Basic Instructions for Inca EDX

Introduction

Energy dispersive X-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis and chemical characterization of a sample. Characteristic X-rays from a specimen are stimulated by the incident electron beam in a SEM. Typical energy resolution of an ED detector is about 140 eV. The x-ray data acquisition is done by Oxford spectrometer attached to the SEM chamber, and the spectrometer is control by the Oxford INCA software.

Procedure

I. Setting up the microscope and INCA (EDS acquisition program)

1. Sign in to the session via the computer in the hallway.
2. At the login screen select 'Quanta ESEM' and enter the user profile.
3. Follow the ESEM instruction (WD = 10 mm, proper focus/stigmatism/lens alignment settings etc.) and locate the area of interest. Depending on the materials of interest, select an appropriate electron beam voltage. As a rule of thumb, in order to excite the desired characteristic x-rays it takes a beam energy at least 1.5X higher than the x-ray energies.
4. If necessary, the log-in information to the INCA computer:
   Username: supervisor
   Password: supervisor
5. Open up INCA program with the computer to the left of the SEM computer. The program operates in the energy mode by default.
6. Select "Full Acquisition Mode"

II. INCA Overview

INCA is the software used for EDS/WDS data acquisition and analysis.

Mode Flowchart

Chart item detail

Acquisition Mode:
- Analyzer
- Point & ID
- Mapping

Spectrometer Mode:
- Energy Dispersive
- Wave Dispersive
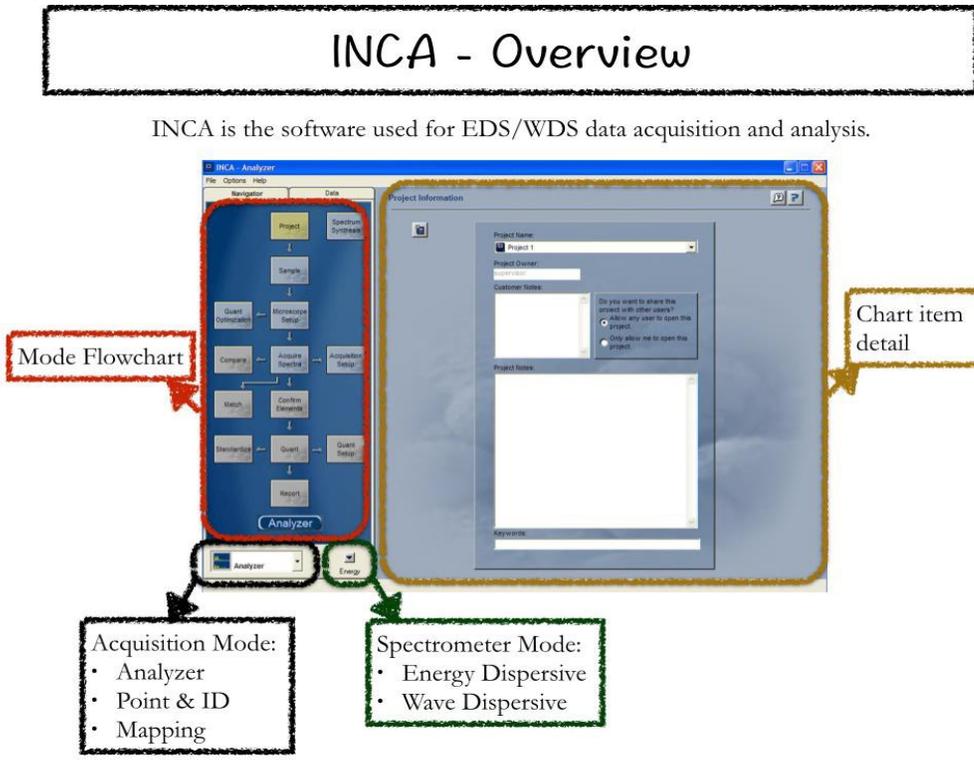
III. Acquisition Modes

The procedures for the two most common acquisition modes (Point & ID and Mapping) are provided in this manual. Use 'Point & ID' mode for quantitative chemical composition (atom % and weight %) analysis of a sample. Use 'Mapping' mode to obtain the spatial distributions (2D map or 1D line scan) of the elements presented in the sample. The mode can be selected from the mode drop-down menu located on the bottom left of the program.

IIIa. Insertion of Detector

Go to top tool bar and select "Option", then "Detector", then Slide. Select "Move In".

IIIb. Point & ID

1. Sample
   Define the sample condition. Is the sample polished? Is the sample coated? Whats the coating material and its properties (thickness and density)?

2. Image Setup
   Default 'Image Resolution' at 1024x896, 'Speed' at Fast, 'Data' at 8 bit. In general high quality images are acquired directly from the SEM not through INCA. Enable 'SiteLock' if image drifting is experienced.

3. Acquisition Setup
   Default 'Livetime' at 60 seconds, 'Process time' at 5, 'Spectrum range' at 0-20 keV, and 'Number of channels' at 2K. *Livetime* is the time for which the system is processing counts into the spectrum. *Process time* is the length of time spent reducing noise from the x-ray signal coming from the ED detector during processing. The longer the *Process time*, the lower the noise but with lower X-ray acquisition rates.

4. Site of Interest
   Press the green button located in the tool bar to acquire the image. By default, the image is acquired from the secondary electron detector.

5. Acquire Spectra
   Select the desired *point/region/line/grid* of points on the previously obtained SE image to carry out the spectra acquisition. For the line and grid x-ray acquisitions, the density of sampling points can be defined by entering desired X and Y values on the right.

6. Confirm Elements
   After the spectra acquisition, you can remove/add elements to the subsequent analysis by double clicking the element buttons. Selected elements would turn yellow.

7. Quant
   Chemical composition based on the obtained spectra is calculated here.

8. Report
   Select a desired report layout using the *Template* dropdown menu and output the report to MS Word. For example,
   Quant Results > Image/Spectrum/Summary of Results
   Warning Do not select a template that has a Graph or Chart in it. There is a bug that causes the system to crash and shut down, losing your data.

IIIc. Mapping

1. Sample, Site of Interest, and Image Setup
   Please refer to the descriptions in 'Point & ID' mode.

2. SmartMap

Element distribution can be obtained either with a 2D map scan or 1D line scan. The options are to scan (1) full field of view, (2) a line, and (3) selected rectangle region of interest. The default setting is to scan over full field of view.

3. Element Maps
    The result of 2D map scan is displayed here.
4. Element Linescans
    The result of 1D line scan is displayed here.
5. Element Setup
    Element addition/removal.
6. Report
    Select a desired report layout using the *Template* dropdown menu and output the report to MS Word.
    2D Map: *SmartMap > Maps > X-ray Maps/Image*
    1D Line Scan: *SmartMap > Linescans > Linescans (Tiled)/Image*

IIId. Retacting Detector

Go to "Option", then "Detector", then "Slide". Select "Move Out".

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:
1. Contact IAC Staff.
2. Leave system as is, Do Not disable vacuum system.
3. Try to shut off the High Tension.
Audible/Siren Emergency Alerts:
    Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

Nan Yao: Office (609)258-6394; Cell (908) 922-2236 Email: nyao@princeton.edu
John Schreiber: Office (609)258-0034; Cell (215) 431-4670 Email: js51@princeton.edu
Paul Shao: Office (609)258-3851; Cell (847) 721-086 Email: pshao@princeton.edu