Development of high lateral and wide angle resolved hard X-ray photoemission spectroscopy at BL47XU in SPring-8

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ABSTRACT

In this study, we have realized a high lateral resolution and wide-angle-resolved hard X-ray photoelectron spectroscopy (HAXPES) facility at BL47XU in SPring-8. The system uses Kirkpatrick–Baez focusing mirrors to achieve a beam size of 1.0 μm (horizontal) × 0.98 μm (vertical) at the photon energy of 7.94 keV and a wide-acceptance-angle objective lens installed in front of the electron energy analyzer. The objective lens system, which was developed originally and has achieved a total acceptance angle of ±34° with a resolution better than that of an acceptance angle of 1.5°. The performance of this system was evaluated through core spectra measurements of a typical multi-layered sample of Ir (8 nm)/HfO2(2.2 nm)/thickness-graded SiO2 (0–10 nm)/Si(0 0 1).

1. Introduction

Hard X-ray photoemission spectroscopy (HAXPES) has become a powerful tool for investigating the chemical states and electronic structures of various materials with third generation high-brilliance synchrotron radiation sources. The most advantageous feature of HAXPES is enabling us to measure intrinsically bulk sensitive electronic structures and buried interface profiles. The probing depths of PES are determined by the inelastic mean free paths (IMFPs) of electrons for the kinetic energy within a solid. The IMFPs in the 8 keV range are about 15 nm, which is almost 5 times larger than those at 1 keV [1]. The HAXPES technique, which was developed around 2001 at SPring8 [9,10] and ESRF [2], has in parallel been broadened, and rapidly expanded to the NSLS, BESSY, PETRA III, and ALS synchrotron radiation centers around the world [3–7]. BL47XU began at SPring8 from research and development associated with the discharge removal problem that is associated with the high retarding voltage for high-kinetic-energy electrons. This frequently occurring electric discharge problem was solved in collaboration with the RIKEN group [11]. In 2004, the HAXPES activities at BL47XU were introduced to limited users in support of nanotechnology, and finally in 2006, those were opened to all users [8]. Fig. 1 shows the configuration of a standard optical and experimental setup at the HAXPES experimental station in BL47XU. The band widths of the X-rays monochromatized by the Si(1 1 1) double crystal beamline monochromator are further reduced by the Si(1 1 1) channel cut post-monochromator. Photon energies of 6, 8, and 10 keV can finally be obtained using the 333, 444, and 555 Bragg reflections with intrinsic band widths of 50, 38, and 15 meV [11], respectively. The available photon flux is 2.8 × 1011 photons/s/0.0005% BW – at 8 keV this band width is achieved with a beam size of 30 μm (horizontal), H × 40 μm (vertical) by making use of both the focusing mirrors. Fig. 2(a) shows a schematic of the apparatus in the experimental hutch at BL47XU. For a hemispherical electron energy analyzer (R-4000–VG-Scienta Co.), the measurable kinetic energy has been extended to 10 keV. The stability of the electronics and the power supplies for the analyzer was confirmed to be extremely high by recording Au 4f spectra for about 10 days. The drifts in peak and integrated intensity were within ±5 meV and ±1%, respectively [12]. The total energy resolution for conventional parameters (pass energy: 200 eV and curved analyzer slit of 0.5 mm width) was estimated to be 228 meV by measuring the Fermi edge of Au at photon energy of 8 keV as shown in Fig. 2(b). A 2D detection system is also adopted in this analyzer, thereby enabling us to obtain spectra resolved along the lateral direction parallel to the entrance slit or in the angle of emission along the same direction, as illustrated below. In addition to the analyzer, the system includes a motorized XYZ stage with a sample manipulator having a flow-type liquid He cryostat, two CCD cameras for viewing the sample position, a flood gun for charge neutralization in studies of insulator samples, and a μ-metal measurement chamber. The whole system, which consists of a...
Fig. 1. Configuration of the standard optical and experimental set up at BL47XU. The lens axis of an electron energy analyzer is placed perpendicular to the incident X-ray beam and parallel to the polarization vector. The incidence angle relative to the sample surface is typically set to 1°, yielding a takeoff angle with respect to the surface of 89°.

