In a recent development of metal oxide semiconductor (MOS) devices, the thickness of a gate oxide has been reduced to 1 nm or even less. X-ray reflectometry is a useful tool for evaluating the thickness, density profiles and surface/interface roughness for thin films. This method is even applicable to very thin films, although it is not easy to evaluate films less than 1 nm thick. We studied the feasibility of evaluating such ultrathin films based on synchrotron radiation in comparison to the conventional X-ray source.

It is well known that the angular dependence of reflected intensity of X-rays from thin films shows the oscillation known as the Kiessig fringe, which provides the information on the layers. The oscillation intervals ($\Delta 2\theta$), which come from the interference of X-rays at a thin film, can be approximated as $\Delta 2\theta = \lambda / t$ [1], where $\lambda$ is the X-ray wavelength, $t$ is a layer thickness, and $\theta$ is the glancing angle of an X-ray. According to the equation, the interference interval increases with decreasing layer thickness. This relation indicates that the measurement of wide area of $\theta$ is requested to precisely evaluate a thin film. Since the reflected X-ray quickly decreases as $1/\theta^4$, a high-flux X-ray is required for obtaining data at high $\theta$ angles.

We compared the performance of X-ray reflectometry for a 1-nm-thick gate oxide using synchrotron radiation from a bending magnet and from an undulator in addition to the laboratory X-ray generator.

In the experiment using synchrotron radiation, an X-ray wavelength of 1.4 Å was selected by the double crystal monochromator. A Rh-coated X-ray mirror in the beamline was used to suppress higher harmonic X-rays which contaminate the monochromatized X-ray.

In reflectivity measurements, a wide intensity range of X-rays needs to be detected, and for this we used the NaI scintillation detector with a Cu attenuator of appropriate thickness to measure each angular division. The Cu attenuator also contributes to the suppression of higher harmonic X-rays with its high attenuation rate above the absorption edge. Vacuum pipes were inserted in the X-ray path to prevent the absorption of X-rays by air, as shown in Fig. 1.

After the measurement, reflectivity from each angular division was corrected for the dead time loss and concatenated into the reflectivity of the whole region.

In the reflectivity measurements taken at high angles, the background events become comparable to specularly reflected X-rays. We measured the background intensity at the off-specular configuration and removed it from the specular intensity.

A thermally grown 1-nm-thick SiO$_2$ layer on an Si substrate was measured with these techniques. At the bending beamline BL16B2, we obtained the reflectivity with the intensity range over 8 decades.
and angular range around 10° (2θ) as shown in Fig. 2. On the other hand, at high-intensity undulator beamline BL16XU the intensity range of 12 decades and angular range around 40° was successfully obtained as shown in the same figure. In the figure, the reflectivity obtained from the laboratory equipment (Rigaku RGXR, 60kV-300 mA at 1.54 Å) is also shown for comparison, where an intensity of around 7 decades and angular range up to 6° (in 1.4 Å equivalent angle) was obtained.

Based on Equation (1), the layer thickness of 1 nm corresponds to the angular interval of 8°, which indicates that the laboratory equipment is insufficient, even for determining its thickness. The measurement at the bending beamline can determine the thickness around to 1 nm. However, it is insufficient for determining the density profile within a film. The measurement taken at the undulator beamline can provide precise information that includes a density profile for the advanced gate oxide below 1 nm.

We analyzed the reflectivity obtained at the undulator beamline by using model optimization based on the matrix formalism of Vidal and Vincent [1]. In the model, we sliced the layer into 0.1-nm (1 Å)-thick sublayers with different densities. The solid line in Fig. 2 (BL16XU data) shows the results of the model calculation, and in Fig. 3 the corresponding density profile is shown. In this 0.1-nm regime, these density profiles give information not only on the compactness of SiO₂ [2], but also on the location of silicon and oxygen atoms in films grown layer by layer [3] through their different electron densities.

Naoki Awaji
Fujitsu Laboratories LTD.

E-mail: awaji@ccg.flab.fujitsu.co.jp

References