

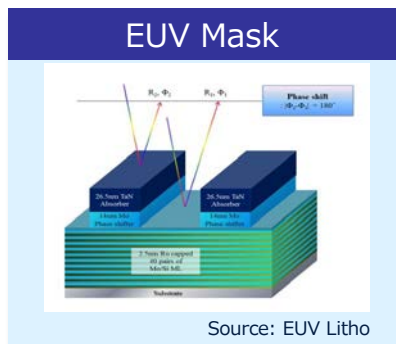
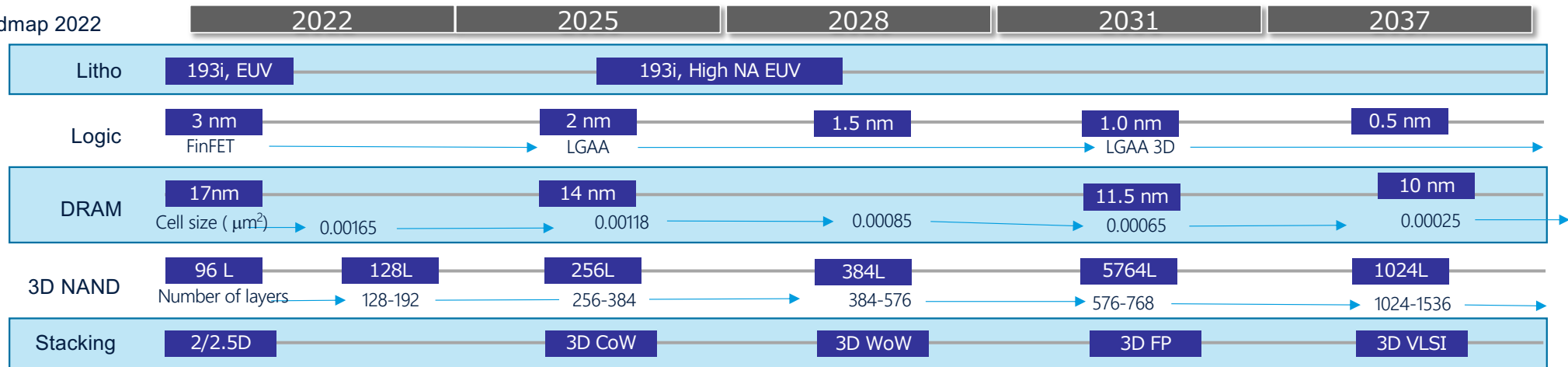
# X-Ray Metrology for Characterizing Advanced Nanoelectronics Structure

April 16<sup>th</sup>, 2024

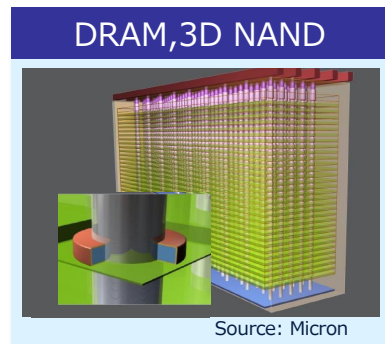
Kazuhiko OMOTE  
X-Ray Research Laboratory, Rigaku Corporation

# Trend of Semiconductor Devices and X-Ray Metrologies

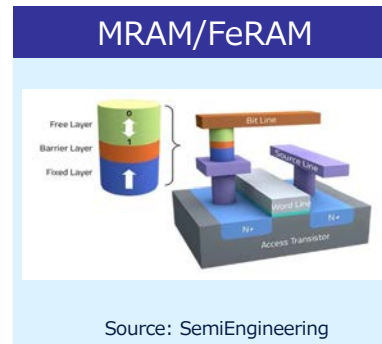
JRS Roadmap 2022



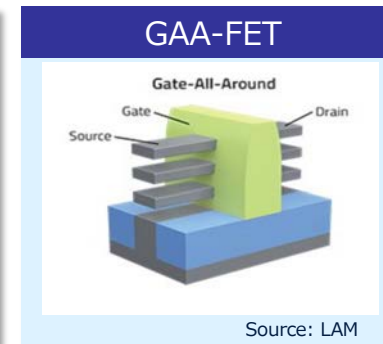
- Ultra thin film => XRR, XRD, XRF
- Contaminations => TXRF



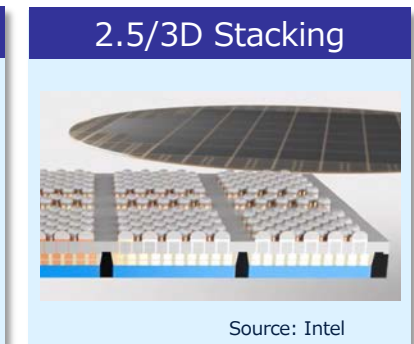
- High AR, 3D => SAXS, X-ray imaging
- W-fill, Si texture => XRF, XRD



- Composition => XRF
- 3D structure => SAXS



- Epitaxial film => HR-XRD, XRR
- 3D Structure => SAXS



- Micro bump, TSV
- Composition, Defects => Micro XRF
- => X-ray imaging

# Why X-Ray?

- **X-ray photons penetrate through materials and enable to investigate inner structure from the size of sub-nanometer (atomic scale) to micrometer of the materials, non-destructively.**

## ✓ **X-ray Diffraction/Scattering**

1. **HR-XRD** : Composition and thickness of epitaxial layers
2. **X-ray reflectivity** : Thickness and stacking structure of multilayers
3. **Topography** : Crystal defects, dislocations
4. **GI-SAXS** : Nanoimprint mold, EUV resist structures, FinFET, GAA, etc.
5. **T-SAXS** : 3DNAND, DRAM structures

## ✓ **X-ray Imaging**

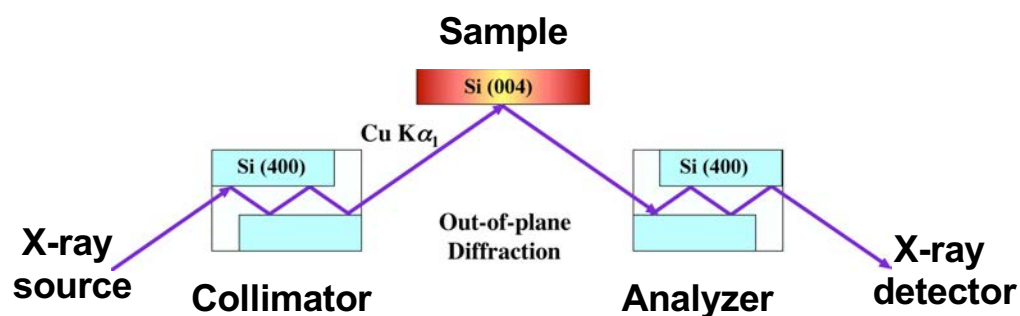
1. **μm Structure** : TSV, Packaging, etc.
2. **nm Structure** : Device scale imaging

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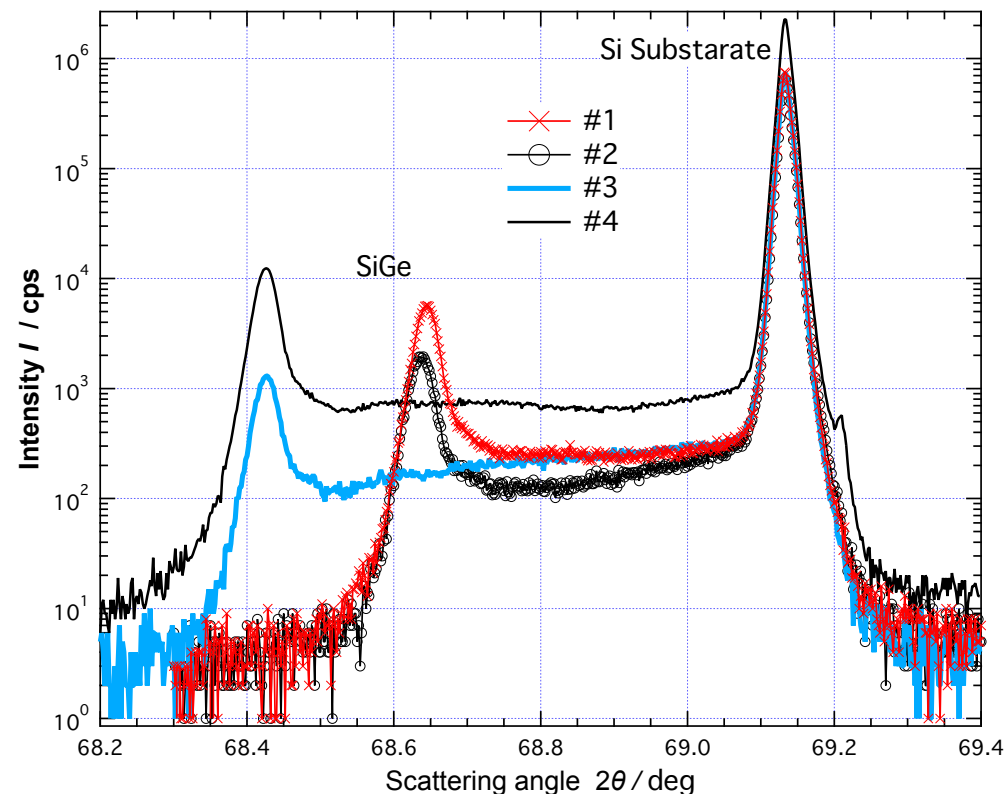
# X-ray Diffraction/Reflectivity/Topography



# Conventional High-Resolution X-ray Diffraction



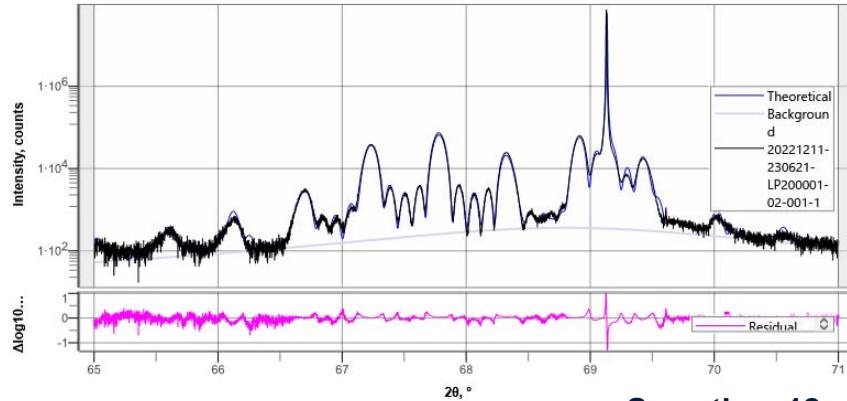
Specimen	Bulk Si			
	#1	#2	#3	#4
Si (nm)	12.54	23.06	12.07	22.90
SiGe	Thick	Thick	Thick	Thick



Parallel beam collimator is utilized for the conventional high-resolution X-ray diffraction and **large analysis area is required** for obtaining reasonable X-ray intensity.

# High-Resolution XRD and XRR for GAA Blanket Wafer

## GAA Stack Scan

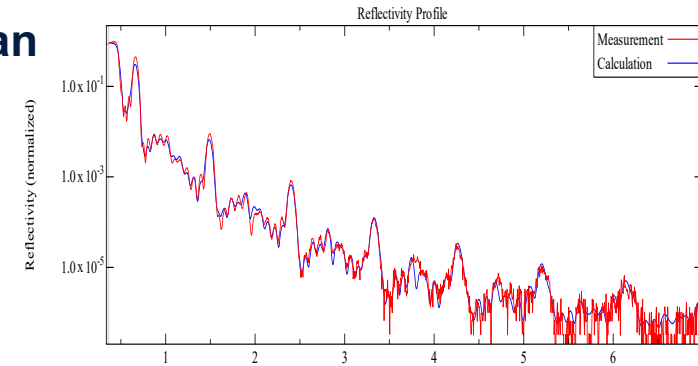


Scan time 12 min

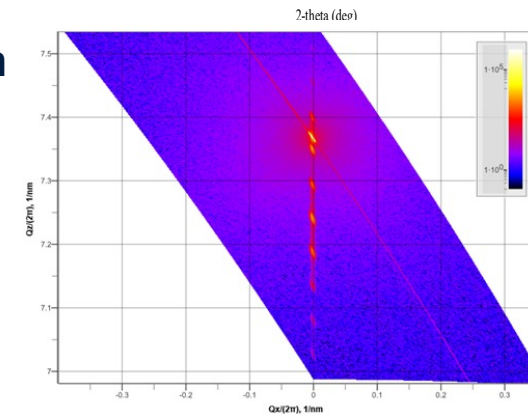
Layer No.	Materials	Thickness (nm)	sigma (nm)	Materials	Concentration Ge(%)	sigma (%)
11	Si	1.652	0.001			
10	Si	16.567	0.001			
9	Si(1-x)Ge(x)	9.871	0.001	Ge conc	28.09%	0.04%
8	Si	10.074	0.002			
7	Si(1-x)Ge(x)	9.740	0.001	Ge conc	28.46%	0.04%
6	Si	9.716	0.002			
5	Si(1-x)Ge(x)	9.349	0.001	Ge conc	28.62%	0.03%
4	Si	9.837	0.009			
3	Si(1-x)Ge(x)	8.993	0.001	Ge conc	28.99%	0.03%
2	Si	9.405	0.002			
1	Si(1-x)Ge(x)	9.574	0.001	Ge conc	27.67%	0.04%
0	Sub					

