Photoemission measurements of Ultrathin SiO_2 film at low take-off angles

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(Received; 04 Dec 2008 and Accepted; 10 Dec 2008)

Abstract
The surface and interfacial analysis of silicon oxide film on silicon substrate is particularly crucial in the nano-electronic devices. For this purpose, series of experiments have been demonstrated to grow oxide film on Si (111) substrate. Then these films have been used to study the structure of the film by using X-ray photo emission spectroscopy (XPS) technique. The obtained results indicate that the photoemission measurements on the ultrathin oxide should be done at low take-off angles to emphasize the oxide features.
Keywords: *Thin film, Surface, Interface, Silicon oxide, XPS technique.*

1. INTRODUCTION

The development of microelectronics has entered the nano metric range due to the continuous shrinking of electronics device dimensions. The semiconductor technology, one of the critical point in this process is progressing in the real measurements which are based on firm experimental techniques.
On the other hands, the interface between gate oxide dielectric and silicon is important, because the transistor's performance and the reliability of devices are determined and controlled by gate and gate oxide dielectric of the transistor. Moreover, the role of this interface becomes even more important as the oxide becomes thinner and thinner due to accommodating the faster speeds in integrated circuits.
Therefore oxide of silicon is a uniquely critical material within silicon technology as we know it is a major component for MOS (Metal-Oxide-Semiconductor) and MIS (Metal-Insulator-Semiconductor) devices. This vital role of very thin dielectrics has been the motivation for characterization of the oxide silicon in the thin and ultrathin film regimes.
In this paper, it doesn't pay attention to the films chemistry in details, because the chemistry of the films are discussed in the literatures [1-15]. The analysis of SiO_2 /Si interface is difficult, scientifically due to so thin interface layer of one to two atomic monolayer(s).
These issues cause practical difficulty of detecting such a small amount of material, bonding-ordering intermixing of the atoms (if only) configurations. However, it has been tried to analyze SiO_2 /Si (111) interface structure by using XPS technique in the different take-off angles. The measurement of ultrathin layer is possible only at low take-off angles down to 1-2 degree(s).

2. EXPERIMENTAL PROCEDURE AND DETAILS

The photoemission experiments were carried out at the electron storage ring with the light from a bending magnet using a spherical grating monochromator. This equipment has a significantly better performance in terms of resolution and transmission than the other systems.
The n-type silicon samples with resistivity of 5 Ω - cm and size of 3 cm × 1 cm were cut out of the wafer then rinsed with ethanol in an ultrasonic bath and then introduced in the UHV chamber. The chamber was then baked before the measurements.

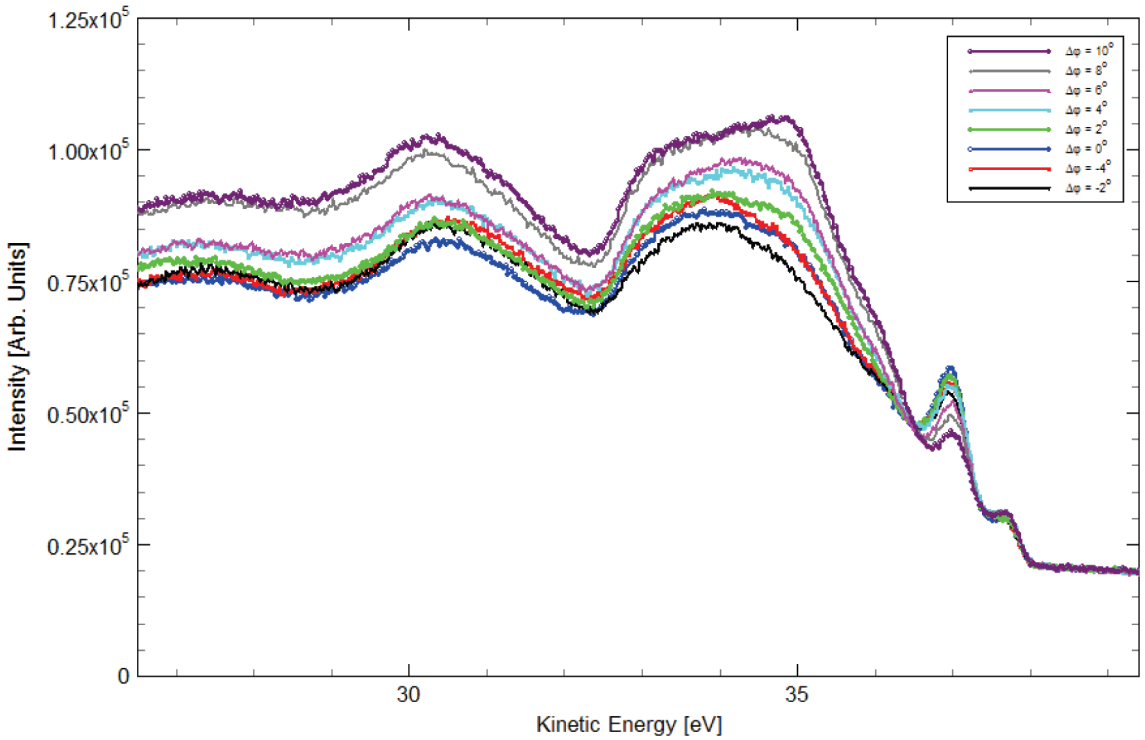

Figure 1: Angle resolved $\Delta\varphi$ valence band of the clean Si(111) surface as recorded with 38 eV photons. The background (due to secondary electron emission) is high.

