Residual stress analysis with neutron diffraction

M. Hofmann\textsuperscript{1}, W.M. Gan\textsuperscript{2}, J. Rebelo Kornmeier\textsuperscript{1}

\textsuperscript{1}FRM II@TUM, \textsuperscript{2}GEMS@MLZ, HZG
outline

Why neutrons?
RS with neutron diffraction - howto
Examples
Summary
Neutron methods for materials science

Why use neutrons?

- **Neutral** → high penetration depth → large samples and/or sample environment (furnaces, cryostats, magnets, etc.) can be used

- **Scattering cross sections independent of atomic number** → detection of light elements, distinction of neighbouring elements

- **Scattering cross section depends on nucleus** → isotopes can be distinguished (i.e. H/D)

- **Neutrons have wavelength** → in the range of atomic distances and can yield information on crystal structures
Surface
angular dispersive X-ray-Methods

Energy range $\approx 5 - 17$ keV
Information depth (steel) $\approx 15 \mu m$

Intermediate Range

E.g.: ED X-ray diffraction

Energy range $\approx 60 \ldots 150$ keV
Information depth (steel) $\approx 150 \mu m$

Volume

Neutron diffraction
High energy synchrotron diffraction

Information depth (steel) $\approx 10-20$ mm

Note: New neutron methods allow to fill the gap $\approx 150 \mu m \ldots 1$ mm
Neutron centres in Europe

- With dedicated instrumentation for strain scanning.
- Most have liaison office and grant special routes for access for industry.
- MLZ (TUM, HZG, FZJ): Diffractometer STRESS-SPEC

from: www.lens-initiative.org
How to measure „stress“

Angular resolution:
< 0.01° for Δd ~ 1 × 10^{-4}

Spatial resolution:
depends on stress gradient, (~mm^3)
typically 0.01° ... 0.1°
How to measure „stress“

Scattering angle (sample) + Reference value

$2\theta$ + $2\theta_0$

$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\sin(\theta_{0,hkl})}{\sin(\theta_{hkl})} - 1$

Hooke’s Law

$\sigma_{ij} = c_{ijkl} \varepsilon_{kl}$

Isotropic case $c_{ijkl} : E, \nu$

Local stress tensor

$\lambda = 2d \sin \theta$
Gauge volume definition at a neutron diffractometer

$2\theta = 90^\circ$
How to measure „stress“ – stress tensor

\[
\sigma_{ii} = \frac{E}{1+\nu} \left( \varepsilon_{ii} + \frac{\nu}{1-2\nu} (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right)
\]

Frequently:
Main axis

\[ \Rightarrow \text{just } \varepsilon_{11}, \varepsilon_{22}, \varepsilon_{33} \]
\[ \Rightarrow \text{just 3 measured directions} \]
Stress Direction (Example: Composite casting Steel-Aluminium)

<table>
<thead>
<tr>
<th>Lattice plane</th>
<th>Steel</th>
<th>Aluminium</th>
</tr>
</thead>
<tbody>
<tr>
<td>2θ_hkl</td>
<td>~91.3°</td>
<td>~86.7°</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Direction</th>
<th>Gauge volume [mm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Axial</td>
<td>1x1x1</td>
</tr>
<tr>
<td>Radial / Tang.</td>
<td>1x10x1</td>
</tr>
<tr>
<td></td>
<td>2x2x2</td>
</tr>
<tr>
<td></td>
<td>2x10x2</td>
</tr>
</tbody>
</table>

How to measure „stress“ - directions
3 Monochromators
- Si(400)
- Ge(311)
- PG(002)

+ $2\theta_M = 35^\circ - 110^\circ$
+ high flux ($8 \times 10^7$ n/cm$^2$/s)

- Optimise:
  - Flux – resolution
  - $\lambda = 1.0 \sim 2.4$ Å
  - $2\theta = 90^\circ$ possible (cubic gauge volume!)
  - Gauge volume ($0.2 \sim 125$ mm$^3$)

+ Additional equipment:
  - Tensile rig
  - Furnaces
  - Dilatometer
SINE2020 projects @ STRESS-SPEC

Brazed Al$_2$O$_3$-ZrO$_2$ / WC plate (*)

RS in Al 7075-T6 plates
- 100 × 50 × 20 cm$^3$
- 285 kg

New ISO standard serves as best practice guide for proprietary research

Leading European neutron centres (F, UK & D) have adopted a further calibration protocol to ensure high accuracy through a Neutron Quality Label (i.e. standardized reporting and positioning routines)

R. Ramadhan et al, NIMA 2021 (in press)
outline

What do we measure?
RS with neutron diffraction - Howto
Examples
Summary
**Motivation:**
Roller straightening is the last process step in production of rails. Residual stresses (RS) introduced should be kept as low as possible (< 250 MPa at the foot, EN 13674, 2008)

**Aims:**
Determination of RS state for comparison with FEM and validation of destructive methods (Contour-Method)
**Motivation:**
Roller straightening is the last process step in production of rails. Residual stresses (RS) introduced should be kept as low as possible (< 250 MPa at the foot, EN 13674, 2008)

**Aims:**
Determination of RS state for comparison with FEM and validation of destructive methods (Contour-Method)
Residual stresses in heterogeneous materials
Residual Stresses (RS) in Metal Matrix Composites (MMC)

- high stiffness & strength
- good resistance to creep
- low thermal expansion
- better dimension stability

RS state in weld is complicated, because it
- includes macro & microscopic RS due to elastic mismatch, thermal misfit and plastic misfit;
- is tough to be measured in MMCs due to difficulties in obtaining unstrained reference parameters ($d_0$).

FSW introduces large RS due to heating + deformation

Friction Stir Welding (FSW)

Reliable experimental method to determine macro and microscopic RS in MMCs using neutron diffraction

X. X. Zhang et al., Mat. & Des. 115 (2017) 364-378
With neutron diffraction all macro & microstrain components can be determined for each phase!

FSW plate
17 vol.% SiC\(_p\)/Al

Input for multiscale FEM model for process optimisation

X. X. Zhang et al., Mat. & Des. 115 (2017) 364-378
Summary and how to get beamtime

- Neutron is a powerful tool for RS analysis, especially for large engineering components
- Most of today facilities have a dedicated strain scanner and offer beamtime
  
  1. Proprietary research (via industrial liaison office) - *usually also some test beamtime for feasibility tests*
  2. Official proposal system – i.e. selected on scientific merit (*NO costs involved here*)

https://mlz-garching.de/user-office

Thanks for your attention!