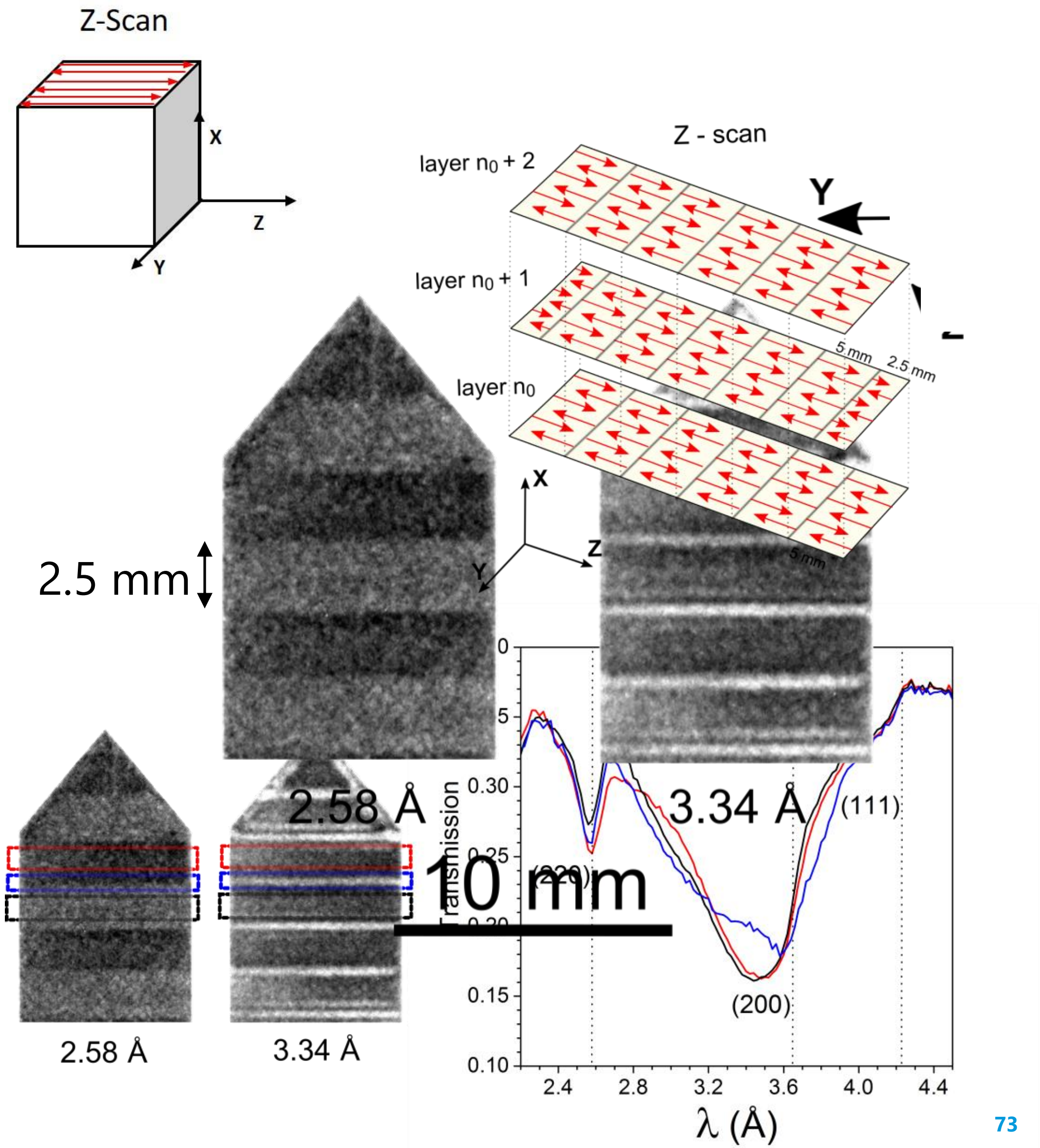
Neutron imaging revealed local differences

- Three different areas:
  - Bright and dark bands
  - Overlaps

→ Local variations. Why?

