This presentation was mainly concerned with imaging. Diffraction was covered by Jochen Fenske (HZG) and Joe Kelleher (STFC). We can share additional information on diffraction as well.

Robin Woracek
Instrument Class Coordinator Imaging & Engineering

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Markus Strobl (Head of Neutron Imaging Group at PSI)
markus.strobl@psi.ch

www.europeanspallationsource.se
Some references related to Neutron Imaging

**Corrosion**
- Examples herein

**Welding**

**Hyrogen studies**

**Strain Mapping**

**Phase Mapping**
AGENDA

ESS: The most intense neutron source for material research

Why Neutrons?
- Example: Neutron Diffraction for residual stress
- Example: Neutron Imaging to detect hydrogen

Introduction: Neutron Imaging (Absorption Contrast)
- Examples 1-5: Corrosion in metals

Introduction: Diffraction Contrast in Neutron Imaging
- Examples 6-8: Phase transformations, Welds

Summary & Discussion

Some references
OECD recommendation 2006
“The Neutron Sources Working Group recommends a scenario which aims at the construction of advanced neutron sources in each of the three regions Asia/Pacific rim, Europe and North America, to be operational within 20 years, and catering for regional needs in a wide range of scientific and technological applications.”

- J-PARC, Japan
- SNS, USA
- ESS, Europe

Existing Neutron Sources

**Spallation**
- no chain reaction
- pulsed operation
- 30 neutrons/proton
- Time resolved exp.

**Fission**
- chain reaction
- continuous flow
- 1 neutron/fission

Source influences the neutron imaging (diffraction) experimental setup
ESS - A European Big Science Project

1843 M€ construction
140 M€/yr operations

- 5 MW proton accelerator
- rotating tungsten target

Sweden and Denmark:
47,5% Construction
15-20% Operations

Partner Countries:
52,5% Construction
80-85% Operations
AGENDA

- **ESS**: The most intense neutron source for material research

- **Why Neutrons?**
  - Example: Neutron Diffraction for residual stress
  - Example: Neutron Imaging to detect hydrogen

- **Introduction: Neutron Imaging (Absorption Contrast)**
  - Examples 1-5: Corrosion in metals

- **Introduction: Diffraction Contrast in Neutron Imaging**
  - Examples 6-9: Phase transformations, Welds

- **Summary & Discussion**
Some Background

Properties of neutrons for measurements

→ High contrast for heavy elements
→ High transparency for hydrogenous / organic material

Attenuation characteristics

<table>
<thead>
<tr>
<th>Atomic number</th>
<th>Hydrogen</th>
<th>Boron</th>
<th>Carbon</th>
<th>Oxygen</th>
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<th>Iron</th>
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X-ray:
- High contrast for heavy elements
- High transparency for hydrogenous / organic material

Neutrons:
- High sensitivity for hydrogenous / organic material
- High transparency for heavy elements
Some Background

Properties of neutrons for measurements

- Different interaction behavior results in different interaction probabilities and attenuation coefficients for the different elements:

| 1a  | 2a  | 3b | 4b | 5b | 6b | 7b | 8  | 1b | 2b | 3a | 4a | 5a | 6a | 7a | 0      |
|-----|-----|----|----|----|----|----|----|----|----|----|----|----|----|----|        |
| H   | 0.02|     |    |    |    |    |    |    |    |    |    |    |    |    |        |
| Li  | 0.06| Be | 0.22|    |    |    |    |    |    |    |    |    |    |    |    |        |
| Na  | 0.13| Mg | 0.24|    |    |    |    |    |    |    |    |    |    |    |    |        |
| K   | 0.14| Ca | 0.26| Sc | 0.48| Ti | 0.73| V  | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb  | 0.47| Sr | 1.61| Y  | 2.47| Zr | 3.43| Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I  | Xe |
| Cs  | 1.42| Ba | 2.73| La | 5.04| Hf | 19.70| Ta | W  | Re | Os | Ir | Pt | Au | Hg | Ti | Pb | Bi | Po | At | Rn |
| Fr  | 11.80| Ra | 24.47| Ac | 75.49| Rf | 34.47| Re | Os | Ir | Pt | Au | Hg | Ti | Pb | Bi | Po | At | Rn | 9.77|

