Residual Stress Evaluation using 2D Area Detector

Stress Analysis Workshop

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Hardware Setup for XRD² Stress Measurement

X-ray Optics used:
- Göbel Mirror
- Capillary
- Collimators
- Soller slits
- Monochromators
Advantages of XRD$^2$ Setup

- Short measurement time
- High accuracy
- Recognition of texture if presents
Measured Area by 0D (point), 1D (line) and 2D (area) Detectors

0D detector
- Small spot measured
- Scans necessary
- Long measuring time

1D detector
- Long 2\(\Theta\) range measured
- Medium measuring time

2D detector
- Long 2\(\Theta\) and \(\gamma\) ranges measured
- Short measuring time
How does Stress show up?

Due to Residual Stresses Debye Ring experiences:
- Distortion
- Shift
- Change of Shape

![Diagram showing Debye Ring and stress effects](image-url)
### Stress Models

**(principal coordinates)**

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<td>( \begin{pmatrix} \sigma &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \end{pmatrix} )</td>
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<td>( \begin{pmatrix} \sigma &amp; 0 &amp; 0 \ 0 &amp; \sigma &amp; 0 \ 0 &amp; 0 &amp; 0 \end{pmatrix} )</td>
<td>( \begin{pmatrix} \sigma_I &amp; 0 &amp; 0 \ 0 &amp; \sigma_{II} &amp; 0 \ 0 &amp; 0 &amp; \sigma_{III} \end{pmatrix} )</td>
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Debye Ring Distortion: Uniaxial Stress

Fe(211) Cr

\( S_1 = -1.271 \times 10^{-3} \text{ GPa}^{-1} \)

\( \frac{1}{2} S_2 = 5.811 \times 10^{-3} \text{ GPa}^{-1} \)

\[
\sigma (\text{GPa}) = \begin{pmatrix} 1 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix}
\]
Debye Ring Distortion: Biaxial Stress

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\[ S_1 = -1.271 \times 10^{-3} \text{ GPa}^{-1} \]

\[ \frac{1}{2} S_2 = 5.811 \times 10^{-3} \text{ GPa}^{-1} \]

\[ \sigma(\text{GPa}) = \begin{pmatrix} 1 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & 0 \end{pmatrix} \]
Debye Ring Distortion: Biaxial and Shear Stress

Fe(211) Cr

\[ S_1 = -1.271 \times 10^{-3} \text{ GPa}^{-1} \]

\[ \frac{1}{2} S_2 = 5.811 \times 10^{-3} \text{ GPa}^{-1} \]

\[ \sigma(\text{GPa}) = \begin{pmatrix} 1 & 0 & 0.5 \\ 0 & -1 & 0 \\ 0.5 & 0 & 0 \end{pmatrix} \]
Diffraction, Detector, Sample spaces and Laboratory coordinate system

- **Diffraction Space**
  - $(2\theta, \gamma)$
  - Crystal structure & wavelength

- **Laboratory Reference Frame**
  - $(X_L, Y_L, Z_L)$

- **Detector Space**
  - $(D, \alpha)$
  - Pixel resolution & angular coverage

- **Sample Space**
  - $(\Omega, \Psi, \Phi, X, Y, Z)$
  - Sample orientation & location
Diffraction Space \((2\theta, \gamma)\)

Diffraction space is used for covariant formulation of stress & strain tensors via Bragg X-ray Scattering formalism;

\(2\theta\) – Bragg angle, \(\gamma\) – polar angle

**Diffraction vector**

\[
\mathbf{H}^L = \begin{pmatrix}
-sin\theta \\
-cos\theta sin\gamma \\
-cos\theta cos\gamma
\end{pmatrix}
\]
Detector Space (D, $\alpha$)

Detector space refers to detector physical position;
D – distance from sample to detector
$\alpha$ – swing angle of the detector center
Sample Space \((\Omega, \Phi, \Psi, X, Y, Z)\)

Sample space is connected to Goniometer axes and refers to all possible Sample rotations and movement.

\[
R(\Omega, \Psi, \Phi) = R_3(-(180^\circ + \Phi)) R_1(\Psi - 90^\circ) R_3(\Omega)
\]

