Physics 7230/4230 FS2012 LAB 5: Qualitative EDS Analysis

NAME:_____ **10/15/2012**

INTRODUCTION

- **Samples**: -Polished metamorphic rock thin section containing silicates, sulfides and other minor minerals.
- **Operating Conditions:**
  - **-SEM:** 20 kV; Spot 4.5, Aperture #6; 12 mm WD.
  - **-Electron Detectors:** Everhart-Thornley SE; Quad BSE detector.
  - **-Imaging:** 640x480; 40 ms dwell time
  - **-EDS:** 60 second acquisition time; deadtime ~20-30%
  - **X-**ray range at 20 keV (horizontal scale)
- **Objectives:** - To combine BSE Z contrast imaging with qualitative EDS analysis, labeling the spots where the spectra were collected on the BSE micrographs

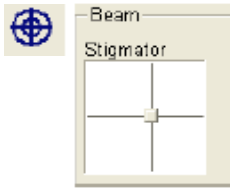
Start-up

1. Log in book. Record date and time
2. See **Lab 4** (Low vacuum lab) for acquiring an image

Remember…

Acquire Image and Set Working Distance ("Link Stage") at 60 mm, then 25 mm and finally at 12 mm

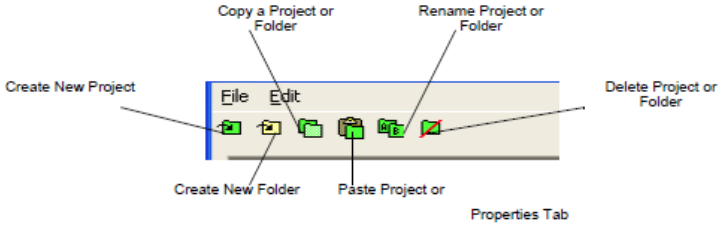
Perform Alignments and Adjust

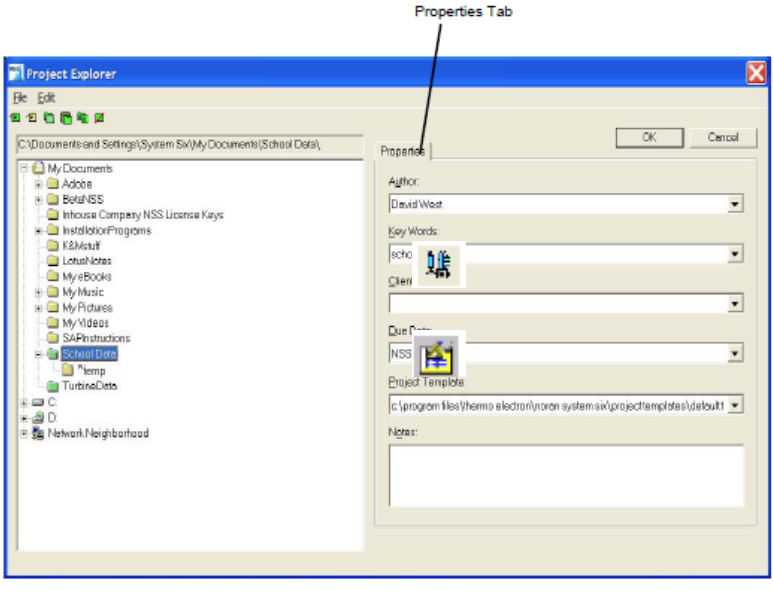


Astigmatism

EDS STARTUP AND INTIAL SET UP

1. Click on the NSS icon on the desktop of left monitor (bottom left corner).
2. Open the folder labeled your name
3. With the right mouse button, make a new project, rename it to Quanta EDS lab with a date.
4. Double click on it to start the program.
5. Make sure Quad 4 (chamber camera) is paused.
6. Click on the EDIT MICROSCOPE PARAMETERS icon, 2nd tool bar down.
   a) Make sure that the KeV, WD, and magnification are correct. If not, get help.
7. Click on the EDIT ACQUISITION PROPERTIES icon, 2nd tool bar down

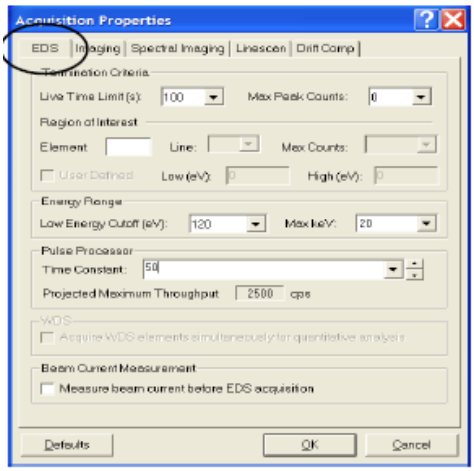




a) Set up the EDS Tab
- **(i)** Set the live time to 60 secs.
- **(ii)** Set the lower energy cutoff to 150 volts (type it in)
- **(iii)** Set the max keV to 20.
- **(iv)** Set the pulse processor time constant to Rate 4.

