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

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27 NOV, 2015 AOFSRR

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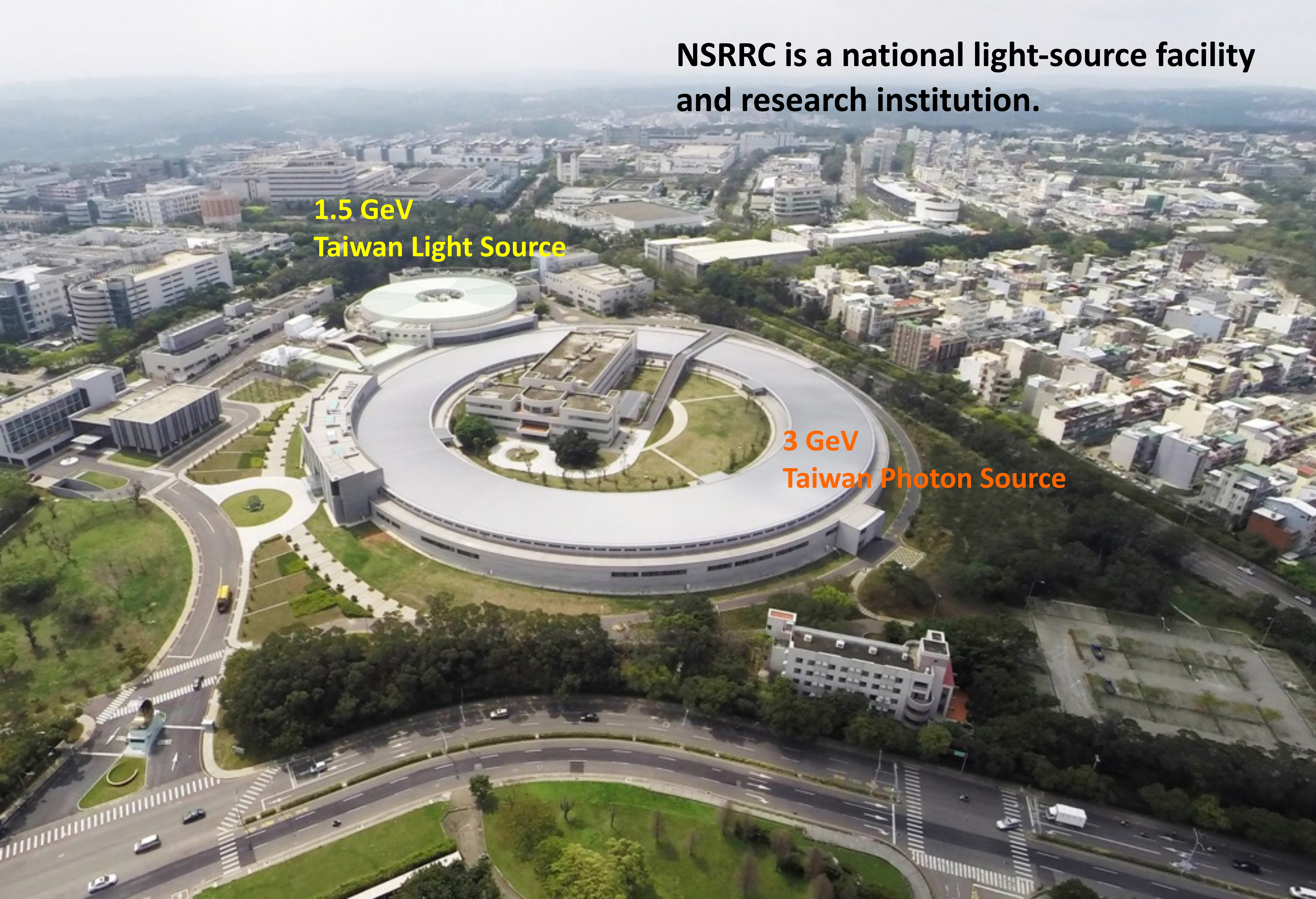
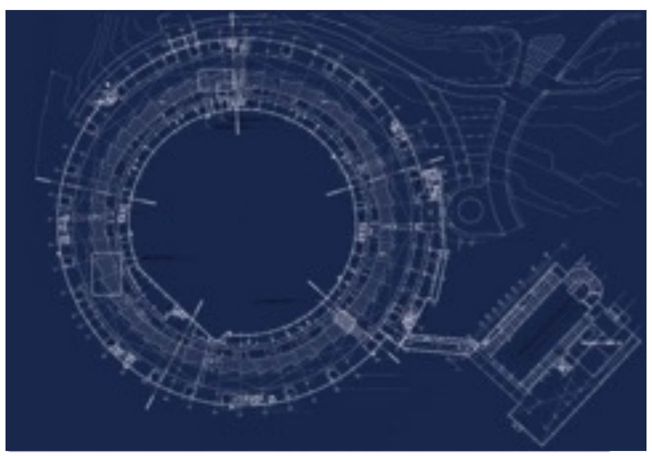


TPS major milestones



Design

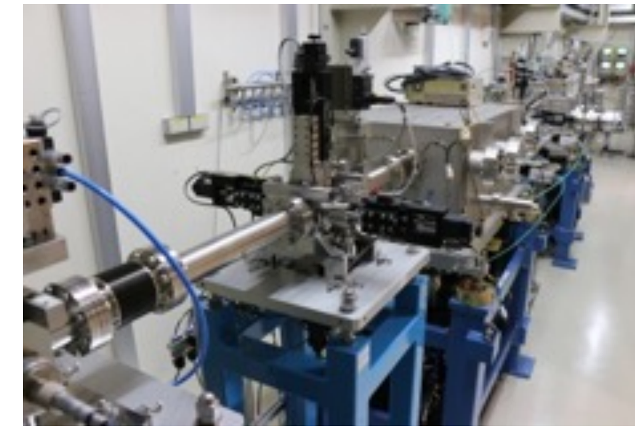
2007

Construction

2010

Commissioning

2014



Available to users

2016

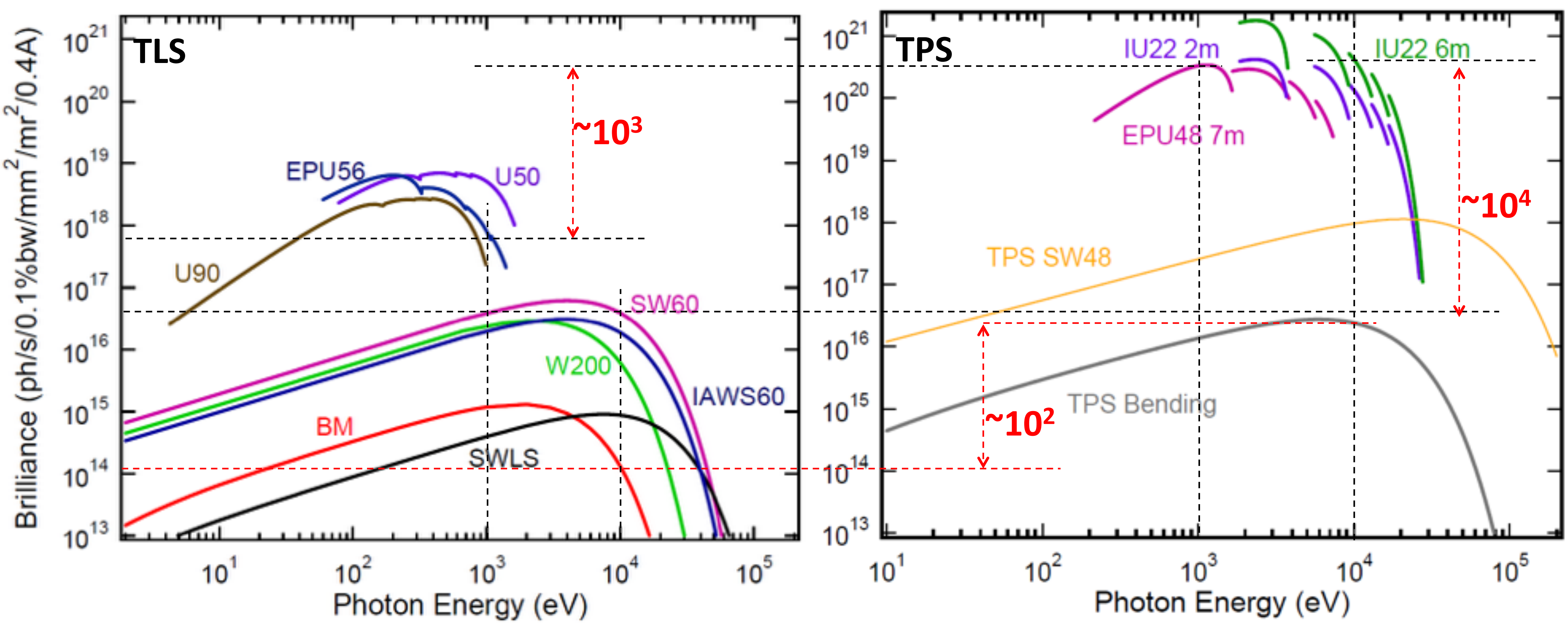
Comparison of Brilliance between TLS and TPS

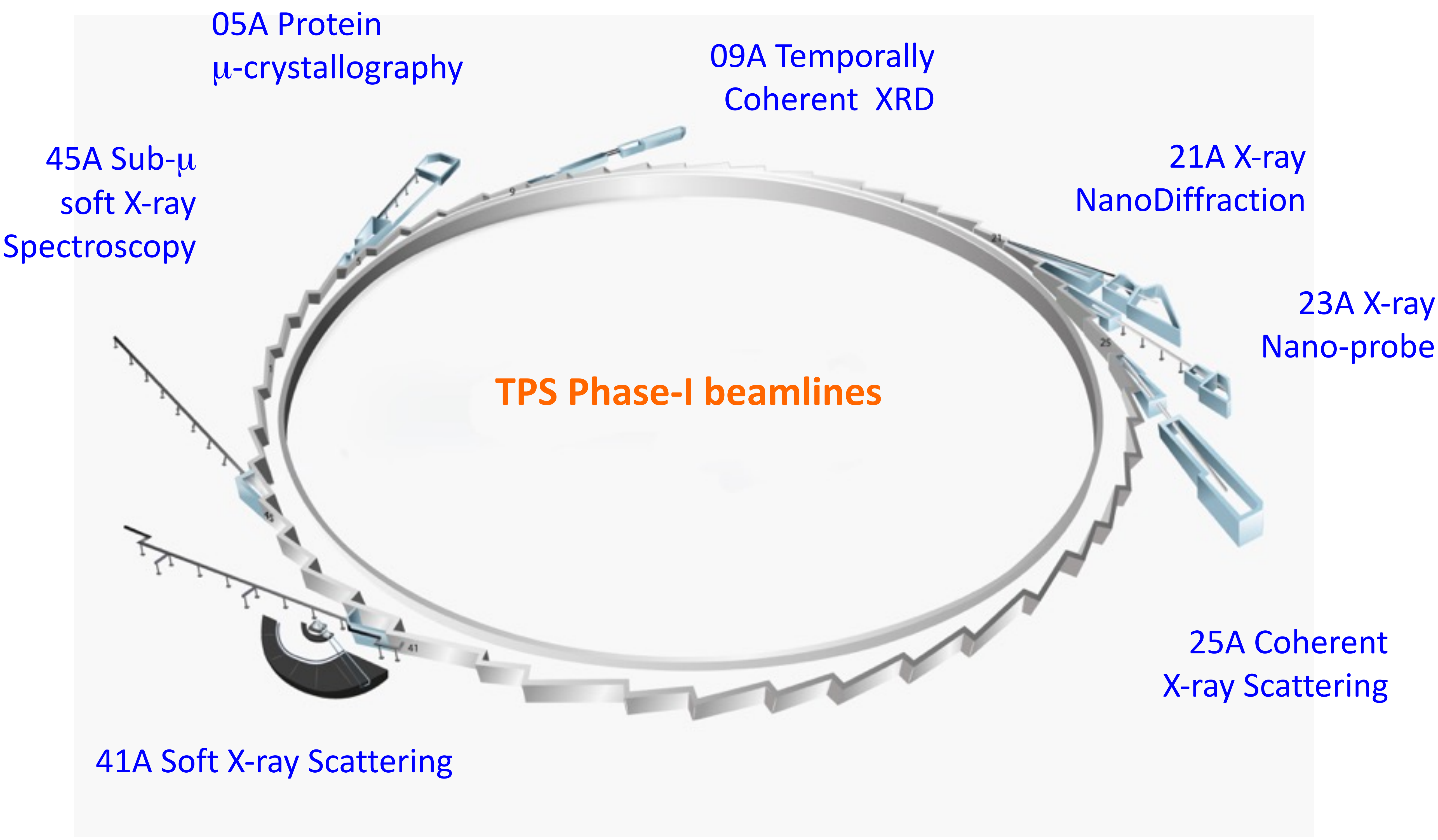
TPS bending BLs @ 10 keV : 10^2 times brighter

TPS ID BLs

@ 1 keV: ~ 10^3 times brighter.

@ 10 keV: 10^4 times brighter.



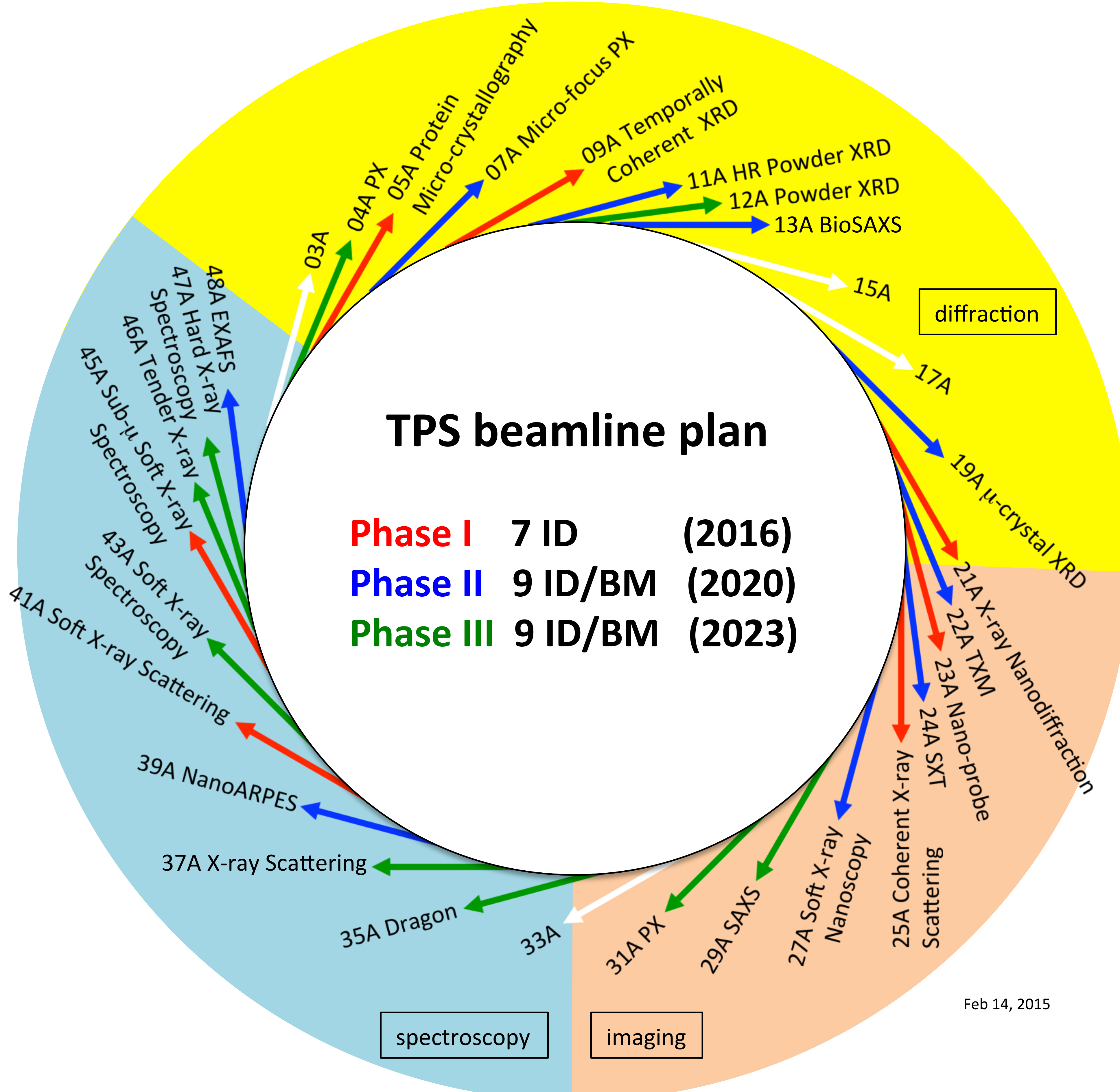


05A Protein
μ-crystallography
09A Temporally
Coherent XRD
45A Sub-μ
soft X-ray
Spectroscopy
21A X-ray
NanoDiffraction
23A X-ray
Nano-probe
TPS Phase-I beamlines
25A Coherent
X-ray Scattering
41A Soft X-ray Scattering

