Strain and Stress Analysis by Neutron Diffraction

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Outline

• Introduction:
  • Definition and classification of residual stresses
  • Principles of diffraction stress measurements

• Use of neutrons

• Experimental aspects

• Examples

• A look to the future
**Residual Stresses:** « *self equilibrating internal stresses existing in a free body with no external forces or constraints on its boundary* » and under uniform temperature conditions

- Residual stress of the **first order** ($\sigma^I$, macro) are nearly homogeneous across large areas (several grains of the material).

- Residual stresses of the **second order** ($\sigma^{II}$, meso) are nearly homogeneous across smaller areas, of the order of some grains or phases of a material.

- Residual stresses of the **third order** ($\sigma^{III}$, micro) are inhomogeneous across submicroscopic areas of the material, several atomic distances within a grain.
Formation of Residual Stresses

Material
- E.g. multiphase materials, inclusion

Material processing
- Casting (thermal residual stresses)
- Reshaping (inhomogeneous plastic deformation)
- Cutting (working RS: grinding, honing)
- Joining (brazing, welding)
- Coating (material properties: case hardening)

Material load
- Mechanical (e.g. rolling)
- Thermal temperature fields
- Chemical H-diffusion

RS superimpose to applied stresses during service life
RS can affect the mechanical behaviour of a component
Their evaluation is fundamental
<table>
<thead>
<tr>
<th>Method</th>
<th>Penetration</th>
<th>Spatial Resolution</th>
<th>Accuracy</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hole drilling</td>
<td>1.2xhole diameter</td>
<td>50 µm depth</td>
<td>± 50 Mpa</td>
<td>Semidistructive, σІ</td>
</tr>
<tr>
<td>X-rays diffraction (atomic strain gauge)</td>
<td>&lt; 50 µm (Al); &lt; 5 µm (Ti); &lt;1mm (if layer removal)</td>
<td>1 mm laterally 20 µm depth</td>
<td>± 20 MPa, from non-linearities in sin^2ψ or surf. cond.</td>
<td>Non-destructive (surface), σІ, σІІ σІІІ</td>
</tr>
<tr>
<td>Hard X-rays (atomic strain gauge)</td>
<td>50 mm (Al)</td>
<td>20 µm lateral; 1mm parallel to beam</td>
<td>± 10 µε, from grain sampling statistics</td>
<td>Non-destructive σІ, σІІ σІІІ; 2D analysis</td>
</tr>
<tr>
<td>Neutrons (atomic strain gauge)</td>
<td>200mm (Al), 30mm (Fe) 4mm (Ti)</td>
<td>500 µm</td>
<td>± 50 µε, from count. Statis. &amp; reliability of reference</td>
<td>Non-destructive σІ, σІІ σІІІ (rather difficult) 3D-analysis</td>
</tr>
<tr>
<td>Ultrasonic (changes in elastic waves velocity)</td>
<td>&gt; 10 cm</td>
<td>5 mm</td>
<td>10%</td>
<td>Microstructure sensitive; σІ, σІІ σІІІ</td>
</tr>
<tr>
<td>Magnetic (variations in magnetic domains)</td>
<td>10 mm</td>
<td>1 mm</td>
<td>10%</td>
<td>Microstructure sensitive (for magnetic materials only), σІ, σІІ σІІІ</td>
</tr>
<tr>
<td>Raman</td>
<td>&lt; 1µm</td>
<td>&lt; 1µm approx.</td>
<td>Δλ≈0.1cm-1=50MPa</td>
<td>σІ, σІІ</td>
</tr>
</tbody>
</table>
### Neutrons vs. X-Rays

<table>
<thead>
<tr>
<th></th>
<th>Neutrons</th>
<th>X-Rays</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\mu$ (cm$^{-1}$)</td>
<td>$t_{50%}$ (mm)</td>
</tr>
<tr>
<td>Al</td>
<td>0.10</td>
<td>69.3</td>
</tr>
<tr>
<td>Ti</td>
<td>0.45</td>
<td>15.4</td>
</tr>
<tr>
<td>Fe</td>
<td>1.12</td>
<td>6.19</td>
</tr>
<tr>
<td>Ni</td>
<td>1.86</td>
<td>3.73</td>
</tr>
<tr>
<td>W</td>
<td>1.05</td>
<td>6.60</td>
</tr>
</tbody>
</table>

- $\mu$: Linear absorption coefficient
- $t_{50\%}$: Half-value thickness

**Graph:**

- Neutrons and X-rays transmission vs. penetration path (mm).
- Materials: Al, Ti, W, Fe, Ni.
- Neutrons data points:
  - $t_{50\%}$ values for different materials are shown.
- X-rays data points:
  - $t_{50\%}$ values for different materials are shown.

