

## Optical Report

# Charge Compensation Studies for Insulators in AES by Low Energy Ion Beam Irradiation

Hideo IWAI, Tomohiko MOROHASHI and Akihiro TANAKA

ULVAC-PHI, Inc., 370 Enzo, Chigasaki, Kanagawa 253-0084, Japan

### 1. Introduction

The recent AES instrument equipped with field emission electron gun (FE-AES) improves the spatial resolution[1], however the primary beam current density becomes extremely high, which makes more difficulty to measure insulators. The most traditional method for reducing the charging is 1) increase of secondary electron emission from the surface by increasing the incident angle of electron beam (or tilting the sample), 2) metal foil mask with small opening for analysis, 3) evaporation of conductive material such as gold and graphite, 4) low energy electron beam irradiation on the insulator sample[2][3][4]. However, those methods are not successfully worked on the high resistivity insulators with FE-AES instrument. The simultaneous irradiation of low energy electron and ion beam had been discussed for the insulator in XPS with monochromatic X-ray[5]. Thus, low energy ion irradiation may be effective for the insulators in AES. In this report, 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, PHI Model 680 FE-AES instrument equipped with the floating column ion gun was used. The maximum ion beam current and ion beam current density at 100 V beam voltage are the order of 100nA and  $10^{-1} \text{ Am}^{-2}$ , respectively[6]. The angle between primary electron beam and ion beam is  $77^\circ$ . Tilt axis of a sample stage is perpendicular to the plane of electron beam and ion beam. In this study, argon gas was used.

### 3. Results and Discussion

Figure 1 shows O KLL peak positions at the surface of fused quartz of 1 mm thickness ( $\rho \sim$

$10^{16} \Omega \text{ m}$ ) with 3 kV 5 nA electron beam in  $100 \times 100 \mu\text{m}^2$  raster as a function of the incident angle of primary electron beam[6]. O KLL peak shifts were observed in any incident angles of electron beam without ion beam irradiation. However the charging was compensated easily over  $40^\circ$  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  $40^\circ$  of incident angle, severe charging was observed even 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 and raising the ion beam voltage up.

High spatial resolution analysis is one of the advantage for FE-AES, so the glass plate of 1 mm thickness was prepared ( $\rho = 5 \times 10^{14} \Omega \text{ m}$ ).

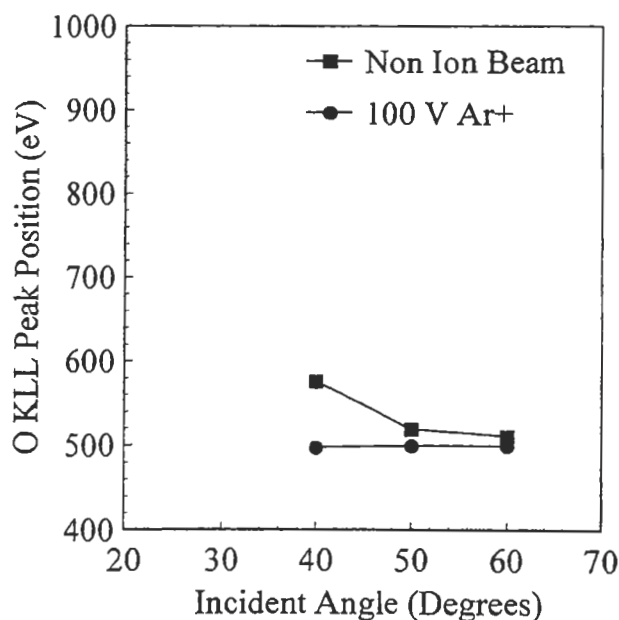


Figure 1[6] O KLL peak position obtained at fused quartz surface ( $\rho = 10^{18} \Omega \text{ cm}$ ) as a function of the incident angle of primary electron beam. The electron beam of 3 kV 5 nA was scanned in  $100 \times 100 \mu\text{m}^2$ .

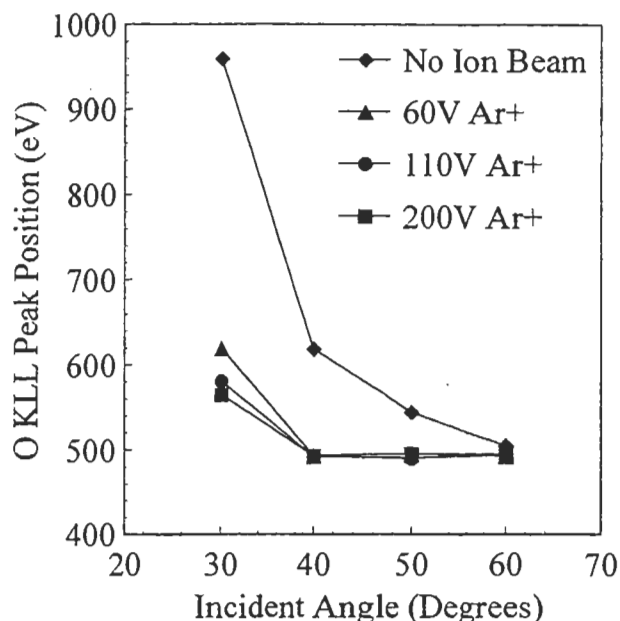


Figure 2[6] O KLL peak position obtained at glass surface ( $\rho = 5 \times 10^{16} \Omega \text{ cm}$ ) as a function of the incident angle of primary electron beam. The electron beam of 3 kV 5 nA spot beam was irradiated, of which beam size was evaluated to be 80 nm in diameter.

Figure 2 shows O KLL peak position measured with 3 kV 5 nA electron beam (beam size is around 80 nm in diameter)[6]. The charging was compensated over  $40^\circ$  of the incident angle, even electron beam was not scanned, which corresponds to the beam current density of  $\sim 6 \times 10^5 \text{ Am}^{-2}$ . The beam current density of primary electron beam is extremely higher 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].

From both results, we have considered the positive charging on the insulator generated by low energy ion beam irradiation. The 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 is much larger than that of electron 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. The

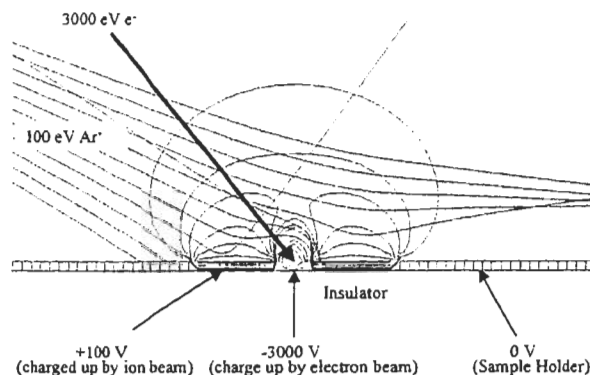


Figure 3. Simulation result of charged up surface.

simulation result is shown in Figure 3, which explains well about our discussion.

In addition, the sample damage by sputtering is to be considered. The performance using helium ion may be expected as well as that of argon ion because the space charge using 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 on the insulator surface

#### References

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