Neutron Imaging in Material Science
Application Examples

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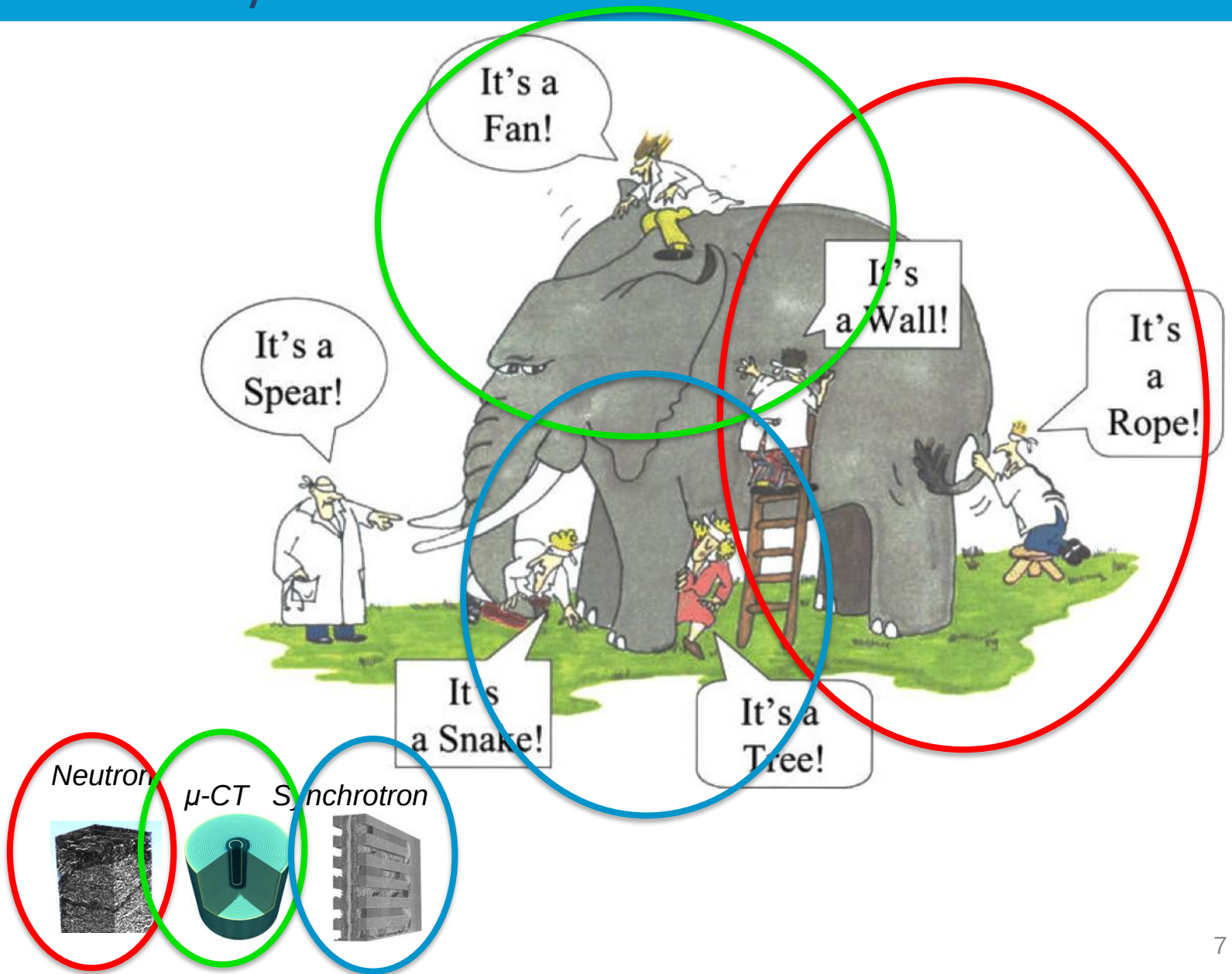
AGENDA

Neutron Imaging in Material Science

- **Intro**
- **Examples:** ‘White Beam’ Imaging
- **Examples:** Diffraction Contrast Imaging
- **Examples:** Phase Contrast Imaging
- **Examples:** Diffractive Imaging

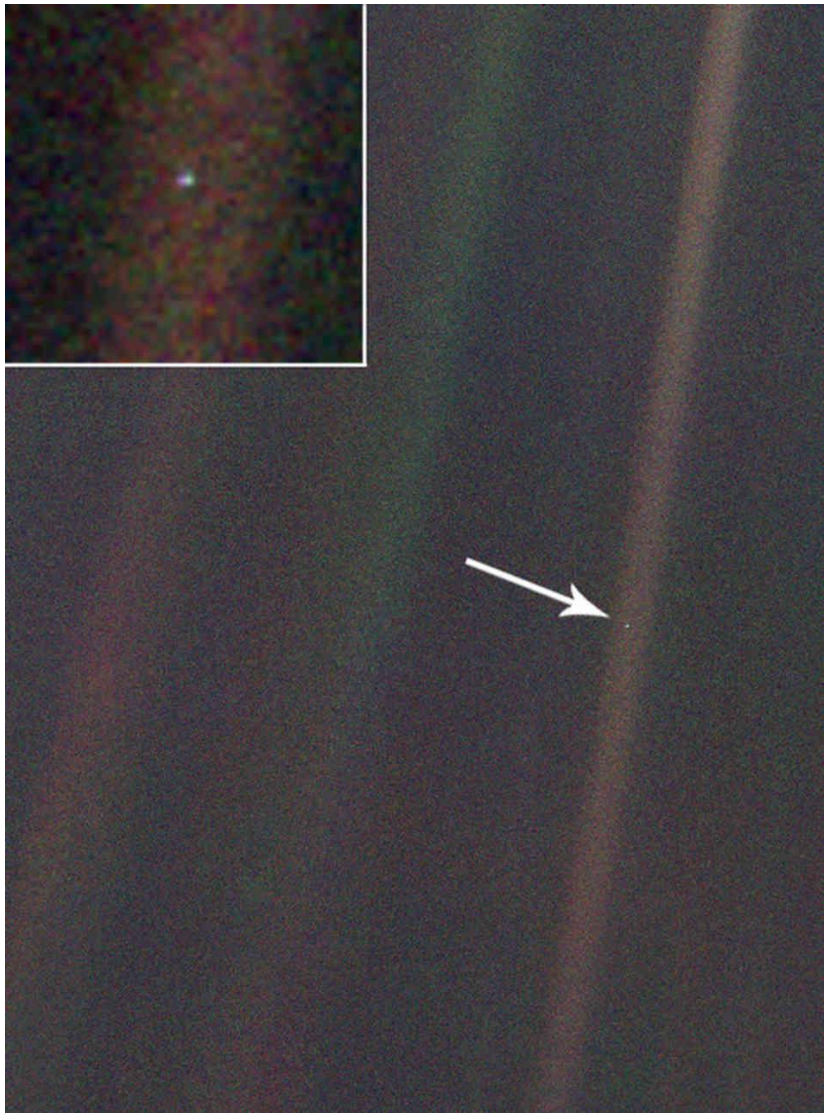
Introduction

Complementarity of methods…



Neutron Imaging

Spatial resolution



The ‘Pale Blue Dot’

Photograph of planet Earth taken on February 14, 1990, by Voyager 1.
Distance: 6 billion km

In the photograph, Earth's apparent size is less than a pixel.

Spatial resolution is not the only thing that is important. Uniqueness of contrast and information!

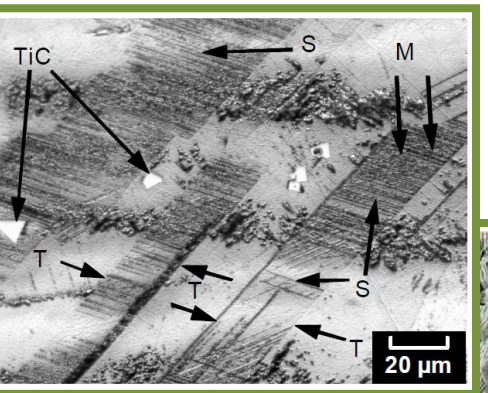
Moreover: Use the synergy of neutrons and x-rays (and other methods) to complement contrast and spatial resolution

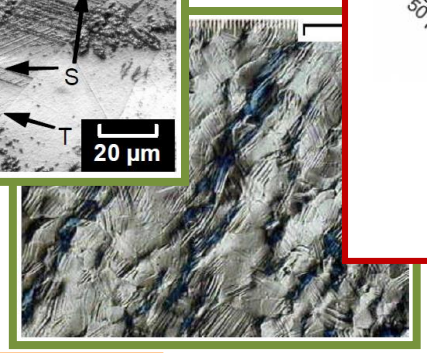
Neutrons for Material Science

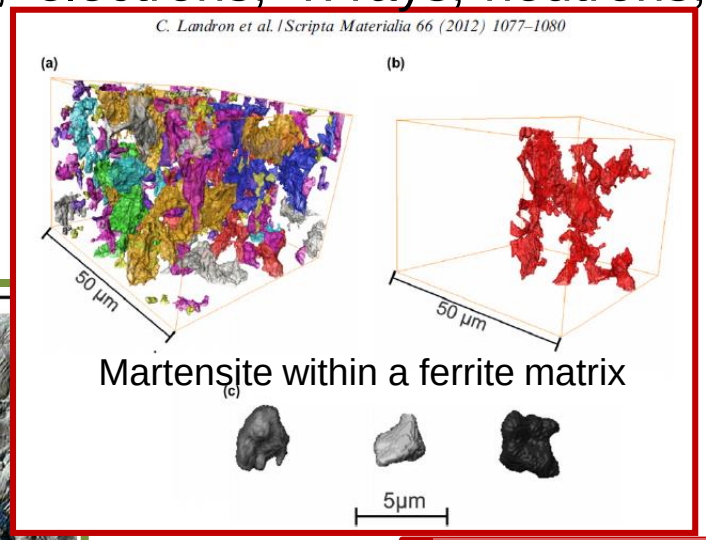
Bridging the gap towards the meso and macroscale

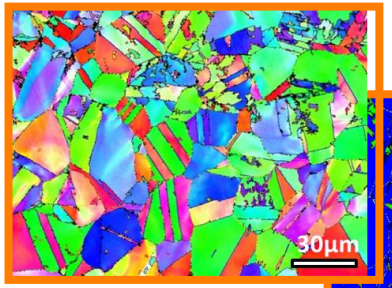
- Different methods provide complementary information and cover various length scales….

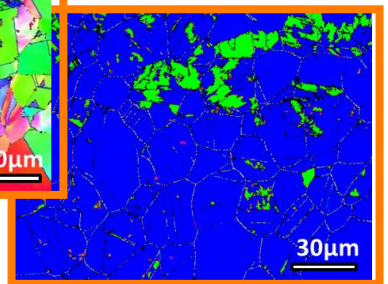
optical microscopy, electrons, x-rays, neutrons, …

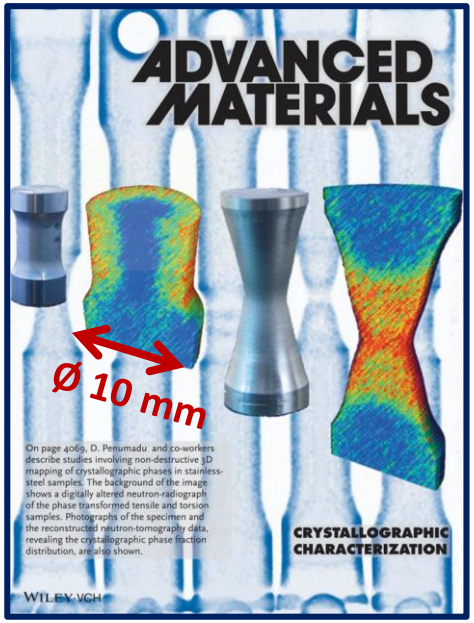








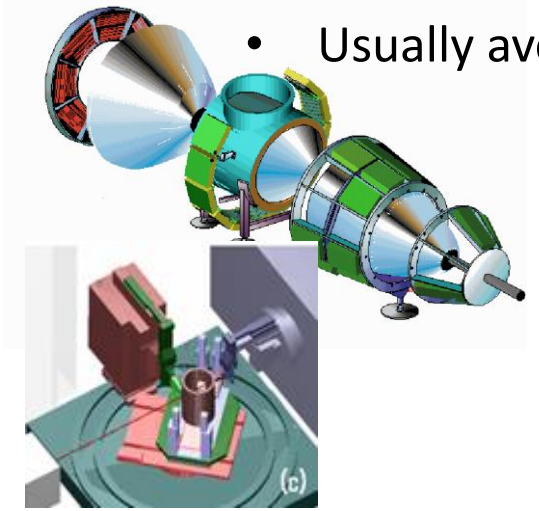


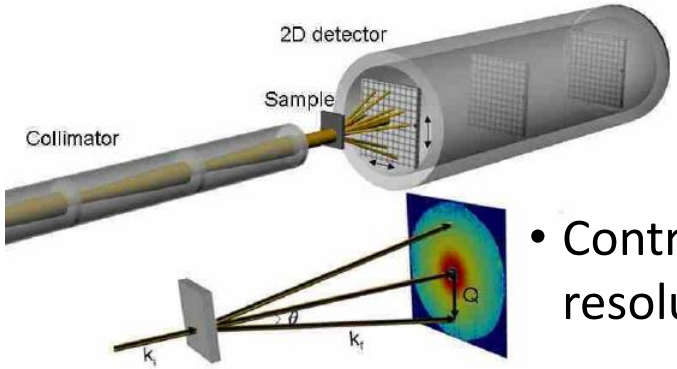


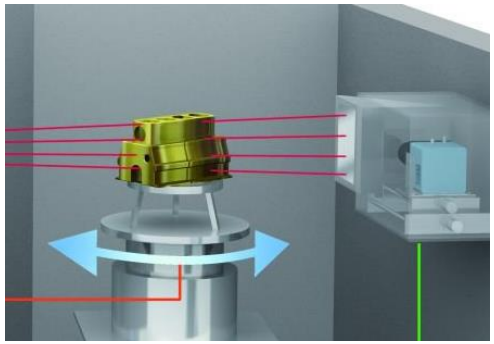
Neutrons for Material Science

Spatial resolution vs 'spatial sensitivity'

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







- Contrast from features (>spatial resolution) resolved in real space

Diffraction regime

SANS regime

Length scale in nm

0.01 0.1 0.3 1.0 3.0 10 100 1000 10000 100000

M. Strobl & F. Grazzi (2015) From scattering in imaging to prospects at pulsed sources, Neutron News 26

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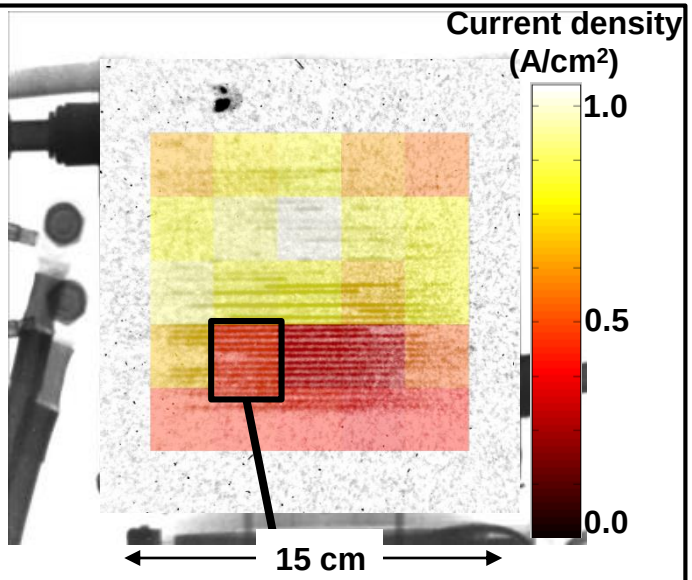
Example 1: Attenuation Contrast

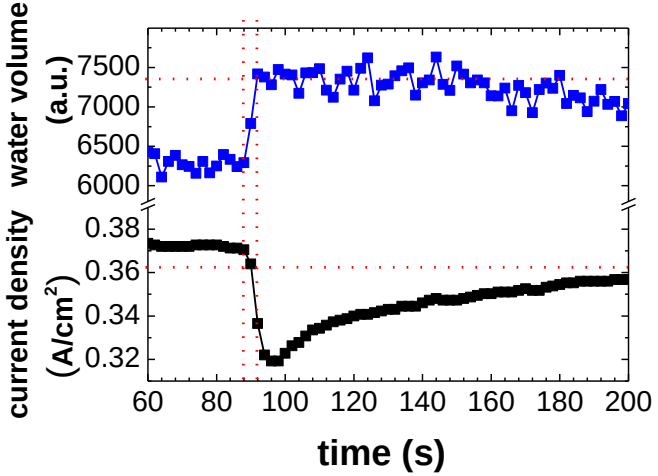
Fuel cells

Courtesy of N. Kardjilov

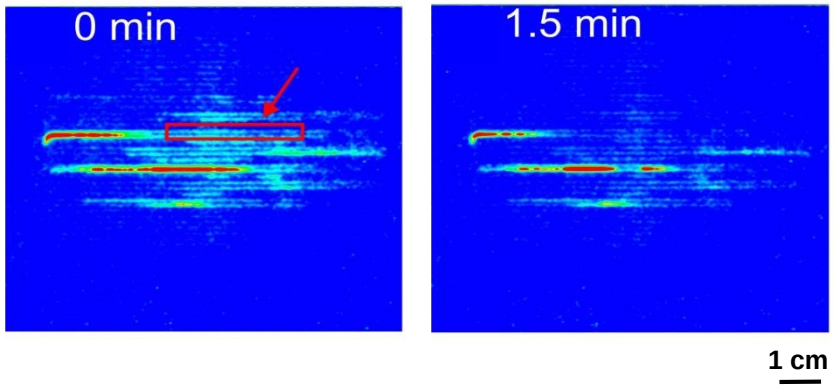
HZB Helmholtz Zentrum Berlin

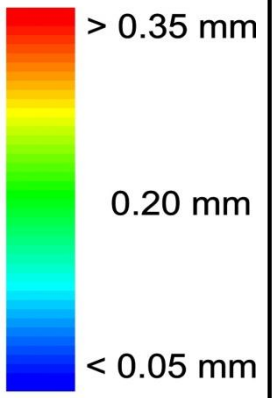
ESS EUROPEAN SPALLATION SOURCE

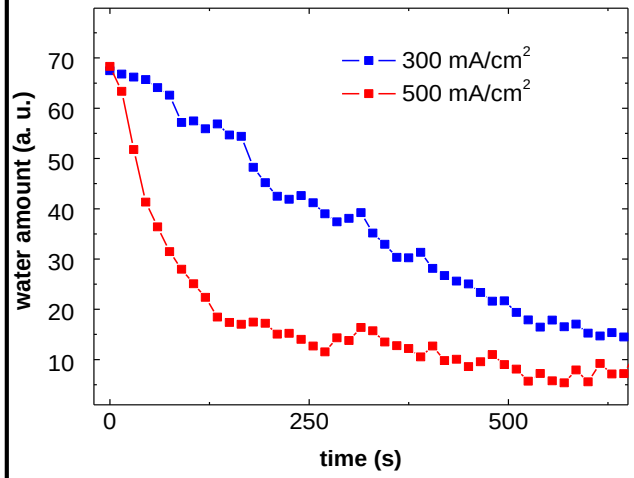




H_2/D_2 contrast radiography







I. Manke et al, APL 92, (2008)

A.Schröder et al, Electrochem Commun (2009)

A. Schröder et al, J Power Sources 195 (2010)

C. Tötzke et al, J Power Sources 196 (2011),

A. Lange et al, J Power Sources 196 (2011)

R. Kuhn et al, Int J Hydrogen Energy 37 (2012)

H. Markötter et al, J Power Sources 219 (2012)

ZSW

JÜLICH FORSCHUNGSZENTRUM

Example 2a: Attenuation Contrast

Hydrogen Storage

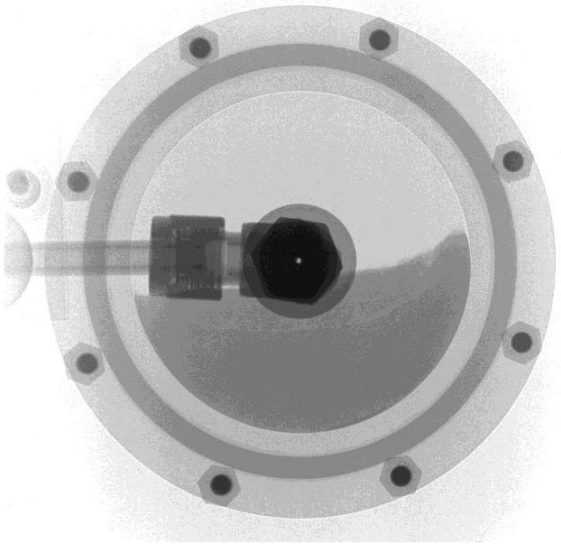
Courtesy of N. Kardjilov

HZB Helmholtz Zentrum Berlin

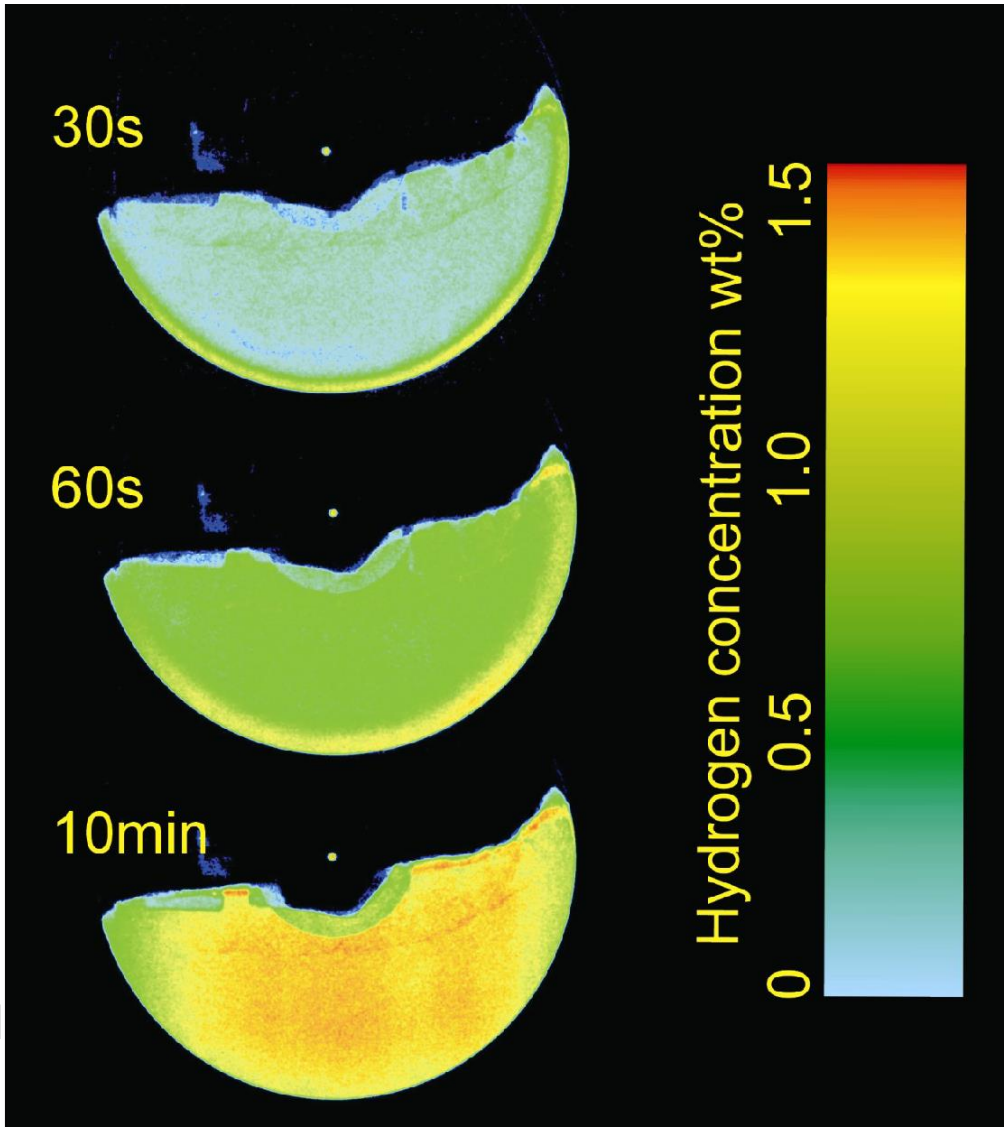
ESS EUROPEAN SPALLATION SOURCE

11 bar of H_2

$(LaNi_{4.8}Al_{0.2})$



Fraunhofer IFAM AGH



L. Gondek et al.,**International Journal of Hydrogen Energy** 36 (2011)

Example 2b: Attenuation Contrast

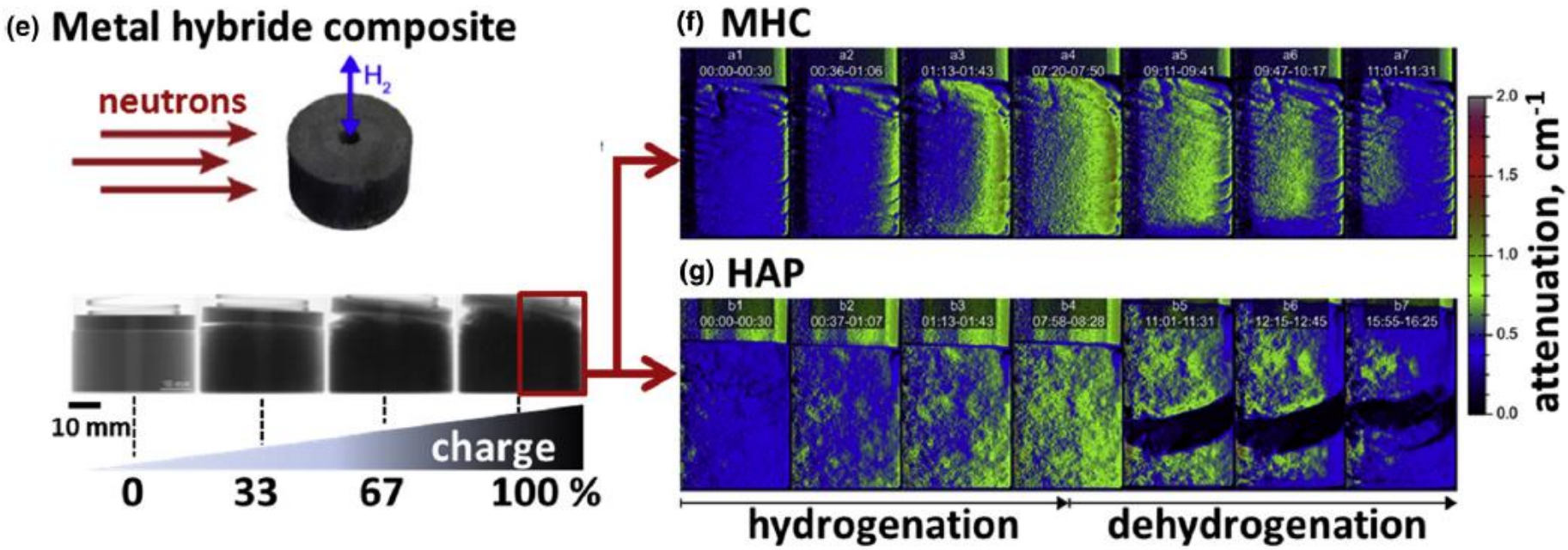
Hydrogen Storage

Courtesy of N. Kardjilov

HZB Helmholtz Zentrum Berlin

ESS EUROPEAN SPALLATION SOURCE

Metal hydride storage materials were investigated during cyclic hydrogenation and dehydrogenation. A general view of the hydrogenation process in a metal hydride composite (MHC) is shown in 4 radiographs.



(f and g) spatio-temporal hydrogen concentrations in a MHC of 40-mm diameter compared to loose hydride–alumina powders (HAPs) both contained in a cylindrical container were studied to evaluate their structural stability.

K. Herbrig et al., J. Power Sources 293 (2015) 109.

C. Pohlmann et al., J. Power Sources 277 (2015) 360.

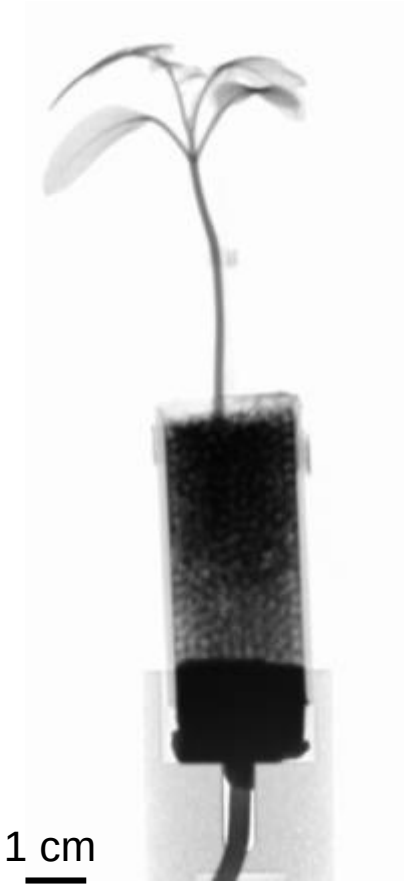
Example 3: Attenuation Contrast

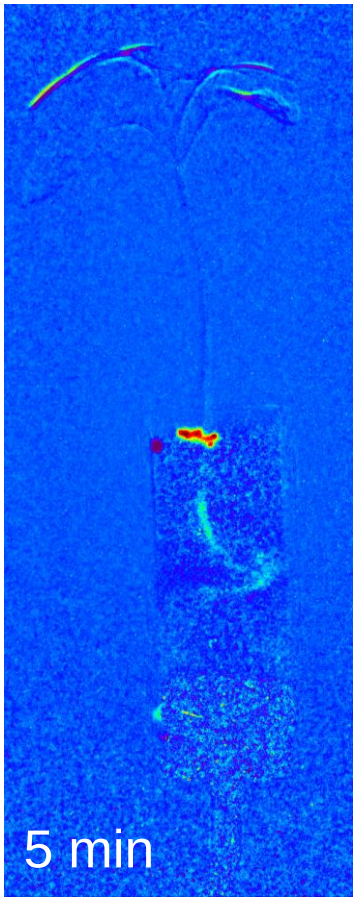
Plants

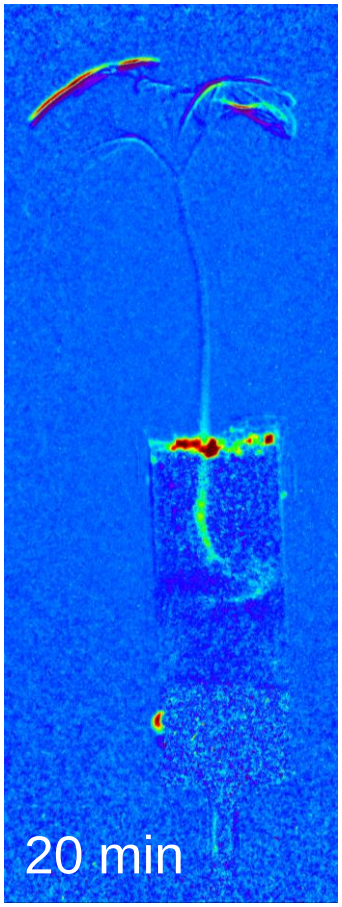
Courtesy of N. Kardjilov

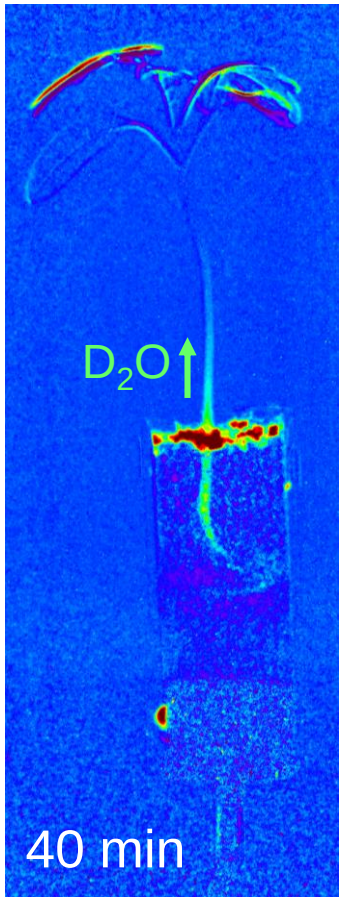
HZB Helmholtz Zentrum Berlin

ESS EUROPEAN SPALLATION SOURCE









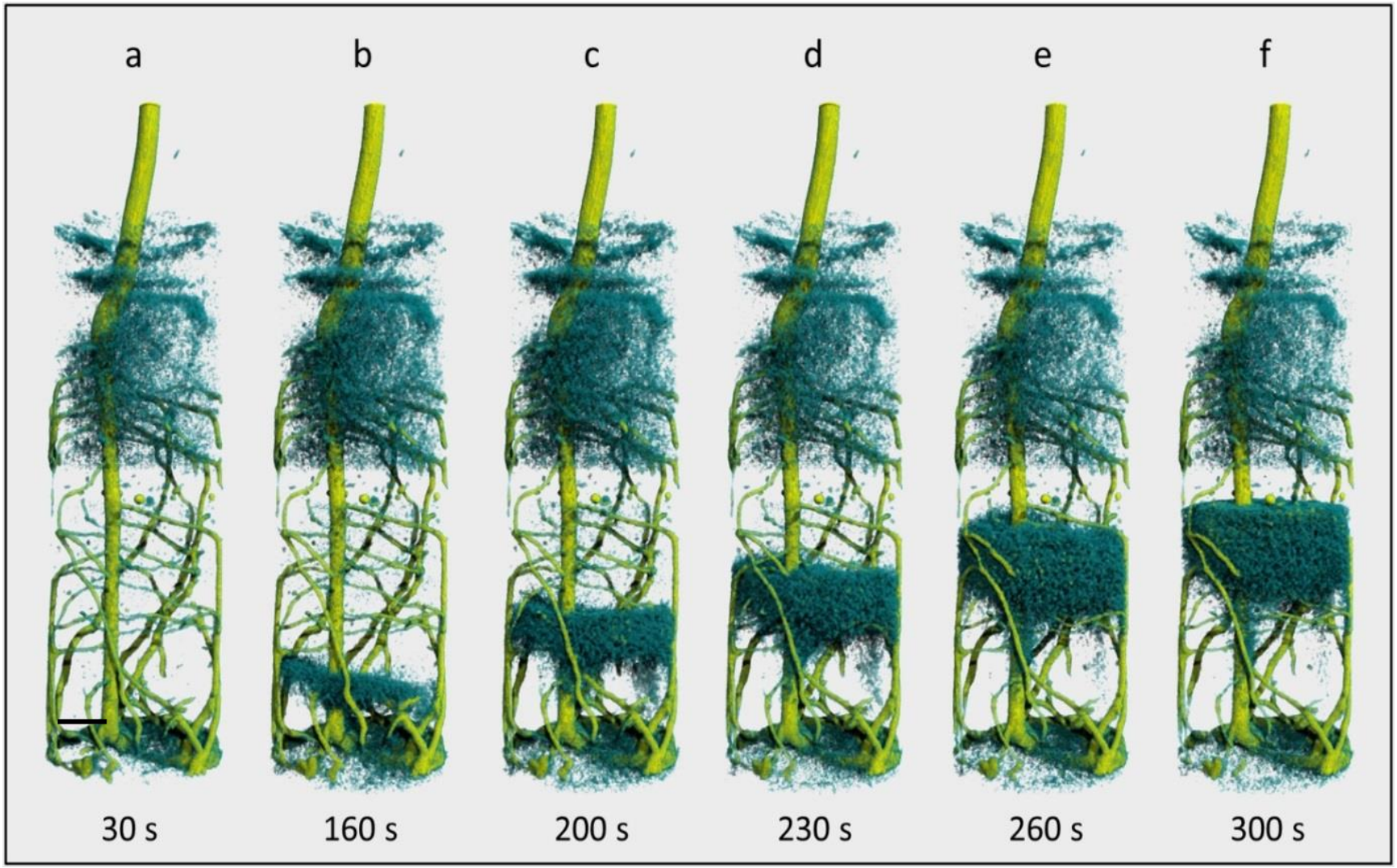
H_2O / D_2O

Tomato seedling

Matsushima, U., et al. "Application potential of cold neutron radiography in plant science research." *Journal of applied botany and food quality* 82.1 (2008): 90-98.