Experimental Techniques of TPS

	imaging	scattering		spectroscopy			
	CDI	structural diffraction	scattering	XAS	XEOL	RIXS	PES
05A Protein μ–crystallography		■		■			
09A Temporally Coherent XRD		■		■			
21A X-ray nanodiffraction		■		■	■		
23A X-ray nano probe	■	■		■	■		
25A X-ray coherent scattering	■	■	■	■			
41A Soft X-ray scattering	■	■	■	■		■	
45A Sub μm soft spectroscopy				■	■	■	■

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)
03A
04A PX
05A Protein Micro-crystallography
07A Micro-focus PX
09A Temporally Coherent XRD
11A HR Powder XRD
12A Powder XRD
13A BioSAXS
15A
17A
19A μ-crystal XRD
diffraction
21A X-ray Nanodiffraction
22A TXM
23A Nano-probe
24A SXT
25A Coherent X-ray Scattering
27A Soft X-ray Nanoscopy
29A SAXS
31A PX
33A
35A Dragon
37A X-ray Scattering
39A NanoARPES
41A Soft X-ray Scattering
43A Soft X-ray Spectroscopy
45A Sub-μ Soft X-ray Spectroscopy
46A Tender X-ray
47A Hard X-ray Spectroscopy
48A EXAFS
spectroscopy
imaging
Feb 14, 2015

Call for proposals

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

Beam time application schedule for cycle 2016-2

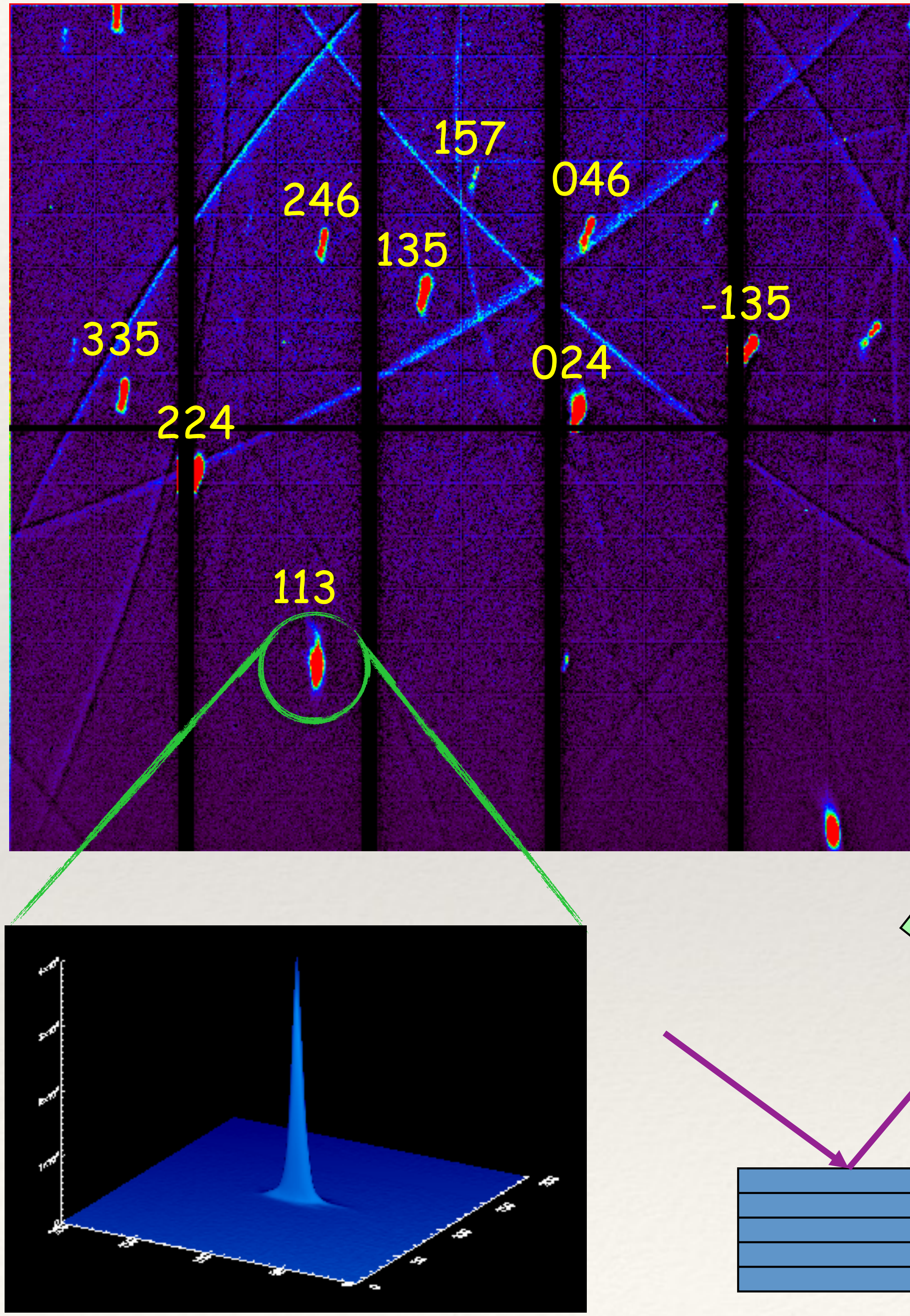
- Proposal submission deadline: April 30
- Beam time announcement: June 30

Cycle 2016-2 beam time

- September 22 – December 27, 2016

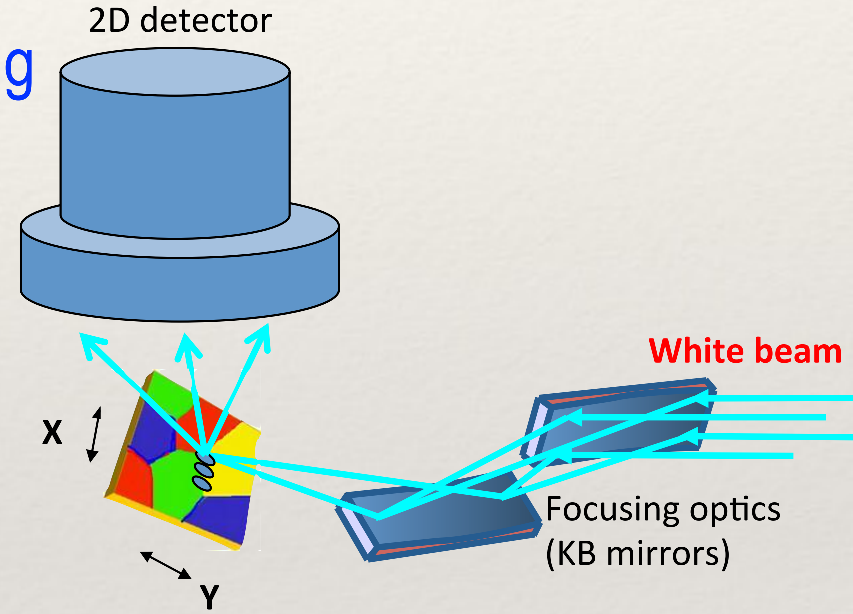
March 24 — June 28 Commissioning and pilot experiments
If you are interesting in our beamline, please contact with us!