\(R_i\) – right-handed rotation matrix
Transformation between Sample and Laboratory Systems

Explicit form of transformation matrix

\[
R(\Omega, \Psi, \Phi) = \begin{pmatrix}
-\cos \Omega \cos \Phi - \sin \Omega \sin \Psi \sin \Phi & -\sin \Omega \cos \Phi + \cos \Omega \sin \Psi \sin \Phi & -\cos \Psi \sin \Phi \\
-\cos \Omega \sin \Phi + \sin \Omega \sin \Psi \cos \Phi & -\sin \Omega \sin \Phi - \cos \Omega \sin \Psi \cos \Phi & \cos \Psi \cos \Phi \\
-\sin \Omega \cos \Psi & \cos \Omega \cos \Psi & \sin \Psi
\end{pmatrix}
\]

Relation of Diffraction vector in Laboratory and Sample coordinates

\[
H^S = R(\Omega, \Psi, \Phi)H^L = R(\Omega, \Psi, \Phi) \begin{pmatrix}
-\sin \theta \\
-\cos \theta \sin \gamma \\
-\cos \theta \cos \gamma
\end{pmatrix}
\]

Components of Diffraction vector in Sample Space

\[
h_1^S = a \cos \Phi - b \cos \Psi \sin \Phi + c \sin \Psi \sin \Phi \\
h_2^S = a \sin \Phi + b \cos \Psi \cos \Phi - c \sin \Psi \cos \Phi \\
h_3^S = b \sin \Psi + c \cos \Psi
\]

P.S. \( \Omega, \Psi, \Phi \)- goniometer angles

a = \sin \theta \cos \Omega + \sin \gamma \cos \theta \sin \Omega \\
b = -\cos \gamma \cos \theta \\
c = \sin \theta \sin \Omega - \sin \gamma \cos \theta \cos \Omega
Measured Strains and Strain Tensor (in Laboratory and Sample coordinates)

\[ \mathbf{H}_{hkl} = (h_1^L, h_2^L, h_3^L) \equiv (h_1^S, h_2^S, h_3^S) \]

\[ \{\varepsilon_{meas}^L\}^{hkl} = \frac{d_{meas}^{hkl}}{d_0^{hkl}} - 1 = \frac{\lambda}{2d_0^{hkl} \sin(\theta_{hkl})} - 1 \]

\[ \varepsilon_{ij}^L, \varepsilon_{ij}^S \] – strain tensor components in the Lab and Sample reference frames, respectively

\[ \{\varepsilon_{meas}^L\}^{hkl} = h_i^L < \varepsilon_{ij}^L > h_j^L \equiv h_i^S < \varepsilon_{ij}^S > h_j^S \]

Equation for Strain Measurement with Area Detector:

\[ \{\varepsilon_{meas}^L\}^{hkl} = h_1^S h_1^S < \varepsilon_{11}^S > + 2h_1^S h_2^S < \varepsilon_{12}^S > + h_2^S h_2^S < \varepsilon_{22}^S > + 2h_1^S h_3^S < \varepsilon_{13}^S > + 2h_2^S h_3^S < \varepsilon_{23}^S > + h_3^S h_3^S < \varepsilon_{33}^S > \]

To determine \( \varepsilon_{ij} \) we have to perform at least 6 independent measurements !!
Explicit form of Equation for Strain Tensor Components

\[
\begin{pmatrix}
  h_1^S \\
  h_2^S \\
  h_3^S 
\end{pmatrix} =
\begin{pmatrix}
  -\cos \Omega \cos \Phi - \sin \Omega \sin \Psi \sin \Phi & -\sin \Omega \cos \Phi + \cos \Omega \sin \Psi \sin \Phi & -\cos \Psi \sin \Phi \\
  -\cos \Omega \sin \Phi + \sin \Omega \sin \Psi \cos \Phi & -\sin \Omega \sin \Phi - \cos \Omega \sin \Psi \cos \Phi & \cos \Psi \cos \Phi \\
  -\sin \Omega \cos \Psi & \cos \Omega \cos \Psi & \sin \Psi 
\end{pmatrix}
\begin{pmatrix}
  -\sin \theta \\
  -\cos \theta \sin \gamma \\
  -\cos \theta \cos \gamma 
\end{pmatrix}
\]

