Development of Hard X-ray Photoemission Spectroscopy for Advanced Materials Science and Technology at BL-47XU/SPring-8

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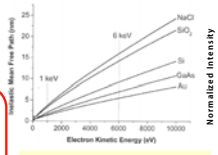
Hard X-ray Photoemission Spectroscopy (HAXPES)

Introduction

■ HAXPES - ***Large probing depth!***

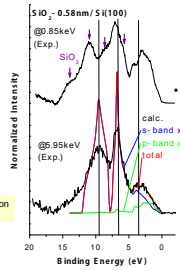
IMFPs
1～4nm @ 1 keV
4～15nm @ 8 keV

- Surface insensitive (Bulk sensitive)
- Free from surface prep. MBE thin film
- Chemical depth analysis Embedded interface (non distractive)



Inelastic Mean Free Path (IMFP) of Electron (From NIST Data Base)

Feature ***Proof of surface insensitivity***

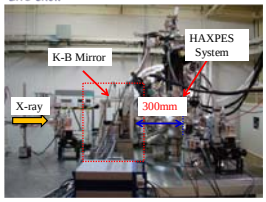


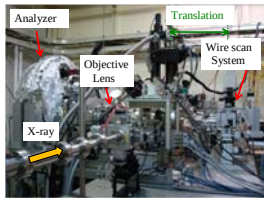
- Contribution of surface SiO_2 is negligible!
  IMFP: Si=9nm, SiO_2=14nm @ 6keV
  Si=1.8nm, SiO_2=3nm @ 0.85keV

Free from surface condition!

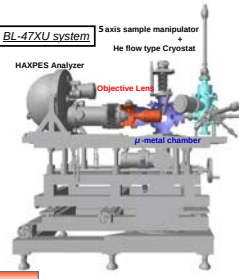
Public Beamline -47XU

Live show





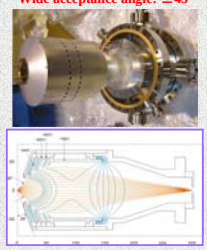
BL-47XU system



Development & Object

Development of Objective Lens

- Present status of solid angle: ±7° (R-4000 SCIENTA Analyzer)
- Wide acceptance angle: ±45°



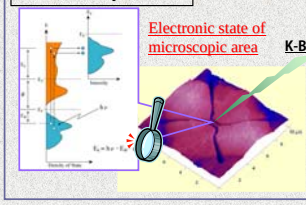
- **High throughput**

Introduce focusing optics

Present status of V: 40 μm H: 35 μm

Both horizontal and vertical focus mirrors have already been installed at BL47XU.

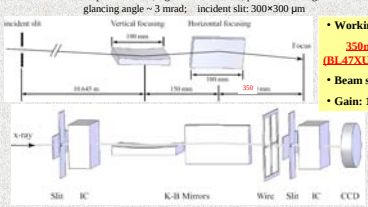
After installation K-B Mirror, Status of beam spot size ⇒ φ1 μm～submicron



In combination with focused beam and x-y scanning of the sample, we will realize the 3D mapping in the chemical states

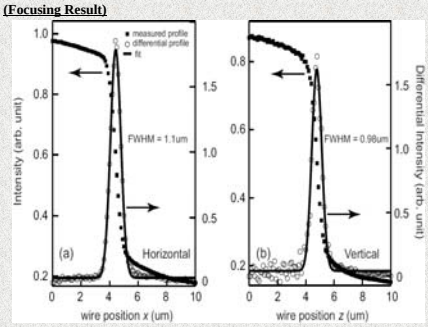
Introduce focusing optics

(Alignment) elliptical mirrors; figure errors ~ 2 nm; platinum coating; glancing angle ~ 3 mrad; incident slit: 300×300 μm



- Working distance: 350mm (BL47XU)
- Beam size: 1um
- Gain: 10^{12-13}

(Focusing Result)

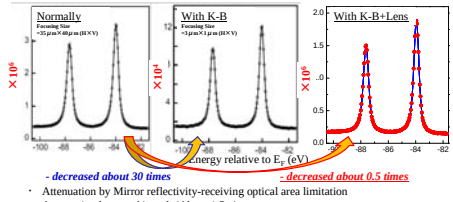


Beam size: 1.1 μm (H) × 0.98 μm (V)

Results

Photon Energy=8KeV

(Evaluation of Intensity) ***Au4f spectra***



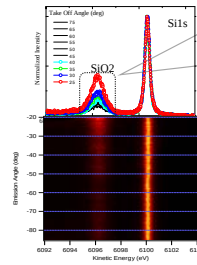
- decreased about 30 times *- decreased about 0.5 times*

- Attenuation by Mirror reflectivity-receiving optical area limitation
- Attenuation by part Air path (About 1.5m).

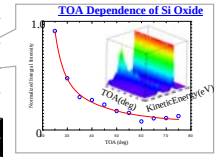
※ The K-B system will be examined to replacement with He gas.

Decreasing of flux efficiency by K-B mirror can be greatly supplemented with object lens.

(Evaluation of Depth Probing) SiO_2(4nm)/Si *spectra* *(RBS inspection finished)*



2D detector Image of Anglar mode (Si1s) (Sample Angle 55deg fixed)

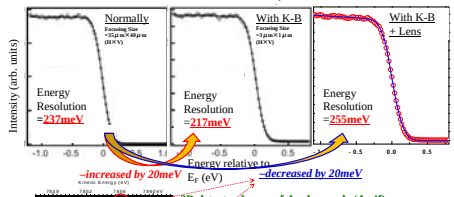


A oxide component increase is confirmed to the surface neighborhood！

The emitted electron angle and depth information can be acquired without changing the angle of the sample.

