A Round Robin Test on XPS Transmission Function

N. Fukumoto[1)]*, S. Tanuma[2)], A. Tanaka[3)], I. Kojima[1)], K. Dohmae[4)],
and SASJ XPS Transmission Project
1) National Institute of Advanced Industrial Science and Technology, AIST Tsukuba Central No.5, Ibaraki, 305-8565, JAPAN
2) National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, JAPAN
3) ULVAC-PHI Inc., 370 Enzo, Chigasaki, Kanagawa 253-8790, JAPAN
4) TOYOTA Central Research and Development Labs., Nagakute, Aichi 480-1192, JAPAN

(Received December 24, 2001 Accepted: October 29, 2002)

In order to perform a quantitative analysis by electron spectroscopy, precise information on the transmission function of the spectrometer is crucial for comparison of spectra obtained with different analytical conditions or by different models of spectrometers. A preliminary round robin test for comparison of transmission functions of different models of XPS spectrometers was conducted by some members of *SASJ*, and it was shown that standardization of the transmission correction procedure is necessary to perform quantitative analysis with relatively small uncertainty.

1. Introduction

The transmission function is the dependence of the sensitivity on the kinetic energy of electrons for electron spectroscopy. It is known that the form of the transmission function is determined by various factors such as the configuration or operating mode of the electron optics including magnetic lenses, the pass energy of the electron analyzer(E_{pass}), the condition of the electron multiplier and the signal counting rate.

An intensive study on spectrometer transmission calibration for Auger electron spectroscopy had been conducted by Yoshitake et al. in 1997.[1] On the other hand, in the field of X-ray photoelectron spectroscopy (XPS), only theoretical possibility of transmission calibration and preliminary experimental results such as by Berresheim et al.[2] and Dohmae et al.[3] were reported.

2.Experimental

By a simple experiment using a pure copper sample and Al monochromatic (1486.6 eV) X-ray source, it was found that the ratio of Cu $2p_{3/2}$ (E_b=933 eV) and Cu 3p (E_b =77 eV) photoelectron intensities changed about 3 times in the 5 - 100 eV pass energy range for a spectrometer. On the other hand, for another model of spectrometer (manufactured by a different company), the intensity ratio of the same pair of photoelectron peaks changed only less than 1.2 times in the same range of pass energy. Thus the necessity of more extensive study, which involves various types of spectrometers manufactured by different companies, was proposed at the regular meeting of the Surface Analysis Society of Japan (SASJ) in June 2001 and SASJ XPS Transmission Function (XPS-TF) project team was founded.

In order to evaluate the characteristics of the transmission function of each spectrometer, XPS peak intensities of low and high kinetic energies for a wide variation of analyzer pass energies were collected. All data were acquired by Al K_α monochromatic X-ray.

The recommended measured peaks were Cu $2p_{3/2}$ for high binding (low kinetic) energy and Cu 3p or Au $4f_{7/2}$ (E_b=84.0 eV) for low binding (high kinetic) energy. For both Cu and Au peak measurements, clean pure metals prepared by each participant were used. Due to the cooperation of the participants of the project team, XPS spectra obtained by 7 different models of spectrometers manufactured by four manufacturers were collected. For most spectrometers studied, pass energies in the rage of 10 eV to 150 eV were studied but one model

This paper was presented at the International Symposium on Practical Surface Analysis (PSA-01), 2001 in Nara, Japan.

could be operated only up to 50 eV due to its specifications.