\[
\{ \varepsilon_{meas}^L \}^{hkl} = h_1^S h_1^S < \varepsilon_{11}^S > + 2 h_1^S h_2^S < \varepsilon_{12}^S > + h_2^S h_2^S < \varepsilon_{22}^S > + 2 h_1^S h_3^S < \varepsilon_{13}^S > + 2 h_2^S h_3^S < \varepsilon_{23}^S > + h_3^S h_3^S < \varepsilon_{33}^S >
\]
Lattice constant is not “constant” for different polycrystalline samples

\[
\{\varepsilon_{\text{meas}}^L\}_{hkl} = \frac{d_{\text{meas}}^{hkl}}{d_0^{hkl}} \pm \Delta d_0^{hkl} - 1 = \left(\frac{d_{\text{meas}}^{hkl}}{d_0^{hkl}} - 1\right) \pm \frac{\Delta d_0^{hkl}}{d_0^{hkl}} = \{\varepsilon_{\text{true}}^L\}_{hkl} + \varepsilon_{ph}^{hkl}
\]

Real situation!!!

As a result we obtain

\[
\begin{pmatrix}
\varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\
\varepsilon_{12} & \varepsilon_{22} & \varepsilon_{23} \\
\varepsilon_{13} & \varepsilon_{23} & \varepsilon_{33}
\end{pmatrix}
+ \begin{pmatrix}
\varepsilon_{ph} & 0 & 0 \\
0 & \varepsilon_{ph} & 0 \\
0 & 0 & \varepsilon_{ph}
\end{pmatrix}
\Rightarrow
\begin{pmatrix}
\varepsilon_{11} + \varepsilon_{ph} & \varepsilon_{12} & \varepsilon_{13} \\
\varepsilon_{12} & \varepsilon_{22} + \varepsilon_{ph} & \varepsilon_{23} \\
\varepsilon_{13} & \varepsilon_{23} & \varepsilon_{33} + \varepsilon_{ph}
\end{pmatrix}
\]

and the strain equation becomes

\[
\{\varepsilon_{\text{meas}}^L\}_{hkl} = h_1^S h_1^S \varepsilon_{11} + h_1^S h_2^S \varepsilon_{12} + h_2^S h_2^S \varepsilon_{22} + h_1^S h_3^S \varepsilon_{13} + h_2^S h_3^S \varepsilon_{23} + h_3^S h_3^S \varepsilon_{33} + \varepsilon_{ph}
\]
For a polycrystal composed of elastically isotropic crystallites, Hooke’s law reads

$$\langle \varepsilon_{ij}^S \rangle = S_{ijkl}^S \langle \sigma_{kl}^S \rangle = S_{ijkl}^C \langle \sigma_{kl}^S \rangle = \left[ s_1 \delta_{ij} \delta_{kl} + \frac{1}{2} s_2 \frac{\delta_{ik} \delta_{jl} + \delta_{il} \delta_{jk}}{2} \right] \langle \sigma_{kl}^S \rangle = s_1 \langle \sigma_{kk}^S \rangle \delta_{ij} + \frac{1}{2} s_2 \langle \sigma_{ij}^S \rangle$$

where $S_{ijkl}^S$ is a compliance tensor of the sample and $S_{ijkl}^C$ of the individual crystallites. Two independent Elastic Constants related to Young’s modulus $E$ and Poison’s ratio $\nu$ of the sample:

$$s_1 = -\frac{\nu}{E}, \quad \frac{1}{2} s_2 = \frac{1+\nu}{E}$$

$\langle \rangle$ denotes an average for all crystallites.

$$\{ \varepsilon_{meas}^L \}^{hkl} = h_i^S \langle \varepsilon_{ij}^S \rangle h_j^S = h_i^S \left[ s_1 \langle \sigma_{kk}^S \rangle \delta_{ij} + \frac{1}{2} s_2 \langle \sigma_{ij}^S \rangle \right] h_j^S = s_1 \langle \sigma_{kk}^S \rangle + \frac{1}{2} s_2 h_i^S \langle \sigma_{ij}^S \rangle h_j^S$$

