Three-Dimensional Analysis for Nanoscale Materials Science

LABORATORY DIRECTED RESEARCH & DEVELOPMENT

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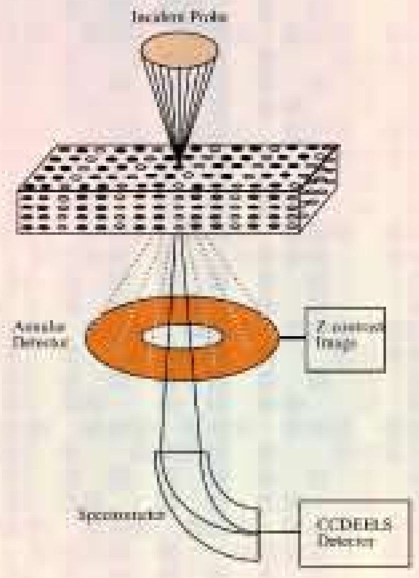
Problem

Nanomaterials pose ever-increasing challenges for characterization with their small dimensions, three-dimensional shapes, demanding internal structures, and novel electronic properties. As such, the technology for characterization must meet these growing demands. The morphology of nanomaterials in 3-D is critical to understand before advances can be made in nanodevices, and for this purpose, we have developed electron tomography in the scanning transmission electron microscope at Sandia/CA.

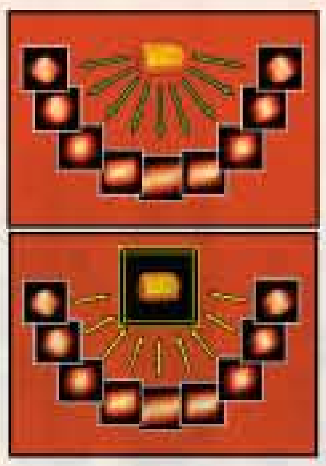
Methods

Below is a schematic for the STEM. Electrons are focused at the specimen plane, and scanned across the material. The scattered intensity is collected at high angles on the high-angle annular dark field (HAADF) detector. This scattering is Rutherford-like, with the intensity in the images approximately proportional to the square of the atomic number of the materials being studied. Electrons that have lost energy while traveling through the specimen can be detected using electron energy loss spectroscopy (EELS).

For tomography, the specimen is tilted across a range of typically +70° to -70°, with the number of images about 140-150. These images are then reconstructed using various reconstruction algorithms such as weighted back projection (WBP) and simultaneous iterative reconstruction technique (SIRT).