3.Results and discussion

Four sets of spectra obtained by spectrometers manufactured by different companies were analyzed. If a spectrometer has a transmission function which depends only on the retarding ratio (E_k / E_{pass}) for various values of pass energies, the shape of the intensity (normalized by pass energy) vs. retarding ratio curve [2], (the *Berresheim's plot*), does not depend on the kinetic energy of the photoelectron. Out of the four sets of spectra studied, only one set showed these characteristics. The intensity ratios of high kinetic energy (approximately 1400 eV; Au 4f or Cu 3p) peak to low kinetic energy (approximately 550 eV; Cu 2p) peak for each spectrometer are shown in Fig. 1. The ratios are normalized to the values for 10 eV pass energy. The intensity ratio of the high E_k peak against the low E_k peak changed only 1.2 times (0.78 to 0.93) in the pass energy range of 5 to 160 eV for instrument B. For instruments A and C the ratios changed about 3 times (0.6 to 1.6 and 0.55 to 1.77 respectively) in this region of pass energy. Instrument D can only be operated up to 50 eV of E_{pass}. Even in this limited region, this (Cu 2p / Au 4f) ratio changed about 1.4 times (0.7 to 1.0). The reason for this difference of the transmission characteristics of the instrument B and other three ones is not clear, but it is supposed that some intentional correction for transmission characteristics could be done in designing spectrometer B. The results shown above suggest that it is necessary to establish standardized transmission correction procedures for various instruments and operating conditions for conducting quantitative analysis by relative sensitivity factor with considerably small uncertainty.

4.Acknowlegements

Other than the co-authors, following members of the XPS-TF project team contributed the XPS spectra for this study: H. Ando (NTT-AT), A Aso (Nippon Mining & Metals), M. Jo (AIST), A. Koizumi (Shinko Electric) and Y Sakai (JEOL). The authors also appreciate SASJ XPS-TF project team members who gave useful suggestions for this study.

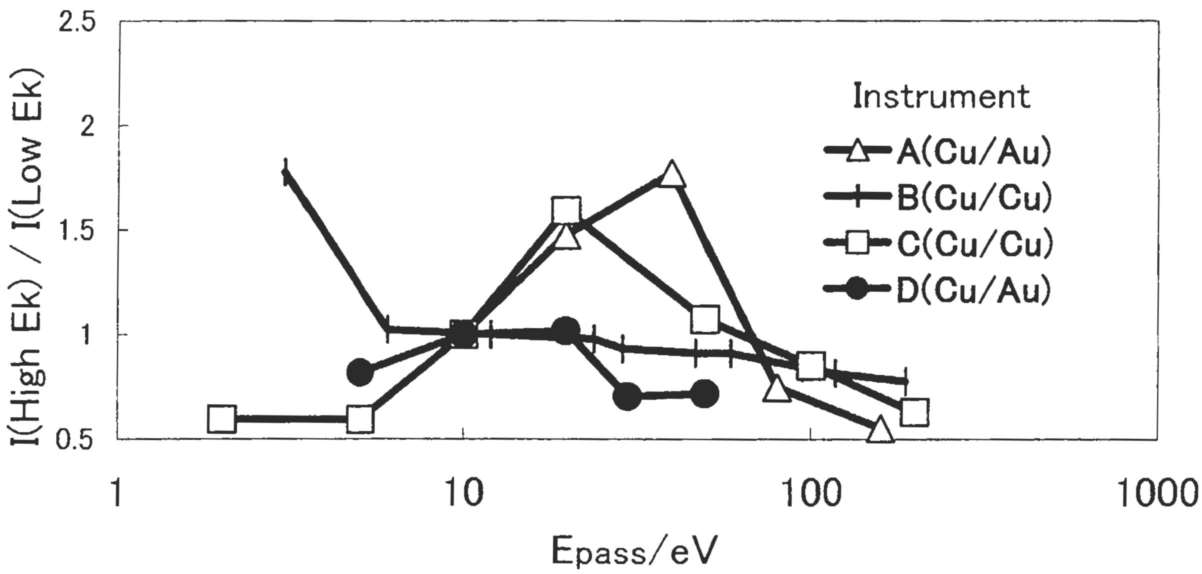

Fig. 1 Normalized intensity ratio of low E_k (around 550eV;Cu2p) and high E_k (around 1400eV;Au4f or Cu3p) peaks for various pass energies obtained by 4 spectrometers manufactured by different companies. All lines are normalized to the values at E_{pass}=10 eV.

5. References

[1] M. Yoshitake and K. Yoshihara, Surf. Int. Anal. **25** 209 (1997)

[2] K. Berresheim, M. Mattern-Klosson and M. Wilmers, Fresenius J. Anal. Chem. **341** 121 (1991)

[3] K. Dohmae, A. Tanaka, J. Surf. Anal. **2** 316 (1996)