The background pressure was 2 $\times 10^{-10}$ Torr after baking. All further cleaning was done inside the UHV chamber by heating with a direct current through the sample, initially up to 1200 °C and later at higher temperatures to restore a clean Si surface. Earlier measurements with a residual gas mass spectrometer in the line of the beam have shown that a very high proportion (about 50 percent) of oxygen is produced with this setup. Typical total pressure in the chamber during exposure was around 5×10^{-7} Torr.

Fig.1 shows a collection angle variation of valence band of silicon oxide film. As one can see in Fig.1, most of the collected electrons originate from atoms near the surface at very low tack-off angles. It means that more electrons under this condition can be collected from cathodes deeper in the sample and exploited to determine composition as a function of depth.

Furthermore, electrons near the exited atom in the solid, respond to the generation of charge, lowering the energy of the ionized final state, and giving the photoelectrons with more kinetic energy (less binding energy).

Practically, XPS experiments were performed with a SPECS PHOIBOS analyzer. This instrument is equipped with Al and Mg x -ray sources and a concentric hemispherical analyzers.

All Si samples were mounted on a holder with Ta clips, which allows direct current heating.

Fig.2 shows the XPS spectrum of a silicon sample using Mg Kα cathode. This figure shows the counting (or intensity) rates acquired, on the vertical scale, and demonstrates clearly the narrow core- like levels labeled 1s, 2s, and 2p.

All XPS measurements were made at different take-off angles with respect to the sample surface plane in UHV. The take-off angles should be chosen so

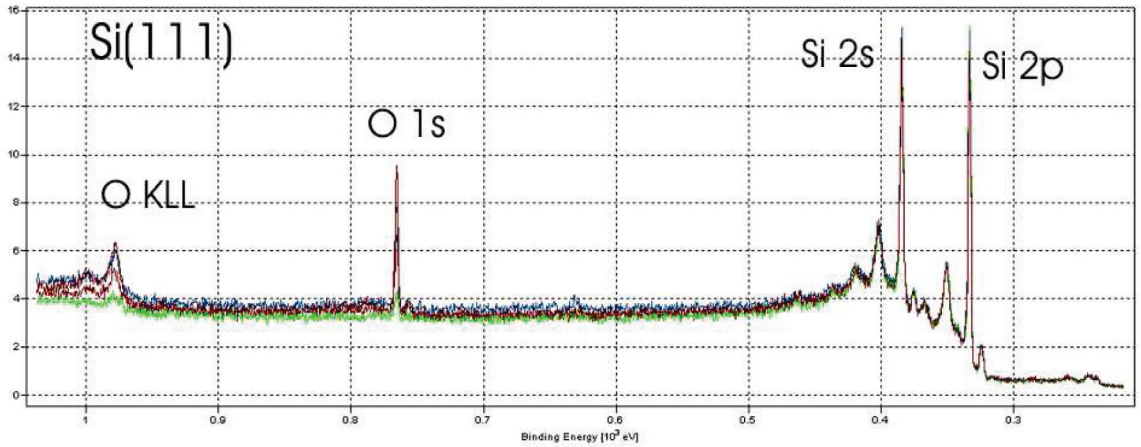

Figure 2: XPS spectra of silicon oxide on Si(111) at 600 °C (Oxygen exposing time: 10, 15, 20 and 30 min) and PO2 = 4.5 × 10^{-8} Torr.

that we could minimize photoelectron direction and get more intensity of electrons which originating from the substrate at certain take-off angles.

3. DISCUSSION

As mentioned above, the analysis of ultrathin film structures need advanced surface sensitive techniques, because the interface between film and substrate consists of 1-2 mono layer(s) which correspond to 3×10^{15} atoms/cm^2 or a more 5×10^{-9} moles/cm^2 . It obviously causes difficulty of evaluating of the interface structures.

Among the surface sensitive techniques, the XPS technique is one of the surface sensitive techniques which can detect impurities and dirties on the film, the surface and the substrate. The reason is due to its limited escape depth of photoelectrons. As we know, in this technique, X-ray typically with an energy range between 20 and 2000 eV, impinges in the sample and causes ejection of electrons from the inner shells of the atoms of the sample.

According to the energy conservation law, the kinetic energy (KE) of the photoelectrons can be obtained from the below relation.

$$KE = h\omega - BE - Wf \quad (1)$$

Where $h\omega$ is the incoming photon energy and Wf is the spectrometer work function. In order to determine the binding energy (BE) of the core electrons, we use both Mg and Al anodes which are rotating anodes for focusing the monochromatic X-ray on the sample in which the intensity of the photoelectrons signal(I), can be found as a function of the sample depth, z, as follow :

$$I = I0 \exp(-z/\lambda \cos(\theta)) \quad (2)$$

Where I0, θ and λ are respectively intensity per surface area, the collection angle of the emitted photoelectrons respect to the normal surface and the escape depth of photoelectrons.

Furthermore, the escape depth for electrons from the Si_2P shell in the silicon oxide film is about 3.7 nm or a little more due to structural defects at the SiO_2/Si (111) interface.

As shown in Fig. 2, it can be seen that the out coming electrons affect significantly the electrons near the exited atom. It will produce some surface Plasmons, interband transition and noise in the XPS spectra. In addition to these effects, the relaxation energy due to intra-atomic and extra-atomic contributions gives the photoelectrons more kinetic energy as it would have if we consider only single electron effects.

The other points in the XPS measurements are

screening the coulomic attraction between the photo electron and the nucleus in which the exited electrons induce several artifacts such as mixing of atoms at the interface and implantation of the penetrating X-ray into the film. These effects will contribute to degradation of the Si_2P, Si_2S and O1s intensity.

4. CONCLUSION

In this work, the efforts concern to obtain valance band spectra of 1 to 2 monolayer(s) silicon oxide film on the silicon wafer with different take-off angles, were followed or paralleled by various ways to achieve oxide growth and see at which take-off angle better spectrum can be measured.

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