

Extended Abstract of PSA-19

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Charge Compensation in Hard X-ray Photoelectron Spectroscopy by Electron Beam of Several Kilo-electron-volts

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The charge compensation for the insulating material in the hard X-ray photoelectron spectroscopy (HAXPES) was investigated using the different energy types of electron flood gun. The electron energy of 5.5 keV resulted in the shifts of photoelectron lines to more proper positions and the lower peak widths compared with the conventional low-electron energy flood gun (3 eV). The charge compensation in HAXPES using the electron beam of several kilo-electron-volts was found to be a practical controlling method for the charging effect.

1. Introduction

Hard X-ray photoelectron spectroscopy (HAXPES) has been attracting considerable attention since it can probe the chemical and electronic states of the bulk and buried interface lying at depths of several tens of nm due to its large probing depth[1]. However, the problem of the charging effect in HAXPES is of importance for attempting to evaluate the small shift in photoelectron line and the small change of spectral shape as well as the soft X-ray photoelectron spectroscopy (SX-PES). In general, for SX-PES, the way of compensating for the charging effect is to supply a flood of low-electron energy. While, the charge compensation in the HAXPES measurement has not previously been reported in detail. Since the probing depth of HAPXES is much larger than SX-PES, it is likely that HAXPES needs to perform the charge compensation at deeper region compared with SX-PES by some suitable sources instead of low-electron energy flood gun. Therefore, in this study, we investigated the procedures of the charge compensation in HAXPES by using the different energy types of electron flood gun as a function of electron energy.

2. Experimental

Hard X-ray photoelectron spectroscopy measurement was performed at BL46XU in SPring-8.

An incident X-ray with a photon energy of 7.94 keV was monochromatized with a Si(111) double crystal and Si(444) channel-cut monochromators. Photoelectron spectra were observed by a hemispherical electron energy analyzer (ScientaOmicron HIPP-2). The aperture of the analyzer slit was 0.5 mm with a rectangular shape, and pass energy was fixed as 200 eV. The analyzer is set perpendicular to the X-ray axis and parallel to the polarization vector. X-ray was incident at the incident angle of 80° and the emitted photoelectrons were detected at the take-off angle of 10° relative to the surface normal. Instrumental vacuum was 1 to 5 × 10⁻⁶ Pa. The charging effect and the neutralization depend on the surface morphology. Thus, we chose to work on samples that were the single crystal in order to eliminate the contribution of the surface morphology for the charging effect and the neutralization. In the present study, we used the polished Al₂O₃ single crystal. Moreover, we employed the 2nm-thick Os coated Al₂O₃ single crystal as a reference. This is because Os coating is an effective method for achieving charge compensation for insulating materials in the photoelectron spectroscopy[2]. In fact, we checked that the peak positions of O1s, Al2s and Al2p lines obtained from Os coated Al₂O₃ corresponded to those of the literature of Al₂O₃ [3]. Also, we employed the two

Table1 Influence of the electron energy on line positions and FWHMs for Al₂O₃

Electron energy	Al1s		O1s		Al2s		Al2p		Charge/eV			
	E_p /eV	FWHM/eV	E_p /eV	FWHM/eV	E_p /eV	FWHM/eV	E_p /eV	FWHM/eV	Al1s	O1s	Al2s	Al2p
3 eV	1560.5	1.4	531.0	1.5	120.0	2.0	75.4	1.5	-1.0	-0.5	0.6	0.7
5.5 keV	1561.5	1.3	531.4	1.3	119.5	1.7	74.7	1.2	-0.1	-0.1	0.0	0.0
Os coating (Reference)	1561.6	1.2	531.5	1.3	119.4	1.6	74.7	1.1	-	-	-	-

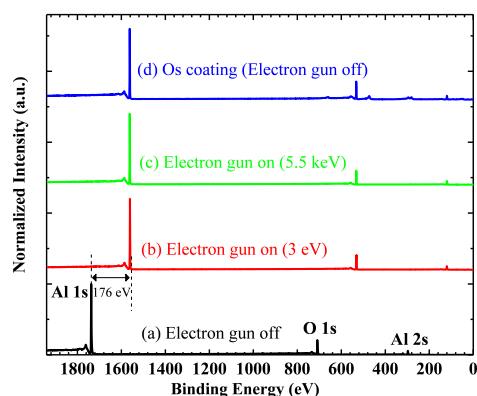


Fig. 1 Survey spectra of Al₂O₃ with (a) electron gun off, (b) electron gun on (electron energy of 3 eV), (c) electron gun on (electron energy of 5.5 keV), (d) Os coating.

energy types of the electron flood gun. The electron beams for charge compensation were performed at the electron energy of 3 eV with the beam current of 6 μ A using a 04-091 (ULVAC Phi), and the electron energy of 5.5 keV with the beam current of 4 μ A using a EGF-3104 (Kimball Physics). We optimized the electron gun settings (the electron energy and the beam current) based on the line position and the peak width (FWHM) of Al2s peak of Os coated Al₂O₃.

3. Results and Discussion

Figure 1 shows the survey spectra of Al₂O₃ with and without electron gun. In addition, survey spectrum of Os coated Al₂O₃ as the reference is shown in Fig. 1. As shown in this figure, the electron gun off (a) is shifted to higher binding energy by 176 eV, indicating that the sample has charged positively. Table 1 summarizes the results obtained by measuring the aluminum and the oxygen photoelectron lines at different energy of the electron flood gun and those of Os coated Al₂O₃. The values of charge in table 1 were estimated by subtracting the peak position of Os coated Al₂O₃ from each peak position.

For the electron energy of 5.5 keV, it was found that the charge of the all lines were influenced almost equally. While for the electron energy of 3 eV, the

charge values of each lines were inconstant. Also, the FWHMs of electron energy of 5.5 keV were close to those of Os coated Al₂O₃. By contrast, the electron energy of 3 eV is insufficient to discharge the Al₂O₃ as reflected in the larger FWHM.

For HAXPES measurement, the photoelectron lines can be measured in the wide range of the kinetic energy. In particular, in the case of high kinetic energy, the emission of photoelectron occurs in the deeper region compared with SX-PES, so that we need to replace the photoemitted electrons with ones at the deep region. Here, the inelastic mean free path (λ) calculated by TPP-2M [4] formula of the Al2p in this study is 13.4 nm. We can consider that some 95% from the photoelectron signal will emanate from a depth of 3λ . Thus, the majority of Al2p peak emitted from outmost surface to 40.2 nm. While, the penetration depth of electrons with the electron energy of 5.5 keV is approximately 300 nm estimated by Kanaya - Okayama Formula [5]. Hence, the electron energy of 5.5 keV have enough penetration depth to compensate the charge effect in hard X-ray region. It is suggested that the electron beam of several keV is suitable for the charge compensation in HAXPES measurement.

4. Acknowledgements

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