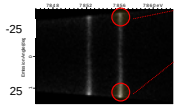
All measurement time is 10min.

(Evaluation of Energy Resolution) ***Au Ef***



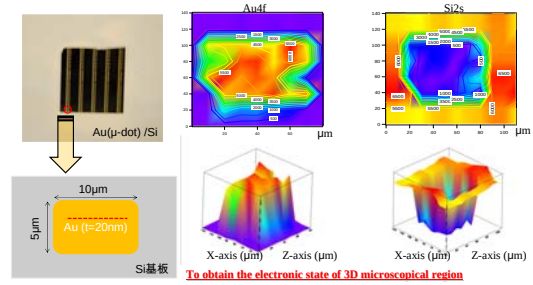
–increased by 20meV *–decreased by 20meV*

2D detector Image of Anglar mode (Au4f)



Resolution decreases when Object Lens is used.
It results in the aberration (high degree side) due to the bend of spheroid shape mesh.
※ Spheroid shape mesh making technology and the hole diameter shape are examined.

(2D mapping) Scanning of sample



To obtain the electronic state of 3D microscopical region

Development of Objective Lens

Wide angle object lens newly developed by 10kV voltage-proof specification (2007. completion in December)

About withstanding voltage・・・

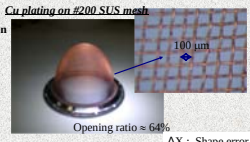
- Stable operation under 8kV application for 48 hrs without discharge.

Main feature・・・

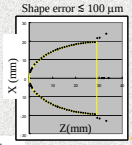
- Discharge prevention measures by simplification of correction electrode part structure
- The surface-roughness that disarranges equipotential is excluded.
- Working Distance=10mm
- Magnification factor=5

（Spheroid shape mesh evaluation:focusing size）

Cu plating on #200 SUS mesh

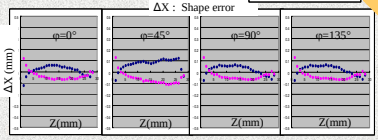


Shape error ≤ 100 μm

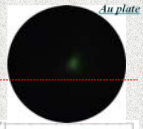


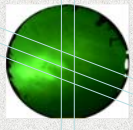
To lens mesh improvement

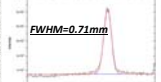
ΔX : Shape error



Phosphor Detector images on focal plane



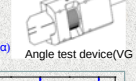




Source: electron gun
Focus size: 2μm@5keV official

(Angle resolving)

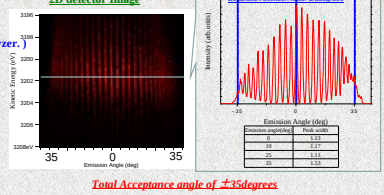
Lens mode: angular
PE=200eV,Slit:S4.0,Fixed
Beam Size200um(50W:Crkα)
Raster Scan(1mm×1mm)



Angle test device(VG SCIENTA)

Au3d(3.2keV)

2D detector Image

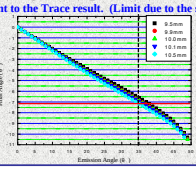


Emission angle(deg)	Peak width
0	1.13
10	1.17
25	1.31
35	1.55

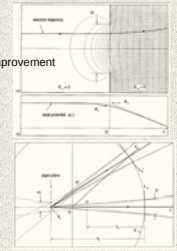
Total Acceptance angle of ±35degrees

- **Why Total Acceptance angle is ±35 result？**

※ It consistent to the Trace result. (Limit due to the solid angle of Analyzer.)



- **Aberration by apertures of lens mesh**



"Evaluation of the dispersive nature of meshes used for the spherical aberration correction of electrostatic lenses"
M. Kato et al., J. Vac. Sci. Technol. A 14(2), Mar/Apr 453 (1996)

The focal image by the effect of the hole is irrelevant to the aberration in the diameter of the hole.

Davisson-Calbick

$$f_M = \frac{4\Phi_0}{E_1 - E_2}$$

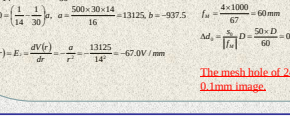
Φ_0 : Final axial potential
Potential difference at Mesh hole position

The hole of the mesh is thought as a lens focal length fM.

$$\Delta d_0$$

$$\Delta d_0 = \frac{S_0 - S_U}{S_U} D = \frac{S_0}{|f_M|} D$$

- **Calculation of focal image when electron is settled by 1kV with the wire of diameter 30 μm**



$$V(r) = \frac{a}{r} + b$$

$$0 = \frac{a}{14} + b, \quad -500 = \frac{a}{30} + b$$

$$500 = \left(\frac{1}{14} - \frac{1}{30}\right)a, \quad a = \frac{500 \times 30 \times 14}{16} = 13125, \quad b = -937.5$$

$$E(r) = E_z = \frac{dV(r)}{dr} = -\frac{a}{r^2} = -\frac{13125}{14^2} = -67.0V/mm$$

$$D = 25.4/100 - 0.03 = 0.224\,mm$$

$$E_z = -67V/mm$$

$$\Phi_0 = 1000V$$

$$Q = \Phi_0 / E_z D = 1000/67/0.224 = 66.6$$

$$f_M = \frac{4 \times 1000}{67} = 60\,mm$$

$$\Delta d_0 = \frac{S_0}{|f_M|} D = \frac{50 \times D}{60} = 0.83D = 0.186 \quad (D = 0.224)$$

$$= 0.02 \quad (D = 0.024)$$

The mesh hole of 24μm is necessary for obtaining an actual 0.1mm image.