MSE 460 TEM Lab 6:
Basic EDS Operation in TEM Mode (and Demo of EDS in STEM Mode)

Last updated on 12/14/2021
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Aims: The aim of this lab is to familiarize you with basic energy dispersive X-ray (EDS) spectroscopy operation. This lab covers
- Preparation of the STEM and Oxford EDS system
- Acquisition of EDS spectra
- Qualitative analysis of EDS spectra- peak identification.
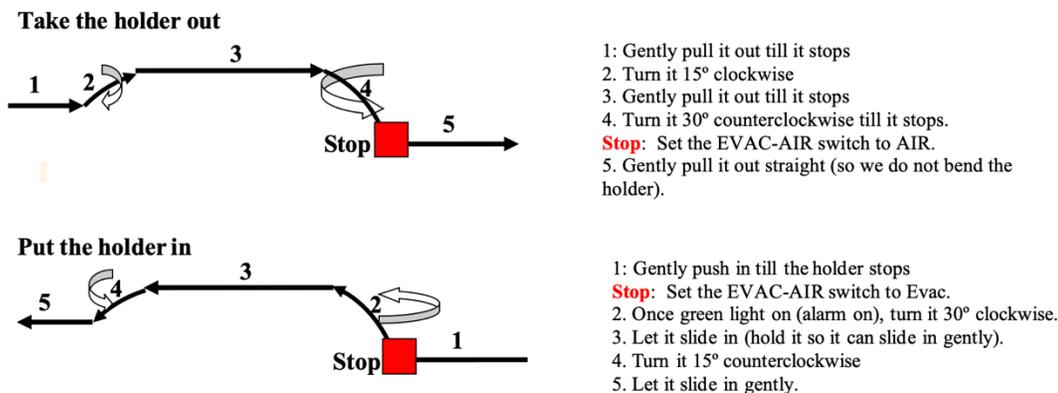- Simple quantitative analysis - composition calculation

TEM: HD2300 STEM (BioCryo)

Time: 3 hours

You may spend the first hour to repeat what you learned in Lab 2. It includes:
I. Get familiar with TEM structure, functions and knobs
II. TEM 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)

Refer to the Daily Operation Guide for specifics about aligning the HD2300 STEM. In particular, note the slight differences in sample holder insertion and removal for this microscope:

Take the holder out

1: Gently pull it out till it stops
2. Turn it 15º clockwise
3. Gently pull it out till it stops
4. Turn it 30º counterclockwise till it stops.
Stop: Set the EVAC-AIR switch to AIR.
5. Gently pull it out straight (so we do not bend the holder).

Put the holder in

1: Gently push in till the holder stops
Stop: Set the EVAC-AIR switch to Evac.
2. Once green light on (alarm on), turn it 30º clockwise.
3. Let it slide in (hold it so it can slide in gently).
4. Turn it 15º counterclockwise
5. Let it slide in gently.

Part 1: Basic EDS operation

1. Prepare TEM and Oxford EDS system
In order to collect a trustworthy EDS spectrum and to protect the EDS detector, it is quite important to bear following points in mind:
- ***Any objective or high-contrast apertures **MUST** be out of the column***
- Find a suitable area for EDS analysis before loading the EDS detector
    - Thinner area should face the EDS detector to avoid strong X-ray absorption
    - When the EDS detector is in the TEM column, do not use the LOW MAG mode or move the thick area into the beam.
    - Avoid focusing the electron beam on Cu grid bars if your sample is supported by a Cu grid.

- Use suitable condenser aperture and select suitable sample thickness to get reasonable X-ray counts and dead time.
- Avoid strong Bragg diffraction condition for normal EDS analysis

Experimental procedure:

a) Make sure that cover of EDS detector is closed

b) Open NSS software from the Thermo computer (right-most monitor) and start a new project.

c) Find an interesting area containing nanoparticles, and set it to the eucentric height.

d) Focus the electron beam to the area of interest.

e) Insert both EDX detectors.

f) Insert the EDX aperture (from **[Setup]** menu select **[Column setup]**).

g) **Take HIGH CONTRAST APERTURE out of the column if it is in. Note: Hard X-rays generated by the aperture may saturate and damage the detector.**

h) Set appropriate EDS acquisition parameters in NSS: select "both detectors", set your "low energy cutoff", and and click on the yellow "Acquisition Property" icon to set up collection time for both detectors, etc.