**Notes:**

- Neutrons and X-rays have different penetration abilities.
- Neutrons are useful for materials with low atomic number.
- X-rays are useful for materials with high atomic number.

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**Graph Details:**

- X-axis: Penetration path (mm)
- Y-axis: Transmission
- Data points for Al, Ti, W, Fe, Ni are plotted.
- Neutrons and X-rays transmission curves are shown for each material.
Neutron Diffraction Applied to Stress Analysis

- Introduced in the 80’s
- Rapid developments: it is the only technique which allows a non-destructive analysis of the stress field in bulk samples
- Neutron can deeply penetrate in most materials
- Intensity of a neutron monochromatic beam is much lower than that of X-ray tube
  - High neutron source
  - Long measuring time
  - Spatial resolution of ~ 1 mm³
Stress Analysis by Neutron Diffraction

Bragg’s Law: \( n \lambda = 2d_{hkl} \sin \theta \)

By differentiating Bragg’s Law:

\[
\frac{\Delta \lambda}{\lambda} = \frac{\Delta d_{hkl}}{d_{hkl}} + \cot \theta \Delta \theta
\]

Strain \( \varepsilon_{hkl} = \frac{d_{hkl} - d_0}{d_0} \)

Stress

Elasticity law

Stress Analysis by Neutron Diffraction

Lattice strain

Position dispersion if \( \Delta \lambda = 0 \)

Wavelength dispersion if \( \Delta \theta = 0 \)
Using diffraction to measure strain

Two cases:

\( \lambda \) is const (reactor)

\[ \varepsilon = \frac{d - d_0}{d_0} = -\frac{1}{2} \cot \theta_0 (\Delta 2\theta) \]

\( \theta \) is const, time of flight technic (spallation source)

\[ \varepsilon = \frac{d - d_0}{d_0} = \frac{\Delta \lambda}{\lambda} = \frac{\Delta t}{t} \]

Strain is given by the change in peak position from the stress-free location
Gauge volume

Detector

Incident beam

Gauge volume

Cd masks

$\theta \approx 90^\circ$
Coordinate System and Strain Relation

- $S_i, L_i$ are the sample and laboratory systems, respectively, and are related by $\phi$ and $\psi$.
- The diffracting planes are normal to $L_3$.
- In terms of the angles $\phi$ and $\psi$, $\varepsilon$ in a given direction is:

$$\varepsilon_{\phi\psi} = \varepsilon_{11}\cos^2\phi \sin^2\psi + \varepsilon_{22}\sin^2\phi\sin^2\psi + \varepsilon_{33}\cos^2\psi + \varepsilon_{12}\sin2\phi\sin^2\psi + \varepsilon_{13}\cos\phi\sin2\psi + \varepsilon_{23}\sin\phi\sin2\psi$$
Converting strain to stress

Hooke’s law

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

\[
\varepsilon_{\varphi\psi} = \frac{1 + \nu_{hkl}}{E_{hkl}} \left( \sigma_{xx} \cos^2 \varphi \sin^2 \psi + \sigma_{yy} \sin^2 \varphi \sin^2 \psi + \sigma_{zz} \cos^2 \psi + \sigma_{xy} \sin 2\varphi \sin^2 \psi \right. \\
+ \sigma_{xz} \sin 2\varphi \sin^2 \psi \sin \varphi \sin 2\psi) - \frac{\nu_{hkl}}{E_{hkl}} \left( \sigma_{xx} + \sigma_{yy} + \sigma_{zz} \right)
\]
Procedure

• General triaxial stress state: 6 unknowns.
  – Make (at least) 6 measurements \( \sigma \) Stress/Strain complete tensor

• Biaxial stress state: \( \sin^2 \psi \) method

• 1D measurements if principal directions are known

• Errors depend on:
  • Counting statistics
  • Array of angles \( \phi, \psi \)
  • Experimental errors
Four ideas

• It is strain that is actually measured.

• Residual strain changes interplanar spacings, which shifts the positions of diffraction peaks.

• Strain is resolved differently in different physical directions in the sample.