- Laser scan strategy introduced local texture differences

→ These local variations would easily remain undetected. Now use EBSD to investigate further.



Applications: Metallography

Localized Texture in SLM 316L stainless steel

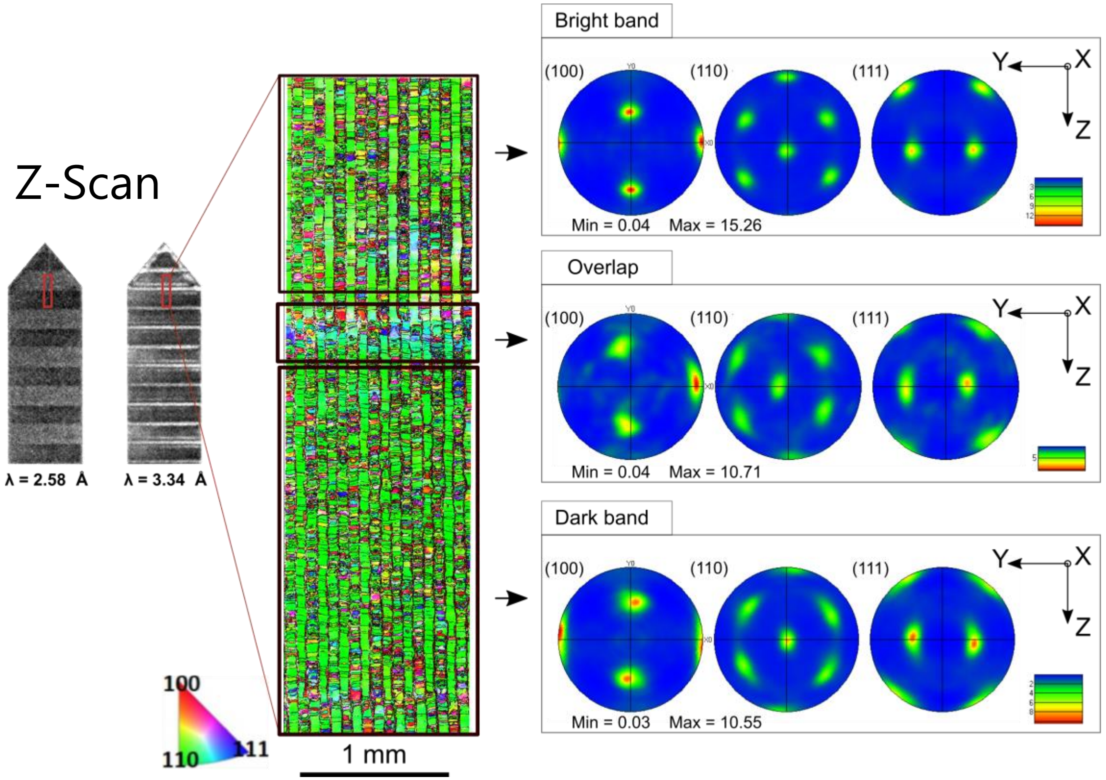
Neutron imaging revealed local differences

- Three different areas:
  - Bright and dark bands
  - Overlaps

→ Local variations. Why?