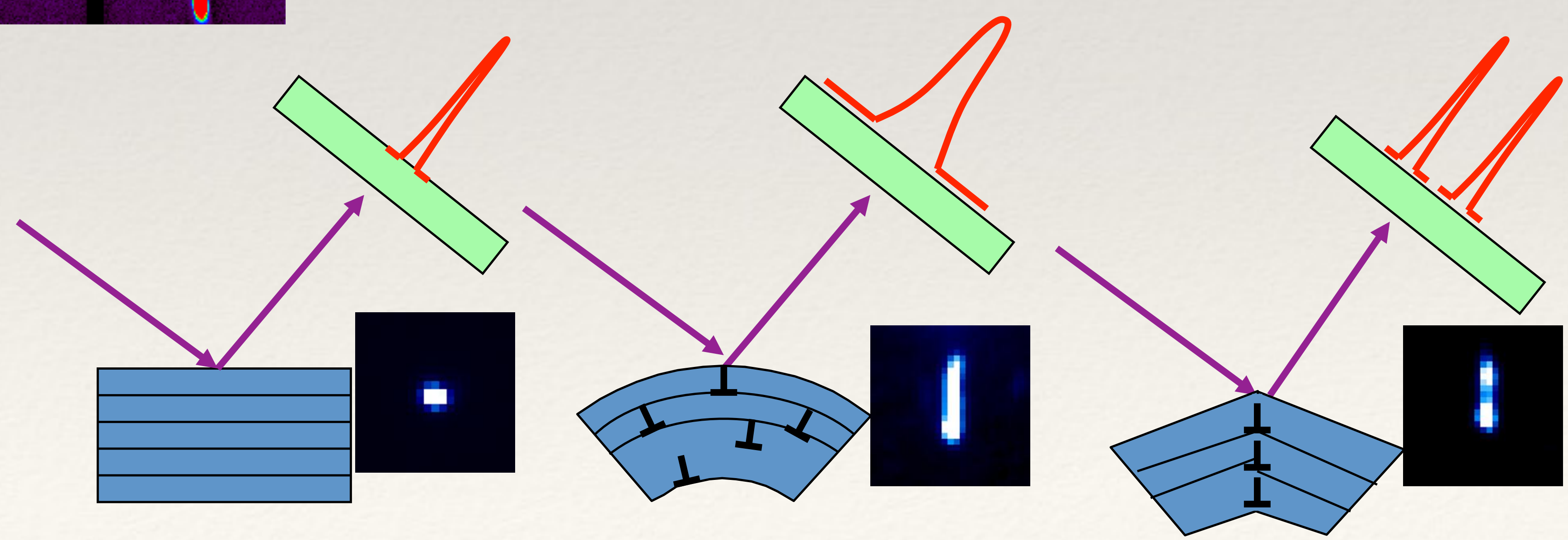
Information from Laue Diffraction



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

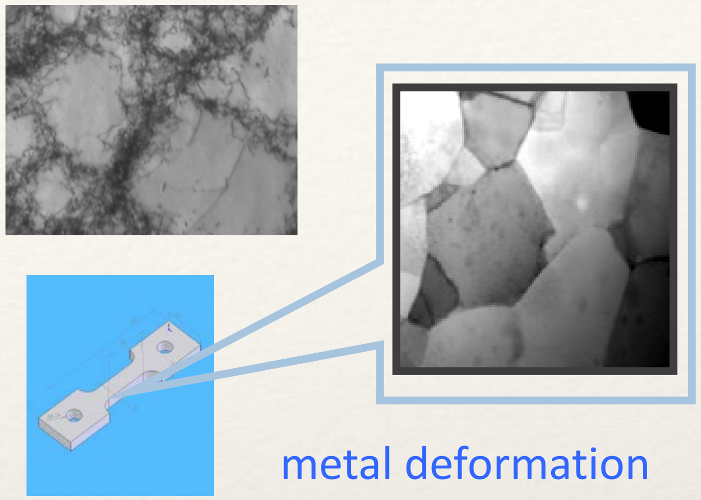


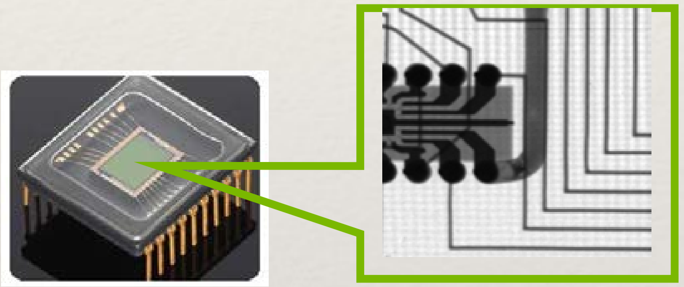


Courtesy Dr. Nobumichi Tamura, ALS

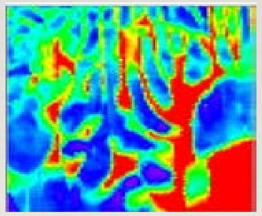
X-ray 3D Laue Diffraction Science

- *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.*

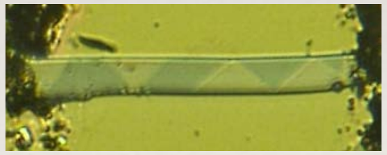




interconnects

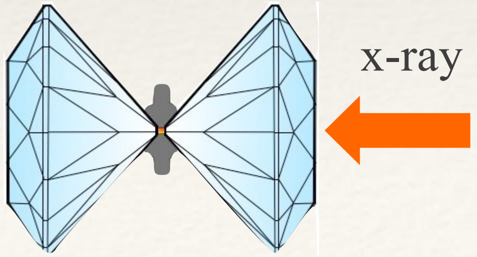


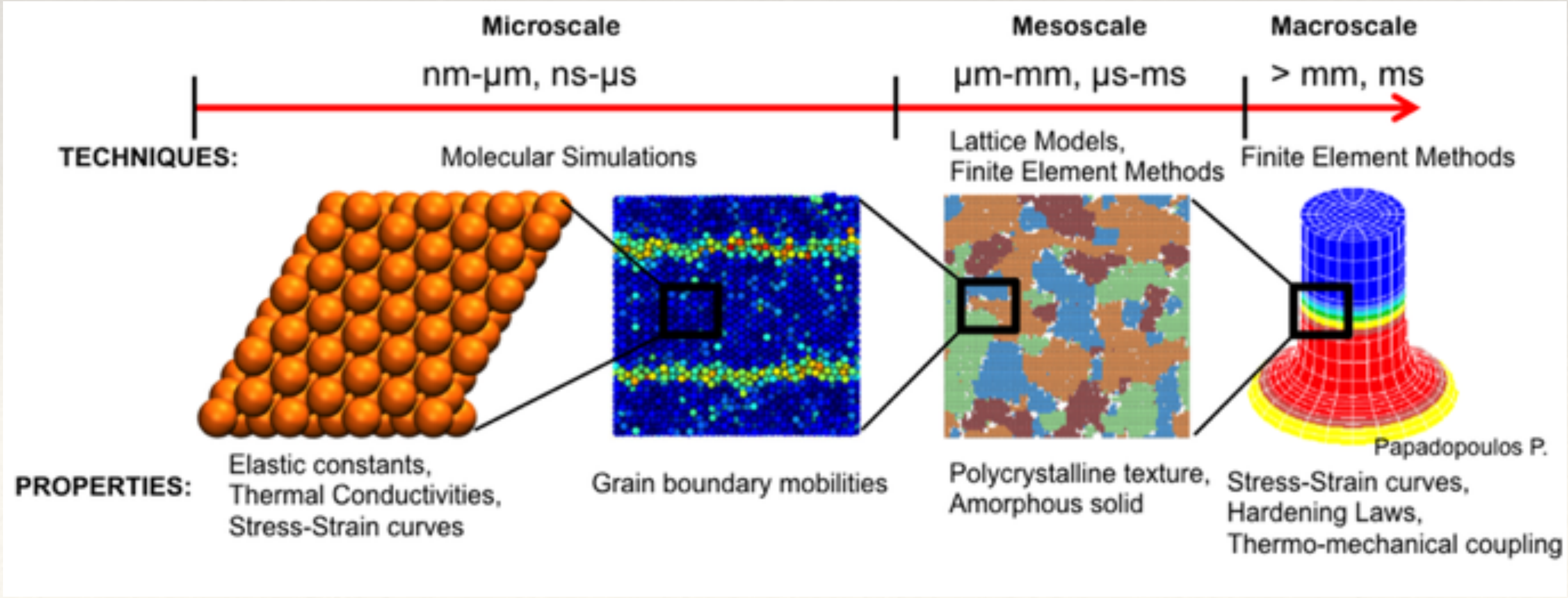
$(LaSr)MnO_3$



VO_2

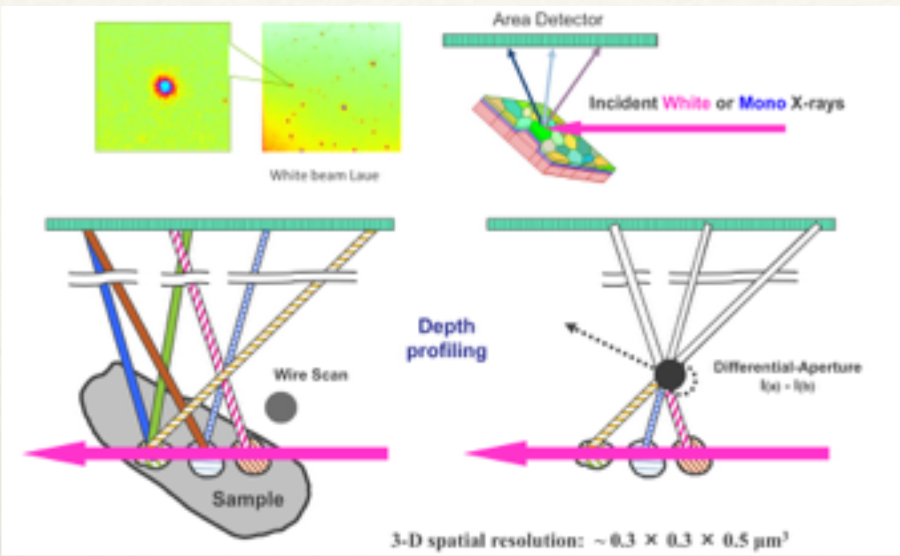
DAC





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

3D-Laue



Scanning type

Non-destructive

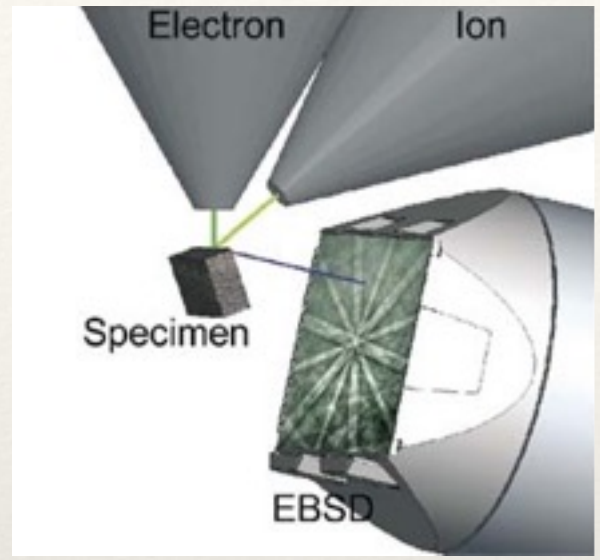
Orientation

Strain/Stress

Highest spatial resolution

~ few hours

3D-EBSD



Scanning type

Destructive

Orientation

Strain/Stress*

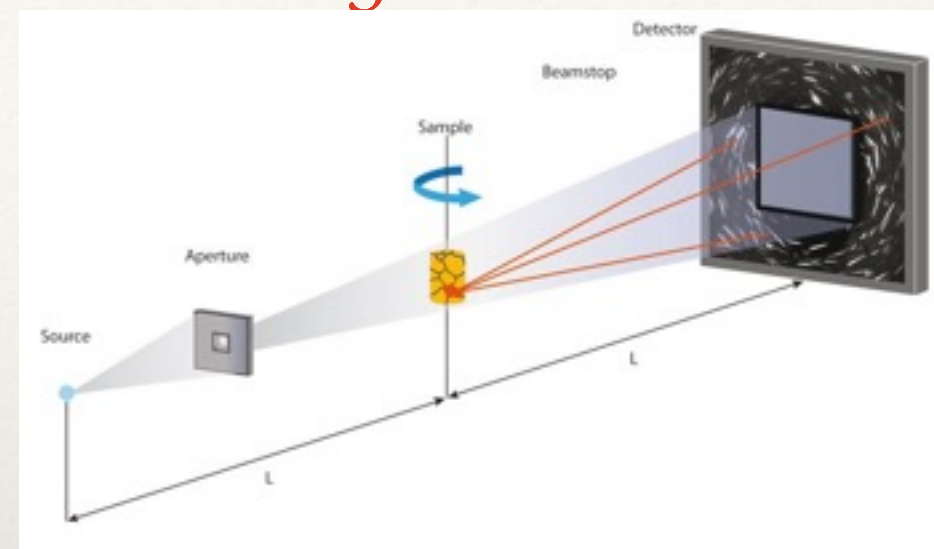
Moderate spatial resolution

~ few weeks*

* uniaxially strain assumed
* included sample preparation

downloaded from Oxford Instruments

3D-DCT



Full field type

Non-destructive

Orientation*

Strain/Stress*

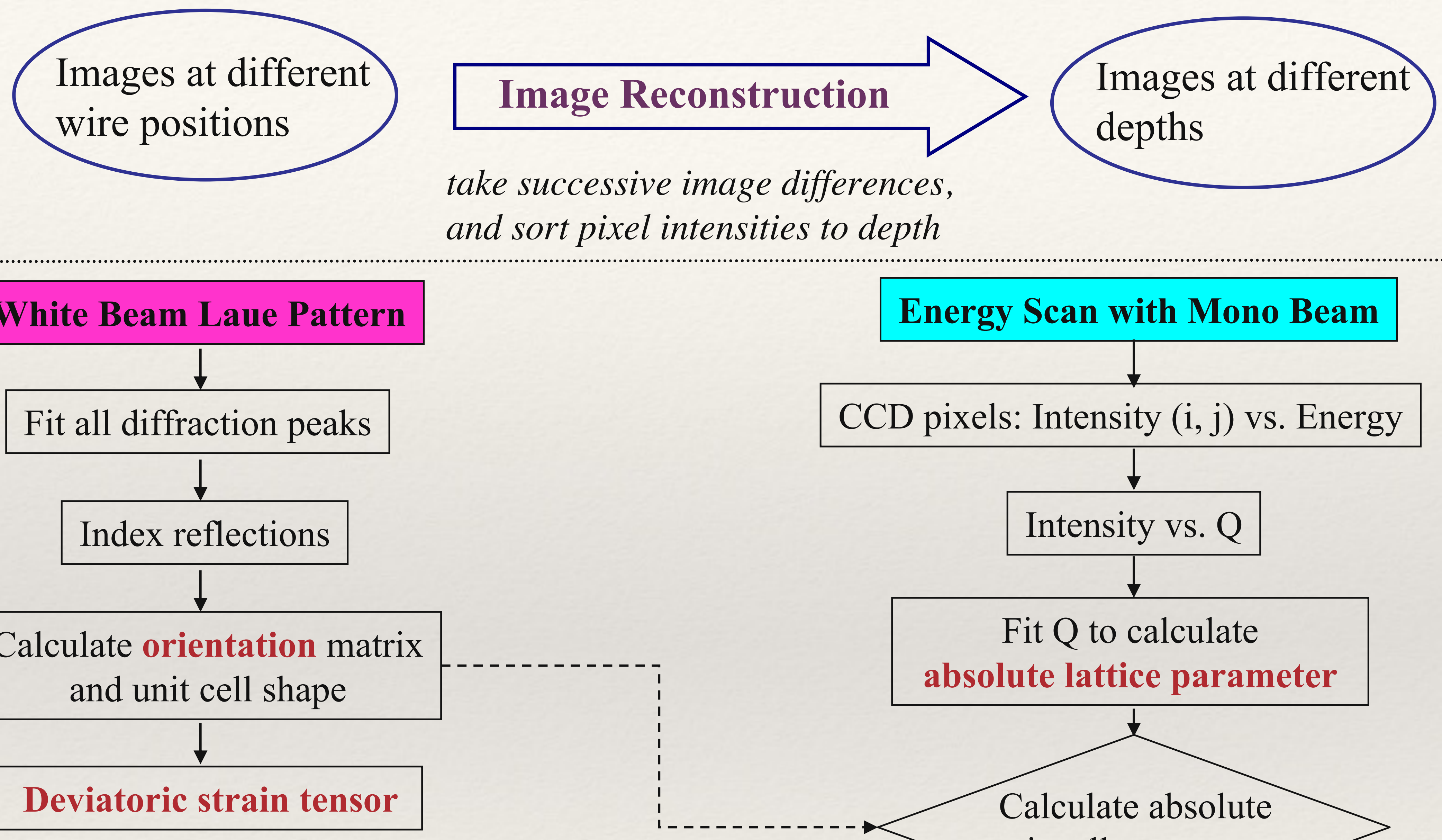
Moderate spatial resolution

~ few hours

*small mosaicity required
* average strain tensors

downloaded from Zeiss.com

Information from Laue Diffraction



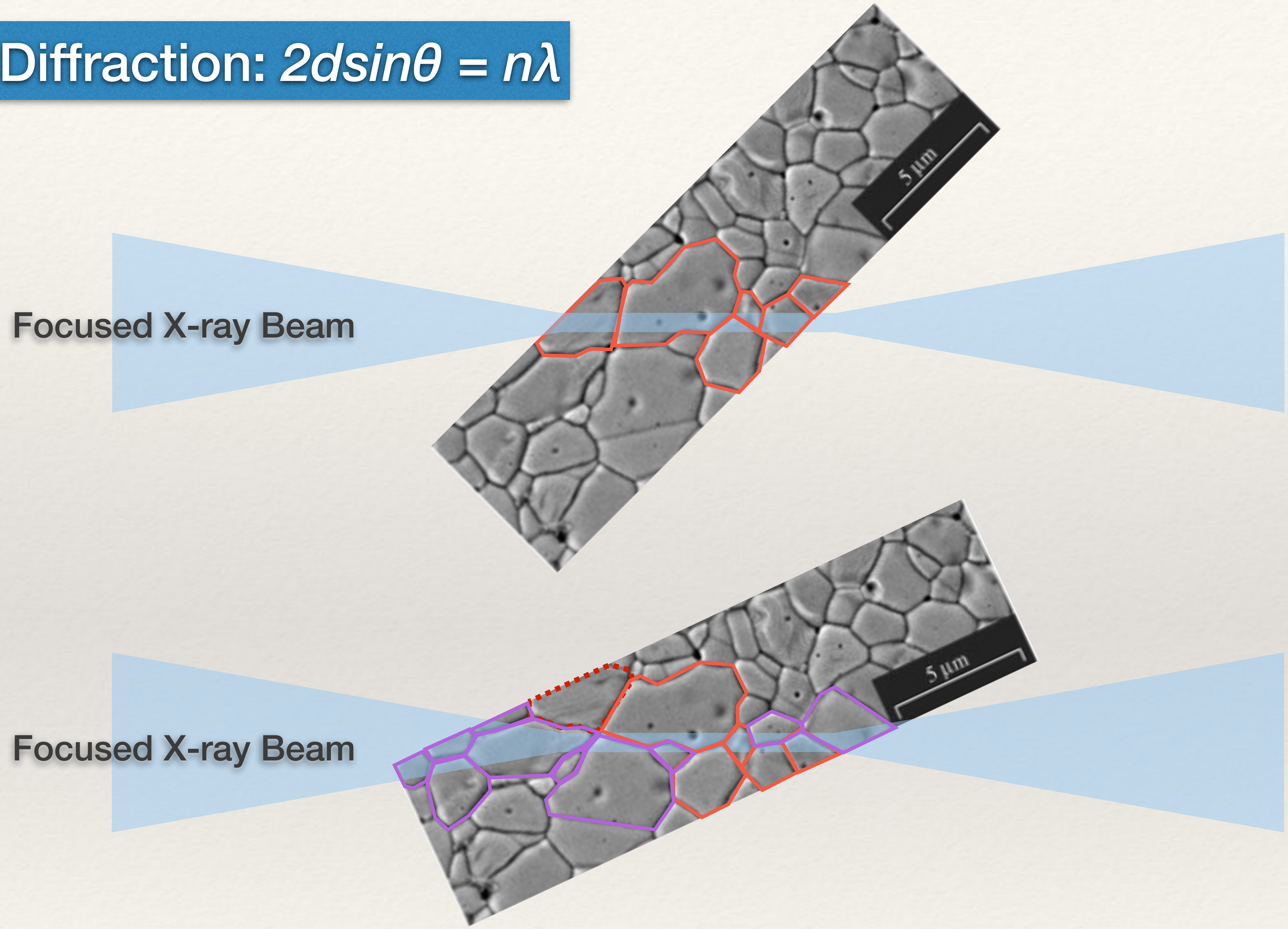
$$\varepsilon = \underbrace{\begin{pmatrix} \varepsilon_{xx}-\Delta/3 & \varepsilon_{xy} & \varepsilon_{xz} \\ \varepsilon_{xy} & \varepsilon_{yy}-\Delta/3 & \varepsilon_{yz} \\ \varepsilon_{xz} & \varepsilon_{yz} & \varepsilon_{zz}-\Delta/3 \end{pmatrix}}_{\text{deviatoric strain (distortion)}} + \underbrace{\begin{pmatrix} \Delta/3 & 0 & 0 \\ 0 & \Delta/3 & 0 \\ 0 & 0 & \Delta/3 \end{pmatrix}}_{\text{hydrostatic strain (dilatation)}}$$

B.C. Larson et al, Nature 415, 887 (2002)

Lyle E. Levine et al, Nature Materials 5, 619 (2006)

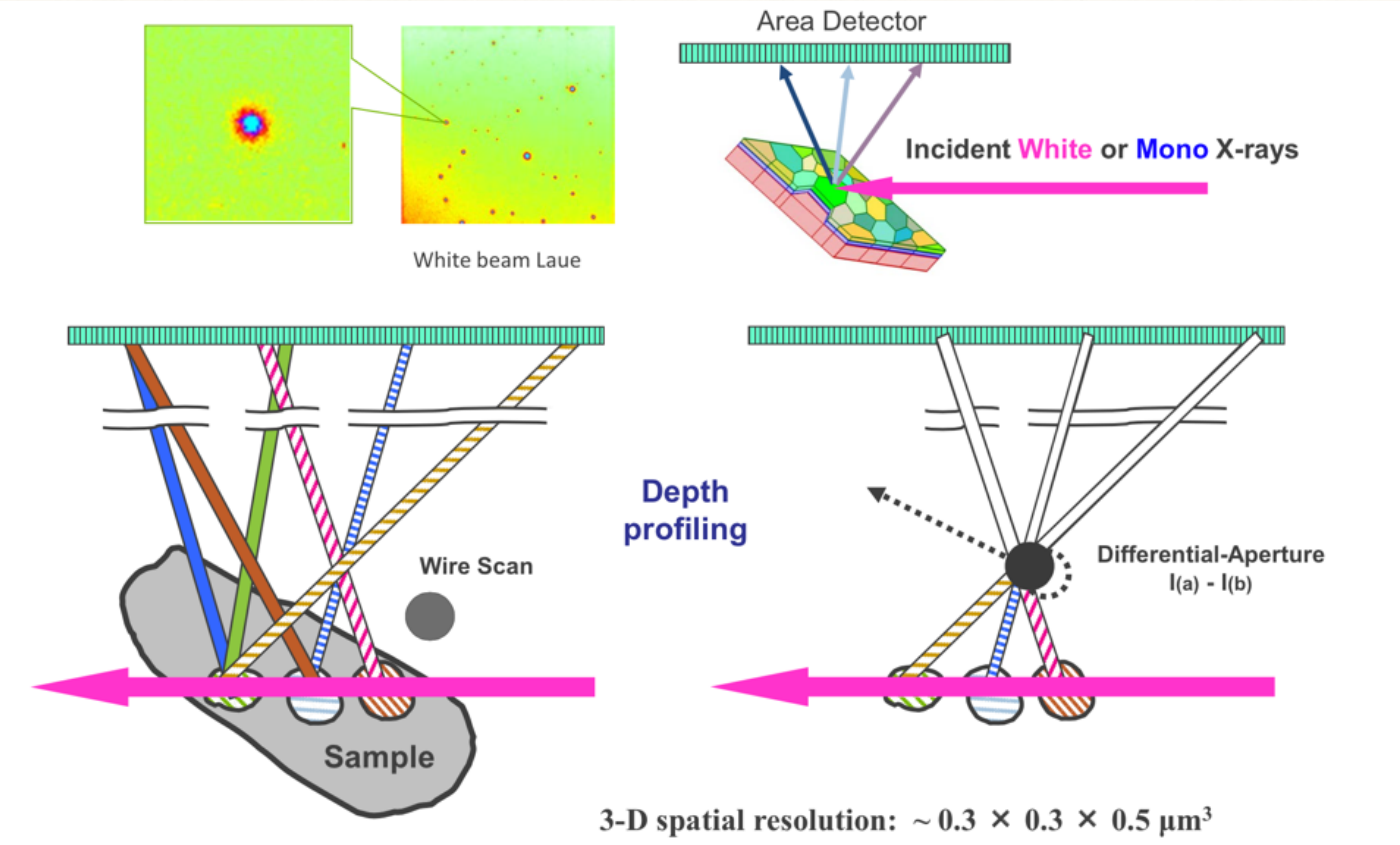
Why Laue for Nano-Diffraction?

Bragg Diffraction: $2d\sin\theta = n\lambda$



Rotation in Bragg Diffraction will Caused the X-ray Beam Probing at Different Sample Region.

