

Extended Abstract of PSA-19 (review)

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# Traceable Thickness Measurement of nm Oxide Films by Mutual Calibration

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Mutual calibration has been known to be a traceable method to determine the absolute thickness of nm oxide films. Recently, the thicknesses of a series of ultra-thin HfO<sub>2</sub> films were compared in a CCQM pilot study P-190 by 11 NMIs using various thickness measurement methods. The reference thicknesses of the films were precisely determined by mutual calibration by the average thickness of x-ray photoelectron spectroscopy (XPS) and x-ray reflectometry (XRR) data. The film thicknesses were also measured by medium energy ion scattering spectroscopy (MEIS). The film thicknesses measured by MEIS were linearly proportional to the reference thicknesses and the thickness difference was about 2%.

## 1. Introduction

Thickness measurement of ultra-thin oxide films is one of the most important measurement issues for semiconductor devices. The thickness measurement of nm SiO<sub>2</sub> films was the subject of first pilot study P-38 [1]. In this pilot study, large offset values in the range from 0.5 nm to 1.0 nm were reported in spectroscopic ellipsometry (SE), transmission electron microscopy (TEM), Rutherford backscattering spectrometry (RBS), MEIS and XRR. Fortunately, however, the offset value of XPS was found to be zero. From this result, mutual calibration method was designed to determine the absolute thickness of nm oxide films [2]. The mutual calibration method by XPS and TEM has been applied to the thickness measurements of ultra-thin SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> films [2-4].

HfO<sub>2</sub> is an important dielectric material that can be used as an alternative to SiO<sub>2</sub> for the fabrication of memory devices. Therefore, a traceable thickness measurement of ultra-thin HfO<sub>2</sub> films by physical or chemical methods is required for the advanced semiconductor industries. In this reason, the thickness measurement of nm HfO<sub>2</sub> films was investigated in the recent pilot study P-190. The certified reference thicknesses of six HfO<sub>2</sub> films were determined by mutual calibration between the average thicknesses of XPS and XRR in P-190.

MEIS is known to be an absolute method to determine the amount of substance of ultra-thin films. In this study, MEIS was investigated as an offset traceable method for the application of

mutual calibration method. MEIS was also investigated as a traceable method to determine the absolute thickness of ultra-thin HfO<sub>2</sub> films without combination with other method.

## 2. Film Thickness by Mutual Calibration Method

Thickness of ultra-thin oxide films is difficult to be determined by a single surface analysis method, such as, XPS, XRR, TEM, SE and RBS due to the large offset value [1]. Mutual calibration by combination of an offset traceable method and a length-unit traceable method was suggested as a useful way to determine the absolute thickness of ultra-thin oxide films as shown in Fig. 1 [2]. XPS and MEIS can be used as offset-traceable methods because the film thickness is proportional to the amount of film materials from the basis of the number density of c-Si. The offset value of XPS and MEIS is theoretically zero because the amount of the film material should be zero when the film thickness becomes to zero.

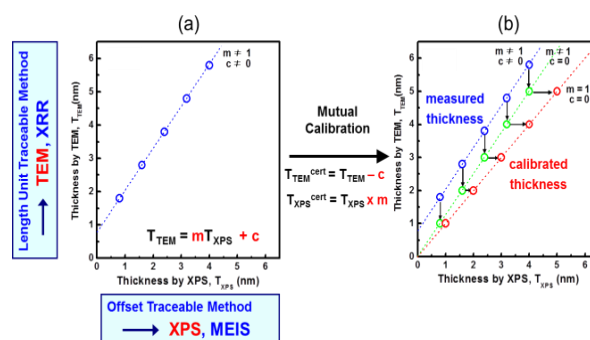


Fig. 1 Schematic illustration of mutual calibration [2].

TEM and XRR can be used as length-traceable methods because the film thickness is based on the lattice constant of crystalline Si and the wavelength of x-ray in HR-TEM and XRR, respectively. From a series of oxide films with the same property and different thicknesses, the absolute thickness can be determined from the slope and offset values in the linear fitting line of the thicknesses by the offset traceable method in the x-axis and a length-unit traceable method in the y-axis.

### 3. Determination of Reference Film Thickness

HfO<sub>2</sub>(1~4 nm)/SiO<sub>2</sub>(2 nm)/Si(100) films were grown on Si (100) substrates by atomic layer deposition. The SiO<sub>2</sub> layers were grown by thermal oxidation to prevent the diffusion of oxygen atoms from the HfO<sub>2</sub> films to the Si substrate. For comparison, the reference thicknesses of the HfO<sub>2</sub> layers were determined by mutual calibration between the average thicknesses by XPS and XRR reported in the pilot study P-190. The perfect linear relationship of the linear fitting line strongly supports the feasibility of the mutual calibration method. The reference thicknesses are 0.76, 1.20, 1.64, 2.08, 2.52 and 3.36 nm, respectively. These reference thicknesses can be regarded as certified values because they are measured by 11 national metrology institutes and they were based on the thickness by XRR where the thickness scale is based on the wavelength of x-ray.

### 4. Results and Discussions

The thickness of the HfO<sub>2</sub> films was measured by a MEIS system (K-120, KMAC, Korea) with a time-of-flight (TOF) analyser as shown in Fig. 2 [5]. TOF analyser is strong for the thickness measurement of ultra-thin oxide films because the sensitivity is high and all scattered ions can be analysed in the same time.

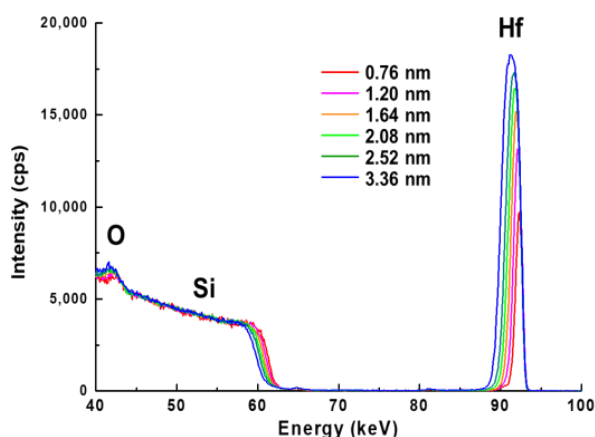


Fig. 2 MEIS spectra of a series of HfO<sub>2</sub>/SiO<sub>2</sub>/Si(100) films used in pilot study P-190 [5].

From the energy distribution of scattered ions, the in-depth locations of the constituent atoms can be determined from the kinematic factor and the amount of the atoms can also be determined from the scattering cross section of the atoms.

The thickness of HfO<sub>2</sub> films could be determined from the measured number of Hf atoms within a unit area and the number density of the bulk HfO<sub>2</sub>. The numbers of the constituent Hf atoms were estimated by simulation (MEISis) of the obtained MEIS spectra including the calibration of stopping power.

The linear fitting result of the measured thickness to the reference thicknesses shows a slope value of 0.981 and an offset value of 0.017 nm. The slope value of 0.981 means that the measured MEIS thicknesses of the HfO<sub>2</sub> films are close to the certified reference thicknesses within 2% [5].

### 5. Conclusions

The thickness of the ultra-thin oxide films can be determined by mutual calibration with an offset traceable method and a length-unit traceable method. The thicknesses of nm HfO<sub>2</sub> films measured by MEIS were traceable to the reference thickness within the thickness difference of about 2%. As a result, MEIS can act as a traceable method to determine the thickness of nm oxide films.

### 6. References

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