Fig. 2. (a) Schematic of the apparatus in the experimental hutch. (b) The Au Fermi edge spectrum obtained for 7.94 keV photon energy. The total resolution is estimated at 228 meV under conventional parameters of the analyzer. (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

load-lock chamber, a preparation chamber, and an analysis chamber, is mounted on a XYZ stage as a rigid base for alignment. The stage can be controlled from outside the hutch. The vacuum of the analysis chamber is around 10^{-7} Pa, and the sample temperature can be lowered to around 20 K without serious vibration in the flowing liquid He.

The large depth of the information provided by HAXPES has opened various applications both in basic and applied research fields. Among them, the numbers of research projects related to lithium ion batteries, solar batteries [13,14], spintronics [15–19], advanced materials (transparent oxide semiconductor, InGaZnO) [20], industrial materials (DVD, LED, and organic) [21–24], and semiconductor technology for large scale integrated wiring material [25–27] have shown strong increases. Such research is targeting nanoscale–microscale material, leading to more requests for observations in this area, and hence it is important to investigate the electronic states over the nanometer to micron scale, including edge structures and cross sections with depth profiles. Anticipating demands from rapidly developing nanoscale–micronscale science and technology, we considered it necessary to introduce microscopic photoelectron spectroscopy with depth profiling capability.

There are two different methods for making photoelectron microscopic observations, as shown schematically in Fig. 3. One is with a photoelectron emission microscope (PEEM), which uses optics to observe the photoelectron emitted from the sample surface. The other is with a sample-scanning-type photoemission microscopes using a nano–micro-focused X-ray beam. The lateral resolution of PEEM, which is less than 10 nm, is very high for the large magnification lens shown in Fig. 3(a), although the energy resolution is moderate at 0.3–0.5 eV and there is typically upper limit of kinetic energy of less than 1 keV. With the sample-scanning-type...
direction of the analyzer in the transmission mode with recorded by the sample-scanning technique along the energy-scanning-type microscope and our motivation for its development. This is an advantage of the dependence of photoelectron spectra. This is an advantage of the scanning-type microscope and our motivation for its development.

Fig. 4 demonstrates the 2D mapping result of a Au micro-dot recorded by the sample-scanning technique along the energy-dispersive direction of the analyzer in the transmission mode with the X-ray beam size of 30 μm (H) × 40 μm (V). The y-axis of the 2D detector maintains the spatial resolution in this mode of analyzer, whereas the x-axis is the energy-dispersive axis. The sample is a Si wafer on which rectangular Au dots of 50 μm × 100 μm size were deposited, as seen in the optical image of Fig. 4(a). By scanning the sample step by step at 5 μm increments, the Au 4f integral intensity for each slice of detector-channel was recorded to obtain the 2D images shown in Fig. 4(b). Fig. 4(c) shows line profiles of the image along the z-axis and along the detector y-channel respectively. The lateral resolution of the 2D detection system along the detector y-channel was estimated to be about 30 μm and gave poor detail in investigations of electronic structures.

Fig. 4. Demonstration of sample-scanning microscopy for a Au micro-dot on a Si wafer with a standard beam size of 30 μm (H) × 40 μm (V). The Au dot size is 50 μm × 100 μm in the optical image (a). (b) Scanning 2D images of Au 4f integral intensity for each slice of detector-channel. (c and d) Line profiles of the image along the z-axis and along the detector y-channel respectively. The full width at half maximum (FWHM) of the image is 90 μm. This value is reasonable considering the Au dot size of 50 μm and the horizontal beam focusing size of 30 μm. On the other hand, the FWHM of the Au dot image profile is as broad as 300 μm in the direction of the detector y-channel as shown in Fig. 4(d), whereas the dot size is 100 μm. This broadening is fairly large compared to the channel array width of 1.605 μm/pixel in the microchannel plate (MCP) of the detector. It seems to have become blurred at the photoelectron spot on the detection system that combines a MCP and a phosphor screen. This result indicates that the focusing of the analyzer system in the imaging mode is rather poor and limits the resolution of the lateral image.

