LAUE MICRODIFFRACTION

French CRG beamline BM32 “InterFaces” @ ESRF
Jean-Sébastien Micha
CNRS UMR SyMMES
micha@esrf.fr

HERCULES Tutorials March 2020
X-ray Laue diffraction microscope for structural imaging
on CRG-IF beamline at ESRF, Grenoble, France

- Polycrystalline Microstructure
- < 500 nm X-ray probe
- Large field of view
- In situ experiments
- Single object internal structure

- Crystal orientation & full strain with high $10^4$ resolution
- 3D Localisation and measurements defects & high stress level

µLaue scanner
Materials x-ray based characterization techniques

- Multi-scale approach
- Multi-modal analysis

- Atom
  - Fine chemical analysis
  - Composition
  - Physico-chemical properties

- Crystal
  - Microstructure
  - Residual strain
  - Phase recognition

- X-ray Diffraction
- X-ray Spectroscopies
- X-ray Scattering

- LAUE MICRODIFFRACTION
- 2D/3D X-ray Imaging

- Final object
  - Failure analysis
  - Compliance
  - Reverse engineering

- Coherence → resolution and contrast
- Hard X-rays → penetration (in-situ, operando)

Courtesy J. Susini
Microdiffraction Laue (μLaue)

Laue Diffraction Microscopy

Beamsize: 0.3 (H) x 0.3 (V) μm²
Energy band: 5 – 23 keV, White & Monochromatic

Visible light
X-ray fluorescence
X-ray Laue pattern
Laue Diffraction Microscopy

Beamsize: 0.3 (H) x 0.3 (V) μm²
Energy band: 5 – 23 keV, White & Monochromatic

Routine
2D mapping
Orientation/Strain/phase
Crystalline defects

Mono- & poly-crystals
Single micro-object

In situ
furnace, stress, light …

Advanced
- Full stress
  (energy resolution)
- 3D mapping
  (depth resolution)

Structure: phase, orientation, strain
Morphology: Grain position & shape
Crystal defects: location & nature

Unique in Europe

µLaue diffraction (µLaue)
X-ray (scattering signal) microscopy for materials studies

• Local (residual) strains measurements

• Surface technique with useful probing depth: ~ several 10 µm

• Small gauge volume: < 500x500nm² x ~10 µm or grainsize limited

• X-ray equivalent to EBSD with:
  • Higher penetration, less sample preparation
  • Conducting or isolating materials
  • Finer resolution in orientation: x 1/100
  • High strain resolution: 10⁻⁴

Still needs:
  • Accelerated acquisition - automated analysis
  • Synchrotron beamline
μLaue diffraction (μLaue)

Strain/orientation resolution: $10^{-4}$

Microstructure & defects in Hard X-ray detector CdTe

High spatial resolution (< 1µm)

Investigation of plasticity damages and stress level

In situ
Maths for Laue in a nutshell (1/2) : mirror reflection

Reflection of incident beam $\mathbf{u}_i$ by a mirror with normal vector $\mathbf{u}_q$ (elastic collision)

$\mathbf{u}_f = \mathbf{u}_i + 2 \sin \theta \mathbf{u}_q = \mathbf{u}_i + 2 (\mathbf{u}_i \cdot (-\mathbf{u}_q)) = \mathbf{u}_i - 2 \frac{\mathbf{q} \cdot \mathbf{u}_i}{||\mathbf{q}||^2} \mathbf{q} = \mathbf{u}_i + \frac{\mathbf{q}}{R}$

$||\mathbf{u}_f|| = ||\mathbf{u}_i||$

$R\mathbf{u}_f = R\mathbf{u}_i + \mathbf{q}$

$R(\mathbf{q}) = -\frac{||\mathbf{q}||^2}{2q \cdot \mathbf{u}_i}$

- Given $\mathbf{q}$ and $\mathbf{u}_i$, it is easy to construct the reflected (or scattered) beam $\mathbf{u}_f$
Maths for Laue in a nutshell (2/2) : Bragg reflection

Atomic planes act as a mirror with normal $q$ (elastic scattering). Direction of the incoming wave (wave vector $k_i$) is changed into a new direction (wave vector $k_f$) due to $q$.

