

Recent development of HAXPES instrumentations at BL-47XU/SPring-8

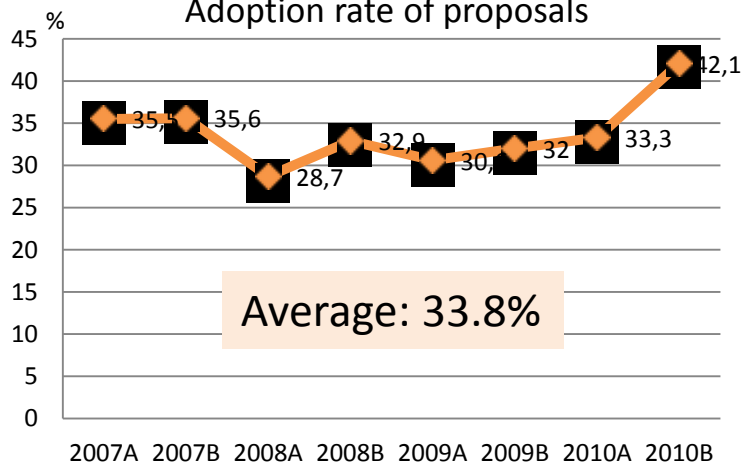


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H. Daimon³, K. Kobayashi²

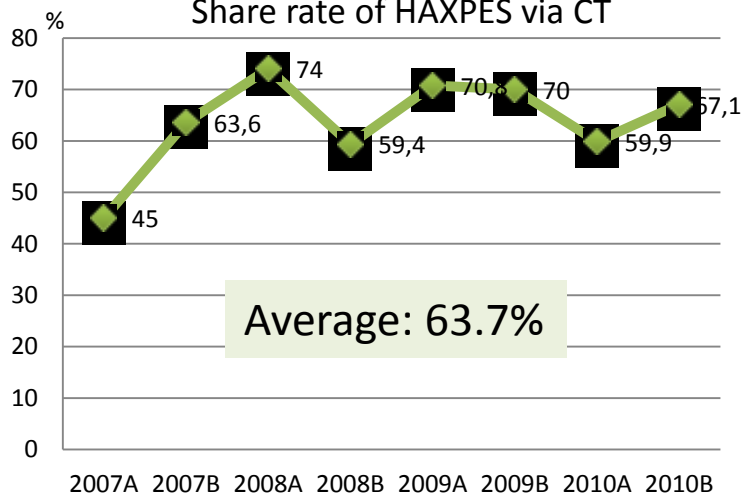
1 SPring-8/JASRI, 2 NIMS Beamline Station, 3 Nara University

Research Market in HAXPES : BL47XU

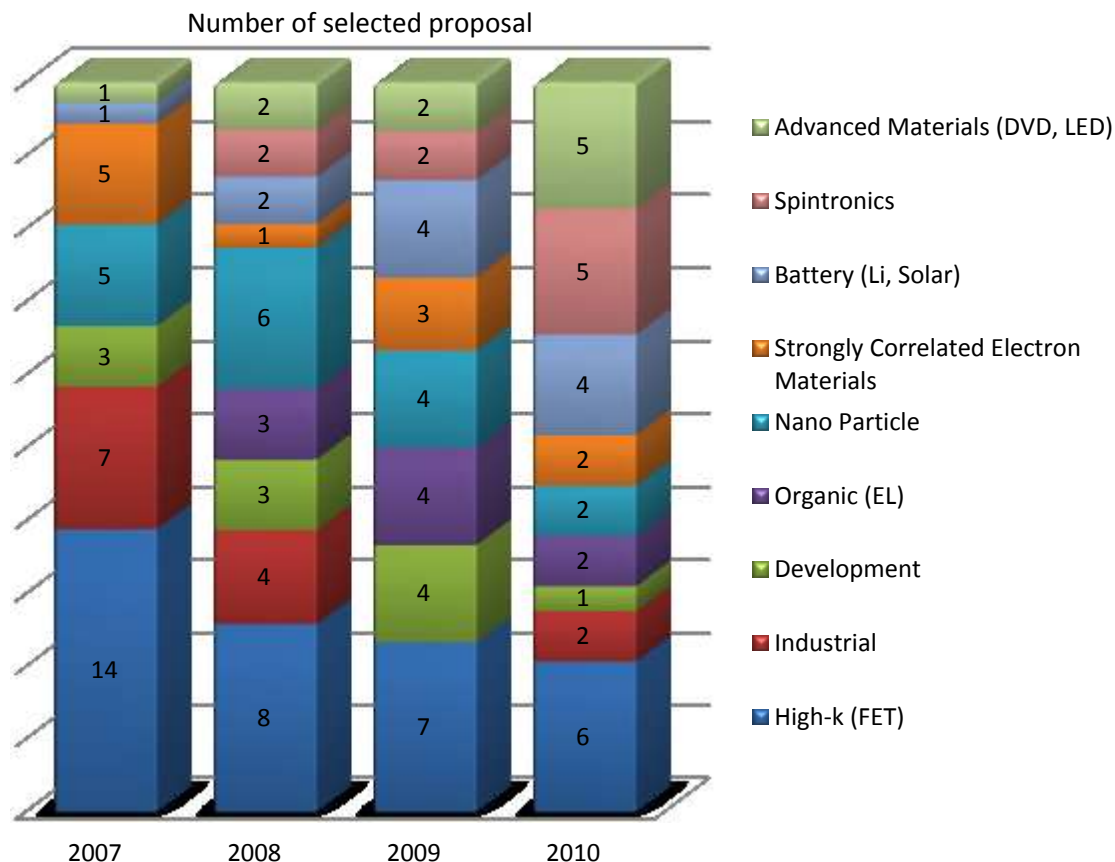
Adoption rate of proposals



Share rate of HAXPES via CT



Research field for 4 years



Researches in Battery(Li, Solar), Spintronics, Advanced Materials(DVD, LED) are increasing.

HAXPES of BL47 has high popularity.

Recent Developments of HAXPES : BL47XU

MCD-HAXPES for Spintronics

200 μ m (100) Diamond @ 8.0 keV **Phase Retarder**



Horizontal Polarization

Linear,
Circular Polarization

- Rapid helicity reversal
- $P_c > 0.95$
- $P_{lin} > 0.90$

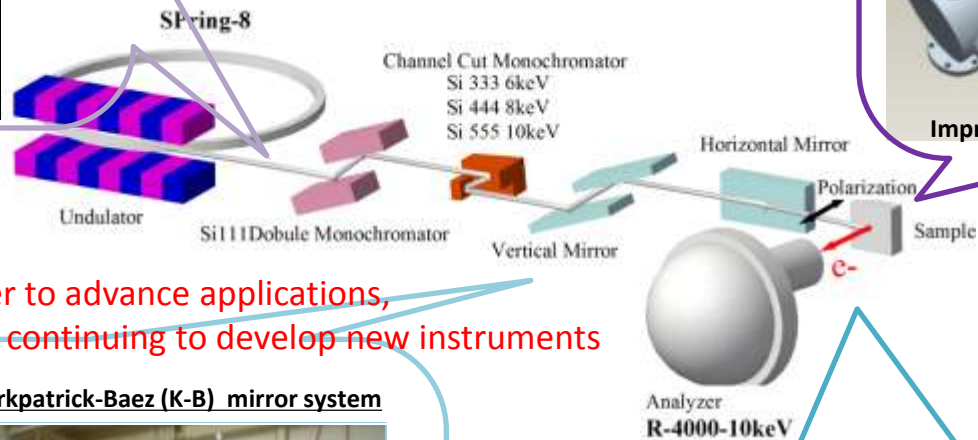
These development was supported by DFG-JST.

Project Leader: Prof. C. Felser

Aims:

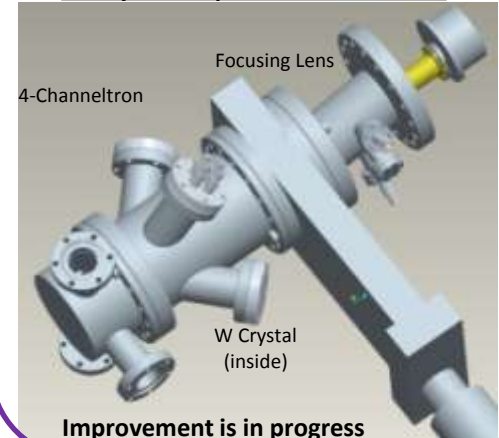
#Development of a new method, spin-resolved HAXPES, for magnetic properties

The SPLEED detector detects the spin polarized secondary electron emitted from the tungsten crystal



SPIN resolved HAXPES

Newly Developed SPLEED Detector

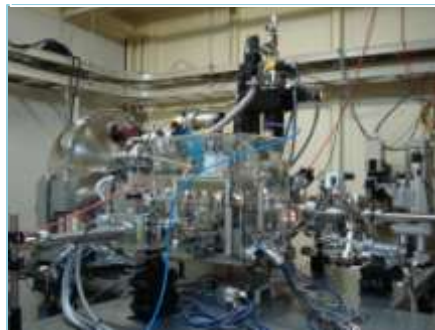


In order to advance applications,
we are continuing to develop new instruments

Focusing optics



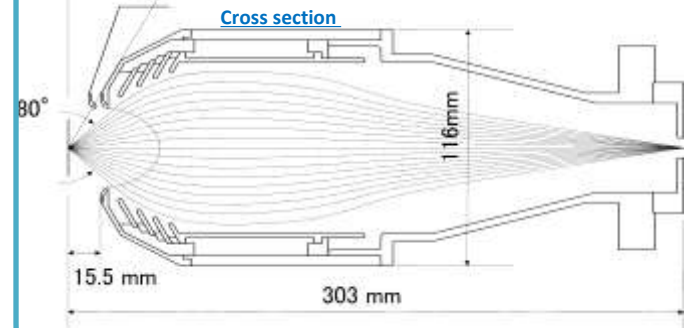
Kirkpatrick-Baez (K-B) mirror system



- Focusing size: $\phi 1\mu$ m

Wide Acceptance Angle

Wide Acceptance Angle Objective Lens

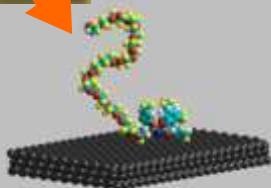


- Acceptance Angle: $\pm 30^\circ \leftarrow \pm 7^\circ$

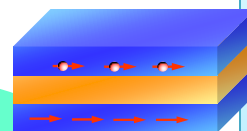
These development was supported by SENTAN, JST.