**X-ray**

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<th>Ce</th>
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Some Background

Properties of neutrons for measurements

- Different interaction behavior results in different interaction probabilities and attenuation coefficients for the different elements:

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* 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 86.3, Bk 61.2, Cf 5.05, Es 1.21, Fm 0.25, Md 0.05, No 0.25, Lr neut.
Some Background

Properties of neutrons for measurements

Neutrons ‘see’ light elements

Courtesy of PSI, NIST
Introduction

Properties of neutrons for measurements

- Charge neutral
  Deeply penetrating

- Magnetic moment (spin)
  Probe of magnetism

- Nuclear scattering
  Sensitive to light elements and isotopes

- H motion in fuel cells

- Solve the high-temperature Superconductivity puzzle

- Efficient high-speed trains

- Active sites in proteins

- Improve electric cars

- Better drugs

Specialized instruments and methods
## Introduction

**ESS: Specialized instruments and methods**

### Large-Scale Structures

<table>
<thead>
<tr>
<th>Diffraction</th>
<th>Multi-Purpose Imaging</th>
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<td>Materials Science Diffractometer</td>
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<td>Extreme Conditions Diffractometer</td>
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### Spectroscopy

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<th>Cold Chopper Spectrometer</th>
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<td>Bispectral Chopper Spectrometer</td>
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<td>Thermal Chopper Spectrometer</td>
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<td>Vibrational Spectroscopy</td>
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<td>Fundamental &amp; Particle Physics</td>
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### Research Areas

- Life sciences
- Magnetism & superconductivity
- Soft condensed matter
- Engineering & geo-sciences
- Chemistry of materials
- Archeology & heritage conservation
- Energy research
- Fundamental & particle physics
Introduction

ESS: Specialized instruments and methods

Large-Scale Structures

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Description</th>
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Diffraction

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Specialized instruments and methods

- life sciences
- magnetism & superconductivity
- soft condensed matter
- engineering & geo-sciences
- chemistry of materials
- archeology & heritage conservation
- energy research
- fundamental & particle physics
Introduction

ESS: Specialized instruments and methods

Beamline for European Materials Engineering Research (BEER)

Optical and Diffraction Imaging with Neutrons (ODIN)
AGENDA

- **ESS**: The most intense neutron source for material research

- Why Neutrons?
  - Example: Neutron Diffraction for residual stress
  - Example: Neutron Imaging to detect hydrogen

- **Introduction**: Neutron Imaging (Absorption Contrast)
  - Examples 1-5: Corrosion in metals

- **Introduction**: Diffraction Contrast in Neutron Imaging
  - Examples 6-8: Phase transformations, Welds

- **Summary & Discussion**
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.

In general, compressive stresses at a surface are beneficial and enhance resistance to failure.
Neutrons for Engineering
Diffraction: Residual Stress

**Surface**
Angle dispersive X-ray methods

Energy range \( \approx 5 – 17 \text{ keV} \)
Information depth (steel) \( \approx 15 \mu\text{m} \)

**Intermediate zone**
Energy dispersive X-ray diffraction

Energy up to about 120 keV
Information depth (steel) \( \approx 100 \mu\text{m} \)

**Bulk**
Neutron diffraction
(High energy synchrotron diffraction)

Information depth (steel): some cm

Nondestructive characterization from surface to volume!
Nondestructive characterization from surface to volume!

Neutrons for Engineering
Diffraction: Residual Stress

Courtesy of M. Boin
Measure Strain and then convert to Stress

**Bragg's Law:**

\[ n\lambda = 2d^{hkl}\sin\theta \]

**Elastic strain:**

\[ \varepsilon = \left( \frac{\Delta d}{d_0} \right) = \frac{\sin\theta_0}{\sin\theta} - 1 \]

**Triaxial stress:**

- **Hooke's Law**

\[ \sigma_{ij} = C_{ijkl} \varepsilon_{kl} \]

\[ \sigma_i = \frac{E}{1+\nu} \varepsilon_i + \frac{\nu E}{(1+\nu)(1-2\nu)} (\varepsilon_x + \varepsilon_y + \varepsilon_z) \]