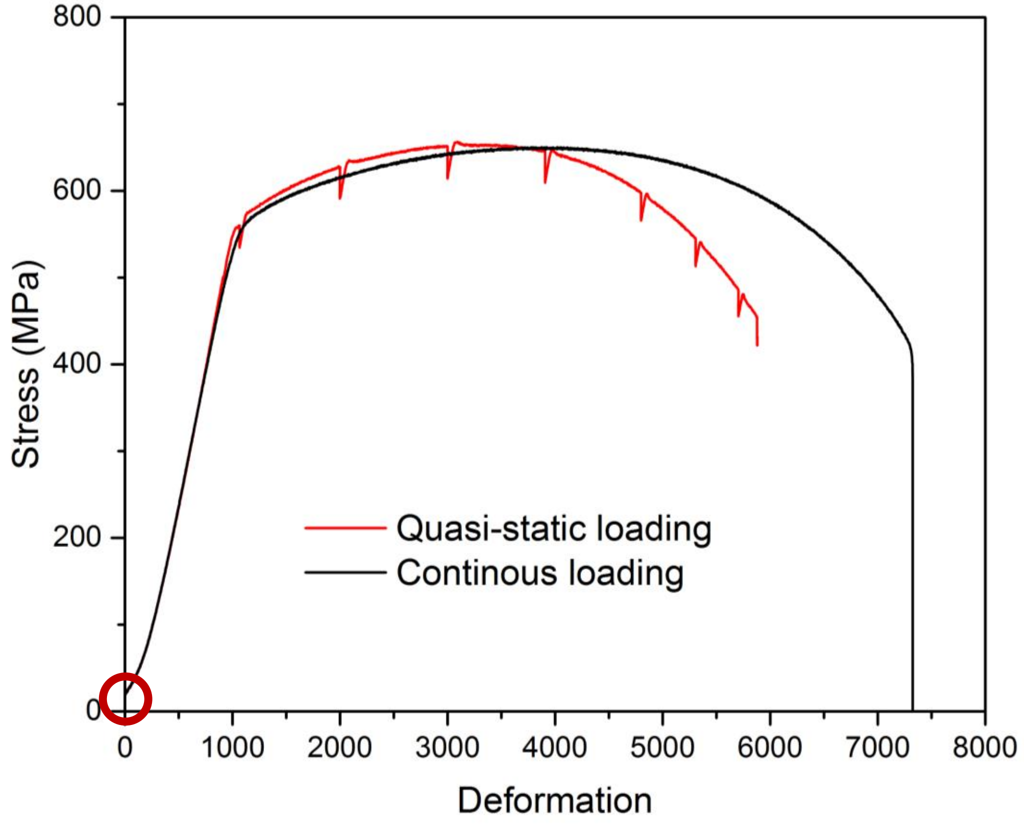
- Laser scan strategy introduced local texture differences

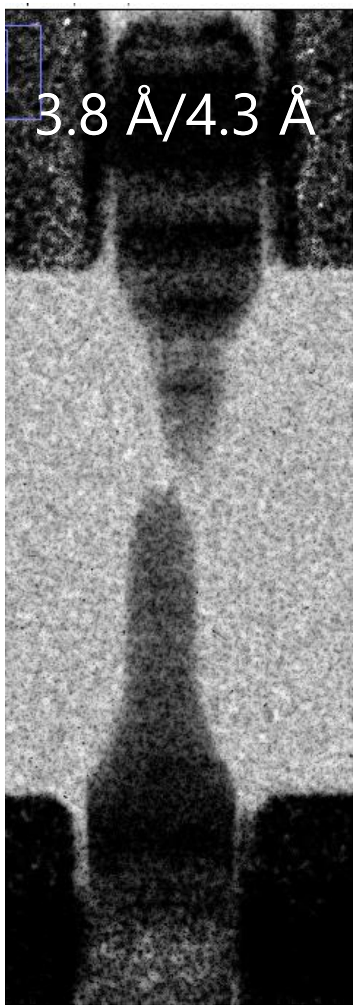
→ These local variations would easily remain undetected. Now use EBSD to investigate further.