Project Leader: Prof. K. Kobayashi.

Our targets



Organic Materials



Strained Manganite Thin Films

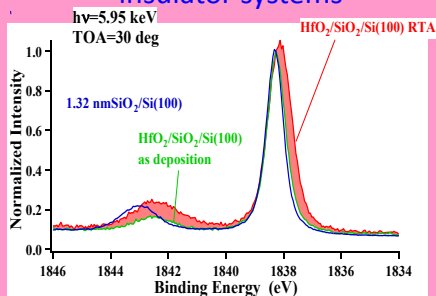
Fundamental Science

Industrial application

Applied Science

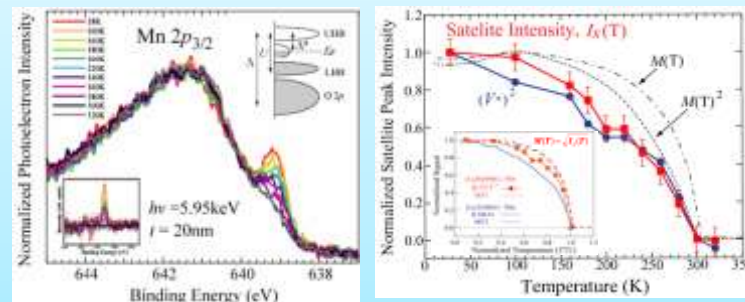
Applied nanotechnology

Typical result Interface reaction of poly-Si/high-k insulator systems

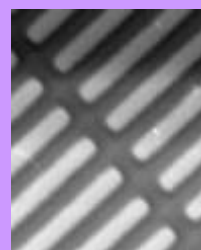


The depth probe is analyzed in high accuracy.

Typical result Electronic Structure of Strained Manganite Thin Films with Room Temperature Ferromagnetism ($\text{LaBaMn}_3/\text{SrTiO}_3$)



The relation that corresponds to temperature-magnetization curve is experimentally clarified.



Transition Metal of Ferromagnetic Oxide



Nano-DNA Chips
(Kawai-Lab nano-bio-chip group)

- These advanced materials has nano or micro structures.
Request of observation in the microscopic area is increased.

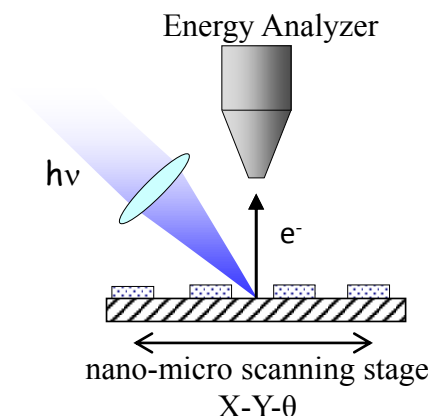
Purpose

Achievement of the **3D chemical states analysis** by scanning sample in microscopic area and taking depth profiles

Scanning (in-plane 2D) + take-off angle (depth 1D)

Plan

Scanning type microscope

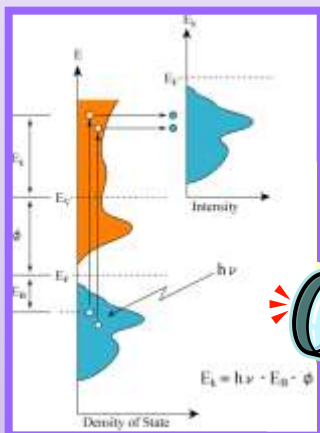


1. Introduce focusing optics

- Standard beam size **V: 40μm & H: 30μm**

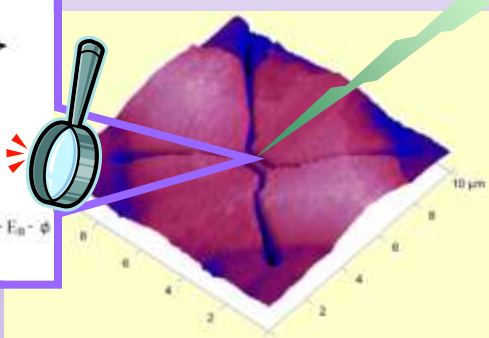
After installation K-B Mirror,
Status of beam spot size

⇒ **φ1μm ~ submicron**



Electronic state of
microscopic area

K-B Mirror

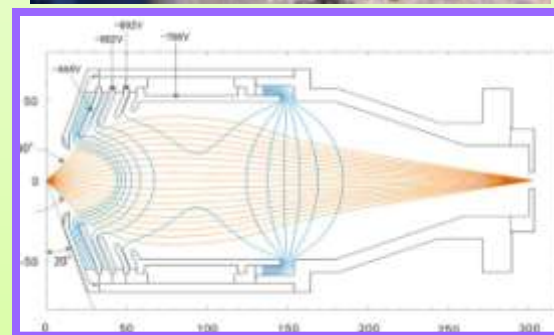


This development of scanning type of photoemission microscope can obtain the electronic states and images in microscopic area.

2. Development of Objective lens

Analyzer lens of solid angle: **±7°**

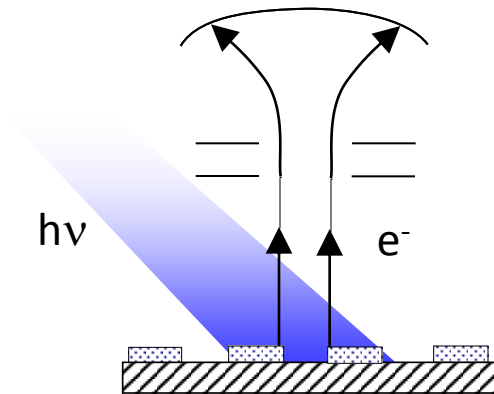
Wide acceptance angle



What is the difference with PEEM ?

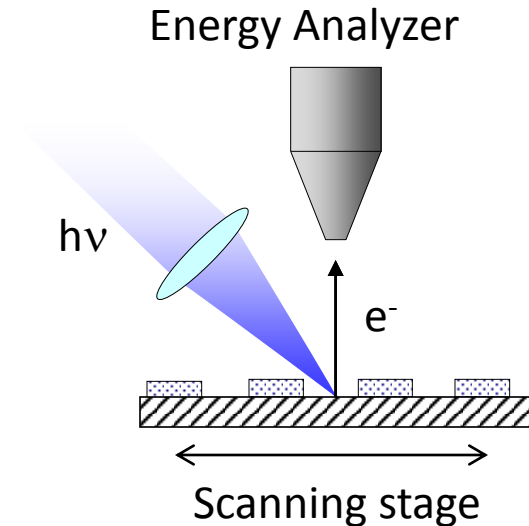
Photoelectron emission microscope (PEEM)

Magnification/Energy selection



- High lateral resolution < 10 nm
- Moderate energy resolution > 0.3 eV

Scanning type microscope Sample scanning (x-y)



- Lateral resolution is limited by spot size
- Advantage for the scanning type microscope (High energy resolution)

High-energy resolution is required
for resolving lateral dependence of electronic structure.

Lateral Resolution of Analyzer Photoelectron Detection

Au micro Dot on Si wafer

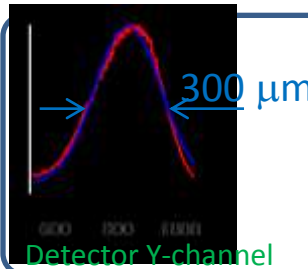
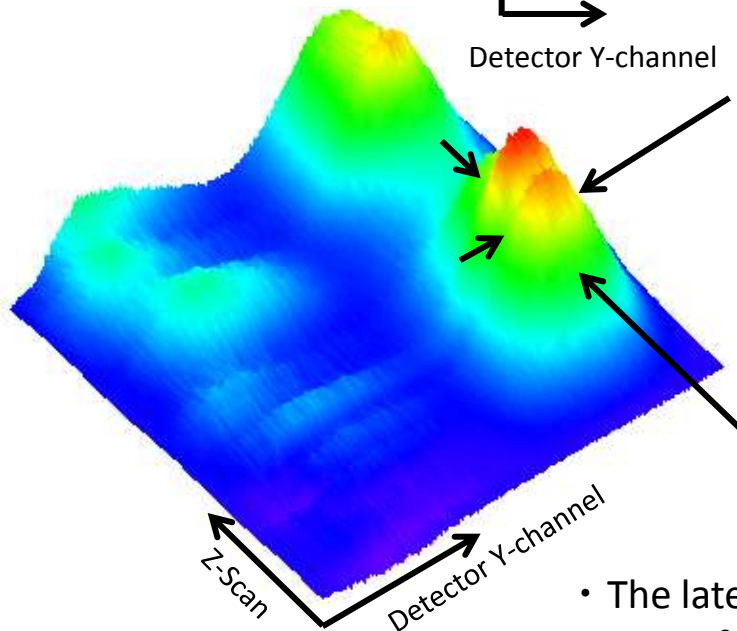
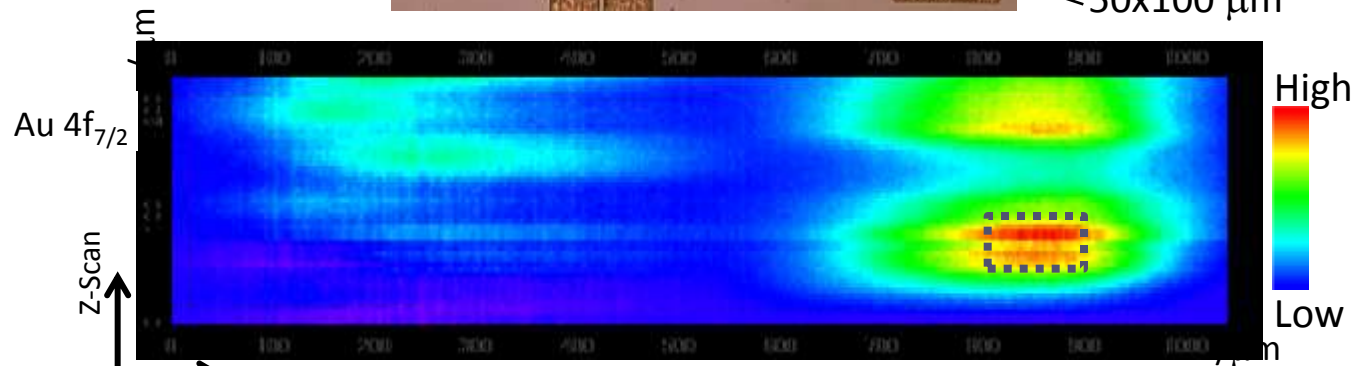
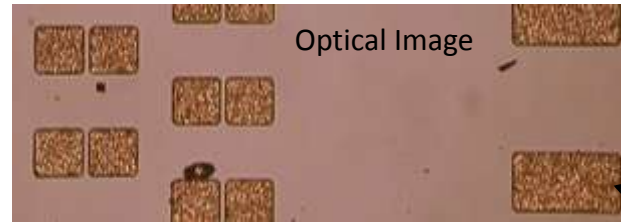
Transmission Mode

