100 nm 3D Laue Diffraction Technique for Ultra-High Spatial and Strain Resolution Combined with Versatile Analytical Probes

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National Synchrotron Radiation Research Center, Taiwan
NSRRC is a national light-source facility and research institution.
TPS major milestones

- Design: 2007
- Construction: 2010
- Commissioning: 2014
- Available to users: 2016
Comparison of Brilliance between TLS and TPS

TPS bending BLs @ 10 keV : $10^2$ times brighter
TPS ID BLs
  @ 1 keV: $\sim 10^3$ times brighter.
  @ 10 keV: $10^4$ times brighter.
# Experimental Techniques of TPS

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<th></th>
<th>imaging</th>
<th>scattering</th>
<th>spectroscopy</th>
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<td>CDI</td>
<td>structural diffraction</td>
<td>scattering</td>
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<td>05A Protein µ–crystallography</td>
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<td>09A Temporally Coherent XRD</td>
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<td>23A X-ray nano probe</td>
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<td>25A X-ray coherent scattering</td>
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<td>41A Soft X-ray scattering</td>
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<td>45A Sub µm soft spectroscopy</td>
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CDI: coherent diffraction imaging
XAS: X-ray absorption spectroscopy
XEOL: X-ray excited optical luminescence
RIXS: resonant inelastic X-ray scattering
PES: photoemission emission spectroscopy
TPS beamline plan

Phase I  7 ID  (2016)
Phase II  9 ID/BM  (2020)
Phase III  9 ID/BM  (2023)
Call for proposals

Apply for beam time at http://portal.nsrrc.org.tw

Beam time application schedule for cycle 2016-2
- Proposal submission deadline: April 30
- Beam time announcement: June 30

Cycle 2016-2 beam time
- September 22 – December 27, 2016

March 24 — June 28  Commissioning and pilot experiments
If you are interesting in our beamline, please contact with us!
Information from Laue Diffraction

Peak position:
1. Phase identification
2. Crystal orientation
3. Grain boundary mapping
4. Twin boundary mapping
5. Deviatoric strain mapping

Peak shape:
1. Dislocation type
2. Dislocation density

Courtesy Dr. Nobumichi Tamura, ALS
Applications

- Metal deformation, stress and strain partitioning, fatigue, grain growth, recrystallization, texture development, etc.;
- Stress effect and strain localization in semiconductor IC devices;
- Phase separation and domain interactions in complex oxides;
- Micro/nano-crystallography: nano-materials and minerals; single crystal diffraction at high pressure.
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

3D-Laue vs. 3D-EBSD vs. 3D-DCT

**3D-Laue**
- **Scanning type**: Non-destructive
- **Orientation**: Non-destructive
- **Strain/Stress**: Highest spatial resolution
- **Highest spatial resolution**: ~ few hours

**3D-EBSD**
- **Scanning type**: Destructive
- **Orientation**: Orientation
- **Strain/Stress**: Moderate spatial resolution
- **Moderate spatial resolution**: ~ few weeks*

**3D-DCT**
- **Full field type**: Non-destructive
- **Orientation**: Orientation*
- **Strain/Stress**: Strain/Stress*
- **Moderate spatial resolution**: ~ few hours

* Uniaxially strain assumed
* Included sample preparation

*Small mosaicity required
* Average strain tensors

Downloaded from Oxford Instruments
Downloaded from Zeiss.com
Information from Laue Diffraction

White Beam Laue Pattern

- Fit all diffraction peaks
- Index reflections
- Calculate orientation matrix and unit cell shape

**Deviatoric strain tensor**

\[ \varepsilon = \begin{pmatrix} \varepsilon_{xx} - \frac{\Delta}{3} & \varepsilon_{xy} & \varepsilon_{xz} \\ \varepsilon_{yx} & \varepsilon_{yy} - \frac{\Delta}{3} & \varepsilon_{yz} \\ \varepsilon_{zx} & \varepsilon_{zy} & \varepsilon_{zz} - \frac{\Delta}{3} \end{pmatrix} \]

- deviatoric strain (distortion)

\[ \frac{\Delta}{3} \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \]

- hydrostatic strain (dilatation)

Energy Scan with Mono Beam

- CCD pixels: Intensity (i, j) vs. Energy
- Intensity vs. Q
- Fit Q to calculate absolute lattice parameter
- Calculate absolute unit cell parameters
- Full strain tensor

Image Reconstruction

- Images at different wire positions
- Images at different depths
- take successive image differences, and sort pixel intensities to depth

Images at different wire positions


Why Laue for Nano-Diffraction?

Bragg Diffraction: \( 2d \sin \theta = n \lambda \)

Rotation in Bragg Diffraction will cause the X-ray Beam probing at different sample regions.

Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)
3D Laue Diffraction (DAXM)

Depth profiling

3-D spatial resolution: $\sim 0.3 \times 0.3 \times 0.5 \, \mu m^3$

Courtesy Dr. Wenjun Liu, APS
3D Image Reconstruction

Image Reconstruction

- 400 images at different wire positions
- 100 images at different depths

Subtract successive differences, and sort to depth

Calculate depths, sort & accumulate

100 µm

60 µm

10 µm

0 µm

Courtesy Dr. Wenjun Liu, APS
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

Data Processing

1. Scan sample & wire
2. Collect diffraction patterns
3. Indexing & refinement
4. 2D/3D crystal information
Wire-scan parameter: (APS)
1. sample surface to wire: 200 µm.
2. wire dimension: 50 µm
3. scan step: 0.45 µm (min.)
4. Detector pixel size: 200 µm
5. Detector to Sample: 50 cm

Depth Resolution:
1/1.414+0.1~0.8 µm (1µm step)  
0.5/1.414+0.1~0.45 µm (0.5 um step)

Wire-scan parameter: (TPS)
1. sample surface to wire: 15 µm.
2. plate dimension: 50 µm
3. scan step: 0.0005 µm (min.)
4. Detector pixel size: 172 µm
5. Detector to Sample: 43~67 cm

Depth Resolution:
50/1.414+6~41 nm (50 nm step, coarse-mode)

We further reduce the parallax contribution from 100 nm down to 6 nm by using SPM feedback technology.
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

Beamline Design

~100 x 100 x 50 nm$^3$ for 21A
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

Specifications

- **Energy Range:** 7 - 25 keV (mono-beam); 5 - 30 keV (pink-beam)
- **Photon Flux:** $3 \times 10^{11}$ photon/sec (7 keV); $3 \times 10^9$ photon/sec (25 keV); $> 1 \times 10^{15}$ photon/sec (pink)
- **Energy Resolution:** $10^{-4} (\Delta E / E)$
- **Spatial Resolution:** < 100x100 nm (lateral); 40 nm (depth resolution in 3D)
- **Effective WD of KB:** 48.8-60.8 mm (remove Be-window)
- **Environments:** Ambient & Vacuum (760 torr ~ $1 \times 10^{-7}$ torr)
- **Sample Temperature:** 100 K - 1,300 K (Vacuum); 300 K - 600 K (Ambient)
- **Maximum Sample Size:** 1.4x1.0 cm; 0.5 cm for thickness
- **Main Functionalities:** 2,3D-Laue XRD, XRF, XEOL/PL, SPM-IV, NanoXAS, PXM, and SEM
Scientific Opportunities

Bennett C. Larson et al.

Robert C. Rogan et al.

John D. Budai et al.

Lyle E. Levine et al.

Felix Hofmann et al.
Nature Communications 4, 2774 (2013)

Most of them are ex-situ, How about in-situ…?
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

Layout of End-station

- PILATUS2 100K (Projection XM)
- In-vacuum PILATUS3 6M (Laue XRD)
- UHV SEM Column
- Load-lock
- Silicon Drift Detector (Fluorescence)
- UHV Slits
- BPM/I₀
- K-B Box
- Sample & Probe Stage
- Adjustment Table
- AVCS

FOcusing X-Ray for MicrO-Structure Analysis (FORMOSA)
Inside the Chamber
The “In-vacuum” PILATUS3-6M detector

- **moving range**: 430~670 mm (from focal point)
- **angle resolution**: 0.02~0.015 degree
- **Triangulation 3D Laue**
Quadruprobe System

DAXM profiler
Indenter tip

Nickel probe
(Unisoku, STM/I-V)

Akiyama probe
(Nanosensors, AFM)

Fiber probe
(Nanonics, SNOM/XEOL/TERS/Gas-deliver/SECM)

Micromechanical probe
(microgripper, microsening)

Sample Holder
3D + 1D = Future of Materials Science

Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

1,2,3D Laue Diffraction

On-line SEM

PXM

SDD

E-field

Temp.

Indenter

Positioner

Laser

AHU

T = T₀ ± 0.02°C

AVCS

Disp. = 5 nm @ 2 Hz

Laser
Schedule

<table>
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<th>Items</th>
<th>Time</th>
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<td>2015.10</td>
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<tr>
<td>Source IUT-22</td>
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<td>Infrastructure</td>
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<td>Installation of End-station</td>
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<tr>
<td>First Experiment</td>
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Now

Functionalities of End-station

1\textsuperscript{st}-stage (2016.03~08)
1. 2D Laue Diffraction Image
2. 2D Fluorescence Image
3. PXM
4. VT Experiments (RT to 1,000K)
5. In-situ E-field & Force
6. SEM navigation + EDS

2\textsuperscript{nd}-stage (2016.08~)
1. 2D Laue Diffraction Image + 3D Laue Diffraction Reconstruction
2. 2D Fluorescence Image
3. PXM + Absorption Images
4. VT Experiments (RT to 1,000K) + Extend LT to 100K
5. In-situ E-field & Force + Optical (XEOL/NSOM)
6. SEM navigation + EDS + CL
Installation of 4BCM, KB & Exp. Chamber
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

Installation of SEM, TFM, BPM, Beamline Components
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

First results from on-line SEM
First results from on-line SEM
Taiwan Photon Source Phase-I Beamline: X-ray NanoDiffraction (XND)

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