To obtain higher lateral resolution, we need to perform 2D scanning of the sample with finer micro-focused X-rays. The lateral resolution is limited simply by the X-ray spot size on the sample surface in this case, as described above. We targeted as a goal here the 3D analysis of chemical states by utilizing the advantage of the large probing depth of HAXPES together with variable takeoff angle provided by a wide-angle lens. Our development approach on the HAXPES scanning microscope has combine the Kirkpatrick–Baez focusing mirrors (K–B) and a wide-acceptance-angle electrostatic lens (WAEL) with angular resolving capability, which is also used in the laboratory HAXPES system described in the present issue; this project was carried out in the framework of the JST Sentan program. In the next section, we describe the developments of K–B mirrors for high lateral resolution and WAEL for angle-resolved measuring at BL47XU in SPring-8.

2. Development of K–B mirror for high lateral resolution

X-ray focusing optical components such as Fresnel zone plates [28], compound refractive lenses [29] and K–B mirrors [30] have been widely applied to X-ray microscopy, diffraction, and spectroscopy. Among them, K–B mirrors, which utilize total reflection, are known to be promising optical elements for achromatic and highly efficient beam focusing. Especially with recent progress in high-precision mirror manufacturing and machining techniques, micron- and submicron-range X-ray focusing sizes with useful throughput have been realized [32]. A beam size of 110 nm (H) × 98 nm (V) has been achieved using high-precision K–B mirrors with an accurate mirror manipulator [32]. A beam size of 47 nm (H) × 25 nm (V) has been reported by Jarre et al., using a combination of K–B mirrors and waveguide optics [31]. Fig. 5(a) shows the optical configuration of K–B mirrors system that we adopted in this development. Both the vertical and horizontal focusing mirrors are elliptical. The ultra-high-precision mirrors were fabricated by elastic emission machining (EEM), plasma chemical vaporization machining (P-CVM) [33,34], and microstitching interferometry [34]. The lengths of both mirrors are 100 mm. The mirror surfaces were coated with a Pt film to increase reflectivity to about 89.5% at 8 keV. The focal length is 258 mm for the vertical mirror and 154 mm for the horizontal mirror. The shape errors of the mirrors are as small as 2 nm. The glancing angle is 4.06 mrad for the vertical mirror and 3.6 mrad for the horizontal mirror. The distance between the two mirrors is 150 mm. The incident slit of 300 μm × 300 μm is located 10.65 m upstream from the center of the vertical mirror. The working distance of the K–B system is 350 mm.

A schematic of the experimental setup for the alignment of the K–B mirrors and for measurements of focused beam profiles is shown in Fig. 5(b). In front of the K–B system, we used a slit to adjust the beam shape and an ionization chamber (IC) to detect the incident beam intensity. Behind the K–B system, a wire scan system was placed at the focal position. A piezo-actuated translation stage was used to enable a wire scan at 10 nm increment steps. Copper wires of 200 μm diameter were used. Behind the scanning wire, we placed a slit to block unfocused beam and an IC to monitor the focused beam. A high-sensitivity CCD camera (HiPic C4742-98) was used for real-time observation of the beam shape and size. After adjusting the beam focusing, the parts behind the mirrors were replaced by the HAXPES system. To ensure the stability of the K–B system, we place the K–B system on a rigid base that is independent from the HAXPES system. The alignment of the K–B mirrors, such as glancing angle and perpendicularity between two mirrors was achieved by using a high-precision mirror manipulator system [32].