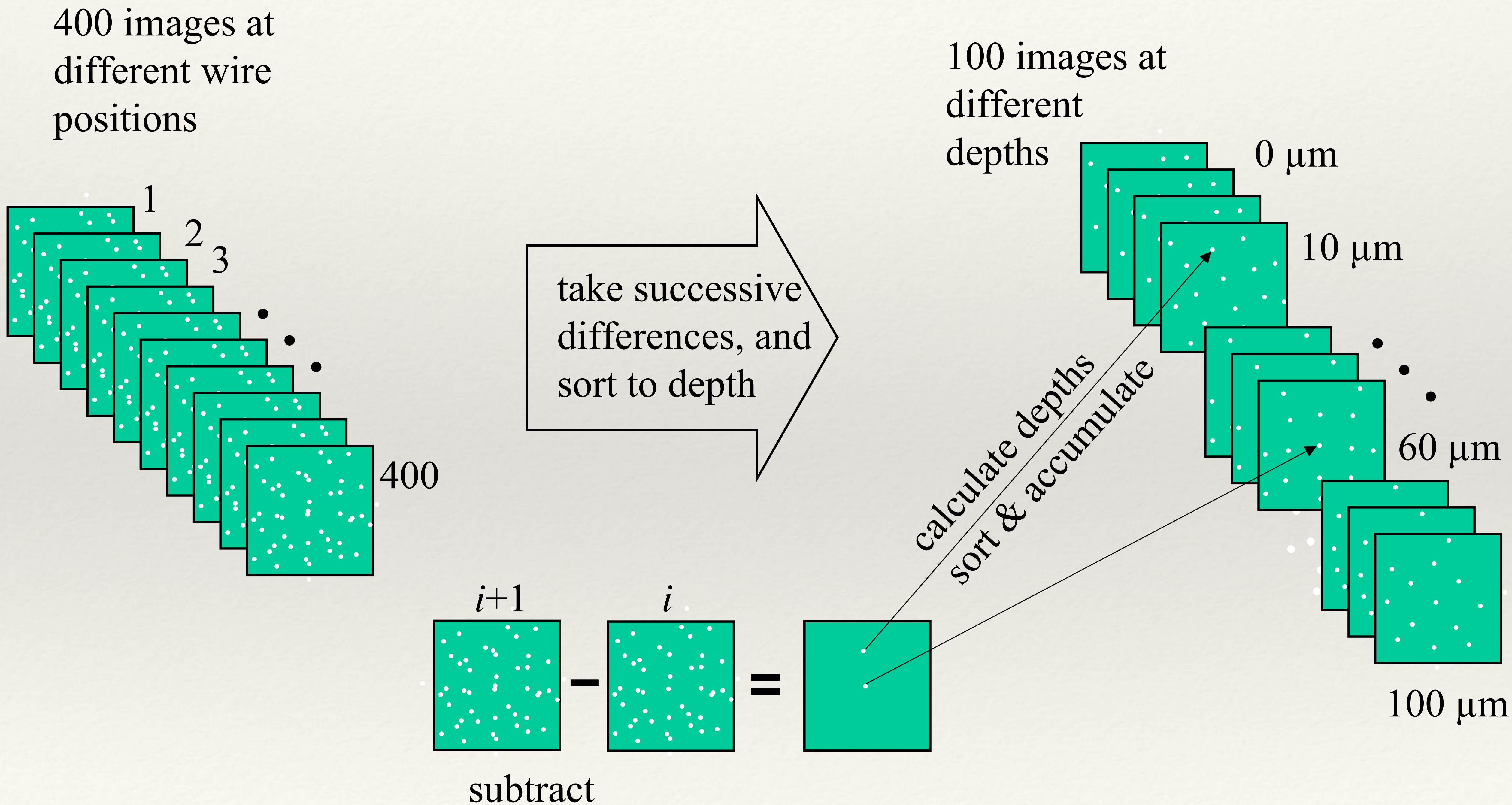
3D Laue Diffraction (DAXM)



Courtesy Dr. Wenjun Liu, APS

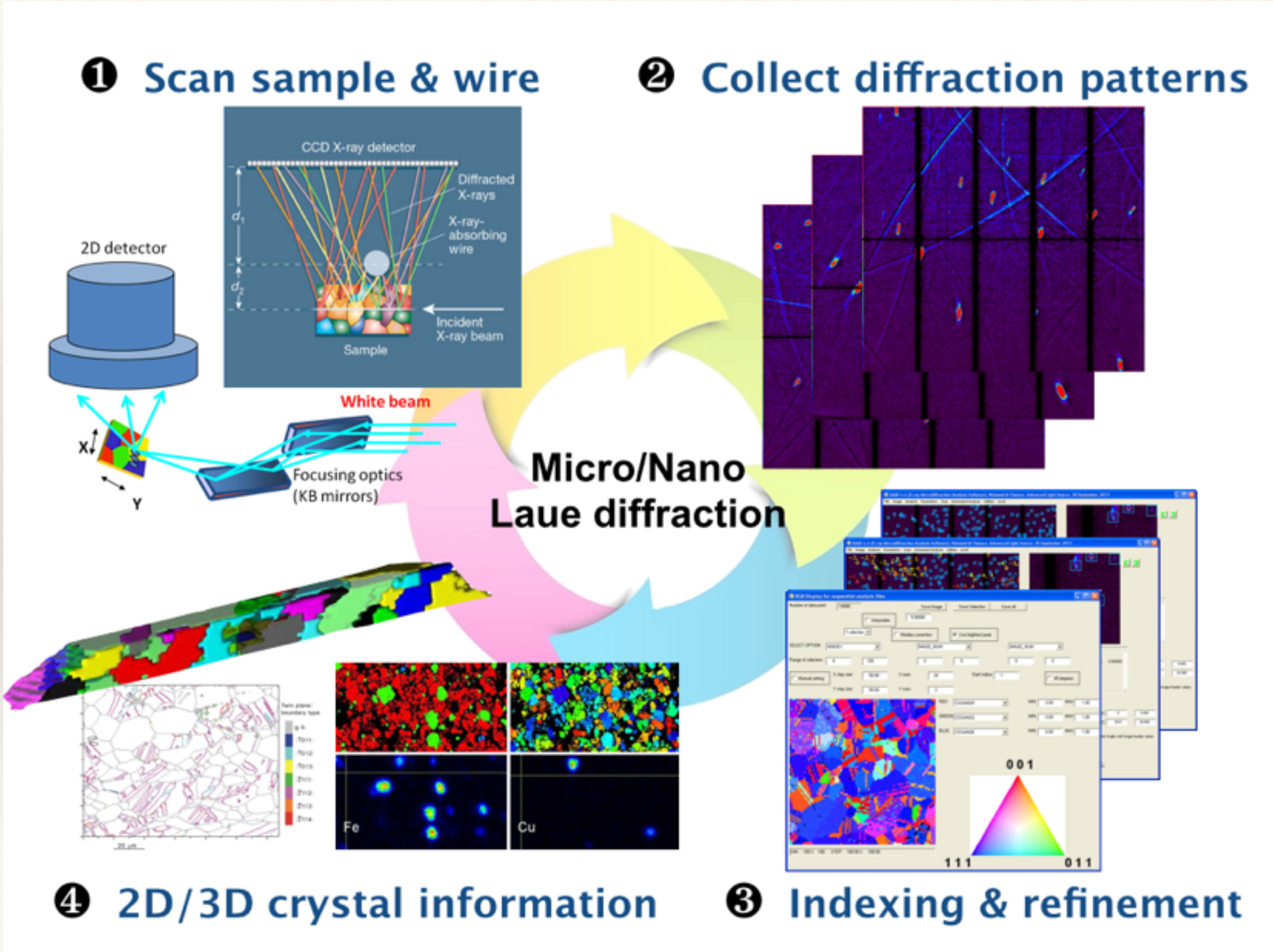
3D Image Reconstruction

Image Reconstruction

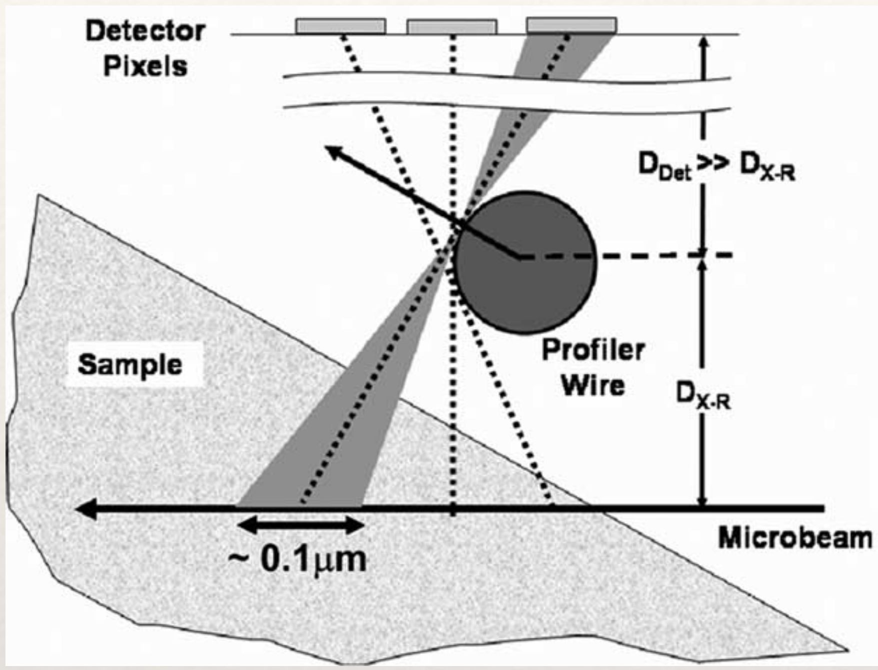


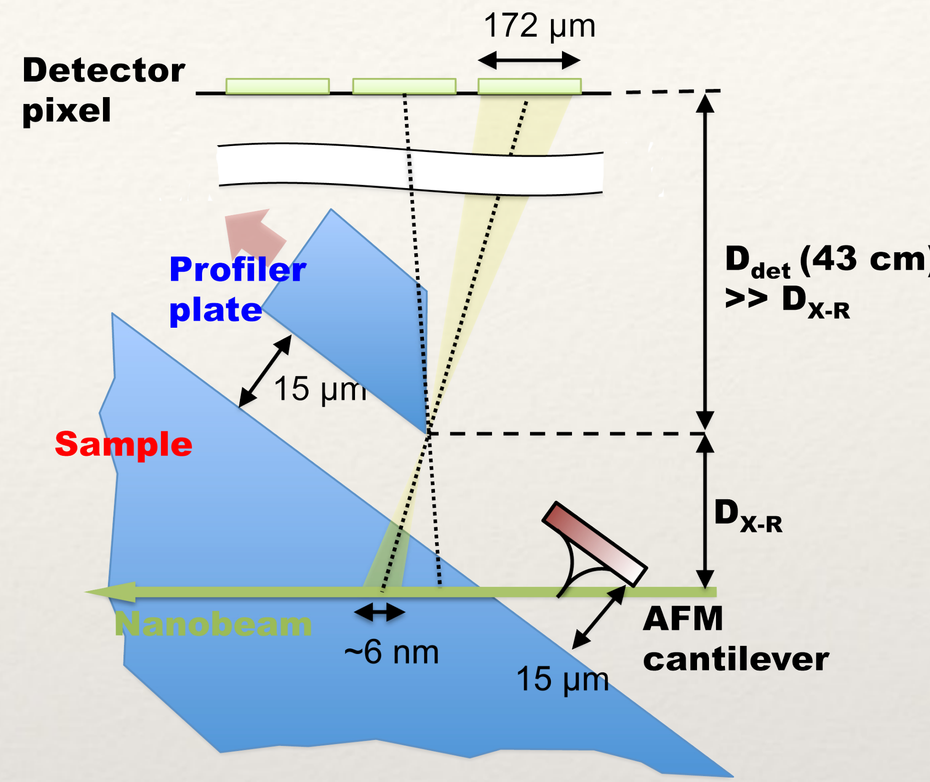
Courtesy Dr. Wenjun Liu, APS

Data Processing



Depth Resolution : 40 nm





Wire-scan parameter: (APS)
1. sample surface to wire: 200 um.
2. wire dimension: 50 um
3. scan step: 0.45 um (min.)
4. Detector pixel size: 200 um
5. Detector to Sample: 50 cm

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

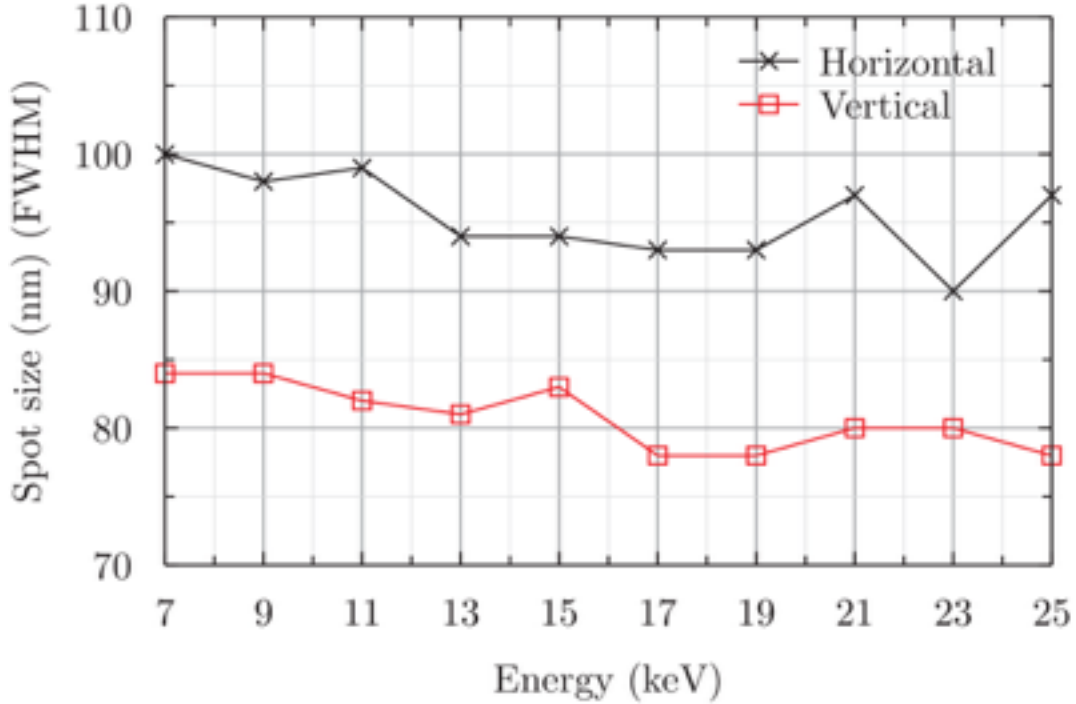
We further reduce the parallax contribution from 100 nm down to 6 nm by using SPM feedback technology

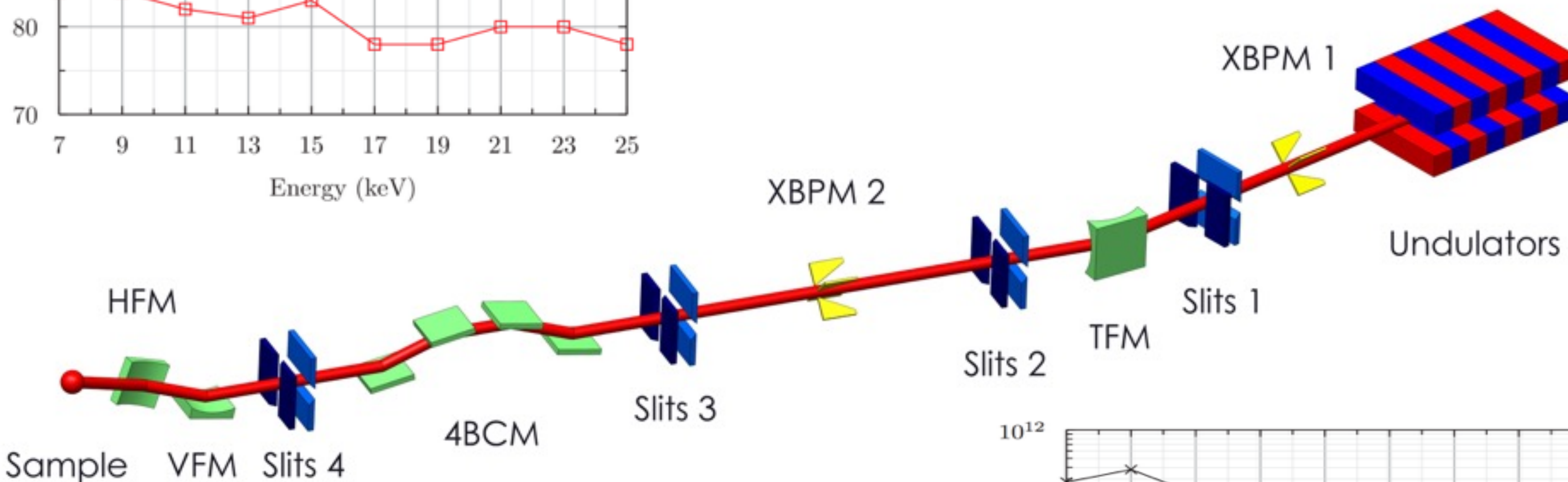
Wire-scan parameter: (TPS)
1. sample surface to wire: 15 um.
2. plate dimension: 50 um
3. scan step: 0.0005 um (min.)
4. Detector pixel size: 172 um
5. Detector to Sample: 43~67 cm

