ELEMENTAL MAPPING OF NANO-MATERIALS IN TEM AND STEM MODES

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For many nano-materials, an important aspect of comprehensive characterization for structure-property-processing correlations is the measurement of composition at a scale of ~1 nm. Two well-established analytical electron microscopy (AEM) techniques will be briefly reviewed: energy-filtered transmission electron microscopy (EFTEM) and spectrum imaging (SI) in the scanning transmission electron microscopy (STEM) mode employing simultaneous energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS). The capabilities and limitations of these techniques will be illustrated through applications to nano-structured materials performed at the ORNL SHaRE User Facility (http://www.ornl.gov/sci/share).

Nano-composites of ~10 nm Ni particles in a continuous amorphous Y-O phase produced by reactive ball milling of NiO and Ni_2Y powders have been mapped by EFTEM and EDS-EELS SI. Focused ion beam (FIB) methods were used to prepare TEM specimens from powders ball-milled at 30 and 100°C. The experimental challenges, such as mapping Y by EFTEM, will be described and the implications of the results for phase transformation mechanisms will be discussed.

EFTEM methods have been applied with great success to Co-Cr-based thin-film longitudinal magnetic recording media for computer hard disks. A critical structural feature for high-density data storage is intergranular Cr segregation that magnetically decouples the sub-10 nm grains to allow independent switching. In CoPt thin-film media for perpendicular recording, magnetic isolation is achieved by co-sputtering reactive-element oxides. Since Pt is not amenable to analysis by EELS and severe electron beam damage to the oxides precludes EFTEM elemental mapping, EDS-EELS SI has been the most useful technique for characterizing these materials. For both media types, quantitative compositional data are essential for making useful structure-property correlations.

Nanoscale $MgAl_2O_4$ spinel has been synthesized by a direct conversion method from nanoscale γ-Al_2O_3 and a $Mg(NO_3)_2$ solution with a final anneal at temperatures in the range 300 to 800°C. To support other characterization data, compositional mapping by EDS-EELS SI was performed but the extreme sensitivity to electron beam damage necessitated the use of probes defocused to ~5nm diameter. Useful information was obtained at this resolution and showed a much higher spinel content after annealing at 800°C but without a significant increase in particle size.

Through next-generation aberration-corrected instrumentation, the transmission electron aberration-corrected microscope (TEAM) project (http://ncem.lbl.gov/team3.htm) seeks to characterize individual nanostructures with atomic resolution in three dimensions. The first of the TEAM test columns, an FEI Titan with probe corrector and monochromator, is scheduled for installation at ORNL in July 2006. The prospects for AEM with single-atom sensitivity and rapid acquisition for dynamic in-situ experiments will be discussed.

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