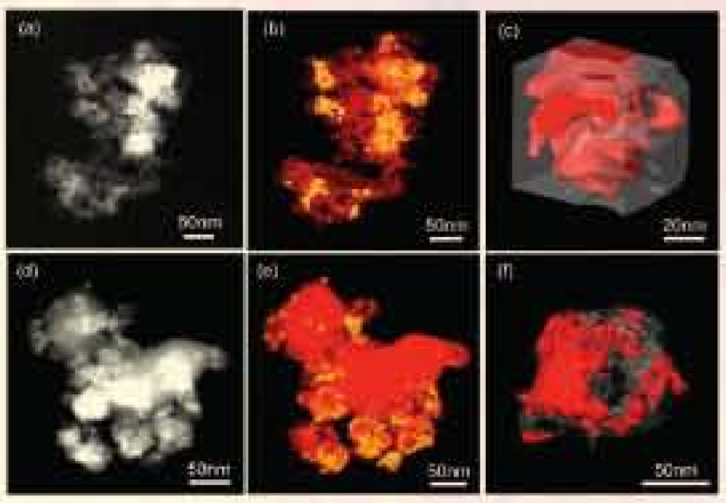
STEM Schematic



Tomography Schematic

Fischer Tropsch Catalysis

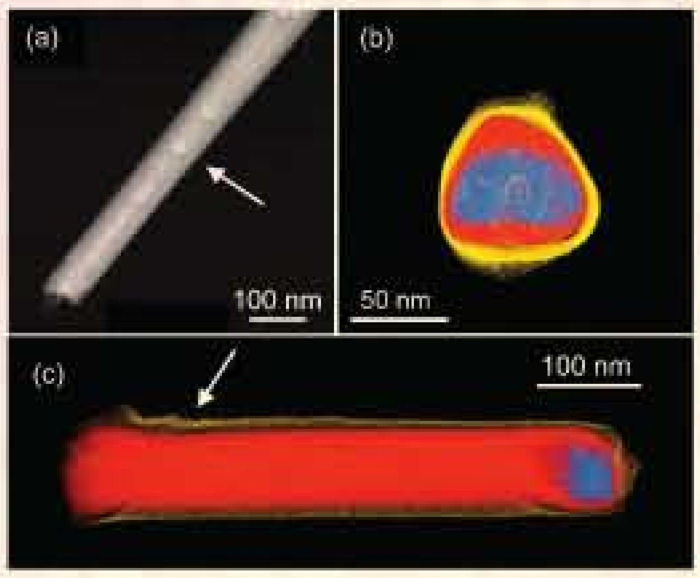
Cobalt-based Fischer–Tropsch systems are widely used to convert synthesis gas to clean hydrocarbon fuel. Two Co_3O_4 catalyst systems with different supports are studied here. The first system (a-c) forms an interlocking structure with its support with no free surface area. The second catalyst (d-f) is more selective and this is attributed to its unique nanocage morphology (f) that allows larger surface area for reactions to take place. This is 3-D nanoscale information that could not be quantified without the use of STEM tomography. Standard bulk chemistry measurements are insensitive to this information.



I. Arslan et. al., JACS 130,5716 (2008).

Semiconductor Nanowires

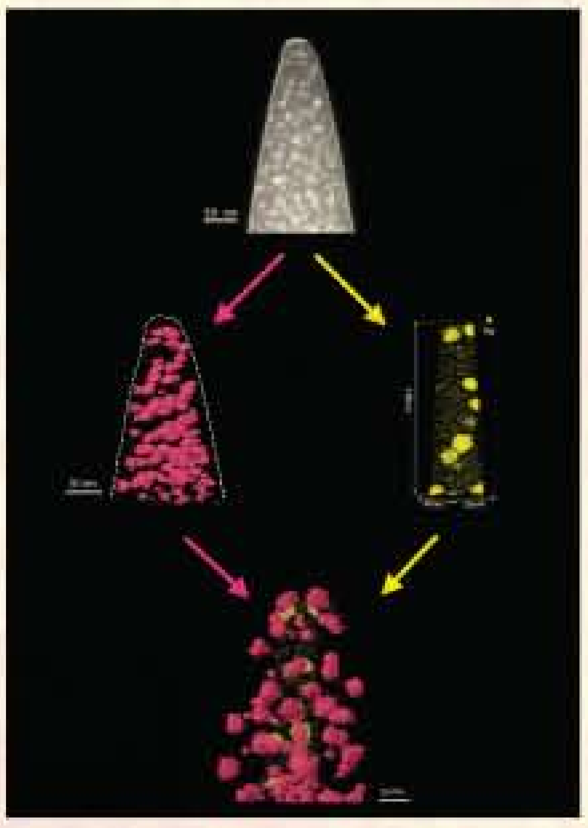
GaN/AlN core/shell nanowires are important for light emitting diodes, diode lasers, and high electron mobility transistors. The surfaces play an important role in these 1-D systems, and the only way to analyze the surfaces and bulk simultaneously is by using STEM tomography. While the defects shown in the 2-D image (a) appear to be bulk defects, the 3-D reconstructions reveal that the defects are actually just on the surface of the nanowires, indicated by an arrow in (c). Yellow, red and blue represent AlN, GaN, and the Ni catalyst nanoparticle respectively.



I. Arslan et. al., J. Phys. Chem. C 112,11093 (2008).

3-D Technique Correlation

Electron tomography (ET) and atom-probe tomography (APT) are both 3-D techniques on the nanoscale. ET provides larger-scale morphological information while APT provides smaller-scale chemical information in 3-D. Correlating these two techniques can yield a level of materials understanding that has never been reached before. Here we show the first such correlation on the very same needle-shaped specimen of a Ag precipitate system in an Al matrix. The pink reconstruction is the ET data, and the yellow is the APT data. This first correlation has allowed us to quantify the artifacts in both techniques, understand the evaporation process in APT, determine the optimal



Significance

The impact of the R&D in this Truman Fellowship is key for the future energy needs of our Nation. In order to make our current energy materials more efficient and to develop new materials for future energy applications, fundamental structure-property relationships must be established on the nanoscale. Electron microscopy is one of the most important tools to meet this demand, and the quantitative 3-D imaging and analysis developed in this Fellowship will play a vital role in moving our Nation's energy needs forward, quickly and competently.

Acknowledgements

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