- **E** – Young's modulus
- **\( \nu \)** – Poisson's ratio
AGENDA

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- Summary & Discussion
Hydrogen loading of duplex stainless steel

Electrochemically loaded

Resolution: 15 µm (pixel size: 6.5 µm), FOV: 13 x 13 mm², 600 proj. x 60 s

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

Imaging: Hydrogen embrittlement of steels

Quantification

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

A. Griesche et al., Acta Materialia 78 (2014)
11 bar of $H_2$

$(\text{LaNi}_{4.8}\text{Al}_{0.2})$

AGENDA

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- **Summary & Discussion**
Neutron Imaging

Introduction

Spatial Resolution

\[ I_0 \sim I_0 e^{-\int \Sigma(x) dx} \]

\( I_0 \) – primary beam
\( \Sigma(x) \) – attenuation coefficient

Spatial Resolution

D – Collimator aperture
L – Distance Collimator-Object
l – Distance Object-Detector

\[ d = \frac{l}{L/D} \]

more: [www.psi.ch/niag/](http://www.psi.ch/niag/)
Neutron Imaging

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

M. Strobl & F. Grazzi (2015) From scattering in imaging to prospects at pulsed sources, Neutron News 26
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- **Summary & Discussion**
Examples 1-5: Neutron Imaging

Corrosion

Neutrons penetrate metals quite easy, while providing contrast for light elements.

Well suited to investigate corrosion

Was a ‘hot topic’ some decades ago, but applications with industrial background were rather limited in the last few years...

X-rays complementary
Example 1: Neutron Imaging

Corrosion in cultural heritage objects

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

Questions:
- Identification of shape/size of lead sheets; inner wood sculpture
- Mounting points / joints (nails and soldering)
- Extent of carbonate corrosion

→ Strategy for restauration

Appraisal as basic information for:
→ Decision for conservatory treatment
→ Development of new conservatory treatments

Courtesy of
Example 1: Neutron Imaging
Corrosion in cultural heritage objects

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

inner wooden kernel

areas affected by corrosion (red)
fixation by nails and soldering (blue)
Example 2: Neutron Imaging

Corrosion: Documentation of processes

X-ray $\rightarrow$ information on density
Neutrons $\rightarrow$ information on element changes (i.e. H, Cl,...)

Courtesy of M. Jacot, C. Gervais, K. Schmidt-Ott
Example 2: Neutron Imaging

Corrosion: Documentation of processes

Plan of experiment

Dechlorination treatment (~3 months)

Courtesy of M. Jacot, C. Gervais, K. Schmidt-Ott
Example 2: Neutron Imaging

Corrosion: Documentation of processes

X-ray

Before

After

Before

After

Difference

Before - After > 0

Before - After < 0

Courtesy of M. Jacot, C. Gervais, K. Schmidt-Ott
Example 2: Neutron Imaging

Corrosion: Documentation of processes

Information from X-ray & neutron CT
→ Verification
→ Base for segmentation

Variable concentration of iron and chloride blur the interpretation

Courtesy of M. Jacot, C. Gervais, K. Schmidt-Ott
Example 3: Neutron Imaging

Corrosion of AL

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

\[ \frac{C}{\delta_{Al}} \approx 0.53 \]
\[ \alpha \approx 0.17 \pm 0.02 \]

\[ \frac{C}{\delta_{Al}} \approx 0.18 \]
\[ \alpha \approx 0.07 \pm 0.01 \]

Example 4: Neutron Imaging

Corrosion process in brass

Joint project of Bern University of the arts, ETH Zurich, PSI, SNM
(Funded by Swiss National Science Foundation):

“Brass instruments of the 19th and early 20th centuries between long-term conservation and use in historically informed performance practice”

Neutron imaging monitoring of the corrosion in brass instruments (Neutron-CT at the start of the project and at the end)
Example 4: Neutron Imaging
Corrosion process in brass
Example 4: Neutron Imaging
Corrosion process in brass
Example 5: Neutron Imaging

Corrosion of steel in concrete

Corrosion of the steel in concrete
(collaboration between PSI and P. Chang, Y. Wang, China)
Example 5: Neutron Imaging

Corrosion of steel in concrete

Corrosion of Steel within concrete matrix

Courtesy of Peng and Wittmann

Visualised 3D-evaluation of Neutron-CT
AGENDA

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- **Summary & Discussion**
Advanced Neutron Imaging

