

## Recent Developments and Applications in AES and XPS

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This paper reports improvement of the spatial resolution in AES analysis and depth profiling of polymer films by C<sub>60</sub> ion beams in XPS. In AES, the acoustic enclosure was integrated to eliminate the image jitter and image drift from the environment. The AES spatial resolution was measured as 6 nm from a gold on graphite sample. In XPS depth profiling, the chemical states on polymers were maintained after C<sub>60</sub> ion beam sputtering.

### INTRODUCTION

Smaller dimensions in new semiconductors and other engineered materials have created a demand for higher magnification measurements. Recently, the incorporation of a Schottky field emitter and multi-channel detector integrated in the electron energy analyzer has made a significant improvement of spatial resolution and sensitivity of AES analysis [1]. While Secondary Electron Imaging (SEI) spatial resolution below 5 nm has been achieved with commercial instruments, the magnification for Auger line scan and mapping measurements has generally been limited below 100,000× with spatial resolution greater than 10 nm. AES spatial resolution will also degrade with the electron beam size broadening associated with larger beam currents and from backscattered electrons [2]. In addition, electron beam jitter and drift with respect to the sample (so called image jitter and drift) is usually observed with high magnification measurements because the acquisition time of an Auger measurement is much longer than that of an SEI image. To avoid this problem, we have developed an AES instrument, named the PHI 700, which is isolated from the environmental affects such as acoustic noise, air circulation and drift of the laboratory temperature.

The miniaturization of many consumer products has also increased the demand for improved spatial resolution in XPS. Today, XPS micro spectroscopy is generally limited to around 8 μm in commercial instrumentation [3]. Another important demand for

XPS is the sample cleaning of contamination from the top surface of polymers and the depth profiling of organic thin films with low sample damage. To address this demand, certain preparation techniques such as ion sputtering, sample scribing or thin-film formation are required *in-situ* [4]. Argon sputtering is typically used for most XPS instruments, however the surface chemistry of the polymer is severely damaged. The buckminsterfullerene(C<sub>60</sub>) ions are known to cause less atomic mixing of samples in time-of-flight SIMS experiments [5-6] and molecular dynamics calculations [7]. The C<sub>60</sub> ion beam sputtering of the surfaces of several polymers has been studied and compared with the chemical damage observed with argon ion beams sputtering [4]. We have also applied C<sub>60</sub> ion beams for depth profiling of organic thin films and considered the potential for broader applications of the C<sub>60</sub> ion beam in XPS.

### DEVELOPMENT AND APPLICATIONS IN AES

#### *AES Instrumentation*

The major causes of the degradation of spatial resolution in Auger line scan and Auger mapping are image jitter and drift during data acquisition. The analytical chamber for AES instrument is based on UHV technology, so the thickness of the chamber is usually thinner than that of the chamber for an SEM instrument. In addition, the other UHV components such as a magnetic coupling sample transfer

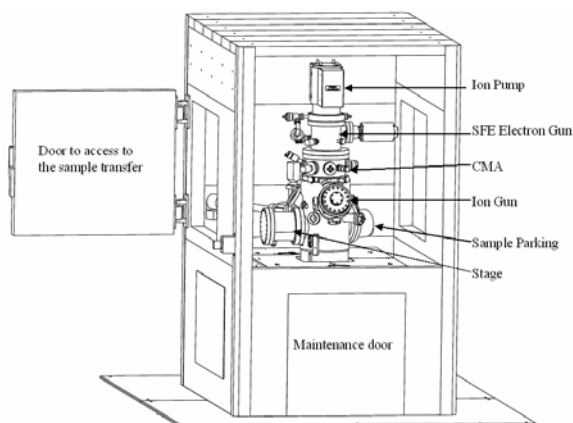


Fig. 1 The drawing of a newly developed AES instrument with an acoustic enclosure [8].

mechanism and an ion gun with differential pumping are integrated into the Auger instrument. These components have resonant frequencies which can transfer energy for mechanical vibration to the sample stage. For example, a sample transfer mechanism has a resonant frequency of 25 to 30 Hz, while the flexible tube for differential pumping has resonant frequencies between 50 to 200 Hz. In general, mechanical vibration is transferred from the floor so vibration isolators are integrated in the instrument. However the vibration isolators do not reduce the vibration originating from acoustic noise. We have designed the instrument surrounded by an acoustic enclosure to reduce the sound pressure from the environment around the instrument, as shown in Fig. 1 [8]. The reduction of sound pressure inside the acoustic enclosure is  $\sim 1/10$  of the laboratory environment in the frequency range of 1-8000 Hz.

