MULTI-TECHNIQUE STUDIES OF ULTRATHIN SiO_2 FILMS

Current gate dielectrics in silicon based devices are only a few nm thick. Optical techniques such as ellipsometry are used to monitor film thicknesses and optical properties in production. However, for the current integrated circuit (IC) generation the accuracy of ellipsometry degrades because parameters such as thickness and index of refraction (which reflects the composition) become strongly correlated. Thus, it is difficult to unambiguously determine these parameters simultaneously, and the accuracy of ellipsometry would benefit from an independent calibration. In reflectometry techniques, on the other hand, these parameters are nearly decoupled. The thickness of a layer is approximately inversely proportional to the oscillation period of the reflected intensity, whereas the differences in scattering length density SLD (also an indicator of composition) between the layers is related to the amplitude of the oscillations. Neutron reflectometry (NR) is better suited than X-ray reflectometry (XR) for the study of the SiO_2/Si system because there is a relatively large contrast (or difference in SLD) between the scattering length densities of the two materials: 65 %, vs. 7.6 % for X-rays.

Consider as an example a sample with a nominally 10 nm thick thermal oxide film on silicon. This moderate thickness was chosen to increase our confidence in the results of the various characterization methods, while remaining thin enough that the results are relevant to film of technological interest. Figure 1 shows spectroscopic ellipsometry (SE) data and corresponding best fits for the sample with surface contamination and after an organic cleansing. Nominally, the only change is a decrease in the thickness of the contamination layer [1].

In XR data (Fig. 2) two oscillation periods are observed for the contaminated sample. The high frequency oscillation corresponds to the SiO_2 film, whereas the low frequency modulation is due to the thinner contamination layer (which is not present after cleaning, indicating removal of the contamination.)

The NR measurements (Fig. 3) were done in a vacuum to reduce the air scattering background. This allowed us to achieve a very large range in reflectivity, over 10^8, which is among the best examples in NR measurements to date. A slightly thinner contamination layer in NR is consistent with the fact that the XR was done in air, during which the contamination was growing. This was confirmed by changes in XR scans immediately following those in Fig. 2.

The average of the 5 measurements of the SiO_2 film thickness was 10.27 ± 0.13 nm. The excellent agreement among the results for the three different techniques increases our confidence in the

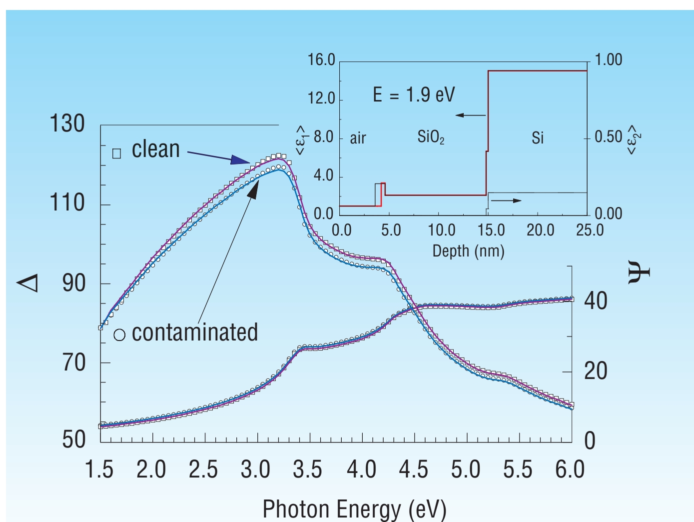

FIGURE 1. Comparison of spectroscopic ellipsometry experimental data, Ψ and Δ, to the fits (solid line) for the clean and surface contaminated sample. The inset shows < ε_1> and <ε_2> as a function of depth determined by the fit.

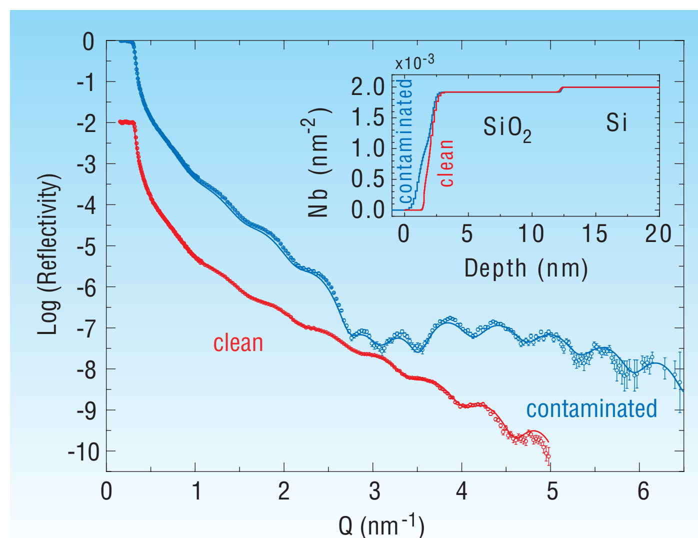

FIGURE 2. X-ray reflectivity and best fits for the clean and surface contaminated sample. The inset shows the scattering length density profile determined by the fits.