Fig. 6 shows the measured intensity profiles (solid squares) and their differential profiles (open circles) from a wire scan. The solid curve is fit by a Gaussian function. Fig. 6(a) and (b) shows the results of horizontal focusing and vertical focusing, respectively. The step increment of the wire scan is 10 nm for both profiles. The FWHM of the fitted curves indicates that a beam size of 1.10 μm (H) × 0.98 μm (V) was achieved at photon energy of 8 keV. The transmission effect at the copper wire edge is ignored.
After aligning of K–B system, we retracted the wire scan system to introduce the HAXPES system. Because the base of the K–B system is separate from the HAXPES system, the replacement did not influence the K–B mirrors. In addition, we improved signal intensity by purging the air in the optical path of the K–B mirror unit with He gas. The air path distance was as long as 500 mm thus, the large photon flux reduction from the atmosphere could not be neglected, even at the high X-ray photon energy of 8 keV. Consequently, we succeeded in recovering 63% of photon flux from $8.1 \times 10^{10}$ to $1.3 \times 10^{11}$ photons/s, and 67% of the Au 4f signal intensity at 8 keV photon energy compared to those without purging. This result confirms that linearity of signal intensity against photon flux is good enough. In this $1 \text{cm}^2$ focusing using the K–B mirrors with He gas, the photon flux is only one-half of that produced with standard focusing, 30 $\mu$m ($H$) $\times$ 40 $\mu$m ($V$) in BL47XU.

### 3. Development of wide acceptance angle electrostatic lens (WAAEL) for angle-resolved measuring

To obtain information about the distribution of elements and their chemical states, angle-dependent measurements of the photoelectron spectra, often called angle-resolved X-ray photoelectron spectroscopy (ARXPS), is a standard method. Development of a WAAEL with angular resolution capability is necessary for realizing 3D chemical analysis via ARXPS, in addition to the K–B mirror system mentioned above. Matsuda et al. proposed a WAAEL design to eliminate spherical aberration using an ellipsoidal mesh in the first stage electrode [35]. We adopted this basic design for an objective lens, which is installed in front of the VG Scienta R-4000 analyzer. The WAAEL designed for the present system effectively reduces the spherical aberration even for electrons impinging into the WAAEL with high angles of up to about $\pm 45^\circ$ as measured.
from the lens axis. Fig. 7 shows the structure and electron trajectory simulation of the WAAEL. Details of the simulation results for this WAAEL are described elsewhere [36]. Although the designed acceptance angle of the first stage is ±45°, it is ultimately restricted to about ±34° after being combined with the analyzer because of the limitation imposed by the final ±7° acceptance angle of the R-4000 analyzer. In addition, the calculation results show that it is very sensitive to the working distance from sample to lens entrance aperture. The working distance is 12 mm with a very small margin. There is a field-free region inside the ellipsoidal mesh electrode (that is on the sample side), such that there is no serious restriction on sample treatment. The structure of the field correction electrodes at the front part of the lens has been revised several times to completely prevent the undesirable discharges that were possible in the original design. Withstanding a voltage of 8 kV is finally realized for stable operation under application without discharge. The WAAEL was installed in front of the electron energy analyzer, as shown in Fig. 2 (a: red part). The magnification factor of both the WAAEL and the R-4000 analyzer lens is 5 times. Thus, the total magnification factor becomes 25 times by combining of the objective and the analyzer lenses. This means that effective view field on the sample with the entire analyzer system becomes 5 times smaller in comparison with the analyzer without WAAEL. The efficiency of the electron detection thus increases with decreasing X-ray spot size on the sample surface. Thus, the combination of the WAAEL and the 1 μm2 focusing of the K–B mirror described previously is very effective in increasing the throughput of the photoelectron measurements.

The angle acceptance and resolution were estimated using a cylindrical multi-slit for collimation. The result of the evaluation of acceptance angle is shown in Fig. 8. Fig. 8(a) shows the 2D image record of a Au valence band spectrum near the Fermi edge region in angular mode without normalizing the intensity. In Fig. 8(a) right and bottom sides, the integrated profile of the slit image along with kinetic energy and the integral of the Au Fermi edge spectrum along with the emission angle are shown, respectively. The number of slice peaks can be directly indicated by acceptance angles larger than ±30°, because the period of the multi-slit is 2.8°. The magnified profile of one slice is shown in Fig. 8 (b: red-circles). The blue curve in Fig. 8(b) is the derivative of the edge shape of the slice profile. The angular resolution was estimated to be 1.32° from the FWHM of the derivative. The spherical aberration is apparent in high emission angles, as shown Fig. 8(c). The aberration of 22 meV at the maximum is remarkably small for the total energy resolution of about 265 meV. It is thought that this aberration originates in the accuracy of an ellipsoidal mesh, in which the gap from a mesh-shaped design is about 50 μm at present. The high-precision fabrication of an ellipsoidal mesh was a most important point, and to remove this aberration, much research and development was done.