## High-intensity Rotating-anode, 9 kW X-ray source

## XRR Scan



## RSM Scan

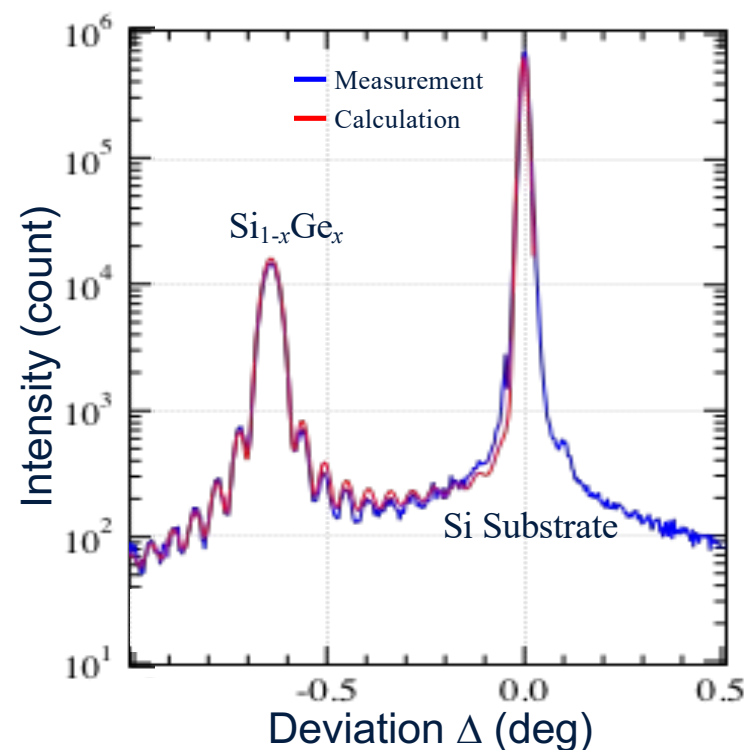
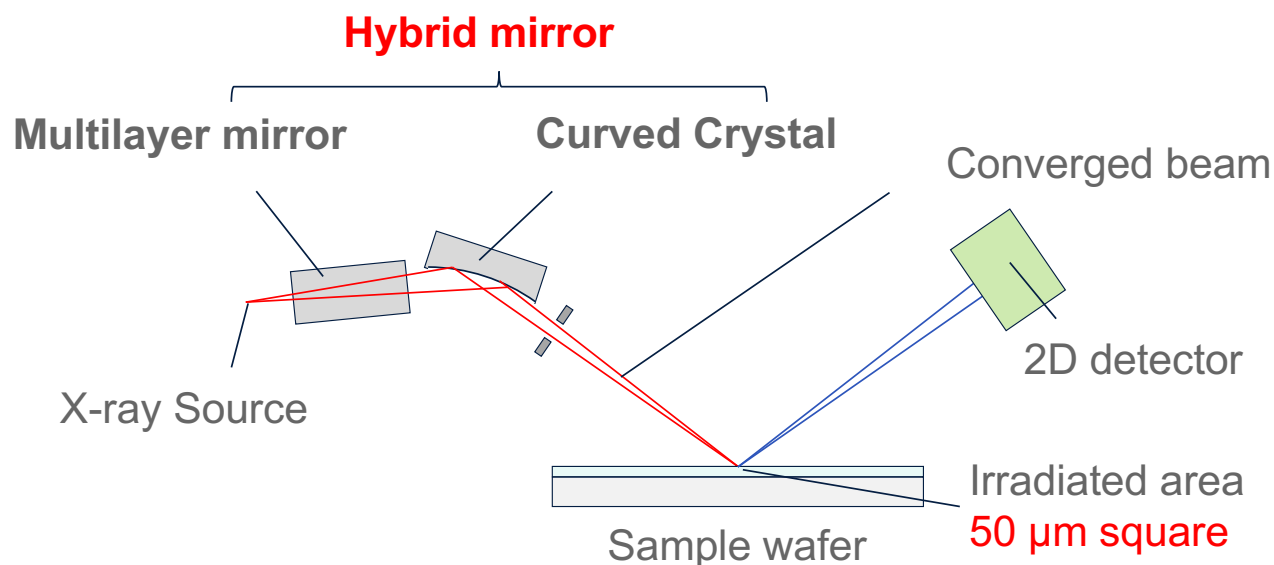


Scan time 10 min

RSM/off-specular scans can assess quality and strain/relaxation

# High-Resolution X-ray Diffraction for Small Area

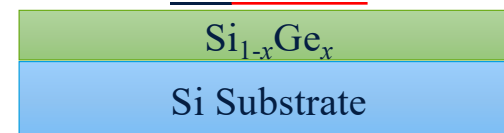
## Micro Focus High-Resolution XRD



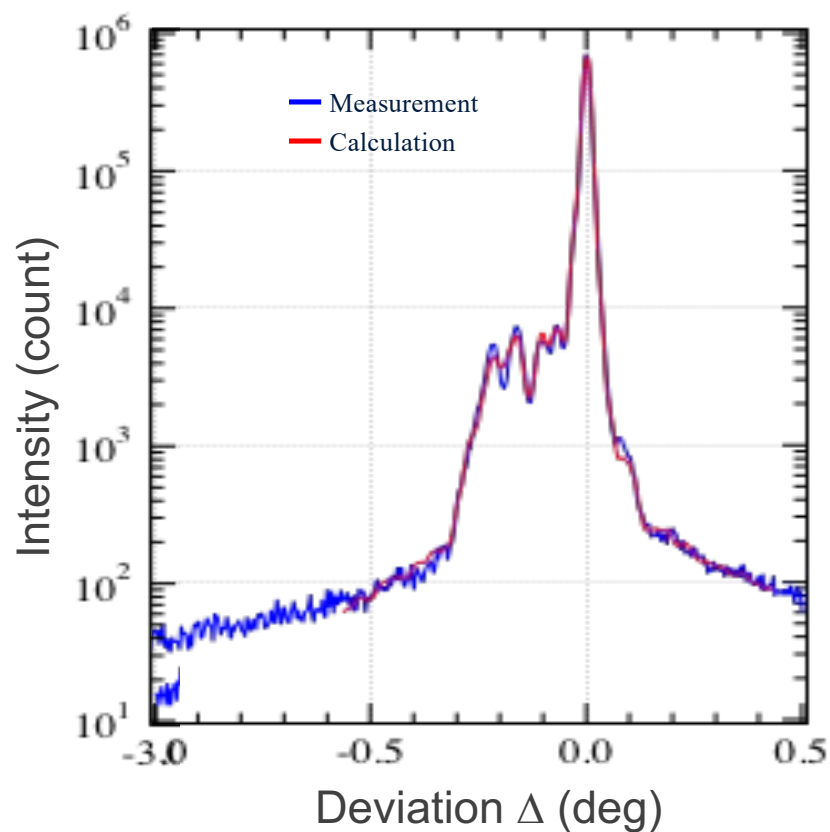
Convergent beam enable to measure diffraction pattern of epitaxial layer in small area at once and typical measurement time is 10 s to 100 s.

Thickness  $t = 96.61 \text{ nm}$

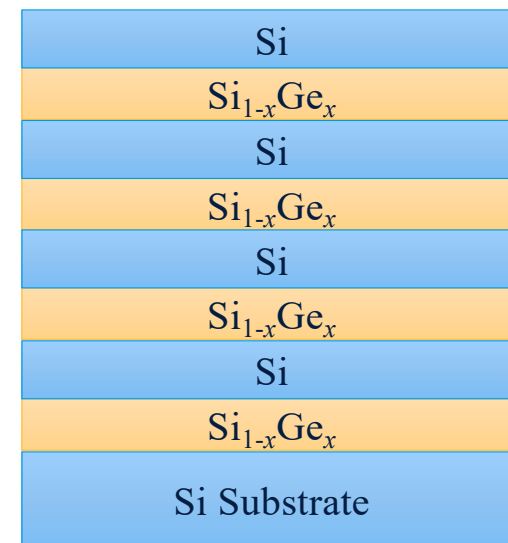
$x = 23.25 \%$



# Analysis for Multi-Stacking SiGe Layer

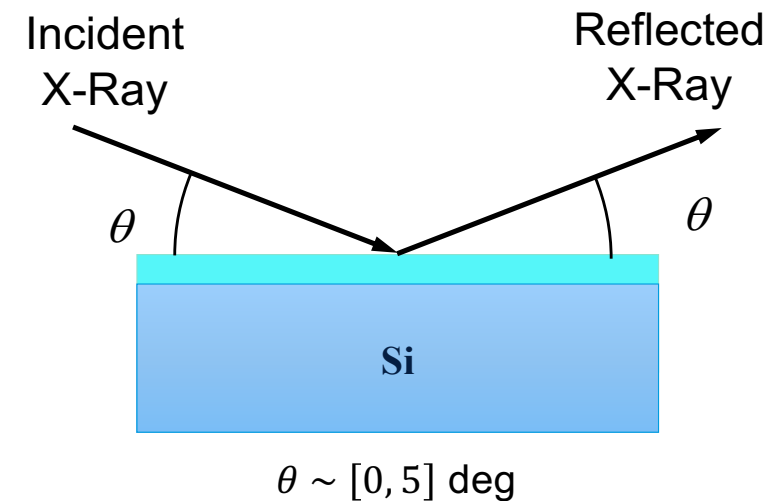
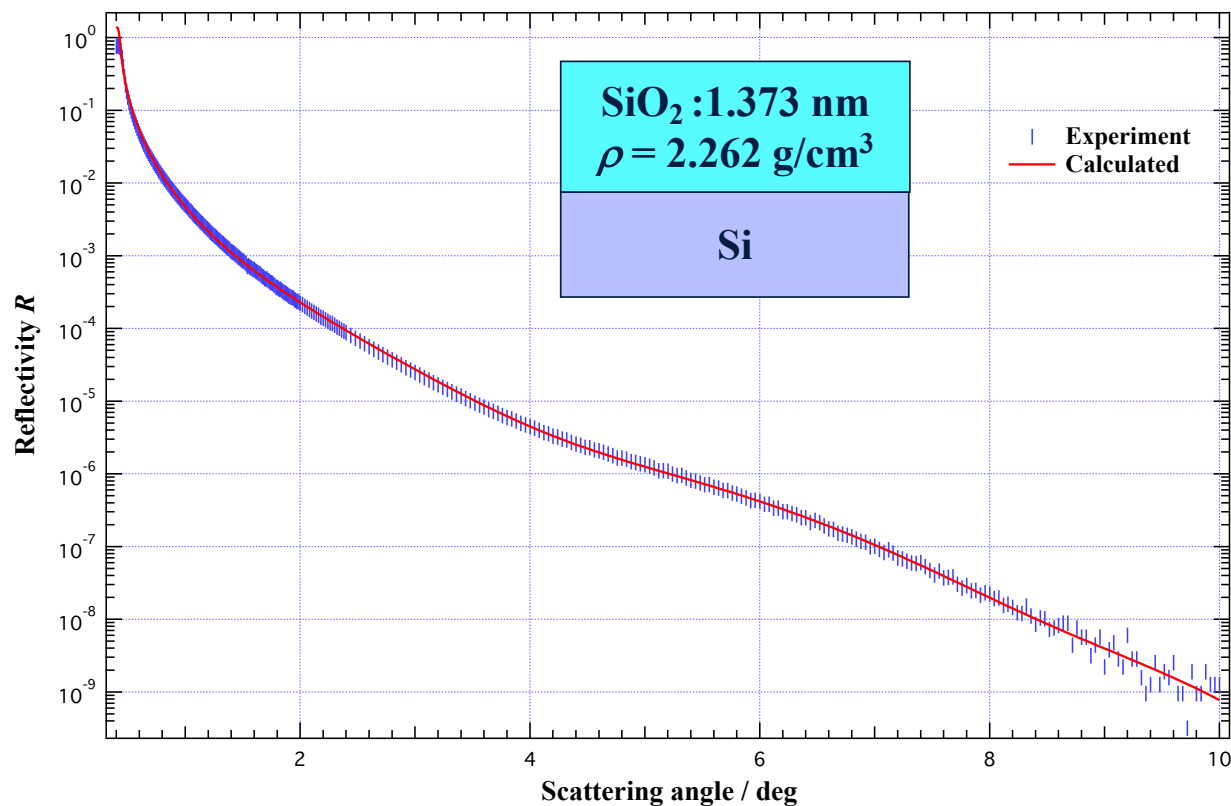


layer	t (nm)	Ge (%)
Si	80.68	-
SiGe	44.12	6.79
Si	65.21	-
SiGe	16.03	4.06
Si	94.12	-
SiGe	50.96	2.58
Si	17.66	-
SiGe	32.71	6.95