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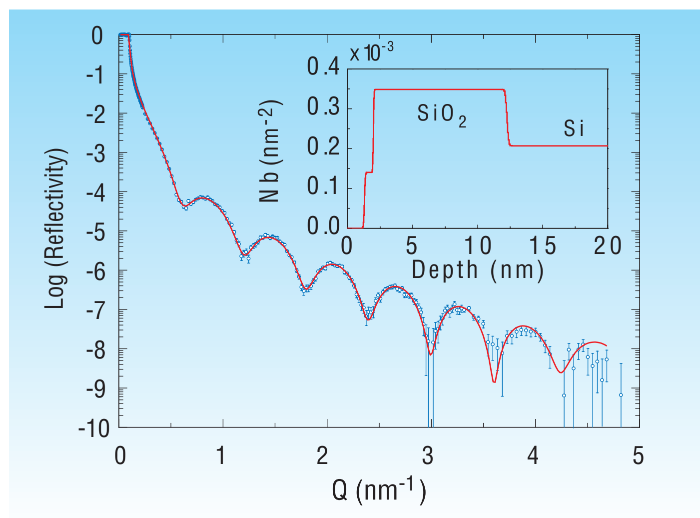

FIGURE 3. Neutron Reflectivity data and best fit for a sample with surface contamination. The inset is the scattering length density profile determined by the fit.

parameters extracted via these models. Thus the XR and particularly the NR corroborate the correct analysis required in SE (which is the technique most practical in monitoring production).

To further investigate the applicability of these techniques to thinner SiO_2 layers, we simulated the SE and the XR and NR curves for 6 nm, 4 nm, and 2 nm thick layers of SiO_2. For a realistic and consistent set of roughness parameters in the models, we used the average values obtained from the actual measurements previously discussed. The SE simulation, Fig. 4a, shows distinct differences in both the magnitude and shape of Δ among the three thicknesses shown. In models of XR, shown in Fig. 4b, only very weak oscillations are seen for even the thickest of the SiO_2 layers because of the low contrast between SiO_2 and Si. However, in NR, for SiO_2 layers as thin as 2 nm strong oscillations are clearly seen above the 10^{-8} lower limit, demonstrated in Fig. 3. Therefore both NR and SE are well suited for the study of SiO_2 films as thin as 2 nm. Encouraged by these models, we obtained NR data for a thinner, 2.4 nm, sample. While these data are not yet fit to a model curve, we note that both the reflected intensities and oscillation amplitude are similar to those of the 2.0 nm model, indicating similar interface widths.

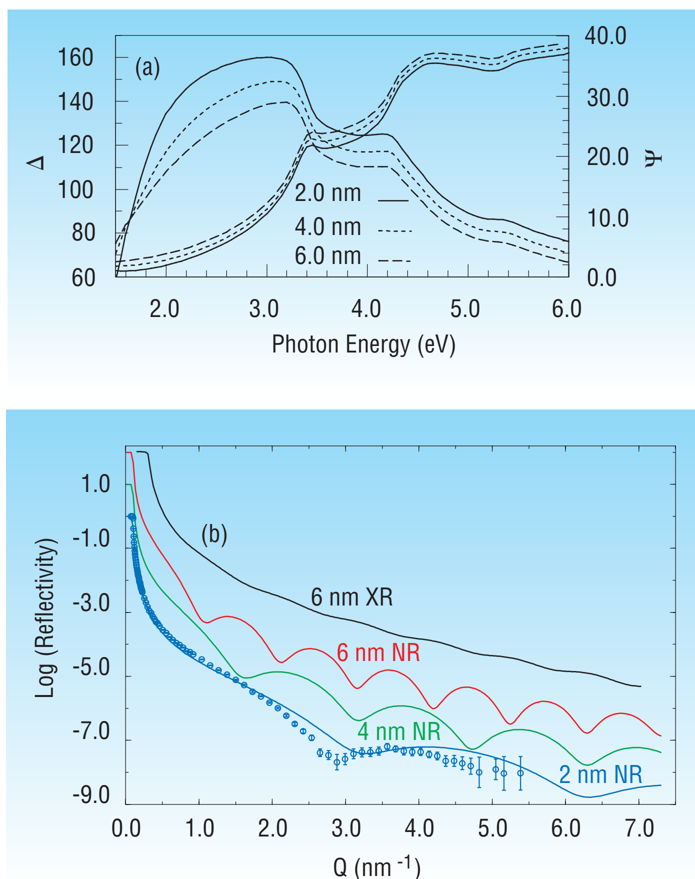

FIGURE 4. Model calculations for thin SiO_2 films of thickness indicated in the figure, on Si. a) Spectroscopic ellipsometry, and b) unless otherwise noted solid lines are neutron reflectometry models. For clarity the 6 nm (4 nm) films are shifted up by 2(1) orders of magnitude. The data points are for a $\approx$ 2.4 nm film.

We have shown that three different techniques can offer complimentary information on the structure of thin SiO_2 films on Si. All offer a significant degree of sub-monolayer thickness sensitivity, although in NR there is a much higher contrast between SiO_2 and Si than in XR.

REFERENCES

[1] For details see: “Neutron Reflectivity, X-ray Reflectivity, and Spectroscopic Ellipsometry Characterization of Thin SiO_2 on Si,” J. A. Dura, C. A. Richter, C. F. Majkrzak, and N. V. Nguyen, Appl. Phys. Lett. **73**, 2131 (1998).