Example 3b: Attenuation Contrast

Plants



Tötzke, C., Kardjilov, N., Manke, I., & Oswald, S. E. (2017). Capturing 3D water flow in rooted soil by ultra-fast neutron tomography. *Scientific reports*, *7*(1), 6192.

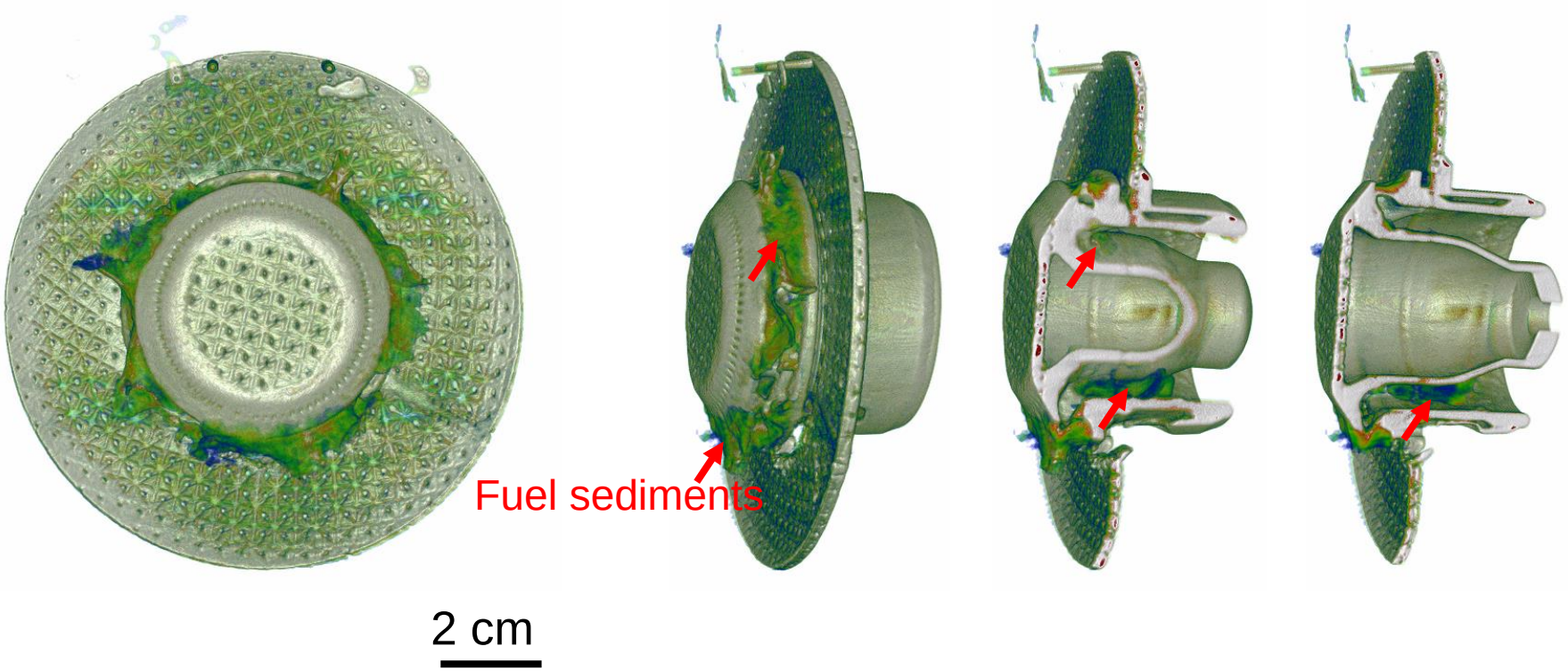
Example 4: Attenuation Contrast

Tomography of Jet Boat Engine

Courtesy of N. Kardjilov

HZB Helmholtz Zentrum Berlin

ESS EUROPEAN SPALLATION SOURCE



Example 5: Attenuation Contrast

Corrosion in cultural heritage objects

Courtesy of PAUL SCHERRER INSTITUT PSI

ess EUROPEAN SPALLATION SOURCE

Lead/Wood sculpture “the violinist”
by Pablo Gargallo
MNAC, Barcelona
Dimensions: 55 cm x 32 cm x 22 cm

2 section-tomography

Mannes et al. (2014) INSIGHT, 56 (3): 137-141

Example 5: Attenuation Contrast

Corrosion in cultural heritage objects

Courtesy of PAUL SCHERRER INSTITUT PSI

ess EUROPEAN SPALLATION SOURCE

areas affected by corrosion (red)
fixation by nails and soldering (blue)

Mannes et al. (2014) INSIGHT, 56 (3): 137-141

Example 7a: Attenuation Contrast

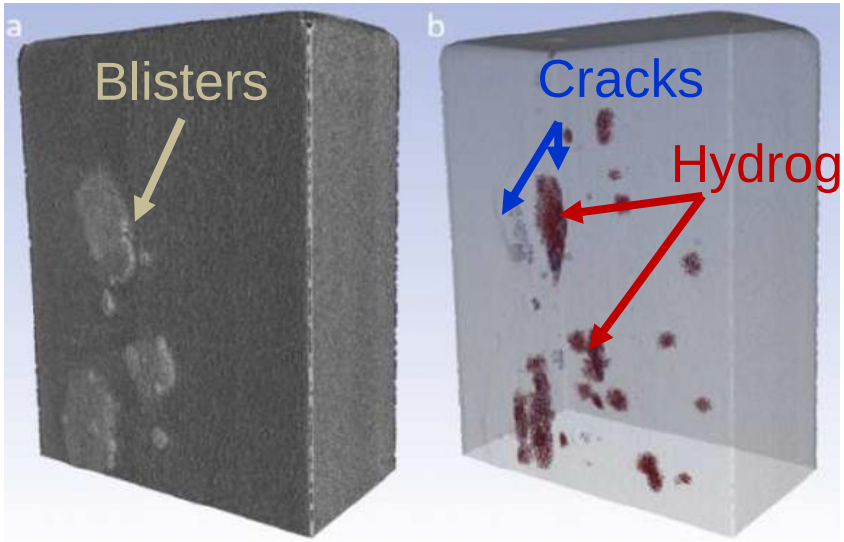
Hydrogen blistering + embrittlement in metals

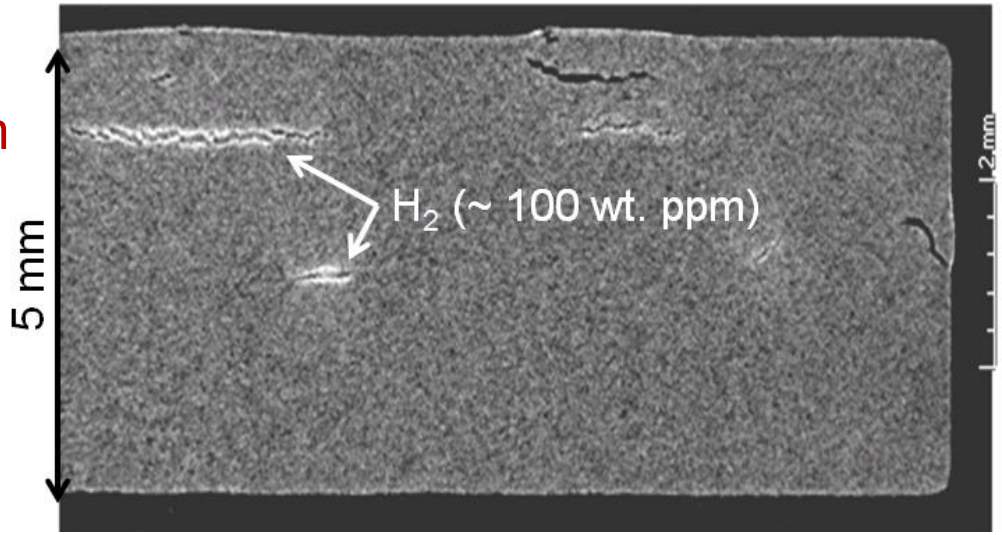
EUROPEAN SPALLATION SOURCE

HZB Helmholtz Zentrum Berlin

BAM Federal Institute for Materials Research and Testing

Hydrogen charged ARMCO (technical iron) sample





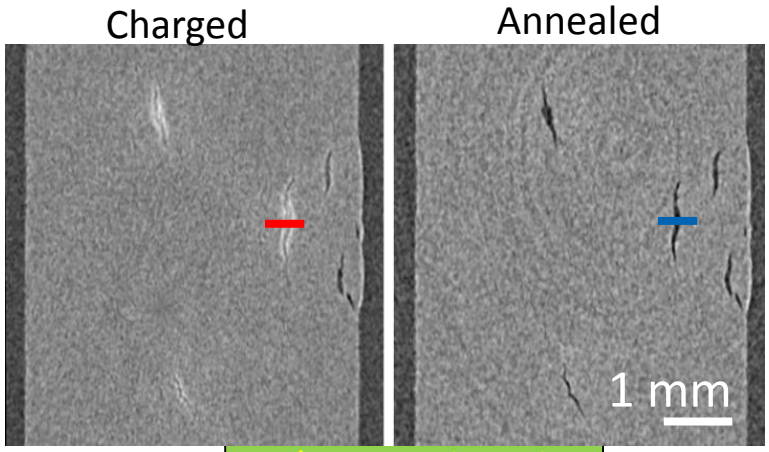
Resolution: 15 µm (pixel size: 6.5 µm), FOV: 13 x 13 mm^2, 600 proj. x 60 s

Electrochemically loaded

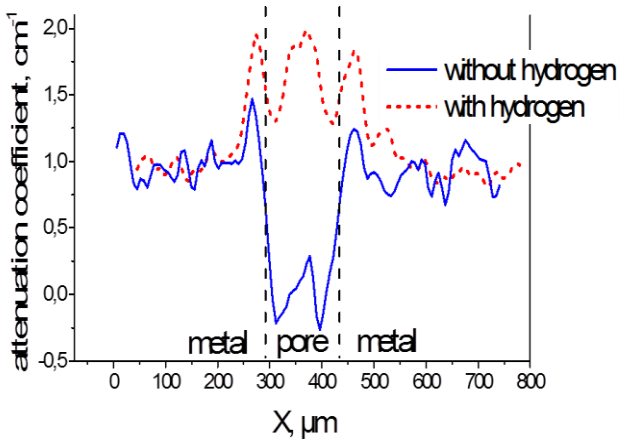
A. Griesche et al., **Acta Materialia 78** (2014)

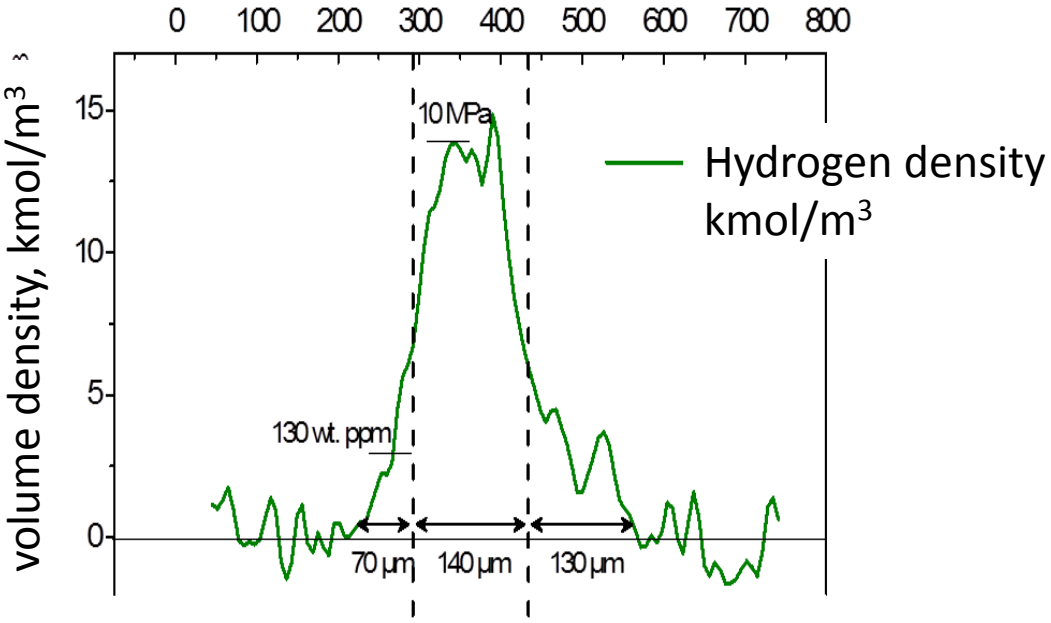
Example 7a: Attenuation Contrast

Hydrogen blistering + embrittlement in metals



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





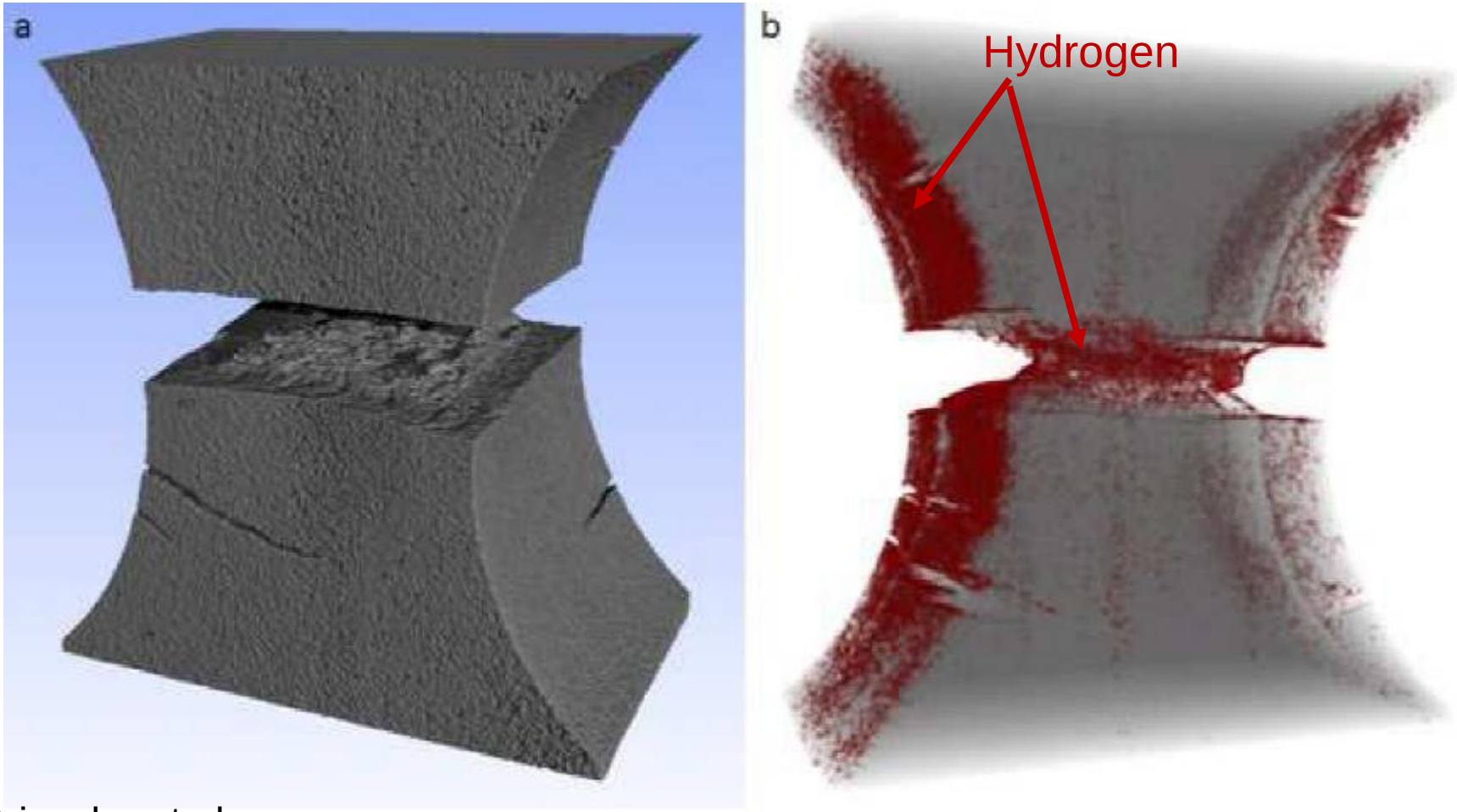
A. Griesche et al., **Acta Materialia 78** (2014)

Example 7b: Attenuation Contrast

Hydrogen blistering + embrittlement in metals

EUROPEAN SPALLATION SOURCE

Hydrogen charged supermartensitic tensile sample



A. Griesche et al

Example 7c: Attenuation Contrast

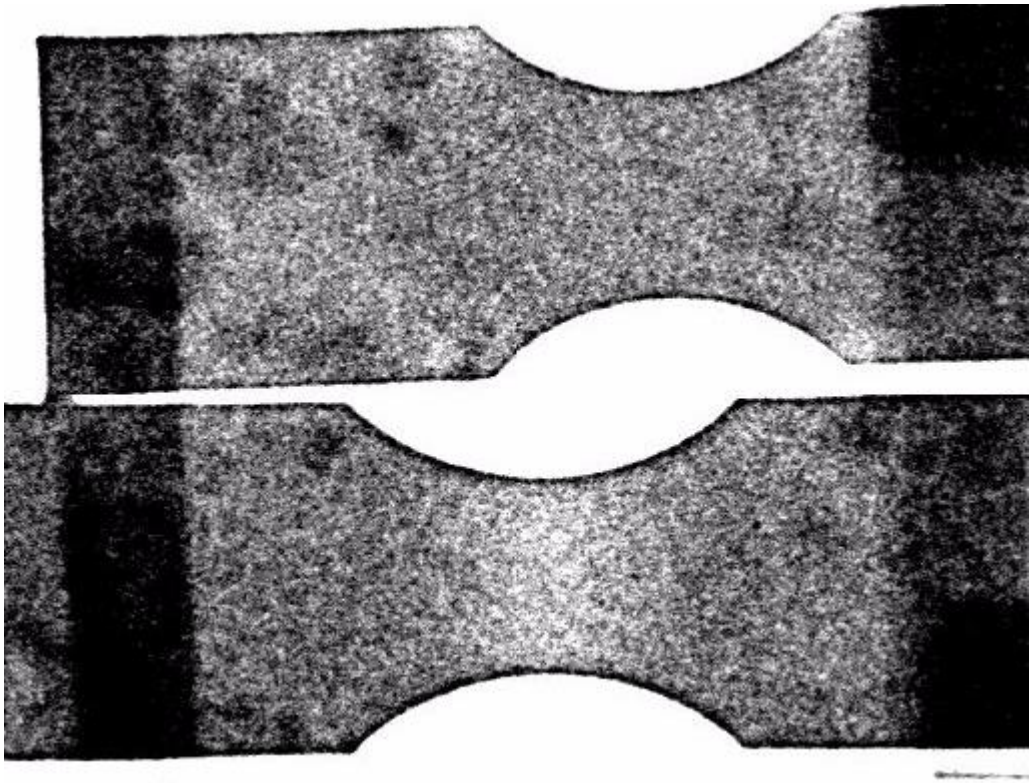
Hydrogen blistering + embrittlement in metals

EUROPEAN SPALLATION SOURCE

In-situ hydrogen diffusion in iron during annealing: Video

Sample A

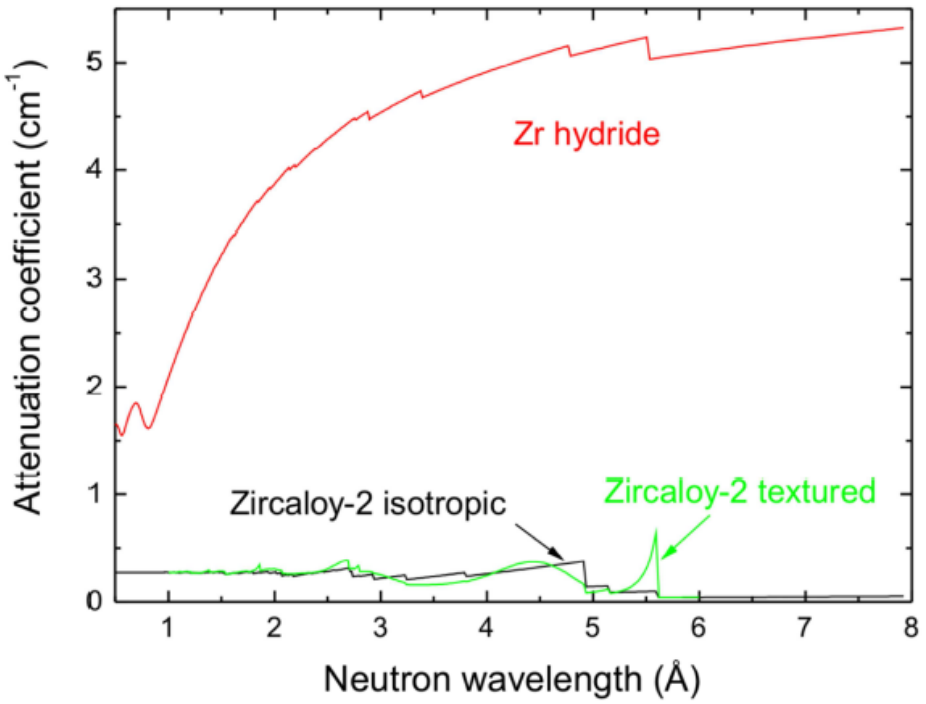
Sample B



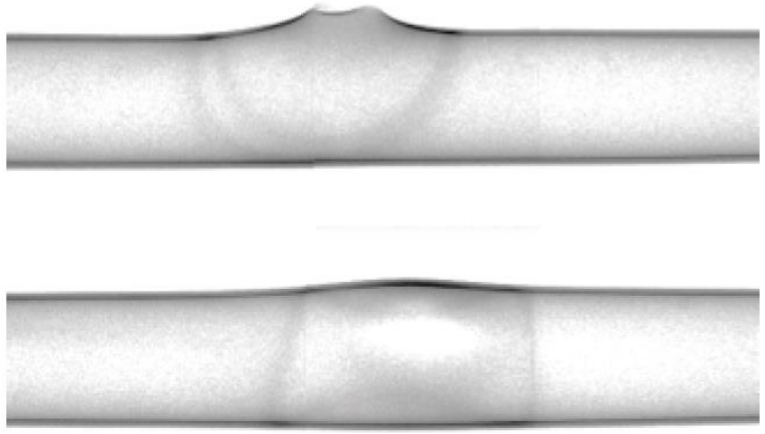
R Woracek et al, unpublished

Example 7d: Attenuation Contrast

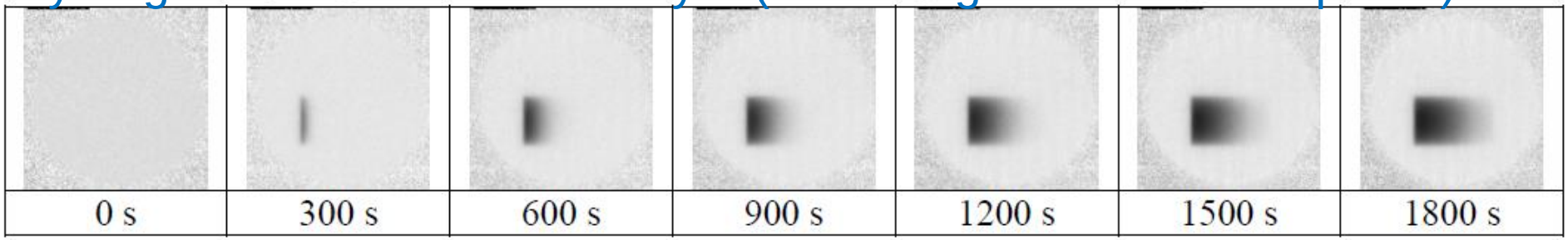
Hydrogen in Zirconium



hydrogen distribution around burst zone of fuel rod simulator cladding



Hydrogen diffusion into Zircaloy-4 (annealing in Ar/H2 atmosphere)



Grosse, M. (2015). Quantitative analysis of hydrogen uptake, diffusion and distribution in nuclear fuel rod claddings made of zirconium alloys. Neutron News, 26(2), 31-33.

Example 7e: Attenuation Contrast

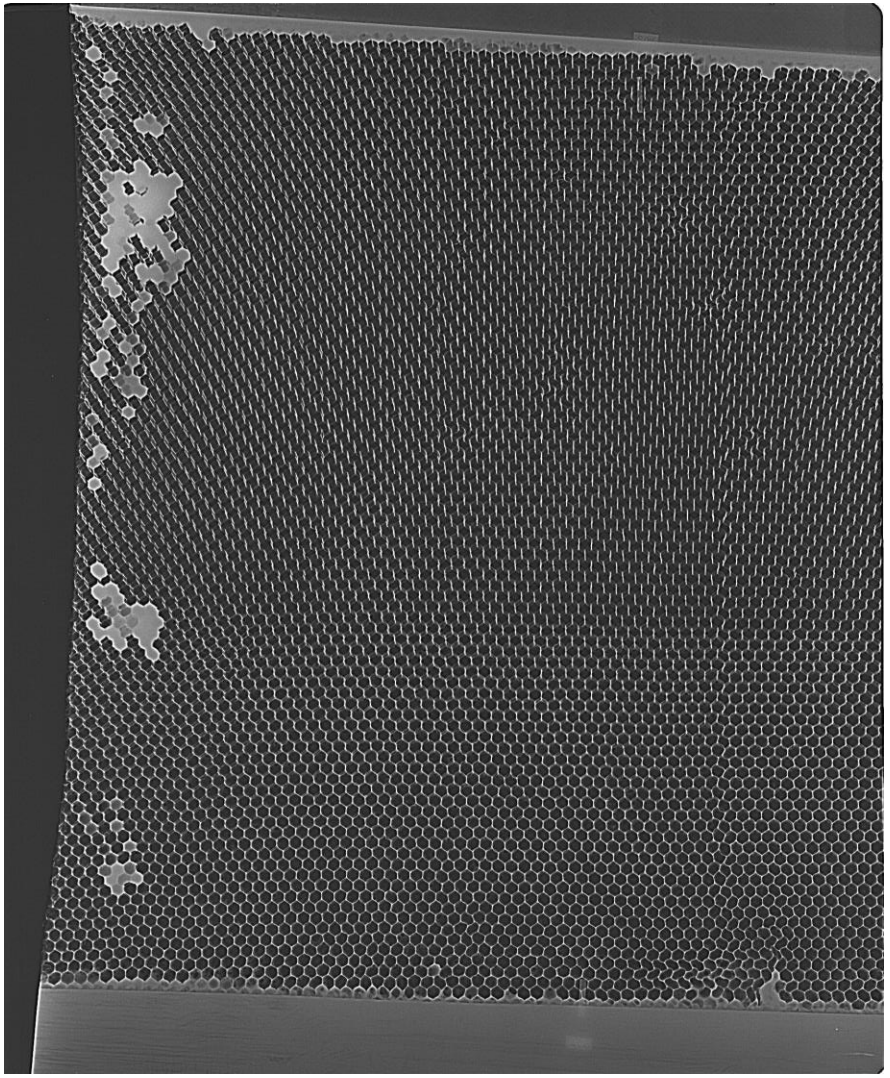
Applications to Composite Materials

EUROPEAN SPALLATION SOURCE

Blade surface can delaminate

Open honeycomb structure attracts moisture

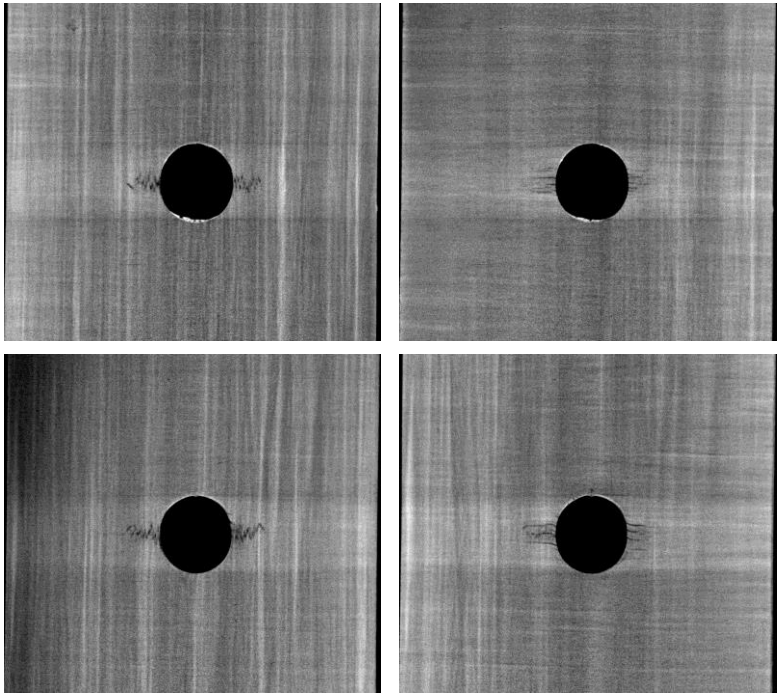
Blade tips may go supersonic thus leading to an explosive vaporization of the water



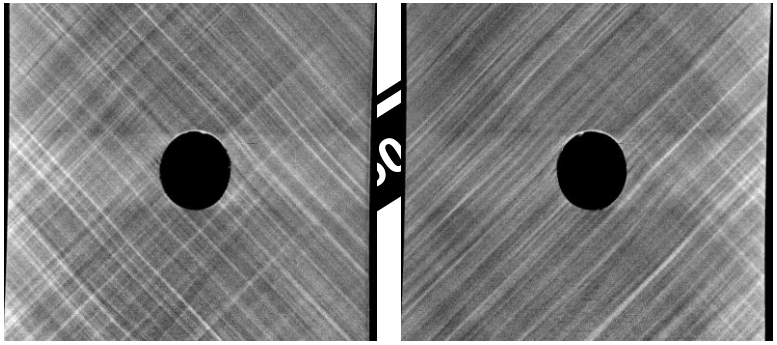
Example 7e: Attenuation Contrast

Applications to Composite Materials

Beim Airbus A380 kommt *Glare* großflächig zum Einsatz.