4. Results and discussion

Here, some results combining K–B mirrors with the WAAEL are described. All the experiments were measured with conventional parameters at photon energy of 7.94 keV. Fig. 9(a) shows the angle-resolved Si 1s spectra of a SiO2 (1 nm) layer buried between a Hf (4 nm) cap and the Si substrate without aberration correction for energy. The intensity is normalized by the intensity of the substrate silicon. The lower image is a single shot of a 2D image. The SiO2 thickness of 1 nm has been inspected by Rutherford Backscattering Spectrometry (RBS). The oxide component clearly increases with decreasing TOA decrease in Fig. 9(a). This means that wide depth profiles can be acquired at once without rotating the angle of the sample. In addition, efficiency is enhanced 30 times, and the 10 min acquisition in this measurement gives a sufficient S/N for the thickness analysis. To estimate the thickness of the SiO2 layer from TOA dependence in a quantitative analysis, we performed a simple angle dependency analysis of the intensity ratio between the signal from the Si substrate I (Si) and the silicon oxide I (SiO2). This TOA dependence of the intensity ratio is shown in Fig. 9(b). The TOA was fixed at 55° so as to minimize photoelectron diffraction effects of the Si(0 0 1) substrate [37], which were reduced in the spectra as can be seen in Fig. 9(b). A simple interlayer-substrate model, with constant thickness d and bulk density of SiO2, was applied to determine the electron attenuation length λs for Si and λo for SiO2 at the measured kinetic energy:

\[
\frac{I_o}{I_s} = \frac{\sin \theta}{\sin \theta_o} \exp \left[ \frac{\lambda_s - 1}{\lambda_o \sin \theta_o} \right]
\]

\[
d = \frac{\sin \theta}{\sin \theta_o} \frac{\lambda_s \lambda_o}{\lambda_o + 1}
\]

Here, ns (5.0 × 10²² N/cm³) and no (2.27 × 10²² N/cm³) denote atomic densities of Si atom in the Si substrate and SiO2 layer, respectively. The attenuation length was determined to be 6.4 ± 1 nm for Si and 9.2 ± 2 nm for SiO2 at the considered kinetic energy of 7940 eV from IMFPs predicted by the TPP-2 M formula [38]. Using the abovementioned parameters, the thickness of the buried SiO2...
Fig. 8. Test results for angular acceptance and resolution of the WAAELs, obtained by using a test device composed of a Au plate, in front of which was mounted a combination of a cylindrical multi-slit (the period of the multi-slit is 2.8°). (a) 2D image recorded in Au Ef region in angular mode. At right and bottom sides, the integrated profile of the slit image along with kinetic energy and the Au Ef spectrum along with emission angle are shown respectively. (b) Magnified profile of one slice (red circles) and the differential curve in edge shape of this slice profile (blue line). (c) Raw plot of Fermi level of Au for binding energy at each acceptance angle (red circles) and after correcting for energy (blue circles). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

oxide layer of each TOA was estimated by Eq. (1). The result of thickness values is plotted as function of TOA in Fig. 9(c). This average thickness was estimated to be 1.004 nm. This is very consistent with the thickness result of RBS and is the proof of quantitative measurement.

The performance of this scanning depth analysis was also evaluated by carrying out core spectra measurements of a typical multilayered sample of Ir (8 nm)/HfO$_2$ (2.2 nm)/thickness-graded (TG) SiO$_2$ (0–10 nm)/Si(0 0 1), schematically shown in Fig. 10(a). By scanning the position of the TG sample surface along the SiO$_2$

Fig. 9. (a) Angle-resolved Si 1s spectra of a 1 nm layer of SiO$_2$ buried between a Hf cap (4 nm) and a Si(0 0 1) substrate with fixed TOA of 55°. The lower panel is a single shot of a 2D image. (b) TOA dependence of the intensity ratio between oxide and substrate of Si 1s spectra. (c) Thickness calculation results for the SiO$_2$ buried layer for each TOA. This average thickness was estimated to be 1.004 nm.
Fig. 10. (a) Schematic structure of the Ir (8 nm)/HfO2 (2.2 nm)/SiO2 (0–10 nm)/Si multi-layer. (b and c) Position dependence of Si 1s and O 1s along TG direction by increments of 50 μm, respectively.