\[
\begin{align*}
\frac{2\pi}{\lambda} u_f &= \frac{2\pi}{\lambda} u_i + \frac{4\pi}{\lambda} \sin \theta u_q \\
|k_f| &= |k_i| + |q| u_q = k_i + q
\end{align*}
\]

avec

\[
|k_f| = |k_i| + |q| \quad \text{R}(q) = \frac{2\pi}{\lambda} = \frac{|q|}{2\sin \theta} = E(\text{keV}) \frac{2\pi}{12,398} = -q^2/(2q.u_i)
\]

Given $q$ and incoming beam vectors $u_i$, the scattered vector $k_f$ (direction and norm) is easily determined geometrically by means of the bisection of $q$.
Laue Principles | measuring reciprocal directions & orientations

Reciprocal lattice nodes
\[ G^* = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* \]

2d \sin \theta = \lambda \iff G^* = k_f - k_i

Advantages
- Always diffraction signal for any crystal orientation
- Large number of Bragg reflections
- No sample rotation
- Determine easily orientation

Mapping:
- 1 Reciprocal direction \(\iff 1\) Laue spot
- \(\iff\) fund. and harmonics reciprocal nodes

\[ \left( X, Y \right)_{\text{detector}} \]

Reciprocal direction \(\iff\) Laue spot
\(\iff\) fund. and harmonics reciprocal nodes
\[ G^* / \parallel G^* \parallel \]
Laue Principles | measuring reciprocal directions & orientations

Matching exp. (red) Theo. (black) spots

Projection sphere of directions

Absolute positions => orientation

Relative positions / ref. => deformation

Simulating strain (black->blue)
Laue Pattern visualisation. Frame

Zone axis \( <110> \)

Pixel Y
Pixel X

Incidence plane

Si(001)

Zone axis \( <110> \)

\( \mathbf{u}_f, \mathbf{u}_i = 2\theta \)
\( \mathbf{u}_f, \mathbf{z} = \chi \)

Scattering angles

\[
\mathbf{q} = 2\sin \theta \begin{pmatrix}
-\sin \theta \\
\cos \theta \sin \chi \\
\cos \theta \cos \chi
\end{pmatrix} = 2\sin \theta \tilde{\mathbf{q}}
\]

\[
\mathbf{u}_i = \begin{pmatrix}
1 \\
0 \\
0
\end{pmatrix}
\]

\[
\mathbf{u}_f = \begin{pmatrix}
\cos 2\theta \\
\sin 2\theta \sin \chi \\
\sin 2\theta \cos \chi
\end{pmatrix}
\]
Laue Principles | Sensitivity to deviatoric strain

Laue =⇒ Deviatoric strain $\varepsilon^* :$ angular unit cell distortions i.e. $b/a, c/a, \alpha, \beta, \gamma$

Laue + measuring 1 spot energy =⇒ 1 reciprocal vector length

=> unit cell volume, $a,b,c, \alpha, \beta, \gamma$ (full strain)
Laue Pattern: Diffraction signal
4 Mpix, 16bits

Optical microscope:
Surface image
40x40 µm² FOV

Focused White beam
5 – 25 keV
Size = 0.5 x 0.5 µm²

Tilt controlled table
(closed-loop control
+/− 0.1 µrad)

KB mirrors focusing

Fluorescence detector
Chemical information
Resolution ~125 eV
μLaue | 2D microscopy

2D lateral resolution < 0.5 x 0.5 μm²

- Surface optical microscope
- Local crystal orientation
- Local unit cell strain

- Indexing
- Refinement

- Element/phase mapping
  - Fluo.
  - Kα Ge

- In situ & operando
  - σ, k_BT,
  - e-, light collection (XEOL),
  ...

