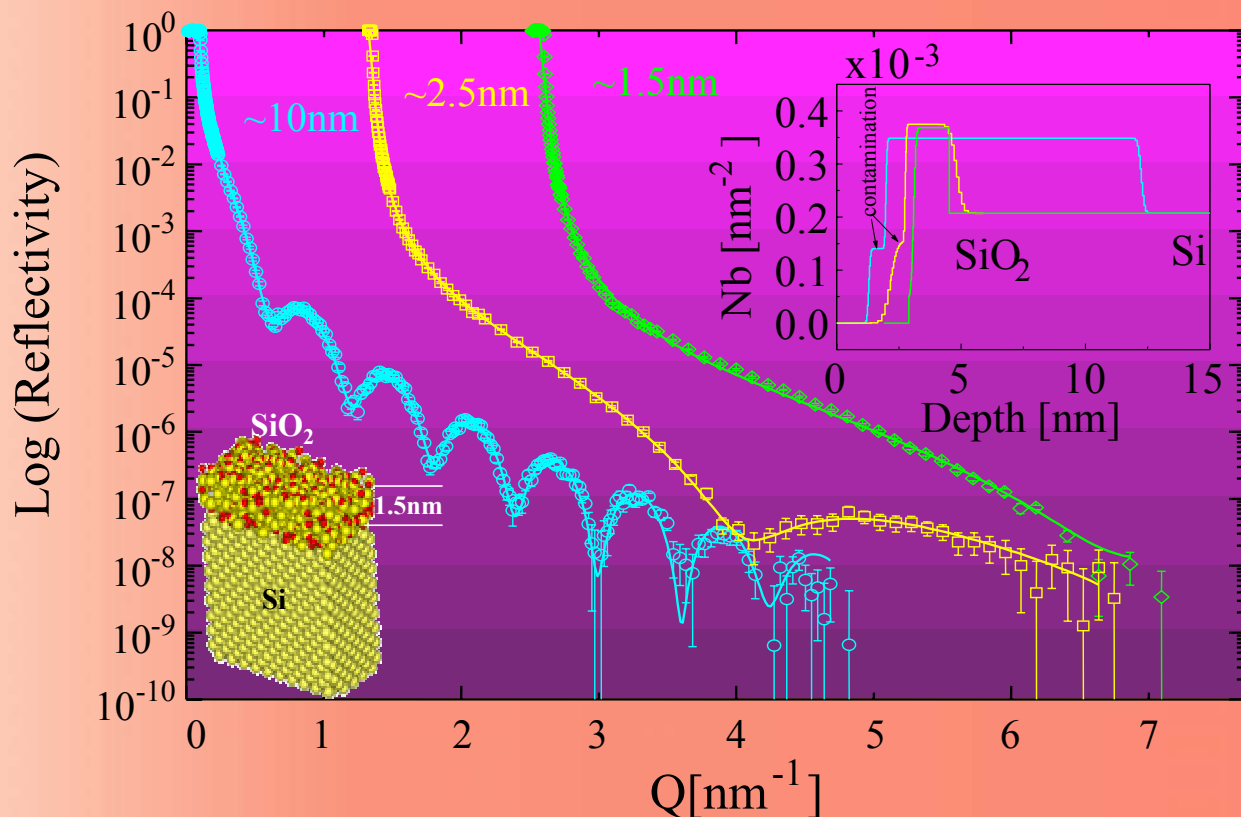# Polarized Neutron Reflectometer

Ultra-Thin Films of SiO_2 on Si

Provides nanometer scale metrology of layer thickness and composition

Neutron Reflectivity data and best fits for SiO_2 films labeled according to thickness. The inset on the right contains the SLD profiles determined by the fits. For clarity the ~2.5nm and ~1.5nm films are shifted to the right by $1.25nm^{-1}$ and $2.5nm^{-1}$ respectively. The inset on the left is a model of 1.5nm of SiO_2 on Si (100).

Semiconductor devices require three components to function: semiconductors for active components, conductors for interconnects, and dielectrics for gates and barriers. It is the ability of Si to form a high quality dielectric oxide that originally distinguished it from the many other semiconductor choices and has made it one of the most technologically important materials in the information age.

Neutron Reflectometry provides a particularly good probe of structure in thin silicon oxide films. Currently, gate dielectrics in silicon devices are as thin as ~2 nm. In production, thickness and optical properties are monitored by ellipsometry. However, the accuracy of optical techniques degrades for very thin films because thickness and index of refraction (which reflects composition) become strongly correlated. **In reflectometry, however, thickness and composition determination are nearly independent.** The layer thickness is approximately inversely proportional to the oscillation period of the reflected intensity, whereas the difference in the scattering length density (SLD) (which is determined by composition) is related to the oscillation amplitude. **Neutron reflectometry (NR) is better suited than x-ray reflectometry (XR) for studying SiO_2/Si due to a relatively large contrast between the SLDs of these materials: 65%, vs. 7.6% for XR.**

We report on three thermal oxides. The **10 nm** film was thick enough to establish confidence in the results, yet thin enough to be technologically relevant. The other samples, a **~2.5nm** oxynitride and a **~1.5nm** oxide are similar in thickness to today's gates. NR measurements of all samples (Fig. 1) were done in vacuum to reduce the background from air scattering. Vacuum also helped to inhibit formation of surface contamination layers, most effectively for the ~1.5nm sample, which was loaded seconds after the cleaning procedure.

Film thickness was easily determined for the thicker two films where at least one oscillation minima was clearly observed. By fitting the data, we were able to accurately determine the SLD depth profiles of all three films (inset) and ascertain that contamination had formed on the thicker two films. The thinner two films were provided by a different supplier than the thickest film and have a larger than typical SiO_2 SLD. In all cases, the quality of the results was enhanced by the very large, **8½ orders of magnitude, range in reflectivity** obtained for these films which are among the best examples in NR measurements to date.

As demonstrated herein, NR is very effective for determining SLD profiles in ultra thin SiO_2 films, and can be used to verify and calibrate more practicable measurements like ellipsometry. We can furthermore extrapolate these successes to ultra thin films of a variety of material systems. Improvements to the cold source and NG1 reflectometer may extend the range of reflectivity to lower values, allowing useful data to higher Q ranges on thinner films. Critical elements include the large dynamic range exhibited here and a careful control over contamination, achievable, for example, by growing and studying films in ultra-high vacuum.