Electron Energy-Loss Spectrometry

Goal

This experiment will introduce you to the practical usage of electron energy-loss spectrometry (EELS) for analyzing the chemical composition of materials with superior spatial resolution provided by transmission electron microscopy. You will learn how to setup the Tecnai F30 for EELS in image-coupled mode and diffraction-coupled mode, how to acquire EELS spectra, and how to interpret them qualitatively and quantitatively in terms of the chemical composition of the specimen.

The specimen of this laboratory is a cross-sectional specimen of a Si single crystal substrate with a thin epitaxial layer of Cu_2O grown on top of it.

Experiment

1. Consult the online documentation for the recommended operating conditions for EELS. This concerns the condenser aperture, the gun lens setting, and the extractor voltage.
2. Start the microscope, load the specimen, load appropriate FEG registers, and check the basic alignment of the microscope.
3. Locate an area where the Cu_2O/Si interface traverses a thin area – near the edge of the hole. Recall that EELS requires a very thin specimen area. should be near the specimen center.
4. First, set up conditions for TEM diffraction mode.
   - Choose a small spot size, e. g. #8.
   - Obtain a diffraction pattern.
   - Center the primary beam at "6:30" on the inner circle of the viewing screen.
   - Chose a camera length of 250 mm.
   - Make sure that the TV camera is inserted.
   - Setup filter control for spectrometry. Choose an entrance aperture of 2 mm in diameter and an energy dispersion of 0.2 eV/pixel.
   - View the spectrum with the TV camera and perform the alignments suggested by “Filter Control.”
   - Obtain a live image of the zero-loss peak on the CCD camera (“Search”).
   - Choose a very short exposure time (0.05 s) and acquire spectra of the zero-loss peak and the low-loss region. Make sure you do not damage the CCD camera! Immediately reduce the beam intensity (spot size) if the displayed spectra turn yellow or even red!
   - Adjust the energy shift (strength of the magnetic prism) to obtain the zero loss peak at $\Delta E = 0$; “nullify” the energy shift.

  - Adjust the drift tube to offset the spectrum such that it includes the energies of the element-characteristic absorption edges that you expect observe (O, S, Cu). Consult the "EELS Atlas" for the corresponding energy values.
  - Record several EELS spectra including characteristic absorption edges of the elements present in the specimen.

5. Perform "qualitative" EELS.
   - Check the spectra for problems.
   - Identify the absorption edges of the individual elements.
   - Compare your results to the "EELS Atlas." (elements? peak families? artifacts?)
   - Save one or two spectra for your report.
   - Estimate the relative concentration of the dominating elements from the absorption edges.
   - Can you recognize any energy-loss near-edge structure (ELNES)?
6. Try "quantitative" EELS.
   - Perform a background subtraction and quantify the concentration ratio of the dominant elements using the provided software.
   - Save your results.
7. *As much as time allows,* repeat the procedure for TEM image mode.
   - Obtain an image of the specimen on the viewing screen.
   - Choose an appropriate magnification and locate a feature of interest on the region that corresponds to the entrance aperture of the spectrometer.
   - Repeat the above procedures for acquiring EELS spectra of the zero-loss peak and low-loss region as well as the element-characteristic absorption edges.
   - Repeat the above procedures of qualitative and quantitative evaluation.

Report

- No more than 5 pages, please!
- Include your spectra and take a critical look at them!
- Describe the set-up and operation of the EELS system.
- Describe how to obtain *high-quality* EELS spectra.
- Describe your results of "qualitative EELS."
- Describe your results of "quantitative" EELS.
- How do your results obtained in TEM diffraction mode compare to the results obtained in TEM image mode?

- What can you say about the spatial resolution and the energy resolution of your spectra?
- How do the results compare to the results of the XEDS experiment?