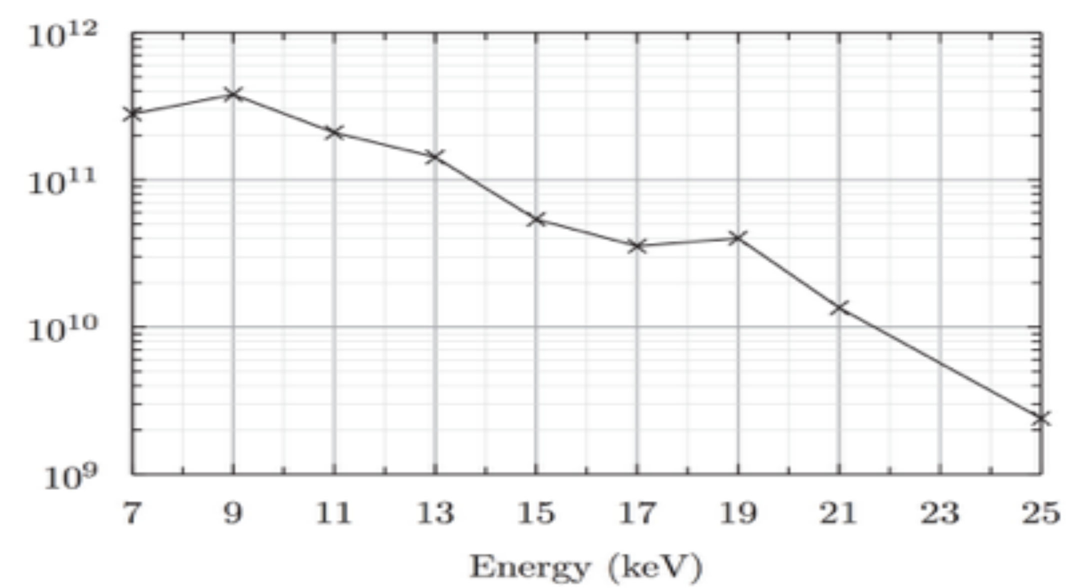
Depth Resolution:
50/1.414+6~41 nm (50 nm step, coarse-mode)

W. Yang et. al. Micron 35, 431 (2004)

Beamline Design





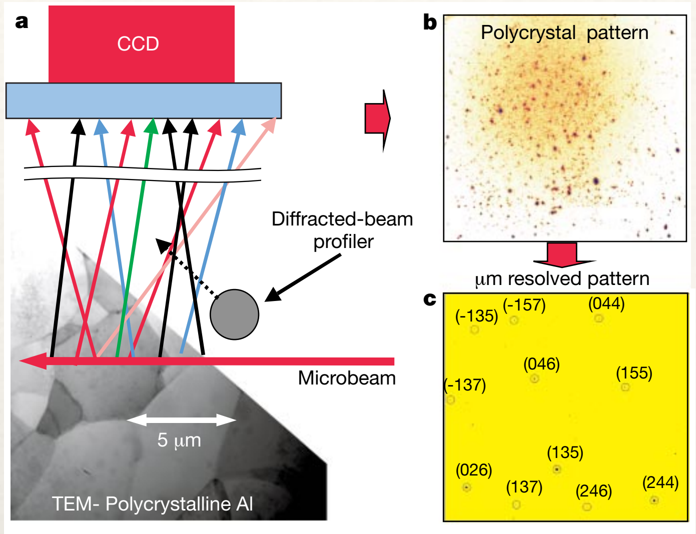


~100 x 100 x 50 nm^3 for 21A

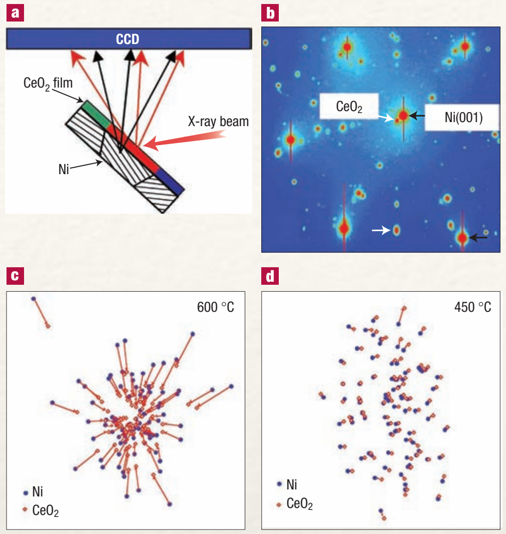
Specifications

- Energy Range: 7 - 25 keV (mono-beam); 5 - 30 keV (pink-beam)
- Photon Flux: 3×10^{11} photon/sec (7 keV); 3×10^{9} photon/sec (25 keV); > 1×10^{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 ~ 1×10^{-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

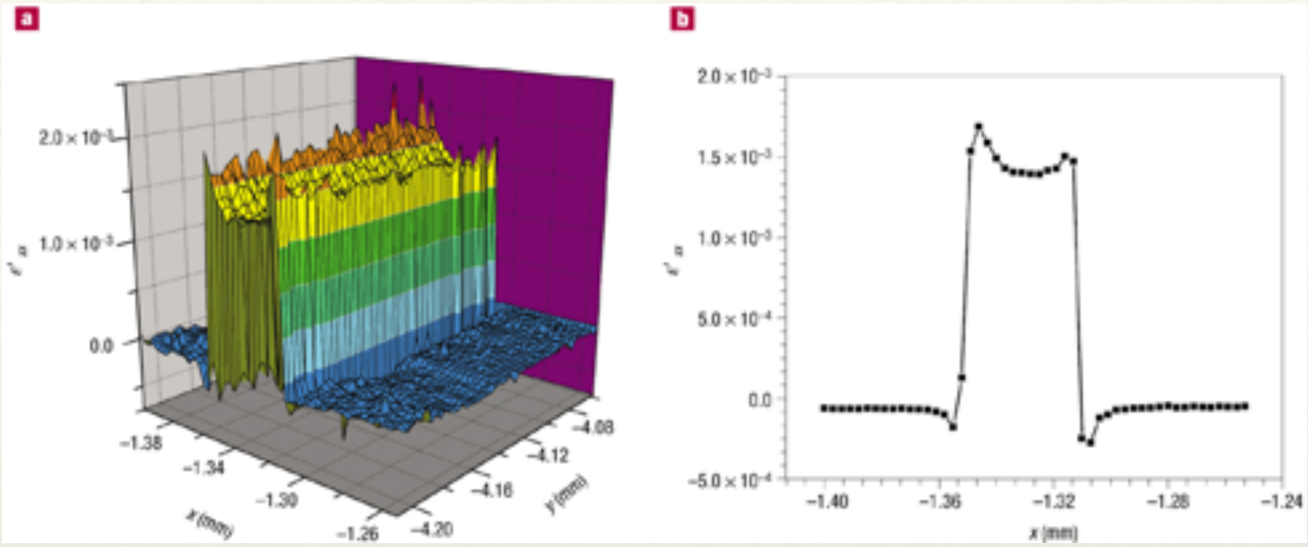
Scientific Opportunities



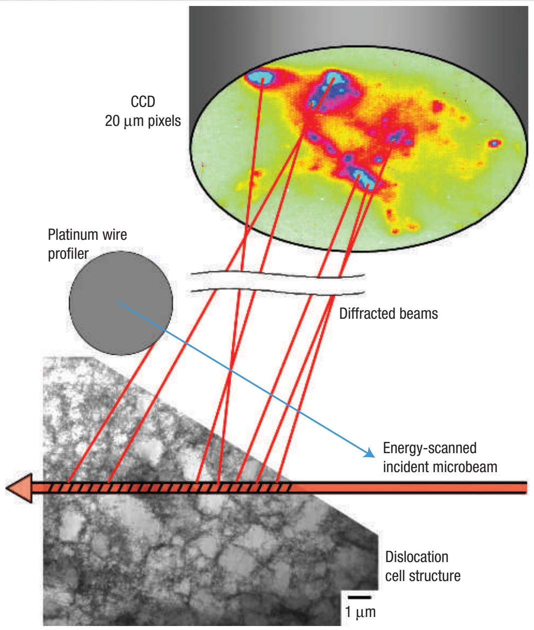
Bennett C. Larson *et al.*
Nature 415, 887 (2002)



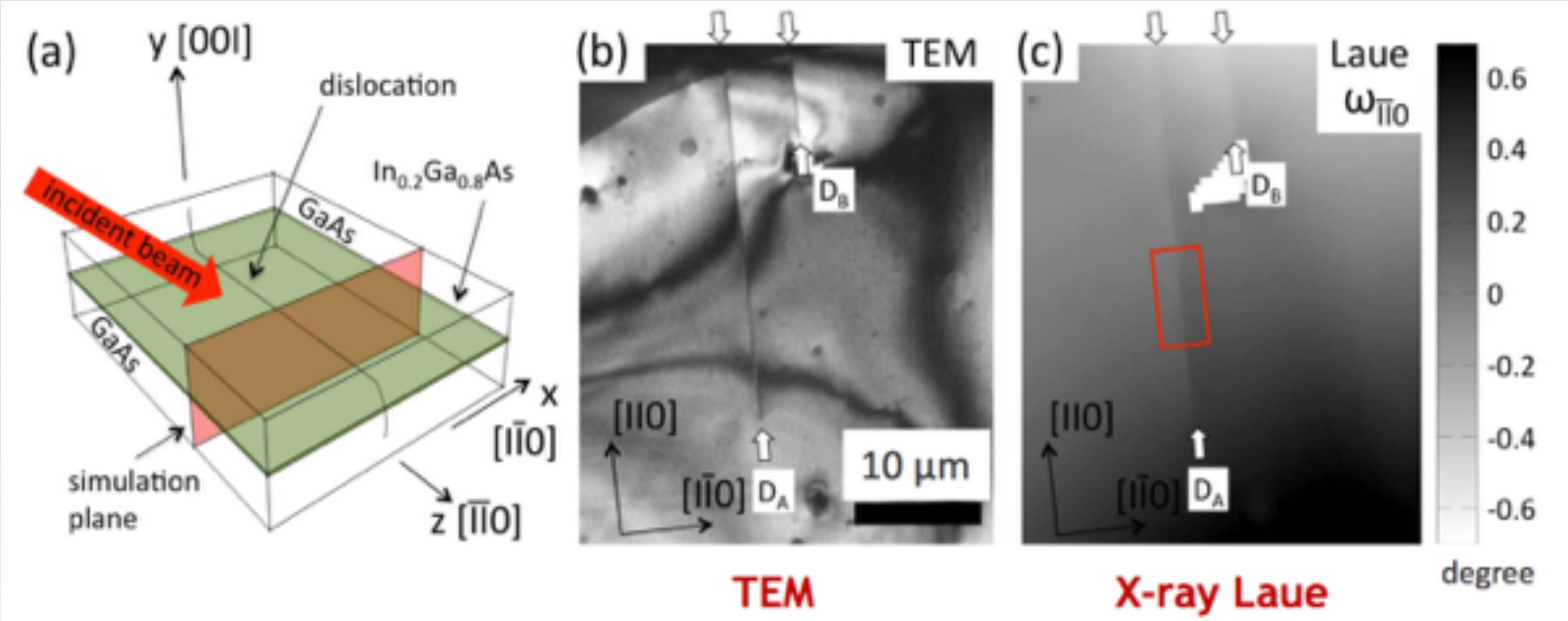
Robert C. Rogan *et al.*
Nature Materials 2, 379 (2003)



John D. Budai *et al.*
Nature Materials 2, 487 (2003)



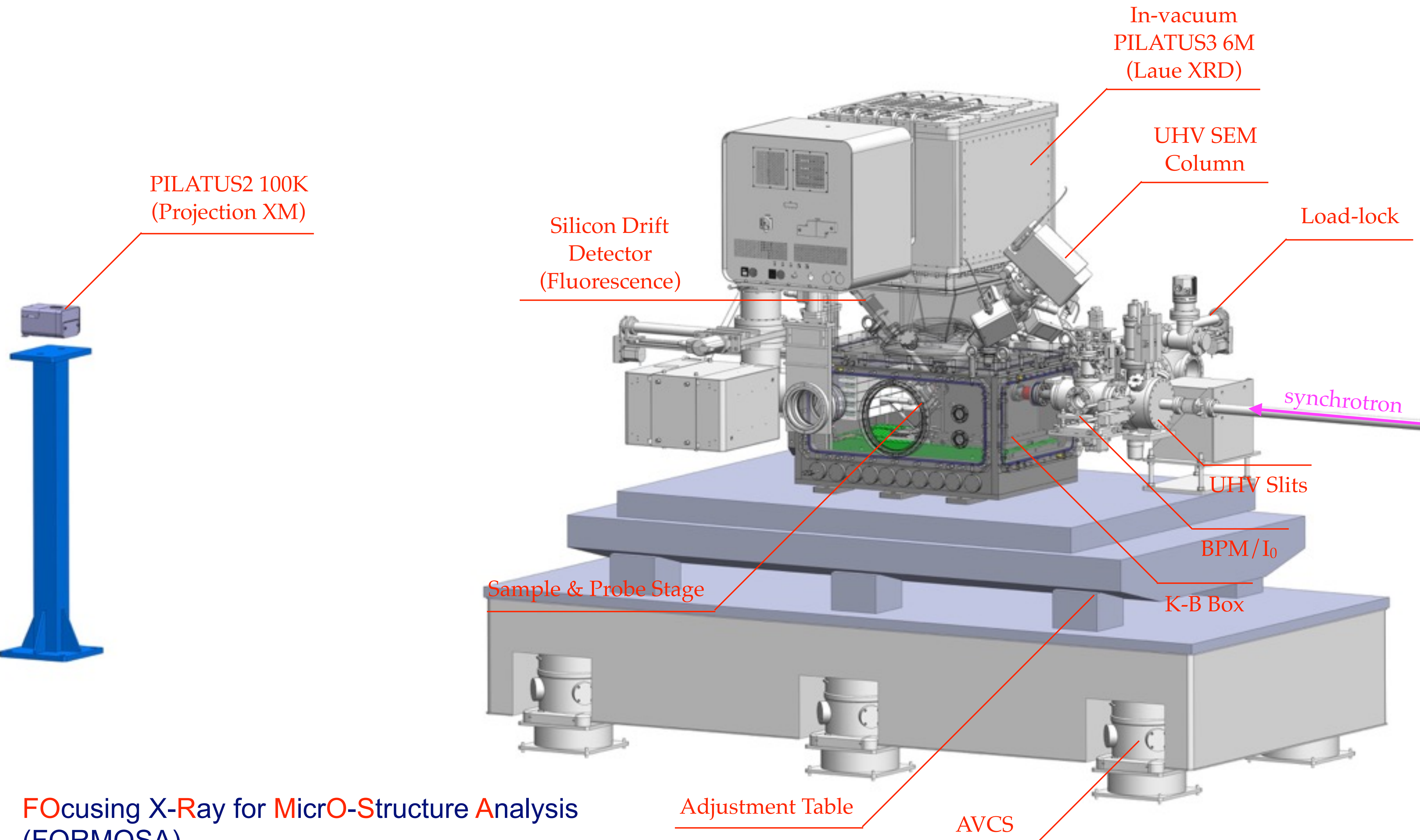
Lyle E. Levine *et al.*
Nature Materials 5, 619 (2006)



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

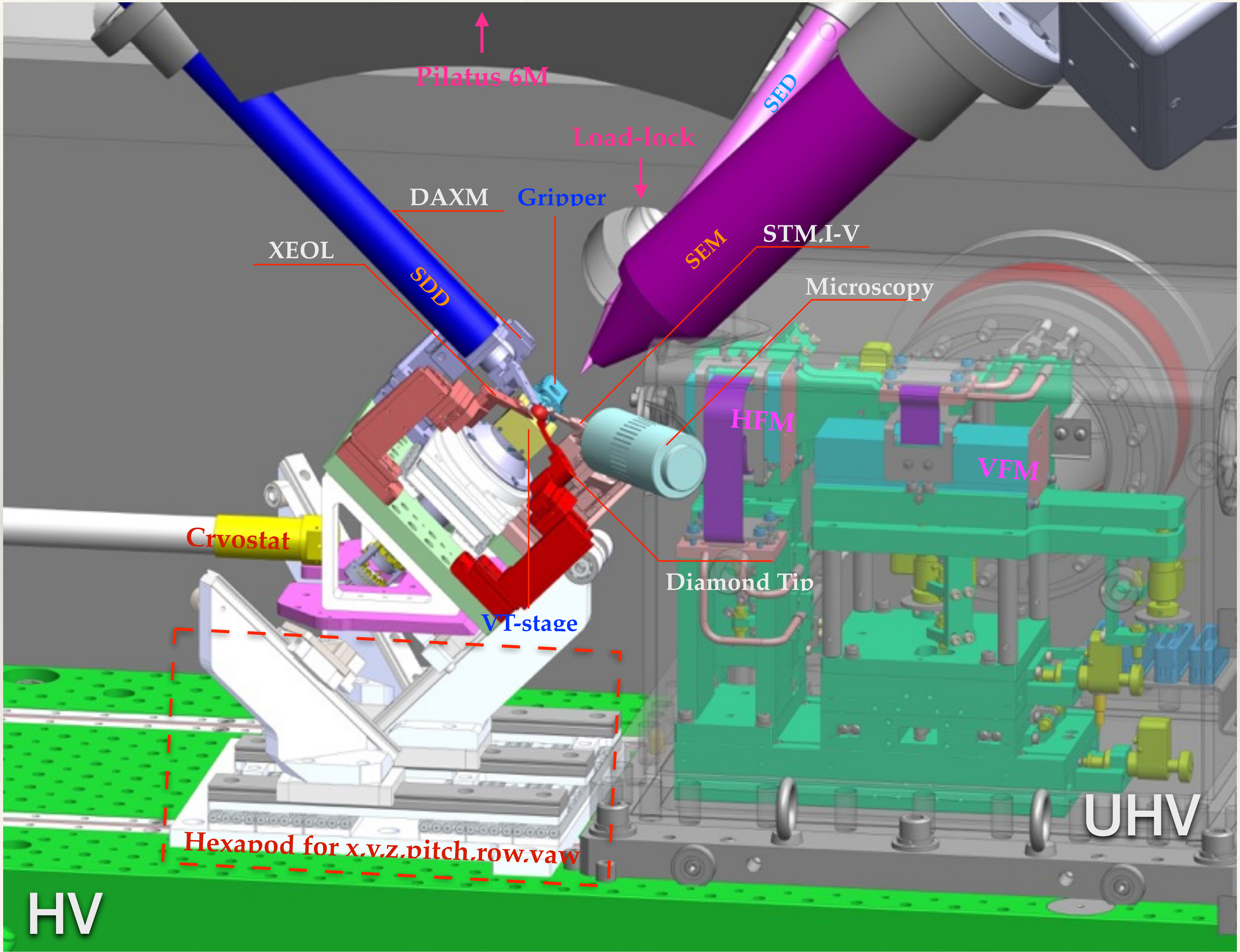
Most of them are ex-situ, How about in-situ…?