- 2D detector
- Tensile test
- Furnace
µLaue | First steps: (1) Locate & Measure

Visible microscope
Field of view: 200X200 to 40X40 µm²

Fluorescence map
Duration time: 2-10 min

- Find micro region in macro sample (can be long!)
- Select single point/ROI for Laue diffraction
μLaue | First steps: (2) Imaging & Tracking

Single crystal & polycrystal mapping

from Images dataset

Images ROI mosaic

Single peak monitoring

from peaks lists dataset

GB & structural defects mapping

μLaue | Second steps: (1) Indexation & (raw) Orientation

Indexing a single Laue pattern

Indexing results
- Miller indices (hkl)
- crystal orientation (U_{raw})

\[ G^* = h.a^* + k.b^* + l.c^* \]

\[ q = U_{raw}.B_0.G^* = k_f - k_i \]

B_0 initial matrix a^*,b^*,c^* in Lab. frame

\[ U_{raw} = U.(I_3 + \varepsilon) \quad \varepsilon \text{ Strain} \]

\[
B_0 = \begin{pmatrix}
a^* & b^* \cos \gamma^* & c^* \cos \beta^* \\
0 & b^* \sin \gamma^* & -c^* \sin \beta^* \cos \alpha \\
0 & 0 & c^* \sin \beta^* \sin \alpha
\end{pmatrix}
\]

\[
\cos \alpha = \frac{\cos \beta^* \cos \gamma^* - \cos \alpha^*}{\sin \alpha^* \sin \beta^*}
\]
Indexing Basic method principles: Recognition of angle between 2 atomic planes normals

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</table>
Indexing: recognition of angle between 2 atomic planes normals

if: \(|\alpha_{k,l} - \alpha_{i,j,\text{ref}}| < \alpha_{\text{max}}\), angle \(\alpha_{i,j}\) in LUT is \textit{recognised} and corresponds to a pair of planes \(\{h_1k_1l_1, h_2k_2l_2\}\) and 2 \textit{potential} solutions:

<table>
<thead>
<tr>
<th></th>
<th>Spot 1</th>
<th>Spot 2</th>
<th>Orientation Matrix</th>
</tr>
</thead>
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<tr>
<td>Solution 1a</td>
<td>(h_1k_1l_1)</td>
<td>(h_2k_2l_2)</td>
<td>UB1a</td>
</tr>
<tr>
<td>Solution 1b</td>
<td>(h_2k_2l_2)</td>
<td>(h_1k_1l_1)</td>
<td>UB1b</td>
</tr>
</tbody>
</table>

Then, 2 Laue patterns are simulated:

\(\alpha_{1,2} = 29,501^\circ\)
\(h_1k_1l_1 = (-1 1 1)\)
\(h_2k_2l_2 = (-1 3 1)\)
Evaluate matching level between two Laue Patterns

Automatic pairing between neighbouring theoretical (hollow red circles) and experimental (filled circles) is performed by (Auto Links) to:

- ① assess the Laue patterns ‘matching rate’
- ② identify unambiguous pairs of (exp., theo.) for structure refinement

### Matching Rate

\[
\text{Matching Rate} = \frac{N_b(\text{green}^+)+N_b(\text{orange})}{N_{\text{spots}_{\text{theo}}}}
\]

### Unambiguous links

- ① Nb of unambiguous links
  \[
  N_{\text{pairs}}(\text{green})
  \]

### Within Matching tolerance angle

- 1 theo. spot linked close to 1 exp. spot
- 1 theo. spot linked close to several exp. spots
- 1 theo. Spot without neighb. Exp. Spots
  \((\text{Missing Reflection} \text{ in exp. Spots list})\)
Matching rates report of potential indexing results

Given two spots angle recognition, several potential indexing solutions (i.e. UB orientation matrix) are listed:

<table>
<thead>
<tr>
<th>#Matrix</th>
<th>Matched</th>
<th>Expected</th>
<th>Matching Rate(%)</th>
<th>std. dev.(deg)</th>
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</thead>
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<td>35</td>
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<td>0.01</td>
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<tr>
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<td>41.94</td>
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<td>2</td>
<td>8</td>
<td>34</td>
<td>23.53</td>
<td>0.01</td>
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</table>

- **Nbmatched**: Nb of exp. Spots (including harmonics) linked to a theo. spot
- **NbExpected**: Number of theo. Spots (including harmonics)
- **Matching Rate**: Nbmatched / NbExpected

A poor de Matching Rate can be due to:
- A small number of exp. Spots (see Peak Search procedure)
- The reference structure for indexing is not well chosen (strong lattice deformation ?)
- Matching tolerance angle is too small
Matching rates report of potential indexing results

Given two spots angle recognition, several potential indexing solutions (i.e. UB orientation matrix) are listed:

