



Standard Operating Procedure for XPS on the Nexsa

Manager information

Dr. Cassie Ward

Email: ward@wayne.edu

Office: 061 Chemistry

Phone: 313-577-2587

Dr. Sameera Perera

Email: samarage.sameera.perera@wayne.edu

Office: 135 Chemistry

Laboratory information

Lumigen Instrument Center NMR and EPR Laboratory

Wayne State University, Department of Chemistry

40 Chemistry Building

5101 Cass Avenue, Detroit, MI 48202

Phone: 313-577-0518

Safety requirements

Access to the lab will be revoked if you do not follow these safety procedures.

1. While working in the NMR Laboratory, researchers are always required to wear Personal Protective Equipment (PPE). The appropriate PPE includes **safety glasses, long pants/skirt covering the legs completely, closed-toe shoes, and gloves**. Do not wear gloves while using the computer keyboard or mouse. All required PPE needs to be supplied by the user.
2. Food and beverages cannot enter the lab. Never eat or drink inside the lab.
3. Please keep the area around the instruments and prep stations clean. If you use the lab's sample holders, clean them after use. Properly dispose of glass waste in the appropriate container. All samples must be properly **disposed of in your research lab**.
4. Following a spill, please clean it up with a paper towel or broom. If you are unsure how to

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clean up the spill, contact the lab manager (Dr. Ward or Mr. Anderson) or the LIC Director (Dr. Westrick). If the spill occurs after hours with personal injury, please contact WSU police department (7-2222) and Dr. Westrick, immediately.

5. The Standard Operating Procedures (SOPs) are next to the instrument. The users are to follow what is written in these procedures.
6. You must log the usage time into the appropriate instrument logbook.
7. All researchers working in LIC must complete the EH&S initial course for Laboratory Safety Training and show proof of completion. Users whose safety training has expired will not be permitted access to the laboratory.
8. In case of an instrument malfunction, turn off the instrument and contact Dr. Ward immediately.

Additional safety requirements for the XPS

1. In case of an instrument malfunction, turn off the instrument by turning the switch in **Figure 1** to the OFF position. Contact Dr. Ward or Dr. Perera immediately.



Figure 1: Picture of the NEXSA. The red arrow is pointing to the back left of the instrument, where the emergency power shut down knob is located. Turn the knob to the "off" position.



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Training and Usage Requirements

Before training

1. All users must pass the online EH&S Laboratory Safety Training and Radiation (X-ray) Generating Machine training (<https://about.citiprogram.org/en/homepage/>). Print the certificate after completing the quiz and bring it to Dr. Ward. The two safety trainings must be completed every January and the printed certificate must be brought to Dr. Ward.
2. You must have an Infinity account and have requested access to the Lumigen X-ray Laboratory. You must also have an index number assigned to you from your PI.
3. Contact Dr. Ward for training.

After training

1. You can reserve time for the XPS using the calendar in Infinity.
2. You must write your first and last name legibly and the hours of use into the Logbook that is next to the instrument.

Overview

1. Determine elemental identification and composition and the chemical state of thin films, metals, plastics, powders etc.
 - a. Samples must be prepped before entering the entry-lock chamber. If samples are volatile, either use heat or vacuum to degas samples.
 - b. Sample height maximum 20 mm.
2. Sputtering with MAGCIS ion gun (Monoatomic And Gas Cluster Ion Source)
3. Angle resolved-XPS (see AR-XPS SOP) for samples with less than 4 nm thickness
4. Rotational holder available (15 mm max. sample thickness) (see Rotational holder SOP) for Zalar Rotation.
5. Fiber holder is available (150 μ m minimum sample thickness) (see Fiber holder SOP)
6. Vacuum Transfer Module (VTM) is available for air-sensitive samples (see the SOP for

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VTM).

Sample preparation

1. Always use gloves and clean tweezers to avoid contamination. Do not touch the areas you wish to analyze.
 - a. You can clean the holders and utensils with **isopropyl alcohol** only.
2. Samples are mounted onto a standard sample holder using the spring clips (**Figure 2**).
 - a. Cu tape can be used alternatively, especially for powders.