Equation for stress evaluation

$$\{ \varepsilon_{meas}^L \}^{hkl} = \left( s_1 + \frac{1}{2} s_2 h_1^S h_1^S \right) \langle \sigma_{11}^S \rangle + 2 \left( \frac{1}{2} s_2 h_1^S h_2^S \right) \langle \sigma_{12}^S \rangle + \left( s_1 + \frac{1}{2} s_2 h_2^S h_2^S \right) \langle \sigma_{22}^S \rangle$$

$$+ 2 \left( \frac{1}{2} s_2 h_1^S h_3^S \right) \langle \sigma_{13}^S \rangle + 2 \left( \frac{1}{2} s_2 h_2^S h_3^S \right) \langle \sigma_{23}^S \rangle + \left( s_1 + \frac{1}{2} s_2 h_3^S h_3^S \right) \langle \sigma_{33}^S \rangle$$
Stress Tensor in Principal Coordinates

Stress Tensor can be transformed to universal principal coordinate system with no shear components (non-diagonal terms)

\[
\begin{pmatrix}
\sigma_{11} & \sigma_{12} & \sigma_{13} \\
\sigma_{21} & \sigma_{22} & \sigma_{23} \\
\sigma_{31} & \sigma_{32} & \sigma_{33}
\end{pmatrix}
\]

\[
\begin{pmatrix}
\sigma_1 & 0 & 0 \\
0 & \sigma_{II} & 0 \\
0 & 0 & \sigma_{III}
\end{pmatrix}
\]

Stress Tensor in arbitrary coordinate system

\[
\sigma_{ij} = \sigma_{ji}
\]

Stress Tensor in principle system is described by values \(\sigma_1, \sigma_{II}, \sigma_{III}\)

The values of the stress tensor’ components depend on the coordinate system chosen. Usually, the selected coordinate system is related to the sample’s geometry, e.g. edges or borders of specimen.
From the measurements and the following data reduction we get 6 stress tensor components; three of them have the same additive pseudo-hydrostatic error:

\[ \sigma_{11}^S + \sigma_{ph}^{hkl}, \quad \sigma_{22}^S + \sigma_{ph}^{hkl}, \quad \sigma_{33}^S + \sigma_{ph}^{hkl}, \quad \sigma_{12}^S, \quad \sigma_{13}^S, \quad \sigma_{23}^S \]

Measuring strains in the sample we obtained only 6 independent values. Possible sextet is shown on the surface of the stress ellipsoid Lame. All the other experimental points only improve the statistics.

Without knowledge of the exact \( d_0^{hkl} \) value it is impossible to calculate simultaneously \( \sigma_{11}, \sigma_{22}, \sigma_{33} \) components of the stress tensor without additive errors.

In stress analysis of polycrystals we can assume \( \sigma_{33}=0 \). The most complicated stress state that can be evaluated with the pseudo-hydrostatic distortion is the **Biaxial + Shear**:

\[
\begin{pmatrix}
\sigma_{11}^S + \sigma_{ph}^{hkl} & \sigma_{12}^S & \sigma_{13}^S \\
\sigma_{12}^S & \sigma_{22}^S + \sigma_{ph}^{hkl} & \sigma_{23}^S \\
\sigma_{13}^S & \sigma_{23}^S & \sigma_{ph}^{hkl}
\end{pmatrix}
\]
Uniaxial Stress

The strain measurement equation reduced to

\begin{align*}
\varepsilon^S &= \begin{pmatrix}
\varepsilon^S & 0 & 0 \\
0 & 0 & 0 \\
0 & 0 & 0
\end{pmatrix} \\
\sigma^S &= \begin{pmatrix}
\sigma^S & 0 & 0 \\
0 & 0 & 0 \\
0 & 0 & 0
\end{pmatrix}
\end{align*}

\[ \varepsilon^S \equiv \frac{1}{E} \sigma^S \]