2. Record EDS spectra

a) Click the **Spectrum** icon to acquire a spectrum from the thin area. If the **dead time** is very high (>50%), recheck objective aperture and make sure it is not in the column. Check other apertures as well.
b) Adjust **BRIGHTNESS** or **SPOT SIZE** to get suitable x-ray counts and dead time (15-30%).
c) Move the beam into a hole and obtain a spectrum. EDS counts from vacuum are normally very low (dead time <2 %). If the counts are high, check apertures, spot size etc. or ask for help
d) Moving an interesting area into the beam
e) Start to **acquire** a new spectrum
f) Repeat d) – e) for the second area
g) Save the EDS project in NSS format. Generate a report by clicking "Export to Word". EDS spectra can also be saved as JPG files by highlighting the spectrum and then clicking "Copy To".

Note: If you have time you may also experiment with other experiment with other functions such as "Line Scanning", "Point and Shoot", and "Spectral Imaging".

3. Qualitative analysis of EDS spectra- peak identification.

a) Switch to an EDS spectrum of interest
b) Under the **Processing** tab, execute a new peak identification by clicking the **Identify Spectrum** icon.

4. Simple quantitative analysis

a) Click **Quant Setup** icon to input appropriate specimen parameters
b) Click **Quantify Spectrum** button to display the results

Tip: The accuracy of the quantitative result is dependent on a lot of factors: detector efficiency, sample absorption, background correction etc. Using sample standards in conjunction with your specimen spectra may greatly improve the accuracy.

Lab required:
1). Record a EDS spectrum from a hole (hole count as background).
2). Record a EDS spectrum from the core of the nanoparticle.
3). Record a EDS spectrum from the shell of the nanoparticle.
4). Spread electron beam and record a spectrum from the whole nanoparticles.
5) Do analysis on all the recorded spectra.

Part II (For your reference): Scanning transmission electron microscopy setup for JEOL 2100F (S)TEM

1) Align microscope under image mode and make sure sample is at eucentric height and Mag above 250K.
2) Make sure CCD camera is selected as "GIF Camera" from "Camera" window.
3) Take out all apertures except the condenser aperture.
4) Start ASID PC-TEM control software from JEOL computer.
5) Open Sirius Client (⬛) software if they are not open (Username: basic ; Password: b) .

6) Press STD FOCUS (right panel).
7) Click 'STEM' button in Detector part of Sirius Client, wait until Sirius Client turns into 'STEM' control interface. Press F1 (right panel) to put down the screen if the screen has been lift up automatically.
8) In SiriusClient: select camera length = 40cm, probe size = 1nm (smaller probe size for dedicated image) and click RONCHI button. Change SMMAG to 200Kx. Observe ronchigram on TEM screen with binocular. Adjust sample height if image is seen instead of ronchigram.
9) If beam rastering motion is observed, go to ASID PC-TEM control panel and turn off Normal Scan Mode (press ⊕).
10) Make sure the disk is at middle of screen (9 O'Clock, 5 mm away from center). If not, center it using PLA (left panel) and DEF/STIG knobs.
11) Select camera length = 2cm and Press F1 (right panel) to lift up screen.
12) DM – Camera View tab – Start View, you should be able to see portion of the Ronchigram in the image window.
13) Make sure Ronchigram disk is at middle of the screen. If not, center it using (PLA + DEF/STIG).

14) Change OBJ focus knobs to obtain Ronchigram with better contrast from amorphous area.
15) Using PLA + DEF/STIG to center the Ronchigram. Correct probe stig (OBJSTIG +DEF/STIG) to make the Ronchigram as circular as possible.
16) Insert appropriate aperture (#3) and center it.
17) In Sirius Client software, change Detector to EXT (Gatan HAADF detector).
18) Press Search under DigScan(Digital Micrograph) DM – DigiScan – Search or Review – Start, you should see image. If necessary, go to DM – HAADF control – Auto PMT Gain to optimize image contrast.
19) Tune **Focus** to get the best contrast.
20) Click on 'Record' to record an image. If necessary, click DigiScan setup button at lower right corner of the tab to change parameters for search/preview/record.