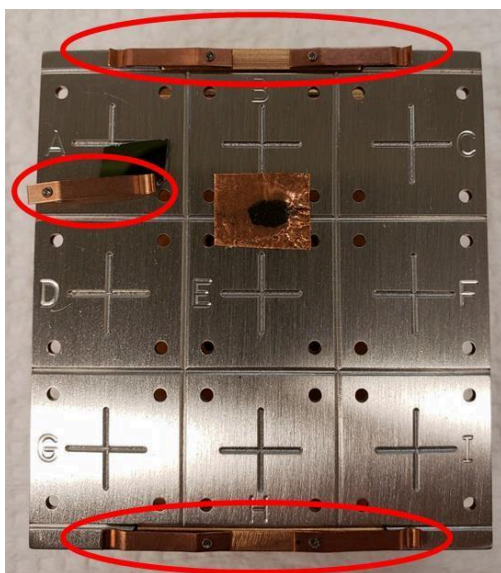


Figure 2: Standard XPS sample holder. Notice the spring clips (circled) and samples do not hang off the edges of the sample holder.

3. Samples, tape, and spring clips must all remain inside the area of the sample holder. **Do not overhang anything over the edges of the holder.**
4. Also, check the height of the sample(s) with the height checker.
5. Turn the sample holder upside down to ensure all samples remain on the holder before placing it into the entry lock.

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Operating the instrument

This SOP will cover how to use the software with this sample holder (**Figure 2**) for XPS standard high resolution and etching measurements. Refer to the other SOPs for the other stages or options on the Nexsa.

1. The program for the NEXSA is called Thermo Advantage. The icon is a pyramid at the bottom of the taskbar. If the program is not already opened, you can click on the pyramid.
2. **Figure 3** shows the “Sample” menu.
 - a. The options at the top shown in **Figure 3** are what should be displayed at the start and end of the experiment. We keep the standard sample holder (**Figure 2**) in entry lock chamber under vacuum.
 - b. Select the left option, “Vent load entry”, labelled **1** in **Figure 3**. Click “Yes” to confirm this action.
 - i. This will open the entry-lock chamber. The chamber door will open automatically. Be patient.

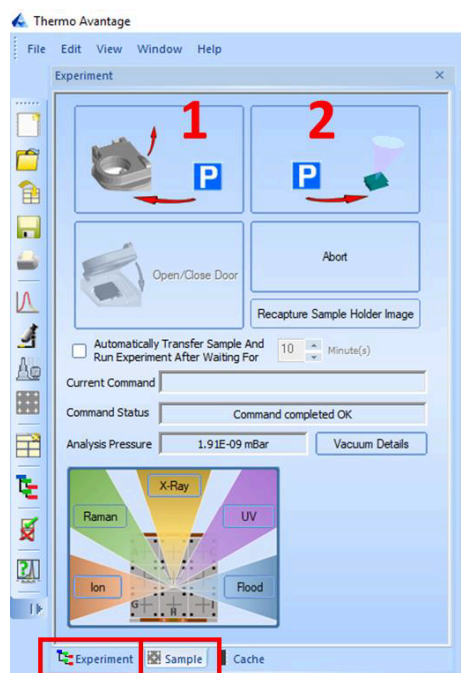


Figure 3: Screenshot of the Sample Menu. 1) Vent the entry lock and open the door. 2) Move sample to

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analysis. To set up an Experiment Tree, click on the “Experiment” tab next to the “Sample” tab.

3. The sample holder can only be positioned in one direction in the entry lock. The “A” on the sample plate should be pointing towards the analyzing chamber (**Figure 4**).
4. Once the holder is in position, select “Pump and park in the entry lock” on the top left (which is labeled **1** in **Figure 3**, but with a different icon since it is closing the door) to close and pump down the entry-lock chamber.
 - a. This will close the door and begins the rough pump. Within 3 min the chamber should reach $\times 10^{-5}$ mbar pressure.