Focusing size: **V: 40 μ m** **H: 30 μ m**

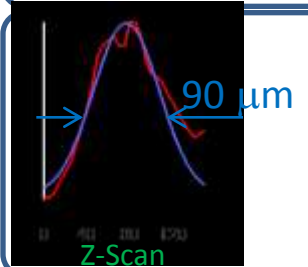
TOA: 88.5, Scan Step: 5 μ m,

CCD Y-channel array:

1.35 μ m/pixel



Broadening by Lateral Resolution of Analyzer Photoelectron Detection
magnification: 25 with Objective Lens, Phosphor Screen & CCD Detection System)



Convolution of
Au Dot Size & Beam focusing Size
50 μ m 40 μ m(FWHM)

- The lateral resolution of 2-D detection system is poor
- Beam focusing is important for well-resolved image with high energy resolution.

Development of K-B mirror system

K-B mirror system

elliptical mirrors; figure errors ~ 2 nm; platinum coating;
glancing angle ~ 3 mrad; incident slit: $300 \times 300 \mu\text{m}$

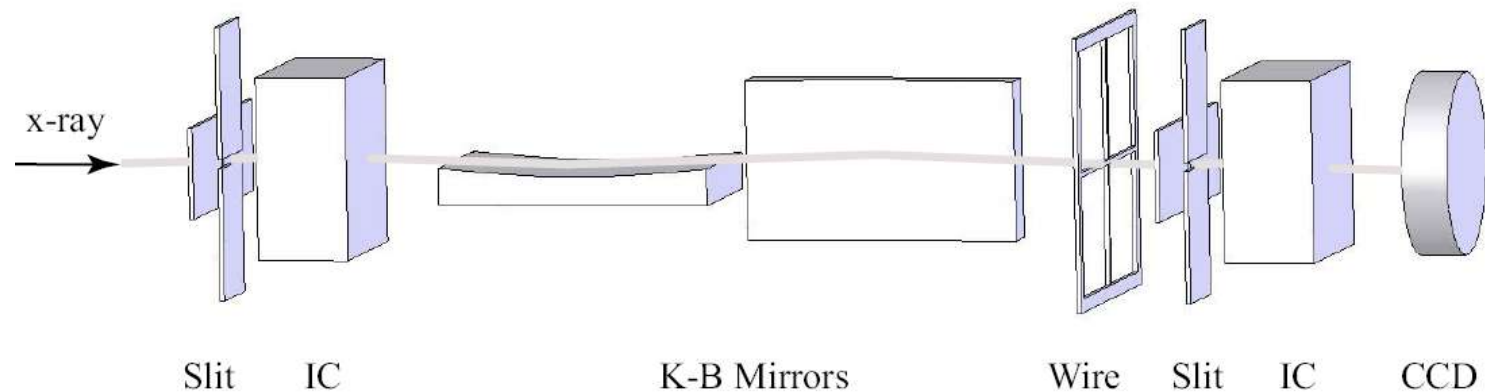
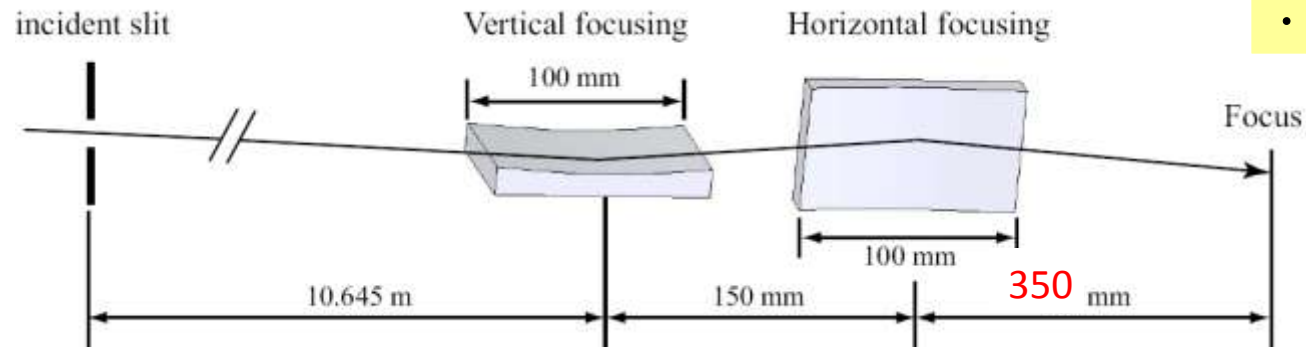
- Working distance:

350mm (BL47XU)

- Beam size: $1\mu\text{m}$

- **Demagnification : 1000**

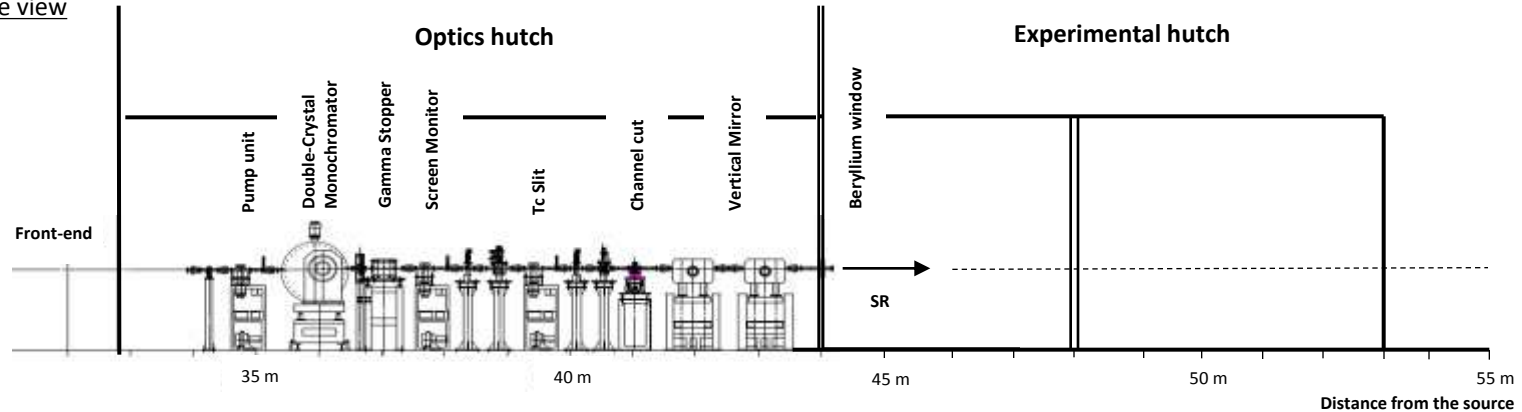
- **Gain: $10^{12\sim 13}$**



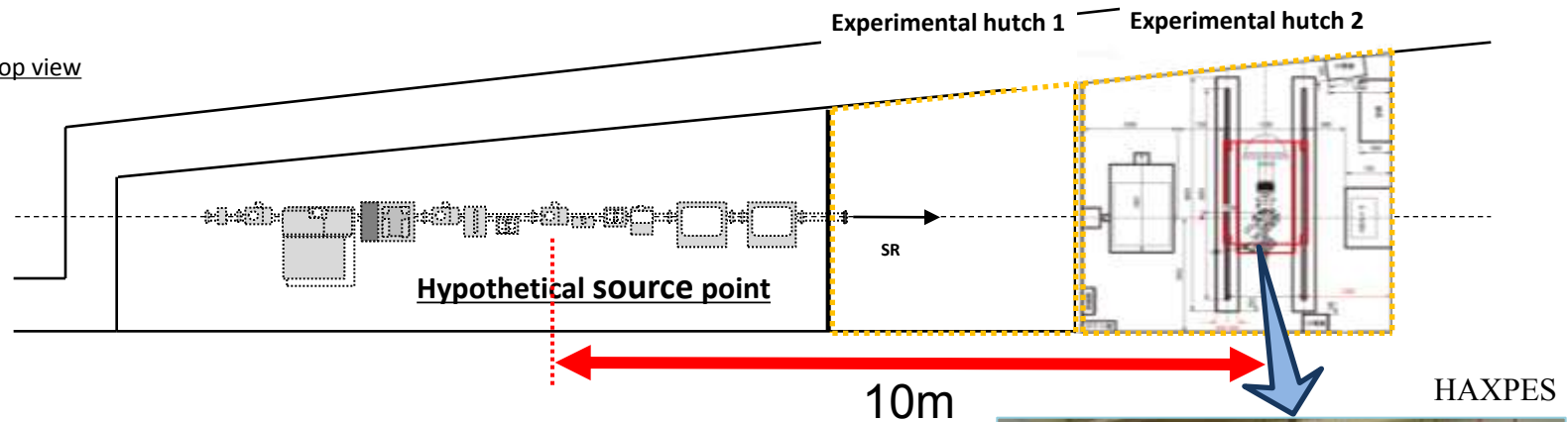
Extended working distance leads to wide space around sample

