# A Study of Charge Compensation for Insulator Samples in AES by Low Energy Ion Beam Irradiation

H. Iwai*,**, H. Namba*, T. Morohashi**, R. E. Negri***, A. Ogata**, T. Hoshi** and R. Oiwa**

* *Ritsumeikan University, 1-1-1 Nojihigashi, Kusatsu, Siga 525-8577, Japan*
** *ULVAC-PHI, Inc., 370 Enzo, Chigasaki, Kanagawa 253-0084, Japan*
*** *Physical Electronics, Inc., 6509 Flying Cloud Drive, Eden Prairie Minnesota 55344, USA*

(Received October 20 1998; accepted February 2 1999)

The insulator analysis in AES has long been a big problem because of the severe charging. We have shown that the ion beam current density using floating column ion gun was obtained as the order of $10^{-1}$ $Am^{-2}$ below beam voltage of 100 V. The charge compensation using a low energy ion irradiation was effective for fused quartz over 40° of the incident angle of primary electron beam with 3 kV 5 nA in 100 × 100 $\mu m^2$ raster. The high resistivity glass plate has also demonstrated for the charge compensation with low energy ion irradiation for a high spatial resolution analysis in AES.

## 1. Introduction

AES is widely used for surface analysis today. The primary beam current density becomes extremely large with recent instrumental development for high spatial resolution analysis such as field emission scanning Auger microscope[1]. For insulator material, irradiated electron supplied by primary electron beam charges on the surface like as a capacitor, which exhibits severe charging problem. The most traditional method for reduction of the charging is increase of secondary electron emission from the surface by increasing the incident angle of electron beam (or tilting the sample). Metal foil mask with small opening for analysis, evaporation of conductive material such as gold and graphite, low energy electron beam irradiation on the insulator sample are effective for some materials to reduce the charging[2][3][4]. A low energy ion beam was considered to be effective for reducing the charging[4]. It was, however, quite difficult to obtain enough beam current below the beam voltage of 100 V to avoid or minimize the sputtering, and it was not commercially available in the past[2].

Recently, using both low energy electron and ion beam for the charge compensation has been developed and demonstrated for the insulator analysis in XPS with monochromatic X-ray[5]. Thus, low energy ion irradiation may be effective for some of the insulator surfaces in AES. In this paper, we describe an evaluation result of low energy ion gun applied to AES instrument, and some of the insulator analysis in AES with a low energy ion beam irradiation are discussed.

## 2. Experimental

For studying charge compensation by low energy ion beam irradiation, Physical Electronics Model 680 AES apparatus was used, which equipped with field emission electron gun and floating column ion gun for improving low energy performance. At first, the performance of the ion gun has been examined. Figure 1 shows a schematic diagram of the ion gun column (model 06-350). An electron impact ionization method is used as an ion source, which is commonly used in a conventional sputter ion gun for depth profiling analyses. The ion gun column is self aligning on assembly for eliminating a beam steering. The voltage of cylindrical grid (2) is supplied at the range of +1 to +5000 V with respect to ground (vacuum chamber), which determines the primary beam energy. Ion beam is extracted from the grid by extractor (3), then transported to the floating column through extraction aperture (4). The transported voltage in the floating column is the sum of the grid voltage and floating column voltage $V_f$. Other optical elements are condenser lens (5) for defining the

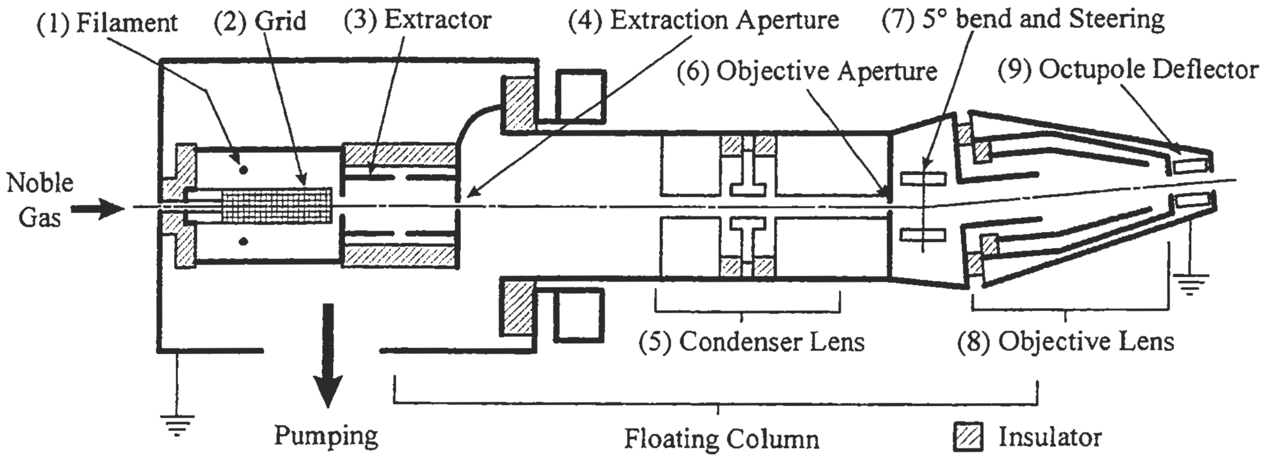

Figure 1. Schematic diagram of floating column ion gun. Ion gun column from Extraction Aperture (4) to Objective Lens (8) is floated from ground.

