

## Announcing the Final Examination of Biao Yuan for the degree of Doctor of Philosophy

Time & Location: March 20, 2012 at 3:00 PM in OTC MCF 304

Title: DIRECT MEASUREMENT OF THICKNESSES, VOLUMES OR COMPOSITIONS OF NANOMATERIALS BY QUANTITATIVE ATOMIC NUMBER CONTRAST IN HIGH-ANGLE ANNULAR DARK-FIELD SCANNING TRANSMISSION ELECTRON MICROSCOPY

The sizes, shapes, volumes and compositions of nanoparticles are very important for their macroscopic properties. Efforts to measure these parameters for individual nanoparticles and to obtain reliable statistics for a large number of nanoparticles require a fast and reliable method for 3-D characterization. In this dissertation, a direct measurement method for thicknesses, volumes or compositions of nanomaterials by quantitative atomic number contrast in High-Angle Annular Dark-Field Scanning Transmission Electron Microscopy (HAADF-STEM) is presented. A High-Angle Annular Dark-Field (HAADF) detector collects electrons scattered incoherently to high angles. The HAADF signal intensity is in first-order approximation proportional to the sample thickness and increasing with atomic number. A new method for the calibration of the sensitivity of the HAADF detector for a FEI F30 transmission electron microscope (TEM) is developed in this dissertation. A nearly linear relationship of the HAADF signal with the incident electron current is confirmed. Cross sections of multilayered samples provided by TriQuint Semiconductors in Apopka, FL for contrast calibration were obtained by focused ion-beam (FIB) preparation yielding data on the interaction cross section per atom. To obtain an absolute intensity calibration of the HAADF-STEM intensity, Convergent Beam Electron Diffraction (CBED) was performed on Si single crystals. A database with several pure elements and compounds has been compiled, containing experimental data on the fraction of electrons scattered onto the HAADF detector for each nanometer of sample thickness. Multislice simulations from Dr. Kirkland's C codes are used for comparison with experimental results. TEM offers high lateral resolution, but contains little or no information on the thickness of samples. Thickness maps in energy-filtered transmission electron microscopy (EFTEM), CBED and tilt series are so far the only methods to determine thicknesses of particles in TEM. The calibrations were used to determine concentration gradients in nanoscale Fe-Pt multilayers as well as thicknesses and volumes of individual Au-Fe, Pt, and Ag nanoparticles. Volumes of nanoparticles with known composition can be determined with accuracy better than 15%. Porosity determination of materials becomes available with this method as shown in an example of porous Silicon. One significant aspect of this study is the orientation dependence of the HAADF intensity. The simulations as well as experiments show that sample orientation plays a small but not always negligible role. One aspect shown in HAADF-STEM imaging is increased contrast at the edge of a high-density layer at the interface to a low-density material. This can be explained by high scattering at the heavy atoms as well as channeling of electrons at the interface with reduced absorption by the lighter atoms.

Major: Materials Science & Engr

Educational Career:

Bachelor's of Inorganic Materials Science & Engineering, 1984, Zhejiang University

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Approved for distribution by Helge H. Heinrich, Committee Chair, on March 6, 2012.

The public is welcome to attend.