5/4 layers, 0° fiber angle, 97% ultimative load



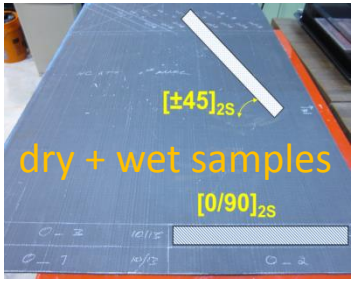
5/4 layers, 45° fiber angle, 97% ultimative load

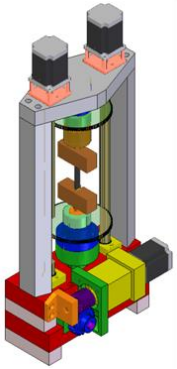
PAUL SCHERRER INSTITUT
PSI

Example 7e: Attenuation Contrast

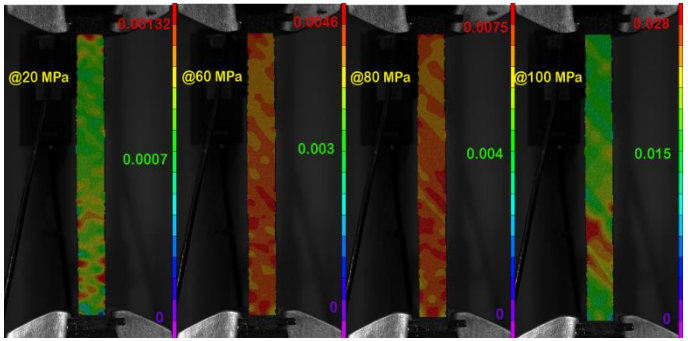
Applications to Composite Materials

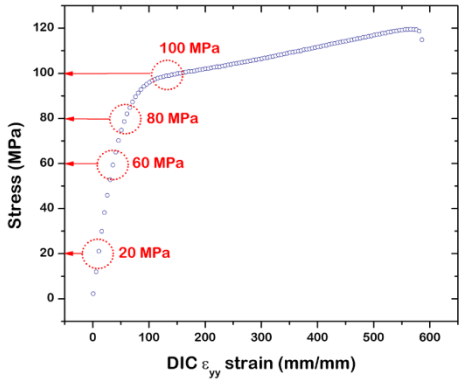
- In-situ loading and tomography



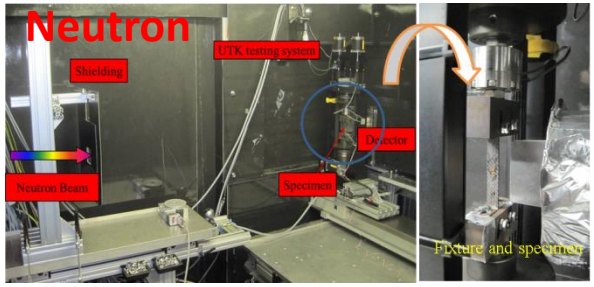


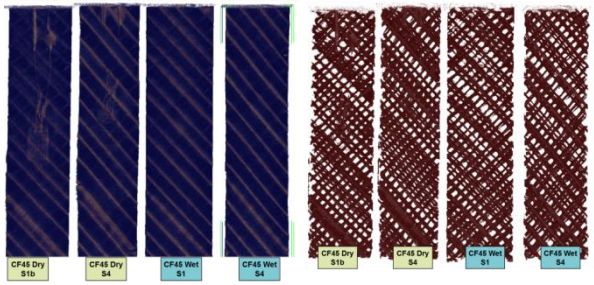
- Surface strains by DIC



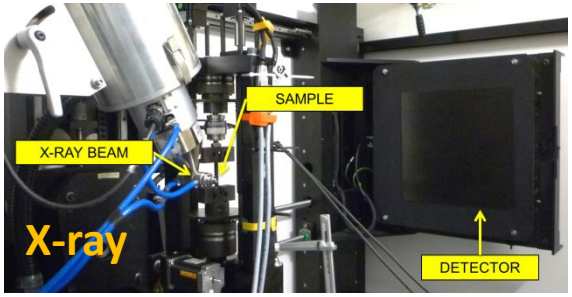


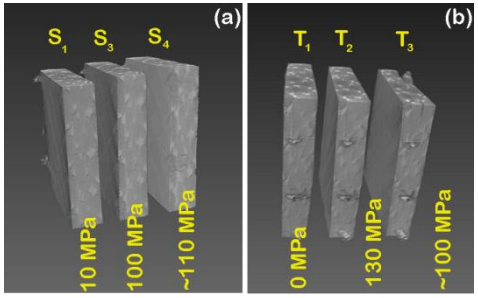
- Combined use of Neutron and X-ray tomography & Image registration





→ Visualization of moisture





→ Details of matrix and delamination effects

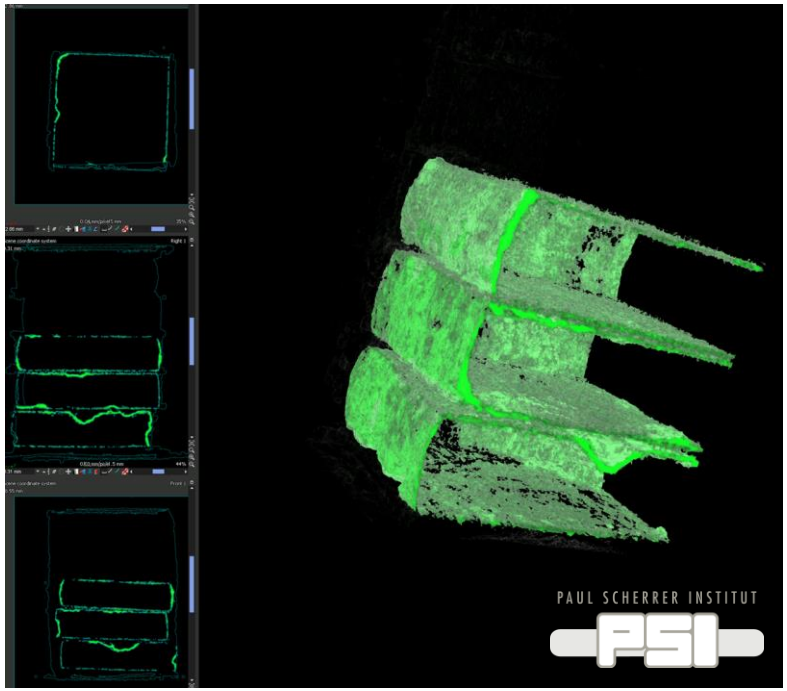
Siriruk, A., et al, Damage evolution in VARTM-based carbon fiber vinyl ester marine composites and sea water effects. Journal of Sandwich Structures & Materials (2017)

Example 7f: Attenuation Contrast

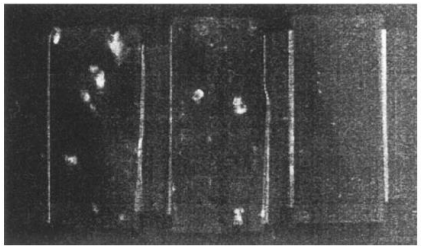
Applications to corrosion

EUROPEAN SPALLATION SOURCE

Mg corrosion



Al corrosion



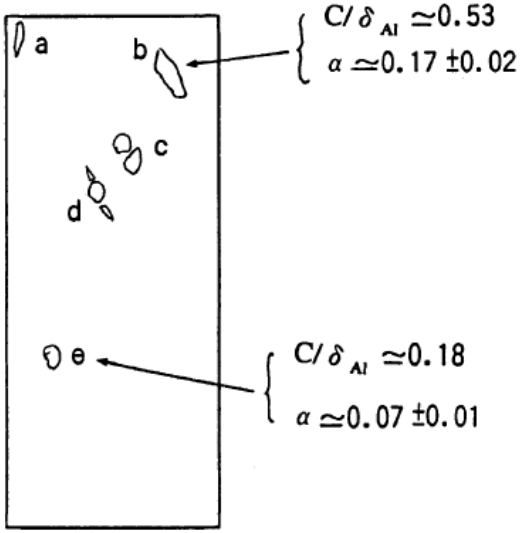


Fig. 6. Practical Al-samples and their NR-images observed.

M. Fujishiro et al. / Applied Radiation and Isotopes 61 (2004) 725–728

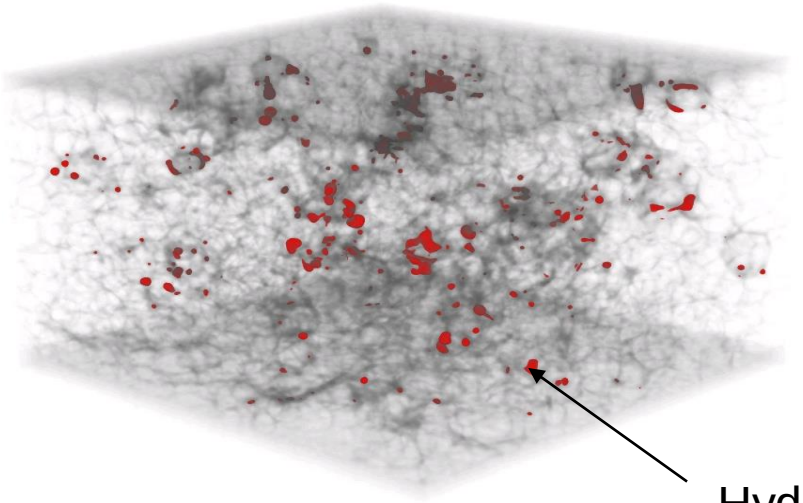
Example 7g: Attenuation Contrast

Applications to Metal Foams

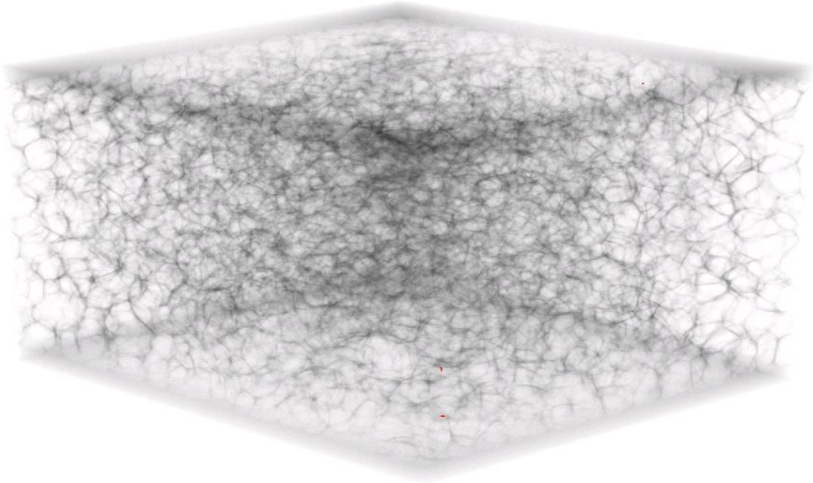
- Metals can be foamed with hydrogen released from hydrides (TiH_2, LiH etc)
  - Problem: hydride agglomerations produce inhomogeneous pore structure

- X-ray tomography reveals spatial structure of metal in great detail
- Neutron tomography gives insight into hydride distribution and relates it to pore structure
- combined measurements allow for superposition of complementary information

Neutron tomography



X-ray tomography



Hydride agglomeration

A. Rack et al. Journal of Microscopy 232(2) 282–292 (2008), M. Strobl et al. J. Phys D (2009)

AGENDA

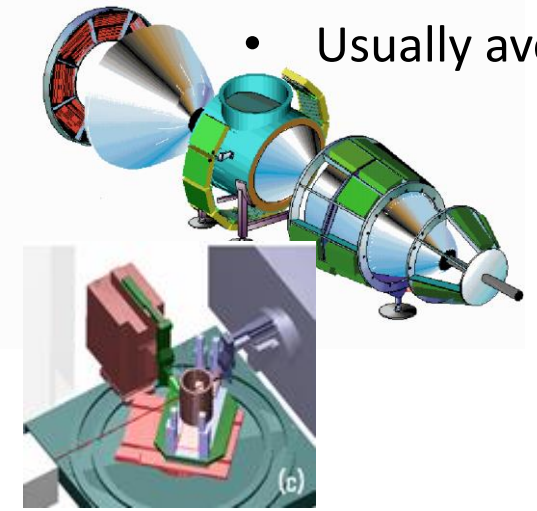
Neutron Imaging in Material Science

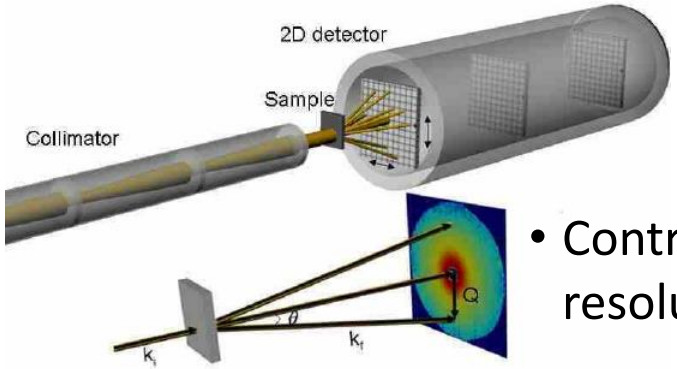
- **Intro**
- **Examples:** ‘White Beam’ Imaging
- **Examples:** Diffraction Contrast Imaging
- **Examples:** Phase Contrast Imaging
- **Examples:** Diffractive Imaging

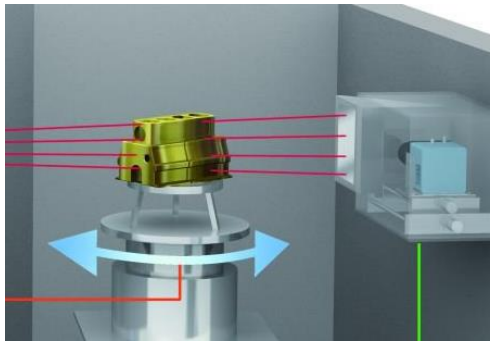
Neutron Imaging

Introduction

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







- Contrast from features (>spatial resolution) resolved in real space

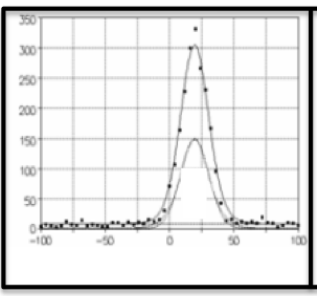
Diffraction regime

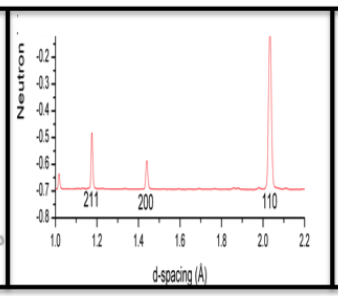
SANS regime

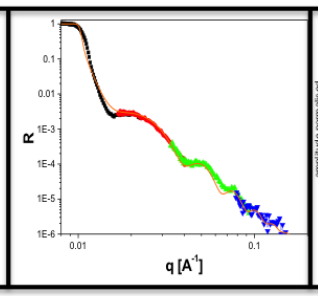
Length scale in nm

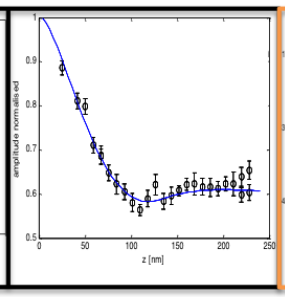
0.01 0.1 0.3 1.0 3.0 10 100 1000 10000 100000

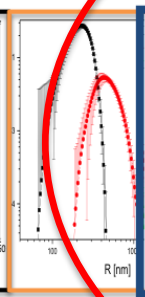
Conventional imaging regime

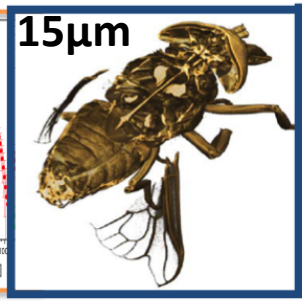


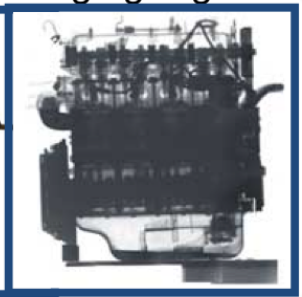












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Systems and components

M. Strobl & F. Grazzi (2015) From scattering in imaging to prospects at pulsed sources, Neutron News 26

Advanced imaging

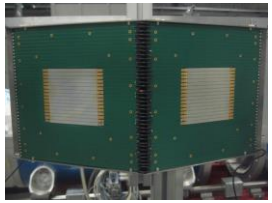
Introduction

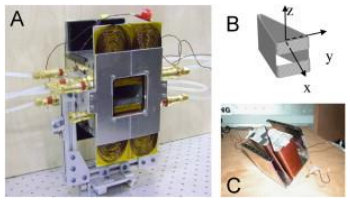
Monochromators

double-crystal monochromator

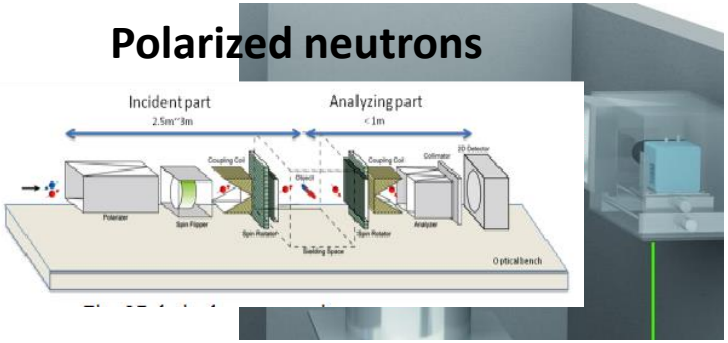
velocity selector

SEMSANS

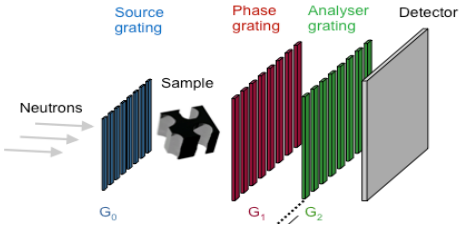




Polarized neutrons



Grating interferometer



- Contrast from features (>spatial resolution) resolved in real space
- Contrast from features (<spatial resolution) resolved in real space

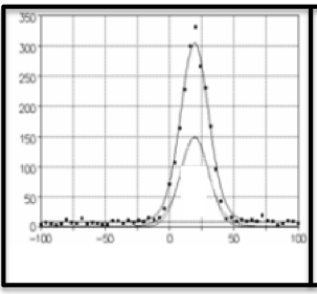
Diffraction regime

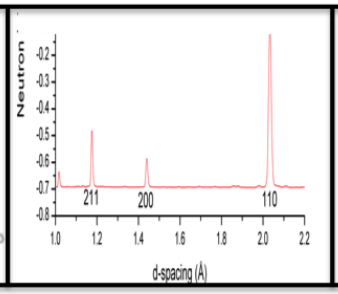
SANS regime

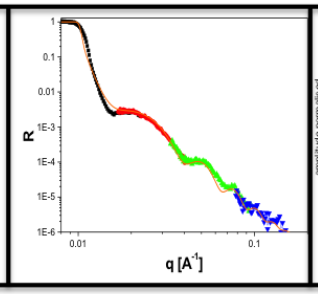
Length scale in nm

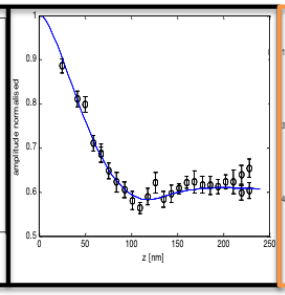
0.01 0.1 0.3 1.0 3.0 10 100 1000 10000 100000

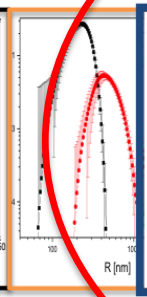
Conventional imaging regime

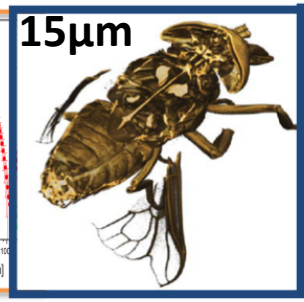


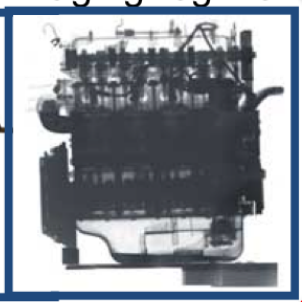












atomic and magnetic structures

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critical phenomena

pharmaceuticals

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internal strain

supermolecules

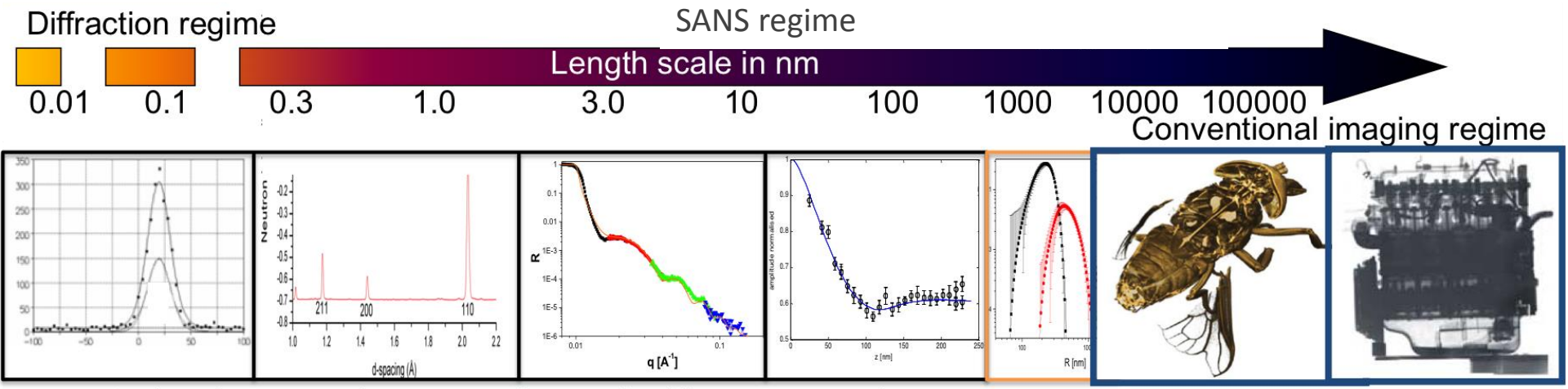
polymers

Systems and components

M. Strobl & F. Grazzi (2015) From scattering in imaging to prospects at pulsed sources, Neutron News 26

Advanced Neutron Imaging

Introduction



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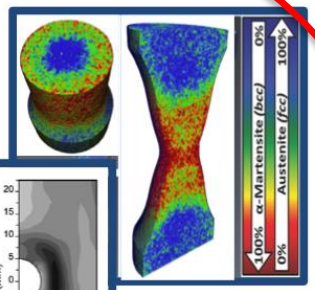
Advanced Neutron Imaging

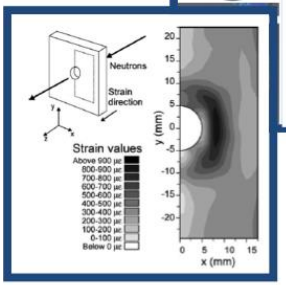
Introduction

Diffraction Contrast

"Bragg Edge Imaging"

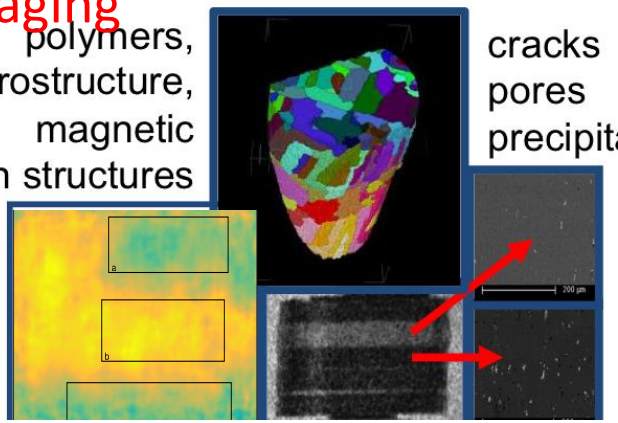
internal strains



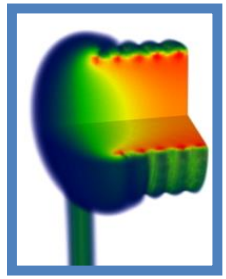


crystalline phases textures

polymers, microstructure, magnetic domain structures



cracks pores precipitates



magn. fields

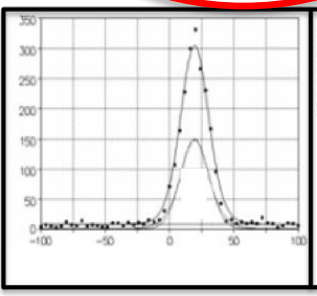
Diffraction regime

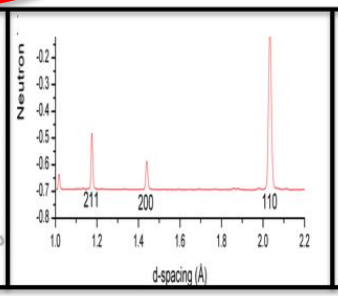
SANS regime

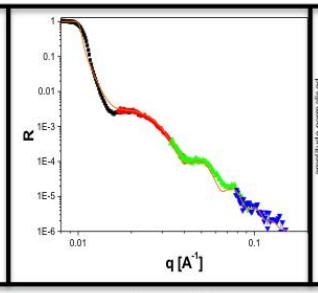
Length scale in nm

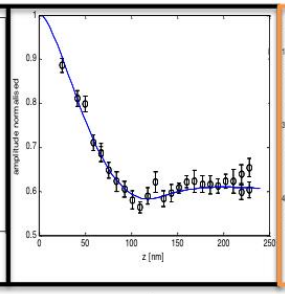
0.01 0.1 0.3 1.0 3.0 10 100 1000 10000 100000

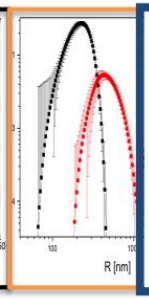
Conventional imaging regime











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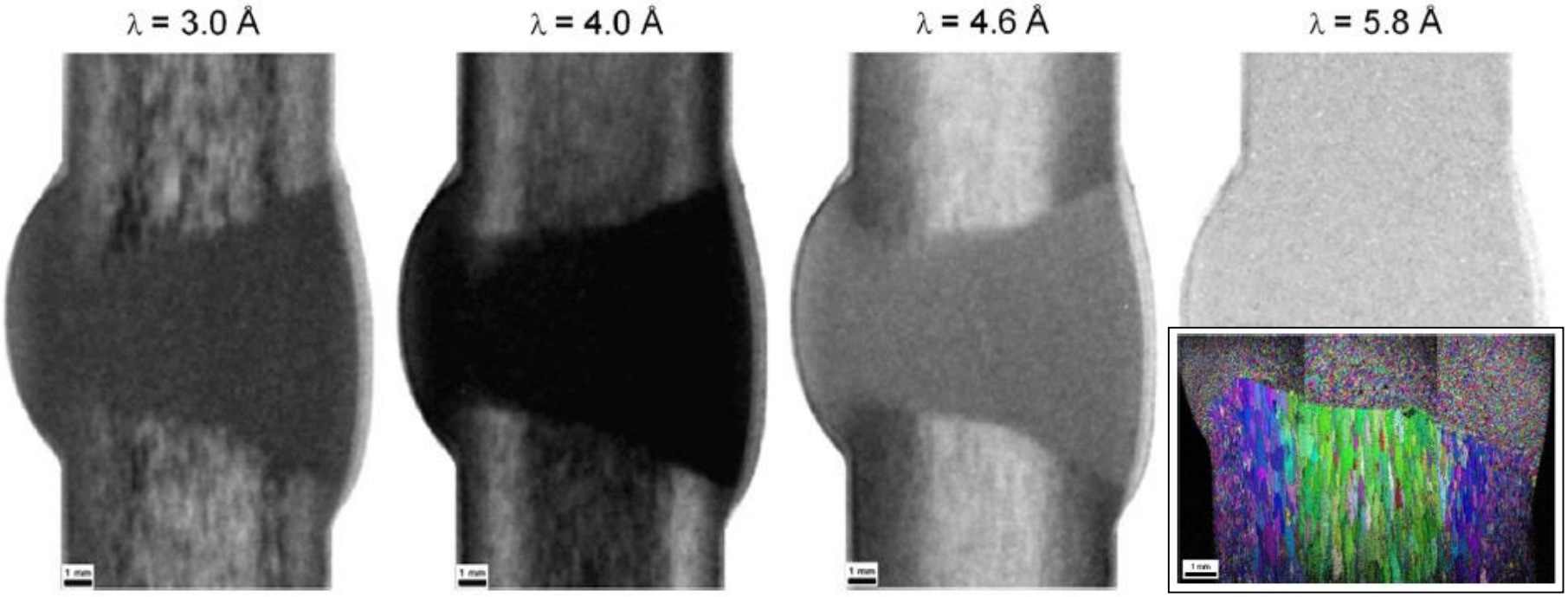
M. Strobl & F. Grazzi (2015) From scattering in imaging to prospects at pulsed sources, Neutron News 26

AGENDA

- **General Introduction**
  - ESS: The Most Intense Neutron Source for Material Research
- **Neutron Imaging**
  - Introduction, Why neutrons?
  - Basic Instrumentation
  - **Examples:** ‘White Beam’ Imaging
- **Diffraction Contrast** in Neutron Imaging
  - **Examples:** Diffraction Contrast Imaging
- **Ongoing Method Development**
- **Summary**

Example 8: Diffraction Contrast

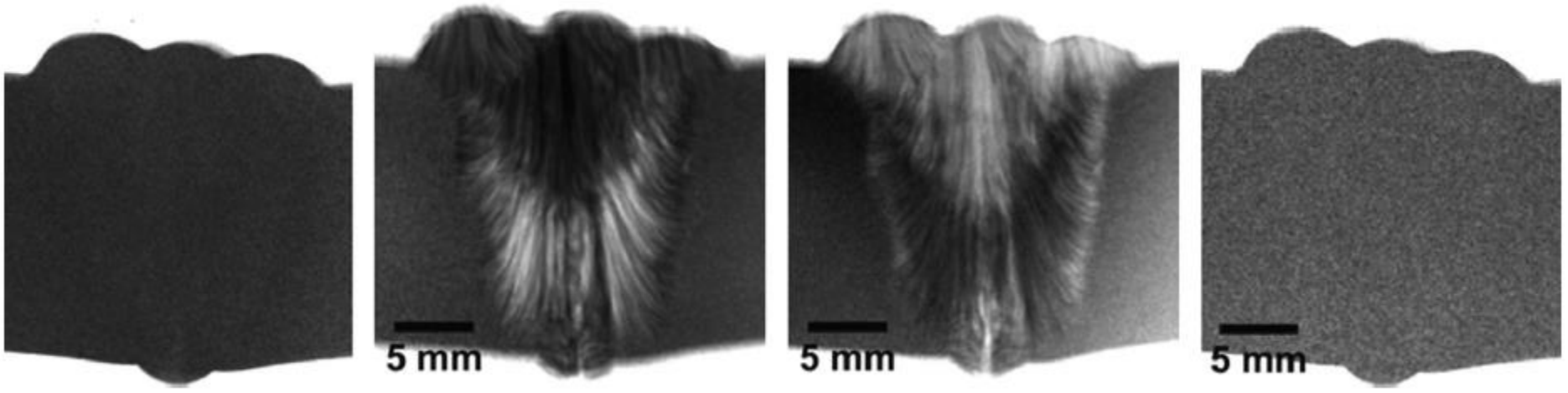
Weld 1

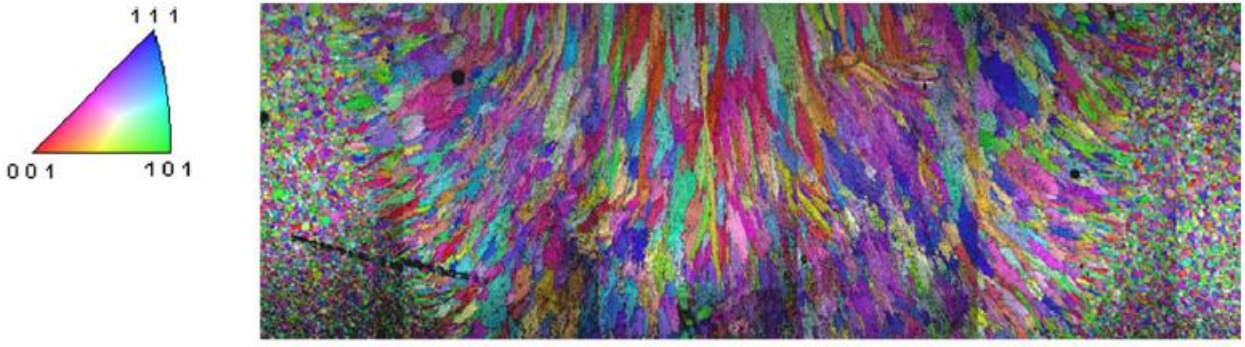


- E.H. Lehmann, S. Peetermans, L.Josic , H. Leber, H. van Swygenhoven, Energy-selective neutron imaging with high spatial resolution and its impact on the study of crystalline-structured materials, NIMA, 2014
- N. Kardjilov, I. Manke, A. Hilger, S. Williams, M. Strobl, R. Woracek, M. Boin, E. Lehmann, D. Penumadu, and J. Banhart , "Neutron Bragg-edge mapping of weld seams", International Journal of Materials Research, 2012