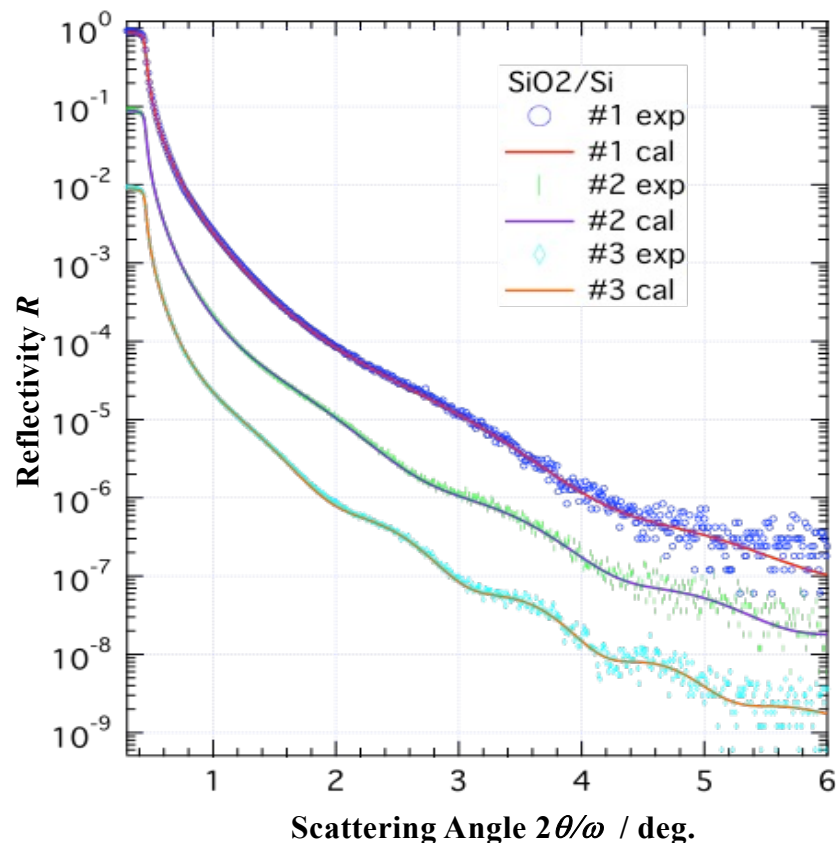
**Multi-Stacking of Si/SiGe layers can be analyzed by small area HR-XRD**

# X-Ray Reflectivity (XRR) - Ultra Thin Oxide Film -



**1 nm thick oxide layer could be analyzed by high-dynamic range XRR measurement**

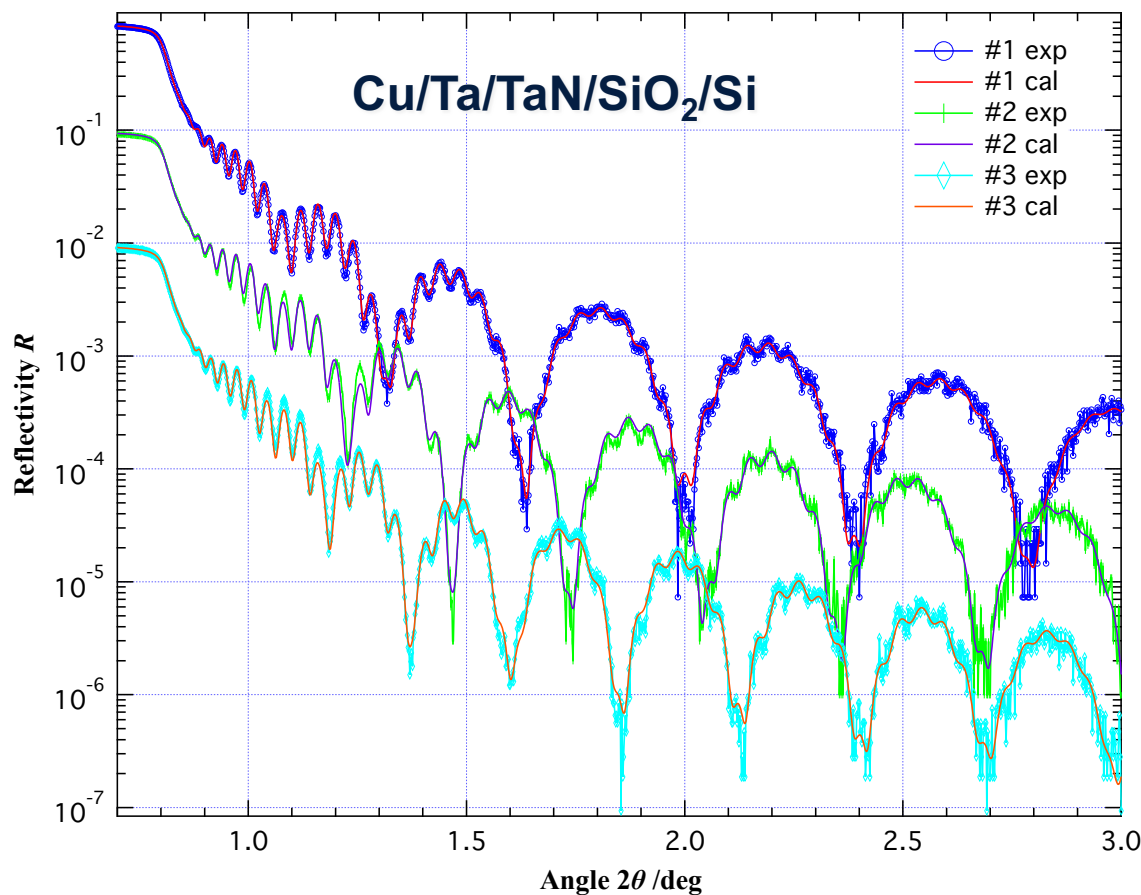
# XRR: Structure of Thermal SiO<sub>2</sub> Film



		Thickness (nm)	Density (g/cm <sup>3</sup> )	Roughness (nm)
#1	SiO <sub>2</sub>	3.26	2.28	0.48
	SiO <sub>2</sub>	0.73	2.38	0.30
	Si	-	2.329	0.10
#2	SiO <sub>2</sub>	5.26	2.24	0.44
	SiO <sub>2</sub>	0.73	2.39	0.49
	Si	-	2.329	0.04
#3	SiO <sub>2</sub>	7.33	2.27	0.46
	SiO <sub>2</sub>	1.16	2.39	0.33
	Si	-	2.329	0.10

- Few nanometer thick SiO<sub>2</sub> films are accurately determined
- Higher density layers are formed on Si substrate

# X-Ray Reflectivity for Metal Layers



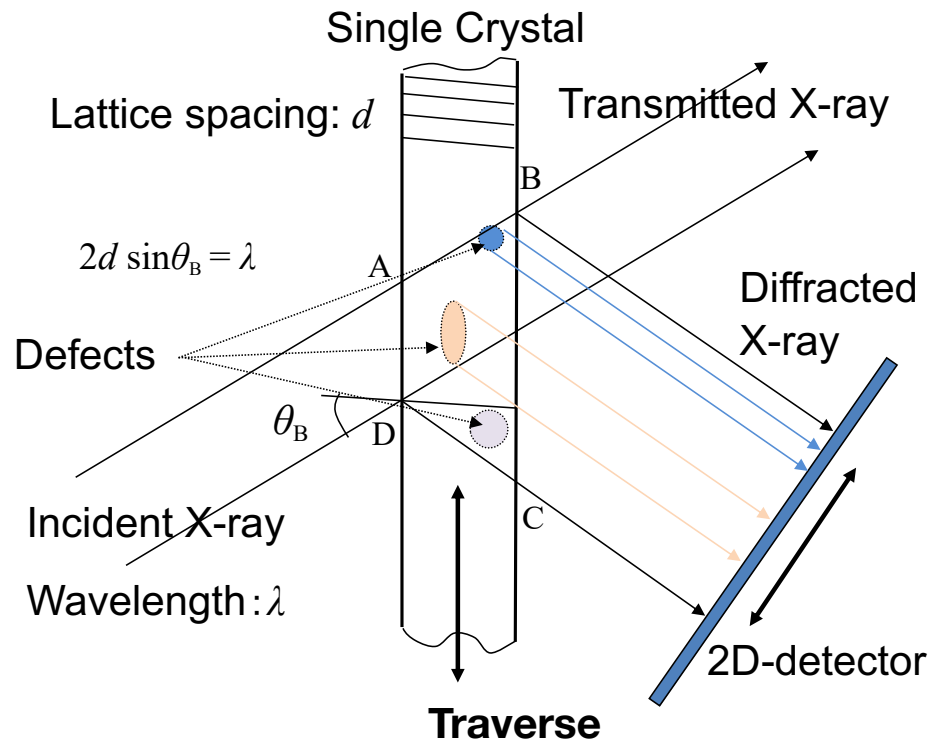
		Thickness (nm)	Density (g/cm <sup>3</sup> )	Roughness (nm)
#1	<b>CuO</b>	<b>2.73</b>	<b>5.2</b>	<b>1.45</b>
	Cu	147.74	9.053	1.17
	Ta	10.94	15.94	0.05
	TaN	9.33	14.52	0
	SiO <sub>2</sub>	-	2.20	0.423
#2	<b>CuO</b>	<b>2.70</b>	<b>5.8</b>	<b>1.59</b>
	Cu	146.98	9.04	0.97
	Ta	14.55	16.46	0.21
	TaN	10.23	14.91	0
	SiO <sub>2</sub>	-	2.20	0.432
#3	<b>CuO</b>	<b>2.79</b>	<b>4.3</b>	<b>1.25</b>
	Cu	146.95	9.089	1.20
	Ta	18.88	16.34	0.427
	TaN	9.34	14.95	0
	SiO <sub>2</sub>	-	2.20	0.441

Top Cu oxidation layer thickness was determined by XRR measurements



# X-Ray Topography (Diffraction Imaging)

## Geometry of the X-ray topography



A. R. Lang, *Acta Cryst.*, **12**, 249 (1959)

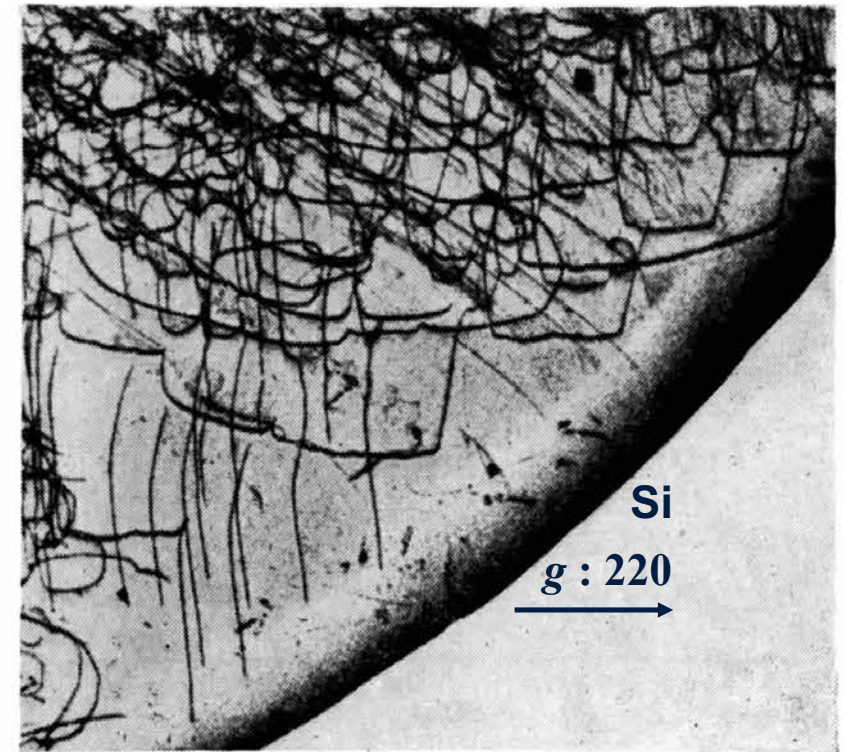
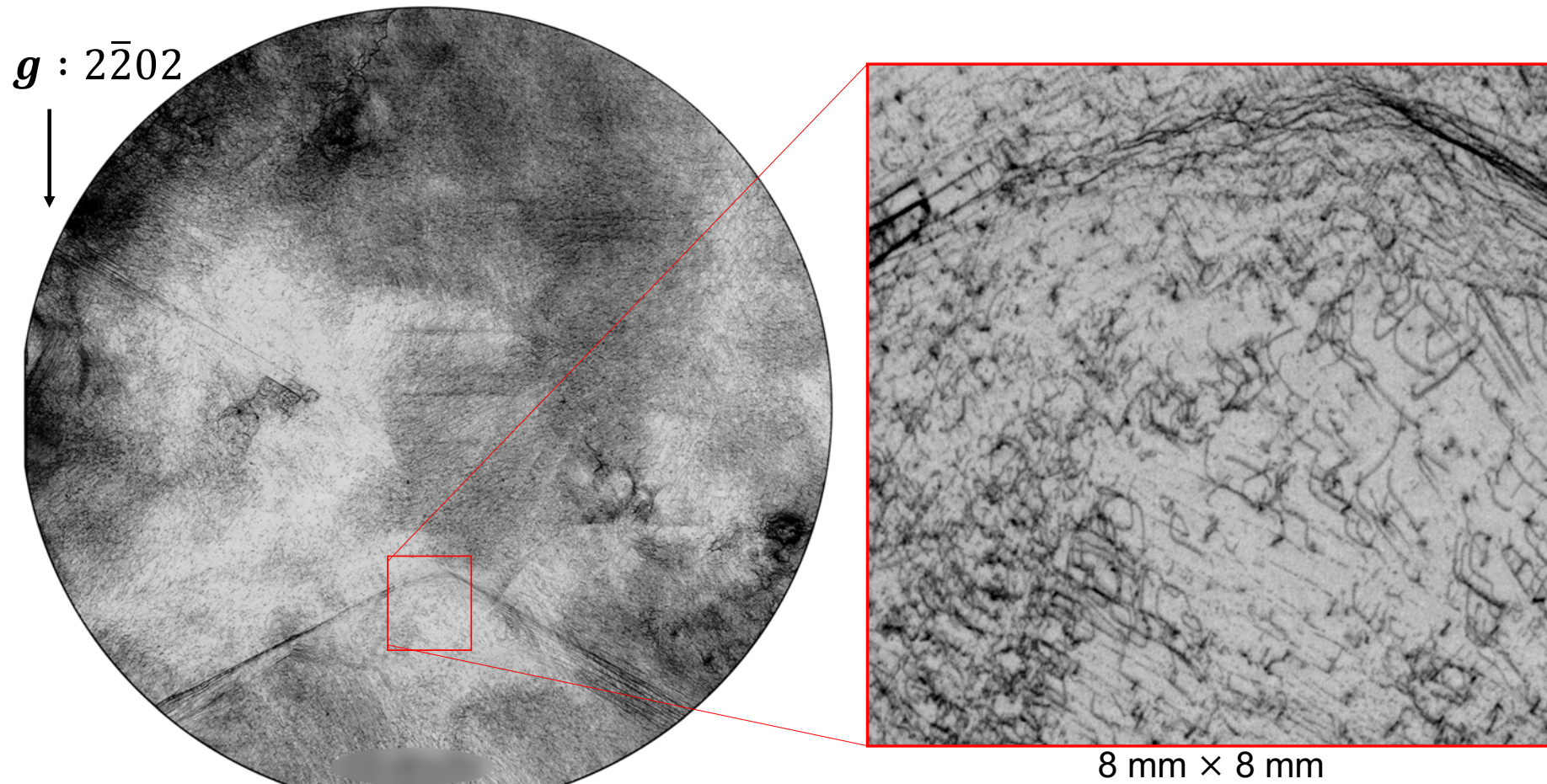


Fig. 2. A projection topograph of dislocations in a  $\{111\}$  slice of silicon, taken with a 220 reflection from planes normal to the slice.