thickness and varying the direction step by step in 50 μm increments, the Si 1s and O 1s spectra are found to exhibit features shown in Fig. 10(b) and (c), respectively. Although Hf 3d5/2 and Ir 3d5/2 spectra were also observed, no significant changes in these core spectra with thickness were recognized. However, for Si 1s there are marked changes. The Si 1s from the SiO2 layer loses intensity and shifts toward lower binding energy as the thickness decreases. The substrate Si 1s peak is still clearly recognized even when it is totally buried under 20 nm thick over-layers in total, indicating the capability of a large probing depth. The intensity of O 1s from SiO2 decreases relatively compared with that from HfO2, and their energy separation becomes smaller as SiO2 thickness decreases. In addition, the depth profile of the structure has been mapped along the SiO2 thickness varying direction with the TOA dependence of these core spectral intensities. These results directly indicate the band bending in thickness dependence caused by the charge trap of interlayer band diagram [39]. This charge trap mechanism proposes that positive charges are trapped on the HfO2 surface and negative charges are trapped in the HfO2 film along thickness of SiO2.

Furthermore, the scanning and angle-resolved results of 2D mapping applied to micro-dots of (Fe2.5Mn0.5)O4 ferromagnetic oxide (thickness: 20 nm) on Nb0.5 wt%–SrTiO3 substrate are shown in Fig. 11. This (Fe2.5Mn0.5)O4 system is a typical magnetic semiconductor exhibiting strong electron correlation effects [40]. Core-level spectra of the (Fe2.5Mn0.5)O4 films showed that Mn and Zn ions were substituted with a divalent cation at the A-site and that Fe2+ components decreased with substitution of Mn. In addition, the valence band spectra revealed that DOS near Ef of (Fe2.5Mn0.5)O4 thin films systematically decreased with increasing thickness.

Fig. 11. Results of 2D mapping via sample scanning applied to a rectangular micro-dot of (Fe2.5Mn0.5)O4 ferromagnetic oxide (thickness, 20 nm) on Nb0.5 wt%–SrTiO3 substrate. (a) The dot size is 50 μm × 50 μm in the optical image. (b and c) The 2D mapping image of integral intensity for Fe 2p3/2 and Sr 3p3/2, respectively.
Mn concentration. The band shift due to changing electron correlation was observed in a wide substitution range and is interpreted by the change on the basis of a Mott–Hubbard model [40]. Tanaka et al. believe that this strong electron correlation plays an important role in small switching devices using carrier-induced phase transition such as a Mott–FET. The dot size is 50 μm × 50 μm in the optical image of Fig. 11(a). In Fig. 11(b) and (c), the 2D mapping image of integral intensity for the Fe 2p3/2 and Sr 3p3/2 shows peaks through step by step at 1 μm increments, respectively. These images show Fe 2p3/2 and Sr 3p3/2 substrate intensities, and reversal distribution of the chemical states in Fig. 11(b). The micro dot is well resolved using a 1 μm beam size. This lateral-resolved investigation of micro-dot thin film suggests that this material system holds great promise for the development of advanced spin electronics.

5. Conclusion

3D chemical analysis combining sample scanning (in-plane 2D) and TOA resolving (ARXPS depth profiling) has been conducted as a 5-year project in the framework of the JST Sentan program. With regard to the use of WAAEL, the total acceptance angle is enhanced by ±34° with resolution of 1.32°. By installing the K–B mirrors, we achieved a beam size of 1 μm as lateral resolution. Through a combination of the K–B mirrors and the WAAEL, chemical state mapping will provide important information about the electronic properties of various materials important to basic science, industry and technology.

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[1] The electron inelastic-mean-free-paths were estimated using NIST Standard Reference Database 71, NIST Electron Inelastic-Mean-Free-Paths Data: Ver. 1.1. It is distributed via the Web site http://www.nist.gov/srd/nist71.htm