Example 8: Diffraction Contrast

Weld 2





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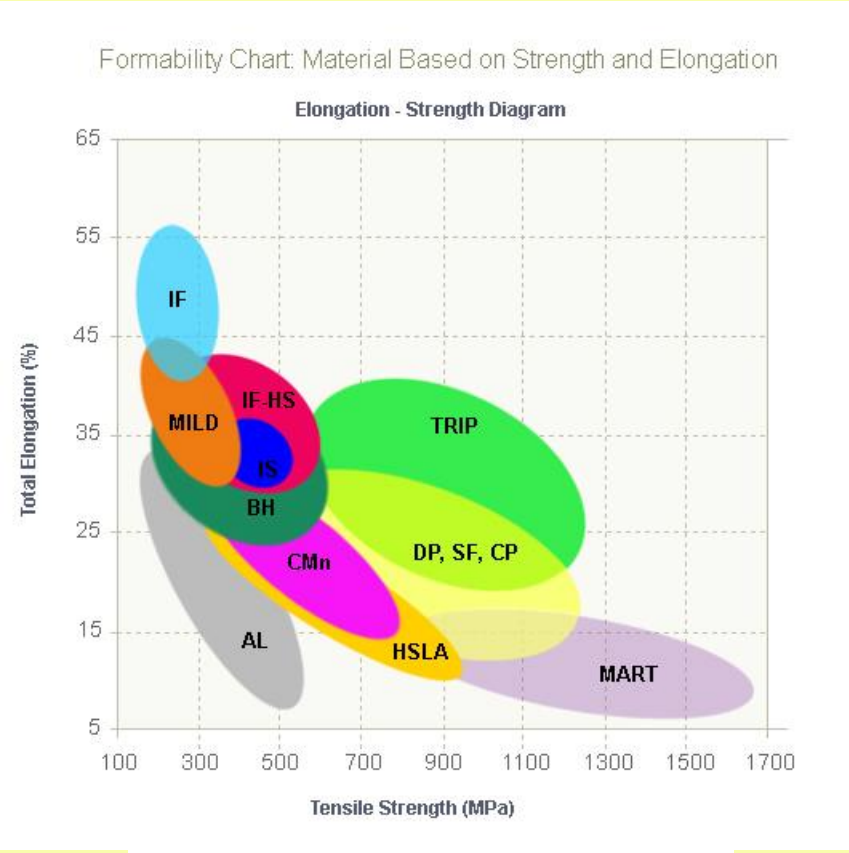
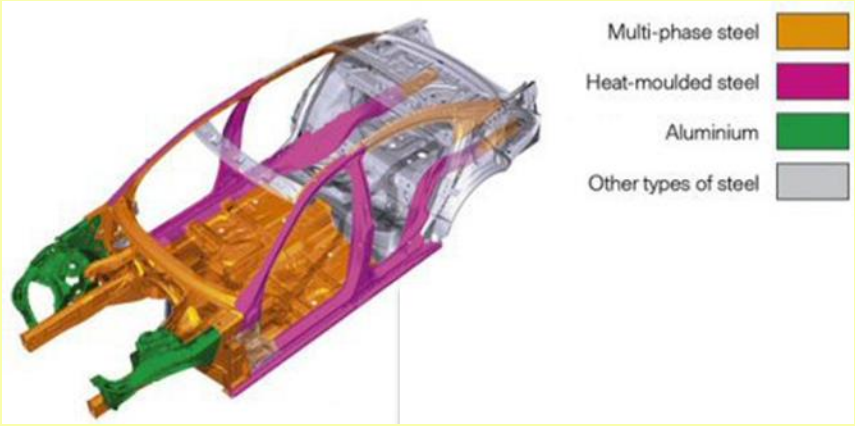
Example 9: Diffraction Contrast

Phase Radiography

- Metastable austenitic stainless (**TRIP**) steel: FCC Austenite transforms to HCP and BCC Martensites under strain





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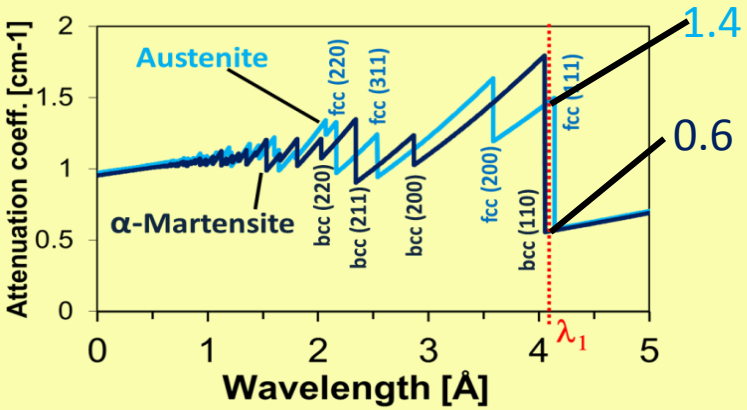
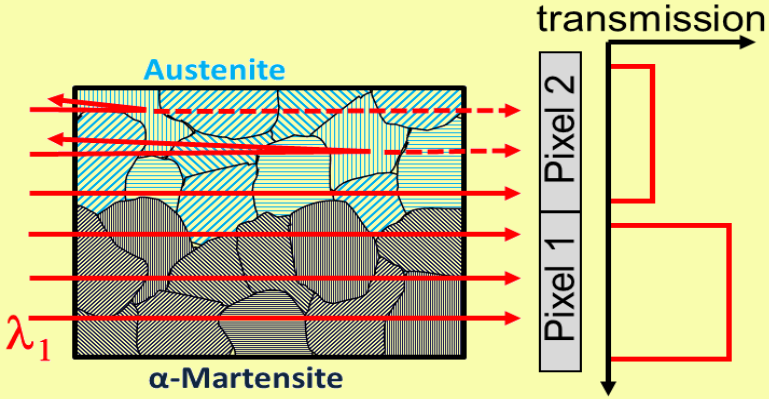
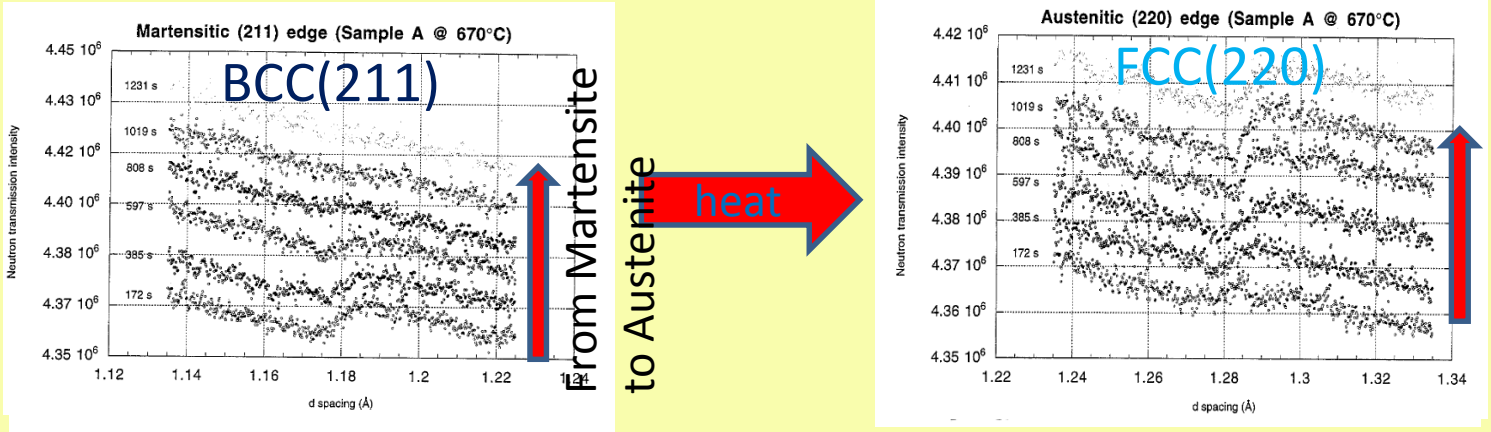




Reverse (α′ → γ) transformation

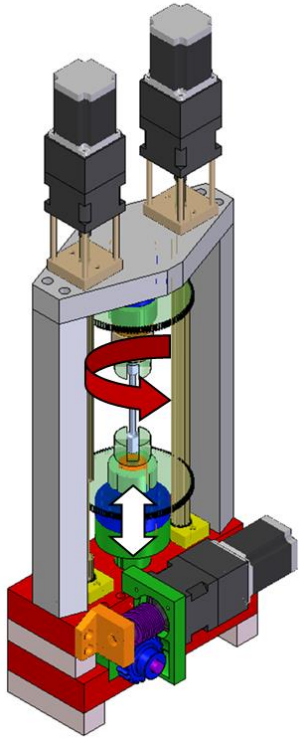


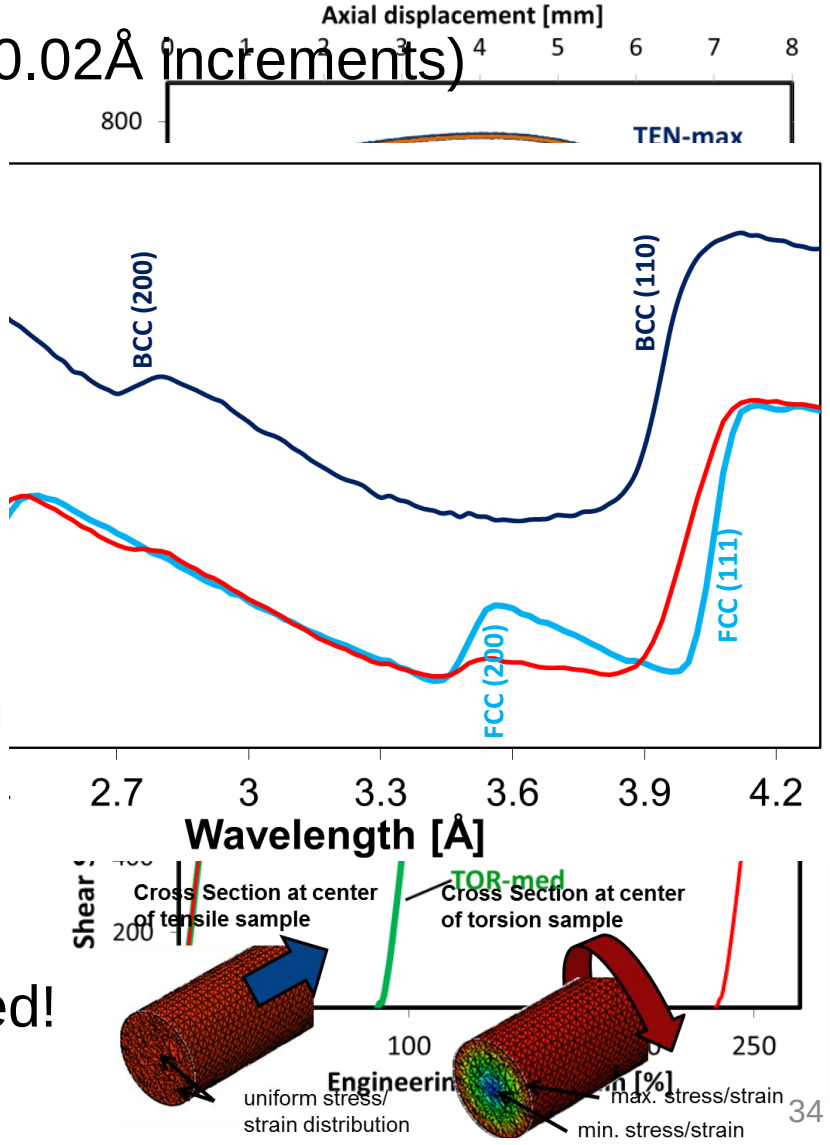
- M.A.M. Bourke, J.G. Maldonado, D. Masters, K. Meggers, H.G. Priesmeyer, "Real time measurement by Bragg edge diffraction of the reverse (α′ → γ) transformation in a deformed 304 stainless steel", Materials Science and Engineering: A, Vol 221 (1996).

Example 9: Diffraction Contrast

Phase Radiography

- 3 samples (304L): *Virgin, 2 x Tension, 2 x Torsion*
- Wavelength Scan from 1.6Å to 4.4Å (0.02Å increments)



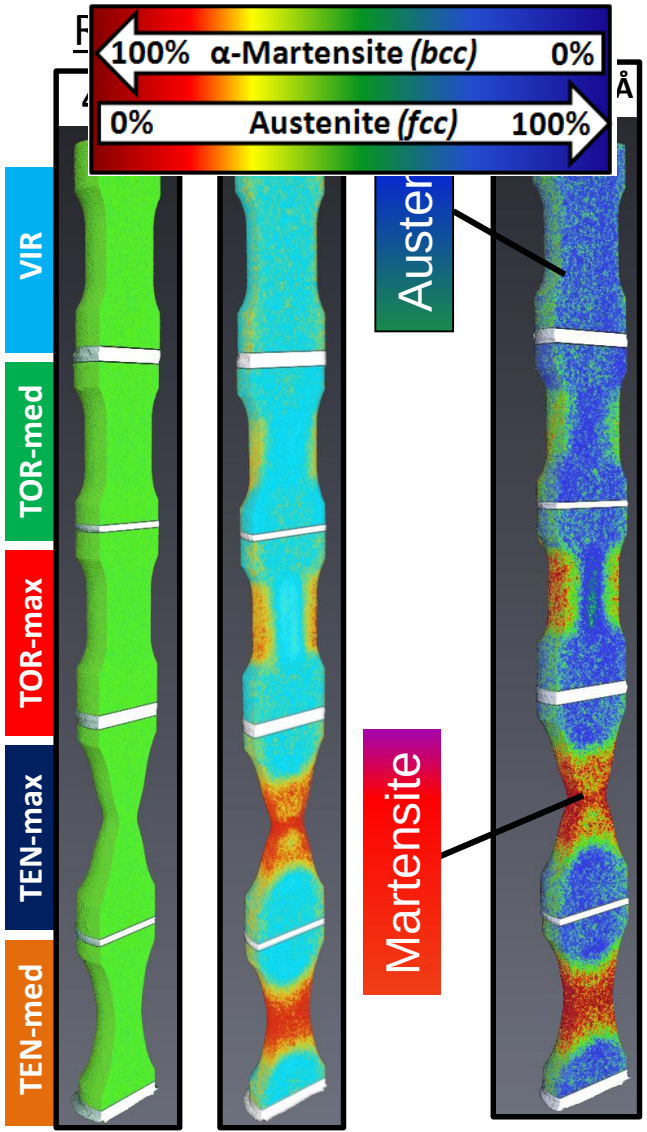


- Phase transformation clearly observed!
- Tomography for further quantification

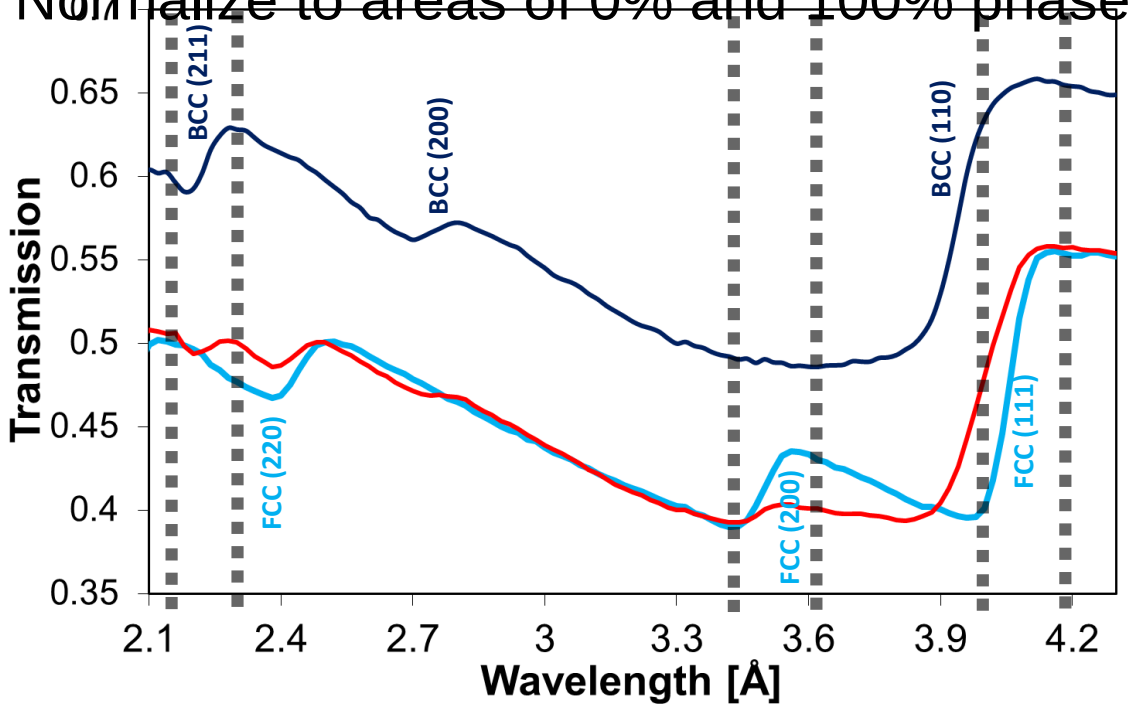
Example 9: Diffraction Contrast

Phase Tomography

- At 6 energies before and after Bragg Edges: 180 projections each



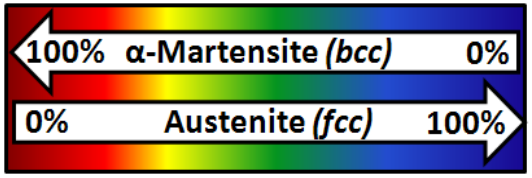
- Edge height proportional to volume fraction
- Normalize to areas of 0% and 100% phase



Example 9: Diffraction Contrast

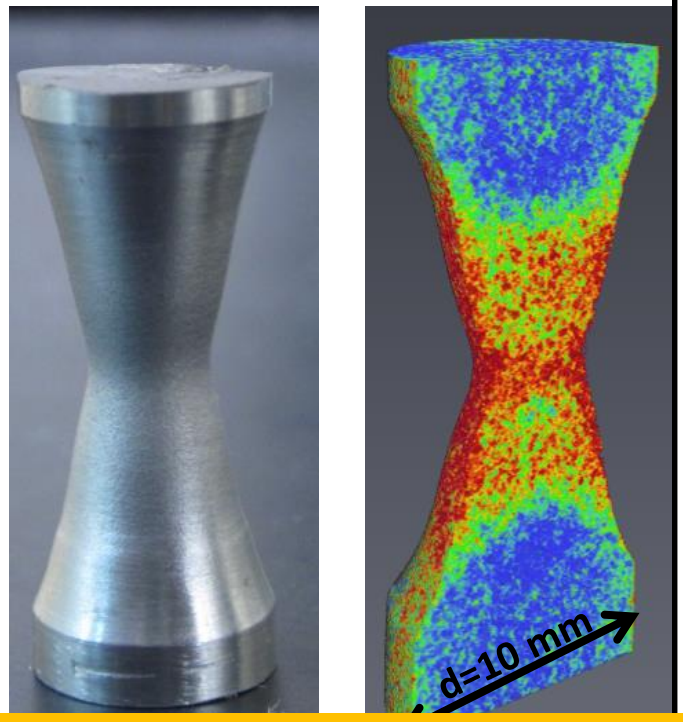
Phase Tomography

- At 6 energies before and after Bragg Edges: 180 projections each

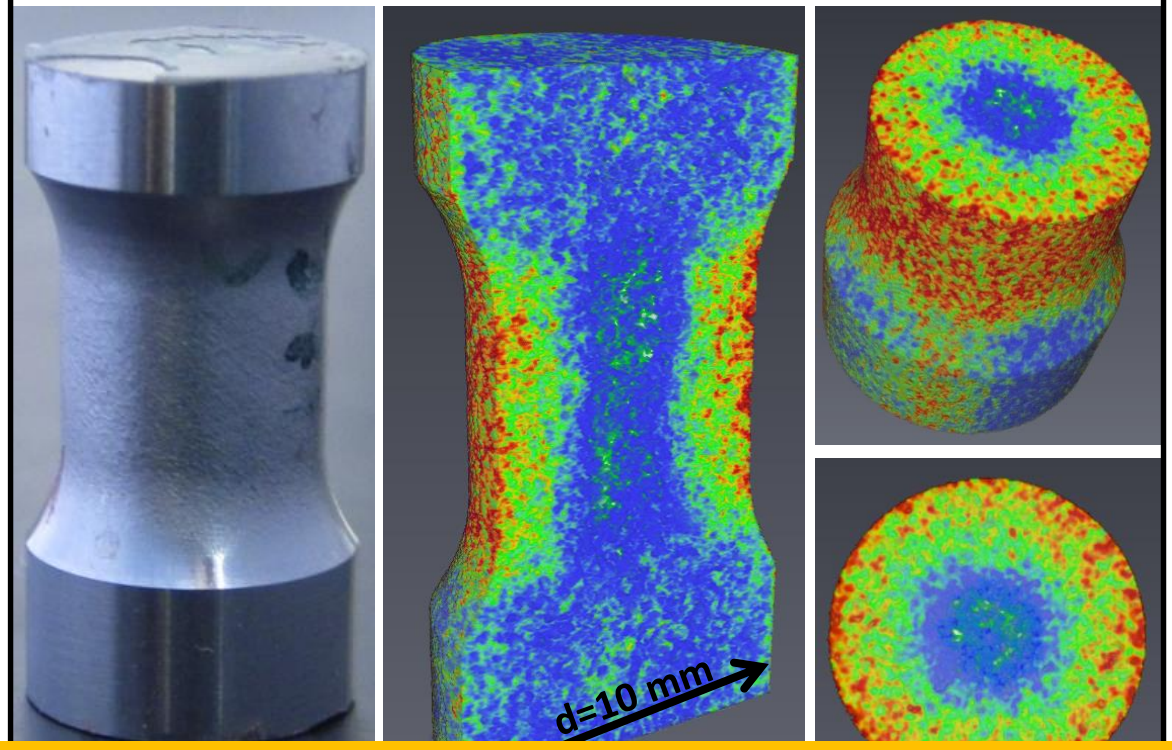


- Edge height proportional to volume fraction
- Normalize to areas of 0% and 100% phase

Tensile



Torsion

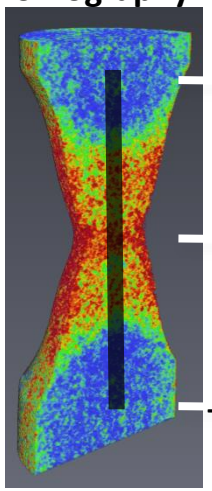


Woracek, R., Penumadu, D., Kardjilov, N., Hilger, A., Boin, M., Banhart, J., & Manke, I. (2014). 3D Mapping of Crystallographic Phase Distribution using Energy-Selective Neutron Tomography. Advanced Materials, 26(24), 4069-4073.

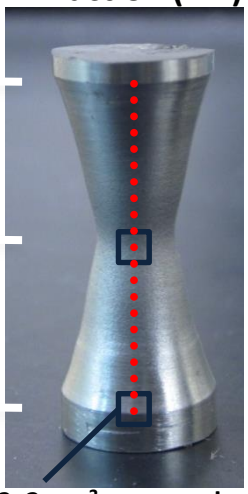
Example 9: Diffraction Contrast

Verify with Neutron Diffraction

Tomography:

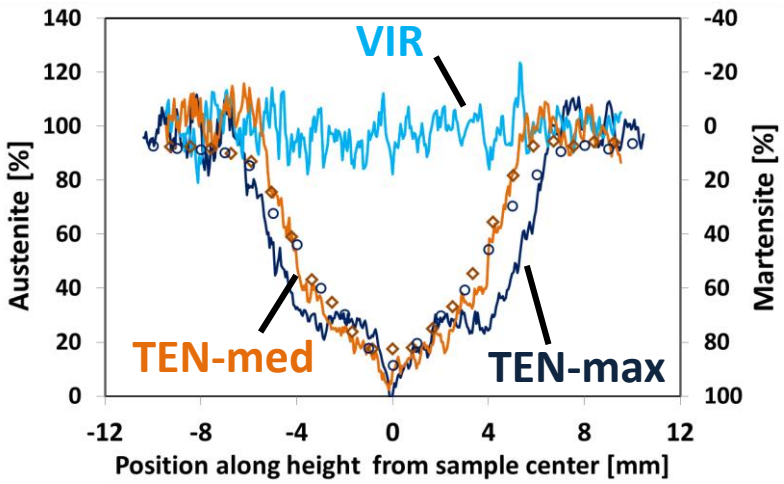


Diffraction (ND):



Line profile through cross-section

2x2x2mm^3 gauge volume: 21 measurement points

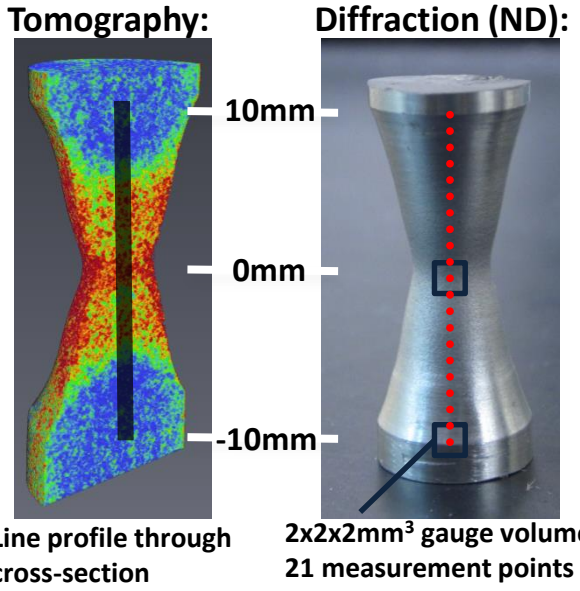


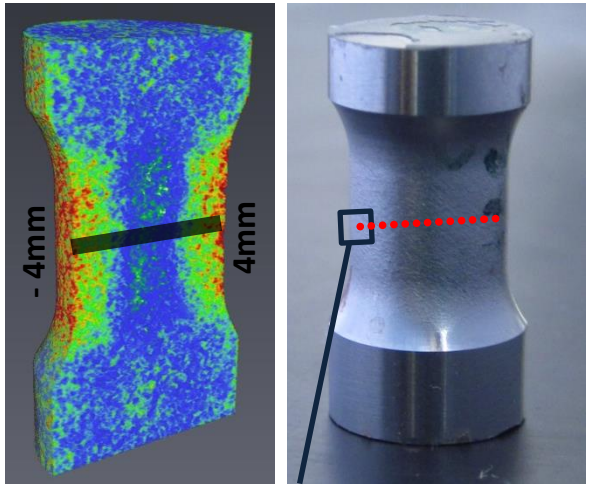
12 hours/ tomography

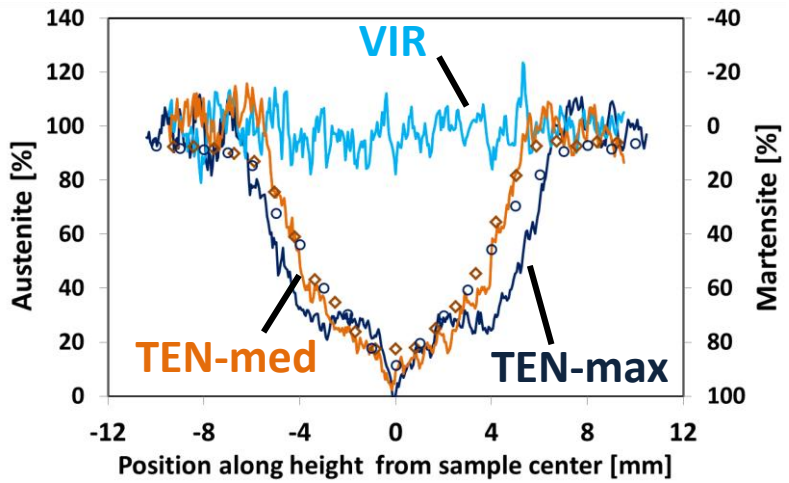
20 minutes/ point

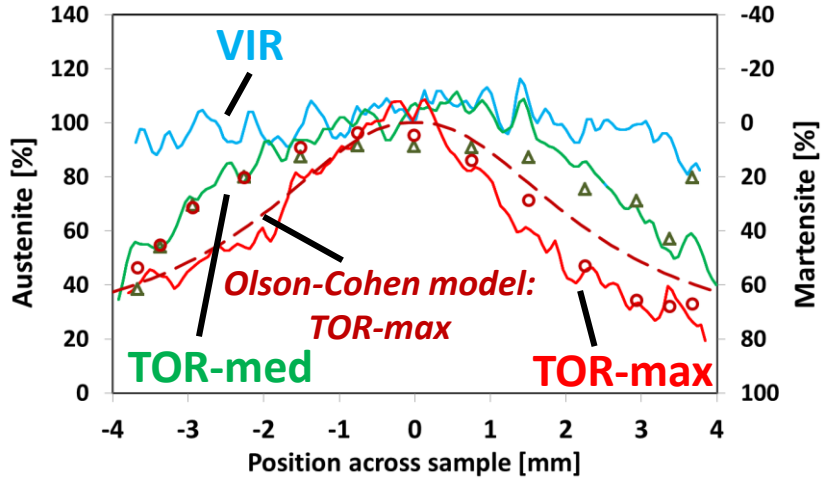
Example 9: Diffraction Contrast

Verify with Neutron Diffraction









12 hours/ tomography

20 minutes/ point

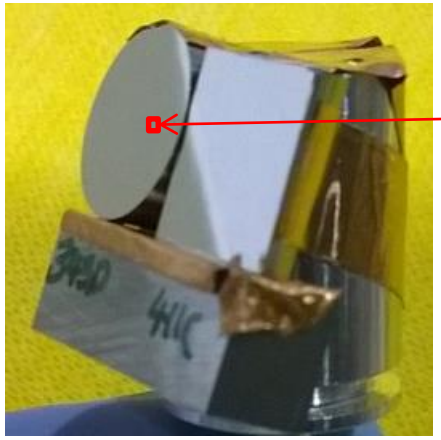
- Good agreement! However, sharp gradients could not be captured because of the relatively large gauge volume used in neutron diffraction.