Individual dislocations in the entire crystal are identified



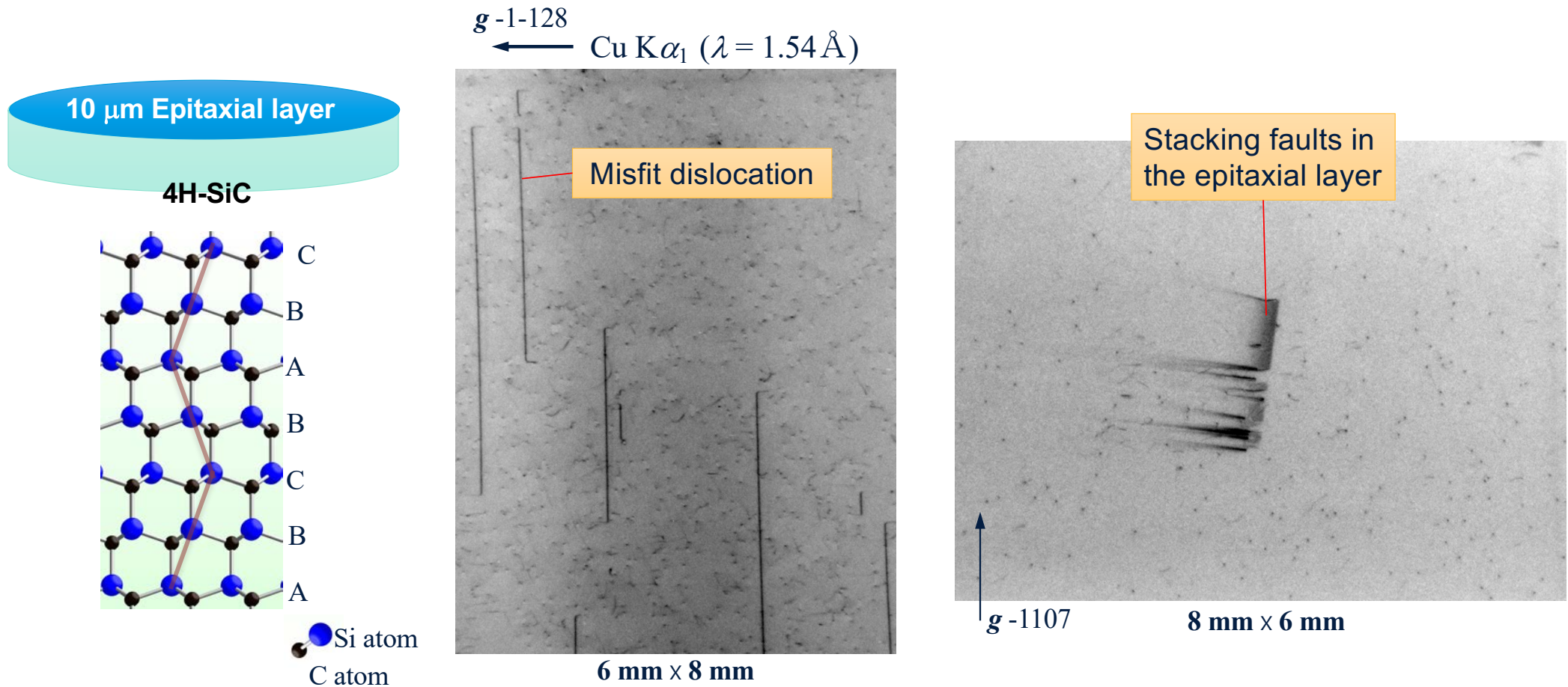
# 4H-SiC 3 inches wafer Transmission Topography



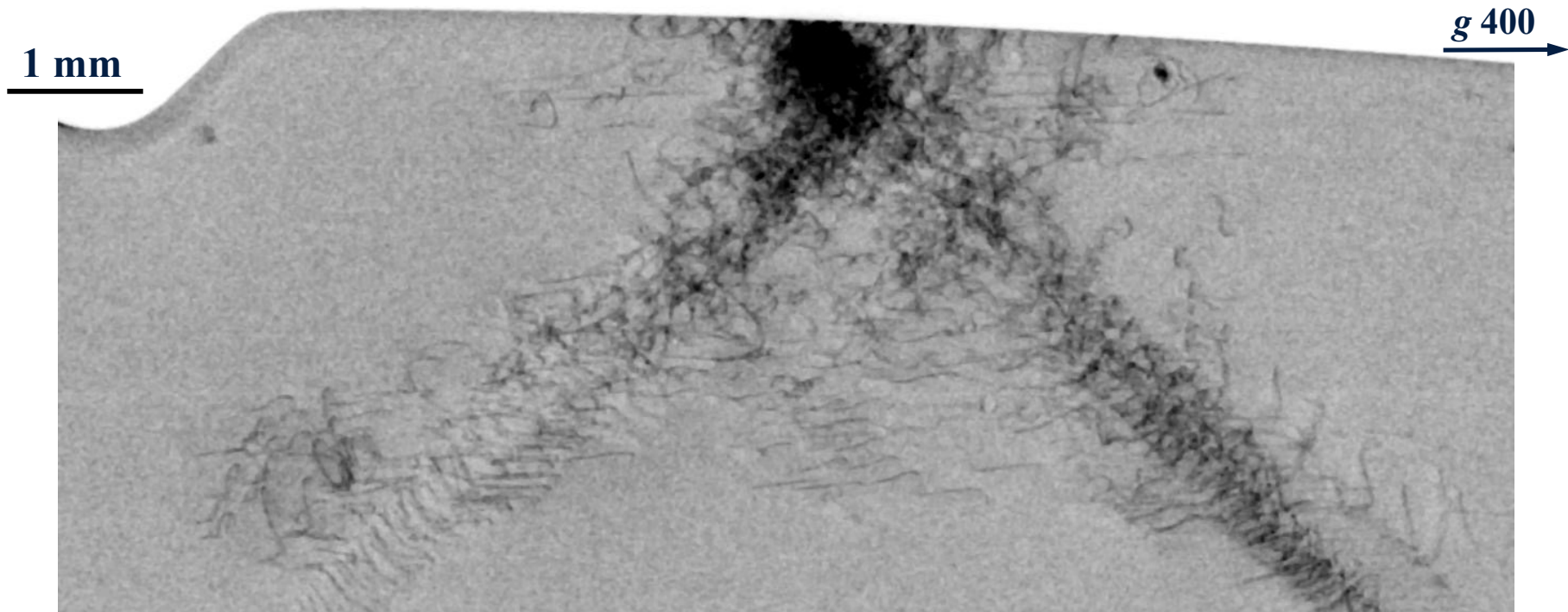
**SiC crystals for the industrial use still contain a lot of dislocations**

# Reflection Topography for 4H-SiC

High-resolution x-ray camera (pixel size: 2.4  $\mu\text{m}$ ) is used for the measurements



# Topography for Process Induced Defects in Si Wafer



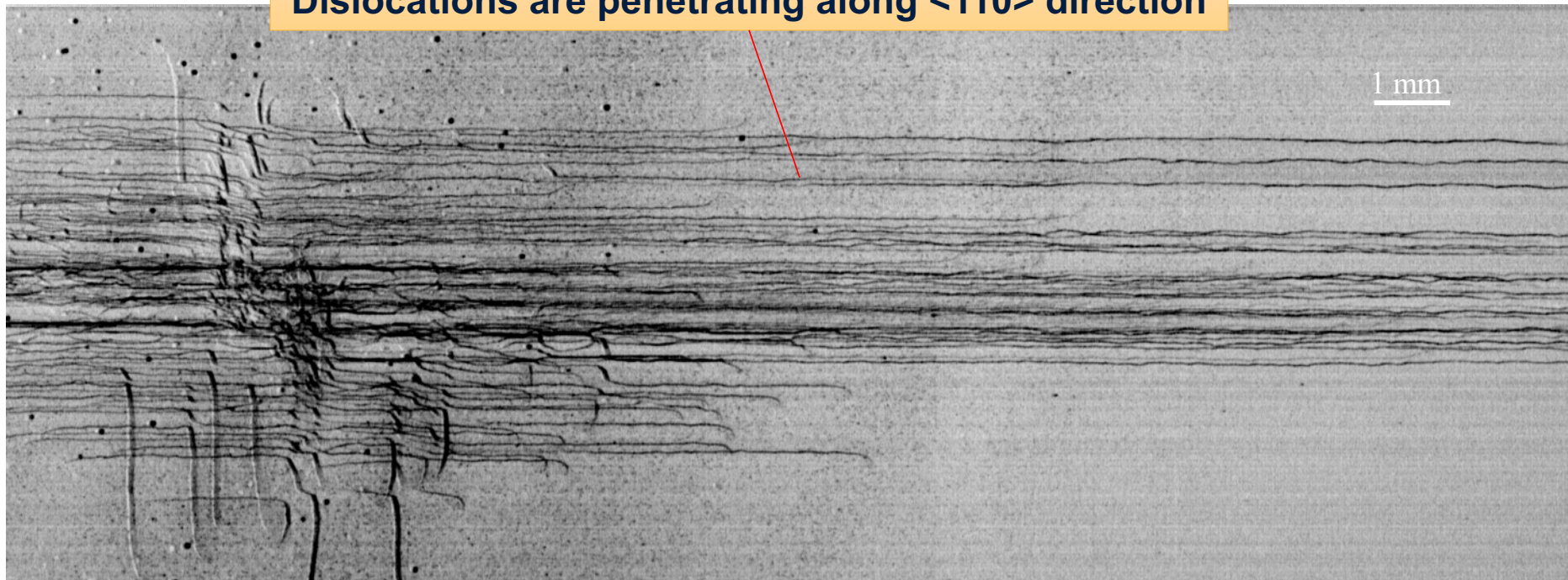
Original Si wafer is dislocation free, but can be created by the device manufacturing process



# 3D Visualization of Dislocations in Si Wafer

Dislocations are penetrating along  $\langle 110 \rangle$  direction

$g = 220 \rightarrow$



$g = 220 \odot$

1 mm



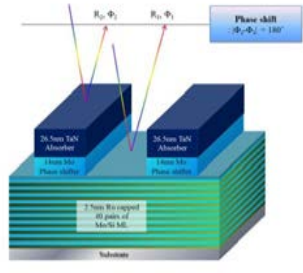
Dislocations are penetrating center of wafer plate

# **Small Angle X-ray Scattering (SAXS) for Device Nano-pattern Analysis**

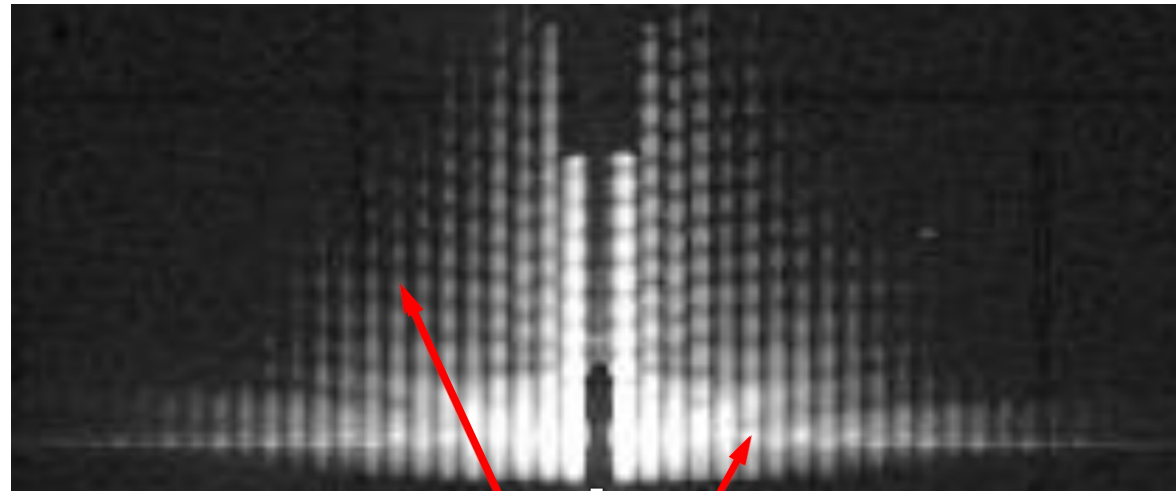
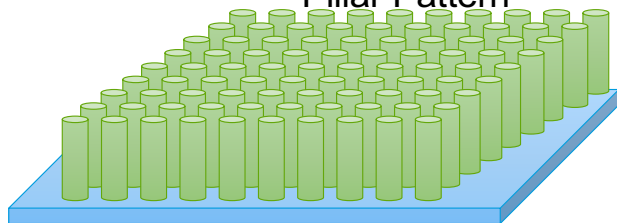
# Grazing Incidence X-ray Scattering (GI-SAXS)