The acoustic enclosure also acts as a thermostatic room. In previous commercial instruments, the image drift is typically about 50 nm/h with image drift correction software (image registration) and the instrument in a stabilized condition. The image drift of the PHI 700 instrument is less than 5 nm/h with image registration every 5 minutes. Fig.2 shows a Au MNN line scan over gold on graphite, acquired with 33 minutes signal averaging at 20 kV 1 nA, overlaid on the SEI image. The AES spatial resolution was determined to be 6 nm at 20% to 80% intensity of the line scan. Monte Carlo simulations show that the 20 kV energy electron beam penetrates deeply into the bulk for

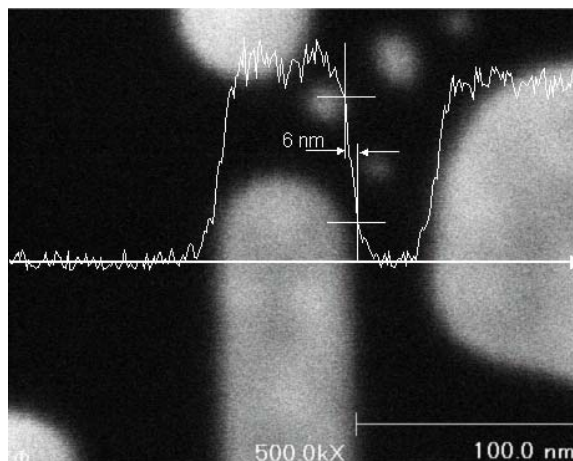


Fig. 2 Au MNN line scan over gold on carbon measured by 20 kV 1nA electron beam is overlaid on the SEI image. Acquisition time was 33 minutes.

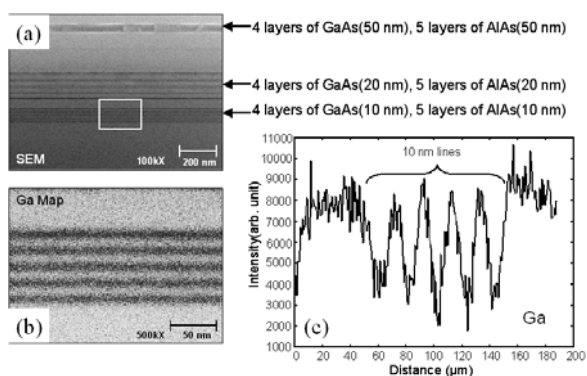


Fig. 3 The SEI image (a) of AlAs/GaAs superlattice. The rectangular line inset on the SEI image was the area for Ga LMM mapping (b). The Ga LMM line scan (c) was acquired in vertical direction on the Ga Auger mapping.

samples such as C and Si, so the degradation of the Auger spatial resolution from backscattering electrons is small [9-10].

#### AES line scan and mapping of gaas/alas superlattice

A GaAs/AlAs superlattice was employed in order to evaluate the Auger spatial resolution. This sample has 5 cycles of AlAs layers and 4 cycles of GaAs layers with 10 nm, 20 nm and 50 nm thickness, respectively, as shown in Fig. 3 (a). The Ga LMM line scan was measured on 10 nm thickness layers in 2 minutes acquisition time, as shown in Fig. 3 (c). The 10 nm layers are clearly resolved. To test the system stability, the Ga LMM mapping was acquired in 200 minutes, as shown in Fig. 3 (b). These results show that the image stability of the PHI 700 is

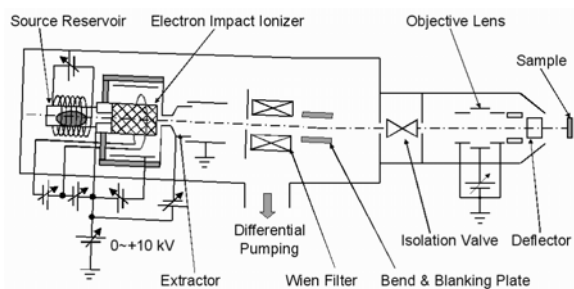


Fig. 4 Schematic diagram of  $C_{60}$  ion gun column [11]. The distance from flange to the sample is 200 mm. A maximum beam current is  $>100$  nA at 10 kV beam voltage. A sputter rate was measured as 2 nm/min on thermally oxidized  $SiO_2$ .

sufficient for high magnification applications in AES and allows high sensitivity AES analysis for sub 10 nm features. For some samples, the AES spatial resolution can be degraded by backscattering electrons. To quantify the AES spatial resolution, we therefore propose that a sample which has a line shape structure between the heavy atomic unit element and light atomic unit element, such as gold on graphite or GaAs/AlAs superlattice.

## DEPTH PROFILING OF ORGANIC FILMS BY $C_{60}$ ION BEAMS

### Experimental

Fig. 4 shows the schematic diagram of the  $C_{60}$  ion gun column [11]. The  $C_{60}$  powder is evaporated and injected into an electron impact ionizer. The  $C_{60}$  ion beam is accelerated through the extraction lens which focuses the beam at the Wien filter

aperture for mass separation. The ion gun column has a 1 degree bend at the blanking plate for eliminating neutral particles. The  $C_{60}$  ion beam is focused at the working distance of 60 mm. The beam current specification is 20 nA at 10 kV beam voltage with mass filtering by the Wien filter. The  $C_{60}$  ion gun was installed on the auxiliary chamber of the PHI Quantera SXM with an incidence angle of  $70^\circ$ . The sample was transferred from the stage to the auxiliary chamber when the sample was sputtered by  $C_{60}$  ion beam. All the XPS data were acquired with monochromatic Al  $K\alpha$  X-ray source at  $100\mu m$  diameter. The photoelectron detection area was defined by the X-ray probe size. The data was acquired at  $45^\circ$  take-off angle and the analyzer acceptance angle was  $\pm 20^\circ$ , for all the measurements. The pass energy of the analyzer was 55 eV. The sputter rate was measured 2 nm/min on a thermally oxidized  $SiO_2$  thin film on a silicon wafer.