Development of K-B mirror system

Side view



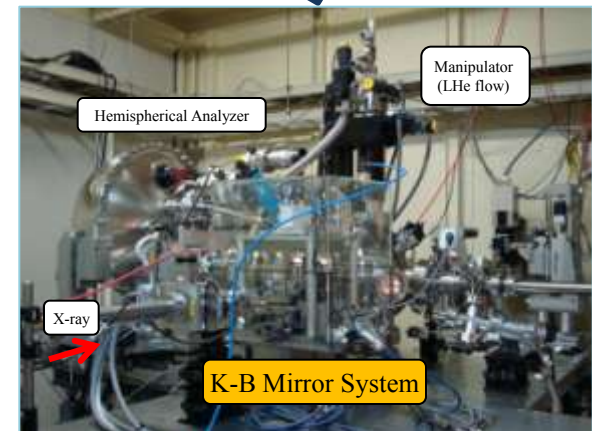
Top view



- In-vacuum planar undulator, 5.9 ~18.9 keV
- Si (111) double crystal monochromator
- liquid nitrogen cooling (not closed cycle system)
- Si (333) channel cut

High Energy Resolution (Band Width=60meV

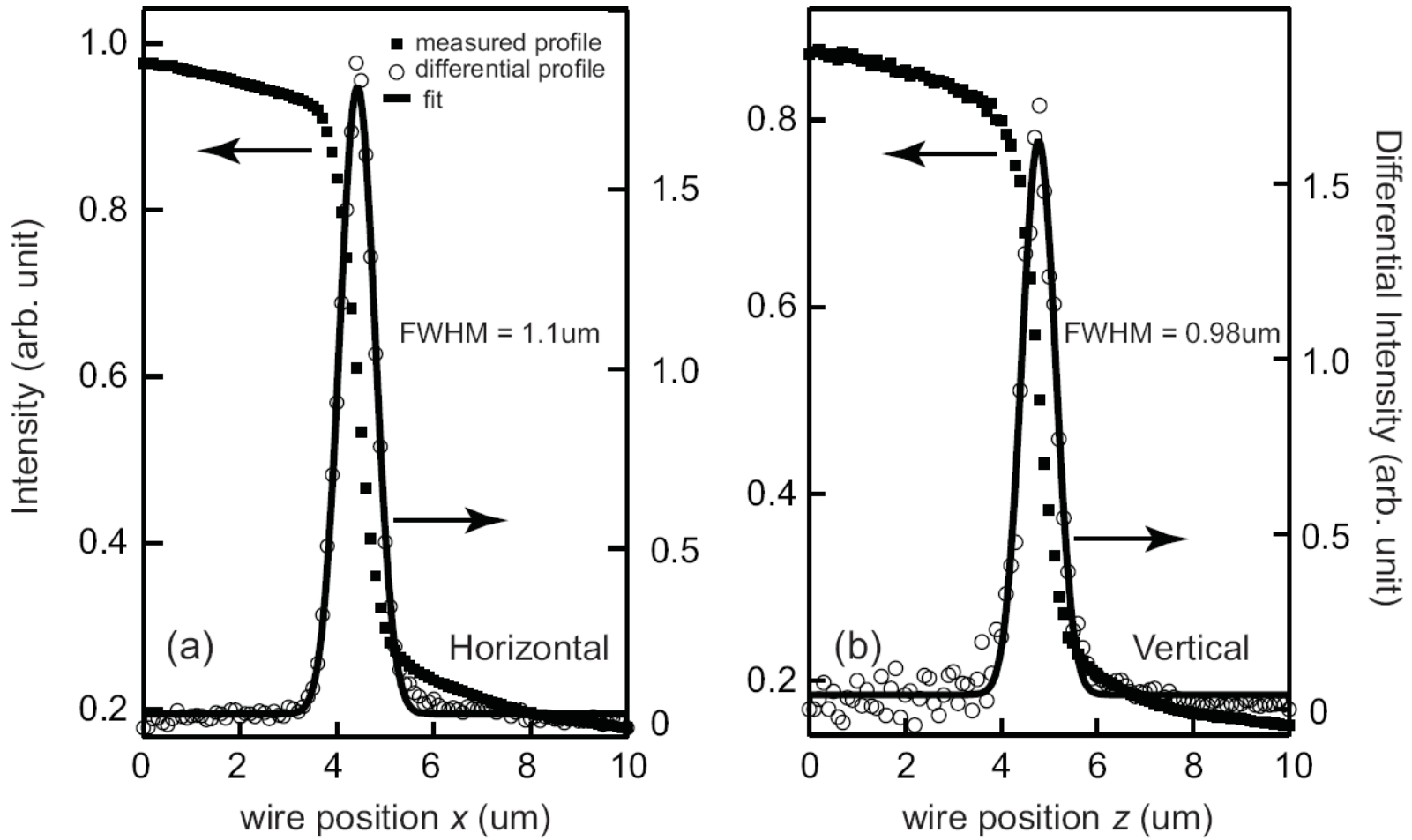
@8keV)



Development of K-B mirror system

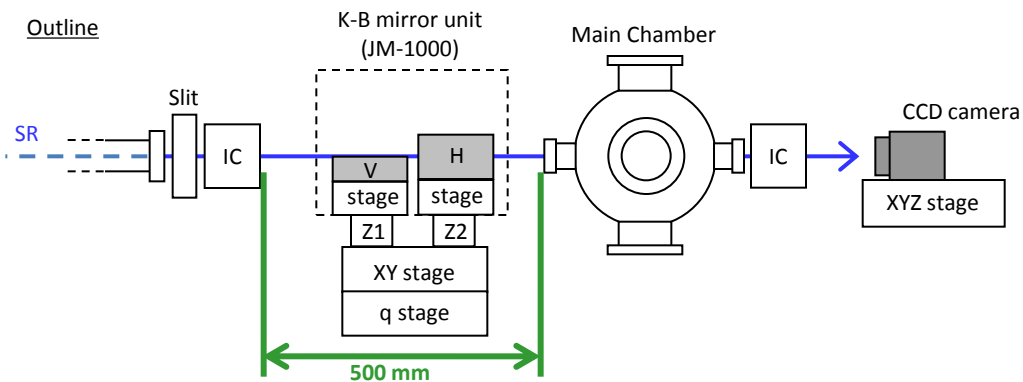
Focusing Result (Photon Energy=7.94keV)

Using Wire Scan monitor



Beam size: $1.1\mu\text{m}$ (H) \times $0.98\mu\text{m}$ (V)

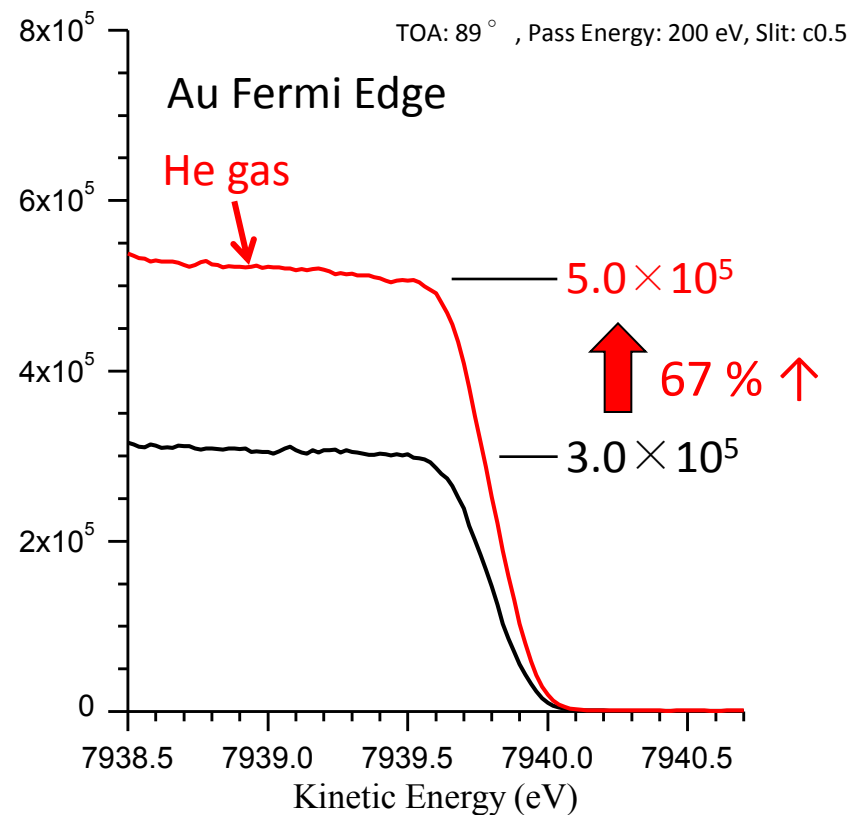
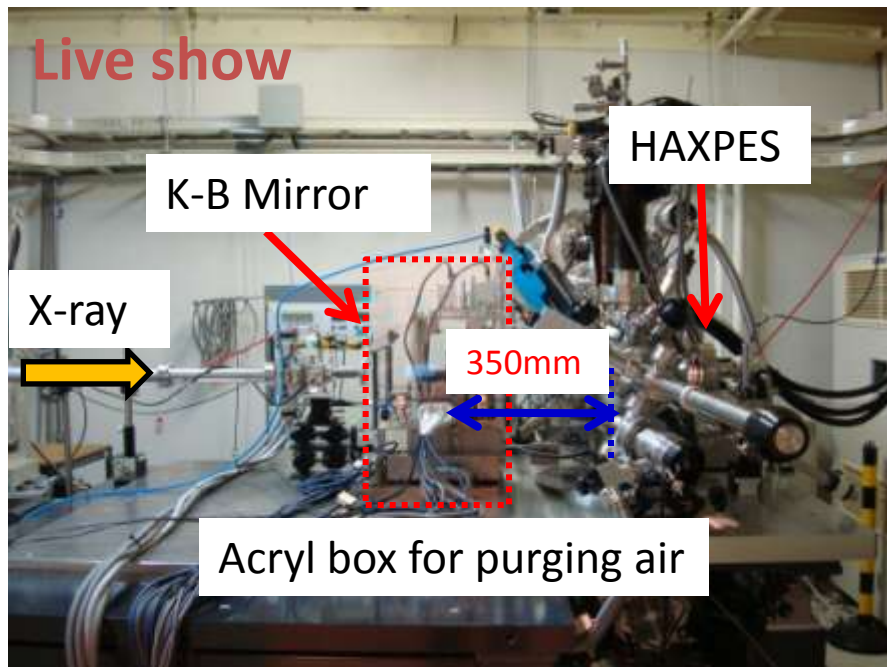
Development of K-B mirror system



