Recent Advances in EELS Instrumentation and Analysis: Quantitative Analysis at the Nanometer Scale

R.D. Twesten, M.M.G. Barfels, C.G. Trevor, P.J. Thomas, N.K. Menon, A. Aitouchen and A.J. Gubbens

Gatan Inc., 5794 W. Las Positas Blvd., Pleasanton, CA 94588, USA

rtwesten@gatan.com Keywords: EELS, Energy Filtered TEM, Tomography, ELNES

Transmission electron microscopy (TEM) reveals details of natural and man-made structures at the micrometer, nanometer, and even sub-nanometer scale. Energy-filtered TEM (EFTEM) and electron energy-loss spectroscopy (EELS) are the ideal analytical partners to the high spatial resolution provided by TEM in both the conventional and scanned (STEM) imaging modes. The additional dimension provided by these analytical techniques allows the researcher to determine not only where the atoms are in the sample, but also which element they are and in some cases, their chemical state (Figure 1).

The first generation Gatan imaging filters (GIFs) [1] were largely tuned by hand and were not integrated with the TEM column rendering them accessible to only the most dedicated operators. Improvements in filter and TEM hardware and, more importantly, improvements in filter automation and filter-TEM integration have made EFTEM acquisition a routine aspect of most TEM laboratories [2]. Continuing improvements in filter optics, design, and stability [3] have allowed these energy filters to routinely achieve sub-100meV energy resolution when coupled with monochromated electron sources [4, 5].

Recently, significant advances have been made in the ability to acquire multidimensional data-sets in the TEM. With the advent of next generation spectrometers and energy filters, high-speed cameras, advanced microscope control, and the widespread availability of high-brightness electron sources, it is now possible to acquire multidimensional data sets with high-information density in a routine and automated manner (Figure 2).

Continuing advances in computing power and processing tools enable rapid identification and extraction of the elemental, chemical, and physical information contained within these data-sets. Such improvements have made these rich data collection and analysis techniques available to nearly all characterization laboratories.

To elucidate these advances, we will discuss recent progress in acquisition and analysis techniques and instrumentation for characterization at the nanometer scale and beyond. We will particularly emphasize the role high-speed acquisition hardware and software automation techniques play in simplifying the acquisition of advanced EELS data.

- 1. O.L. Krivanek, et al., Mircosc. Microanal. Microstruct. 2 (1991) 315
- 2. A.J. Gubbens, et al., Micron 29 (1998) 81
- 3. H. A. Brink, et al., Ultramicroscopy 96, (2003), 367
- 4. G. Kothleitner, et al., Micron 34 (2003) 211
- 5. D. S. Su, et al. *Micron*, **34**, (2003) 235
- 6. K. Jarausch, et al., Ultramicroscopy 109, (2009), 326



Figure 1. Elemental and chemical state mapping of a WS₂ coated multi-wall carbon nanotube (MWCNT). a) HAADF STEM image, b) graphitic carbon-K map with weak π^* coupling, c) graphitic carbon-K map with strong π^* coupling, d) amorphous carbon-K map, e) sulfur-K map, and f) color montage of b-e showing spatial relation among signals. Sample courtesy Univ. of Surrey. Data was recorded on an Hitachi HD2300 STEM coupled to a Gatan Enfina dedicated EELS spectrometer using Gatan spectrum imaging software.



Figure 2. Color composite image of a W to Si contact from a failed semi-conductor device[5]. Volumetric elemental distribution maps for Ti, N, Co and Si were obtained by tomographic (SIRT) reconstruction from the corresponding elemental map tilt-series.