X-ray Energy Dispersive Spectrometry

Goal

The purpose of this experiment is to understand the technique of X-ray energy dispersive spectrometry (XEDS) 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 XEDS, how to acquire spectra, and how to interpret them quantitatively and quantitatively. You will be introduced to spot measurements as well as line scans.

The specimen of this laboratory is a crosssectional 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 XEDS. 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. Ideally, the region of interest should be near the specimen center and opposite of the XEDS detector (mind the rotation of the image).
4. Remove the objective aperture and make sure that you are not in low-magnification mode. Reduce the spot sizes to #6.
5. Insert the XEDS detector.
6. Tilt the specimen *towards* the detector by 16°.
7. Obtain a “live view” of the XEDS spectrum (“search”).
8. Make sure that the dead-time ratio is no larger than ≈25 %. If necessary, increase or reduce the spot size.
9. Record XEDS spectra with an acquisition time of 1 min.
10. Perform ”qualitative” XEDS.
    - Check the spectrum for problems.
    - Identify all peaks in the spectrum (elements? peak families? artifacts?)
    - Save one or two spectra for your report.
    - Estimate the relative concentration of the dominating elements from the peak heights.
11. Try “quantitative” XEDS.

- Select peaks for evaluation (K peaks preferred).
- Perform a background subtraction and quantify the concentration ratio of the dominant elements – k factor?
- Save your results.

12. Intentionally record spectra under "poor" XEDS conditions.
    - Record spectra in thick regions of the specimen.
    - Tilt the specimen away from the detector.
    - Exceed the recommended dead-time ratio (e. g. to 80 % – but not *too* much!)
13. Prepare the instrument for an XEDS line scan across the Cu_2O/Si interface.
    - Insert the HAADF detector.
    - Obtain a STEM image.
    - Draw a scan line and setup appropriate parameters for a line scan in the corresponding OCX.
14. Obtain and XEDS line scan across the interface.
15. Use the TIA software to obtain the line profiles of the dominant elements.

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 XEDS detector.
- Describe how to obtain *high-quality* XEDS spectra.
- Describe your results of "qualitative XEDS."
- Describe your results of "quantitative" XEDS.
- Compare the first spectra with the spectra you recorded under "poor" conditions – artifacts?
- Describe and discuss the results of the line scan. What can you say about the spatial resolution?