Increasing of Photon Flux

8.1e+10 \rightarrow 1.3e+11 (photons/s)
63 % \uparrow

Improvement of signal intensity by replacing the air path of the KB mirror with helium gas



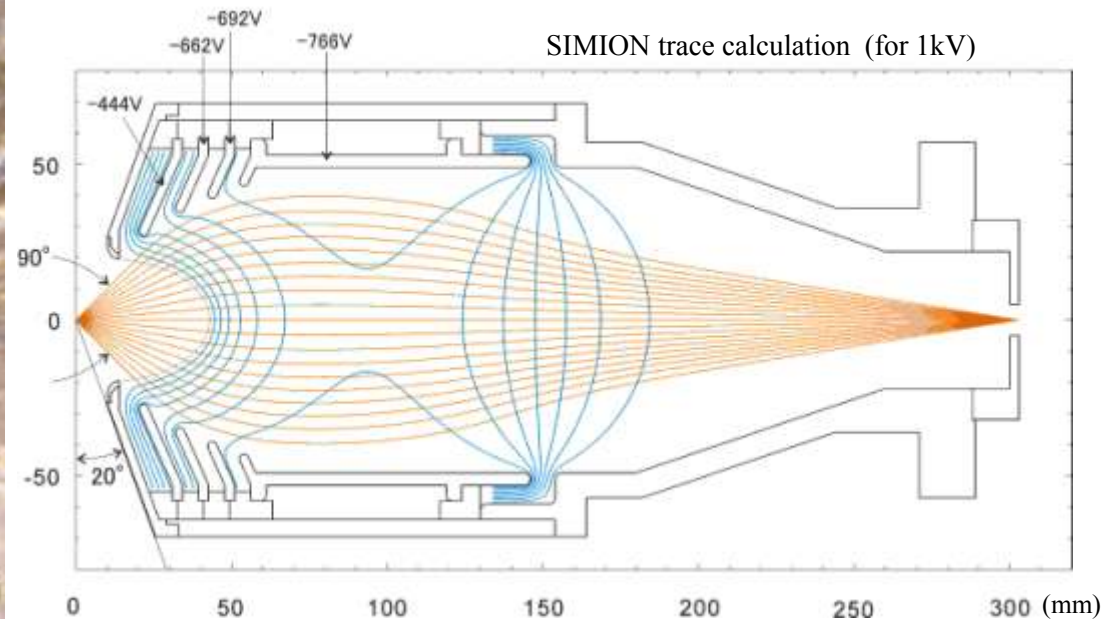
This result confirms linearity of signal intensity against photon flux is good enough.

Wide Acceptance Angle Objective Lens

In Collaborate with Drs. Daimon and Matsuda

About withstanding voltage • • •

Stable operation under **8kV application** without discharge.



Main feature • • •

- Discharge prevention measures by simplification of correction electrode part structure
- **The entrance shape mesh of a spheroid type**
- **Working Distance=15mm**
- **Magnification factor: 5**
- **Total magnification factor becomes 25**
by combination of this objective and the analyzer lenses



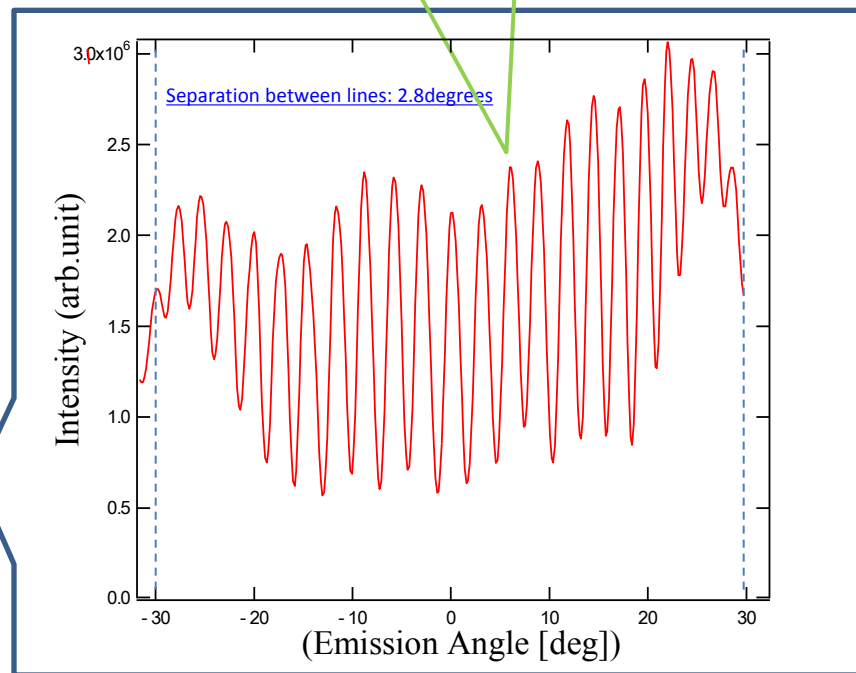
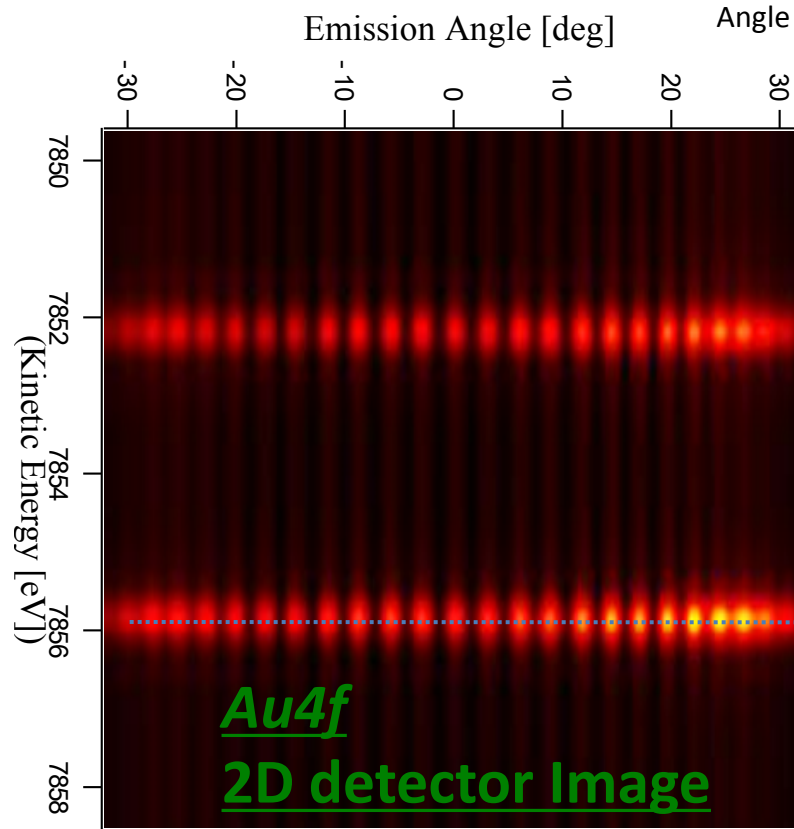
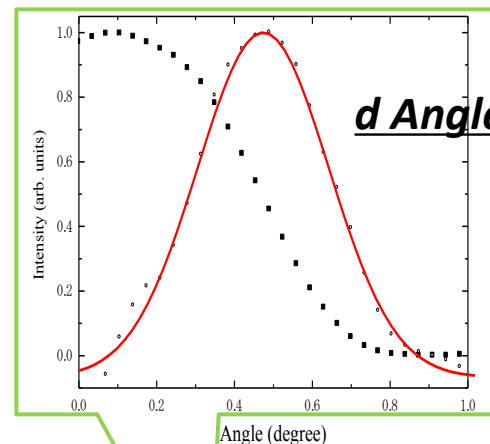
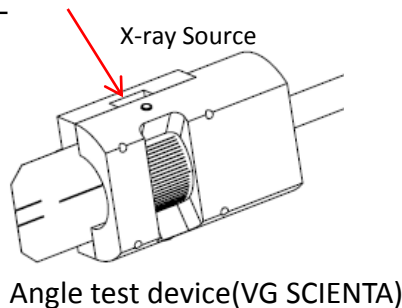
Wide Acceptance Angle Objective Lens

(Evaluation of acceptance angle)