## Analysis for Surface Nanostructure

EUV Mask

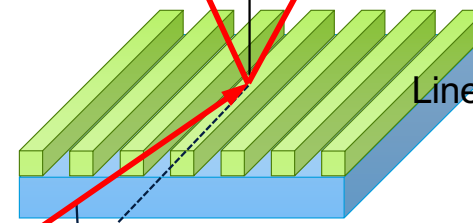


Pillar Pattern



$\phi$  : Sample rotation angle  $\phi$  :  $-10^\circ \sim +10^\circ$

Line & Space Pattern



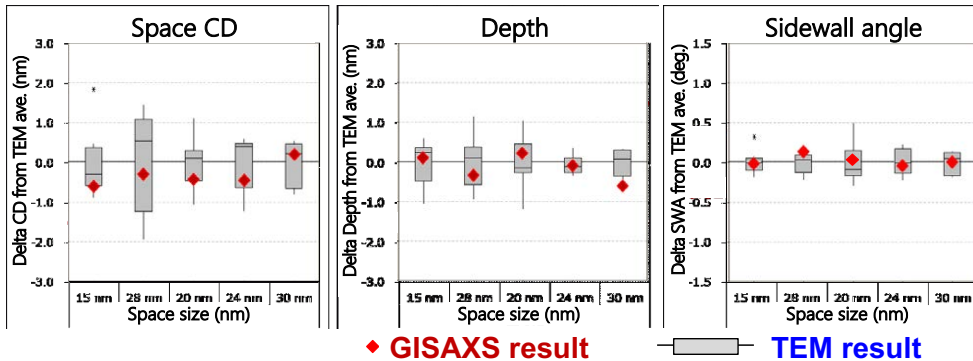
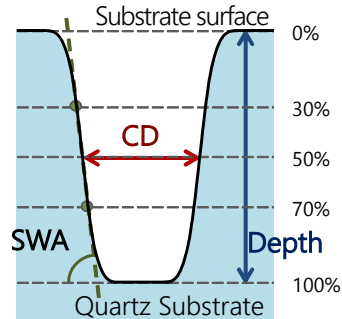
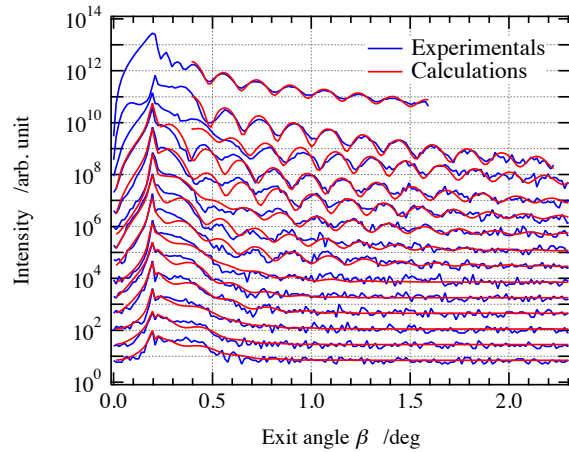
Incident X-ray  
 $\lambda = 0.154 \text{ nm}$   
(Cu K $\alpha$ )

Incident angle :  $\alpha \sim 0.2^\circ$

K. Omote, Y. Ito, and Y. Okazaki, *Proc. of SPIE*, 7638, 763811 (2010)

**GI-SAXS enables to analyze surface structure for the nanoscale device patterns**

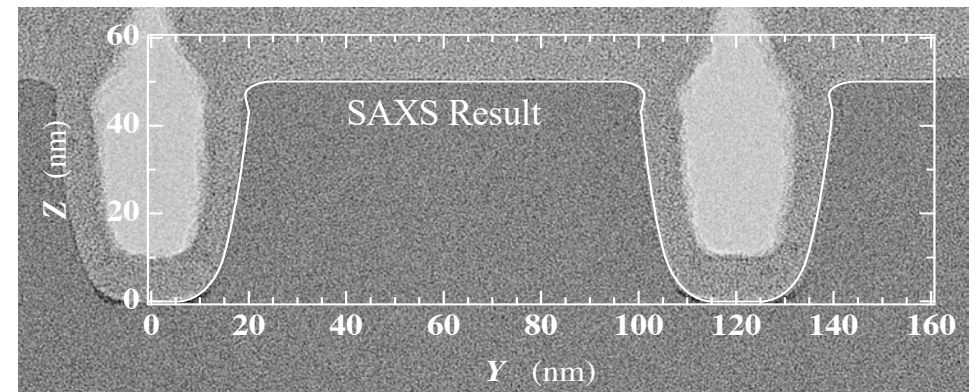
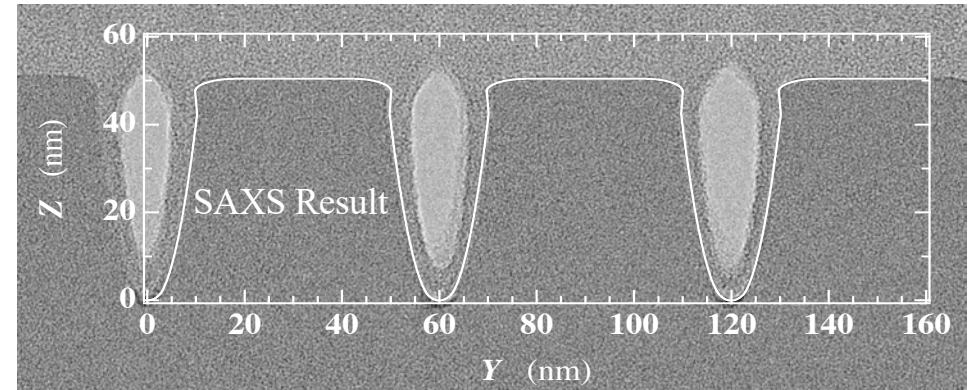
# Nano-Imprint Mold L&S Cross-Section Profile



◆ GISAXS result

▢ TEM result

Cross-section TEM image



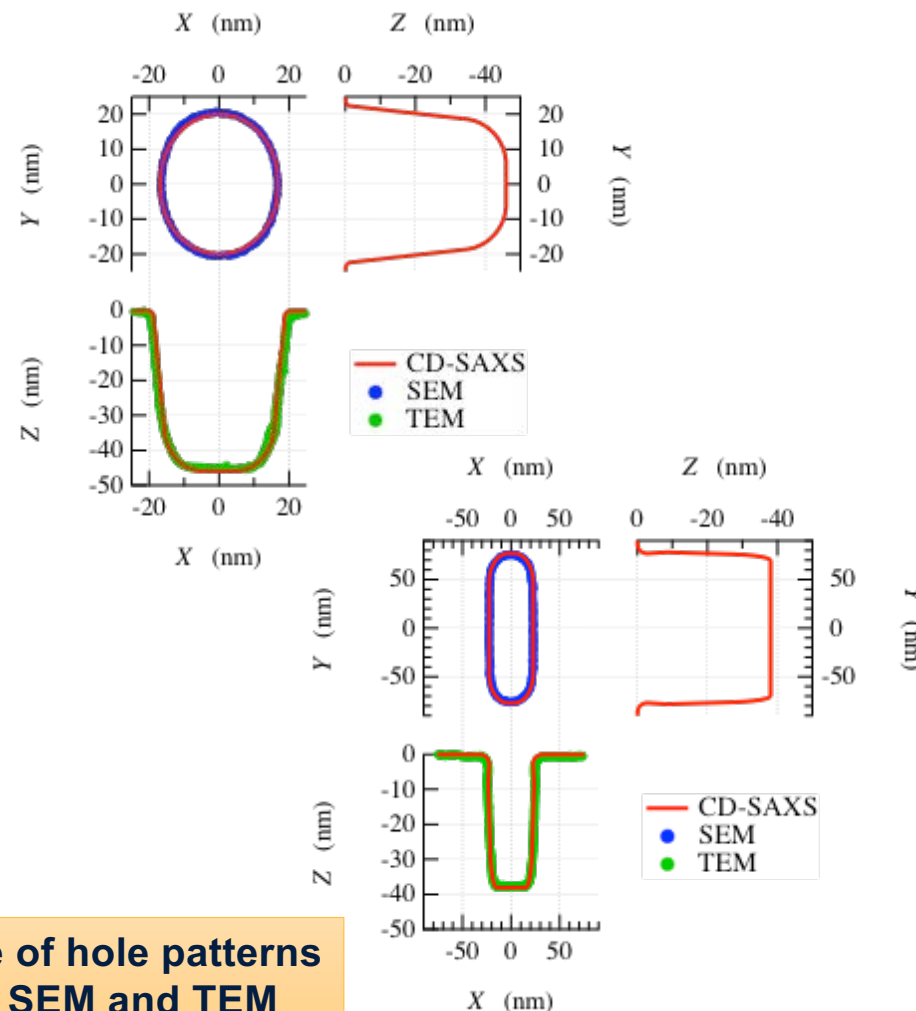
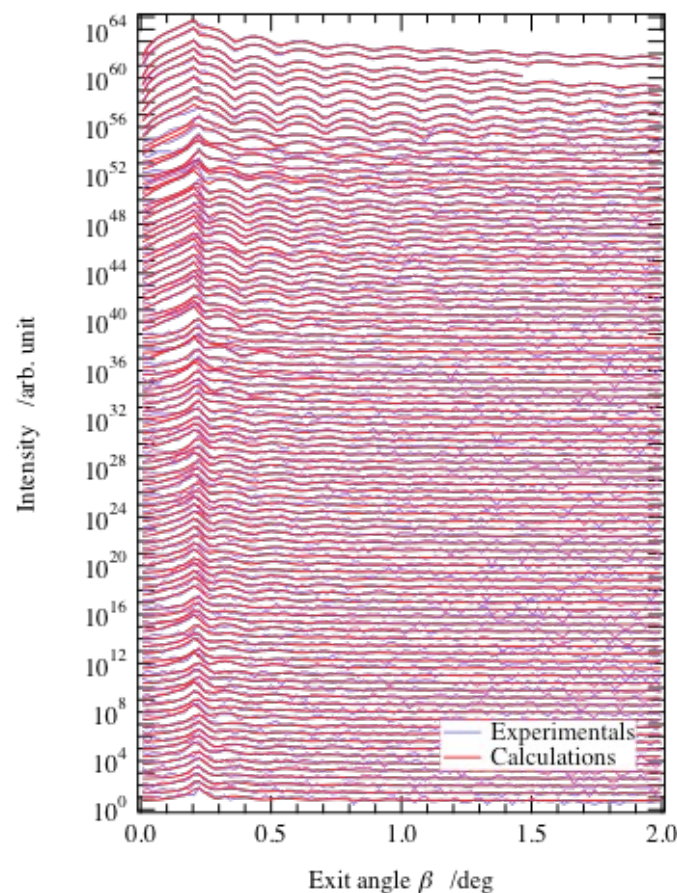
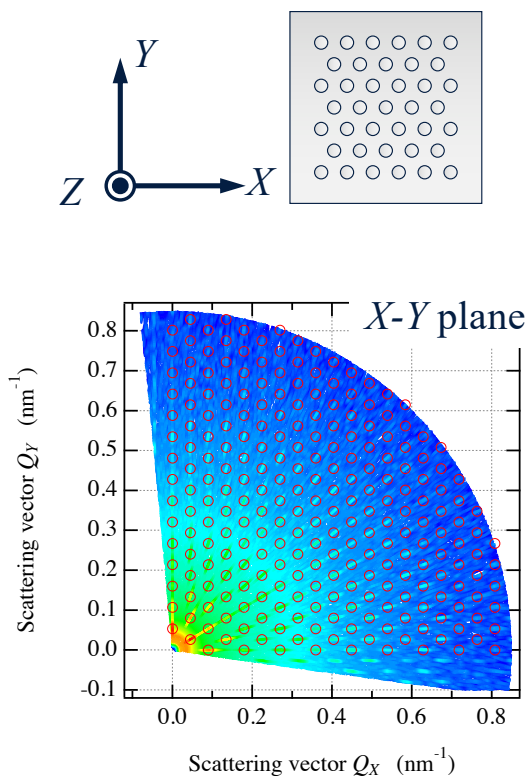
**GI-SAXS results agree very well with that of cross-section TEM**