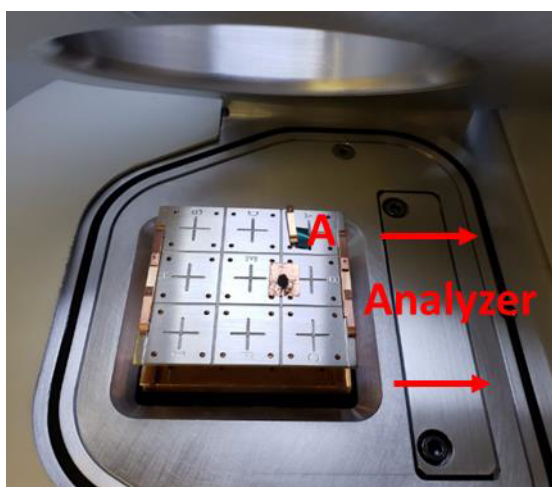


Figure 4: Sample holder in the entry lock with the “A” at the top right, which is pointing towards the analyzer.

5. The “Command Status” will read “Command completed OK” when the pressure reaches 10^{-6} mbar, but it is best practice to leave the sample in the entry lock for about 10–15 mins.
6. Once about 10^{-6} to 10^{-7} mbar is reached, transfer the holder to the analysis position by selecting the “Move sample to analysis” on the top right, labeled **2** in **Figure 3**.
7. The analysis chamber will need to reach at least 10^{-8} mbar to run an experiment, but most likely should be allowed to reach 10^{-9} mbar after about 20 mins.
8. To select the sample for analysis, double click on the sample shown in the Platter View window or double click on the sample in the Live View. You can also move the stage around by using arrows in the Live View (**Figure 5**).
 - a. To toggle between Sample View (live view of the sample inside the analysis chamber) and Transfer View (live view of the entry-lock chamber), right-click on the Live View window, go to Channel, then select **Transfer View** or **Sample View**.



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Setting up an experiment

When setting up an experiment, it is recommended to change the default names for every Experiment Control chosen in the Experiment Tree. You can change the names by clicking on the specific control or by using the **General** tab in the Properties Panel (**Figure 5**).

1. Go to “**File**”, “**New Experiment**” or “**Open Experiment**.”
2. Click on the “**Experiment**” tab (**Figure 3**). **Figure 6** shows an example experimental run for depth profiling and **Figure 7** of a Point analysis. To fill out the Tree, follow the instructions below with **Figure 7**.
 - a. Click on “**Source**” Insert icon (labeled a).
 - i. Select the appropriate source for the experiment. Options are:
 1. Gun shutdown: Enables all sources to be turned off at the end of the experiment.
 2. ISS Source- Ion Scattering Spectroscopy (a.k.a. LEIS-low energy ion scattering) to obtain elemental information from the first atomic layer of the surface by collecting positively charged ions instead of electrons.
 3. UV Source- Used for UPS (UV photoelectron spectroscopy) measurements. Similar to XPS, UPS uses UV radiation to generate photoelectrons in the KE range 0–50 eV and corresponds to the valence electrons involved in bonding.
 4. Manual Source Gun- This is used for Raman.
 5. X-Ray Gun- For XPS measurements.
 - ii. Select the “X-Ray Gun” for XPS measurements. With X-ray Selected in the Tree, there is an “X-ray” tab option in the Properties Panel. Enter the desired spot size (μm) and select to “Turn on” or “off” the Flood Gun (FG). In general, it is better to have the flood gun on if you are uncertain about your sample’s properties. It does not hurt the XPS experiment to have the gun on.

