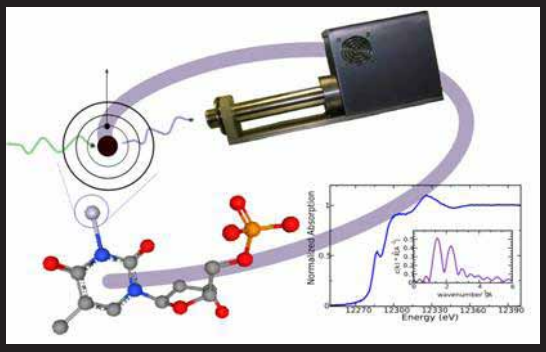
Synchrotron X-ray Measurements

Objective

Our objective is to provide comprehensive descriptions of the structure of advanced materials and devices by performing synchrotron-based measurements to enable the development and optimization of such materials and devices. Our research will establish structure-property relationships for advanced materials, thereby accelerating the introduction of these materials into devices and systems with advanced functionality for a broad spectrum of high-technology applications.



Impact and Customers

- Establishing structure-property relationships for advanced materials is critical to the development and optimization of products in many technology sectors. Synchrotron measurements provide structure data that cannot be attained by other methods.
- Synchrotron measurements of the depth dependence of the interfacial structure (*e.g.*, chemistry and local bonding) in high dielectric constant gate stack devices have enabled SEMATECH to optimize processing of such devices.
- Synchrotron measurements have elucidated the surface chemistry of an environmentally friendly automotive oil additive produced by Afton Chemical.
- NIST researchers collaborate with more than 15 companies to provide synchrotron-based structure measurements of novel materials for next generation device applications.

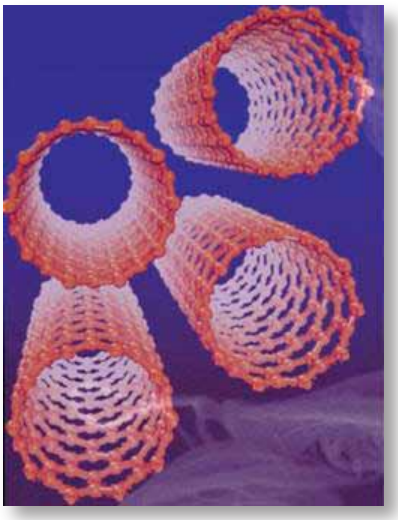
SEMATECH

Afton CHEMICAL

DOW

Approach

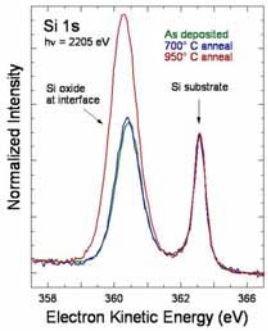
Our approach is to apply state-of-the-art synchrotron measurement capabilities at the National Synchrotron Light Source (NSLS) and the Advanced Photon Source (APS) to generate structure data that cannot be measured by other methods. We collaborate with leading researchers from industry, other government agencies, and universities to address the nation's most pressing measurement needs. The methods that we employ include: (1) near-edge X-ray absorption fine structure (NEXAFS) spectroscopy; (2) extended X-ray absorption fine structure (EXAFS) spectroscopy; (3) variable kinetic energy X-ray photoelectron spectroscopy (VKE-XPS); (4) grazing incidence X-ray diffraction; and (5) small angle X-ray scattering and reflectivity. Methods (1) – (3) are available at the NIST beamlines at the NSLS, while methods (4) and (5) are located at the APS.



NANOMATERIALS

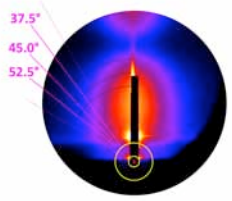
Accomplishments

One of the semiconductor industry's "Grand Challenges" is to develop an alternative to the SiO_2 gate dielectric. Integrated circuits exhibiting greater speed and lower power consumption are no longer attainable with ultrathin (≤ 2 nm) SiO_2 gate dielectrics due to their high direct tunneling leakage currents. Our collaboration with SEMATECH has led to extensive evaluation of conductive metal gate electrodes and thin film metal oxides as promising high dielectric constant (high-k) replacement insulator materials. We have measured core level binding energy spectra as a function of annealing temperature for $Al_2O_3/SiO_2/Si$ gate stacks. An Al_2O_3/SiO_2 interface effect is detected by the shift of the oxidized Si^{4+} 1s core peak to higher binding energy with increased annealing temperature, indicating interfacial modification of the Si sub-stochiometric chemical oxide.