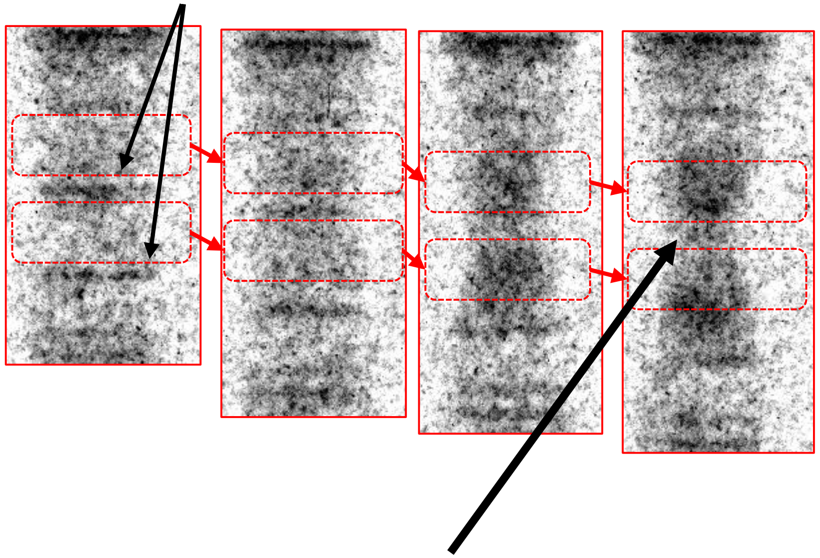
Applications: Metallography

In-situ tensile testing of SLM 316L stainless steel





Grain rotations cause reorientation of <111> direction



Failure occurs between these regions

Applications: Additive Manufacturing

Local texture variations revealed

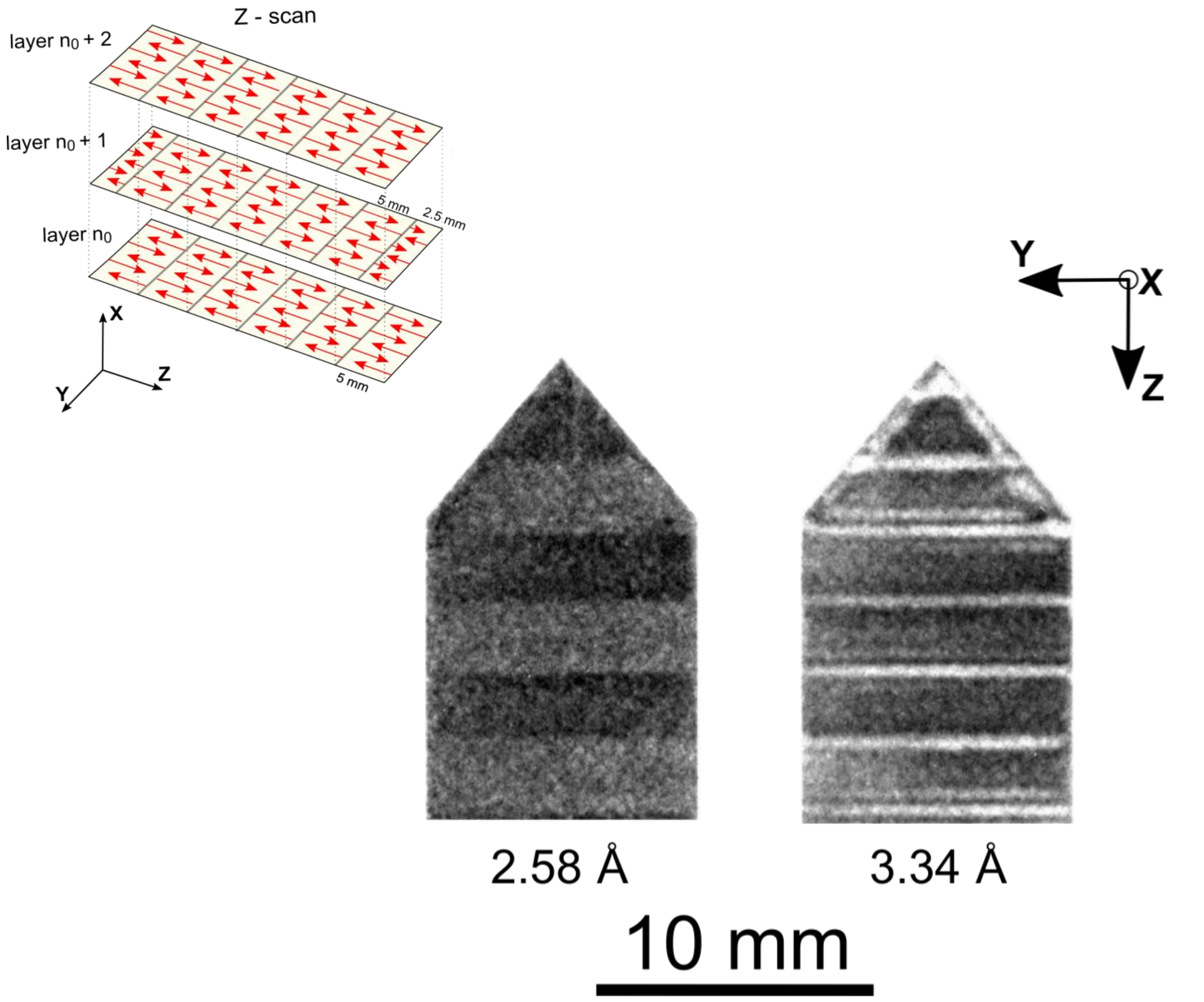
Additive Manufacturing:

fabrication methods based on **layer-by-layer** approach using a **digital model**