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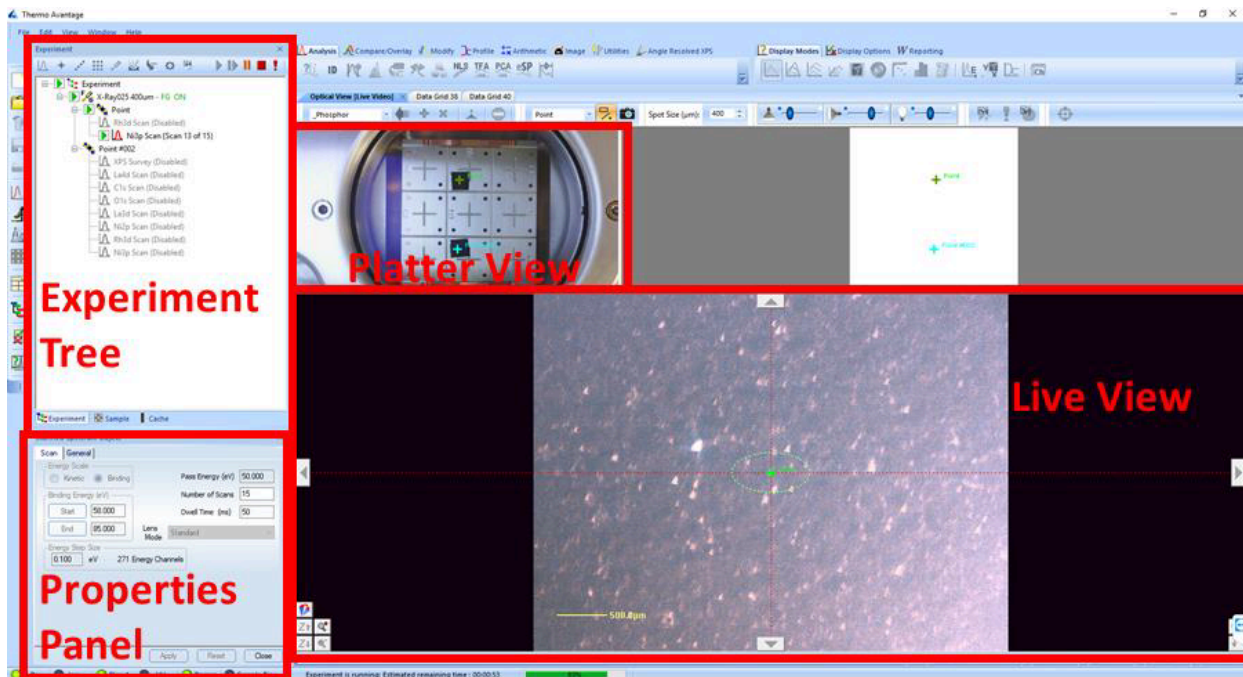


Figure 5: Avantage program of the sample views and experimental list.

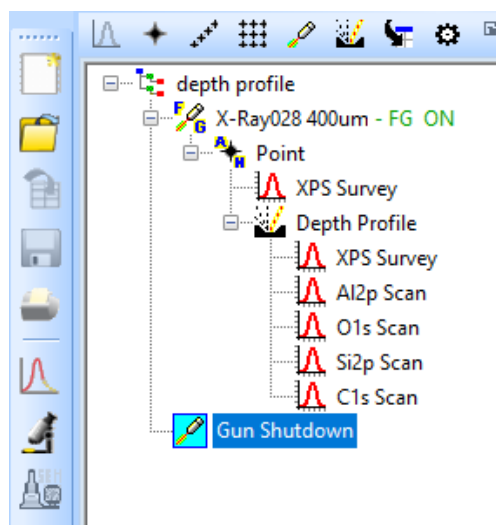


Figure 6: Example of a depth profile experiment.

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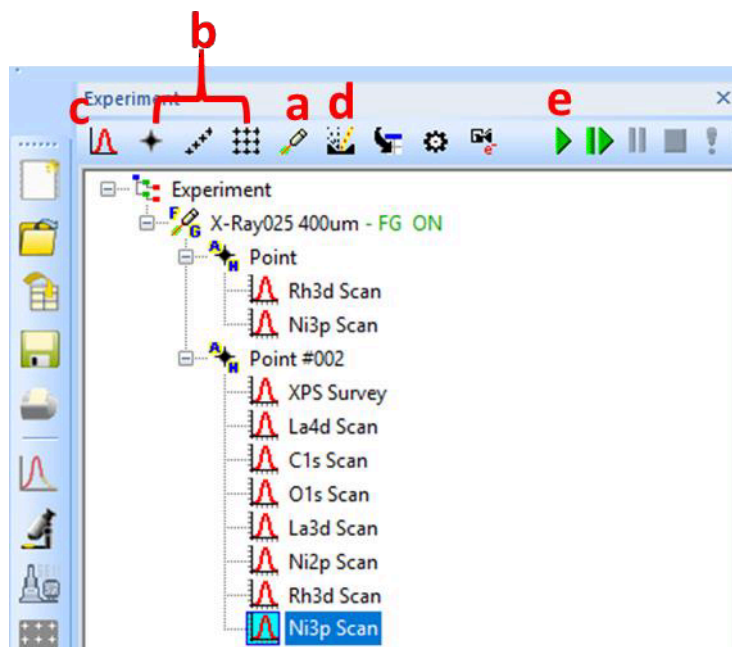