Si 1s core-level spectra from an $Al_2O_3/SiO_2/Si$ gate stack as a function of annealing temperature.

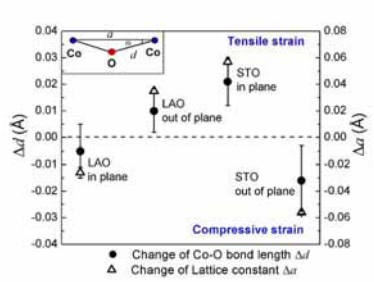
X-ray reflectivity and grazing-incidence small-angle X-ray scattering (GISAXS) were used to study the annealing behavior of ultrathin complementary-metal-oxide-semiconductor (CMOS) gate high-κ dielectric HfO_2 films grown by atomic layer deposition (ALD). Although dense, internal film structures exist, attributed primarily to 2 nm "missing island" porosity close to the substrate, and most likely associated with coalescence defects during initial ALD growth. Some larger features are also present, that may indicate a widespread modulation in the film density. Changes in film microstructure with annealing appear sufficient to affect CMOS gate dielectric performance after extended high temperature exposure and cycling.



GISAXS data for ALD HfO_2 film.

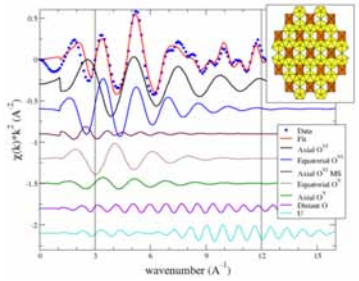
We have used a combination of high resolution EXAFS and XRD to measure the local structure of strained $LaSrCoO_3$ thin films grown under both compressive and tensile strain on substrates with dissimilar lattice constants. The lattice mismatch strain is found to affect both the bond lengths and the bond angles in the films. The popular double exchange model for ferromagnetism in these compounds provides a qualitative but not quantitative description of the measured changes in Curie temperature, with the predicted changes in Curie temperature being much smaller than observed. This finding demonstrates that a microscopic model for ferromagnetism with a much stronger dependence on structural distortion is needed.

An understanding of pentavalent uranium and U redox chemistry is of critical importance to the nuclear fuel cycle and to



Changes in Co-O bond length (EXAFS) and lattice constant (XRD).

environmental concerns centered around man made and natural sources of U ground water contamination. Although nature provides only a small number of examples of U^V minerals, exotic U^V species can form in the unusual geochemical environments existing in nuclear fuel storage facilities and in the vadose and ground water regions contaminated by leaking storage facilities. The synthesis, crystal structure and spectroscopic characterization of $[U^V(H_2O)_2(U^{VI}O_2)_2O_4(OH)](H_2O)_4$, a novel, stable U^V/U^{VI} oxide material not requiring the incorporation of carbonate, silicate, or organic ligands, were recently reported. EXAFS on a powdered form of this mineral was measured at beamline X23A2, verifying the mixed-valence of the U and the structure determined by single crystal x-ray diffraction.



EXAFS of the mixed valence uranium mineral

Learn More

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Publications

Guevremont J, Guinther G, Szemenyei D, Devlin M, Jao T, Jaye C, Woicik JC and Fischer DA *Enhancement of Engine Oil Wear and Friction Control Performance Through Titanium Additive Chemistry* Tribology Transactions., 51(3) 324-331(2008)

Belai N, Frisch M, Ilton ES, Ravel B and Cahill CL *Pentavalent Uranium Oxide via Reduction of [UO] Under Hydrothermal Reaction Conditions* Inorg. Chem., 47 (21) 10135-10140 (2008)

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