Spatial resolution vs ‘spatial sensitivity’

Diffraction Contrast
“Bragg Edge Imaging”

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

- Polymers, microstructure, magnetic domain structures
- Cracks, pores, precipitates
- Magn. fields

Diffraction regime
- Internal strains
- Crystalline phases, textures

SANS regime
- Length scale in nm
- 0.01, 0.1, 0.3, 1.0, 3.0, 10, 100, 1000, 10000, 100000

Systems and components
- Atomic and magnetic structures
- Organic molecules
- Surfaces and multilayers
- Viruses
- Cracks and voids

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

- **Phase**
- **Texture**
- **Strain**
AGENDA

- **ESS**: The most intense neutron source for material research

- **Why Neutrons?**
  - Example: Neutron Diffraction for residual stress
  - Example: Neutron Imaging to detect hydrogen

- **Introduction: Neutron Imaging (Absorption Contrast)**
  - Examples 1-5: Corrosion in metals

- **Introduction: Diffraction Contrast in Neutron Imaging**
  - Examples 6-8: Phase transformations, Welds

- **Summary & Discussion**
Example 6: Bragg Edge Imaging

Phase Transformation: TRIP steel

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

- 44kN
- 11Nm (110Nm)
- tomography
Example 6: Bragg Edge Imaging
Phase Transformation: TRIP steel

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

- Phase transformation clearly observed!
- Tomography for further quantification
Example 6: Bragg Edge Imaging
Phase Transformation: TRIP steel

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

![Tensile and Torsion Phase Transformation](Image)

Example 6: Bragg Edge Imaging

Complementarity of methods...

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

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

Martensite within a ferrite matrix
Example 7: Diffraction Contrast

Weld 1

Example 7: Diffraction Contrast

Weld 2

Example 8: Diffraction Contrast
Dissimilar welds by ToF imaging

Example 8: Diffraction Contrast

Dissimilar welds by ToF imaging

Anton S. Tremsin et al. • Investigation of m...
Example 8: Diffraction Contrast
Dissimilar welds by ToF imaging

Spatially resolved strain maps

Example 8: Diffraction Contrast
Dissimilar welds by ToF imagingc

Resonance absorption contrast:
Elemental contrast: E.g. check diffusion processes

Example 9: Diffraction Contrast

Strain distribution in threads

• Strain Mapping in a bolt assembly, where a standard thread is compared with ‘spirallock’ thread
• The experimental data visualizes the strain redistribution inside the screw and base plate

Standard
A conventional thread is axial loaded, increasing probability of shear, especially in soft metals

Spiralock
A spiralock thread form distributes the load radially beyond the threads, significantly increasing the strength of a connection

A. Tremsin, T. Yau, W. Kockelmann, Non-destructive Examination of Loads in Regular and Self-locking Spiralock® Threads through Energy-resolved Neutron Imaging, Strain, 52 (2016)
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- **Summary & Discussion**
Neutron Imaging is useful in many scientific areas

New methods (not covered herein) rapidly evolving: Guidance from industry needed to develop methods that will benefit society

Corrosion certainly a topic that should be addressed using neutron imaging: underrepresented in recent years

We are no experts in corrosion, welding, hydrogen embrittlement etc...

We have good collaborations with existing neutron facilities worldwide!

ESS operates a testbeamline in Berlin: test measurements + feasibility studies are welcome!

Design and discuss experiments together! Now is a good time!

We should strive to take advantage of the possibilities ESS will offer, by (now) designing suitable experimental setups, sample environments, modelling tools,...

Contact us: robin.woracek@esss.se
Journey to deliver the world’s leading facility for research using neutrons

2003: European Design of ESS Completed

2009: Decision to Site ESS in Lund

2012: ESS Design Update Phase Complete

2014: Construction Starts on Green Field Site

2020: Machine Ready for 1st Beam on Target

2023: ESS Starts User Program

2025: ESS Construction Phase Complete
THANK YOU!

Courtesy of
Sample Environment

Portable loading system

- 44kN tension, 11Nm (110Nm) torsion, tomography mode
- Available for joint experiments.