→ Complex designs

Selective laser melting is the most industrially matured technique

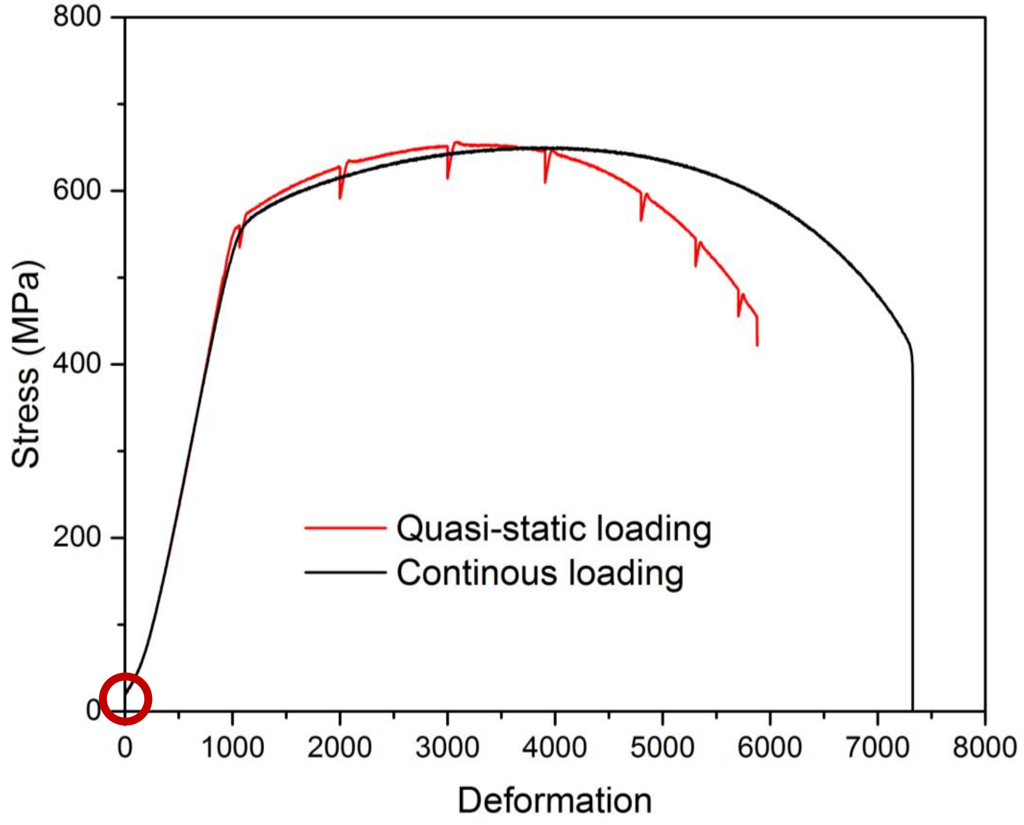
→ Neutron imaging revealed local texture differences due to Laser scan strategy