Layout of End-station



FOcusing X-Ray for MicrO-Structure Analysis (FORMOSA)

Inside the Chamber



The "In-vacuum" PILATUS3-6M detector


DECTRIS®

detecting the future

Technical Specifications and Operating Procedure

PILATUS3 S 6M-NSRRC Detector System

Version: V1.0

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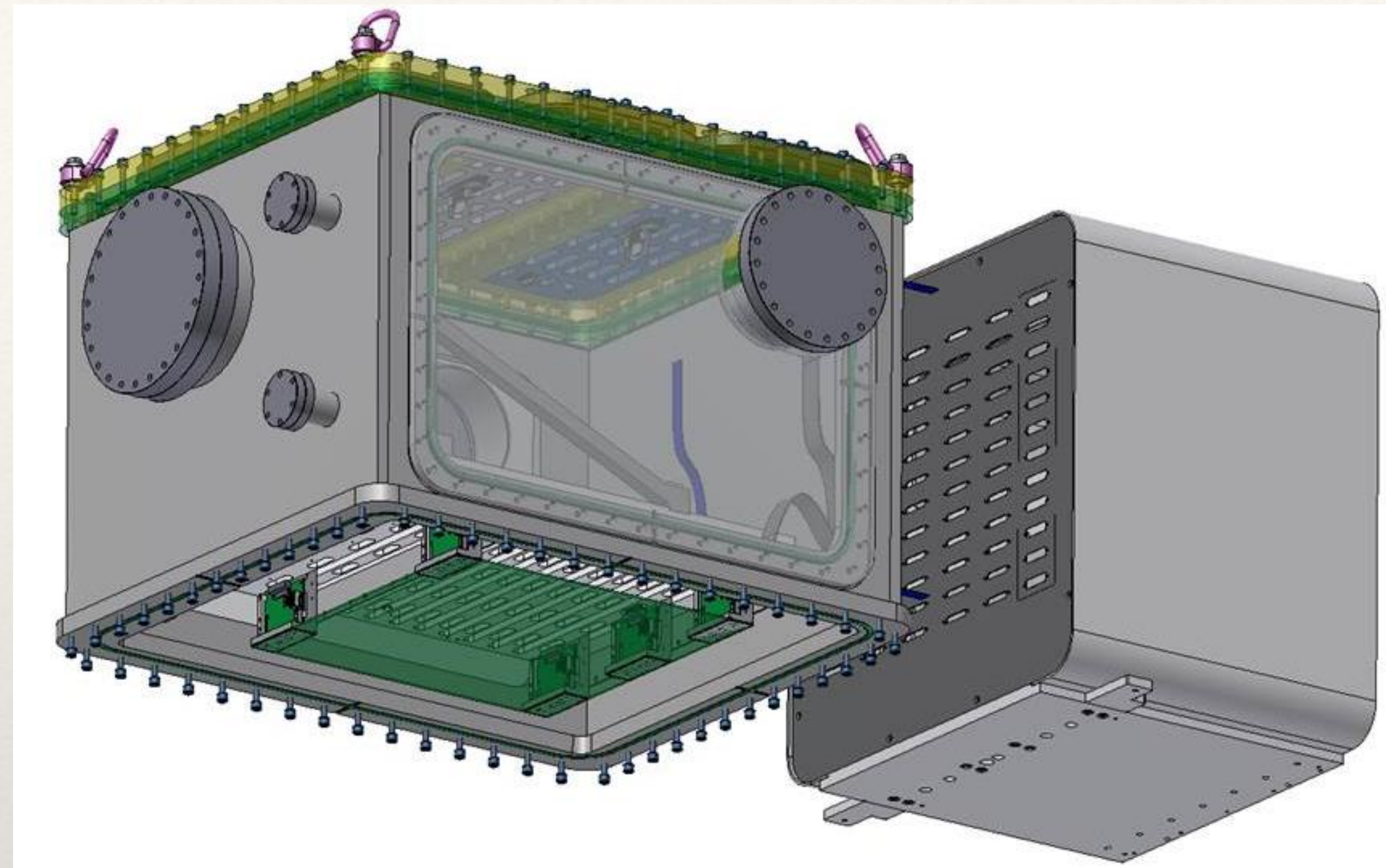
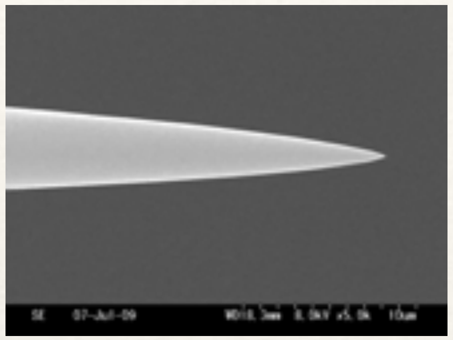
www.dectris.com



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

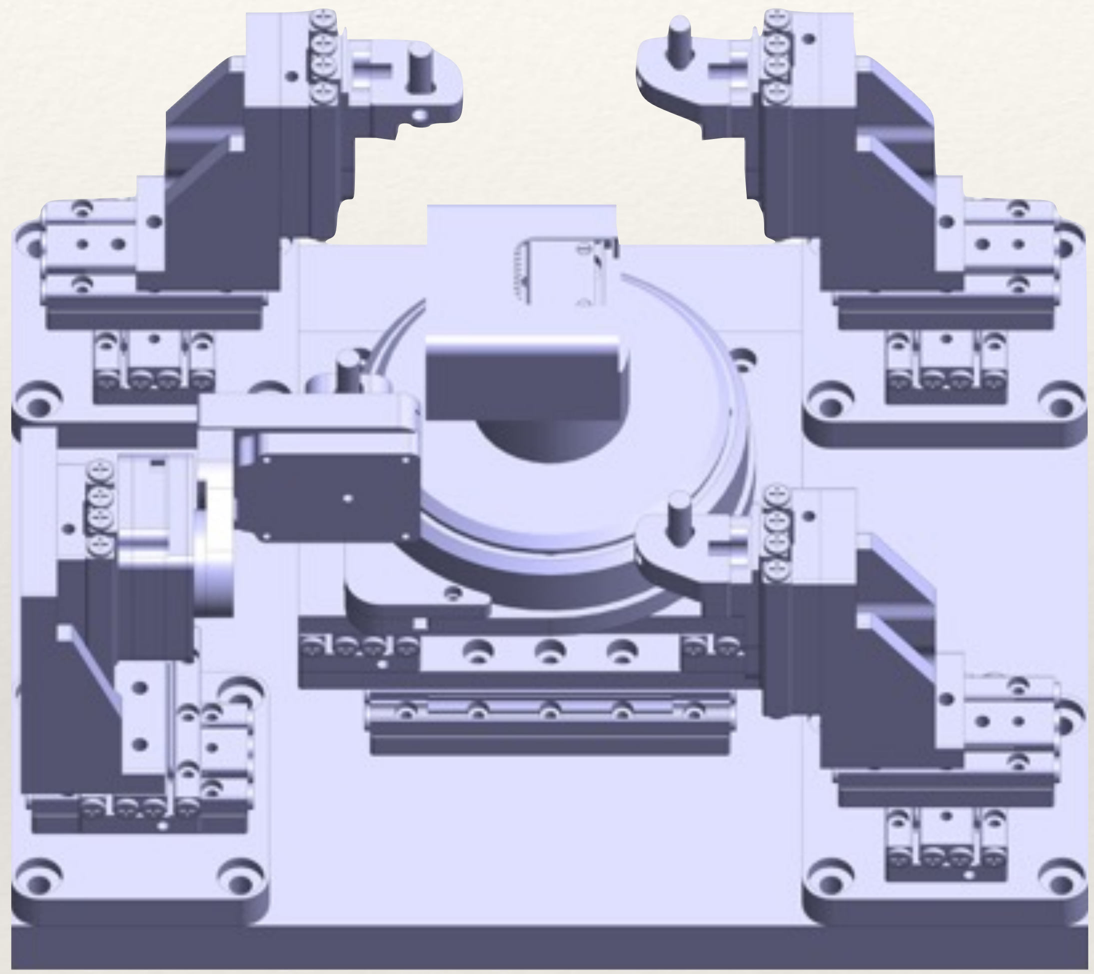
Quadruprobe System

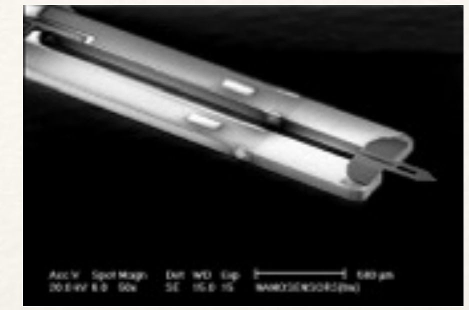


Nickel probe
(Unisoku, STM/I-V)

DAXM profiler

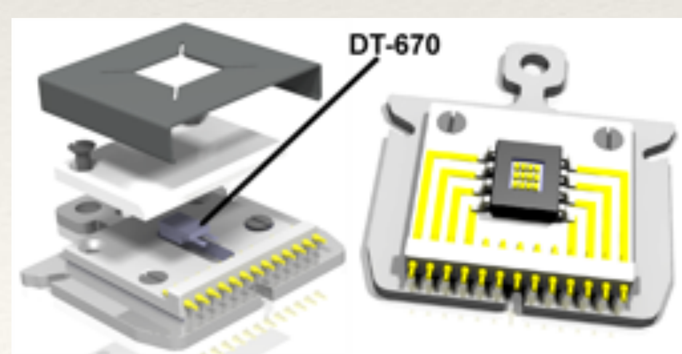
Indenter tip



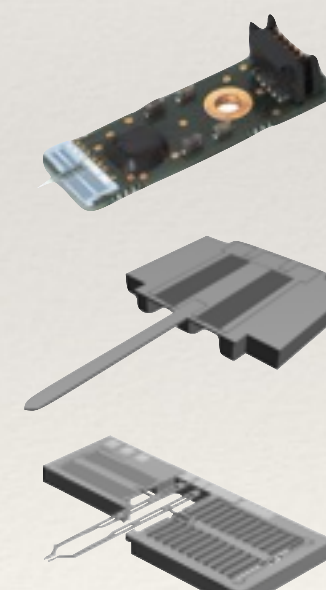


Akiyama probe
(Nanosensors, AFM)

