# Nanoscale Film Thickness, Interfacial Roughness, and Density Measured Using X-Ray Reflectivity

Thin films are an important part of countless industrial applications, such as transistors, LEDs, and anti-reflective coatings. The physical dimensions of thin films are often critical to their functionalities. For example, the efficiency of an LED depends on the thickness of the layer that allows electrons and holes to recombine so as to emit light. In addition to the film thickness, the roughness of the film's interfaces can also be critical to its properties. Interfacial roughness affects the amount of light scattered from transparent coatings. It also affects the electro-magnetic coupling between two layers in the read head of a computer hard drive. While there are many techniques to measure layer thickness at the macroscale and microscale, x-ray reflectivity (XRR) has become the first choice for nanoscale thin film characterization.

X-ray reflectivity works on the same principles as other reflectivity techniques, but uses smaller wavelengths of light. A beam of parallel, monochromatic light shines on a thin film and scatters from the film's top and bottom interfaces. The two scattered beams interfere with each other, creating oscillations in the reflected intensity as the scattering angle changes. The intensity oscillation period is directly related to the film thickness. The slope of the intensity as a function of scattering angle provides information needed to determine the interfacial roughness. Finally, the angle at which total external reflection ceases offers information on the density of the film.

To demonstrate the usefulness of x-ray reflectivity, a low-κ dielectric film on a silicon wafer sample was analyzed using a Rigaku SmartLab X-Ray Diffractometer. On the SmartLab, a copper x-ray source with $\lambda$ = 1.5406 Å and a Ge 2x(110) monochromator was used to create a low-divergence (0.01°) x-ray beam. The XRR pattern of the sample was measured with the D/TeX 1D detector at 2°/min over a $2\theta$ range of 0 to 3°. Rigaku GlobalFit reflectivity analysis software was used to fit the experimental data and determine the film thickness, roughness, and density.

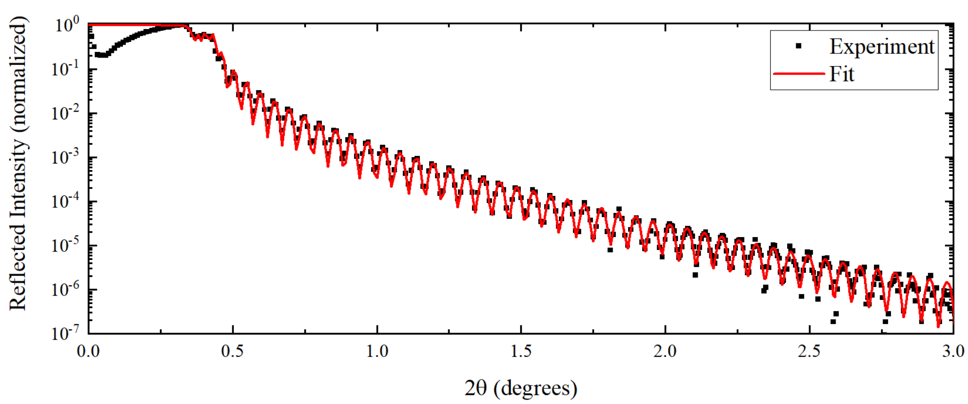


Figure 1. Experimental and simulated XRR patterns of the low-κ dielectric film on silicon wafer sample.

Address: 10025 Valley View Road, Ste 150, Eden Prairie, MN 55344, U.S.A.
Tel: +1 (952) 941-2199, Fax: +1 (952) 746-8086, Email: natlab@ebatco.com
www.ebatco.com

The x-ray reflectivity intensity measured from the sample is shown in Figure 1. Parameters for thickness, density, and interfacial roughness were entered into the reflectivity simulation software in order to find the best fit for the experimental results. During the fitting process, an additional layer between the organo-siloxane film and the silicon substrate was needed to generate a satisfactory fit. The best-fit results were produced by adding a thin layer of silica between the film and the substrate. This added layer is consistent with a well-known phenomenon that native oxide spontaneously forms on silicon wafers. As can be seen in Figure 1, with this treatment, the simulated data is an excellent match with the experimental results. Once the simulation process is finished, the film properties can be determined. The results for this sample are summarized in Table 1.

Table 1 XRR Measurement Results for Low-κ Dielectric Film on Si Substrate

| Layer | Thickness (nm) | Density ($g/cm^3$) | Interfacial Roughness (nm) |
|---|---|---|---|
| Organo-siloxane | 146.24 | 1.46 | 0.85 |
| Native oxide ($SiO_2$) | 1.35 | 1.85 | 0.30 |
| Silicon substrate | - | 2.32 | 0.85 |

As exemplified by this analysis on a low-κ dielectric film on silicon substrate system, XRR is a powerful technique for measuring the thickness, density, and interfacial roughness of thin films. The technique is well suited for non-destructive analysis of multilayer thin films that other characterization methods may not be able to perform. The ideal thin film system for XRR in our lab would have a thickness of 5-200 nm, a density of 1-7 $g/cm^3$, and an RMS roughness below 5 nm.

As knowledge of this technique becomes increasingly wide-spread, x-ray reflectivity is expected to find more and more applications in the semiconductor, microelectronics, medical devices, glass and many other industries.

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