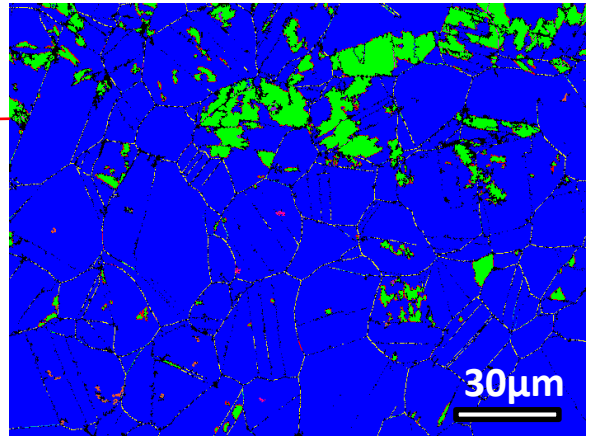
Example 9: Diffraction Contrast

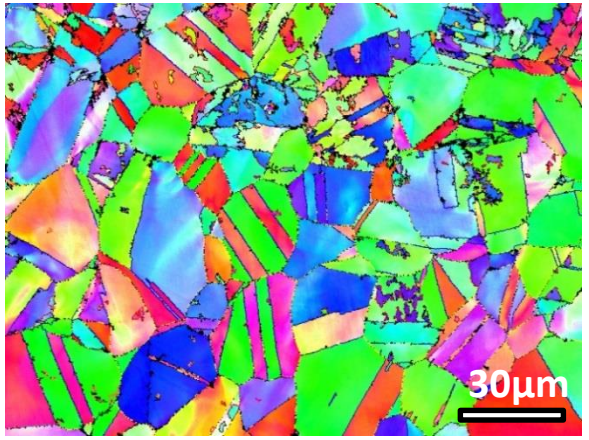
Verify with EBSD

EUROPEAN SPALLATION SOURCE

❖ **Virgin Sample**: 95% Austenite (blue) vs 5% Martensite (green). Orientation mostly random.

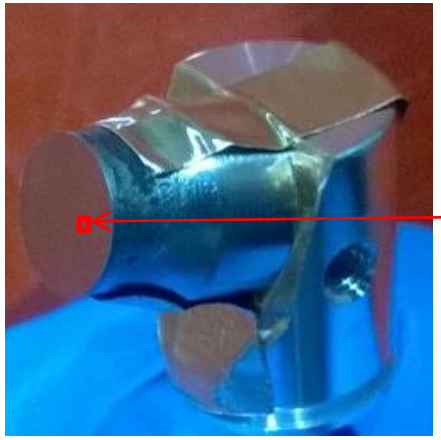


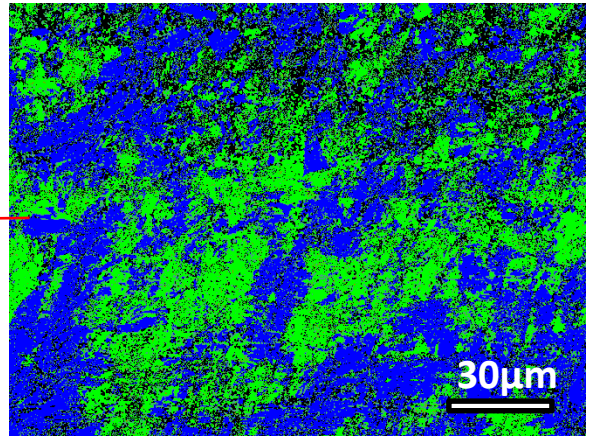


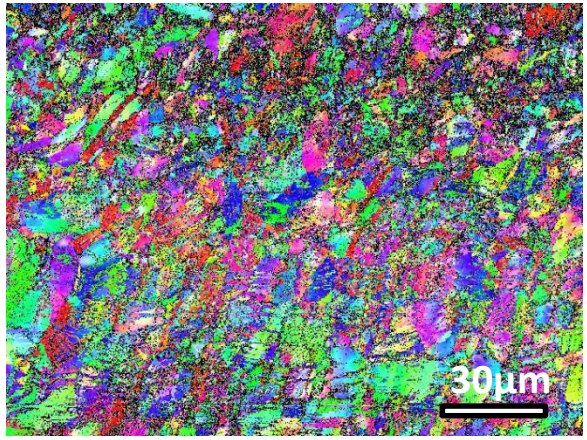


40 min/ map

❖ **Torsion sample** (TOR-max) **at r = 2 mm**: 45% Austenite vs 55% Martensite







5 hours/ map

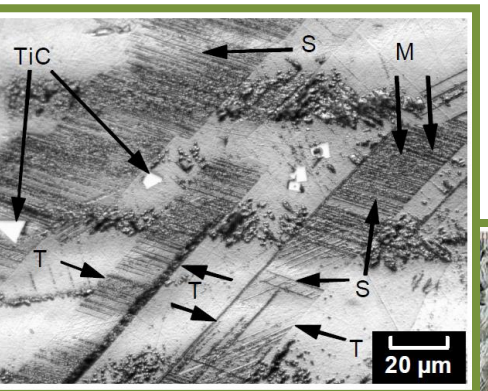
- EBSD only successful in regions up to medium deformation (close to center)
- EBSD indexing fails for regions with high plastic strain!

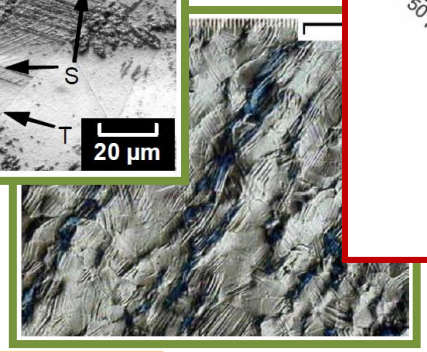
Example 9: Diffraction Contrast

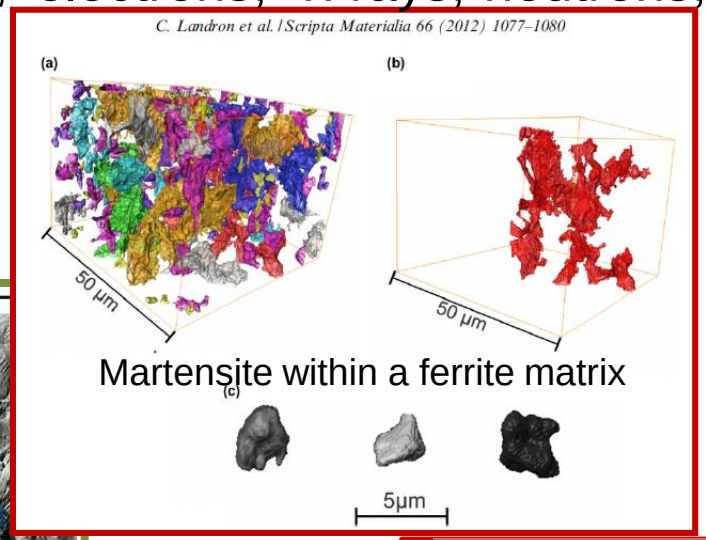
Complementarity of methods…

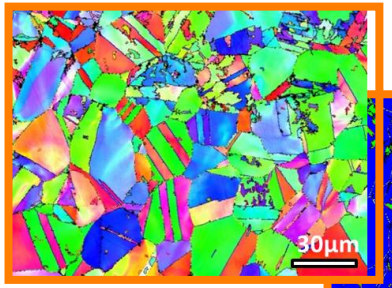
- Different methods provide complementary information and cover various length scales….

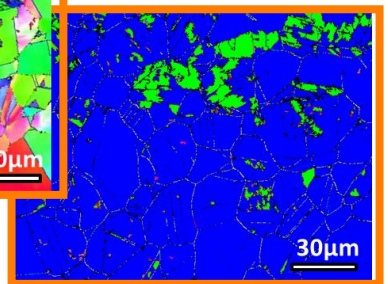
optical microscopy, electrons, x-rays, neutrons, …

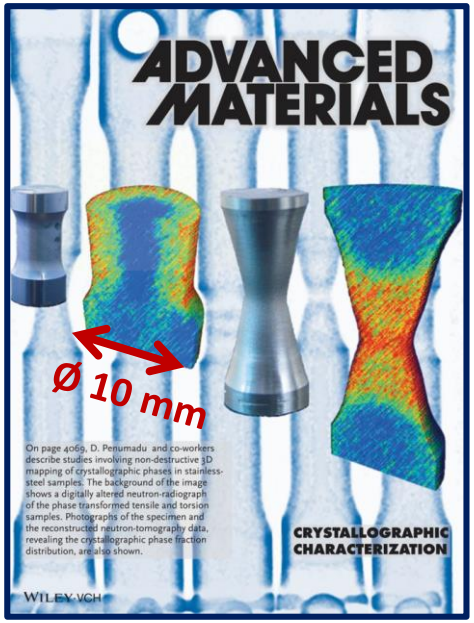








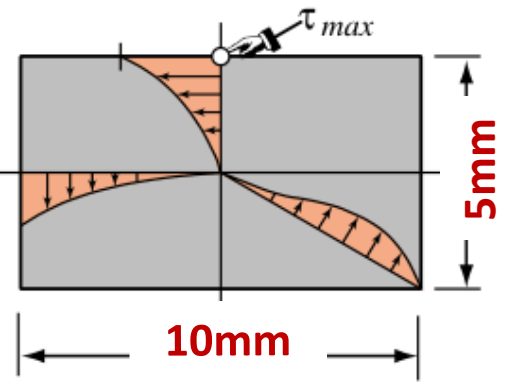




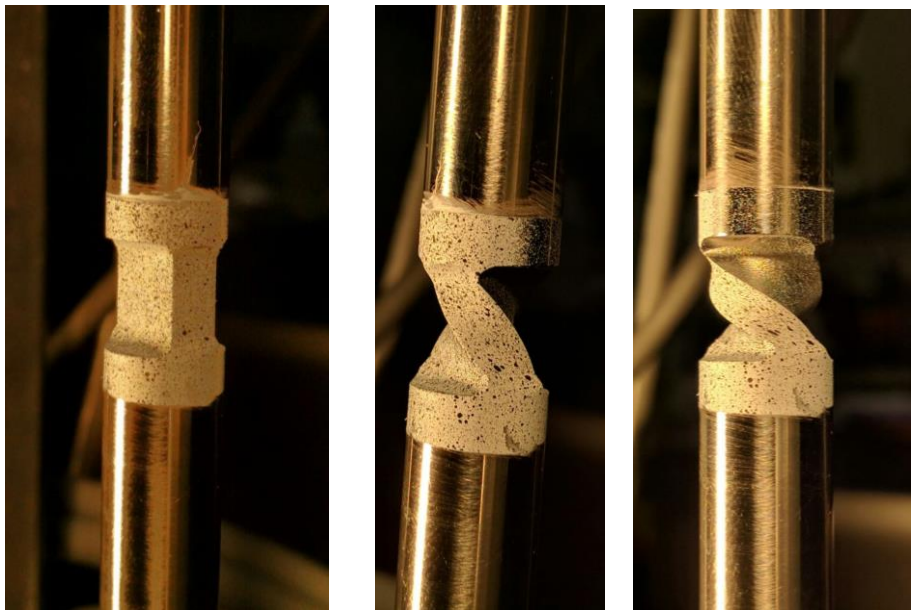
Example 10: Diffraction Contrast

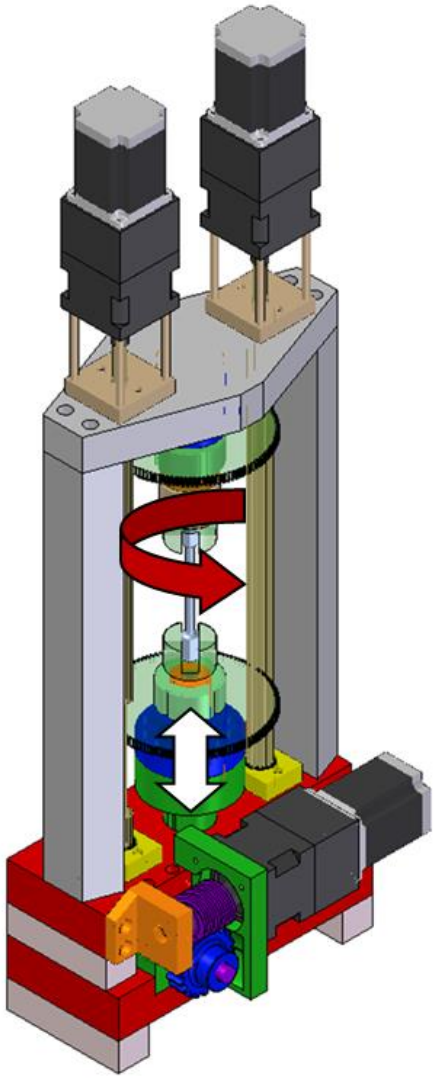
Complicated geometry

- Torsion of rectangular cross-section (304L)
- In-situ Tomography at 3 wavelengths



- Follow the phase transformation in high strained material with complex stress field

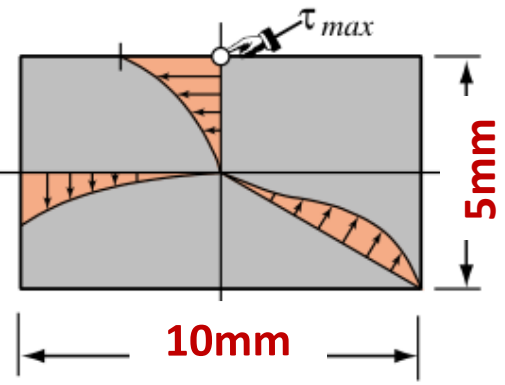


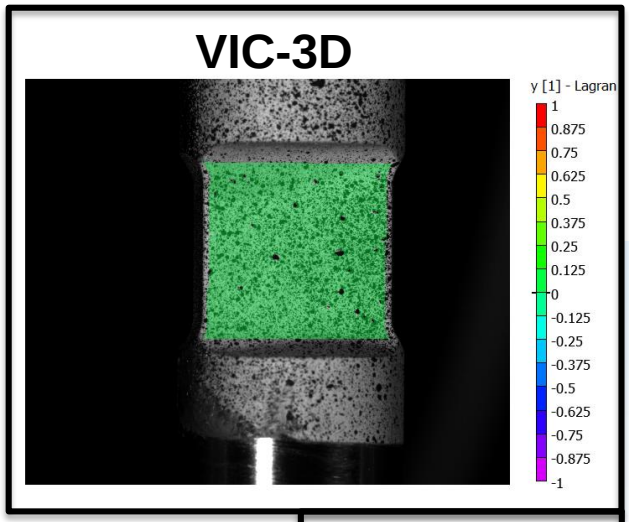


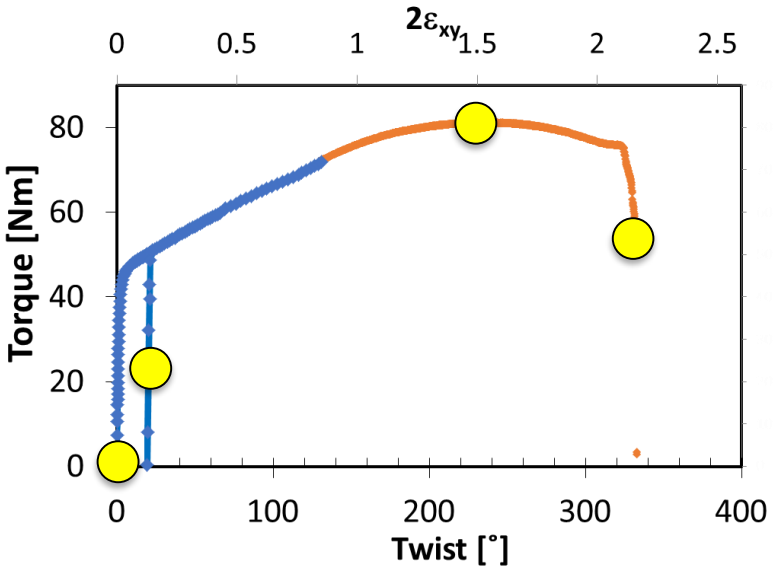
Example 10: Diffraction Contrast

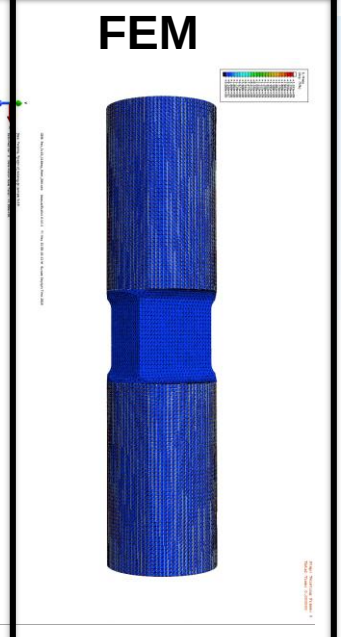
Complicated geometry

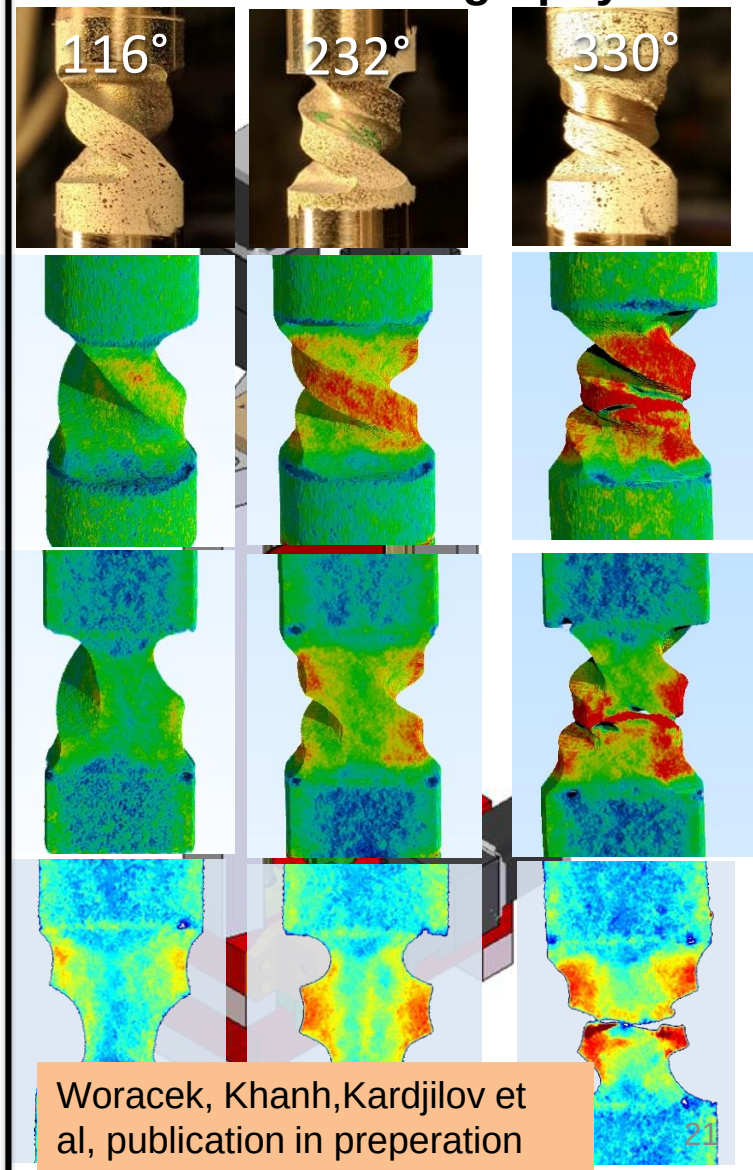
- Torsion of rectangular cross-section (304L)
- In-situ Tomography











Woracek, Khanh,Kardjilov et al, publication in preperation

Example 11: Diffraction Contrast

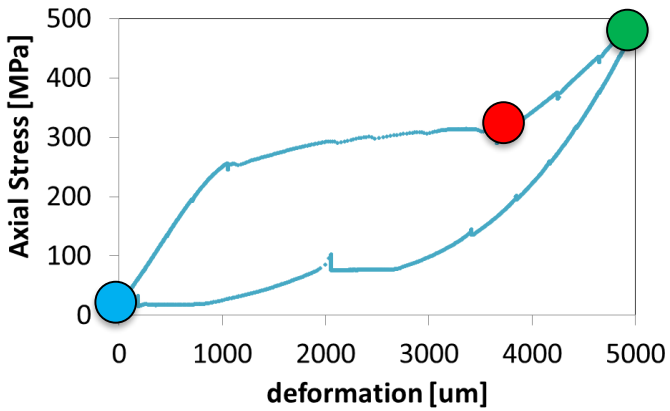
Phase & Texture: NiTi

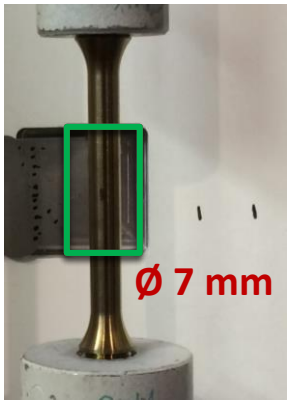
- So far: random (low) textured samples

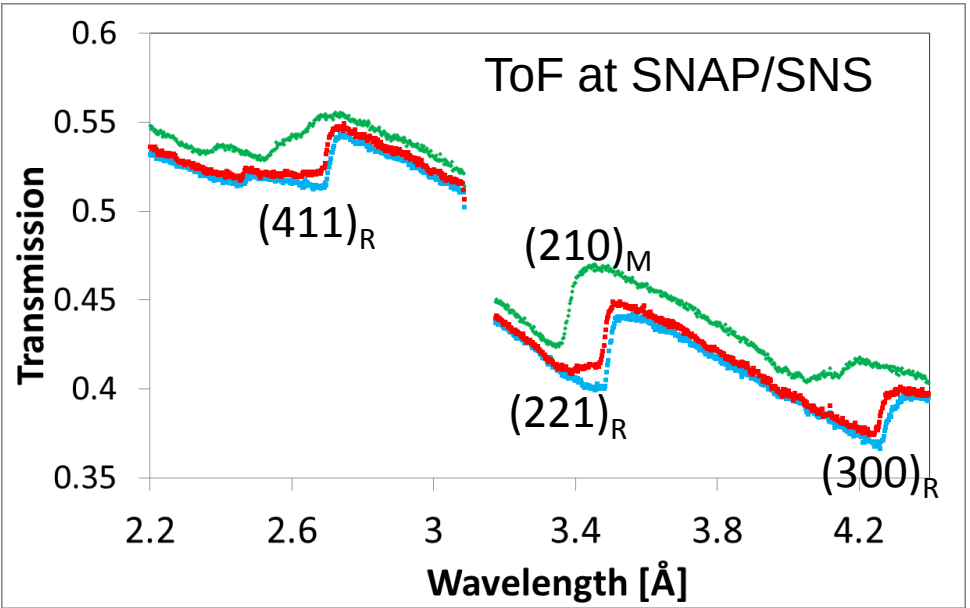
Example 11: Diffraction Contrast

Phase & Texture: NiTi

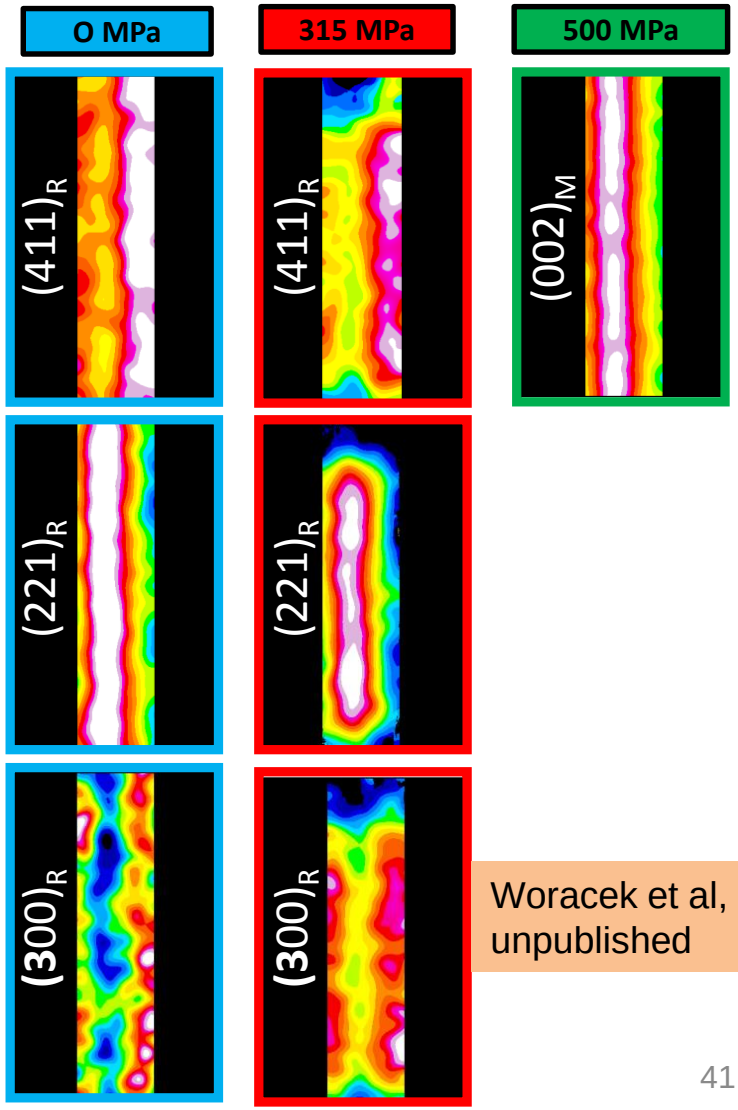
- So far: random (low) textured samples
- In-situ tensile loading of NiTi







Edge height



Woracek et al, unpublished

Example 12: Diffraction Contrast

Textured samples: Challenges and Opportunities

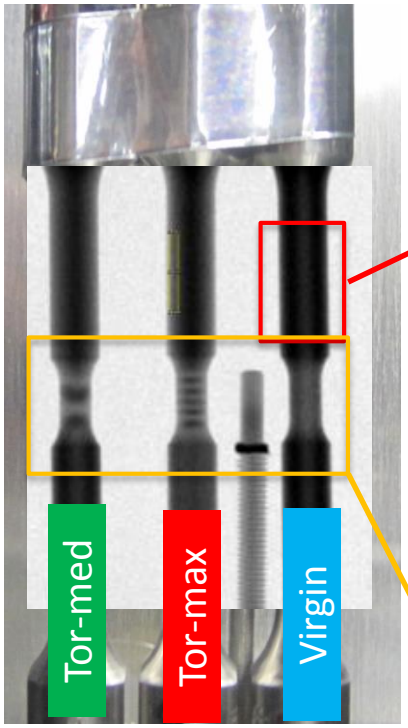
- Again TRIP (304L) steel: *initially textured*

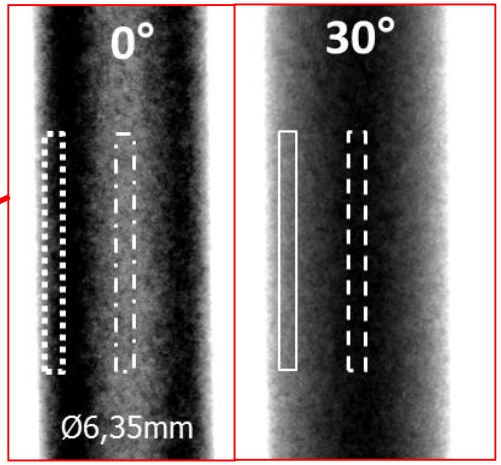
Woracek, Dissertation

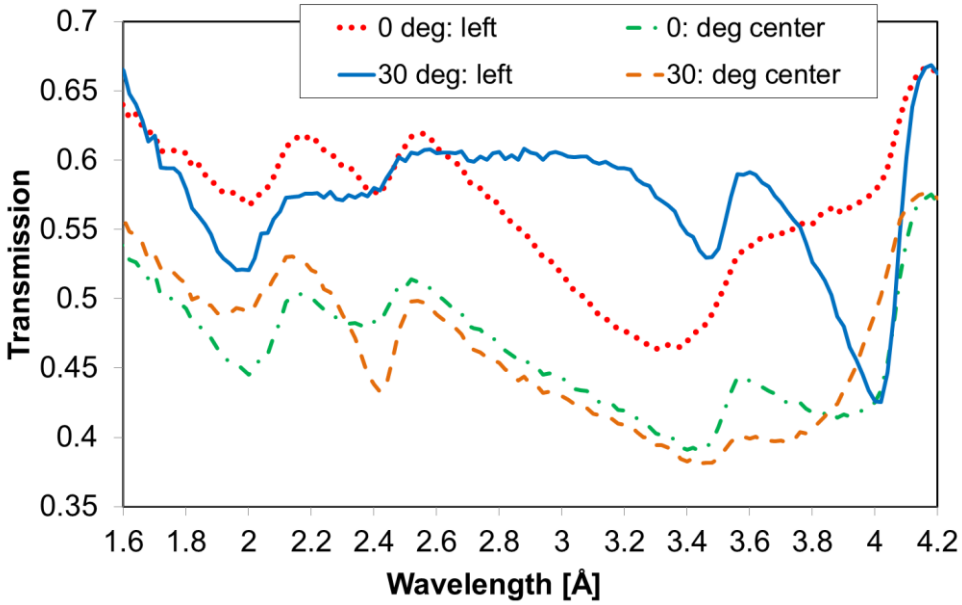
Example 12: Diffraction Contrast

Textured samples: Challenges and Opportunities

- Again TRIP (304L) steel: *initially textured*







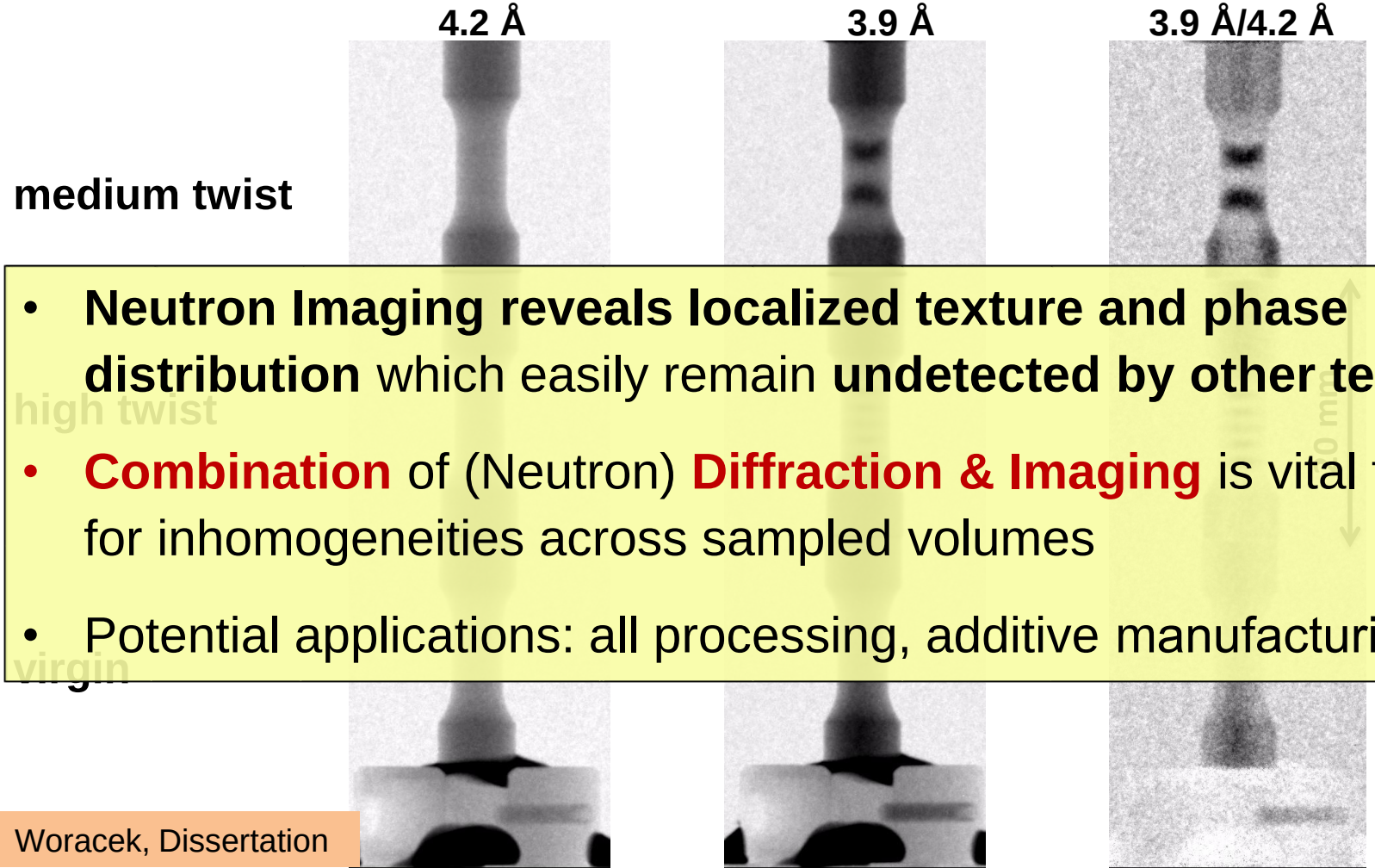
3.9 Å/4.2 Å

Woracek, Dissertation

Example 12: Diffraction Contrast

Textured samples: Challenges and Opportunities

- Again TRIP (304L) steel: *initially textured*

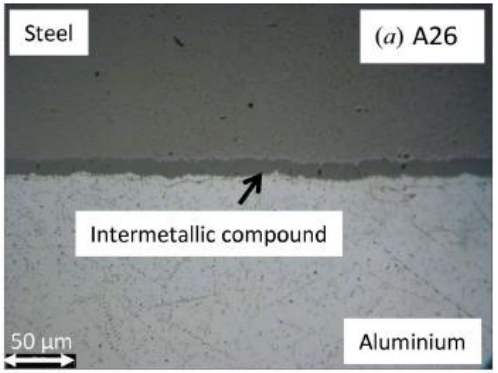


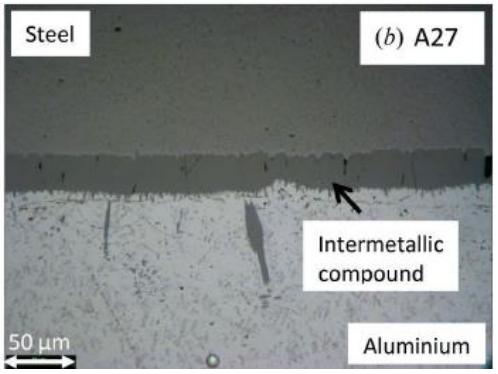
- **Neutron Imaging reveals localized texture and phase distribution** which easily remain **undetected by other techniques**
- **Combination** of (Neutron) **Diffraction & Imaging** is vital to monitor for inhomogeneities across sampled volumes
- Potential applications: all processing, additive manufacturing, …?