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

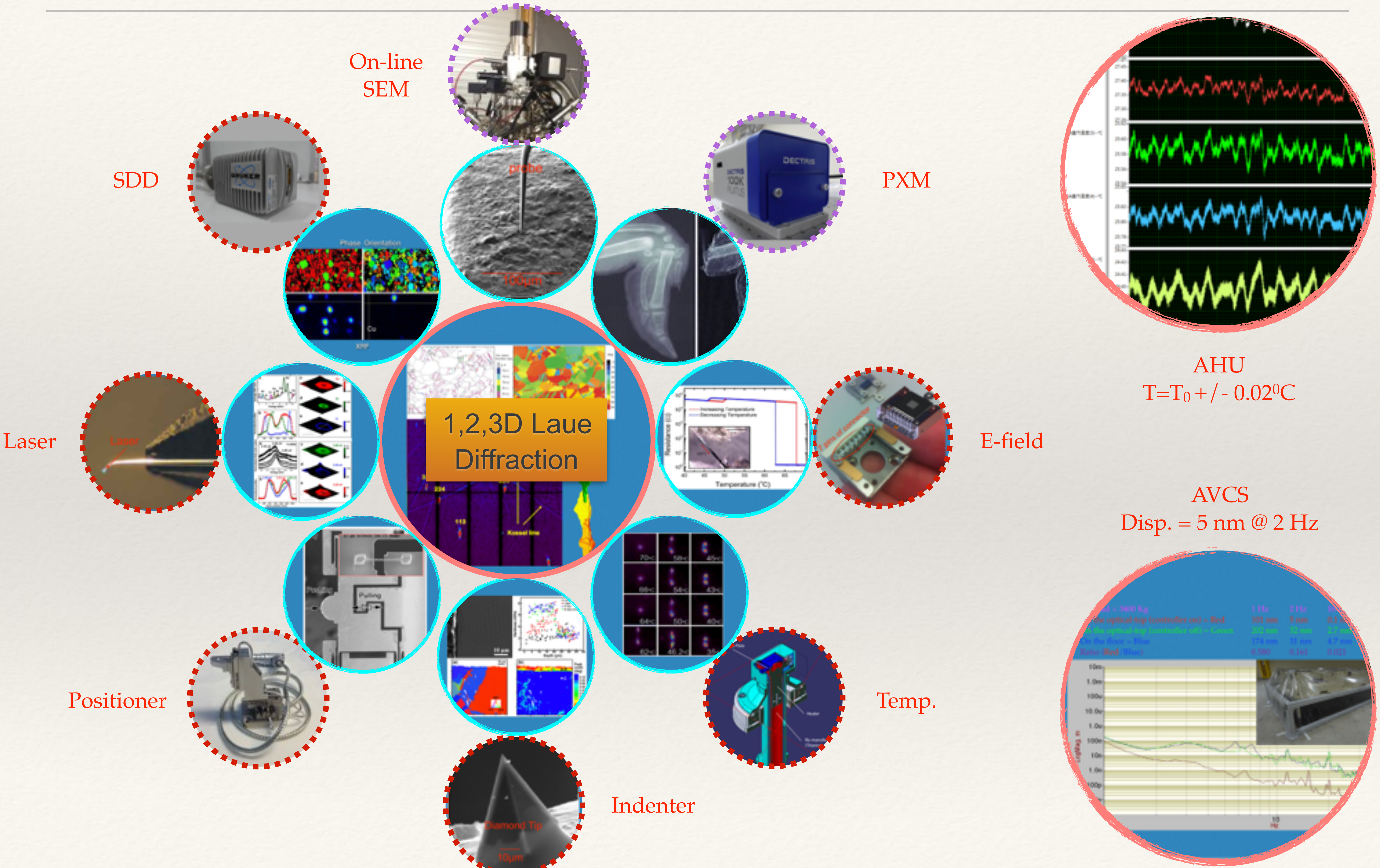


Sample Holder

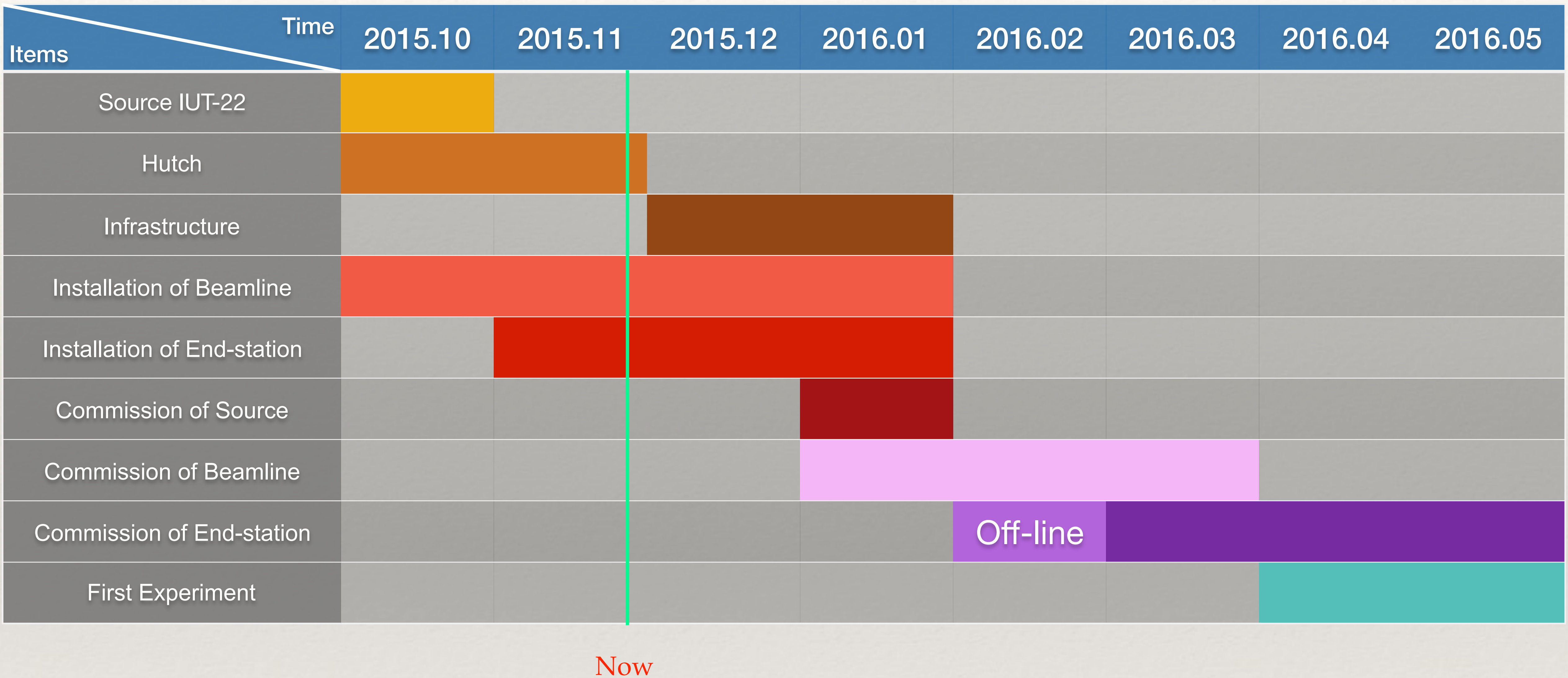


Micromechanical probe
(microgripper, microsening)

3D + 1D = Future of Materials Science



Schedule



Functionalities of End-station

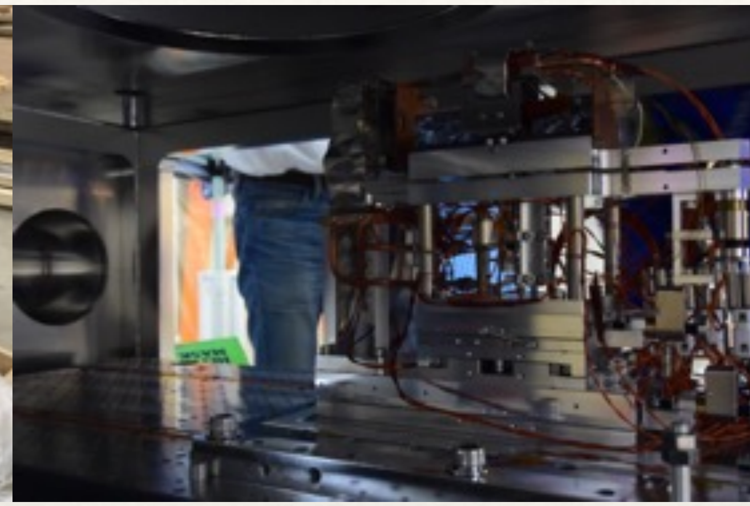
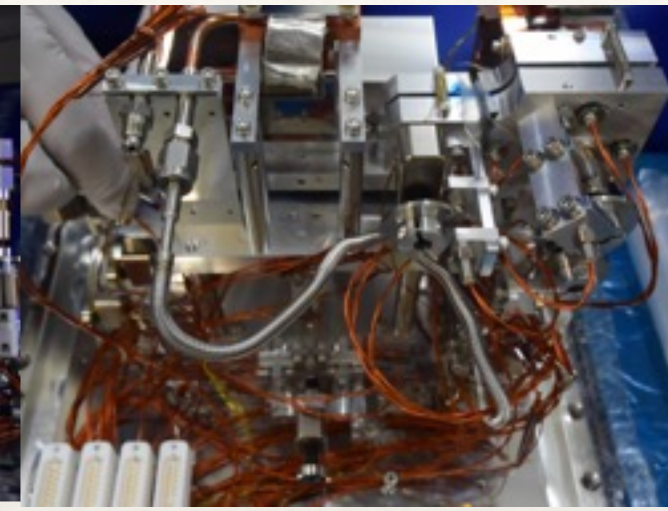
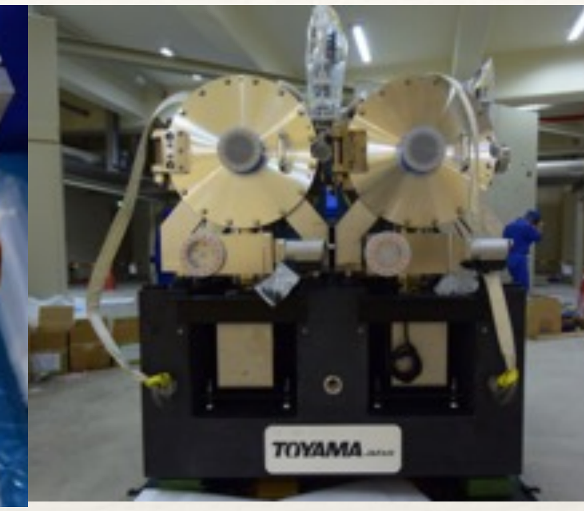
1st-stage (2016.03~08)

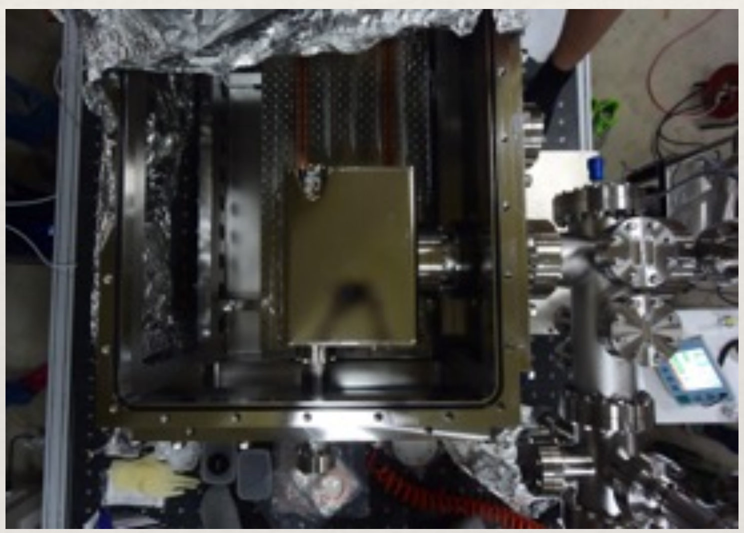
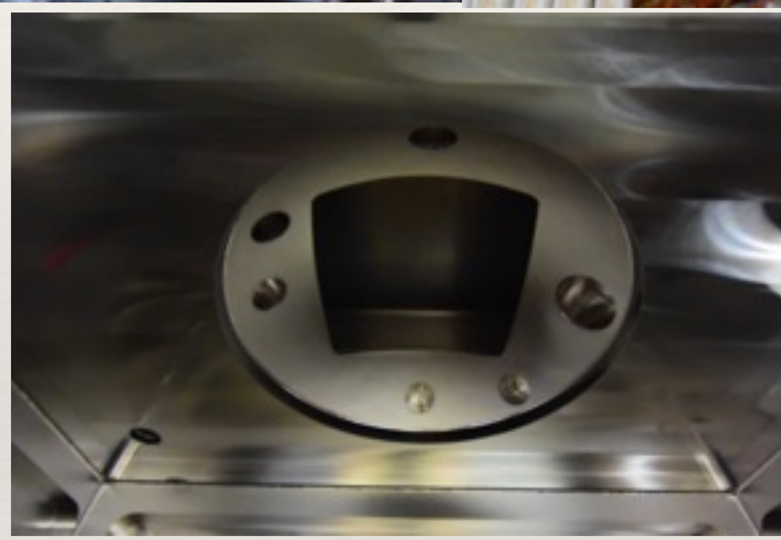
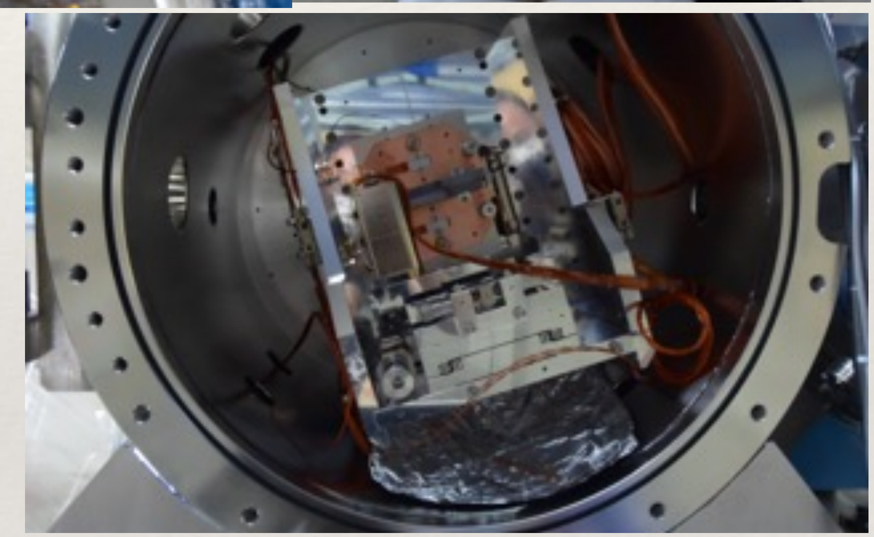
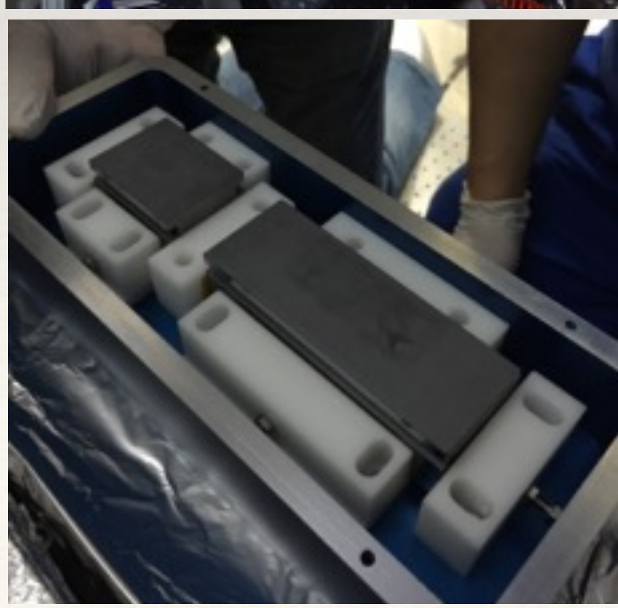
1. 2D Laue Diffraction Image
2. 2D Fluorescence Image
3. PXM
4. VT Experiments (RT to 1,000K)
5. In-situ E-field & Force
6. SEM navigation + EDS

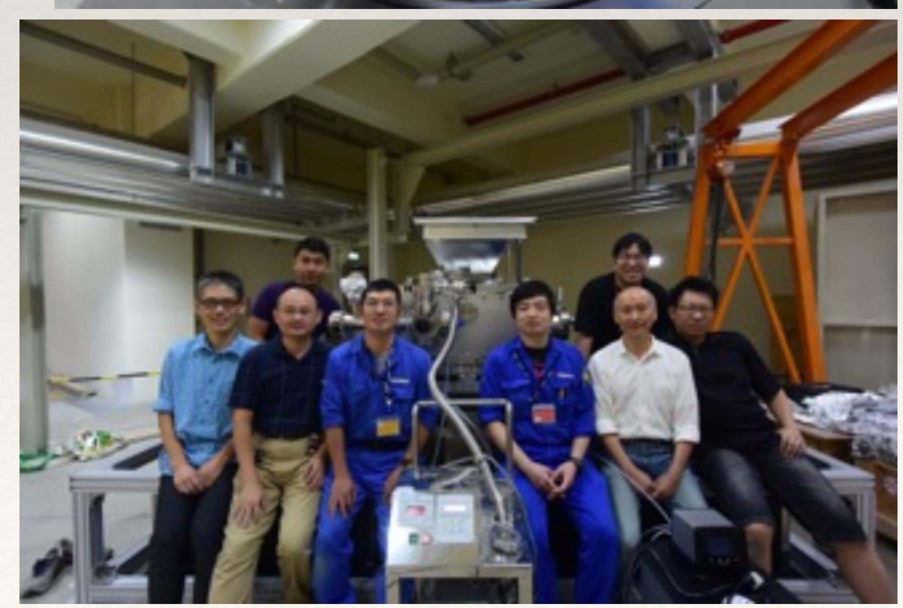
2nd-stage (2016.08~)