b) Set up the IMAGING tab
- **(i)** Set the image resolution to 1024
- **(ii)** Set the frame time to 30, type it in.
- **(iii)** Set the number of frames to 1

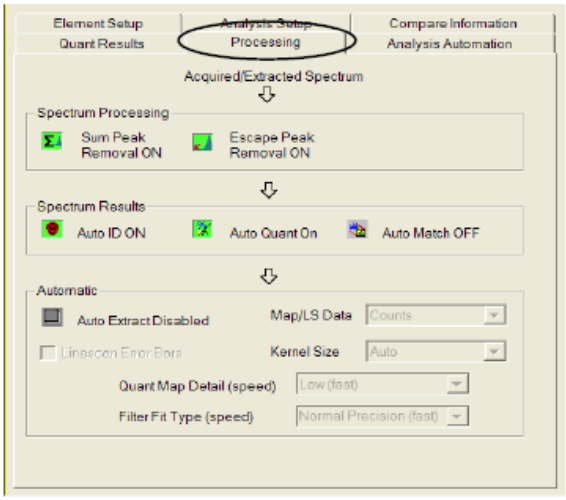
8. Click OK, and look at the dead time in the lower window. It should be around 27%, but no higher than 35%.



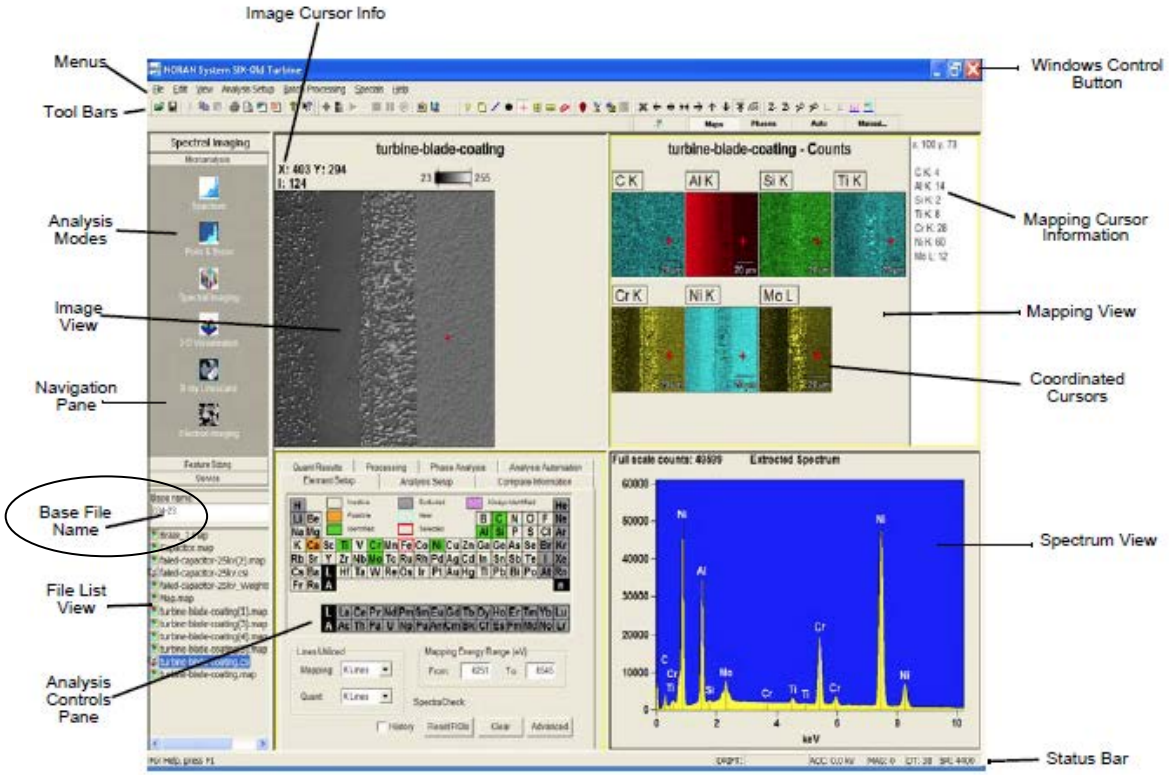
EDS DATA COLLECTION I and OTHER TOOLS

9. On the SEM, find an area with both dark and bright minerals including various grey phases at ~500x.

10. Processing Setup. In the lower left quad select the Processing tab
- **a)** Turn on both the Remove Sum Peaks and Remove Escape Peak options
- **b)** The Auto ID option should be on, if not, turn it on.
- **c)** Turn on Auto Quant.
- **d)** Go back to the Element table tab.