E. Yamanaka, et. al., *Proceedings of SPIE*, 9984, 99840V, (2016)



# Nano-Imprint Mold Hole Pattern 3D-Shape

## Hole Pattern

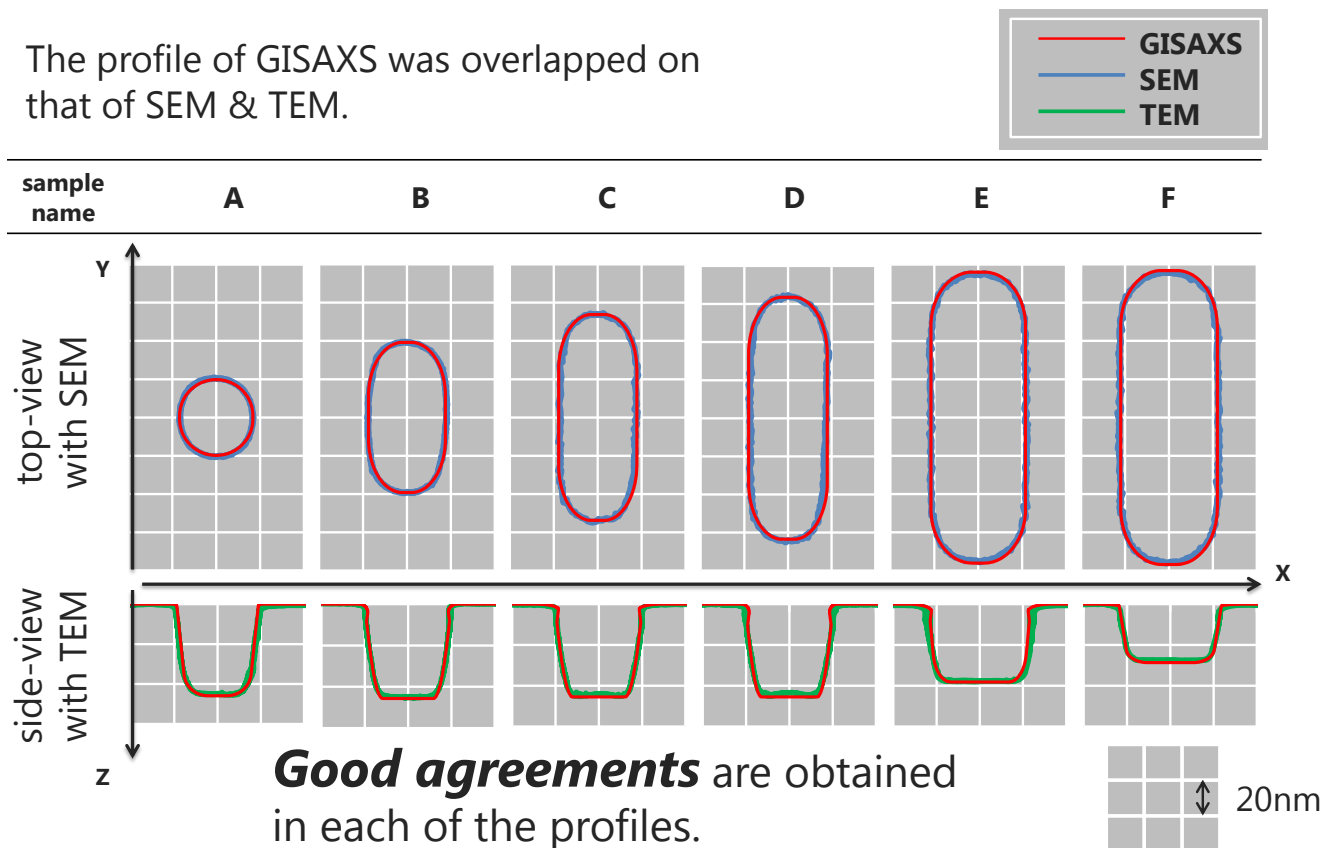


**GI-SAXS can also analyzed three-dimensional structure of hole patterns and the results agree very well with that observed by SEM and TEM**



# Nano-Imprint Mold Hole Pattern SAXS vs. SEM & TEM

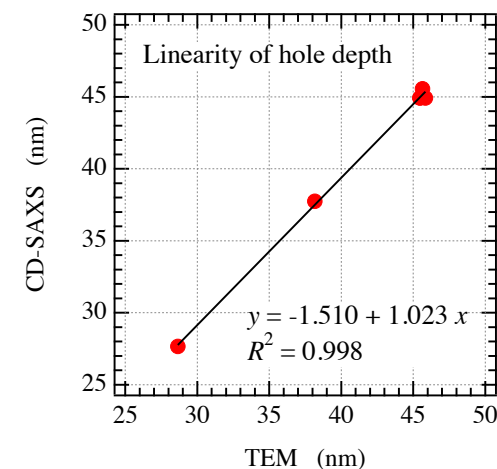
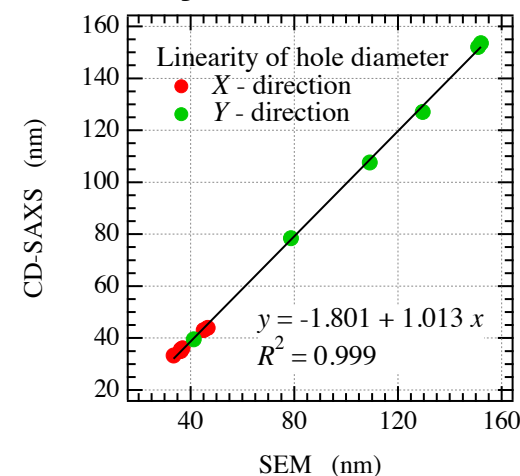
The profile of GISAXS was overlapped on that of SEM & TEM.



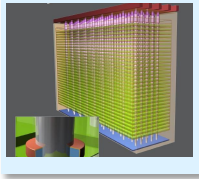
**GI-SAXS results agree very well with that of SEM and cross-section TEM**

K. Hagihara, et. al., *Proceedings of SPIE*, 10451, 104510H (2017)

## Linearity with SEM & TEM

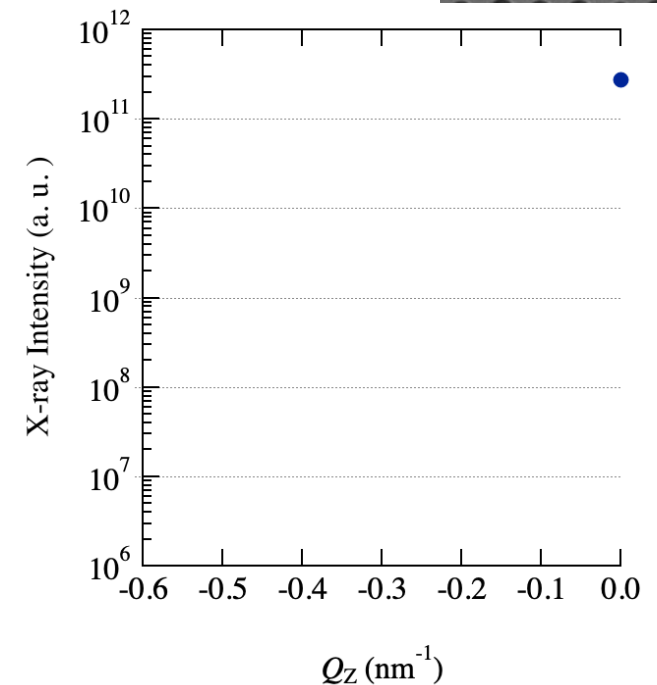
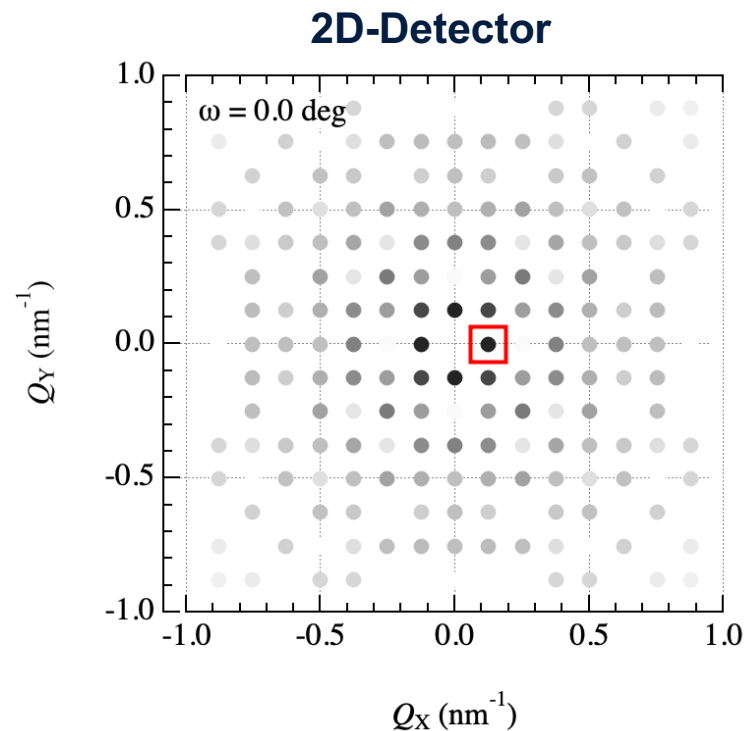
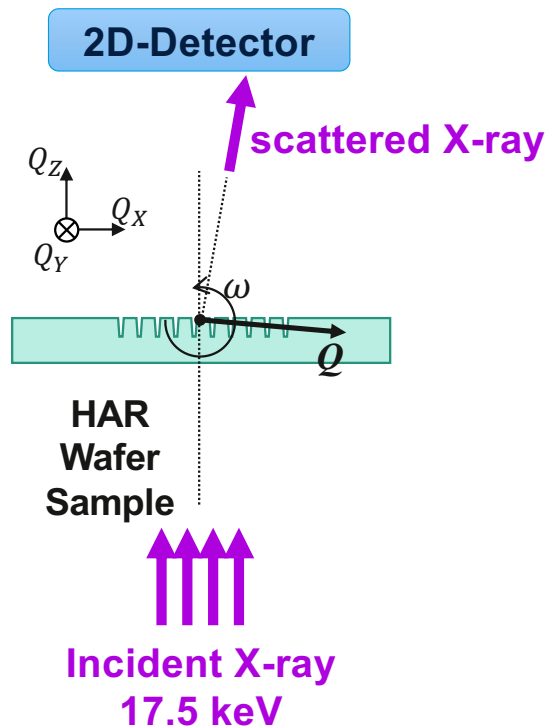
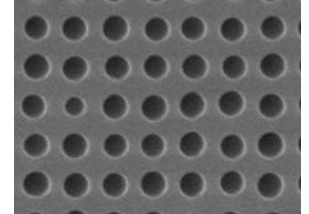


DRAM, 3D NAND



# Transmission X-ray Scattering (T-SAXS)

## Analysis for HAR (High Aspect Ratio) Structures



T-SAXS enables to analyze HAR structure for the process wafers as is

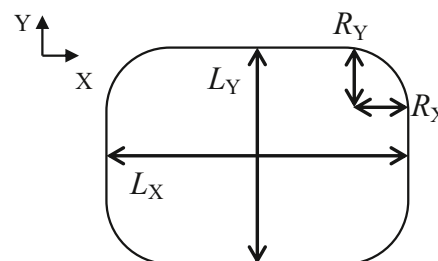
# Shape Modeling for HAR Hole Structure

- The X-Y cross-sectional shape of the hole was represented with “Koban model”.

**Parameters: Koban parameter  $R_X/L_X$  ( $R_Y/L_Y$ )**

- The structure in the depth direction of the hole was represented by stacking of a number of thin layers.

