EFTEM Spectral Imaging: A Screening Tool for the Discovery of New Nanostructures

Nanotechnology promises to bring about the "next industrial revolution," and is expected to play a major role in the 21st century US economy. The fulfillment of this promise will rely upon the discovery of new classes of materials and structures that could not exist but for the methods of self-assembly that are native to the nanoscale. Combinatorial methods, commonly used for the discovery of new drugs, provide a mechanism for testing thousands of distinct variations in the "recipe" used for synthesizing a particular class of nanostructures in a single experiment. However, there is currently no efficient screening tool for sampling the chemical makeup of these variations to identify novel structures.

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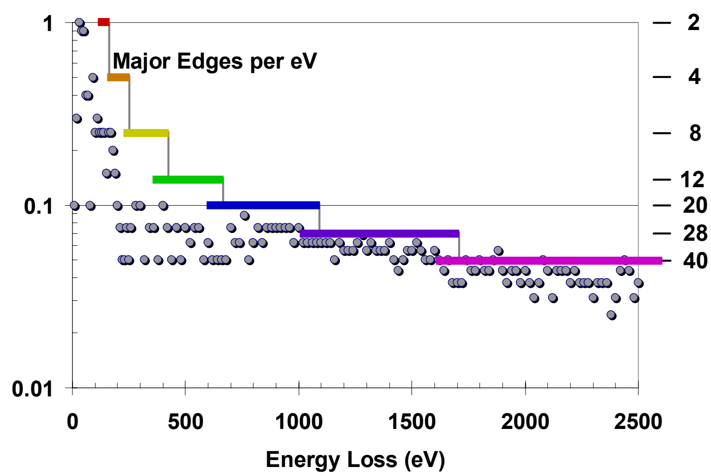
Energy-filtered transmission electron microscopy (EFTEM) has proven to be a powerful method for the quantitative compositional imaging of select materials systems, for example advanced structural alloys, semiconductor devices, and magnetic recording media. However, the sharp and well-separated spectral features (edges) of the electron energy-loss spectrum (EELS) that are used for these studies account for less than 1% of the spectral intensity, and more than 99% of the intensity is discarded as uninteresting background.

This overlooked energy-filtered transmission electron microscopy (EFTEM) spectral intensity can be effectively used for the screening of nanostructures, for which robust nanometer-scale *qualitative* analysis – "what's in there?" – is more crucial than quantitative analysis – "and how much?"

This qualitative analysis draws on the strengths of the EFTEM technique, such as its rapid data acquisition and excellent spatial sampling of the nanostructure, while mitigating its well-known limitations, such as the complexity of the EELS spectrum and its poor sensitivity for quantifying most elements in the periodic table. Recently developed spectral imaging methods for data acquisition and analysis are well suited to optimizing EFTEM as a screening tool for nanoscale structures. EFTEM spectral imaging is also an ideal technique for three-dimensional (3D) chemical imaging at the nanoscale, the subject of a CSTL-led NIST competence project.

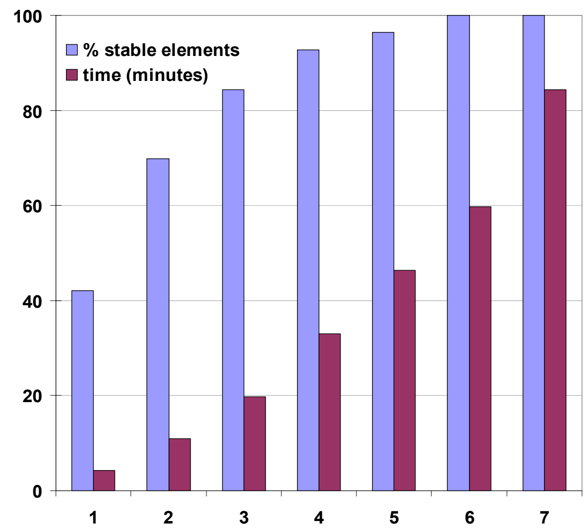
A preliminary method of EFTEM data acquisition and analysis for robust qualitative analysis has been devised. Whereas for most spectroscopic techniques, the full spectral range can be sampled with a fixed energy window (e.g., 10 eV for X-ray energy-dispersive spectroscopy, XEDS), this is not practical for EFTEM because the available signal in the EELS spectrum varies by several orders of magnitude. We determined that the practical EELS spectral range (E ≤ 2500 eV) can be effectively sampled with seven equally sized spectral images, with the sampling density chosen to be proportional to the number of available spectral features, as shown in Fig. 1, and the acquisition time chosen to achieve high signal with acceptable specimen drift.



Semilog plot of major characteristic edges per eV in the EELS spectrum is shown. The spectrum up to 2500 eV energy loss can be sampled in proportion to the number of spectral features present with seven different sampling densities, as shown on the right-hand axis (eV). The sampling density at the lowest energy losses (red) is 20 times finer than that at the greatest energy losses (violet).

Major EELS edges from all stable elements of the periodic table can be sampled with the first six spectral ranges; however, 70% of the elements have major edges within the first two spectral ranges, and a significant time savings per specimen can be realized if the number of ranges is reduced. The integration of effective image registration routines, necessary to correct for residual specimen drift, is being explored.

Trade-off between the percent of stable elements with major edges represented and time of acquisition. Major edges from 70% of all stable elements in the periodic table can be sampled in 10 minutes (first two spectral ranges); an hour would required to sample those of all elements (first six ranges).



Future Plans: This work is still in its early stages, having been in progress for just a few months. The next step is to effectively integrate the EFTEM spectral imaging data acquisition method described above with efficient data analysis. This protocol will then be applied to a variety of model nanostructures to evaluate the efficacy of the technique for specimens with severe spectral overlaps and the consequences of truncating the spectral image series with a limited number of spectral ranges.

A rapid and effective sampling tool for screening nanoscale structures would have a significant impact on the progress of nanoscale science, engineering, and technology.

Publications:

I.M. Anderson and J.H.J. Scott, **"EFTEM Spectral Imaging: A Screening Tool for the Discovery of New Nanostructures"** (2006) *Microscopy and Microanalysis* 12 (Suppl. 2).

Note: to be distributed at the annual *Microscopy and Microanalysis* meeting, August, 2006.