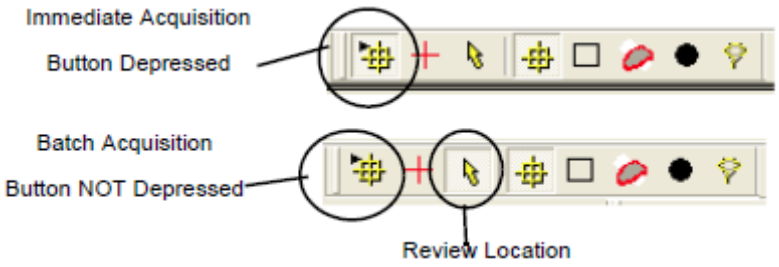


11. Collect a spectrum over the area of interest (Bulk Analysis) as follows
- **a)** Label the spectrum, Base name field on the left, to area scan 1.
- **b)** Select Acquisition icon to begin (Arrow Play button)
- **c)** When the acquisition is done, the spectrum name will appear in the bottom left field.



12. EDS collection with Point and Shoot as below

- **a)** Click on the Point and Shoot icon
- **b)** Make sure the SEM image is in focus with good brightness and contrast
- **c)** Label the Point and shoot file.
- **d)** Click on the Acquire Average Image icon, to the left of the EDS Play button.
- **e)** In the Point and Shoot tool bar field,



- **(i)** Deactivate instantaneous collection, first button one on left.

- **(ii)** Select points by clicking on the image over various minerals of image, 6 or so. Note: the acquisition mode is in the default Point method
- **(iii)** Click on the EDS acquisition button (play arrow)

B. EDS DATA COLLECTION II and III with POINT and SHOOT

1. Select another area at higher magnifications with a variety of mineral phases. For #2 go to ~2000x and for #3, go to ~5000x
2. Repeat Steps D3 and D5 for both magnifications.

C. SHUTDOWN the SEM

1. Go back to Quad view if in single screen mode.
2. Unpause the Chamber scope.
3. Return the Stage to Center Position under the Stage menu.
4. Set Z at 20 mm.
5. Click the VENT button. Answer Yes. You will hear an initial hiss, this is the bam blanker being inserted into the column.
6. Wait until the venting sound stops, ~ 1 min.
7. Open the CHAMBER DOOR. Pull the door easily out with the handle at bottom.
8. Remove the sample, loosen the screw with allen wrench.
9. Make sure Quad 4 is still active.
10. **Gently close door!!** While watching the chamber scope**,** slide the door in slowly. Stop when it rests against the o-ring and then gently push it closed.

****Remember if the door is closed to hard and fast, the resulting pressure could damage the thin Be window on the EDS detector!!**

11. Select HIGH VACUUM if needed.
12. Click on the PUMP button.
13. Wait for the SPECIMEN CHAMBER to turn green.
14. Log Out. At the top of the screen, click on STOP UI.

D. EXPORTING THE EDS DATA TO A WORD DOCUMENT

1. Under FILE, go to PAGE SETUP
2. Under HEADER fill out your name, company info (Physics 7230/4230, and title (Qualitative EDS Lab 5)
3. Under the SPECTRUM Tab
   - **a)** Click on Discrete at the top
   - **b)** All other options should be checked except ROI results.
   - **c)** Click OK
4. Under the POINT and SHOOT tab, make sure all the options are on and Click OK
5. Select the first POINT and SHOOT file
6. Click on the WORD icon at the top and the tool bars and it will create a file with the image, spectra and quantitative results. It will also save it automatically in your project folder.
7. Select the other Point and Shoot files, and hit the Word icon to add them to the same Word file.
8. Go to Spectrum and repeat the same procedure for the three spectral files.

E. CLOSE the NORAN SYSTEM SIX PROGRAM and RETRIEVE WORD FILE

1. Under FILE, select CLOSE PROJECT
2. In the new window, click OK to save the files.
3. In the next window, click on Quit SYSTEM SIX.
4. Program should end and take you back to the desktop
5. Click on the desktop “shortcut to Shareddata.
6. Transfer your data to the Userdata folder