MSE 460 TEM Lab 7:
Basic EELS Operation

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Aims: The aim of this lab is to familiarize you with basic Electron Energy Loss Spectroscopy (EELS) operation. This lab covers

- Preparation of the STEM system
- Acquisition of EELS spectra from two regions with different composition
- Qualitative analysis of EELS spectra- peak identification.
- Simple quantitative analysis - composition calculation

TEM: HD2300 STEM (BioCryo)

Time: 3 hours

You may need your notes from previous labs. Please bring them with you.

You may spend the first hour to repeat what you learned in Labs 2 and 6. This includes:

I. Get familiar with STEM structure, functions and knobs
II. STEM startup
III. Obtain a good electron illumination
IV. Set the sample at eucentric height
V. Condenser lens alignment
VI. Beam tilt purity
VII. Objective lens alignment (Voltage center)
VIII. Image focus and astigmatism correction (Fresnel-fringe method)

Again, please refer to the Daily Operation Guide for specifics about the HD2300 STEM.

IX. Basic EELS operation

1. System alignment

- Start DigitalMicrograph software.
- Do normal TEM alignment (See HD2300 daily operation guide).
- Find a thin sample area of interest.
- Apply suitable TEM magnification.
- Switch to Preview to use CCD camera and observe image on PC screen. Check stigmatism with Process/Live/reduced FFT, etc.

- Click Acquire to get a picture. Save it into your own folder in the TEM-Server. Data in Disk C and other directories will be deleted without any notice.
- You may save you images in standard formats (either TIF or JPG), however, these formats do not keep notations and magnification information. The file format DM3 will keep this information.

2. Electron Energy Loss Spectroscopy

- While spectroscopy can also be done with either TEM imaging mode or TEM diffraction mode, we will use (STEM) imaging mode only for this lab.

- Find a very thin region of interest and focus properly with an objective lens strength very close to the optimum and re-correct objective astigmatism if using a magnetic specimen.

- On the Gatan computer, in Digitalmicrograph (DM), open the EELS software: In 'Help', select "Power user" for user mode; In "Window", click "floating window layouts" and select "EELS-2012". On the "Enfina Control", type in beam energy (EELS is only aligned for 200 and 80 kV; we will generally use 200 kV).

- In the Hitachi STEM-control, click the red EELS button to select EELS mode.

- Make sure you are still focused on the region of interest on the sample.

- On the Gatan computer, in the DigiScan window, click "Search" to scan in an image. Then click "Stop" to stop scanning, and click on the beam control icon (red triangle) to park the beam on the area of interest where the spectrum will be collected.

- In the EELS window, select 1 mm aperture, use 0.0025 s as the starting exposure time, and 0 as energy-loss, click on "View" to look at the zero-loss peak. (Convergent angle: aperture 1,2,3,4 – 24, 12, 8, 5 mrad, respectively; collection angle: 1,2,3,5 mm – 7, 14, 21, 30 mrad. **Please do not use 5 mm aperture: CCD camera will be damaged! Stop acquisition immediately if you see warnings about oversaturation or the spectrum turns red.**)

- Click "Align" to set zero for the zero-loss peak (ZLP).

- On the "Enfina control", double-click "Focus-X" and press on left or right arrow to focus (i.e. make the ZLP as thin and high as possible). Then repeat this with "Focus-y".

- Set up "Acquire" condition and acquire a zero-loss EELS spectrum (making sure the ZLP is now calibrated). Set up "energy-shift" and gradually increase exposure time to record a core-loss spectrum. You may select a larger EELS aperture and different dispersion (eV/channel) for this.

- Use this information to set the parameters for your acquisition. The larger apertures naturally give more signal intensity but poorer energy resolution and the smaller apertures vice versa. Use small apertures for low loss experiments and large apertures for core-loss experiments; it may even be helpful to quantify the energy resolution under the conditions you intend to use.

- Acquiring a spectrum:
  a) Once the zero-loss is focused, and the correct dispersion is set, acquire EELS spectra by increasing the integration time.
  b) For acquiring core-loss spectrum you may input expected Spectrum Offset. Generally 50 eV before the expected major edge of the sample element. Set desired dispersion (eV/channel) and entrance aperture size.
  c) While performing core loss, it is necessary to have a much more intense beam, since the cross section for EELS excitations decreases drastically with ΔE. You must increase the integration time to at least 2s before being able to see the core loss peaks.

- Acquire spectrum. Increase exposure time if necessary. Use cumulative acquisition for better S/N ratio.

- Move your sample and acquire EELS from the second area.

3. Qualitative analysis of EELS spectra- peak identification.

a) In DigitalMicrograph software, go to Menu – EELS – quantitative analysis.
b) Select elements from the Edge List and transfer to Quantitative Analysis List.
c) Click Label button to show labels on the spectrum.

4. Simple quantitative analysis

a) In the Quantification window, click on Quantify button to do quantitative analysis.

5. Exit EELS mode

a) Click on the green triangle icon to release beam control; it will turn red, indicating you have let Hitachi re-control the beam.
b) Save all your data in the .dm3 format and it can be opened and analyzed again later on an EPIC computer where DM is installed.
c) In the Hitachi STEM control, click the "EELS" icon again to quit EELS mode and return to normal S/TEM mode.
d) When you're ready, follow shut down procedures (Lab 2 and Daily Operation Guide).

NOTES:

- Pay attention to how aperture size and dispersion affect your quantification.
- Think about what other aspects of the sample can be quantified making use of the data you've collected!