beam current, differential pumping aperture (6) for isolating the analysis chamber, and a retarding immersion lens as an objective lens (7). Ion can be scanned by octupole deflector (9). A neutral beam is eliminated by 5° bending of the column at the steering deflector (7). Ion beam is finally decelerated at the objective lens area. The floating column is differentially pumped by turbo molecular pump. The pressure of analysis chamber was maintained at $1 \times 10^{-6}$ Pa during the operation. The working distance of the ion gun was 30 mm to reduce the beam size. The angle between primary electron beam and ion beam is 77°. Tilt axis of a sample stage is perpendicular to the plane of electron beam and ion beam. In this study, argon was used for the low energy ion beam. For evaluating the charge compensation, O KLL peak positions were recorded.

## 3. Results and Discussion

### 3A. Performance of low energy ion gun

Figure 2 shows the maximum beam current and maximum beam current density as a function of the ion beam voltage. Below a beam voltage of 200 V, the floating mode operation shows significant improvement of both beam current and beam current density. The ion beam current density at $V_p$ = 100 V is the order of $10^{-1}$ Am$^{-2}$, which is approximately equivalent to the beam current density of primary electron beam for the beam current of 1 nA in 100 × 100 μm$^2$ raster. The maximum beam current was also obtained as the order of 100 nA around $V_p$ of 100 V. In addition, the energy spread of 50 eV ion beam was obtained as full width at half maximum of 3.6 eV defined by elastic scattering ion beam spectrum. It is, therefore, enough beam current density with reasonable energy spread for the charge compensation on the insulator sample.

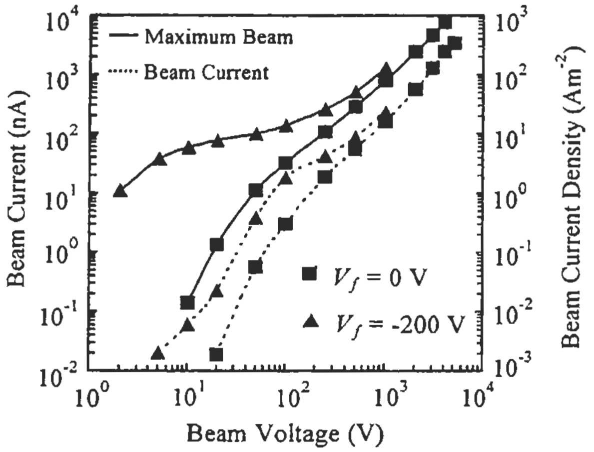

Figure 2 Maximum ion beam current and maximum ion beam current density as a function of the primary beam voltage. Beam current density was measured by Faraday cup with ϕ250μm hole in diameter.

### 3B. Result of electrically insulated sample with low energy ion beam irradiation

Figure 3 (a) shows the sample current of primary electron beam at gold and silicon surfaces as a function of the incident angle of the electron beam. In case of gold surface with 3 kV electron beam incidence, the sample current shows positive values for any incident angles, which explains that the secondary electron current exceeds the incident beam current. We have also experimentally obtained that gold Auger peak positions were not shifted