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UB0

UB2

UB1
Multiple indexation of superimposed Laue patterns

Indexation
- Miller indices (hkl)
- Crystal orientation ($U_{\text{raw}}$)
- Separate grains contributions

Sequential indexation in case of no orientation relationships between grains
\( \mu \text{Laue microscopy challenges} \)

Single image analysis of complex Laue patterns

- Raw binary image
- Polycrystals Laue patterns
- Segmentation
- Recognition by Template matching or standard angle recognition
- Crystal structure refinement

✓ parallel indexation to handle orientation relationships between grains
Dealing with $\Sigma 3$ twins in fcc

Twins
Ambiguous superimposition of Laue spots (of different grains and hkl)

100% matching

~33% matching

UB_parent

UB_child_1

UB_child_2

UB_child_3

UB_child_4
Dealing with sigma3 **twins** in fcc

Checking various Laue spots presence
(+ Knowing Laue spot energy solves the ambiguity!)

-511 15700 ev
-111 5166 eV
111 5166 eV

-202 6934 ev

-202 6888 ev
282 20847 eV

-15-1 15612 ev
-1-15 15492 ev
-1-11 5166 ev
Fine Texture can be resolved (at least manually)

111 textured polycrystalline Cu
μLaue | Second steps: (1) Indexation & (raw) Orientation

Orientation map

Useful to Correlate

**scattering** – **spatial** informations

Orientation, texture, Grains size, shape, positions, phase

But Also: strain, defects,...

Laue Diffraction | Data analysis

LaueTools Suite Software for Laue microdiffraction data analysis

1- Peak Search
2- Indexing
3- Unit Cell Refinement
4- Map results plot
5- Automatic Analysis

https://gitlab.esrf.fr/micha/lauetools
http://sourceforge.net/projects/lauetools/

µLaue Diffraction | Phase Identification

Oxide layer growth at UO₂ pellet surface

Single shot measurement:
3 variants with quadratic strain c/a ~ 1.03

In situ Phase transitions of (GeTe)ₙSb₂Te₃ (6 <= n <= 15)

Spatially resolved study of defects and distorted structures distribution

DVD


M. Schneider et al., Chem. Comm. 48 (16), 2192 (2012)
Strain refinement by least squares minimization
- needs refined detector geometry (calibration)
- provides :
  – unit cell deviatoric strain $\varepsilon^*$
  – crystal orientation $U$

$$ q = U(l_3 + \varepsilon) B_0 = k_f - k_i $$

Normal-to-surface strain level in polycrystalline He-implanted \( \text{UO}_2 \)


Mechanical properties under load Data => input for FEA
Defects induced by He implantation in UO$_2$ polycrystal

Radial streaking => Surface swelling

=>$\Rightarrow$Intragrain fine misorientation
 =>$\Rightarrow$Activated slip system

=>$\Rightarrow$ Stress release modes, Slip system recognition

μLaue Diffraction | *in situ* mechanical test

**Data measurements & analysis validation**

**Errors estimation**

*In situ elastic* bending

- Validation:
  - uncertainties on strain
  - reliability of data analysis chain
- In situ structural characterization
- Determine behaviour law

---

Reversible phase transformation (SMA) during cycle in CuBeAl

E. Plancher Thesis 2015

S. Berveiller *et al*, Acta Mat. 59 (2011) 3636

E. Plancher Thesis 2015
**µLaue Diffraction | in situ mechanical test**

- Well controlled micromechanical tests
  - Crystal & GB orientation
  - Fine Force resolution
  - Tension, compression, bending, torsion, ...

- Measurements of **peak elongation direction and width**
  → Activated slip systems
  → Dislocations density

- **μLaue Add-ons** | Going beyond phase/orientation/strain determination

- **Full stress/6 lattice parameters** measurements

  \[ \varepsilon^* = \varepsilon - \bar{\varepsilon}I_3 \]

  Deviatoric strain
  Standard Laue Pattern analysis
  Full strain
  hydrostatic strain

- **Mechanical assumption**  e.g.  \( \sigma_{33} = 0 \)

  Solve \( \sigma = \mathbf{C} \varepsilon \) for \( \bar{\varepsilon} \) and 5 \( \sigma_{ij} \)

- **Laue spot energy fine experimental measurement:**

  \[ \varepsilon_{hkl} = \bar{\varepsilon} + (\varepsilon^* \tilde{\mathbf{G}}) \tilde{\mathbf{G}} \]

  Determine \( \bar{\varepsilon} \) then \( \varepsilon \) then \( \sigma = \mathbf{C} \varepsilon \)

  How?