Photon Energy=7.94keV

PE=200eV, C0.5

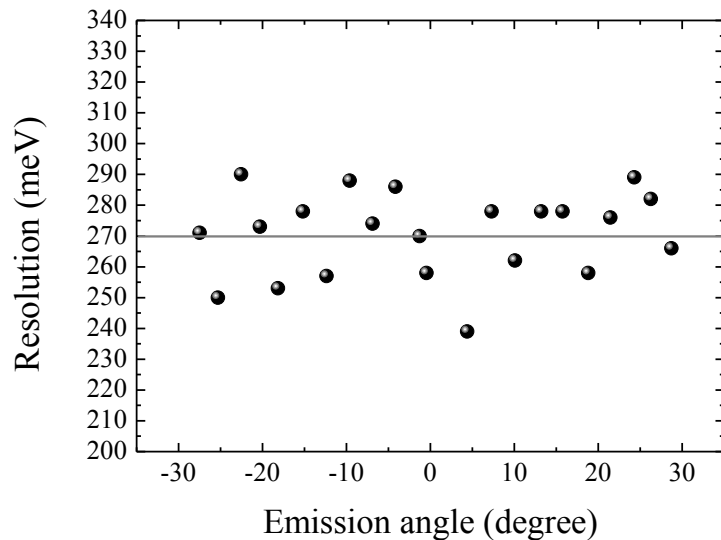
TOA85deg, Lens mode: Angular



Total Acceptance angle ± 30 degrees

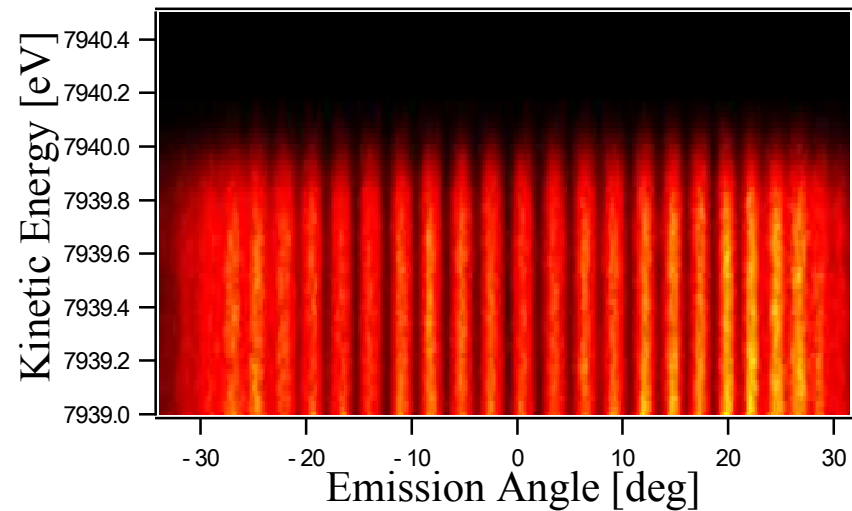
Wide Acceptance Angle Objective Lens

(Evaluation of Energy Resolution - aberration)



270 ± 20 (meV)

Fermi edge of Au

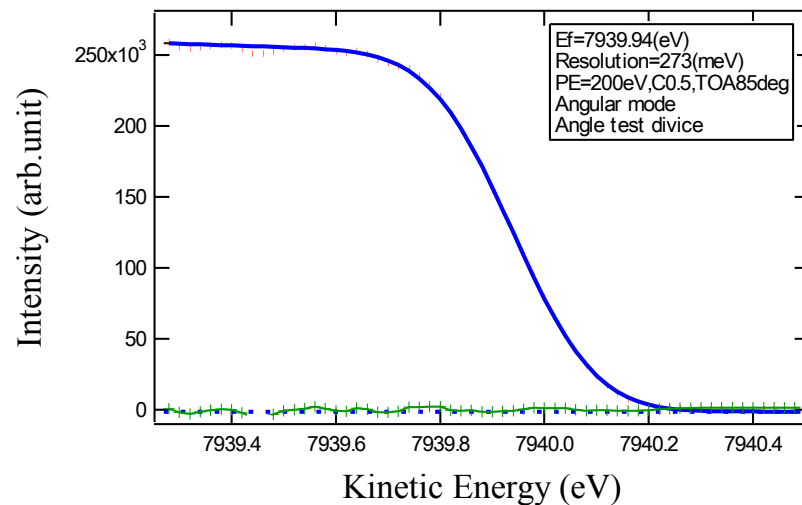


Aberration due to the bend of shape mesh in lens



Decrease of energy resolution

**Decrease of energy resolution with
objective lens is suppressed within 20 meV !**



Total Resolution=275meV

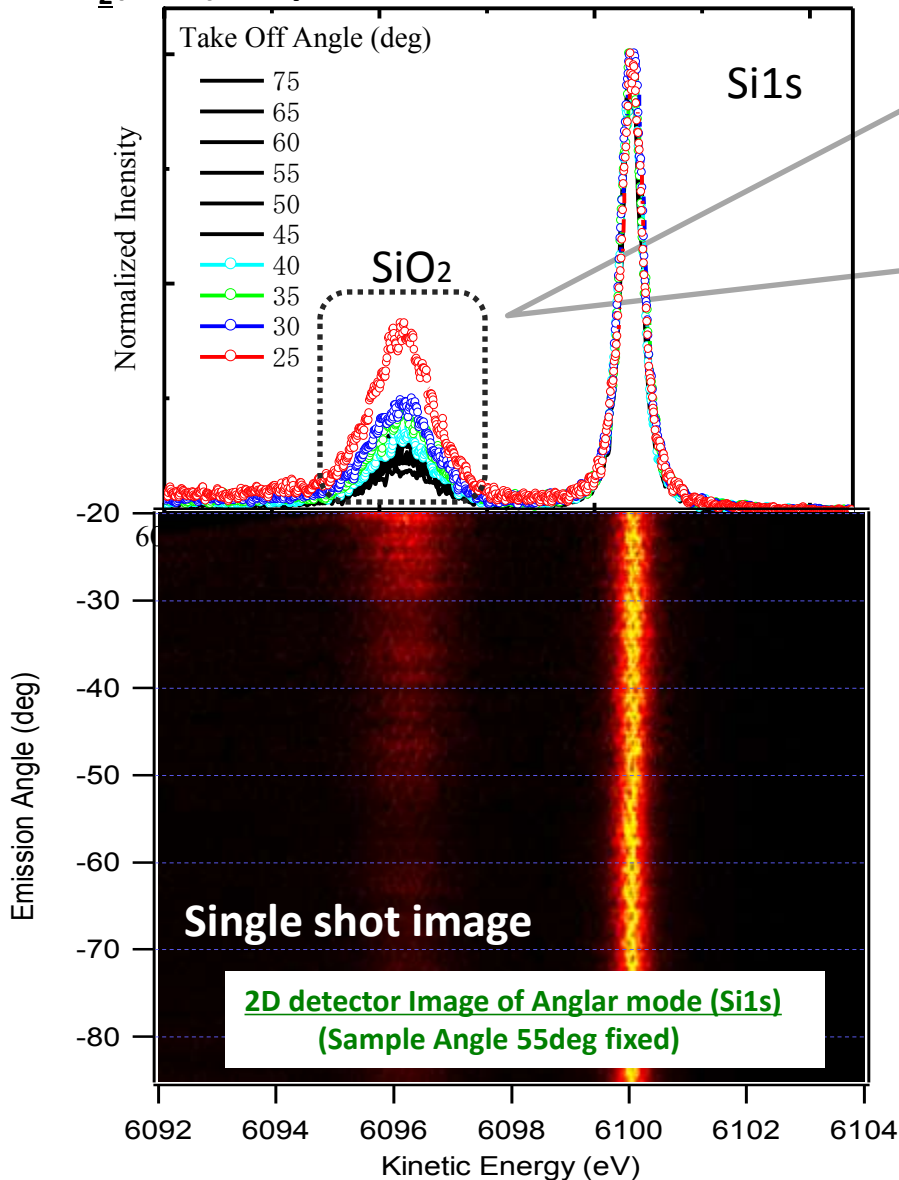
Wide Acceptance Angle Objective Lens

(Evaluation of Depth Probing)

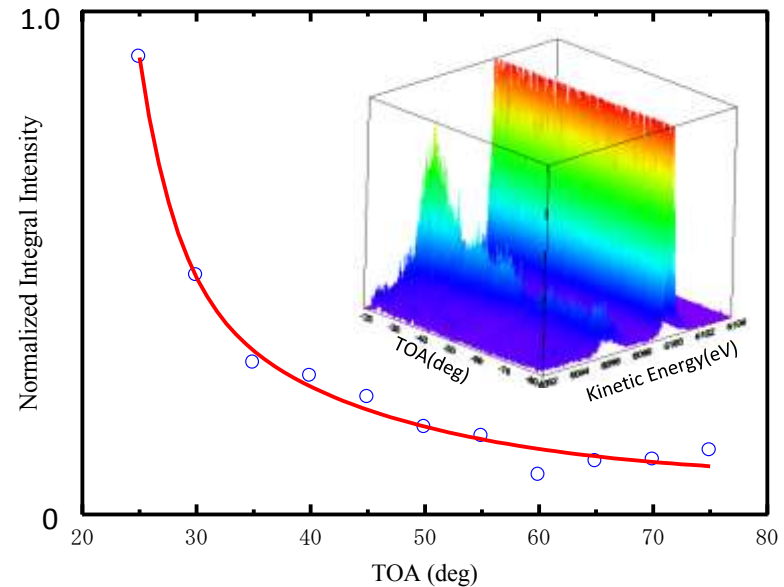
SiO₂(4nm)/Si spectra

depth probing in Si 1s spectra of 4nm SiO layer on Si substrate.
Normalized by substrate Si1s intensity

Photon Energy=7.94KeV
Pass Energy=200eV
Slit:=C 0.5



TOA Dependence of Si Oxide



Oxide component increases with decreasing TOA



Wide Depth profiles can be acquired at once
without changing the angle of the sample!