Woracek, Dissertation

Example 13: Diffraction Contrast

Dissimilar welds by ToF imaging



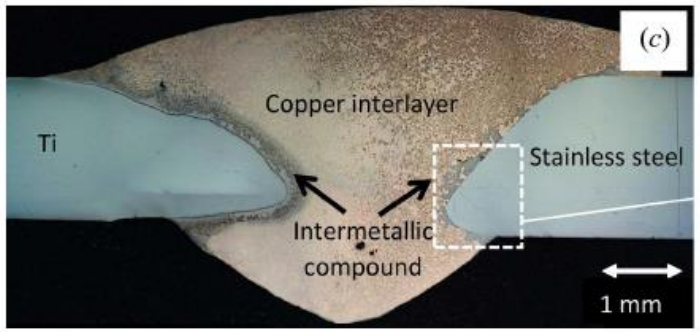


Aluminium to steel alloy.

	Elements (wt%)											
	Al	Fe	C	Si	Mn	P + S	N	Ti	Cu	Mg	Zn	Cr
XF350 (steel)	0.045	Balance	0.0002	0.015	0.613	0.024	0.017	0.002				
5083-H22 (aluminium)	Balance	0.4		0.4	0.5			0.15	0.1	2.6–3.6	0.2	0.3

Titanium to stainless steel alloy.

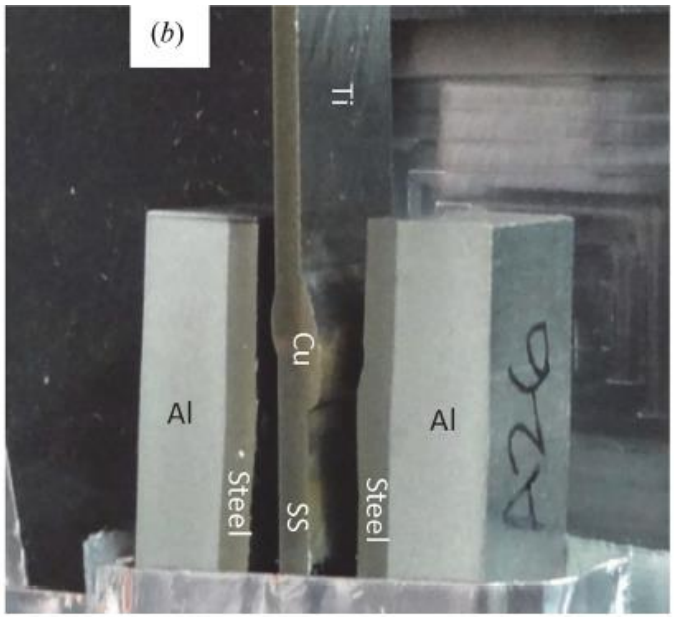
	Elements (wt% maximum)														
	Si	Fe	Mo	Mn	Ni	Cr	C	P	S	Ti	N	Al	V	O	H	Y
AISI 316 L (stainless steel)	0.45	Balance	2.07	1.73	10	17.2	0.02	0.032	0.01		0.054					
Ti-6Al-4V (titanium)		0.3					0.08			Balance	0.05	6.75	4.5	0.2	0.15	0.05

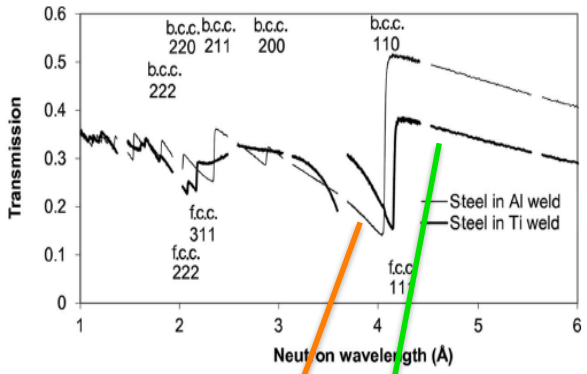


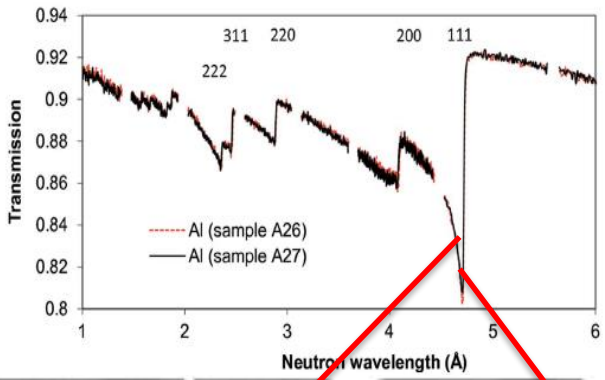
Anton S. Tremsin *et al.* • Investigation of metal welds by neutron imaging *J. Appl. Cryst.* (2016). **49**, 1130–1140

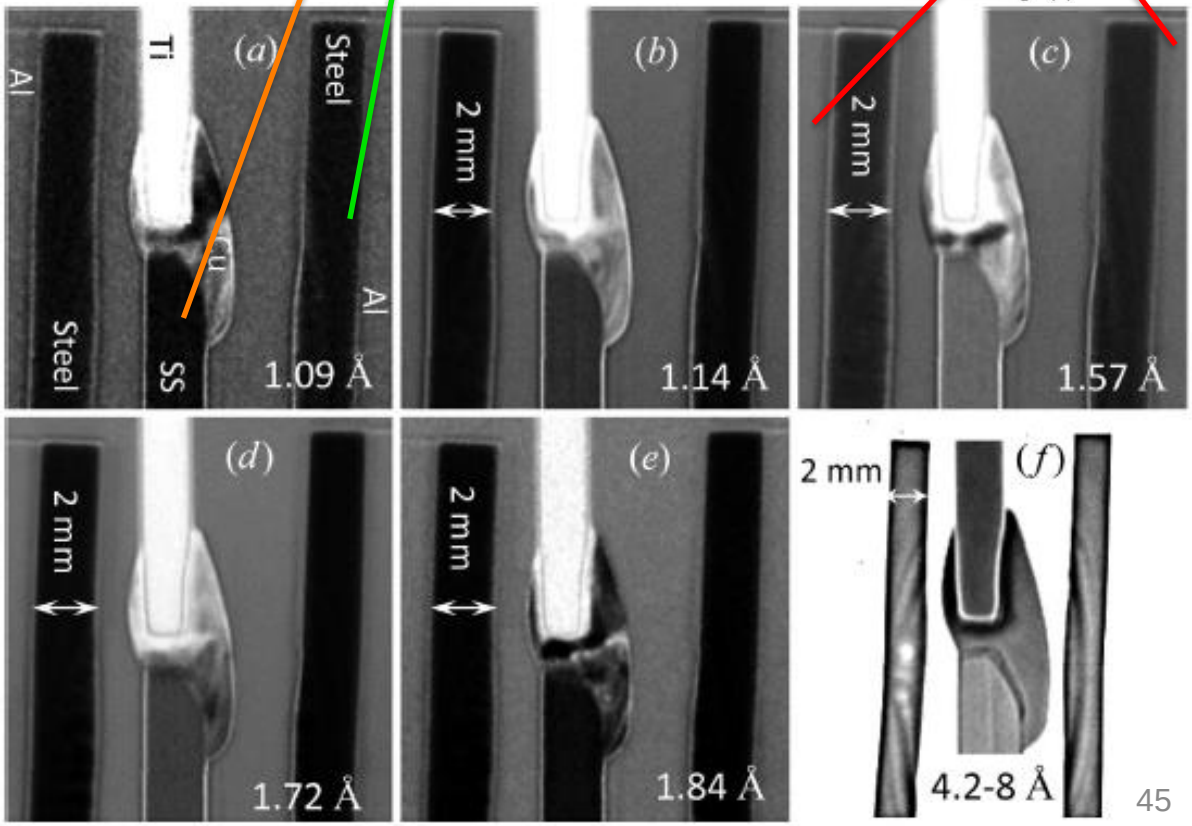
Example 13: Diffraction Contrast

Dissimilar welds by ToF imaging



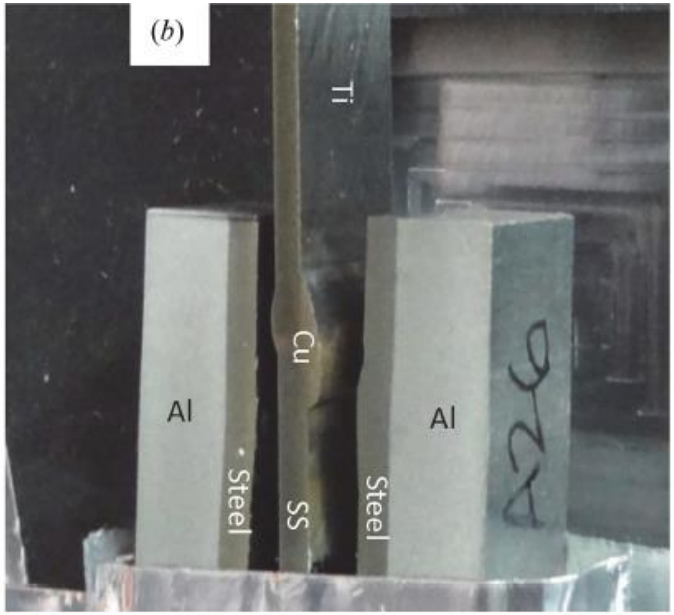


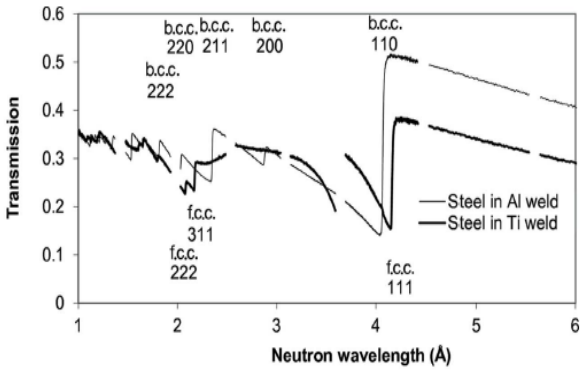


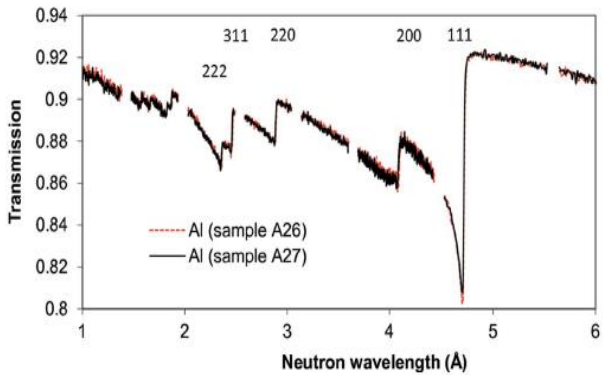


Example 13: Diffraction Contrast

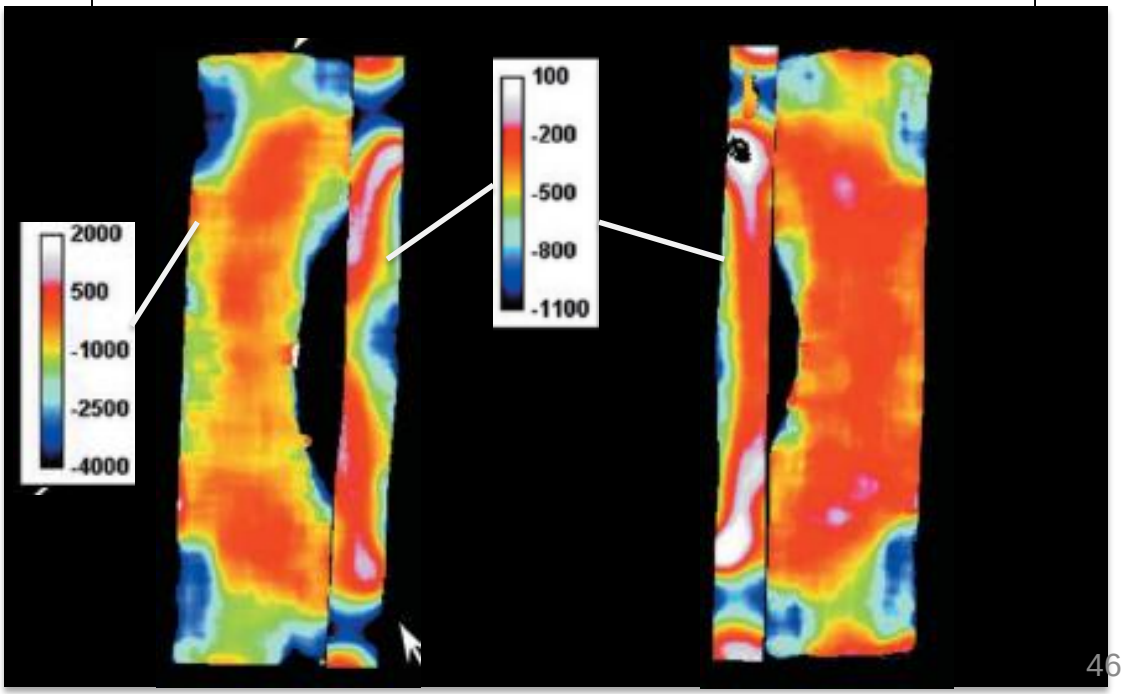
Dissimilar welds by ToF imaging





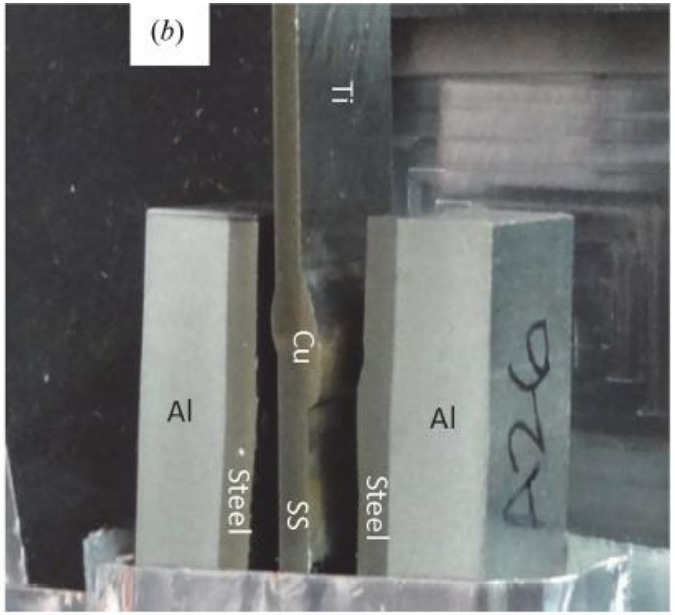


Spatially resolved strain maps



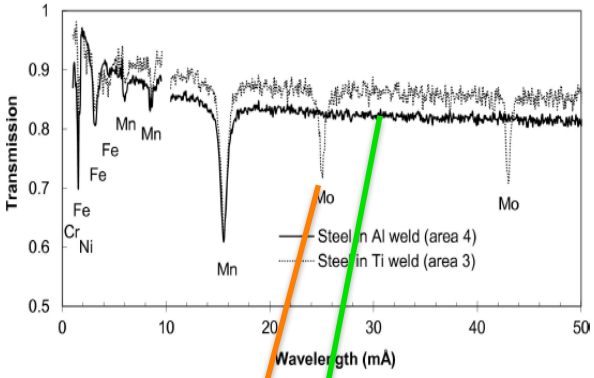
Example 13: Diffraction Contrast

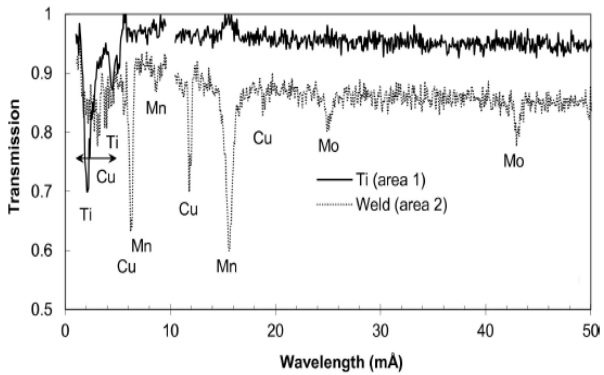
Dissimilar welds by ToF imagingc

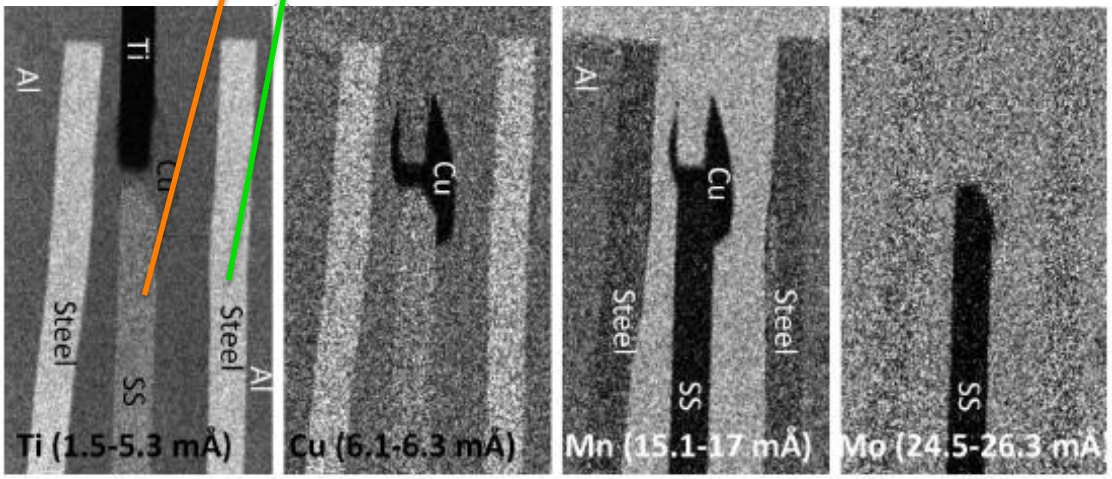


Resonance absorption contrast:

Elemental contrast: E.g. check diffusion processes



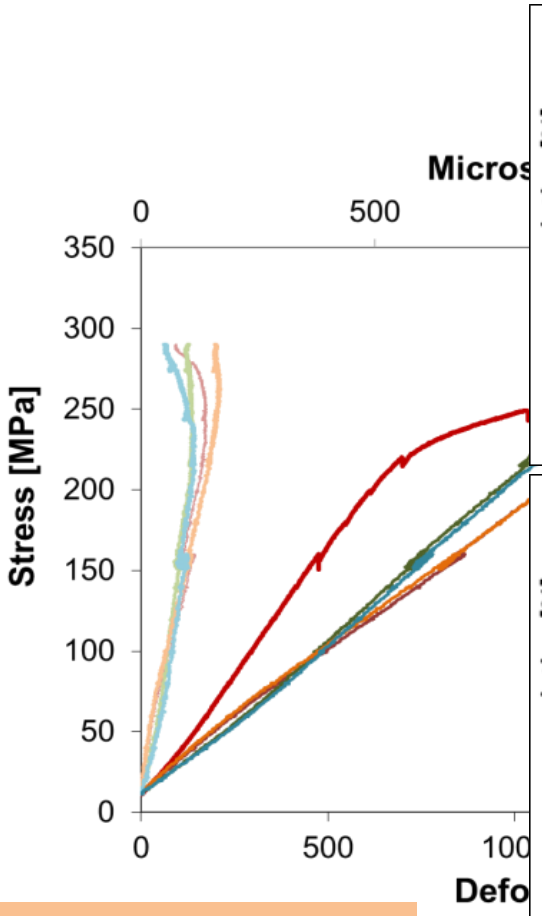


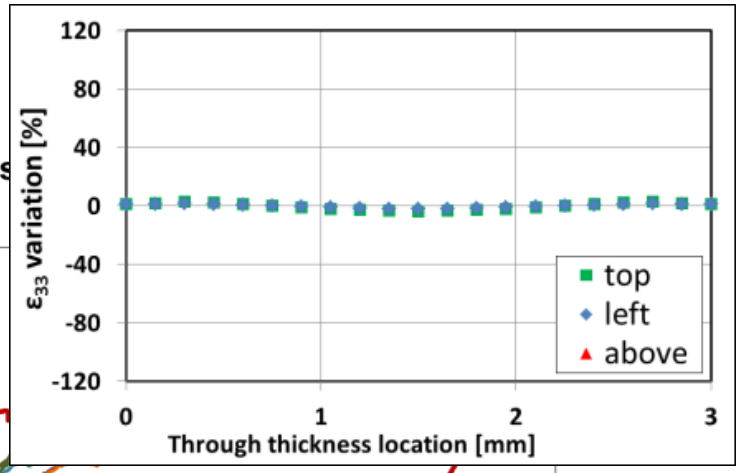


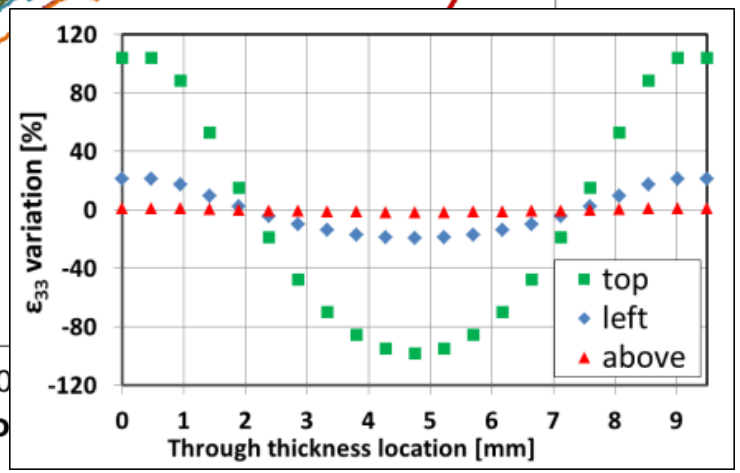
Example 14

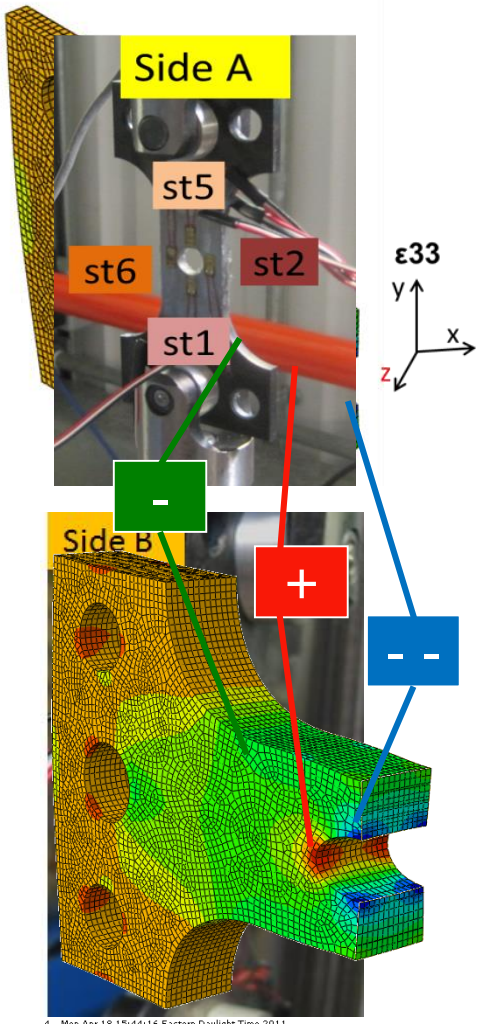
Bragg Edge Imaging: Strain Radiography

- Sample (low carb steel) with stress concentration: Demonstrate high spatial resolution at TOF source using novel Imaging detector
- 2 thicknesses considered







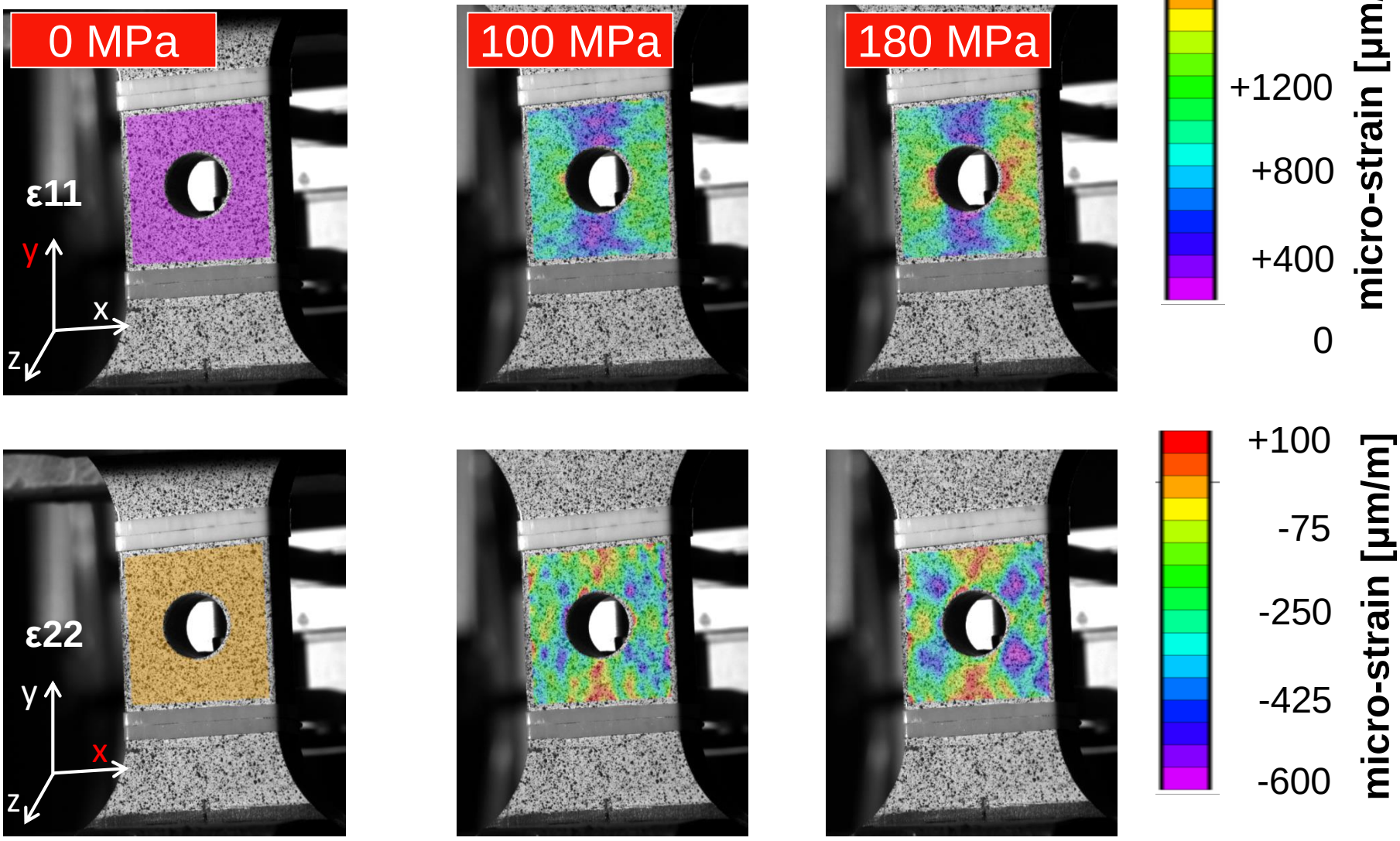


Woracek, Dissertation

Example 14

Bragg Edge Imaging: Strain Radiography

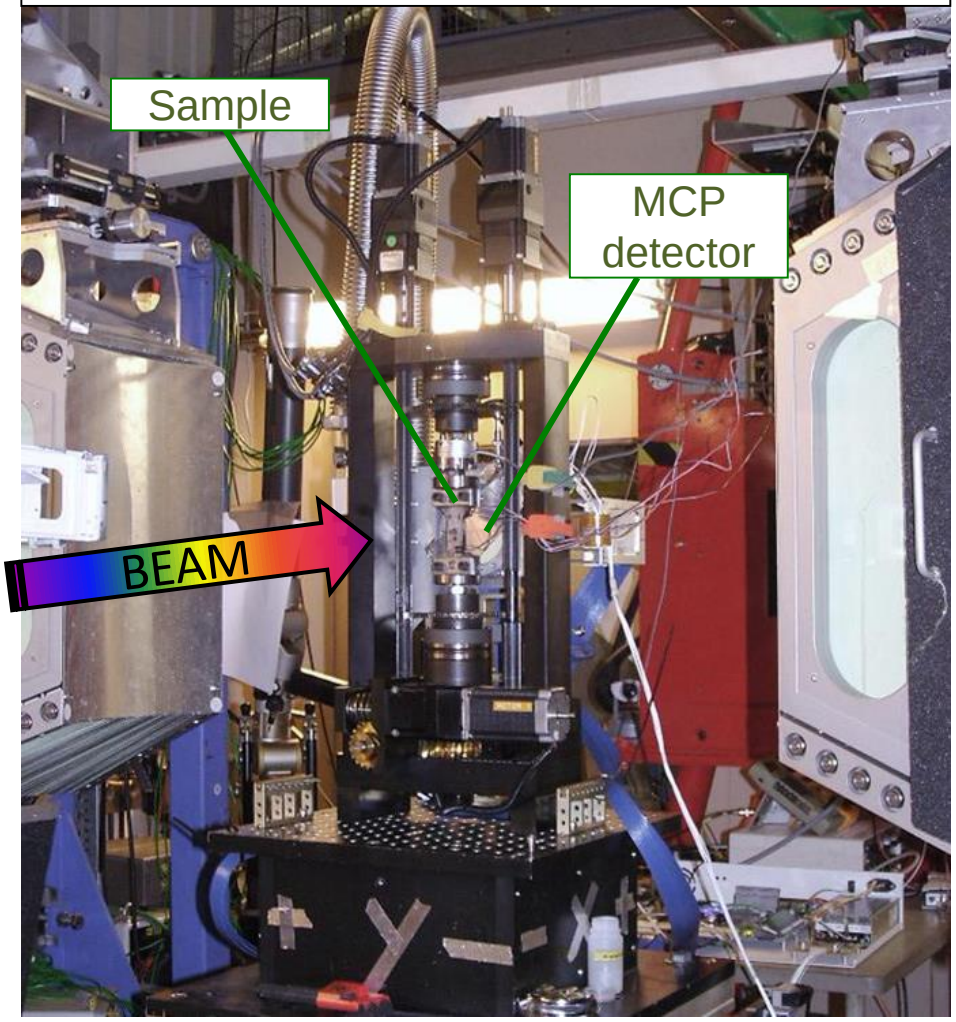
Digital Image Correlation (VIC 3-D): Surface strains