  - insert monochromator (optics hutch)
  - use of a 0D movable energy-resolved detector
  - insert monochromator (diamond) in transmission (in exp. Hutch)

- **Depth resolution**: 3D microscopy
From the deviatoric to the full strain tensor

**Hypothesis**: No stress perpendicular to the free surfaces ($\sigma_{33} = 0$)

Hooke’s law $\mathbf{F} = k \mathbf{x}$ ... *with tensors and Voigt’s notation*:

\[
\begin{pmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{33} = 0 \\
\sigma_{23} \\
\sigma_{13} \\
\sigma_{12}
\end{pmatrix} =
\begin{pmatrix}
c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\
0 & 0 & 0 & c_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & c_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & c_{44}
\end{pmatrix}
\begin{pmatrix}
\varepsilon'_{11} + \varepsilon_h/3 \\
\varepsilon'_{22} + \varepsilon_h/3 \\
\varepsilon'_{33} + \varepsilon_h/3 \\
2\varepsilon'_{23} \\
2\varepsilon'_{13} \\
2\varepsilon'_{12}
\end{pmatrix}
\]

$\sigma =$ stress (unknown)

$c_{11}, c_{12}, c_{44} = 126, 44.0, 67.7$ GPa = elastic coefficients of Ge

$\varepsilon'$ = deviatoric strain (measured)

$\varepsilon_h =$ hydrostatic strain (unknown)

Solve the 6 equations for the 6 unknowns to get the full strain tensor
Local stress agrees with macroscopic stress (by $\sin^2 \psi$)

$$\sigma = \mathbf{C} \varepsilon$$

$$\sigma = \begin{pmatrix} -986 & -35 & 30 \\ -35 & -878 & 25 \\ 30 & 25 & -209 \end{pmatrix} \text{Mpa}$$

Spatial distribution of full stress tensor in polycrystalline ZrO$_2$-based electrolyte for Solid Oxide Fuel cell

Direct energy measurement with energy-resolved detector (SDD)

Local energy spectra acquired with the Laue diffraction pattern (no overhead)
Energy calibration required (using fluorescence or known diffraction peaks)
Quest for the last parameter: The Rainbow filter technique

"Inverse" monochromatic diffraction

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"Inverse" monochromatic diffraction

The Rainbow filter technique

Relationship between filtered out energies and diamond filter angle

The full bandwidth of the beamline (5-25 keV) can be covered
Rainbow filter technique: full strain tensor determination
Stress free surfaces hypothesis vs rainbow technique

Excellent agreement up to 5% strain, the stress-free surface hypothesis is valid!

Precision is lower at higher strain (smaller devices, harder to measure)
Evidencing non-linear strain effects in Ge

Raman spectral shift for <100> uniaxial stress

Strain measurements: μLaue as reference for Raman spectroscopy

A. Gassenq, et al., APL 108, 241902 (2016)
μLaue Diffraction | towards 3D

Determine orientation & strain gradients in depth

1. Ybeam position of scattering source
Pixel intensity = f(y\text{wire})

2. Reconstruction:
Laue pattern = f(y\text{beam})

Depth resolution: 0.3\text{µm}

10^{-4} Resolution preserved in depth
μLaue Diffraction | Getting more & more accurate

Combine μLaue with

DIC
Combine local and macro strain fields

EBSD
EBSD- based selection & Laue Measurements

Improve μLaue with

2D energy resolved detector
Faster and more accurate Full stress measurements

Laue DIC
Enhance relative strain accuracy

Beam scan
Keeping sample free during in situ experiments
μLaue Diffraction | Conclusions

**X-ray Laue Microscopy**

- Intense, narrow beam (<500x500 nm²)
- Complementary x-ray white and energy resolved (5 – 23 keV)
- Dedicated installation:
  for basic 2D orientation map to advanced quantitative structural data
- 'simple', 'fast', non destructive, *in situ* experiments

**Open to a large scientific community**

- Metallurgy, structure and functional materials
- μ-electronics & -device, μ & nano-object mechanics
- mineralogy, cultural heritage

**Fundamental and applied sciences**

- local structure
- metrology, control and reliability
μLaue Diffraction | Conclusions

μLaue @ ESRF

Next proposals submissions round  September 2020

Access through CRG (SOLEIL) and program committees

http://sunset.synchrotron-soleil.fr/sun/

Open to free tests and collaborating developments!