• Engineering materials are polycrystalline, so some grains are always oriented to diffract enabling stress tensors to be determined.
Gauge volume - Shape

\( \theta = 45^\circ \)

- Front surface: \( L_{\text{max}} = L_{\text{min}} = \frac{W}{\sin \theta} \)
- Back surface:

\( \theta < 45^\circ \)

- Front surface: \( L_{\text{max}} = \frac{W}{\sin \theta} \)
- Back surface: \( L_{\text{min}} = \frac{W}{\cos \theta} \)

\( 45^\circ < \theta < 90^\circ \)

- Front surface:
- Back surface:

\[ L_{\text{min}} = \frac{W}{\cos \theta} \]
\[ L_{\text{max}} = \frac{W}{\sin \theta} \]
Gage volume - issues

- **Size**
  - Small enough for spatial resolution
  - Large enough for sufficient intensity

- **Shape**
  - Confine measurement to region of interest
  - Neutron measurements around $90^\circ 2\theta$

- **Geometry and optics**
  - Can be tricky
Texture - preferred orientation

• Conversion of strain to stress:
  – Elastic constants altered
  – Can change with sample orientation

• Peak intensities and shape:
  – Intensities can vary strongly with orientation
  – Shapes can also change, affecting accuracy

• Can be checked by:
  – Measurements of intensity versus sample orientation
  – Powder pattern intensity analysis

• If possible, use experimentally determined diffraction elastic constants from same material
Stress-free standards

- Generally require peak positions for the stress-free state ($d_0$ values) to determine strains.

- **Approaches:**
  - Stress-free region
  - Stress-free piece
  - Reference powders
  - Use of equilibrium requirements:
    - Two phases
    - Force/moment balance
Characteristics of a Strain Dedicated Diffractometer

- two axis diffractometer with good instrumental resolution ($\Delta d/d \sim 10^{-3} - 10^{-4}$) combined with a good luminosity
- high/medium neutron flux
- rigid mounting and adequate space for manipulation of the samples

ENGIN-X (ISIS), SMARTS (Los Alamos), DIANE (LLB), POLDI (SINQ), D1A (50%, ILL), E3 (HMI)

- devices for the positioning and the alignment of the samples
- translation/rotation table $XYZ\omega$
- slits for the definition of the "gauge volume"
Spallation Source

\[ \varepsilon = \frac{\Delta \lambda}{\lambda} = \frac{\Delta t}{t} \]

- Good gauge volume definition
- Diffraction angle fixed, wavelength varies
- Peaks from many planes recorded simultaneously
- Useful for anisotropic systems
Steady State Reactor

- One peak analysis;
- Data analysis quite simple
- Wavelengths from about 1-2 Å (2-5 Å for cold neutrons)
- Resolution adequate, throughput varies.

\[ \epsilon = -\frac{1}{2} \cot \theta_0 (\Delta 2\theta) \]
Measurement of radial strain in the cross section of a railway wheel hoop

- Recent railway disasters are essentially caused by material failure of the wheel
- A better understanding of failure mechanism is needed

The life time of railway wheels is limited by:

- Accumulation plastic strains and formation of micro-cracks due to cyclic loading
- Manufacturing engineering and structural modifications «local cold work hardening» induces residual stresses formation
Strain Scanning

Material:
- perlitic steel
- Sector (35°) of an ICE train
- used 1.400.000 Km (life limit)

Diffractomètre G52, \( \lambda = 3A \)
- Fe-\( \alpha \) (110)
- résolution spatiale: 2x2x10 mm\(^3\)
Results

Conclusion

• Compression in the central region, tension in the regions near the wheel-rail contact regions

• Correlation between strain distribution and fracture mechanism

• the stress re-distribution during the wheel service life is fundamental.
Residual Stress Analysis in **Metal Matrix Composites**

**Diffraction methods give access to phase stresses**

- Improved combination of mechanical characteristics
- Bad ductility
- Misfit Stress between phases due to $\neq$ CTE and elastic properties

**Aim of the study**: effects of plasticity on the residual stresses of thermal origin

**Material**: Al 2124 + 17% SiC$_p$ (505°C 2h, cold water quench) : before and after bending

**Theoretical model**:

\[
\sigma_i^T = \sigma_i^M + \sigma_i^{mT} + \sigma_i^{mE}
\]

- Wearing resistance
- high temperature resistance
- Stiffness

\[
\begin{align*}
\sigma^M &= f\sigma_P^T + (1-f)\sigma_M^T \\
\sigma^{mE} &= B\sigma^M
\end{align*}
\]
Results