Example 14

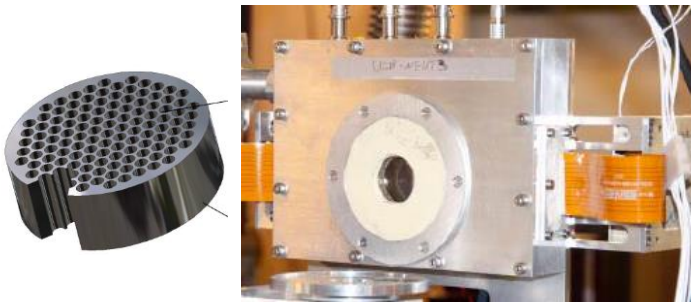
Bragg Edge Imaging: Strain Radiography

Transmission Measurements at ENGIN-X

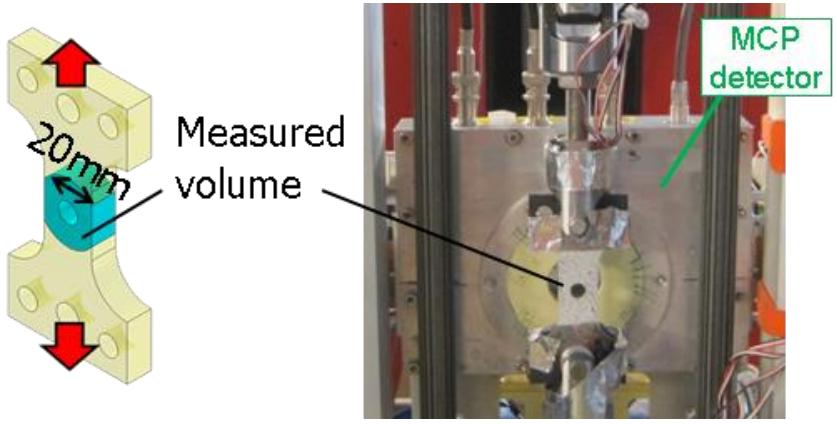


MCP detector (UC Berkeley)

- 28mm FOV, 55um pixels,



(i) A. Tremsin, UC Berkeley

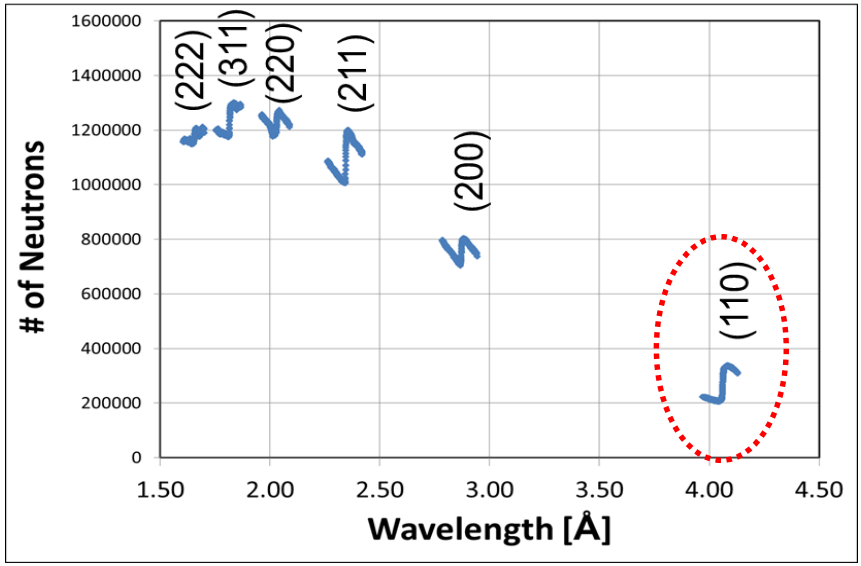


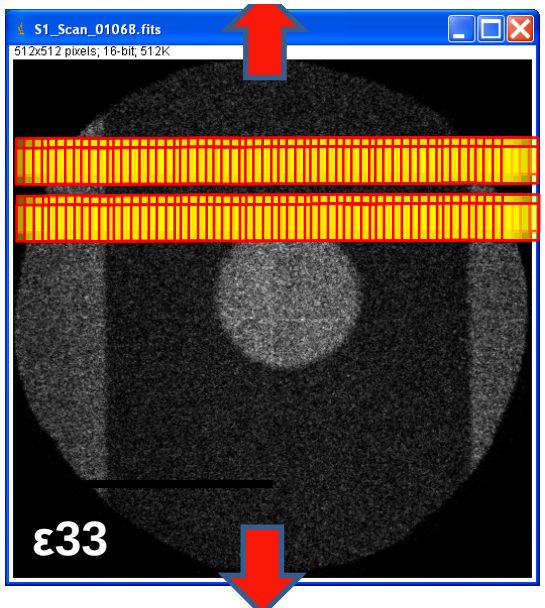
Example 14

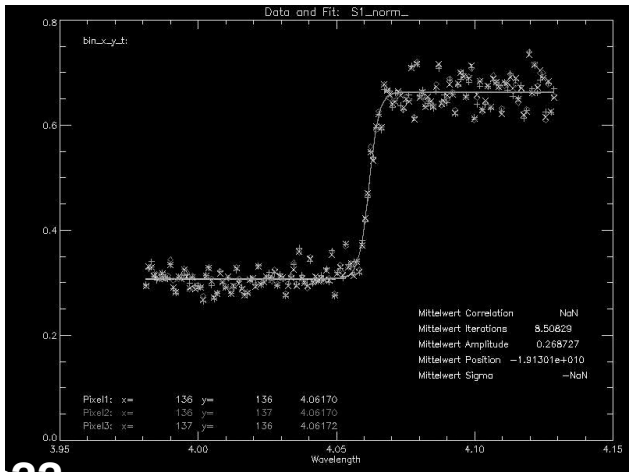
Bragg Edge Imaging: Strain Radiography

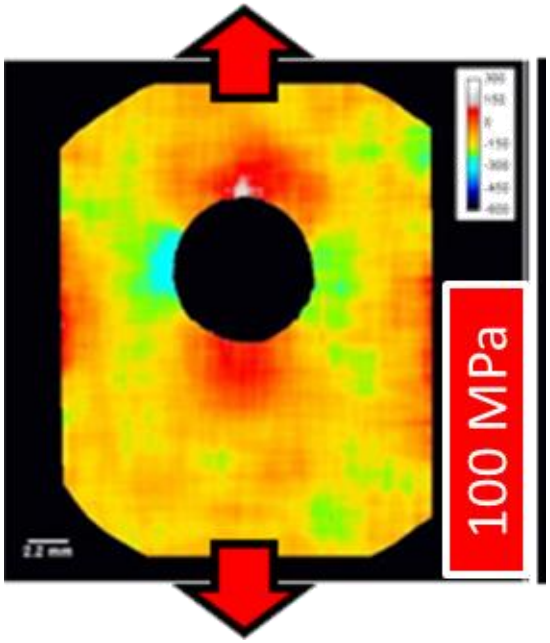
- Record data at few loads
- Pixels are spatially grouped (box 2.1mm) and TOF channel binned (3x15ms; ≈0.001Å)
- Fit is performed for individual edge:

$$f(x) = \frac{1}{A_0 e^{-\left(\frac{x-A_1}{A_2}\right)} + 1} + A_3$$









Example 14

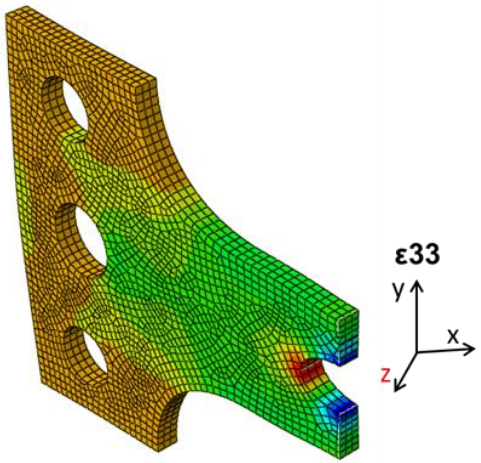
Bragg Edge Imaging: Strain Radiography

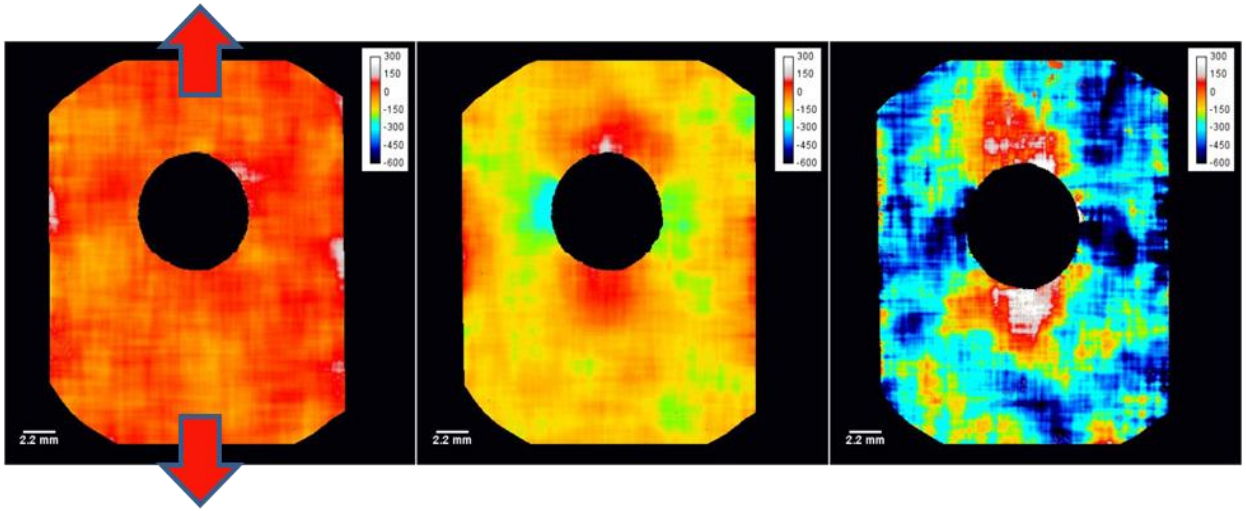
Edge position (strain) parameter for the (110) lattice

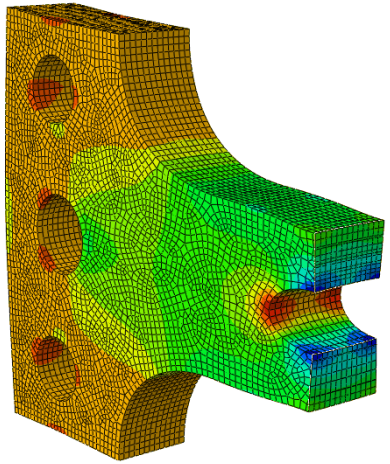
10 MPa

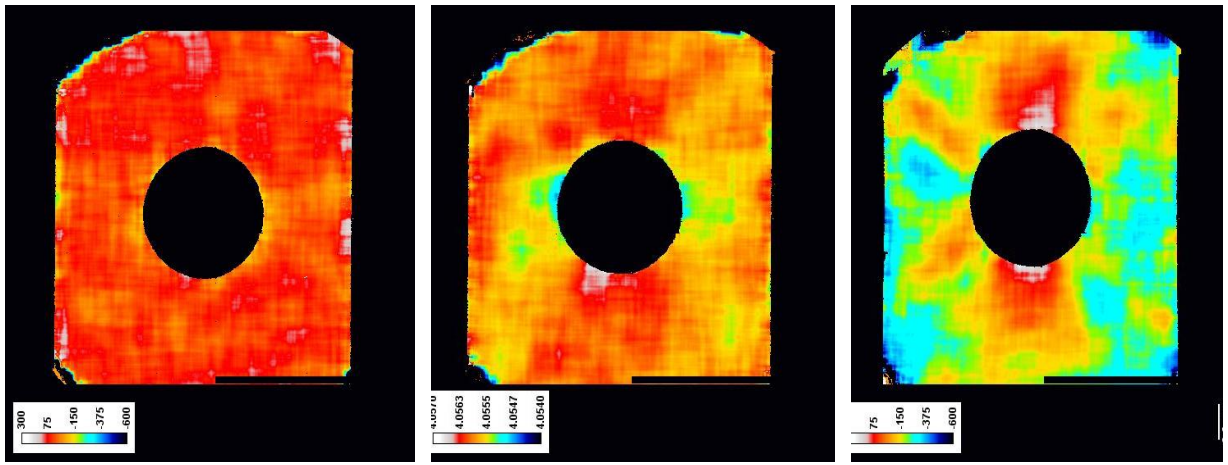
160 MPa

300 MPa









Example 14

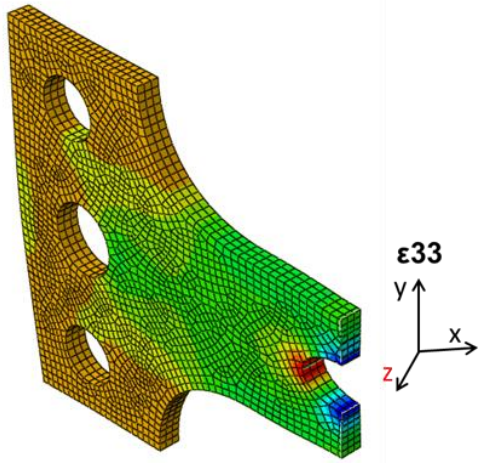
Bragg Edge Imaging: Strain Radiography

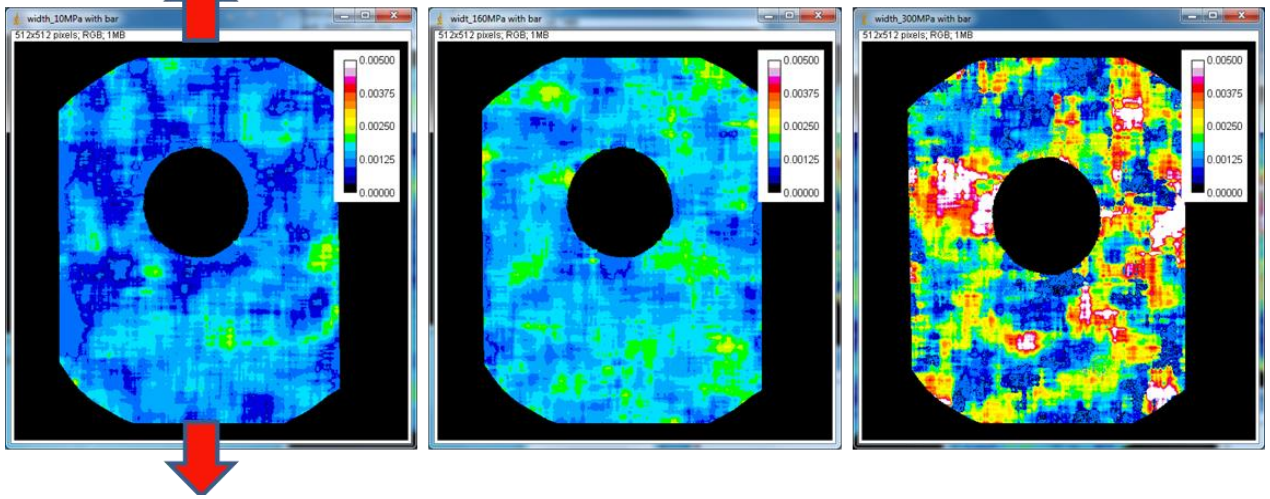
Edge width parameter for the (110) lattice

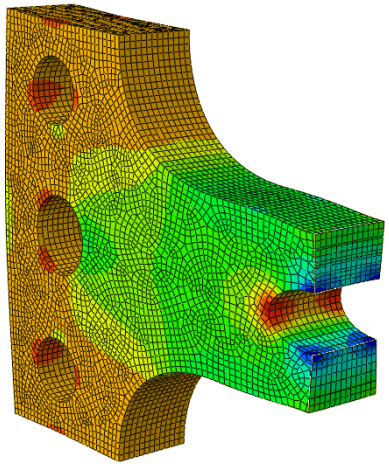
10 MPa

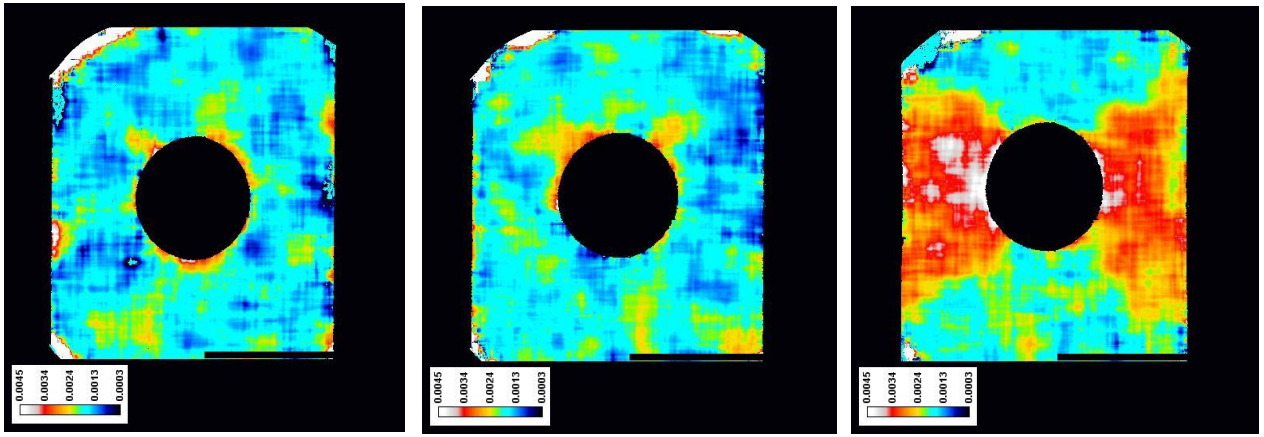
160 MPa

300 MPa









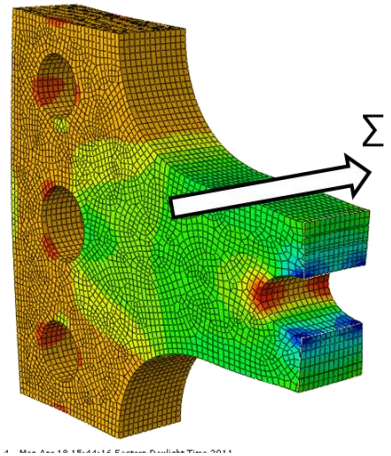
Example 14

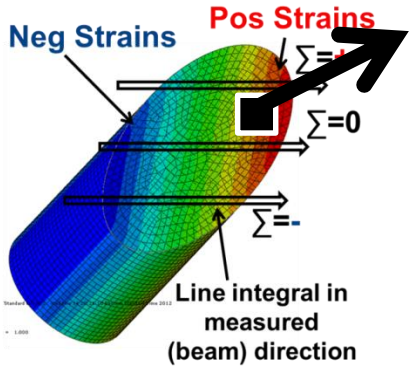
Bragg Edge Imaging: Strain Radiography

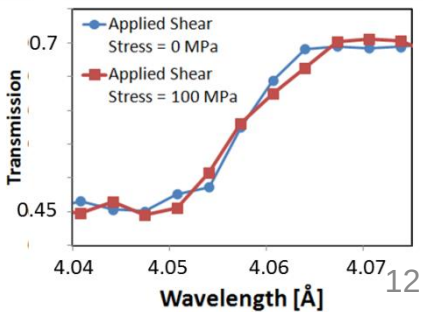
EUROPEAN SPALLATION SOURCE

Conclusions

- Gauge Volume contains complete beam path
- Strain variations though beam paths lead to Bragg edge widening… making quantification difficult
- … but allowing to detect local differences
- Limited choice of scattering vector
- **Neutron and X-Ray diffraction remain method of choice for bulk strain measurements in most cases**
- Useful in specific cases (e.g. plane stress) where precise mapping in one plane is required but check if EDXRD better option

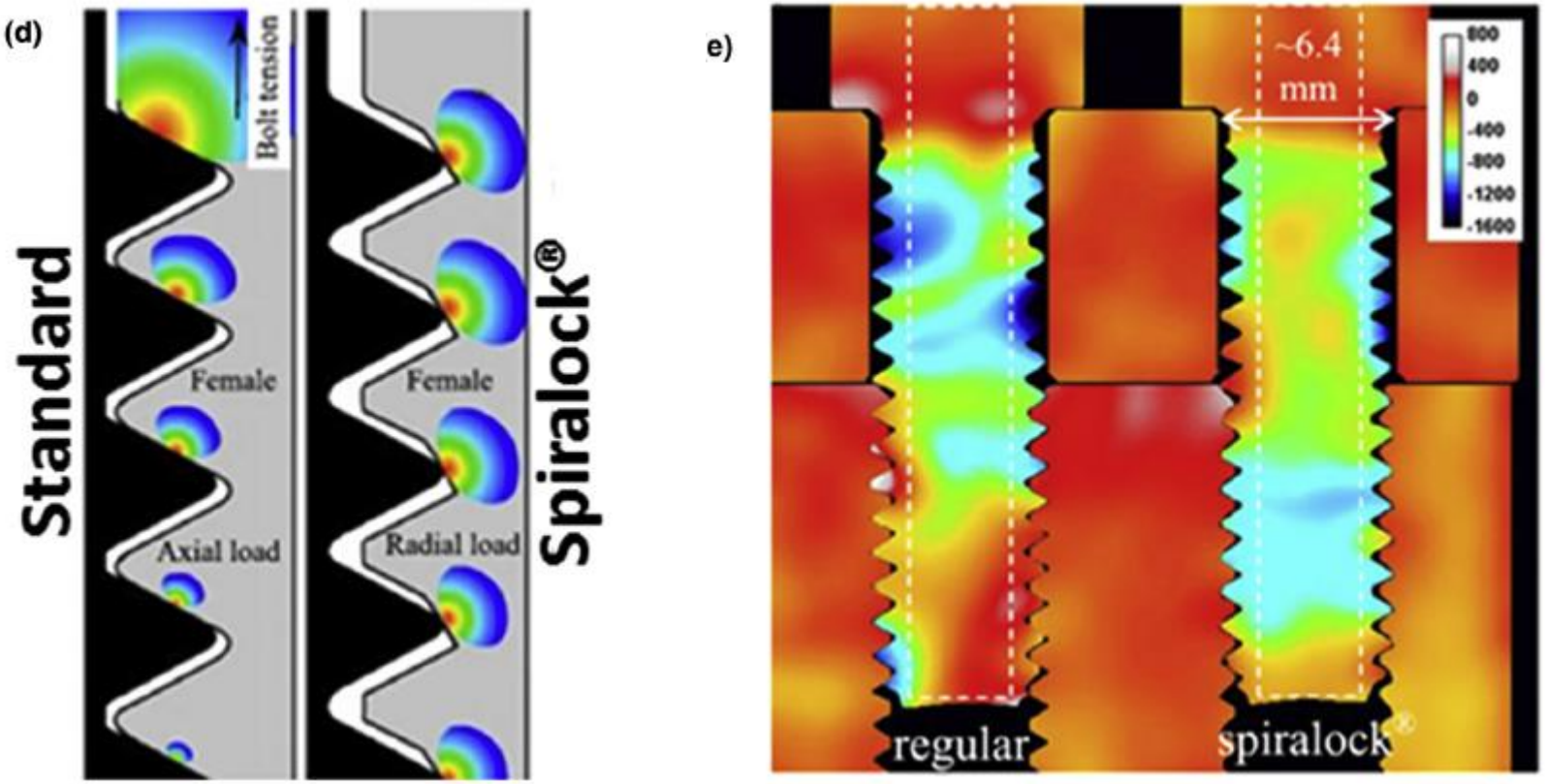






Example 13: Diffraction Contrast

Strain distributions in fasteners



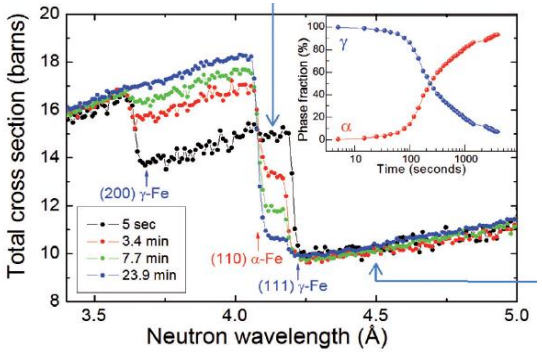
Tremsin, A. S., Yau, T. Y., & Kockelmann, W. (2016). Non-destructive Examination of Loads in Regular and Self-locking Spiralock® Threads through Energy-resolved Neutron Imaging. Strain, 52(6), 548-558.

Advanced Neutron Imaging

Diffraction Contrast

EUROPEAN SPALLATION SOURCE

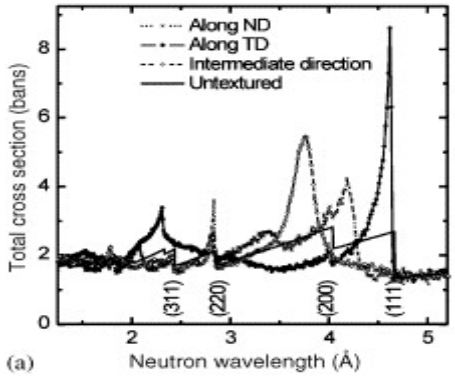
- **Phase**



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)*

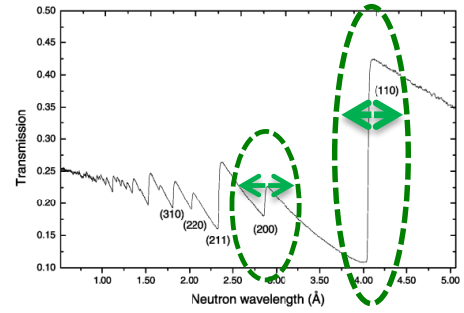
- **Texture**



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).*

- **Strain**



Selected References:

- **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, 093506 (2011)
- Anton paper screw!
- **A. S. Tremsin, T. Y. Yau, W. Kockelmann**, „Non-destructive Examination of Loads in Regular and Self-locking Spiralock® Threads…", Strain 52, 548–558 (2016)

AGENDA

Neutron Imaging in Material Science

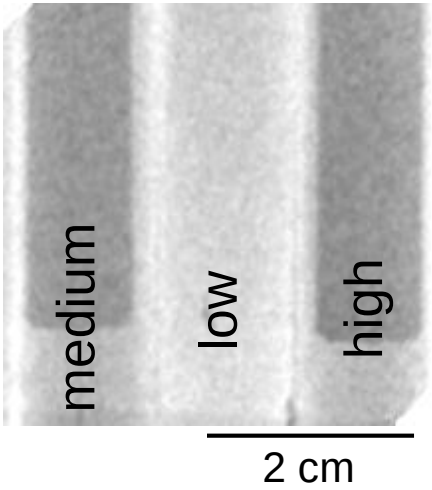
- **Intro**
- **Examples:** ‘White Beam’ Imaging
- **Examples:** Diffraction Contrast Imaging
- **Examples:** Phase Contrast Imaging
- **Examples:** Diffractive Imaging

Example 15: Phase & Dark Field Contrast

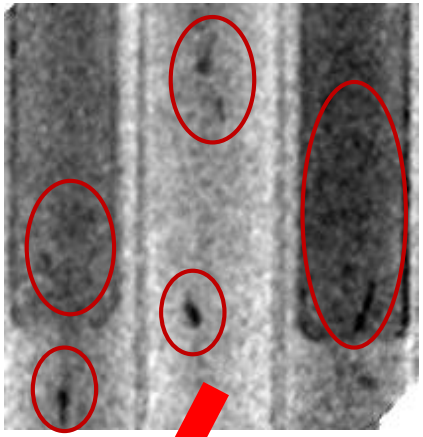
Cast ingots

Cast AlSi ingots

Attenuation

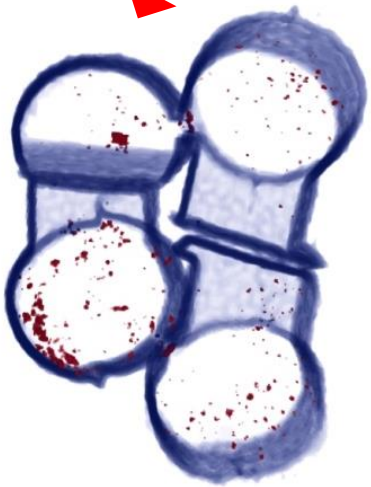


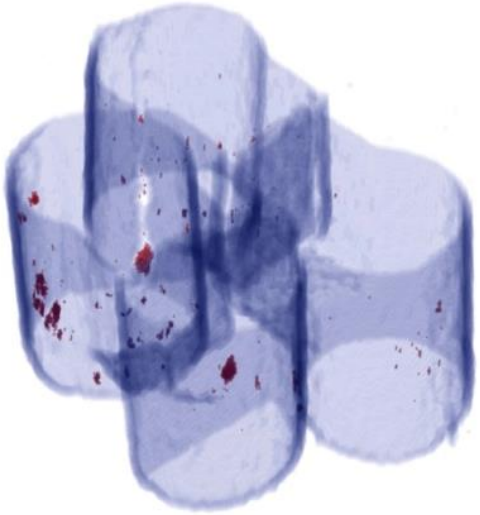
Darkfield/small angle scattering



Nano-/microsized pores + precipitates

Small angle scattering in 3D reveals nano/micro defects





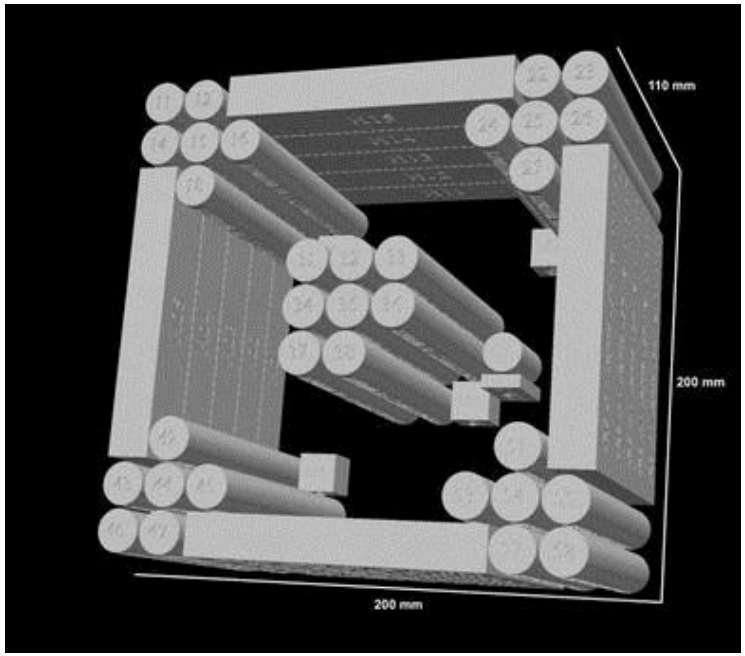
Hilger, A., et al. "Revealing microstructural inhomogeneities with dark-field neutron imaging." (2010)

Example 16: Phase & Dark Field Contrast

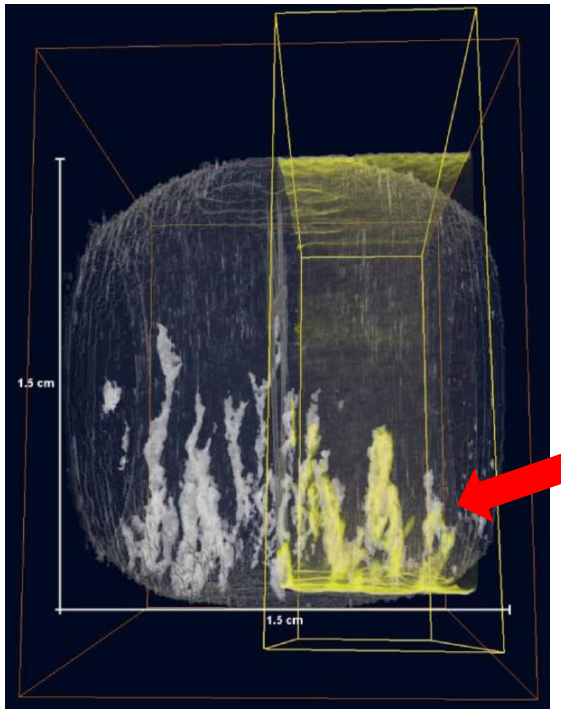
3D printing

Example: Additive manufacuring

Printed test sample



Defects caused by printing process



defects

Porosity detection in electron beam-melted Ti-6Al-4V using high-resolution neutron imaging and grating-based interferometry

A. J. Brooks, J. Ge. M. M. Kirka. R. R. Dehoff, H. Z. Bilheux, N. Kardjilov, I. Manke, L. G. Butler
Progress in Additive Manufacturing 2, pp 125 (2017)

AGENDA

Neutron Imaging in Material Science

- **Intro**
- **Examples:** ‘White Beam’ Imaging
- **Examples:** Diffraction Contrast Imaging
- **Examples:** Phase Contrast Imaging
- **Examples:** Diffractive Imaging

Ongoing Method Development

3D metallurgy in bulk samples

What we can do now:

Phase	Radiography & Tomography	non-textured samples
Texture (qual.)	Radiography	textured samples
Strain	Radiography	(non-)textured samples