Thank you for your attention
Appendix: μLaue | Optics setup

2 steps demagnification with secondary source

White mode

Beamsize (FWHM) < 0.5x0.5 μm²

Monochromatic mode

Fixed microbeam position and incident angles = Image of $S_2$

Experimental Hutch | Optics Hutch
Appendix: μLaue Diffraction | Energy measurements

Movable Energy dispersive detector (SDD)

Beamsize (FWHM) < 0.5x0.5 μm²

Laue pattern ε* refinement

ε_{hkl} = \bar{c} + (\epsilon^* \tilde{G}) \cdot \tilde{G}

=> Determine \bar{\epsilon} then \epsilon

Advantage:
Measurements of ε* and ε_{hkl} at same (r,t)
=> better accuracy
Several spot energy measurements
=> better statistics

Fast & reliable full strain tensor \epsilon

Readings:

Appendix: μLaue Diffraction | Energy measurements

Monochromator Transmission Mode (Rainbow)

Beamsize (FWHM) < 0.5x0.5 μm²

Advantages:
- Monochromators working in parallel
  => even better statistics

Fast & reliable full strain tensor ε

Monochromator Transmission Mode (Rainbow)

Transmission C_{dia}
monochromator

Missing colors
all colors

Laue pattern ε* refinement

Spot intensity

Peak photon energy

e^−

BM32

Φ

(535)_{Ge}

(311)_{dia} (111)_{dia}

(11-1)_{dia}

40%

ε_{hkl} = \bar{ε} + (ε*\tilde{G}).\tilde{G}

Ge (-311)
Appendix: Fine structural metrology for applied physics
Works of Samuel Tardif, CEA, CRG-IF BM32 beamline

Why straining Ge?

Strain engineering: from indirect to direct bandgap

Conduction band

Valence band

Γ

0.80 eV

0.66 eV

indirect

direct

L

HH

LH

SO

Poor light emitter

Good light emitter: on-chip Ge laser source!
How to strain Ge?

Strain concentration in GeOI membranes [Alban Gassenq]

Built-in thermal biaxial tensile stress

Strained microstructure

Underetched relaxed pad

How to strain Ge?

Model objects to study any arbitrary uniaxial or biaxial stress

Strain can be statically tuned by changing the design parameters (width, length, ...)

Measuring the strain at the µm scale: Raman spectroscopy

- Strain increases depending on bridge geometry.
  - Bulk Ge shift to strain conversion: 0.649 % per cm\(^{-1}\) (uniaxial)
  - 0.236 % per cm\(^{-1}\) (biaxial)

- [100] = traction direction
- Probe the Ge-Ge resonance

- Shift to strain conversion:
  - 0.649 % per cm\(^{-1}\) (uniaxial)
  - 0.236 % per cm\(^{-1}\) (biaxial)

Raman spectroscopy measurements with Guilherme Osvaldo Dias, SEM image by Alban Gassenq
Raman spectroscopy mapping

Good check of the homogeneity of the strain but analysis is difficult when the strain is not pure bi- or uniaxial
Laue µdiffraction at ESRF CRG BM32 in a nutshell

**Diffraction peaks**

<table>
<thead>
<tr>
<th>2θ</th>
<th>X</th>
<th>Intensity</th>
</tr>
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<td>...</td>
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**Sample**

Microbeam
typ. 0.6 µm (H) x 0.8 µm (V)

**2D detector**

X-ray Laue diffraction

**Achromatic microfocusing optics**

White beam
5 – 25 keV
300 µm x 300 µm

**LaueTools**
Analysis software
(developed at BM32 by J.S. Micha, O. Robach)

**Orientation + Deviatoric strain tensor**

(= sensitive to **symmetry changes**
but insensitive to **homothetic transformations**)

\[
(\text{Full Strain}) = (\text{Hydrostatic Strain}) + (\text{Deviatoric Strain})
\]
Direct measurement of the deviatoric strain tensor
Distortions and rotations of the lattice (uniaxial stress)