V -

The strain measurement equation becomes

\[ \{ \varepsilon^L_{\text{meas}} \}^{hkl} = \left( h_1^S h_1^S - v h_2^S h_2^S - v h_3^S h_3^S \right) \langle \varepsilon^S \rangle = \left[ -v + (1 + v) h_1^S h_1^S \right] \langle \varepsilon^S \rangle \]

and the stress equation becomes

\[ \{ \varepsilon^L_{\text{meas}} \}^{hkl} = \left( S_1 + \frac{1}{2} S_2 h_1^S h_1^S \right) \langle \sigma^S \rangle \]
Biaxial Stress

In the Biaxial Stress case

\[
\sigma^S = \begin{pmatrix}
\sigma_{11}^S & \sigma_{12}^S & 0 \\
\sigma_{12}^S & \sigma_{22}^S & 0 \\
0 & 0 & 0
\end{pmatrix}
\quad \varepsilon^S = \begin{pmatrix}
\varepsilon_{11}^S & \varepsilon_{12}^S & 0 \\
\varepsilon_{12}^S & \varepsilon_{22}^S & 0 \\
0 & 0 & -\frac{\nu}{1-\nu}(\varepsilon_{11}^S + \varepsilon_{22}^S)
\end{pmatrix}
\]

The strain measurement equation reduced to

\[
\{\varepsilon_{meas}^L\}^{hkl} = \left(h_1^S h_1^S - \frac{\nu}{1-\nu} h_3^S h_3^S\right)\langle \varepsilon_{11}^S \rangle + 2h_1^S h_2^S \langle \varepsilon_{12}^S \rangle + \left(h_2^S h_2^S - \frac{\nu}{1-\nu} h_3^S h_3^S\right)\langle \varepsilon_{22}^S \rangle
\]

and the stress equation becomes

\[
\{\sigma_{meas}^L\}^{hkl} = \left(S_1 + \frac{1}{2} S_2 h_1^S h_1^S\right)\langle \sigma_{11}^S \rangle + 2\left(\frac{1}{2} S_2 h_1^S h_2^S\right)\langle \sigma_{12}^S \rangle + \left(S_1 + \frac{1}{2} S_2 h_2^S h_2^S\right)\langle \sigma_{22}^S \rangle
\]
Rotationally Symmetric Biaxial Stress

The strain measurement equation reduced to

\[
\{\varepsilon_{meas}^L\}_{hkl} = \left( h_1^S h_1^S + h_2^S h_2^S - \frac{2\nu}{1-\nu} h_3^S h_3^S \right) \langle \varepsilon^S \rangle = \left( 1 - \frac{1+\nu}{1-\nu} h_3^S h_3^S \right) \langle \varepsilon^S \rangle
\]

and the stress equation becomes

\[
\{\varepsilon_{meas}^L\}_{hkl} = \left[ 2S_1 + \frac{1}{2} S_2 \left( 1 - h_3^S h_3^S \right) \right] \langle \sigma^S \rangle
\]
The strain–stress relation between a diffracting crystallite and the surrounding matrix depends on a large set of structural features like shape and dimensions, the interface structure between grain and matrix, their different elastic constants, etc. In most of the models (Voigt, Reuss, Neerfeld-Hill, Kröner, etc.) it is assumed that the elastomechanical properties of the matrix are isotropic. The elastic anisotropy is then related only to the crystalline nature of grains contributing to the measured Bragg reflection.

Even if the individual crystallites of a polycrystal are elastically anisotropic, the whole body can still be microscopically elastically isotropic (quasi-isotropic). This is the case if crystallographic texture is not observed and if the grain interaction is isotropic.

**X-ray Elastic Constants**

\[
\begin{align*}
\varepsilon_{\text{meas}}^{L}^{hkl} &= \left( s_{1}^{hkl} + \frac{1}{2} s_{2}^{hkl} h_{1}^{S} h_{1}^{S} \right) \langle \sigma_{11}^{S} \rangle + 2 \left( \frac{1}{2} s_{2}^{hkl} h_{1}^{S} h_{2}^{S} \right) \langle \sigma_{12}^{S} \rangle + \left( s_{1}^{hkl} + \frac{1}{2} s_{2}^{hkl} h_{2}^{S} h_{2}^{S} \right) \langle \sigma_{22}^{S} \rangle \\
&+ 2 \left( \frac{1}{2} s_{2}^{hkl} h_{1}^{S} h_{3}^{S} \right) \langle \sigma_{13}^{S} \rangle + 2 \left( \frac{1}{2} s_{2}^{hkl} h_{2}^{S} h_{3}^{S} \right) \langle \sigma_{23}^{S} \rangle + \left( s_{1}^{hkl} + \frac{1}{2} s_{2}^{hkl} h_{3}^{S} h_{3}^{S} \right) \langle \sigma_{33}^{S} \rangle
\end{align*}
\]
**XRD\(^2\) Stress Evaluation Equation: Reduction to Conventional XRD\(^1\)**

