Study on the behavior of the photoelectrons with lower energies in the ultra thin film of silicon oxide

Motoyasu Imamura *1 ¹AIST, 1-1-1 Higashi, Tsukuba 305-8565, Japan

1 Introduction

X-ray photoelectron spectroscopy (XPS) has been used for the quantitative and qualitative analysis methods in the developments of functional materials because of its high surface sensitivity, and it is useful to determine the thickness of the thin film. For determination of the the thickness using XPS, the attenuation length (AL) of the photoelectron is an important parameter. For a long time, the inelastic mean free path (IMFP) has been used as an alternative of AL because the values of IMFP can be calculate easily using the predictive equation TPP-2M [1-3]. However the effect of the elastic scattering is not considered in the evaluation of IMFPs. Recently effective attenuation length (EAL) [1, 4, 5] is proposed and used for the practical purposes. The author has been experimentally measured the EAL of the photoelectron in the SiO_2 thin films with the energy range of 100– 1000 eV [6] at old BL13C station [7,8].

For experimental determination of EAL in SiO_2 , the SiO_2 thin film and bulk samples of Si and SiO_2 are necessary. The thickness of the thin film must be certified by another method. As shown Fig. 1, photoelectron intensity from each sample was measured. From the bulk samples of the silicon and silicon oxide,

$$R_o(h\nu,\theta) = \frac{I_{Si}^{\infty}}{I_{SiO_2}^{\infty}} \tag{1}$$

is caluculated where I_{Si}^{∞} and $I_{SiO_2}^{\infty}$ are the photoelectron intensities from the Si substrate and the substrate of oxide only, respectively.



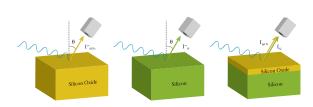


Fig. 1: Samples for EAL detemination

is calculated where I_{Si} and I_{SiO_2} are the photoelectron intensities from the Si substrate and SiO₂ thin film, respectively.

The EAL is calculated from the equation below:

$$\lambda_{SiO_2}(h\nu,\theta) = \frac{d}{\cos\theta} \cdot \frac{1}{\ln\{[R_o(h\nu,\theta)/R_d(h\nu,\theta)]+1\}}$$
(3)

For determination of the EAL in the SiO₂ thin film, it is important to measure the R_o precisely.

2 Experiment

The measurements of XPS spectra were carried at BL₃B using the apparatus built for the angle-resolved x-ray photoemission spectroscopy (ARPES II). In this apparatus, the hemispherical electron analyzers with a 50 mm mean radius (VSW HA54) are fixed on the goniometer, which enable the analyzer to be rotate in the vacuum chamber as shown in Fig. 2. The energy step was 0.05 eV in the measurements of the XPS spectra. The pass energy was set as 10 eV. The acceptance angle was $\pm 1^{\circ}$. The angle between the incident x-ray and the surface normal of the sample was fixed as 55°. The emission angle of the photoelectrons from the surface normal was adjusted to 0° , 15° , 30° , 45° , 60° , and 75° by rotating the electron analyzer. The energy of the incident x-ray was set as 130 eV, 150 eV, 180 eV, 200 eV.

As the bulk samples, Si(100) wafer with a thickness of 0.5 mm and SiO_2/Si thin film with a thickness of 100 nm prepared by a thermal process were used. The thickness 100 nm of the SiO_2 thin film is thick enough to avoid the photoelectron signal from the substrate and thin enough to suppress the charging by the irradiation of x-ray. Both samples were cut as

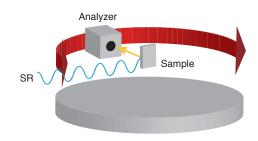


Fig. 2: Alignment of sample and analyzer

the size of 5 mm \times 3 mm using a diamond cutter and fixed on the same stub for the measurement using carbon tape as shown in Fig. 3, because two samples must be used for measurement by turns in the same condition to suppress the time dependent influence.

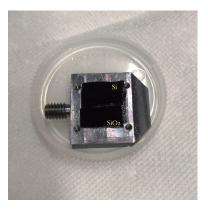


Fig. 3: Samples on the stub

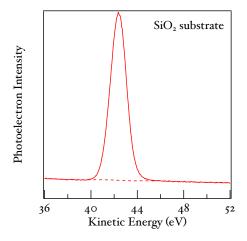


Fig. 4: XPS spectra of SiO₂

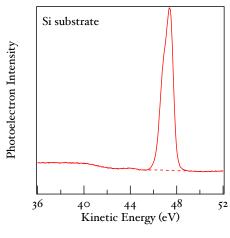


Fig. 5: XPS spectra of Si

3 Results and Discussion

Fig. 4 and 5 shows the Si 2p spectra of SiO₂ and Si, respectively which measured with the emission angle of surface normal excited by x-ray with the energy of 150 eV. Si peak and Si oxide peak are recorded clearly. To estimate the photoelectron intensities in the spectra, curve fitting procedure was carried out using Shirley function for the background and Gauss function for the peak. The R_0 is calculated using Eq.(1) for each energy and emission angle, which is mentioned in the experimental section. Some of the R_0 data were plotted as functions of the emission angle in Fig. 6.

Acknowledgement

I would like to thank Prof. Ozawa of Tokyo Tech and Prof. Mase of PF for their help in my experiments at BL₃B station.

References

- [1] ISO 18115:2001-surface chemical analysis vocabulary.
- [2] S. Tanuma et al., Surf. Interface Anal., 17 (1991)
 927; 20 (1993) 77; 21 (1993) 165.
- [3] C. J. Powell and A. Jablonski. NIST Electron Inelastic-Mean-Free-Path Database: Version 1.1. National Institute of Standards and Technology (2000).
- [4] C. J. Powell and A. Jablonski. NIST Electron Effective-Attenuation-Length Database - Version 1.0. National Institute of Standards and Technology (2001).
- [5] A. Jablonski and C. Powell, Surf. Sci. Rep. 47 (2002) 33.
- [6] M. Imamura et al., Meas. Sci. Technol. 22 (2011) 024007.
- [7] N. Matsubayashi *et al.*, *Rev. Sci. Instrum.* 63 (1992) 1363.
- [8] H. Shimada et al., Rev. Sci. Instrum. 66 (1995) 1780.

*motoyasu@ni.aist.go.jp

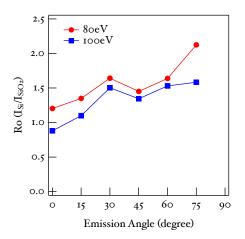


Fig. 6: Ro of several emission angles