Ongoing Method Development

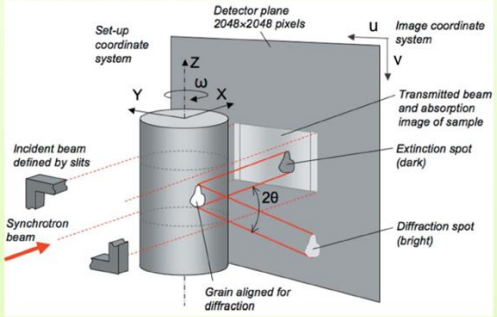
3D metallurgy in bulk samples

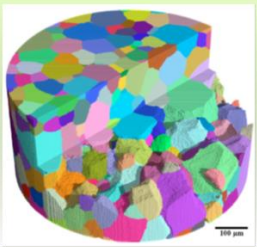
What we **want to do**:

Phase	Radiography & Tomography	textured samples
Texture (**quant.**)	Radiography **& Tomography**	textured samples
Strain	Radiography **& Tomography**	(non-) **textured samples**

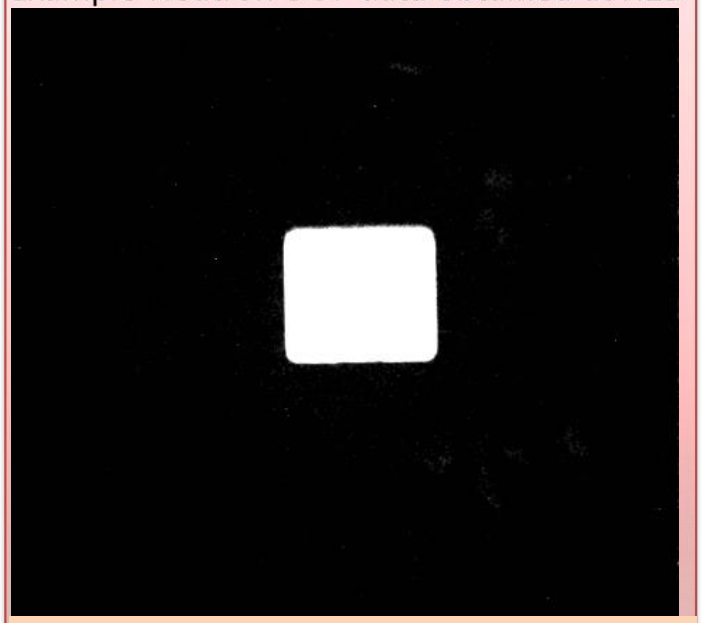
How to do it? **Combined Imaging & Diffraction**

From W. Ludwig et al.: Experimental Setup and 3-D grain orientation map obtained at ESRF

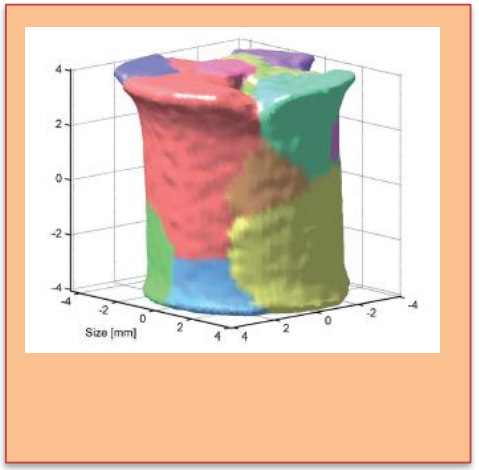




Example Neutron-DCT data obtained at HZB



R. Woracek, N. Kardjilov, W. Ludwig, unpublished



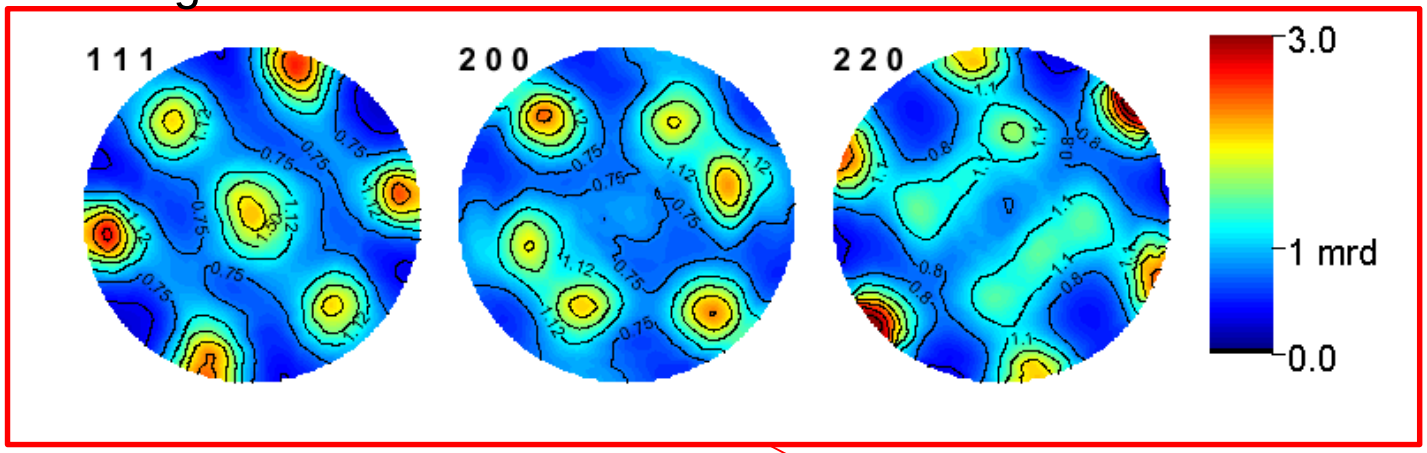
S. Peetermans, A. King, W. Ludwig, P. Reischig, E. Lehmann, **Cold neutron diffraction contrast tomography of polycrystalline material**. *Analyst* 139, (2014).

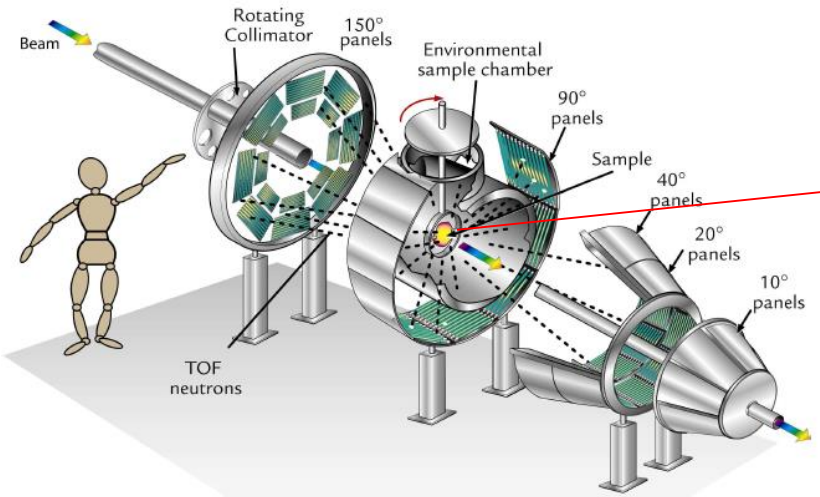
Ongoing Method Development

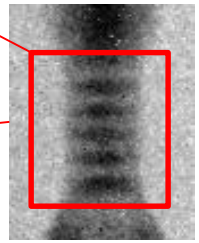
Motivational example

- The TRIP samples were investigated at neutron texture instruments:
  Complete gauge section is averaged

Pole Figures measured at HIPPO:



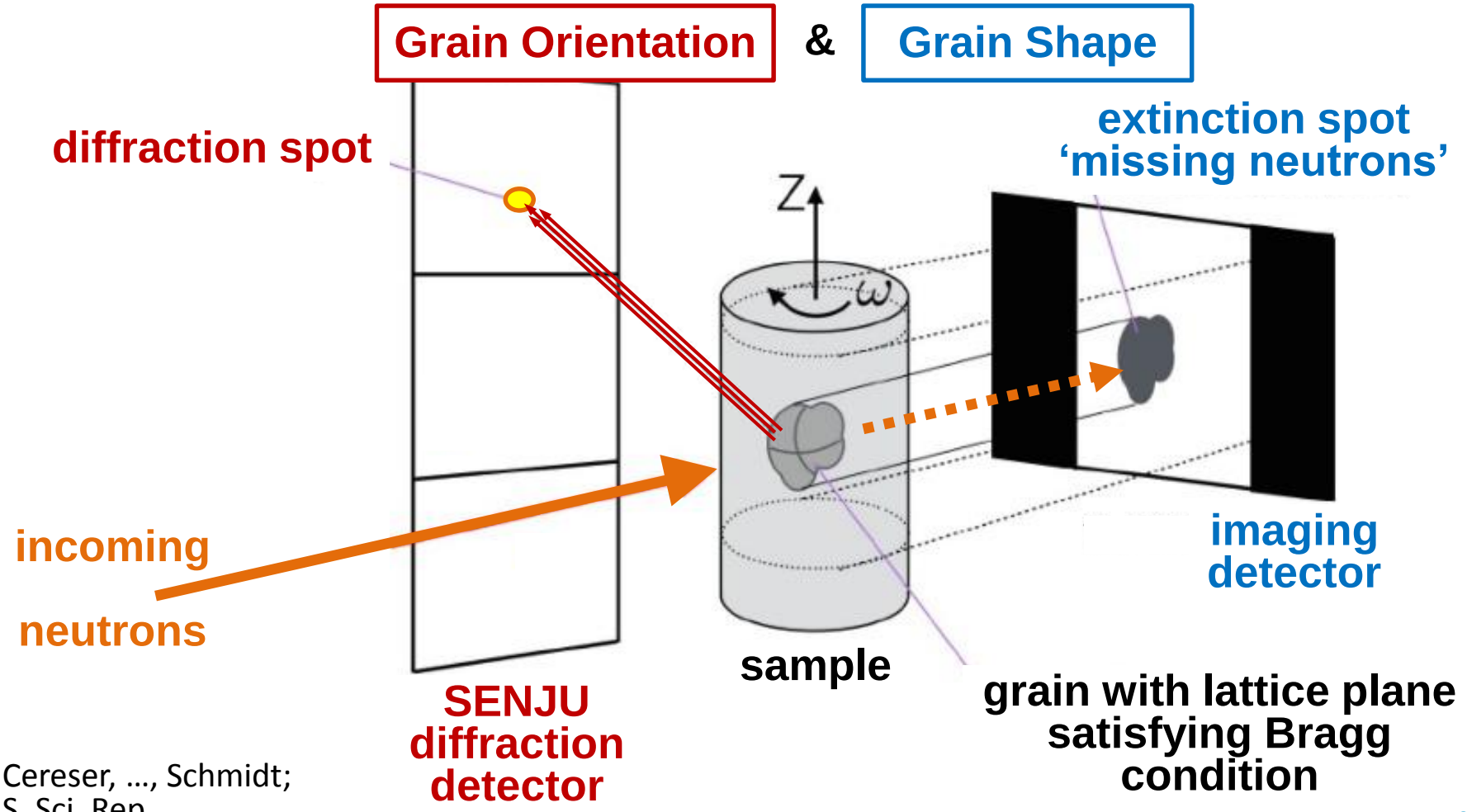




Ongoing Method Development

3D metallurgy in bulk samples

3 Dimensional Neutron Diffraction (3DND)



Cereser, …, Schmidt;
S. Sci. Rep.,
2017, 7, 9561.

Ongoing Method Development

3D metallurgy in bulk samples

3DND *in situ* Deformation at SENJU @ J-PARC

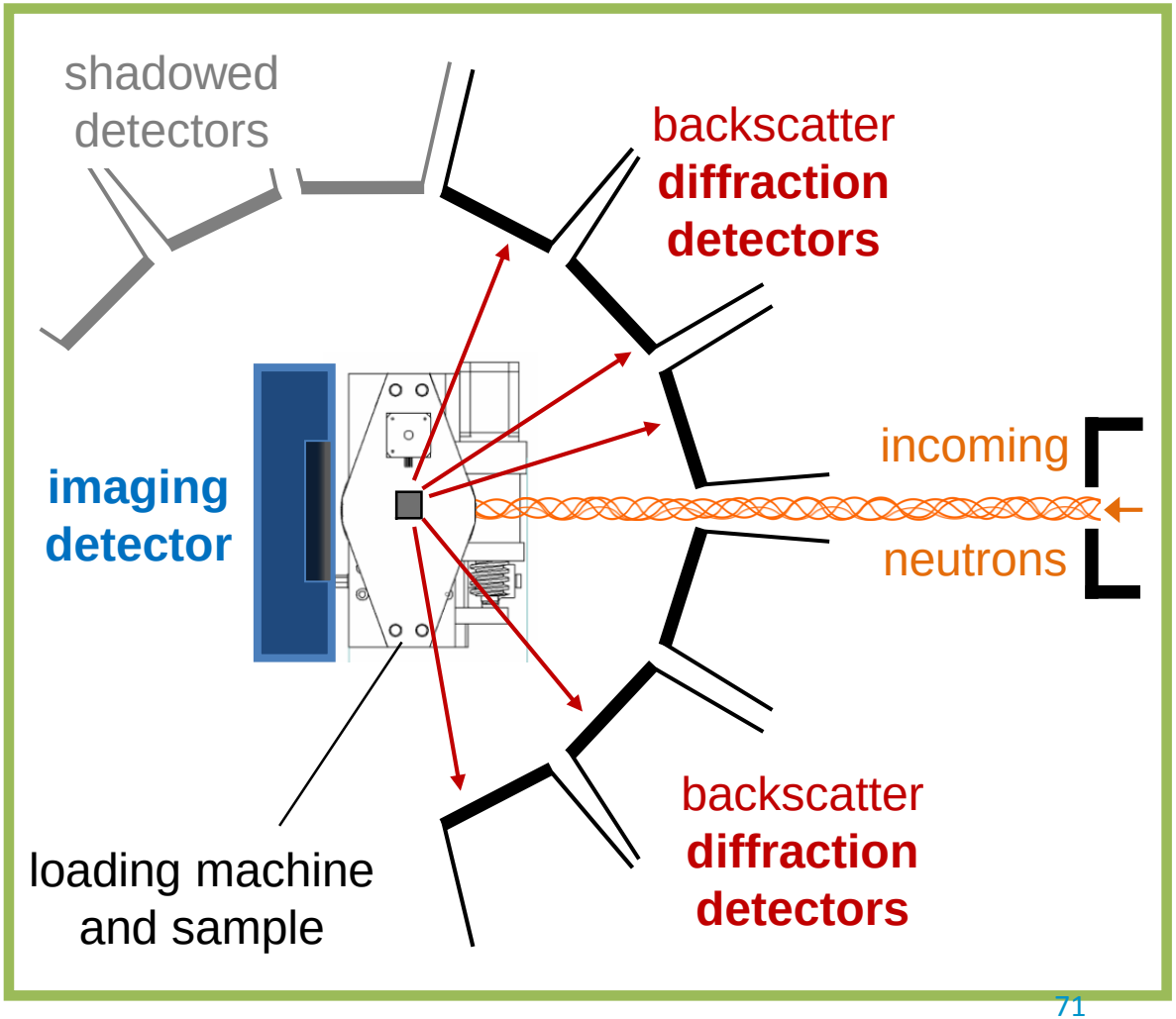
Multicrystalline
CuAlMnZn
Shape Memory Alloy
(mm grains)

Tomography
+
Diffraction

- **Grain Shape**
- **Grain Orientation**
- **Austenite/Martensite**

→ **Grain Interactions**

Top View

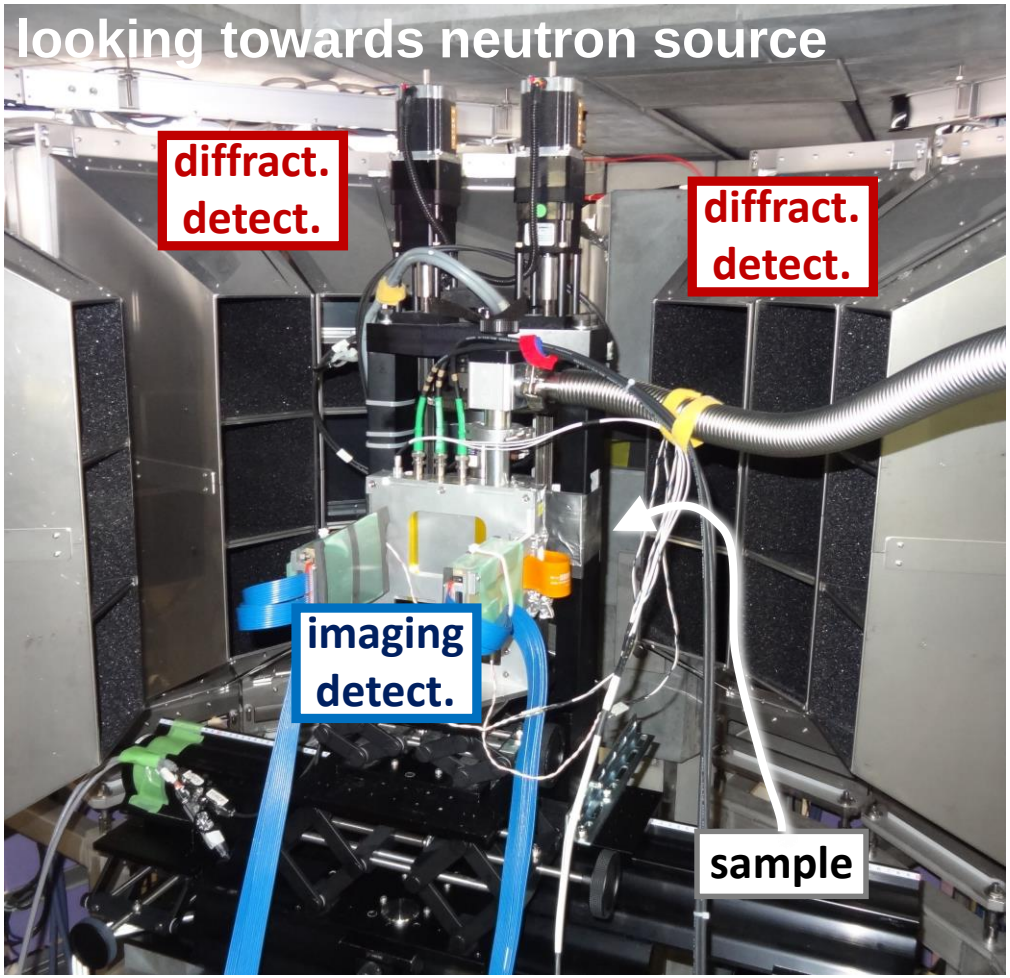


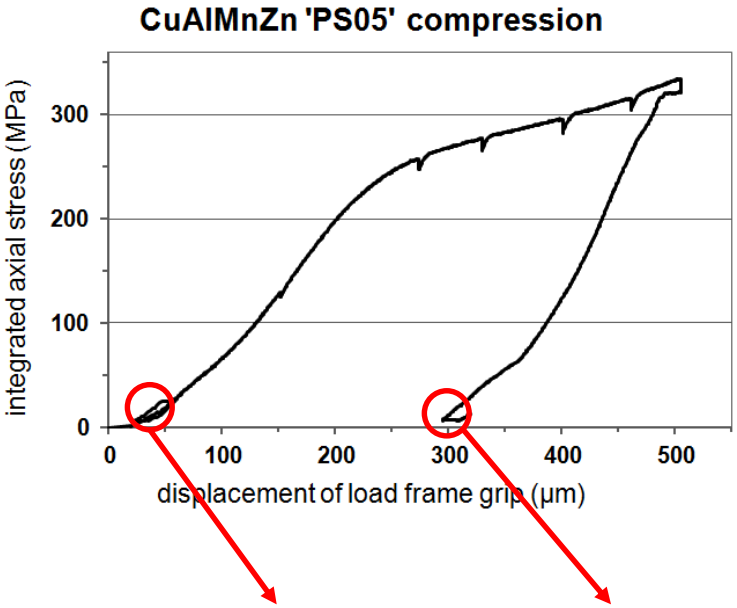
Ongoing Method Development

3D metallurgy in bulk samples

EUROPEAN SPALLATION SOURCE

3DND *in situ* Deformation





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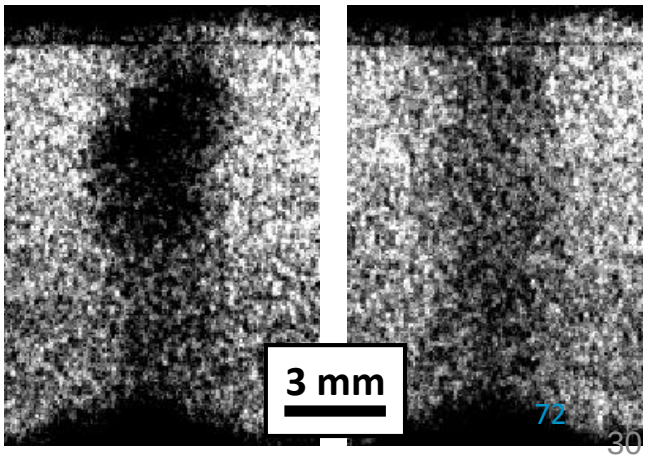
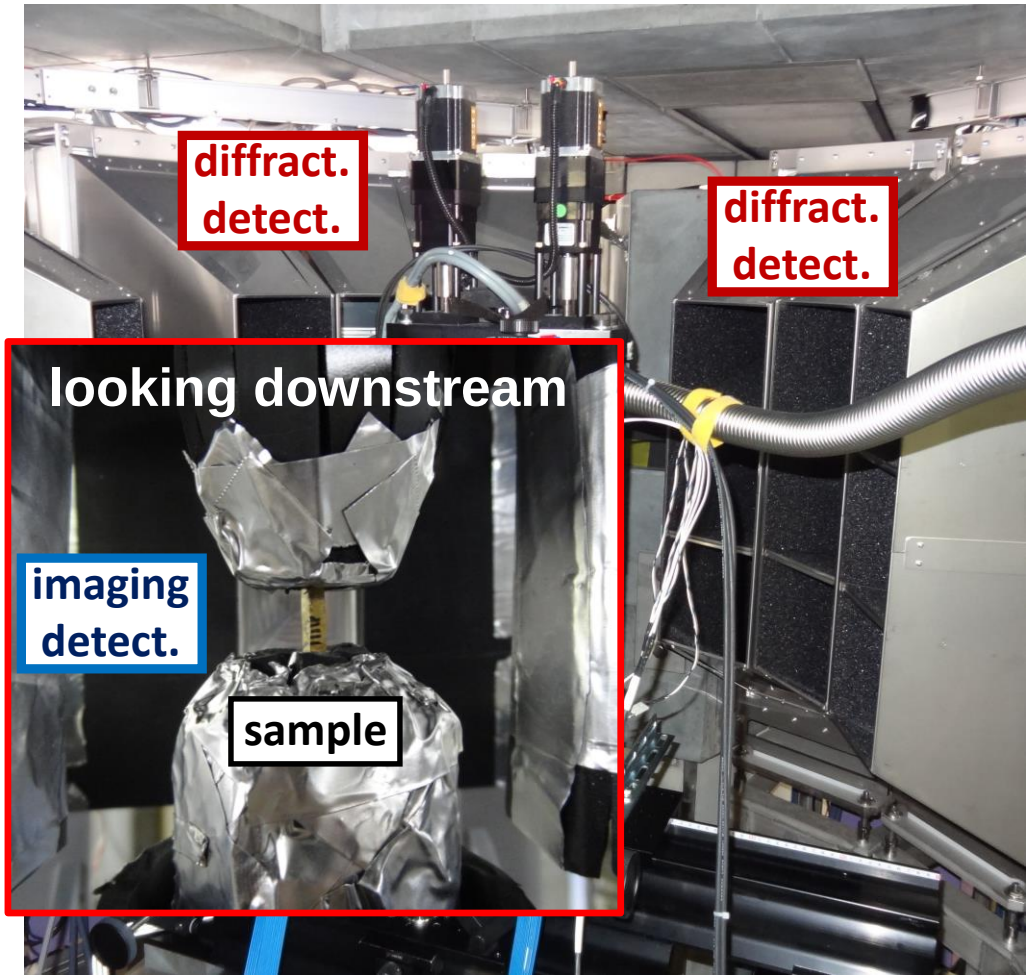


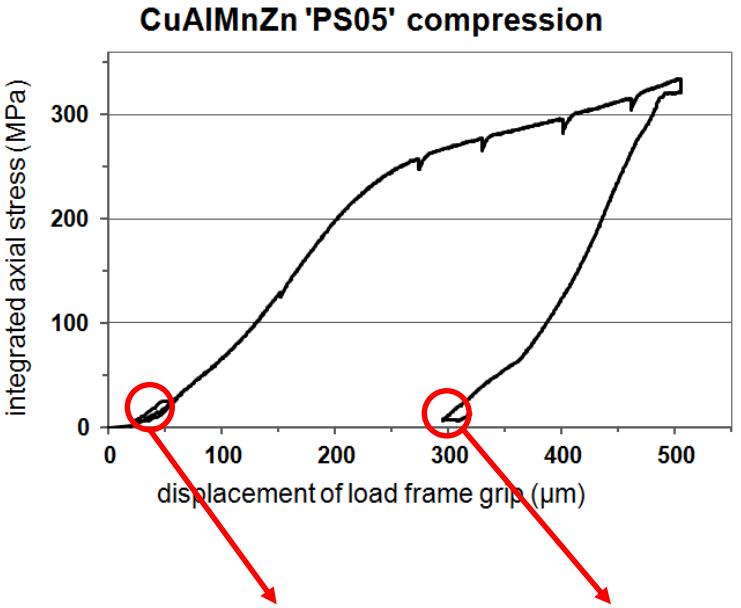
Ongoing Method Development

3D metallurgy in bulk samples

EUROPEAN SPALLATION SOURCE

3DND *in situ* Deformation





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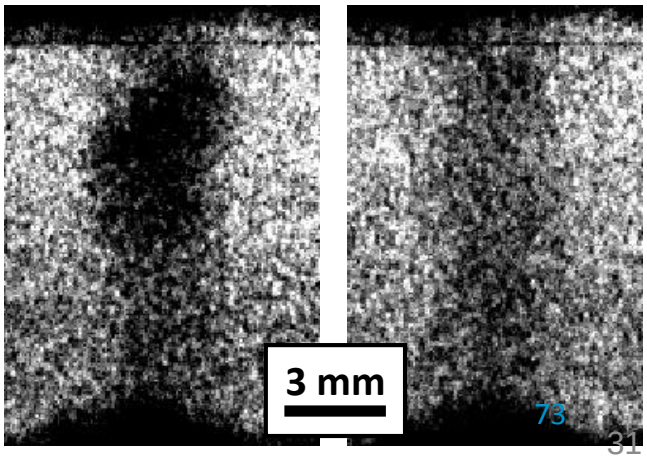
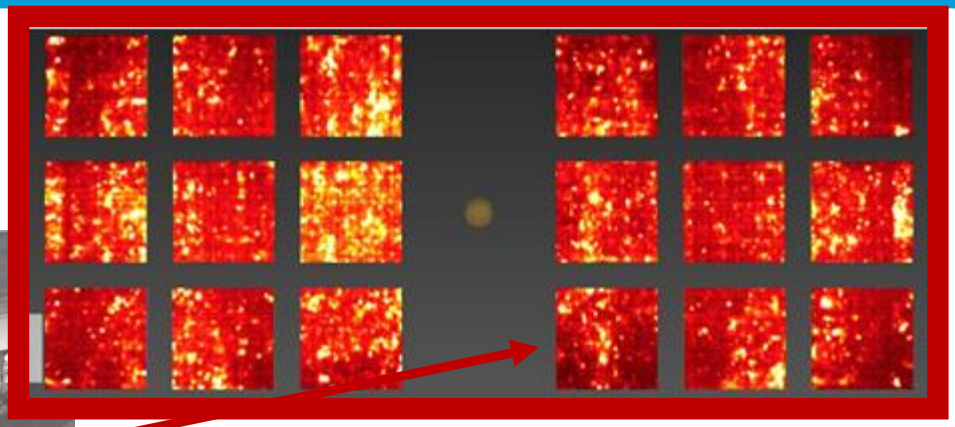


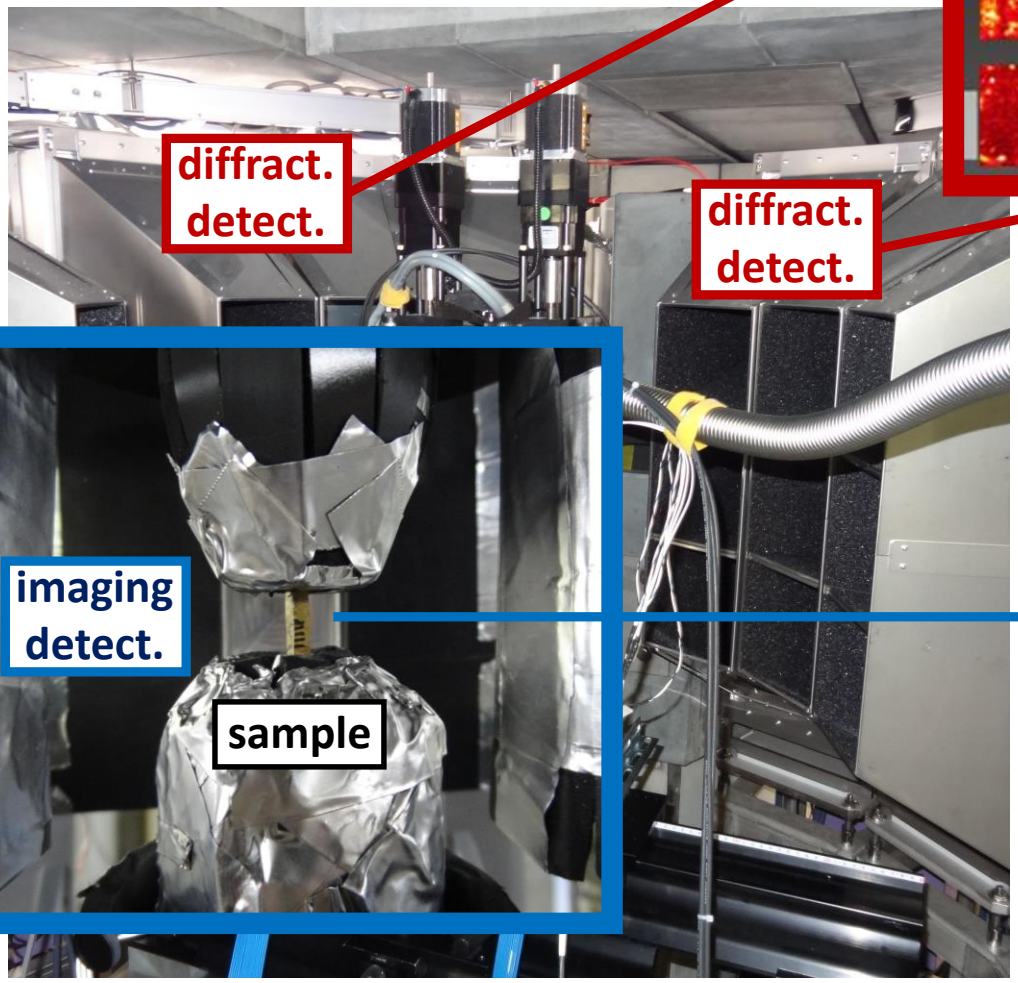
Ongoing Method Development

3D metallurgy in bulk samples

EUROPEAN SPALLATION SOURCE

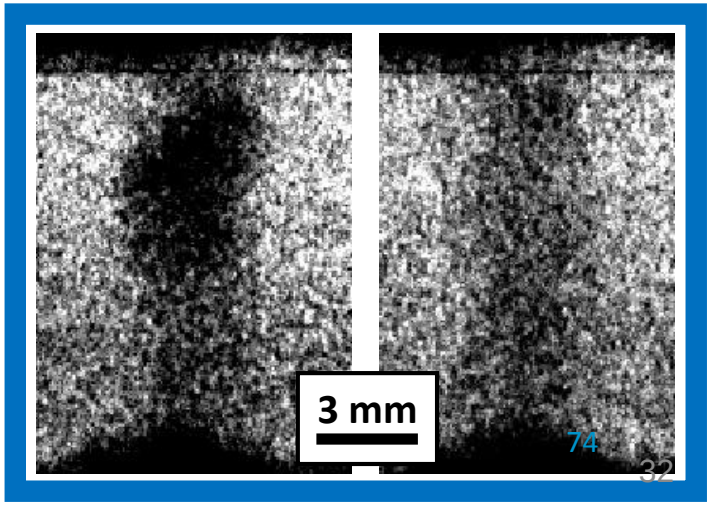
3DND *in situ* Deformation





3D representation of crystalline microstructure with austenite and martensite

before loading after loading



THANK YOU!

EUROPEAN SPALLATION SOURCE