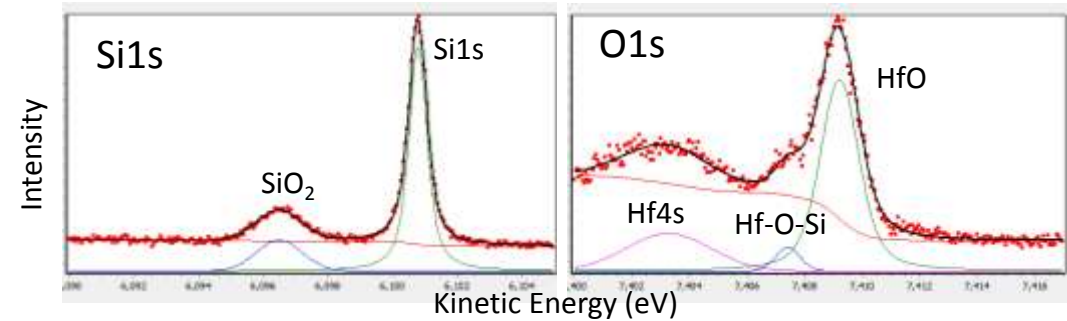
- Wide Depth Profiles -single shot image
- Efficiency 30times ↑
- 10 min acquisition

Wide Acceptance Angle Objective Lens

Photon Energy=7.94KeV
 Pass Energy=200eV
 Slit:=C 0.5

(quantitative analysis of thickness)

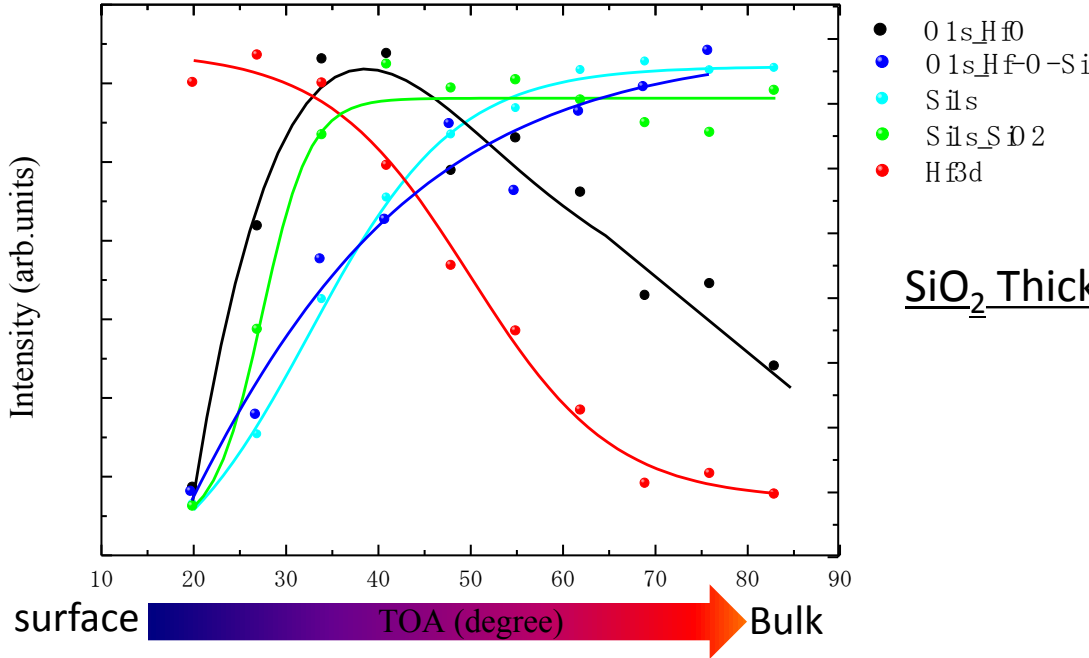
HfO₂(4nm)/SiO₂(1nm)/Si (The SiO 1nm thickness has been inspected by RBS)



$$d = \sin \theta \ln \left(\frac{N_s \lambda_s I_f}{N_f \lambda_f I_s} + 1 \right)$$

Atomic density	N/cm ³
Si(N _s)	5E+22
SiO ₂ (N _f)	2.27E+22
The attenuation length ※TPP2M Formula	(nm)
Si(λ _s)	6.4
SiO ₂ (λ _f)	9

Angle dependence of each components



SiO₂ Thickness

Thickness	Angle	
d(nm)	θ	If:SiO2/Is:Si
1.092	83	0.0831
0.996	76	0.0772
0.941	69	0.0757
0.941	62	0.0802
0.969	55	0.0897
0.925	48	0.0947
0.991	41	0.1166
1.037	34	0.1461
1.125	27	0.2024
1.021	20	0.2512

This is well consistent with the thickness result of RBS.

Average Thickness : 1.004nm

Combination of K-B mirror and Objective Lens

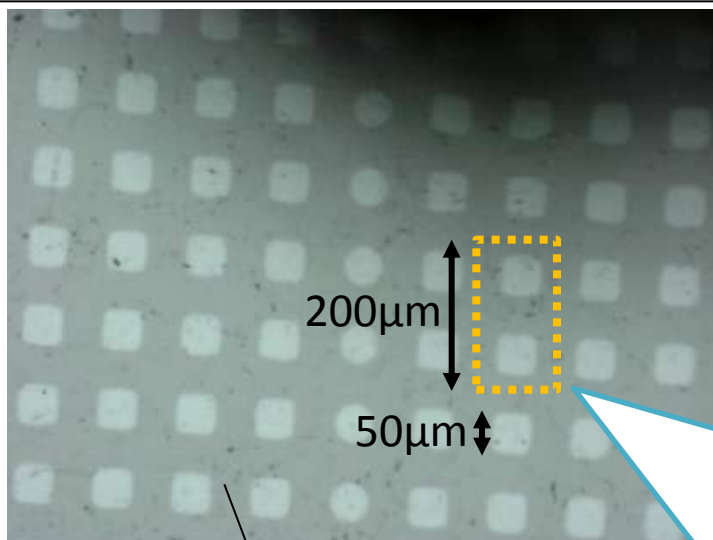
Photon Energy=7.94KeV

Pass Energy=200eV

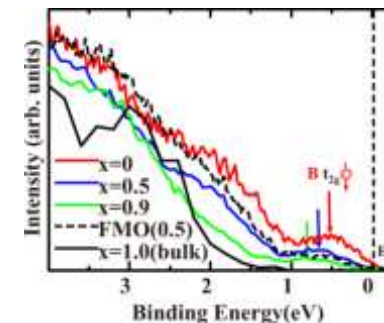
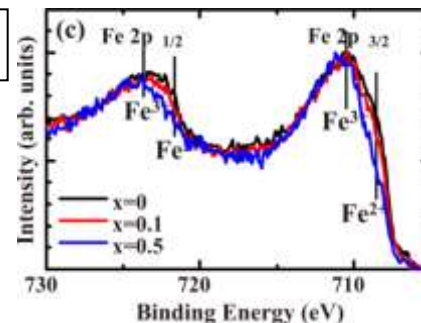
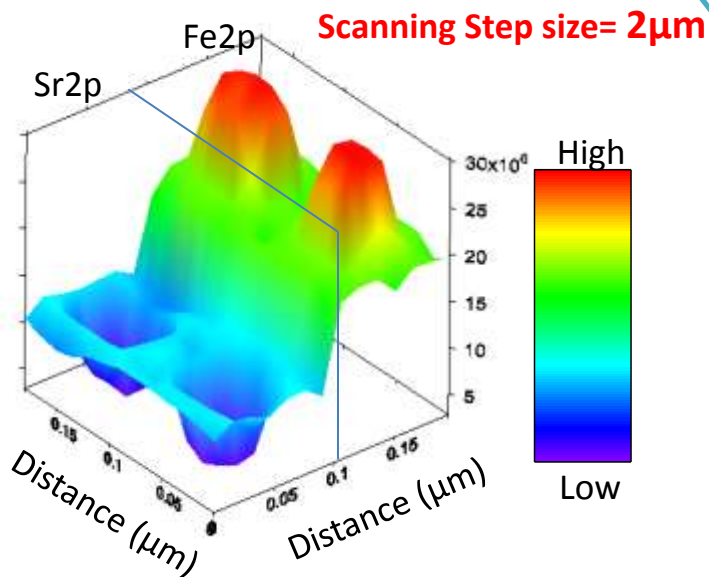
Slit:=C 0.5

(sample scanning -2D mapping)

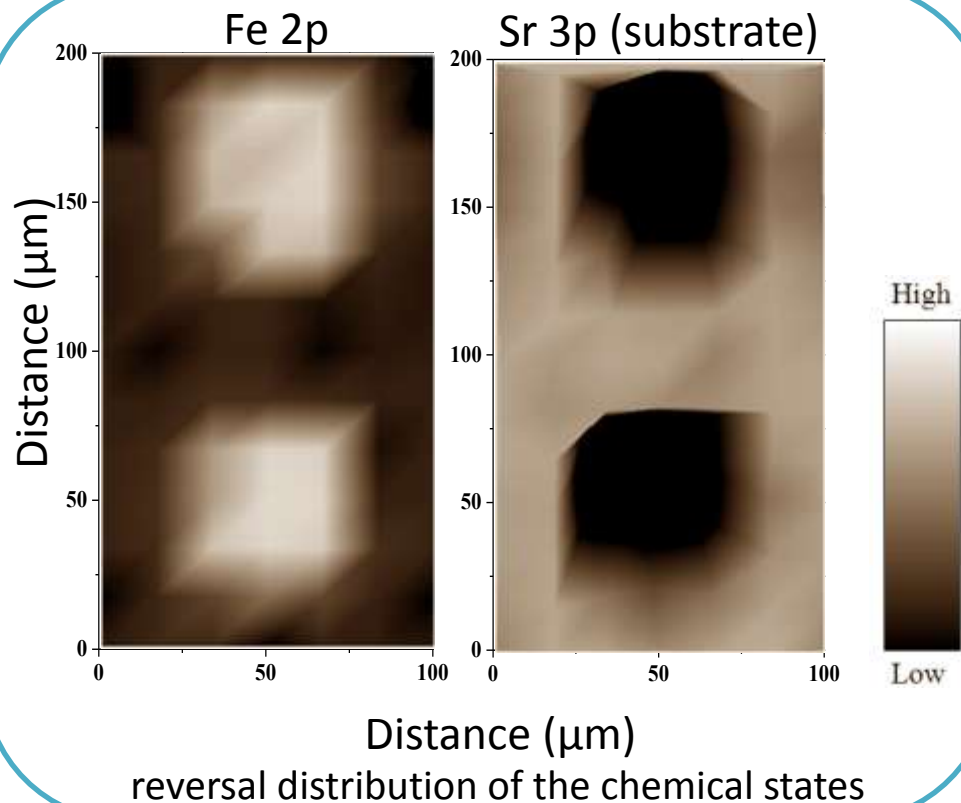
$(\text{Fe}_{2.5}\text{Mn}_{0.5})\text{O}_4$ dot $50 \times 50 \mu\text{m}$ thickness : 20nm



Substrate: Nb0.5wt%-SrTiO₃



Phys. Rev. B, 76 (2007) 205108

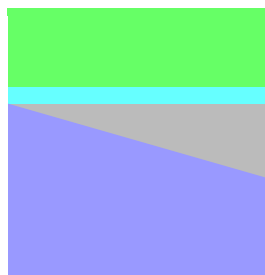


Micro dot is well resolved by using beam size of 1 μm.