\[ \gamma = 270^\circ \]

\[
\begin{align*}
    h_1^s &= \sin(\theta - \Omega) \cos \Phi + \cos(\theta - \Omega) \sin \Psi \sin \Phi \\
    h_2^s &= \sin(\theta - \Omega) \sin \Phi - \cos(\theta - \Omega) \sin \Psi \cos \Phi \\
    h_3^s &= \cos(\theta - \Omega) \cos \Psi
\end{align*}
\]

**Iso mode:**
\[
\begin{align*}
    \varphi &= \Phi, \\
    \psi &= \theta - \Omega, \quad \Psi = 0
\end{align*}
\]

**Side Inclination mode:**
\[
\begin{align*}
    \varphi &= \Phi - 90^\circ, \\
    \theta &= \Omega, \quad \psi = \Psi
\end{align*}
\]

\[ \psi, \varphi \text{ - diffraction vector angles} \]

**Method \(\sin^2\psi\)**

\[
\{\varepsilon^L_{meas}\}_{hkl}^{\psi} = \left[ <\varepsilon_{11}^s> \cos^2 \varphi + <\varepsilon_{12}^s> \sin(2\varphi) + <\varepsilon_{22}^s> \sin^2 \varphi \right] \sin^2 \psi
\]

\[
+ \left[ <\varepsilon_{13}^s> \cos \varphi + <\varepsilon_{23}^s> \sin \varphi \right] \sin(2\psi) + <\varepsilon_{33}^s> \cos^2 \psi
\]
How is Stress measured with 2D detector?
Measured Frames
(Segments of Debye Ring @ different $\Omega$)
Data Reduction and Analysis

- X-ray Intensity Integration

- Physical Corrections
  - Absorption
  - Background
  - Lorentz & Polarization
  - $K_{\alpha_2}$

- Bragg Peak Evaluation/Search
  - Gravity, Sliding Gravity, Pearson, Parabola, etc

- Stress/Strain Calculation
The measured Debye ring is divided into segments, where the integration over $\gamma$ is performed. The result is a set of $2\theta$ peaks @ different $\gamma$ values.
Data Reduction: Absorption Correction

\[ f_{\text{abs}}(\alpha) = \frac{\sin(2\theta - \alpha)}{\sin \alpha + \sin(2\theta - \alpha)} = \frac{1 + \cot \theta \tan(\theta - \alpha)}{2} \]

\[ l_{\text{det}} \sim f_{\text{abs}}(\alpha) \times l_0 \]

\[ \cos \alpha = \frac{n_{\text{inc}} \times n_{\text{diff.pl.}}}{\sin \delta} = \left| -\frac{\cos \Omega \sin \gamma \cos \Psi + \cos \gamma \sin \Psi}{\sin \delta} \right| \]

\[ \cos \delta = \cos \Omega \cos \gamma \cos \Psi - \sin \gamma \sin \Psi \]
Data Reduction: Lorentz and Polarization Corrections

\[ LP(\theta) = \frac{1 + \cos^2 2\theta}{\sin^2 \theta} \]

\[ LP_M(\theta) = \frac{1 + \cos^2 2\theta_M \cos^2 2\theta}{\sin^2 \theta} \]

In the presence of a monochromator:
Peak Position Determination

The determination of the peak position is performed on the basis of the intensity distribution being corrected previously. There are different methods of peak position determinations. Some of them are:

- **Center of Gravity**
- **Sliding Gravity**
- **Profile Fitting** (*Pseudo-Voigt, Pearson VII functions*)
- **Parabolic** (upper part of the profile fitting)