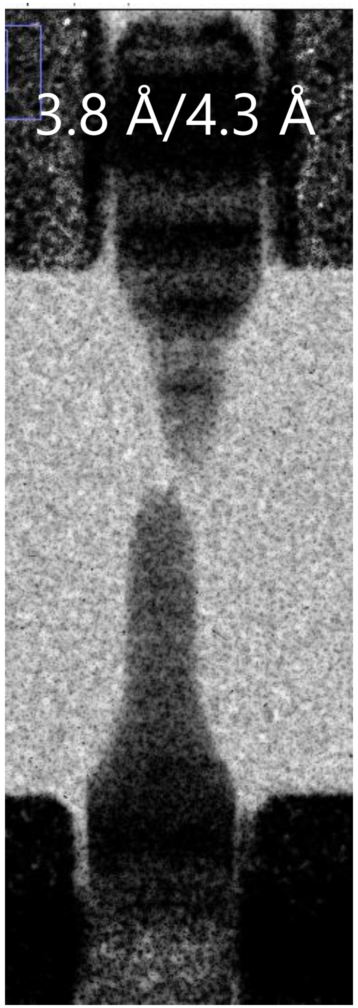


Pacheco, Woracek, et al., to be submitted

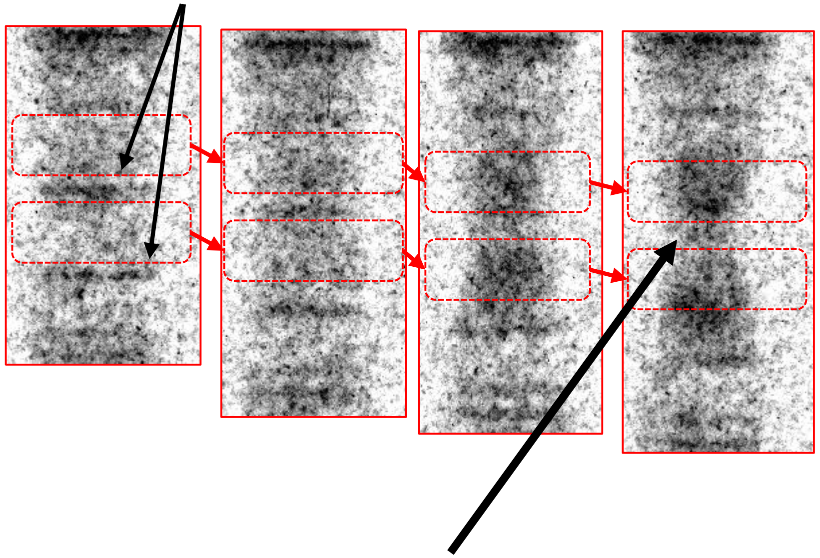
Applications: Additive Manufacturing

Local texture variations revealed





Grain rotations cause reorientation of <111> direction



Failure occurs between these regions

Pacheco, Woracek, et al., to be submitted

Non-destructive testing with neutrons: Engineering materials and components revealed

- ❑ Characterization Techniques & Contrast Mechanisms
- ❑ Neutron Methods & Length Scales
- ❑ Applications using Imaging (Attenuation Contrast)
- ❑ Applications using SANS and Diffraction
- ❑ Applications using Imaging (Diffraction and Scattering Contrast)
- ❑ Summary & Outlook

Summary & Outlook

Why you should start using neutrons …

- ✓ Neutrons are a non-destructive, penetrating probe of structure on the atomic to macroscopic scale.
- ✓ Neutrons provide chemical sensitivity being especially sensitive to light elements.
- ✓ Neutron imaging may help you to reveal local differences in the microstructure of your sample that otherwise remain undetected
- ✓ New techniques and instruments will enable unprecedented in-situ and in-operando studies.

Thank you! Any Questions?

Want to know more?

Website : **http://www.europeanspallationsource.se** Contact me : **robin.woracek@ess.eu**

ASP Online Seminar Series: Photons and Neutrons in the quest to solve societal challenges

Watch the recordings: https://www.youtube.com/cdarve

African School of Fundamental Physics and Applications

Accelerate Webinar Series – Watch the recordings: https://vimeo.com/acceleratehorizon2020

ACCELERATE

14th October :	Neutrons-A Natural Tool for Industrial Research Dr. Andrew Jackson
4th November :	Nanoscale to Microscale Structural Analysis with Neutrons Dr. Judith Houston
11th November :	Neutron protein crystallography reveals molecular details of inhibitor binding to clinical targets. Dr. Zöe Fisher
25th November :	Non-destructive testing with neutrons: Revealing (micro-) structural properties and providing unique contrast inside large samples and assembled components Dr. Robin Woracek