Combination of K-B mirror and Objective Lens

Ir(8nm)/HfO₂(2.2nm)/SiO₂ (0-10 nm)/Si(100)

[4mm] ← [0mm] Scanning 200μm step



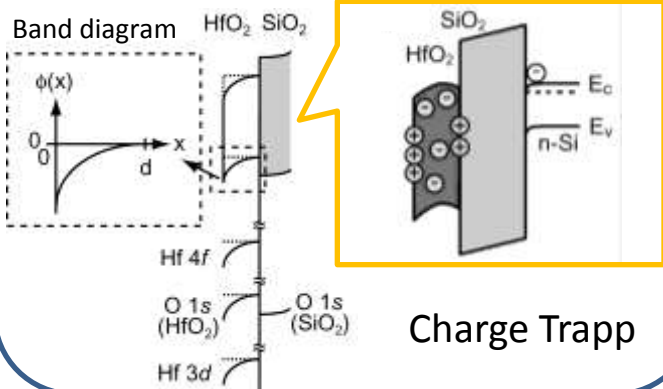
Ir(8nm)

HfO₂(2.2nm)

SiO₂(0-10nm)
thickness-graded

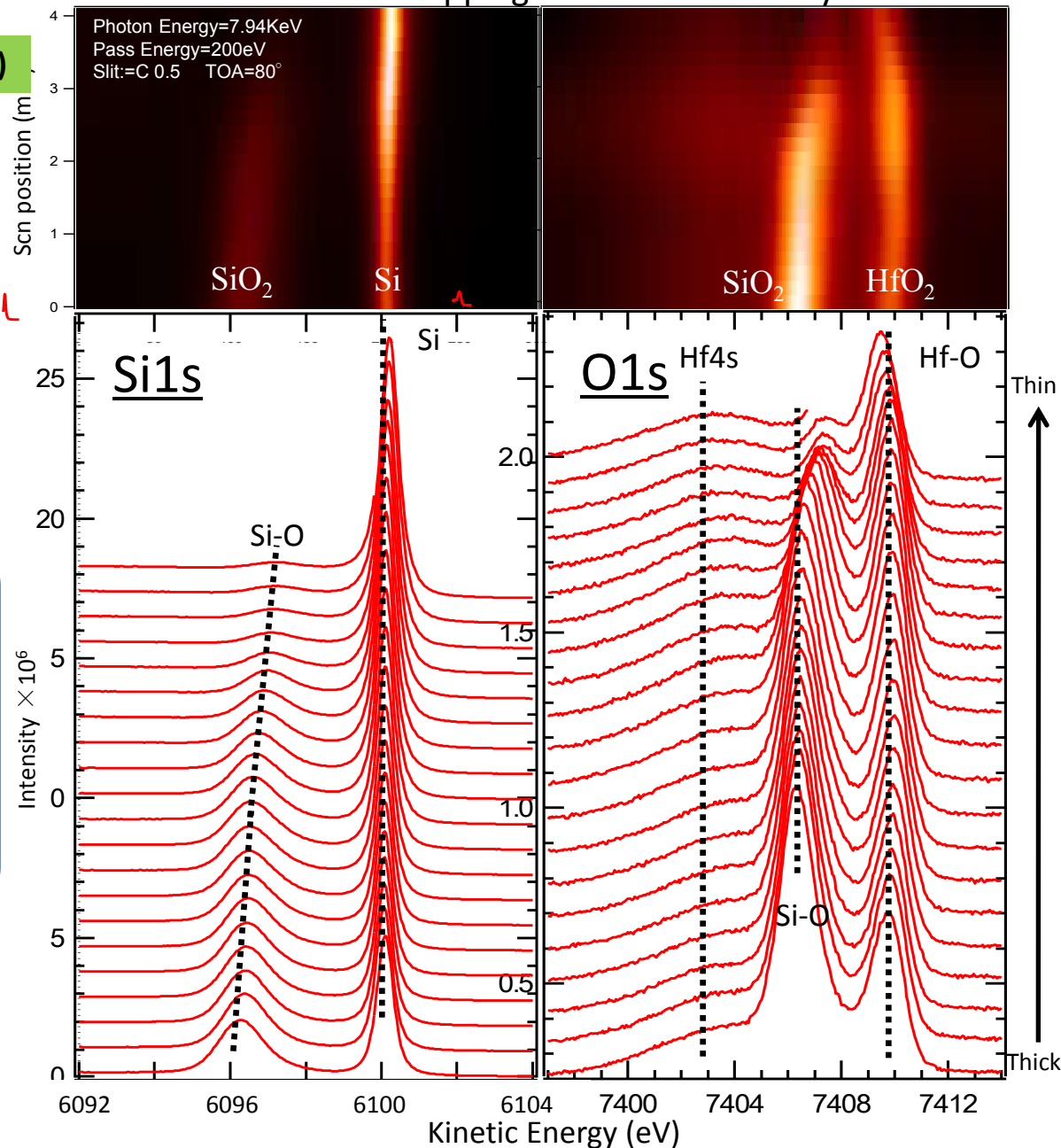
Si(100)

Band Bending in thickness dependence



These results indicate the band bending in thickness dependence caused by charge trap of interlayer

chemical state mapping result for a multilayer film

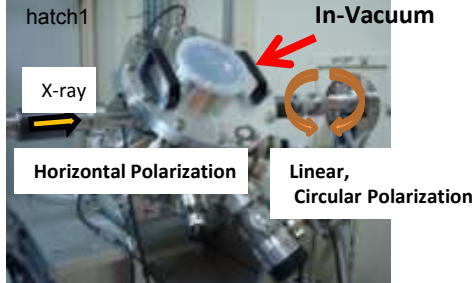


These development was supported by DG-JST.

Project Leader: Prof. C. Felser

MCD-HAXPES for Spintronics

200 μ m (111) Diamond @ 8.0 keV Phase Retarder



- $P_c > 0.95$
- $P_{lin} > 0.90$
- Rapid helicity reversal

Summary

(1) Objective Lens

- Total Acceptance angle = ± 30 degrees

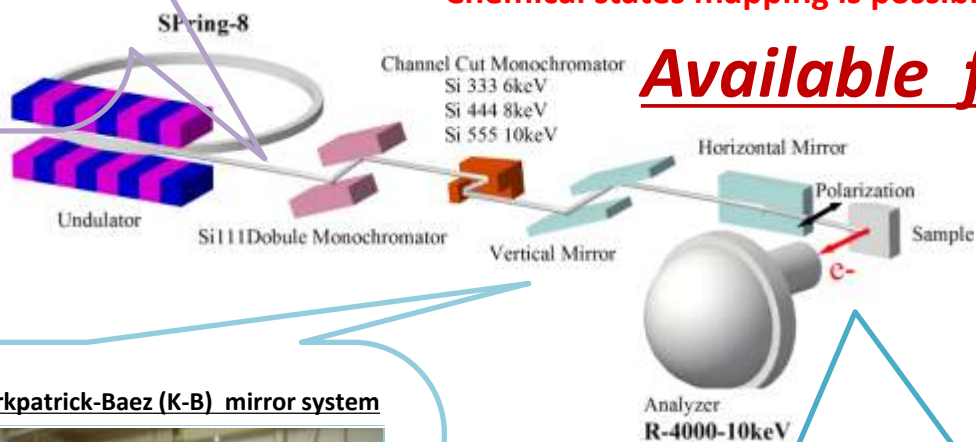
(2) K-B mirror

- Beam Size = $\Phi 1\mu$ m

(3) Combination

- Chemical states mapping is possible!

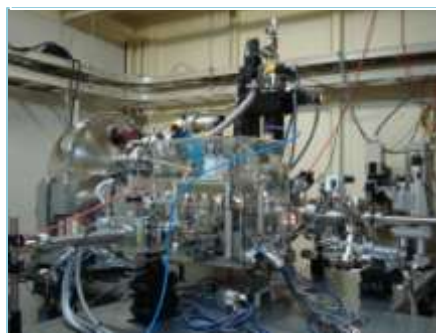
Available for all users !!



Focusing optics



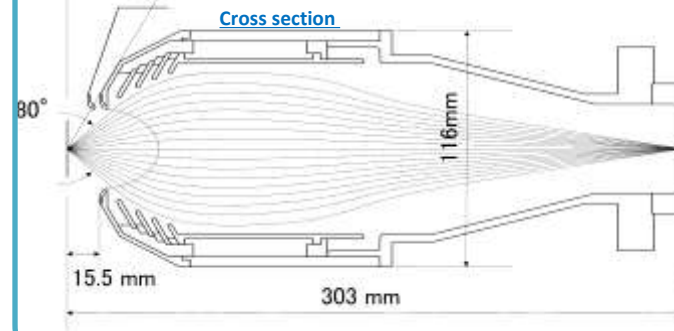
Kirkpatrick-Baez (K-B) mirror system



- Focusing size: $\phi 1\mu$ m

Wide Acceptance Angle

Wide Acceptance Angle Objective Lens



- Acceptance Angle: $\pm 30^\circ \leftarrow \pm 7^\circ$

These development was supported by SENTAN, JST.

Project Leader: Prof. K. Kobayashi.

Thank you for attention !