**Center of Gravity**

\[
 d_{CG} = \frac{\int_{d_1}^{d_N} xI(x)dx}{\int_{d_1}^{d_N} I(x)dx}
\]

**Sliding Gravity**

\[
 d_{SG} = \sum_{i=1}^{6} \omega_i \, d_i
\]

\( \omega_i \) – weighting factors
Strategy of 2D Stress Measurement

Measurement strategy for $\sigma_{11}$ Stress Evaluation

Measurement strategy for complete Stress Tensor Evaluation (6 independent measurements by dashed lines)

Stress tensor is then calculated from the condition:

$$\chi^2 = \sum_i \left[ \varepsilon_i^{meas}(hkl; (\Omega, \Psi, \Phi)_i) - \varepsilon_i^{calc}(\sigma^S; hkl; (\Omega, \Psi, \Phi)_i) \right]^2 = \min$$
Example:
Biaxial Stress from Fe(211) @ Cr-Kα

Measurements under 21 goniometer positions:

- $\Omega = 102^\circ$, $\Phi = 0^\circ$, $\Psi = 0^\circ, 15^\circ, 30^\circ, 45^\circ, 60^\circ$;
- $\Omega = 102^\circ$, $\Phi = 45^\circ$, $\Psi = 15^\circ, 30^\circ, 45^\circ, 60^\circ$;
- $\Omega = 102^\circ$, $\Phi = 90^\circ$, $\Psi = 15^\circ, 30^\circ, 45^\circ, 60^\circ$;
- $\Omega = 102^\circ$, $\Phi = 135^\circ$, $\Psi = 15^\circ, 30^\circ, 45^\circ, 60^\circ$;
- $\Omega = 102^\circ$, $\Phi = 180^\circ$, $\Psi = 15^\circ, 30^\circ, 45^\circ, 60^\circ$.

$2\theta_0 = 156.084^\circ$

$S_1 = -1.271 \cdot 10^{-6} \text{ MPa}^{-1}$

$\frac{1}{2}S_2 = 5.811 \cdot 10^{-6} \text{ MPa}^{-1}$

$\sigma_{11} = -737.3 \pm 3.8 \text{ MPa}$

$\sigma_{22} = -706.5 \pm 3.3 \text{ MPa}$

$\sigma_{12} = \sigma_{21} = -6.4 \text{ MPa}$

Corrections applied:
- Absorption
- Background
- Polarization
- Kα$_2$ elimination
- Smooth
Precision of Debye Ring Distortion
Technique: Si(533) Powder @ Cu-Kα

Sample: NIST640c (assuming Biaxial stress model)
\[ \theta_0 = 136.908°, \quad S_1 = -1.322 \times 10^{-6} \text{ MPa}^{-1}, \quad \frac{1}{2}S_2 = 7.303 \times 10^{-6} \text{ MPa}^{-1} \]

**XRD² Technique**

Measurements under 21 goniometer positions:
- \( \Omega = 112°, \Phi = 0°, \Psi = 0°, 15°, 30°, 45°, 60° \)
- \( \Omega = 112°, \Phi = 45°, \Psi = 15°, 30°, 45°, 60° \)
- \( \Omega = 112°, \Phi = 90°, \Psi = 15°, 30°, 45°, 60° \)
- \( \Omega = 112°, \Phi = 135°, \Psi = 15°, 30°, 45°, 60° \)
- \( \Omega = 112°, \Phi = 180°, \Psi = 15°, 30°, 45°, 60° \)

**σ**₁₁ = 0.7 ± 2.2 MPa
**σ**₂₂ = -1.0 ± 1.9 MPa
**σ**₁₂ = **σ**₂₁ = -0.1 MPa

**XRD¹ Technique (sin²ψ)**

\[ \sigma_{\text{nom}} (\psi=0°) = 1.1\pm1.4 \text{ MPa} \]
\[ \sigma_{\text{nom}} (\psi=90°) = 16\pm8 \text{ MPa} \]

\[ \sigma_{11} = -4.0 \pm 6.5 \text{ MPa} \]
\[ \sigma_{22} = -11.6 \pm 5.7 \text{ MPa} \]
\[ \sigma_{12} = \sigma_{21} = -1.2 \text{ MPa} \]
Accuracy of XRD$^2$ Technique: Carbon Steel (211) @ Cr-Ka