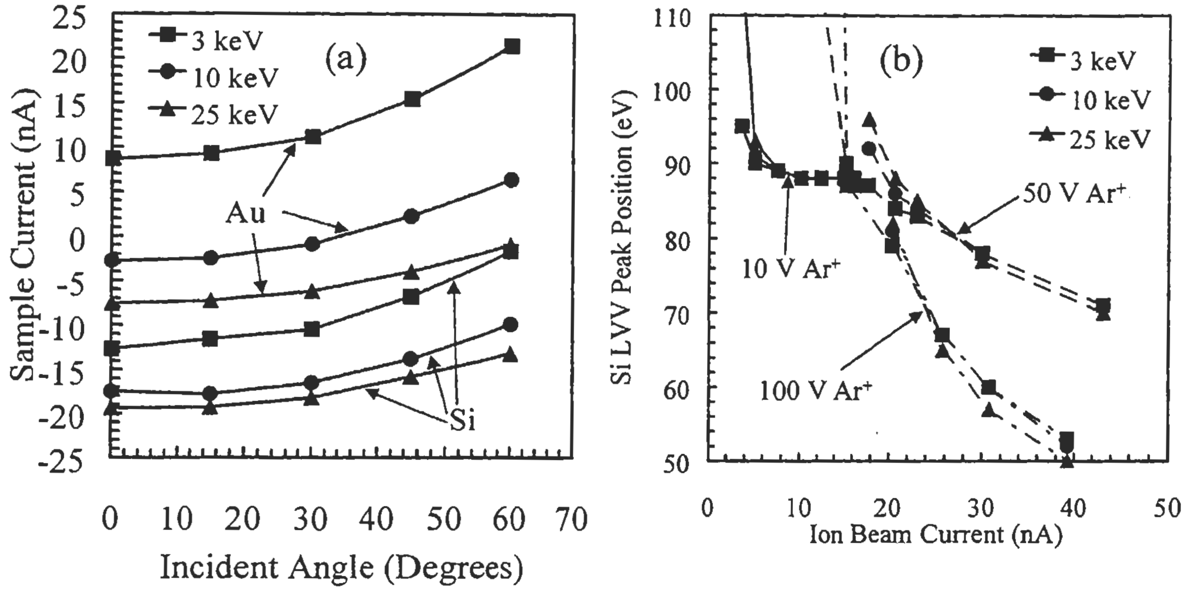

Figure 3. (a) Sample current at gold and silicon surface as a function of the incident angle of primary electron beam. (b) Si LVV peak position measured at the electrically insulated silicon surface by disconnecting the sample current cable as a function of the ion beam current. Electron beam was scanned in 100 × 100 $\mu m^2$ at 25 nA for both experiments

at 3 kV in any incident angles and at 10 kV over 35° of the incident angle and silicon Auger peaks were shifted at any beam voltages on electrically insulated surfaces. It is to be concluded that the surface potential will be positive potential with respect to ground when the sample current shows positive value, and some of the abundant low energy secondary electrons will move back to the sample, which will self-adjust the sample potential to ground[2]. Figure 3 (b) shows Si LVV peak positions at the electrically insulated silicon surface as a function of the ion beam current for 30° incident angle of 25 nA primary electron beam scanned in 100 × 100 $\mu m^2$. The incident angle of the low energy ion beam was fixed at 47°. Silicon is a conductive sample, so that the charging is compensated by adjusting the ion beam current as well as the absorption current of primary electron beam rather than adjusting the beam current density. The critical ion beam currents for charge compensation at each electron beam voltages are in good agreement with absorption current of silicon. The peak position was not shifted by over irradiation of 10 V ion beam in any electron beam voltage. It is, however, difficult to adjust the peak position by ion beam irradiation for 50 V and 100 V ion beam voltage in any beam voltage. It is to be concluded that the ultra low energy ion beam irradiation is more effective for the charge compensation.

**3C. Results of Fused Quartz and Glass plate**

Figure 4 (a) shows O KLL peak positions at the surface of fused quartz of 1 mm thickness ($\rho \sim 10^{16}\ \Omega$ m) with 3 kV 5 nA electron beam in 100 × 100 $\mu m^2$ raster as a function of the incident angle of primary electron beam. O KLL peak positions were shifted in any incident angles without ion beam irradiation, however the charge was compensated easily over 40° of the incident angle with ion beam irradiation. The ion beam current was around 1 to 3 nA at 100 V, which was same order of the primary electron beam current. Below 40° of incident angle, severe charging was observed with ion beam irradiation, and O KLL peak was not observed. The electron beam current density was still equivalent to that of irradiated ion beam, however the charge was not compensated at all even increasing the ion beam current by raising the ion beam voltage. Another condition of the ion beam need to be examined.

For a high spatial resolution analysis on the insulator material in AES, the glass plate of 1 mm thickness was prepared ($\rho = 5 \times 10^{14}\ \Omega$ m). Figure 4 (b) shows O KLL peak position obtained with 3 kV 5 nA electron beam which corresponded to the beam size of 80 nm in

