MSE 460 TEM Lab 8:
Basic High Resolution Electron Microscopy Operation

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Aims: The aim of this lab is to familiarize you with basic HREM operation

TEM: JEOL 2100F TEM or JEOL ARM-300 (EPIC)

Time: 3 hours

You may spend the first hour to repeat what you learned in previous labs. This 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 by live Fast Fourier Transformation of HREM image of amorphous carbon.

IX. High Resolution Electron Microscopy (HREM)

In this lab, you will learn to basic method of HREM, images showing phase contrast originated from the interference of the transmitted beam and diffracted beams. The quality of HREM images are influenced by the coherence of electron beam, beam alignment, sample thickness, sample orientation, objective lens focus and astigmatism. The better you align the TEM, the better quality the images have.

If the instrument you are using has a high resolution polepiece, a highly coherent source, and the operating conditions are perfect (vibration, vacuum, alignment, etc.), atomic resolution images showing projected atomic columns can be obtained. We will learn step by step how to align the TEM and collect such images with close to atomic resolution. Note that a better method called coma-free alignment is required to obtain images which are easily interpretable at the atomic scale, without this there are positional errors of 1nm or more for where the fringes are.

Procedure:

1. Align JEOL-2100F TEM under image mode. Especially, please check the HV center at MAG 100k or higher. It is worthwhile to get this aligned as well as possible. However, for really good HRTEM you need to do coma-free alignment.
2. Start DigitalMicrograph (DM) software if they are not started.
3. DM – Find and expand Camera View and Camera Acquire tabs.
4. Find amorphous area on specimen, click Camera View and Start to view CMOS image. At high mag (10kx or up) use live FFT (process menu) to correct objective stigmatism. You want to correct the astigmatism for a small defocus. Unfortunately if you do not use coma-free alignment then you will not be able to fully correct the apparent astigmatism at all defocus – tilt misalignment leads to an effective defocus dependent astigmatism.
5. Coma-free alignment: start beam tilt wobbler at frequency of 3 (as shown in the menu). By adjusting beam tilt so that the FFT patterns are close to the same for both plus and minus tilts directions of beam tilt. You should try and do this for more than one defocus. Since the beam tilt can be wobbled along x- and y- direction, you should do one after the other, i.e. wobble the beam tilt along x-axis and correct the beam-tilt along x-axis; then wobble the beam tilt along y-axis and correct the beam-tilt along y-axis.
6. Go back to 12 and recorrect the astigmatism – it will appear to have changed! If you have enough time iterate Steps 12 & 13 a few times; if you do not then just move ahead to 15.
7. Expand Camera Acquire tab and click Start Acquire button to record image. Adjust exposure time if necessary. Save image to your own folder in DM3 format. You will obtain better images by defocusing the illumination a bit and using a longer exposure. How long an exposure you can use will depend upon how much the sample is drifting.
8. Slightly turn FOCUS knob and try to record few more HREM images with different defocus values. See how the defocus changes the HREM image contrast and where the fringes appear.