Totally 7 frames were measured @ $\Omega = 33, 48, 63, 78, 93, 108, 123^\circ$

<table>
<thead>
<tr>
<th>Conventional $\sin^2\psi$</th>
<th>XRD$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>-776 ±62 MPa</td>
<td>3 points</td>
</tr>
<tr>
<td></td>
<td>-769 ±38</td>
</tr>
</tbody>
</table>
Ellipsoid Lame

\[ \sigma_{II} \]

\[ \sigma_{I} \]

\[ \sigma_{III} \]

Stress vector
Advanced XRD$^2$ Techniques: Stress Gradients

Sample:
Wolfram Standard Probe, stress free

Single Frame:
W(310)+W(222)@Co

$\Omega=101.77^\circ$; $\Psi=60^\circ$; $\Phi=0^\circ$;
$2\theta_{\text{det}}=217^\circ$
Advanced XRD² Techniques: Stress Gradients

\[ \sigma_{22} = 9.2 \pm 52.4 \text{ MPa} \]

Different incidence angles – different penetration depths \(\rightarrow\) stress profile
Advanced XRD\(^2\) Techniques:
Multiple HKL Reflections

**Sample:**
NIST 640c, Si powder

21 Frames:

\[ \Omega = 112^\circ; \Psi = 0^\circ; \Phi = 0^\circ; \]
\[ \Omega = 112^\circ; \Psi = 15^\circ; \Phi = 0^\circ; \]
\[ \Omega = 112^\circ; \Psi = 15^\circ; \Phi = 45^\circ; \]
\[ \Omega = 112^\circ; \Psi = 15^\circ; \Phi = 90^\circ; \]
\[ \Omega = 112^\circ; \Psi = 15^\circ; \Phi = 135^\circ; \]
\[ \Omega = 112^\circ; \Psi = 15^\circ; \Phi = 180^\circ; \]
\[ \Omega = 112^\circ; \Psi = 30^\circ; \Phi = 0^\circ; \]
\[ \Omega = 112^\circ; \Psi = 30^\circ; \Phi = 45^\circ; \]
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\[ \Omega = 112^\circ; \Psi = 60^\circ; \Phi = 135^\circ; \]
\[ \Omega = 112^\circ; \Psi = 60^\circ; \Phi = 180^\circ; \]

Frame: Si(620)+Si(533)@Cu
Advanced XRD\(^2\) Techniques: Multiple HKL Reflections

<table>
<thead>
<tr>
<th></th>
<th>Si(620)</th>
<th>Si(533)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biaxial</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(\sigma_{11}) (MPa)</td>
<td>-0.9±2.7</td>
<td>-0.6±2.2</td>
</tr>
<tr>
<td>(\sigma_{22}) (MPa)</td>
<td>-1.3±2.4</td>
<td>-1.3±1.9</td>
</tr>
<tr>
<td>Single(620)</td>
<td>1.1E2</td>
<td>8E1</td>
</tr>
<tr>
<td>Single(533)</td>
<td>8E1</td>
<td>1.1E2</td>
</tr>
<tr>
<td>Multiple(620)+(533)</td>
<td>1.1E2</td>
<td>1.1E2</td>
</tr>
</tbody>
</table>
Stress in Textured Samples

Presence of texture influences the evaluation of residual stresses due to intensity variation along the Debye ring.
Modern Area Detector Implementation

- 0D mapping
- 1D mapping
- 2D:
  - semiconductor
  - image plate
  - multiwire
  - CCD

HiStar (MW)
Vantec 2000 (µGap)
APEX II (CCD)

Saturn 944 (CCD)

R-Axis IV (IP)

Mar (IP)

PIŁATUS100K

The PILATUS 100K detector system is a versatile instrument which is very easy to handle and can be used for demanding X-ray applications.

It is based on technology that is used in all DECTRIS high-resolution X-ray detectors. PILATUS 100K thus provides a highly integrated solution that is therefore ready to use live in your lab. PILATUS 100K offers unsurpassed benefits in terms of detection efficiency and analysis speed.
References