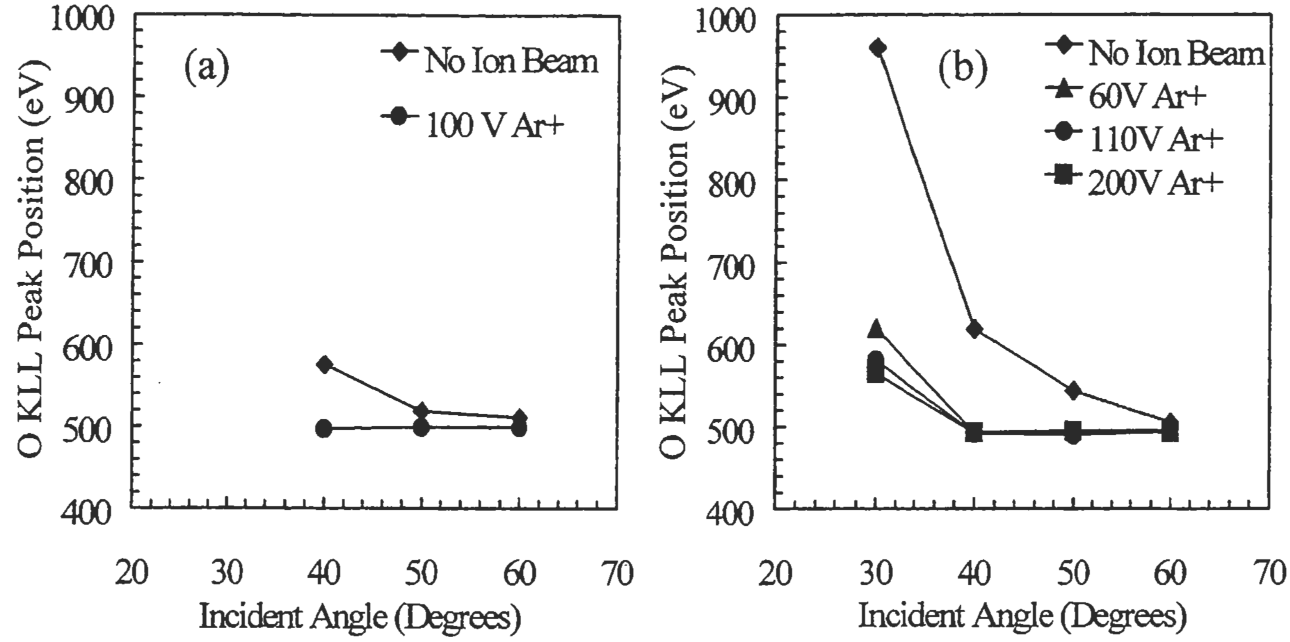

Figure 4. O KLL peak position obtained at fused quartz surface ($\rho = 10^{18}$ Ω cm) (a) and glass surface ($\rho = 5 \times 10^{16}$ Ω cm) (b) as a function of the incident angle of primary electron beam. The electron beam of 3 kV 5 nA was scanned on fused quartz (a) in 100 × 100 $\mu m^2$ and not scanned for glass plate (b), of which beam size was evaluated to be 80 nm in diameter.

diameter. The charge was compensated over 40° of the incident angle even electron beam was not scanned, which corresponds to the beam current density of $\sim 6 \times 10^5$ $Am^{-2}$. The beam current density of primary electron beam was extremely larger than that of ion beam. This glass contains alkaline-earth metals (Mg and Ca) and boron, so that diffusion and segregation of these elements need to be considered, which may reduce the electrical resistivity[3].

In charge compensation process, low energy ion is irradiated beyond the irradiation area of the primary electron and the insulator surface becomes positive potential with respect to ground because of the beam size of the ion beam. Therefore low energy ions are decelerated by positive potential, then ions will not reach to the irradiation area of the primary electron beam with certain incident angle of the primary electron beam even increasing the ion beam current. In this case, the irradiation area of the primary electron beam has severe charging. Increasing the ion beam voltage, ions will reach to the irradiation area of electron beam, which explains the result of Figure 4 (b). In addition, the sample damage by sputtering is to be considered. The performance using helium ion will be expected as well as that of argon ion because the space charge of helium ion is smaller than that of argon ion in the floating column of the ion gun. Therefore the sample damage by sputtering can be avoided by low energy helium ion irradiation.

## 4. Conclusions

Charge compensation using low energy ion irradiation by floating column ion gun was demonstrated. In conclusion, low energy ion irradiation is more effective than only changing the incident angle of the primary electron beam for high electrical resistivity sample. For improving the charge compensation, additional "low energy electron" irradiation needs to be considered to compensate the positive potential generated by low energy ion around the electron beam irradiation area.

## 5. References

[1] D. H. Narum: J. Vac. Sci. Technol. B 11, 2487 (1993)
[2] S. Hoffmann: J. Electron Specrosc. Relat. Phenom. 59, 15 (1992)
[3] J. Cazaux and P. Lehunede: J. Electron Spectrosc. Relat. Phenom. 59, 49 (1992)
[4] S. Ichimura, H. E. Bauer, H. Seiler and S. Hofmann, Surf. Interface Anal. 14, 250 (1989)
[5] P. E. Larson and M. A. Kelly: J. Vac. Sci. Technol. A 16, 3483 (1998)