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

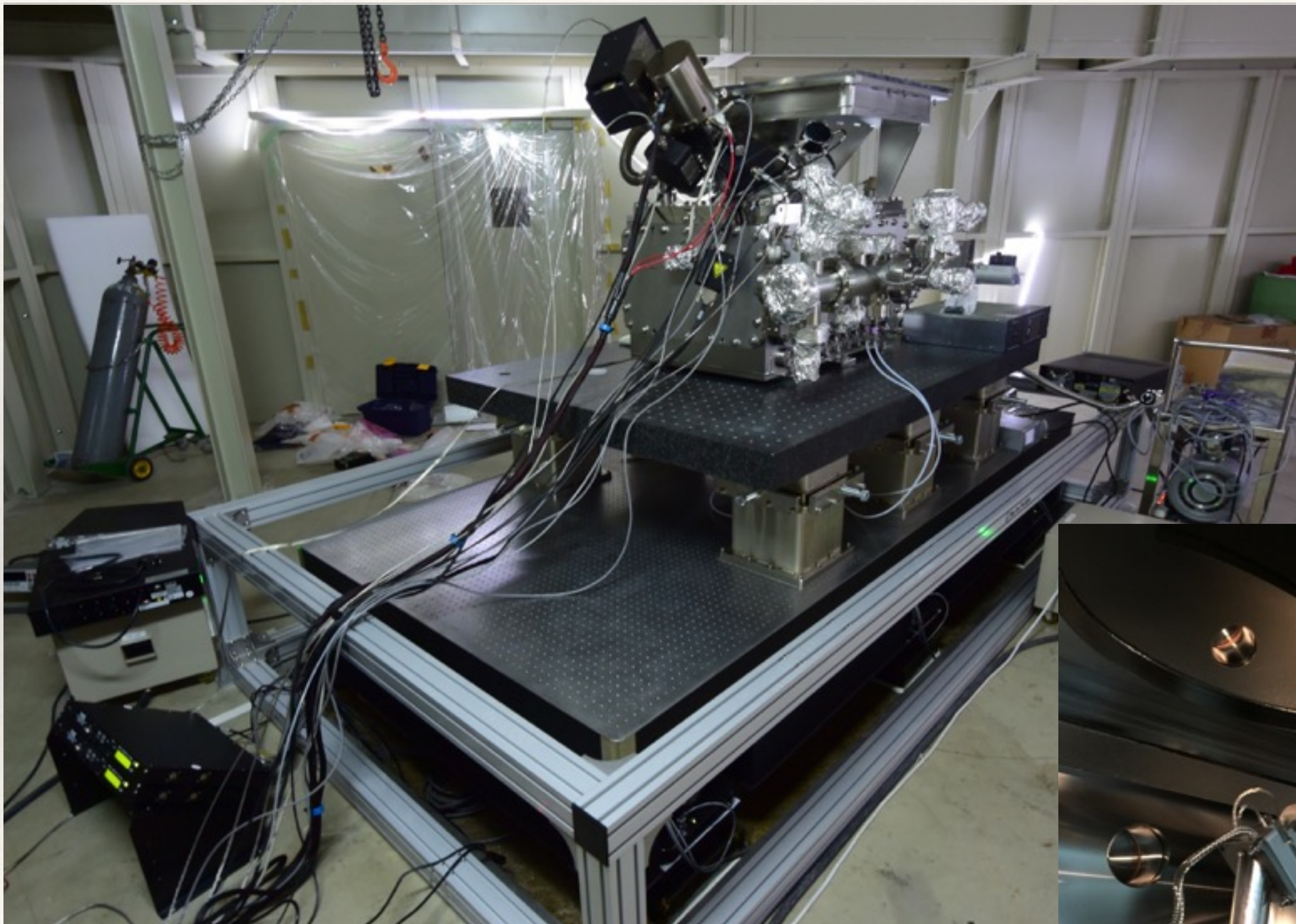





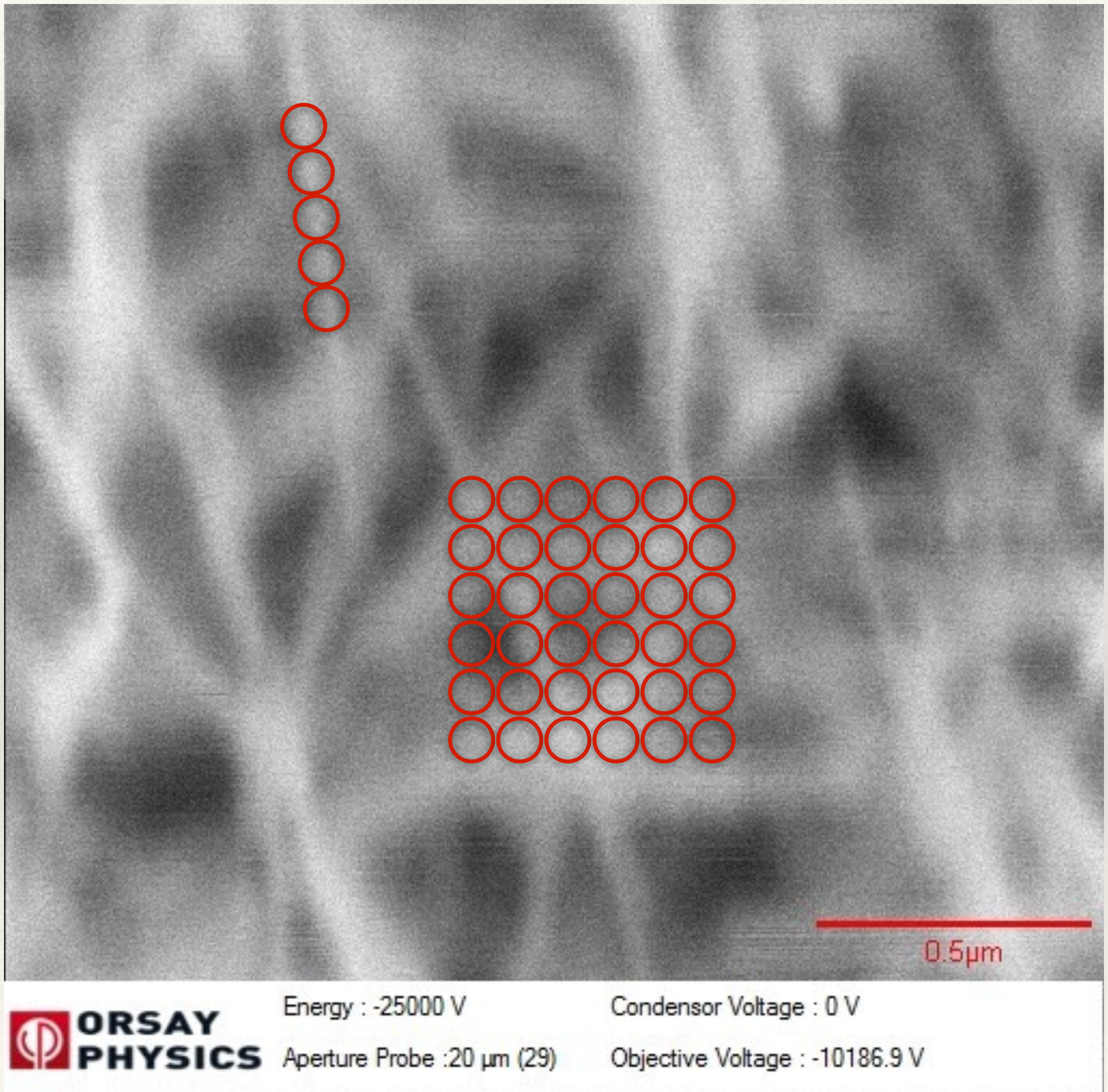


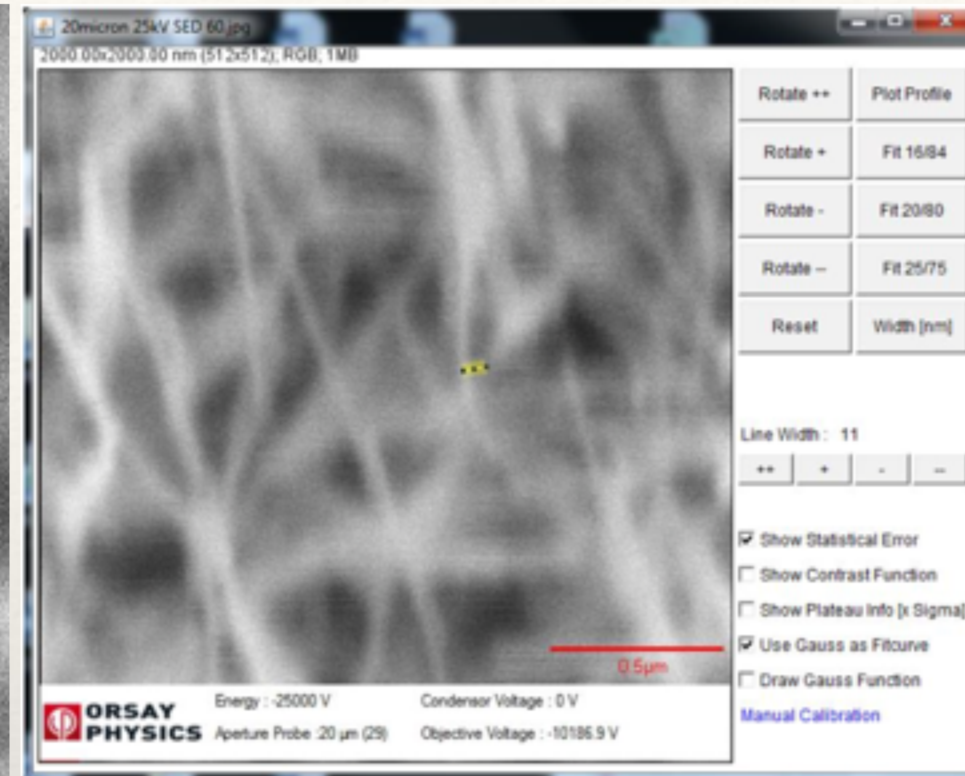
Installation of SEM, TFM, BPM, Beamline Components

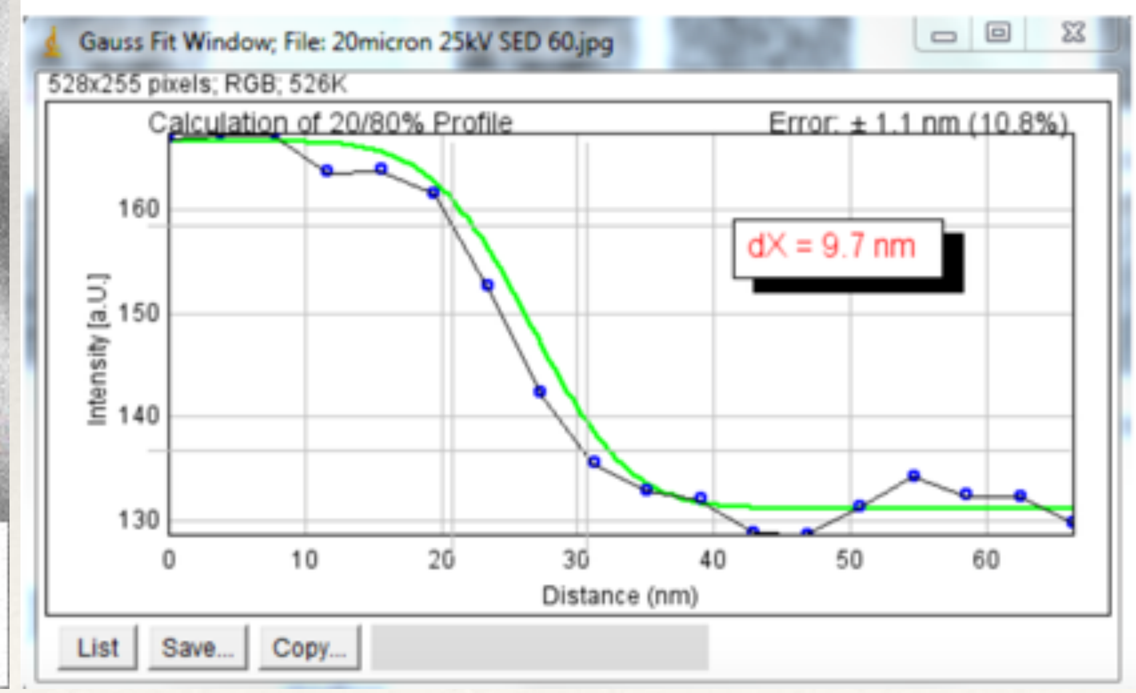
First results from on-line SEM



First results from on-line SEM







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https://www.facebook.com/groups/submicron/

History & Project Team

2009-2010
- LOI for Sub-μ
- DAXM technology
- 1st, 2nd Sub-μ Workshop

2011
- CDR for Sub-μ
- SPM combined DAXM
- SEM navigation
- Workshop for phase-I Beamline

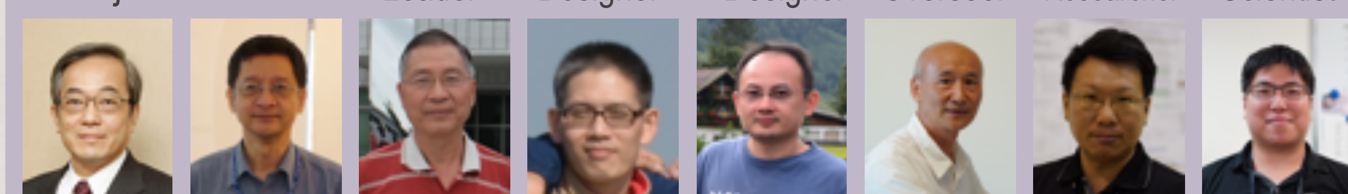
2012
- CDR v.2 to SAC
- Concept for Vacuum Operation
- Announce at SRI 2012

2013
- PDR & DDR to SAC
- 2-stage focus beamline
- Core-user meeting
- Special SEM Develop.
- Fackbook vs. User

2014
- Progress Report (SAC)
- HV PILATUS3 Det.
- 4-probe manipulator
- VT-IV sample stage
- Finial Design of ES

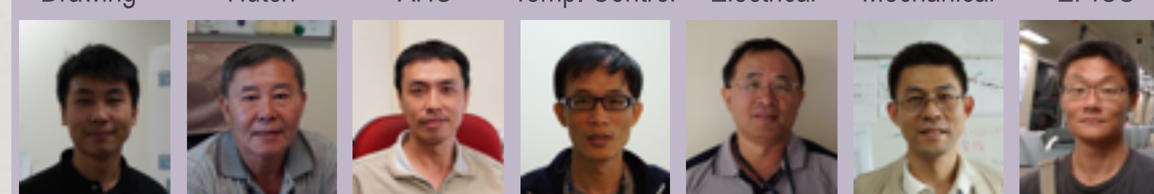
2015~
- SAC 2015
- Announce at SRI 2015
- BL & ES commission
- Commissioning

Construction Team



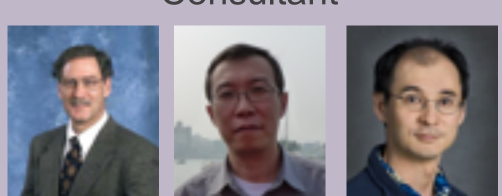
Project Leader	Project Leader	Construction Leader	Beamline Designer	End-station Designer	Overseer	THU Joint Researcher	Scientist
Haydn Chen (2010-2013)	I-K Yang (2013-)	H. Y. Lee	C. Y. Huang	C. S. Ku	C. C. Chen	L. Lee	S. J. Chiu

NSRRC Support Team



Drawing	Hutch	AHU	Temp. Control	Electrical	Mechanical	EPICS
H. Y. Yan	J. M Juang	Z. D. Tsai	C. C. Chiu	C. Y. Liu	C. H. Chang	H. Y. Chen

Consultant

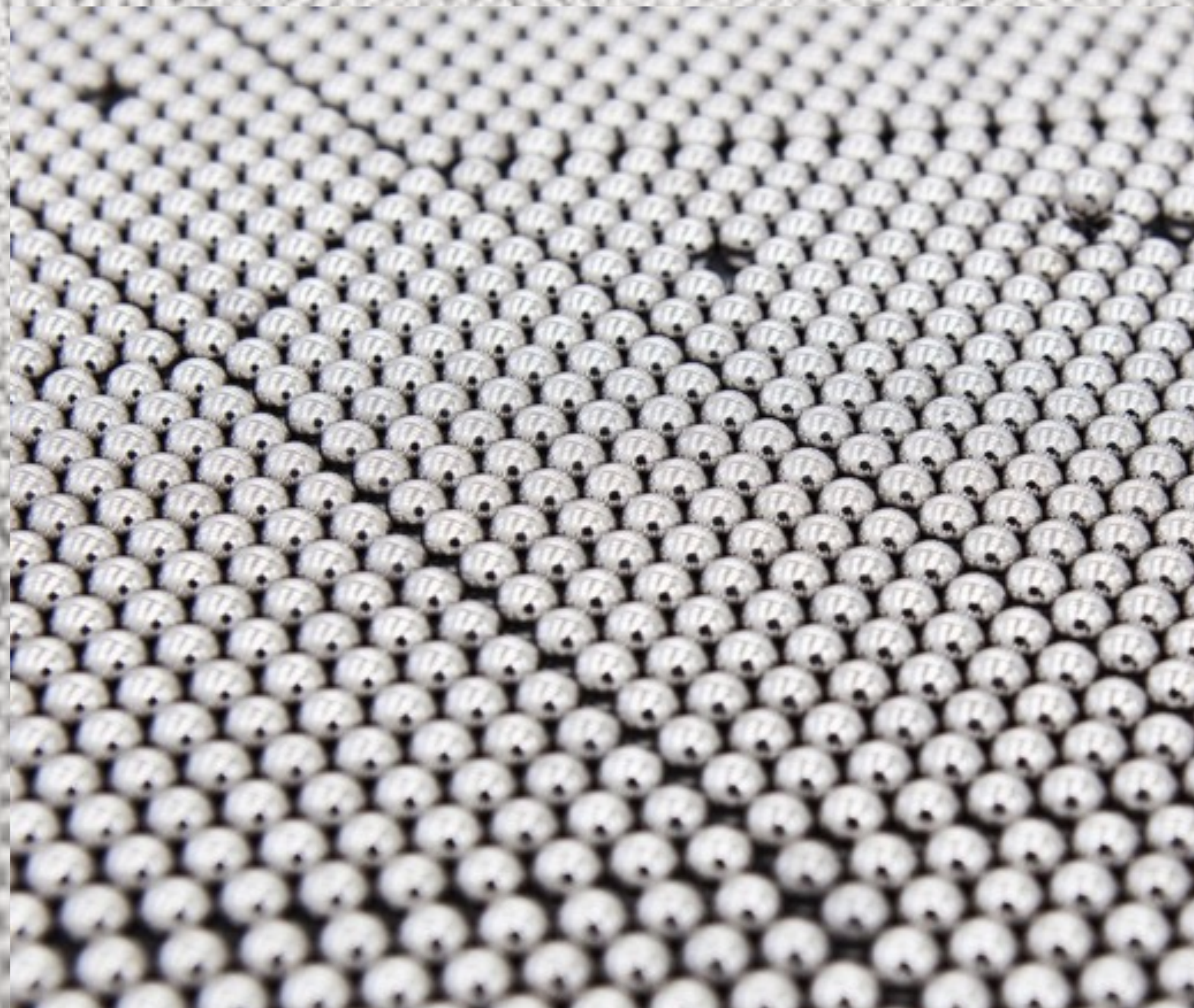


G. E. Ice (Oak Ridge), W. J. Liu (APS), N. Tamura (ALS)

Core-Users

W. C. Chou, Kai Chen, J. L. Huang, J. H. Huang, E. W. Huang, C. E. Ho, K. Y. Lo, David Mao, Y. D. Wang, Albert T. Wu

Thanks for your kind attention!



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