**Parameters: diameter, center line shift, thickness, etc.**



$R_X/L_X = 0.5 \rightarrow$  ellipse  
 $R_X/L_X = 0.0 \rightarrow$  rectangular



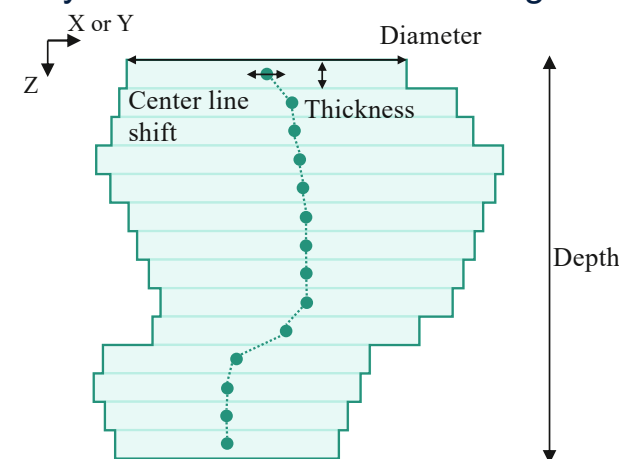
“Koban”  
Japanese oval gold coin in Edo period

$$F_j(\mathbf{Q}) = \sum_{l=1}^M \int_{X,Y} \rho_j(z_l, x, y) e^{-i(Q_x x + Q_y y + Q_z z_l)} dx dy \Delta z_l$$

$$I(\mathbf{Q}) = (r_c P)^2 \langle F_j(\mathbf{Q}) F_k^*(\mathbf{Q}) e^{-i\mathbf{Q} \cdot (\mathbf{u}_j - \mathbf{u}_k)} \rangle \sum_{j,k=1}^N e^{-i\mathbf{Q} \cdot (\bar{\mathbf{R}}_j - \bar{\mathbf{R}}_k)}$$

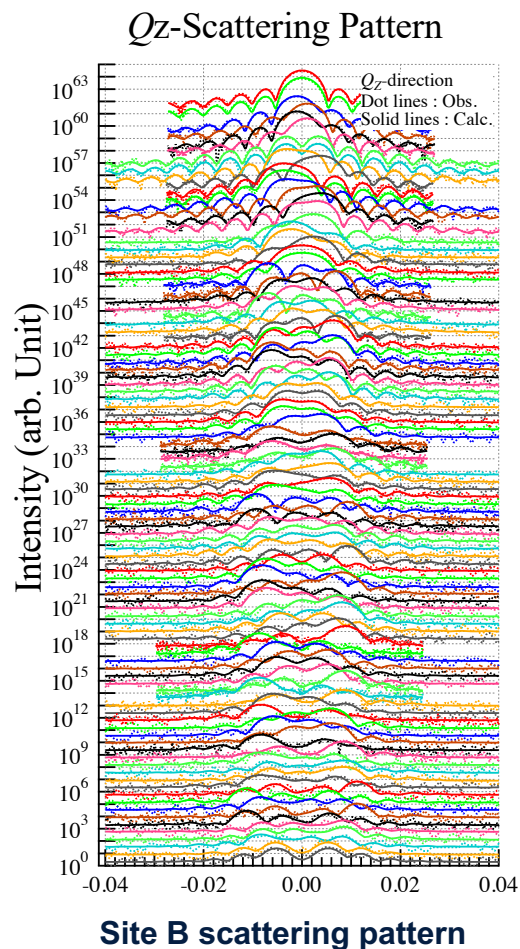
$$= (r_c P)^2 \langle F_j(\mathbf{Q}) F_k^*(\mathbf{Q}) e^{-i\mathbf{Q} \cdot (\mathbf{u}_j - \mathbf{u}_k)} \rangle \frac{\sin\left(\frac{N_x Q_x L_x}{2}\right) \sin\left(\frac{N_y Q_y L_y}{2}\right)}{\sin\left(\frac{Q_x L_x}{2}\right) \sin\left(\frac{Q_y L_y}{2}\right)}$$

Stacked layer structure schematic diagram

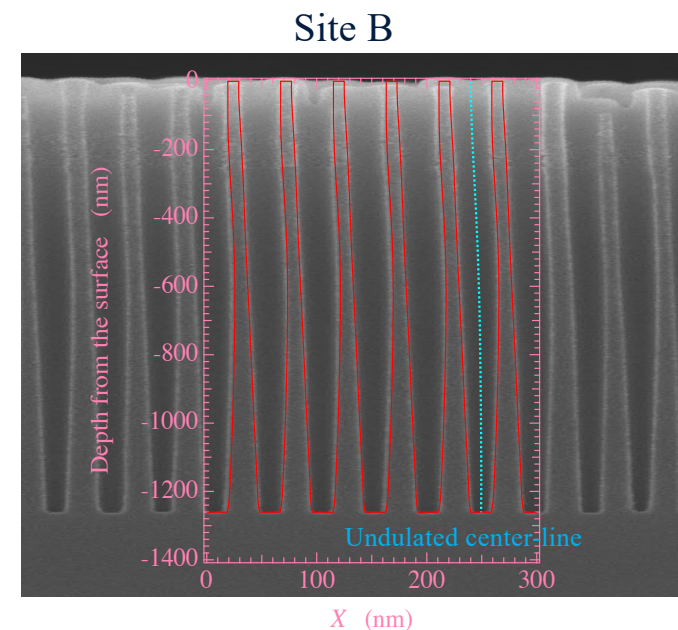
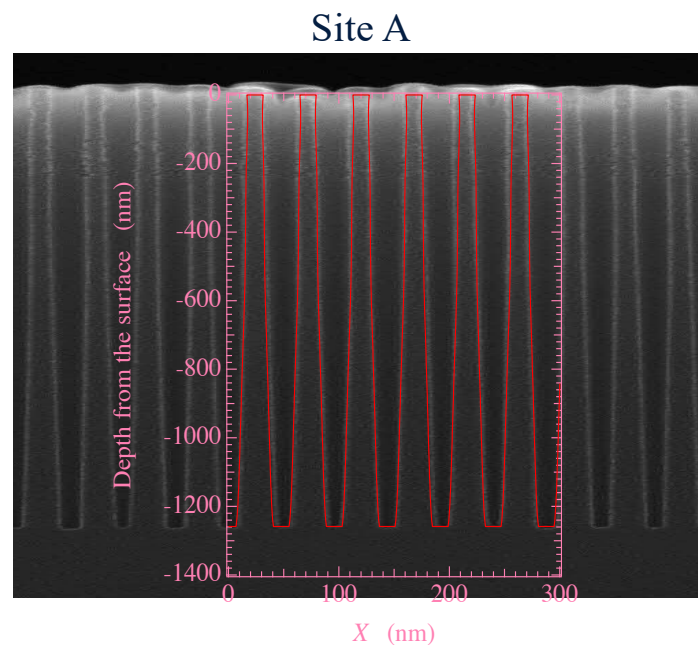


Free slicing for the HAR structure is used and analyzing without any specific models of the hole structure

# Example of HAR Structure Analysis by SAXS



Red line indicates the obtained profile to optimize measured and calculated scattering intensity.

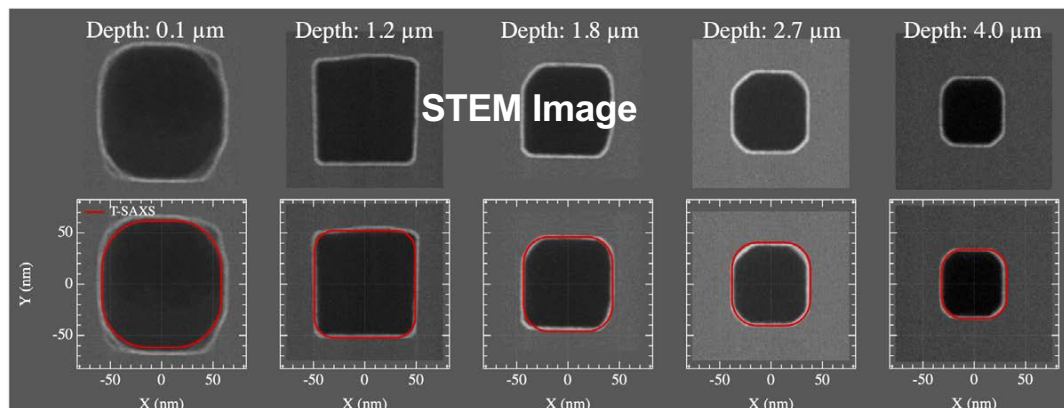


The X-Z cross section profile estimated by SAXS are consistent with that measured by the cross-sectional SEM as a reference.

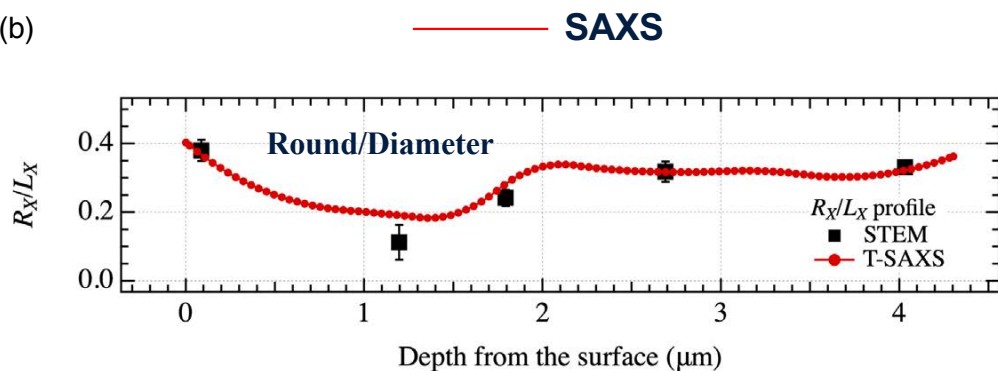
Yoshiyasu Ito, *et. al.*, Japanese Journal of Applied Physics, **62**, 046501 (2023).

# Precise Analysis for The Hole Shape by SAXS

(a)



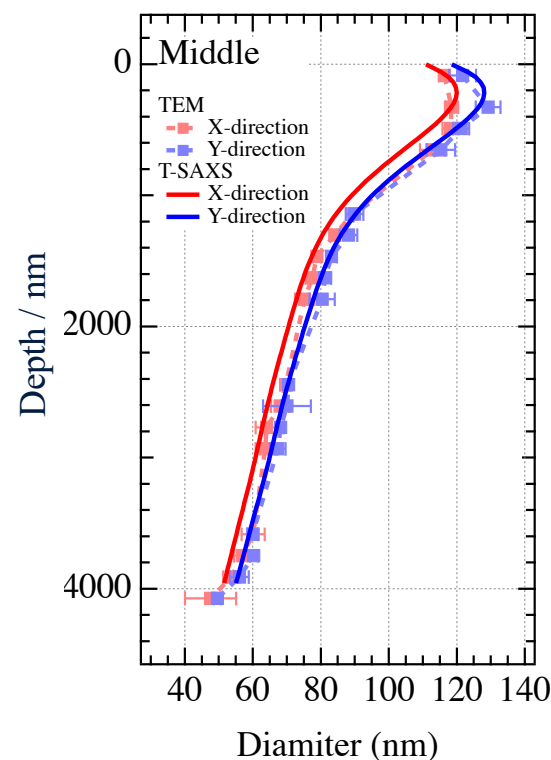
(b)



Similar result with TEM was observed for cross sectional shape

Hole depth shape

Hole 3D shape



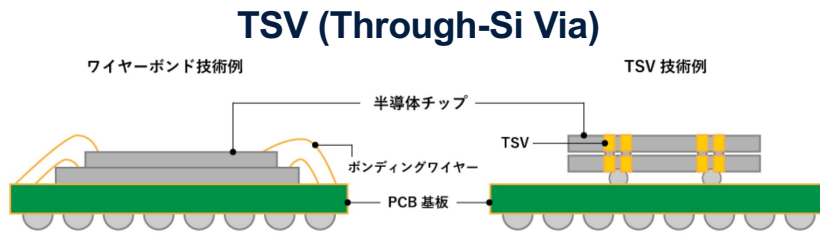
Rieko Suenaga, *et. al.*, Japanese Journal of Applied Physics, **62**, 096502 (2023).

X-ray scattering is superior to analyze average structure with 0.1 nm resolution, non-destructively, but not good for detecting local defects. Therefore, it is strongly demanded a metrology to observe local defects.

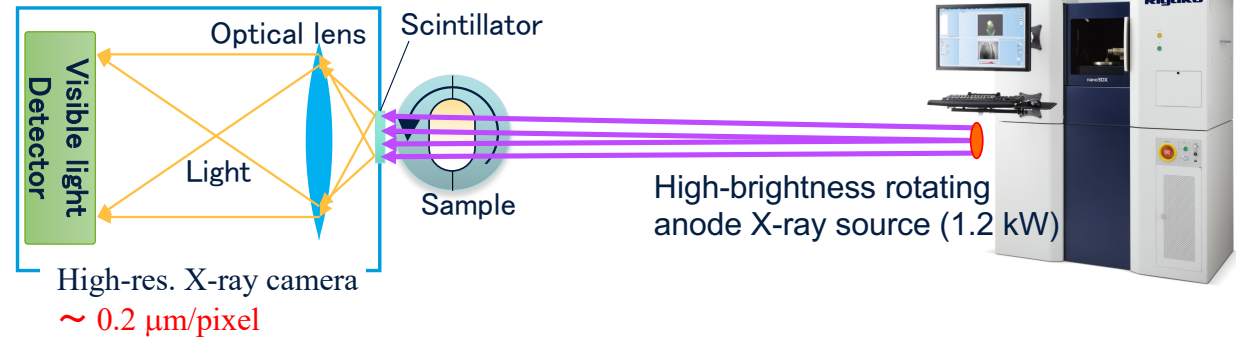
# X-ray Imaging



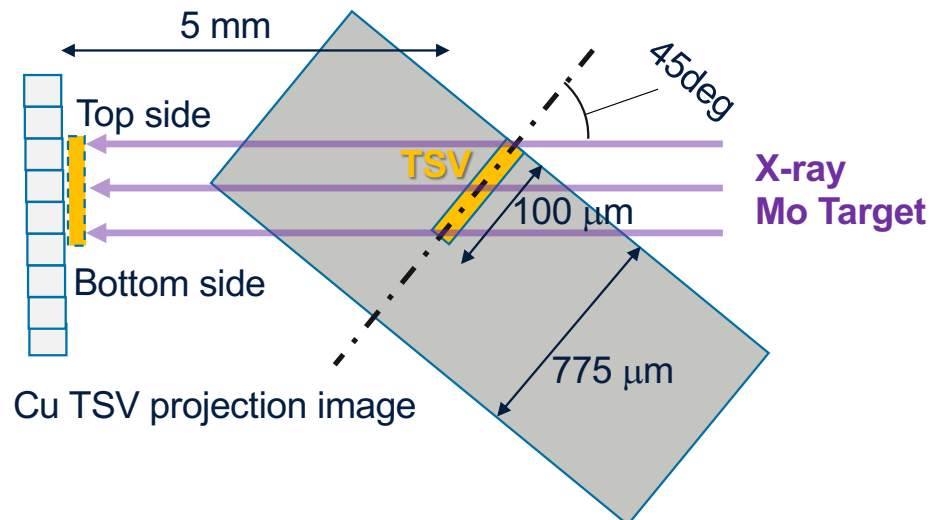
# Needs for Visualization of Buried Metal Structure