- Dominance of the thermal expansion misfit on the residual stresses evolution before bending

• After bending the mismatch in the centre of the bar remain unchanged, but the plasticity in the surface region has a large effect relaxing the misfit stresses
Residual Stresses and Microstrain Investigation in Fatigued Specimen

AISI 316L Compact Tension specimens

- **CT_1**: $R=0.1$, 26000 cycles $\Delta K=30.48$ MPa mm $^{1/2}$
- **CT_2**: $R=0.5$, 20000 cycles $\Delta K=30.48$ MPa mm $^{1/2}$
- **CT_3**: tension up to 5mm crack opening (10000N)
Internal Elastic Stresses by Neutron Diffraction and FE calculation

Neutrons
- 3D measurements
- Spatial resolution: 1x1x1 mm$^3$

FE calculation
- 3D
- Elastic-plastic behaviour

- In all the three specimens a 3D stress state is observed.
- CT_1: the residual stresses obtained are rather weak.
- CT_2: higher tensile stresses with a maximum at 1.5 mm from the crack tip.
- CT_3: the maximum stress level is at 2 mm from the tip. At 8 mm from the tip, $\sigma_{xx}$ and $\sigma_{yy}$ are very low, whereas $\sigma_{zz}$ becomes large and compressive.
Steep gradients at a surface

Wavelength longer, on average
Center of scattering mass is shifted in space

Specimen
Sampling volume

Detector finds peak at higher angle = apparent compressive strain
Example: Shot-peening

- Ni Superalloy
- shot-peening
- high spatial resolution: 0.3x0.3x20 mm\(^3\)

Example: Shot-peening

![Graph showing stress vs depth](image)

- Stress (MPa)
- Depth (mm)
- Σy corrected
- Σz corrected
- Σy exp
- Σz exp

- 50 mm
- 5 mm
Residual Stresses in Notches Resulting from Impacts

**Aim of the study**: determine the effect of a high-energy projectile impact on the fatigue life of the target material.

To validate the elasto-plastic Finite Element calculation, **neutron diffraction** has been used to determine the evolution of the residual stresses under the notch.

(spatial resolution: 1x1x1mm³)

Disagreement under the impact notch, due to the adiabatic heating of the steel and the formation of adiabatic shear bands made by very high energy impact;

residual stress relaxation

Impact of projectile with a speed of 100km/h

σ (MPa)

Z (mm)

σ_{rr} (calc)

σ_{zz} (calc)

σ_{zz} (exp)

σ_{rr} (exp)

XC38 STEEL

TRACTION

COMPRESSION
Recommandations

• **Experience is really feasible?**
  - Path length, spatial resolution, number of samples ....

• **Define an efficient strategy:**
  - How large are the stress gradients? ® Dimensions of the gauge volume
  - Are $D$ vs $\sin^2\psi$ linear (or approx.)? ® Yes measurements in the 3 principal directions
  - What is the ratio of macro- to microstress? ® Use of single reflection line or several
  - Define meaningful region of the sample to be investigated
  - Choose of an adequate \{hkl\} reflection

• **Reference sample for determining $d_0$**
A look to the future

- Triaxial measurements
- Anisotropic materials
- Small strain systems (e.g. ceramics)
- Real time (parametric) studies
- Small gage volumes (e.g. gradients, buried interfaces)
- Higher temperature and stress
- High Z materials

High flux, high resolution

3rd high flux neutron sources: SNS, JNS, ESS
# Neutrons vs. Synchrotron Radiation

<table>
<thead>
<tr>
<th></th>
<th>Neutrons</th>
<th>Synchrotron Radiation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Penetration</strong></td>
<td>10-20 mm</td>
<td>Qq. mm</td>
</tr>
<tr>
<td></td>
<td>40-60 mm</td>
<td>10-20 mm</td>
</tr>
<tr>
<td><strong>Spatial resolution</strong></td>
<td>1mm³</td>
<td>µm in one direction, mm in the other (0.1mm³)</td>
</tr>
<tr>
<td><strong>Strain maps</strong></td>
<td>3D</td>
<td>2D</td>
</tr>
<tr>
<td><strong>Stress complete tensor</strong></td>
<td>Yes</td>
<td>With problems</td>
</tr>
<tr>
<td><strong>Coarse grain probleme</strong></td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Industrial real component study</strong></td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td><strong>Counting time</strong></td>
<td>½ h – 1h</td>
<td>Minutes-seconds</td>
</tr>
</tbody>
</table>