# I. Introduction to Scanning Transmission Electron Microscopy
Fig. 1
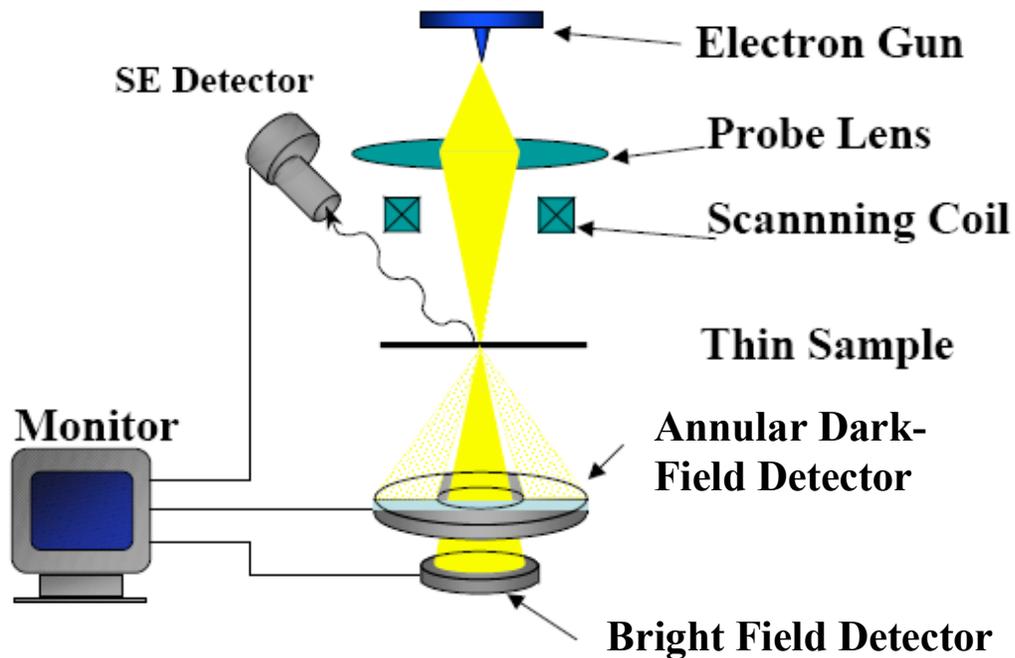
In Fig. 1, a small electron beam (0.13 nm-2.5 nm) can be formed by probe-forming lens (condenser lens and objective pre-field lens). Similar to SEM, the beam can be controlled by a scanning coil to scan an area of a thin sample. After passing through the sample, the electron beam will be selected by bright field detector and annular dark field detectors to form images. Normally, the direct beam and some diffracted beams may hit Bright Field Detector to form a BF or lattice image, other diffracted beams are collected by Dark Field Detector to form a DF image. The magnification of recorded image is dependent on the scanned area.

The detector size is fixed, but imaging conditions may be adjusted by changing camera length and projector deflector, and tilting the sample, which enables operator to achieve different goals (for example, Z-contrast image, diffraction contrast image, phase contrast image). There is no BF detector on JEM-2100F, the BF image may be achieved by using ADF detector + projector deflector. The image quality is highly influenced by the beam condition (size, shape, alignment and focus), which may be monitored by Ronchigram (Fig.2). In general, the Ronchigram must be circularly symmetric in order to get a good image.

Fig. 2
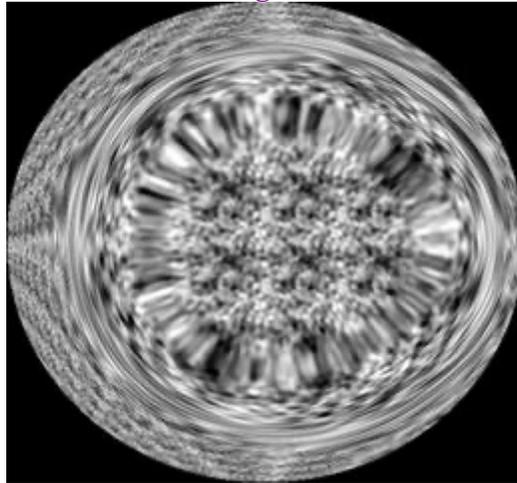
In order to get quantitative results, operator needs to know/measure convergence angle (2αs) and collection angle (2βs), as shown in Fig. 3.

Fig. 3

$$2\alpha = 2\theta_B \frac{a}{b} \qquad [5.6]$$

Incident beam

α_S

Sample

β_S

Detector

calculated from simple geometry. Depending on the diameter (d) of the spectrometer entrance aperture and the distance from the specimen to the aperture (h), β (in radians) is given by

$$\beta \approx \frac{d}{2h} \qquad [37.1]$$