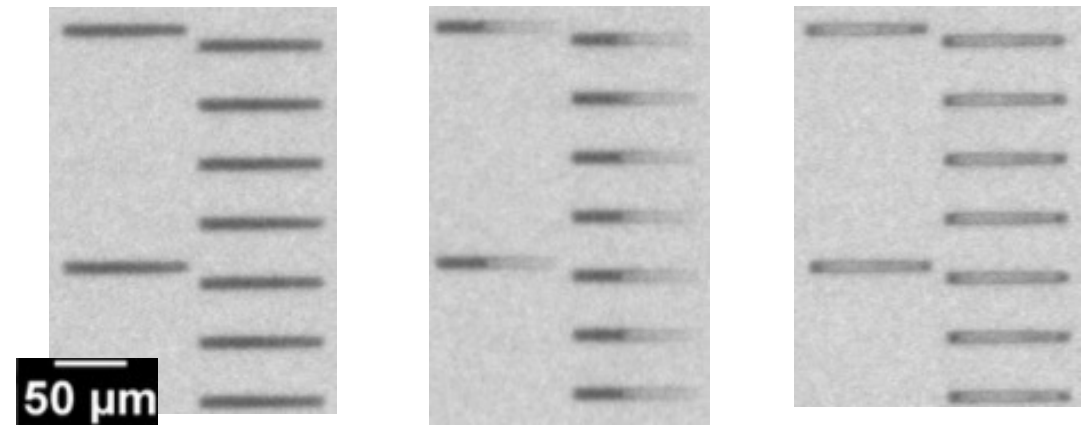
<https://www.inrevium.com/pickup/tsv/#03>



## Measurement geometry



## X-ray projection images for TSVs



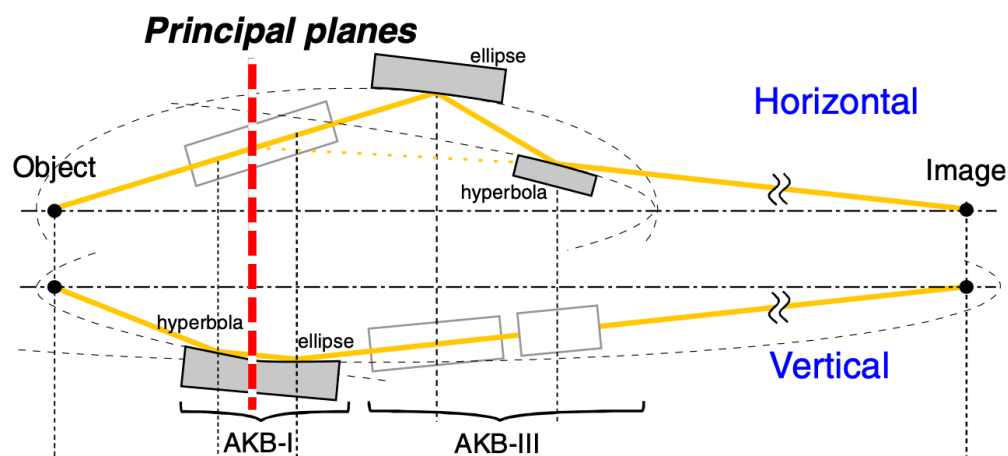
**Cu filling structures are clearly visible**

# Developing Reflective Imaging Lens for Hard X-Ray

## X-ray imaging lens

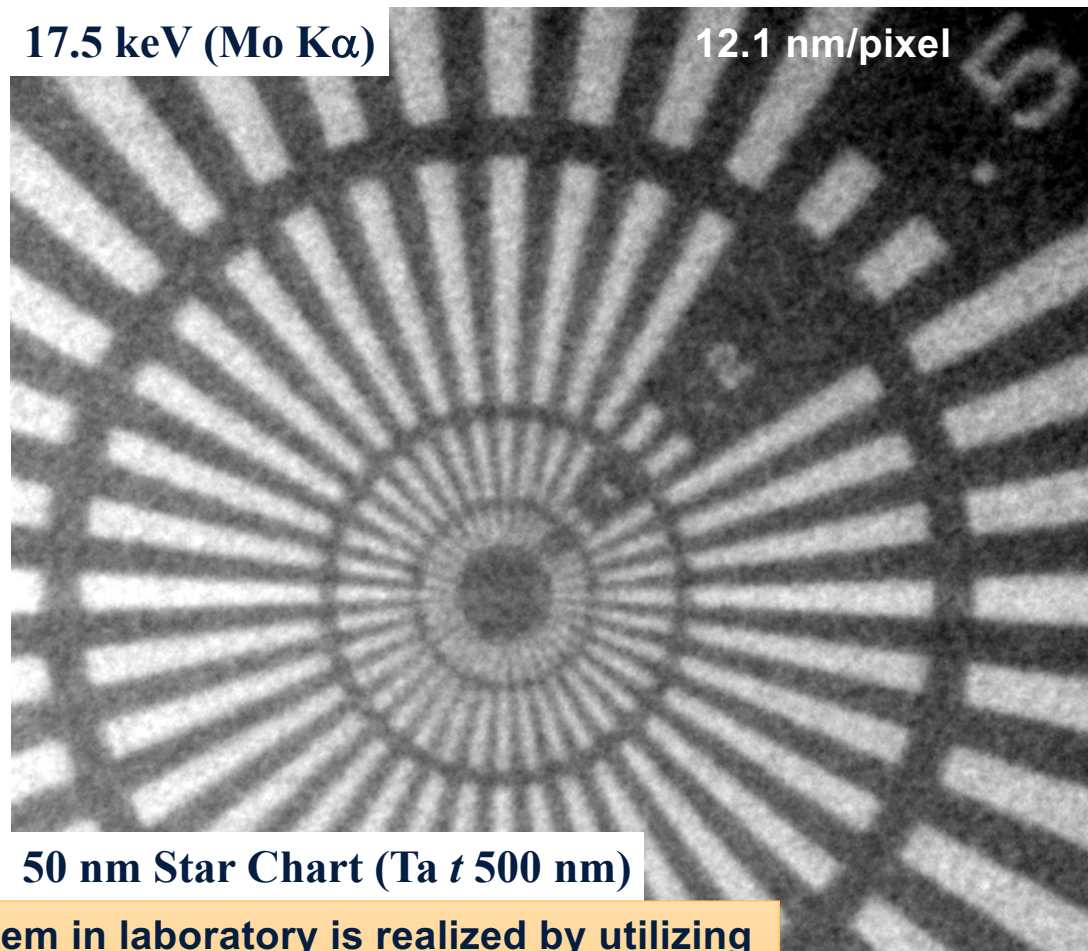


Advanced Wolter type KB mirror  
for 17.5 keV X-ray



17.5 keV (Mo K $\alpha$ )

12.1 nm/pixel

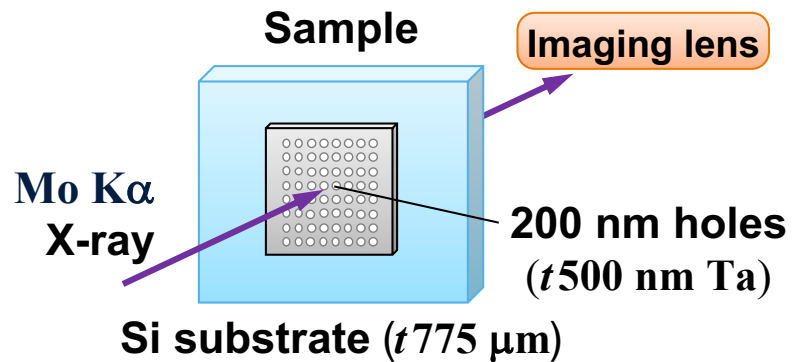
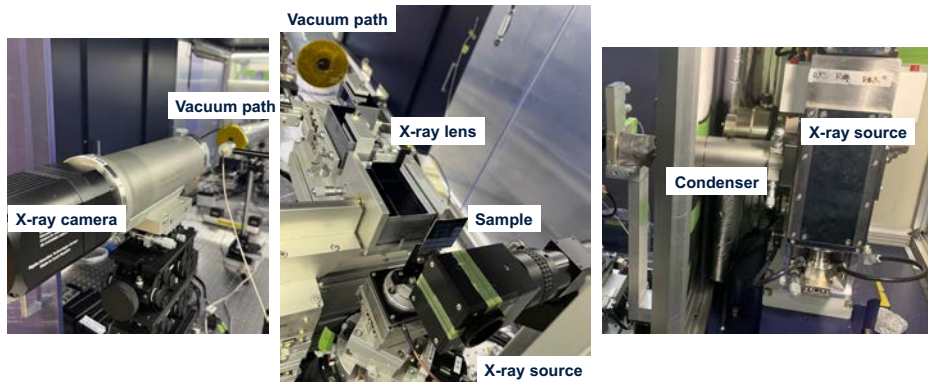


50 nm Star Chart (Ta  $t$  500 nm)

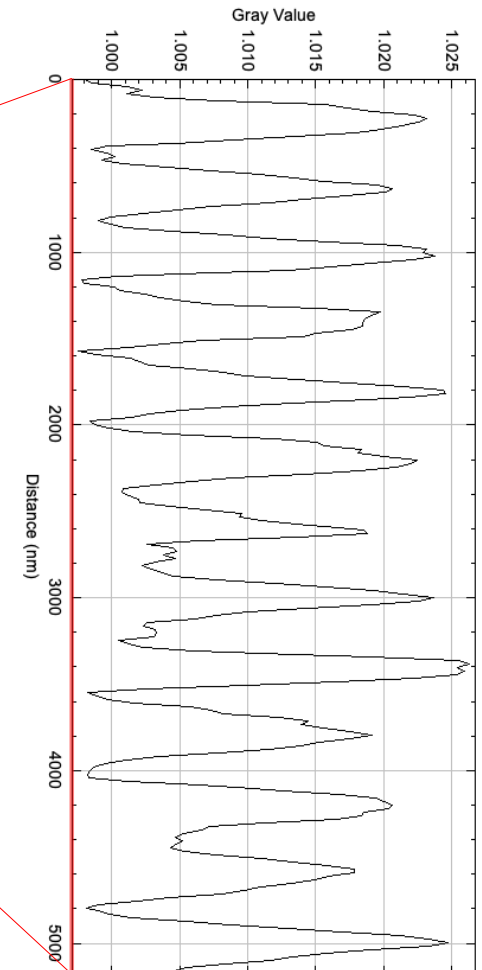
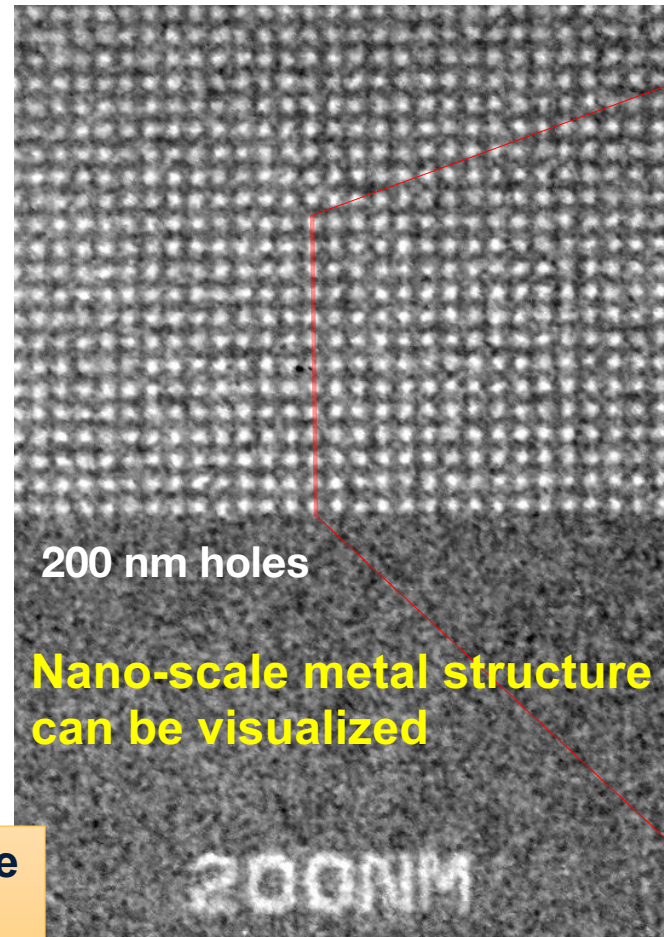
Hard X-ray nanoscale imaging system in laboratory is realized by utilizing reflective lens and High-brightness X-ray source



# Nanometer Resolution Hard X-Ray Imaging in Lab



We can measure nanoscale metal structure without destroying Si substrate



# Future Challenges

## ➤ X-ray Diffraction/Scattering

- ✓ Measurement for thinner film crystallinity, more complex film characterization, e.g. GAA
- ✓ Higher sensitivity for crystal distortion and identification of the defect species
- ✓ Shorter footprint for Grazing Incidence SAXS
- ✓ More accurate shape analysis for HAR structures by Transmission SAXS

## ➤ X-ray Imaging

- ✓ Higher resolution and shorter exposure time

**Continuing improvements for X-ray sources, optics, and detectors are the key!**