### Depth profiling of a laminated polymer film

A laminated polymer film (gelatin and latex layers on the polyester) was employed to evaluate the depth profiling by a  $C_{60}$  ion beam. Fig. 5 (a) shows the XPS depth profile of the C 1s, O 1s, N 1s and S 2p spectra. The gelatin layer, the latex layer and the polyester base film could be defined from the intensity profiles of the N 1s and S 2p peaks. The total thickness of gelatin and latex layers was estimated to be 20 nm defined by  $SiO_2$  sputter rate

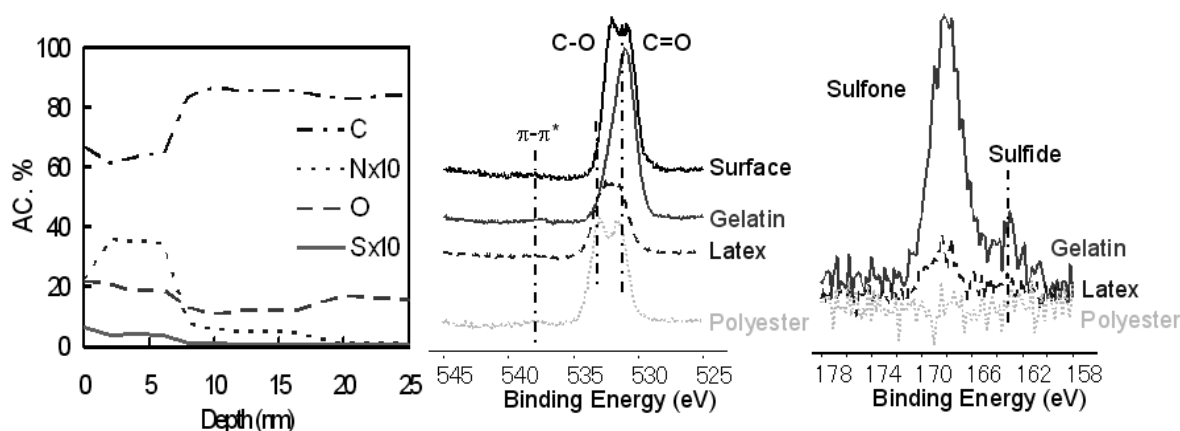


Fig. 5 The depth profiling of a laminated polymer film (gelatin (~100 nm) / latex (~100 nm) layers on the polyester) (a) and the XPS spectra of O 1s and S 2p (b) by 10 kV  $C_{60}$  ion beam. The sputtering depth was estimated by the  $SiO_2$  sputter rate.

(2nm/min), while the expected total thickness of the gelatin and latex layer is 200 nm. The sputter rate for this polymer laminating film is therefore estimated to be ~20 nm/min. In the latex layer, N and S were also detected. At present, it is difficult to determine if this is diffusion from the gelatin layer or sample damage. The O 1s and S 2p XPS spectra are shown in Fig. 5 (b) to evaluate the chemical states in each layer. The O 1s spectra show 3 patterns. The base polyester and latex layers have the expected 1 to 1 ratio of C-O and C=O peaks that shows that no chemical state damage has occurred, even to a sputter depth of 200 nm. Above the base polyester and the latex layers, a peak to slightly lower binding energy to the C=O peak can be assigned to the gelatin chemistry. The surface of the gelatin film has a third O 1s spectrum. The chemical states of S 2p in the gelatin layer were assigned to be sulfone and sulfide. It is well known that sulfone can be damaged by an ion beam to form sulfide. The depth profile results show minimum chemical reduction of sulfone to sulfide in the gelatin layer. These results demonstrate that the chemical state information can be maintained while performing a C<sub>60</sub> ion beam sputter depth profile for some organic materials.

## SUMMARY

The new PHI 700 AES instrument has been designed with an acoustic enclosure to minimize the image jitter and image drift introduced by the laboratory environment. An AES spatial resolution of 6 nm was measured for a gold on graphite sample. The AES mapping of a AlAs/GaAs superlattice with 200 minutes data acquisition also shows no degradation in spatial resolution. The reduction of image jitter and image drift has been shown to significantly enhance spatial resolution.

A new XPS depth technique for polymer materials has been developed with a C<sub>60</sub> ion beam. The depth profiling of a laminated polymer film was employed for the evaluation. The results showed 3 layers of organic structure. The chemical states of O 1s and S 2p were observed in each layer without evidence of damage associated the C<sub>60</sub> ion beam sputtering. These results indicate that C<sub>60</sub> ion beam sputtering may be a powerful method for the depth profiling of organic materials.

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