Small anticlastic relaxation of the stretching arms and strong distortion + rotation near the fillets (max shear stress)
Direct measurement of the deviatoric strain tensor
Out-of-plane rotations are negligible (no anticlastic or synclastic relaxations). Strong distortions and rotations near the fillets.
From the deviatoric to the full strain tensor
Without measuring Laue spots energy

**Hypothesis**: No stress perpendicular to the free surfaces ($\sigma_{33} = 0$)

Hooke's law $\mathbf{F} = k \mathbf{x}$ ... *with tensors and Voigt’s notation*:

\[
\begin{pmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{33} = 0 \\
\sigma_{23} \\
\sigma_{13} \\
\sigma_{12}
\end{pmatrix} =
\begin{pmatrix}
c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\
c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\
0 & 0 & 0 & c_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & c_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & c_{44}
\end{pmatrix}
\begin{pmatrix}
\varepsilon'_{11} + \varepsilon_h / 3 \\
\varepsilon'_{22} + \varepsilon_h / 3 \\
\varepsilon'_{33} + \varepsilon_h / 3 \\
2 \varepsilon'_{23} \\
2 \varepsilon'_{13} \\
2 \varepsilon'_{12}
\end{pmatrix}
\]

$\sigma = $ stress (unknown)
$c_{11}, c_{12}, c_{44} = 126, 44.0, 67.7$ GPa = elastic coefficients of Ge
$\varepsilon' = $ deviatoric strain (measured)
$\varepsilon_h = $ hydrostatic strain (unknown)

Solve the 6 equations for the **6 unknowns** to get the full strain tensor
XRD measurements vs FEM simulations

<table>
<thead>
<tr>
<th>FEM</th>
<th>μLaue</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\varepsilon_{xx}$</td>
<td>$\varepsilon_{yy}$</td>
</tr>
<tr>
<td>$\varepsilon_{yy}$</td>
<td>$\varepsilon_{zz}$</td>
</tr>
</tbody>
</table>

\[
\varepsilon_h (\%) \quad \text{Nbr of fitted peaks} \quad \text{fit dev (pix)}
\]

\[
\begin{align*}
\text{strain} & \quad \text{strain} \\
\text{X (μm)} & \quad \text{X (μm)} \\
\text{Y (μm)} & \quad \text{Y (μm)}
\end{align*}
\]
XRD measurements vs FEM simulations

FEM | µLaue

\[
\varepsilon_{xx}, \varepsilon_{yy}, \varepsilon_{zz}, \varepsilon_{xy}, \varepsilon_{yz}, \varepsilon_{xz}
\]

\[
\text{strain} \% \
\text{Nbr of fitted peaks} \
\text{fit dev (pix)}
\]
Uniaxial [111] strain in the range 0 % – 1.5 %, bandgap energies consistent with theoretical model

Describing how the lattice is strained (deformed) under stress (~force)

General case

\[
\begin{pmatrix}
\varepsilon_{xx} & \varepsilon_{xy} & \varepsilon_{xz} \\
\varepsilon_{xy} & \varepsilon_{yy} & \varepsilon_{yz} \\
\varepsilon_{xz} & \varepsilon_{yz} & \varepsilon_{zz}
\end{pmatrix}
\]

Pure uniaxial tensile stress

\[
\begin{pmatrix}
\varepsilon_{xx} & 0 & 0 \\
0 & -\nu \varepsilon_{xx} & 0 \\
0 & 0 & -\nu \varepsilon_{xx}
\end{pmatrix}
\]

Pure biaxial tensile stress

\[
\begin{pmatrix}
\varepsilon_{xx} & 0 & 0 \\
0 & \varepsilon_{xx} & 0 \\
0 & 0 & -2\nu \varepsilon_{xx}
\end{pmatrix}
\]

\[\varepsilon_{ij} = \frac{\Delta i}{j} = \frac{\Delta j}{i}\]

\(\varepsilon_{xx}, \varepsilon_{yy}, \varepsilon_{zz}\) = axial strain

\(\varepsilon_{xy}, \varepsilon_{xz}, \varepsilon_{yz}\) = shear strain