Thin Film X-ray Reflectometry

Objective

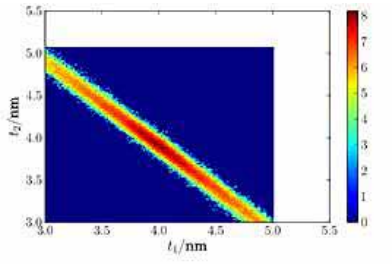
Our goal is to develop Standard Reference Materials (SRMs) and quantitative, reproducible X-ray reflectometry (XRR) data analysis methods to enable accurate measurement of film thickness, roughness, and density in thin, multilayer structures used predominantly in the microelectronics industry. Our approach is to measure robust, temporally stable, uniform thin films on a NIST-constructed X-ray reflectometer that is traceable to the International System of Units (SI), and analyze the measured data with statistical analysis methods capable of quantifying uncertainties in structural information.

Impact and Customers

- XRR SRMs will provide the $43B U.S. semiconductor industry with SI traceability for thin film thickness for ISO compliance and internal fabrication line standardization
- XRR SRMs will provide absolute thickness calibration for material structural modeling in nanotechnology and related fundamental research fields
- NIST provides technology transfers of metrological XRR characterization approaches to SEMATECH
- NIST has collaborations with key XRR instrument vendors (BEDE, Bruker AXS, Jordan Valley, PANalytical, and TECHNOS) to enhance calibration methods for XRR instruments in the field
- NIST has informal collaborations with semiconductor manufacturers for SRM materials development and acquisition

Approach

State-of-the-art microelectronic devices are patterned from nanometer-scale thickness films. Process development and manufacturing of these devices relies heavily on accurate thickness determination. XRR measurement tools provide the semiconductor industry with internally reproducible film thickness determination; however, different instrumentation and analysis software often produce divergent modeling results. NIST will address this problem by providing the community with SRM thickness standards that can be used to calibrate XRR laboratory and Fab-line instrumentation.



There are three steps in linking an XRR SRM with SI-traceable quantities. First, industry partners will produce a robust, inert, and homogeneously deposited layer structure as an SRM feedstock. Second, NIST will use its SI-traceable X-ray reflectometer to measure multiple wafer areas for certification of physical properties. Third, NIST will develop metrological, first-principles statistical analysis approaches for estimating structural parameters and uncertainties from NIST XRR measurements.

SEMICONDUCTORS

852-08-82b

Accomplishments

Several 2008 accomplishments have enabled significant progress in the NIST pre-standards effort. We will be circulating our first XRR round robin study in 2009.

First, we have completed construction and assembly of a new goniometer base for the Ceramics Division Parallel Beam Diffractometer (CDPBD), NIST's SI traceable X-ray reflectometer. Our NIST-designed and-constructed spherical air-bearing base can be seen in the figure below. This bearing allows us to completely separate sample translation and tilt, facilitating high accuracy XRR measurements.

Spherical air bearing base for CDPBD

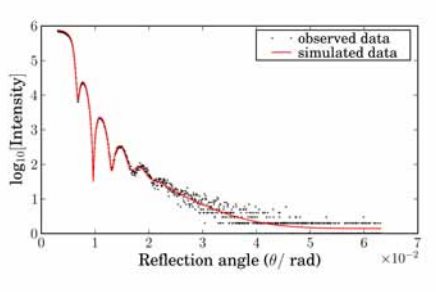
Second, the CDPBD has been reconstructed on a granite base, further enhancing structural stability for high resolution, high accuracy XRR measurements. The instrument is shown during an alignment study.

Third, we have partnered with the International SEMATECH Manufacturing Initiative (ISMI) to develop pre-standards for our round robin and, eventually, feedstock structure for an XRR SRM. In 2008, ISMI manufactured its first test structure based on a calibration-optimized bi-layer developed at NIST.

The NIST structural approach is to use a SiO_2 capping layer to protect a high electron density (high-atomic number, Z) layer that is deposited on a silicon wafer. The thickness of the high-Z layer will provide the SI-traceable quantity in the final SRM. The surface oxide will be treated as a variable in the XRR analysis to accommodate any surface contamination in the field. The requirements for both layers in the structure are: 1) smooth deposition processing (low roughness), 2) uniform lateral deposition across the entire wafer surface, 3) no interdiffusion or interface layers formed during deposition, 4) good shelf stability. Thermal SiO_2 was chosen for the capping layer due to its high uniformity, ubiquitous availability, and excellent stability.

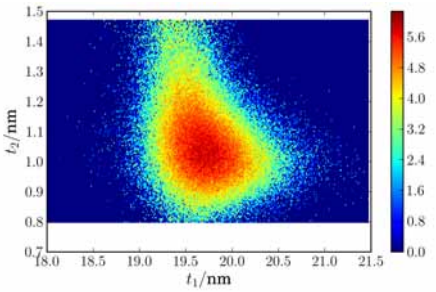
CDPBD during alignment on granite base

The high-Z layer poses a significant challenge in structure development. In the first round of depositions, we chose Atomic Layer Deposition (ALD) TiN as the high-Z structural layer. Unfortunately, the deposition process generated a ≈1-nm interface layer, presumably at the Si substrate interface. This additional layer creates difficulties for XRR modeling and would reduce the overall effectiveness of the structure for instrument calibration. The following figure shows a Genetic Algorithm (GA) refinement performed at NIST using ISMI-measured XRR data for the (TiN deposition) on the test structure. The measurements were taken while the wafer was still within the SVTC Fab, and before SiO_2 cap deposition.



XRR GA refinement of TiN on Si

We then used a statistically-based, Monte Carlo (MC) technique to determine the thicknesses of the TiN and TiSi interface layers. The figure below shows the posterior probability distribution for the TiN (t_1) and TiSi (t_2) from the MC analysis. The 1nm TiSi layer precludes accurate modeling of the TiN layer and Si substrate roughness, making this material and/or process inadequate for the XRR SRM.



Posterior probability of TiN (t_1) and TiSi(t_2)

In 2008, we presented our project at the spring ISMI Advanced CD Metrology Advisory Group (AMAG) meeting, established our development partnership, and presented preliminary results from our collaboration on XRR data analysis at the Denver X-ray Conference.

Learn More

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Publications

Windover D, Gil DL, Cline JP, Henins A, Armstrong N, Hung PY, Song SC, Jammy R and Diebold A *NIST Method for Determining Model-independent Structural Information by X-ray Reflectometry* AIP Conf. Proc., 931: 287 (2007)

Windover D, Gil DL, Henins A, Cline JP, Armstrong N, Hung PY, Song SC, Jammy R and Diebold A *X-ray Reflectometry Determination of Structural Information from Atomic Layer Deposition Nanometer-scale Hafnium Oxide Thin Films* MRS Online Proceedings, 0996-H07-05 (2007)

Windover D, Armstrong N, Cline JP, Hung PY and Diebold A *Characterization of Atomic Layer Deposition using X-ray Reflectometry* AIP Conf. Proc., 788: 161 (2005)