Figure 7: Experiment Tree for setting up an automotive list of measurements. a) Source insert icon, b) (left to right) Point, Line, and Area insert icons, c) Spectrum insert icon, d) Ion Gun insert icon, and e) Experiment Run icon.

- b. Select either Point, Line, or Area (labeled **b** in **Figure 7**).
 - i. Point-Analysis a single point. **Figure 8** shows the different menus for the Point Position. Double click on the sample position of interest (Figure using Live View or Platter View). Then click “Read” to insert the x, y, and z positions under the “Position” tab.
 - ii. Line Scan- Similarly, adjust the number of points between a “Read Start” and a “Read End” to create a series of points along a line.
 - iii. Area- Select the “Step Size” or “Point Count” to change the number of points in an area. You can use the mouse to adjust the area coverage over the sample in the Live View window.



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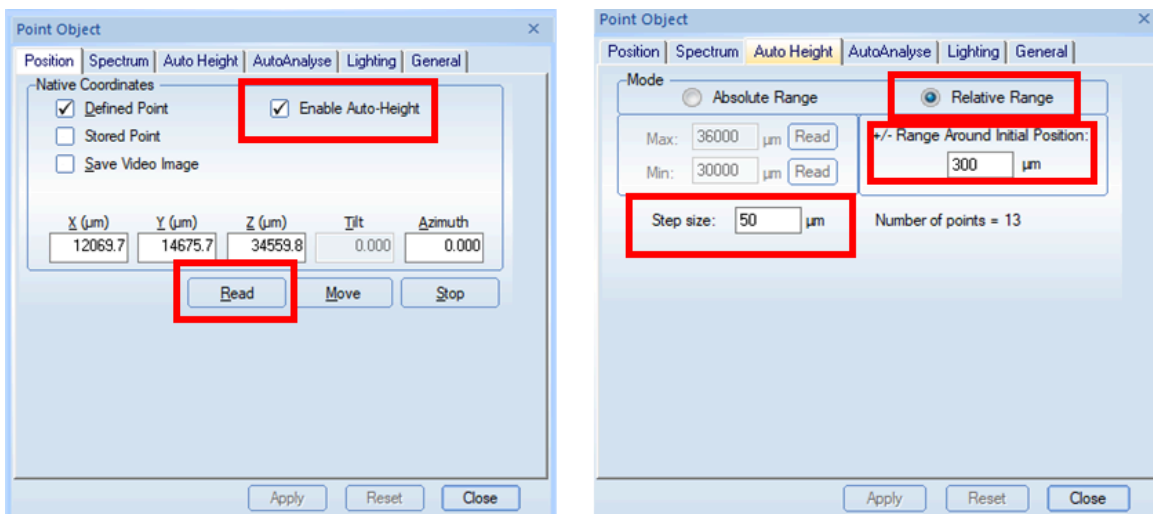


Figure 8: When Point is selected in the Experiment Tree, these are the tabs available to change details about the point position. Under the “Position” tab (left) click on the “Read” button to enter the current position shown in the Live View window. Also, check the “Enable Auto-Height” option, which will optimize the sample height position for optimal signal before collecting. In the “Auto Height” tab (right), select “Relative Range” and enter 300 μm with a “Step size” of 50 μm .

- c. Select “Multi Spectrum” under the Spectrum icon (labelled c in **Figure 7**).
 - i. The menu shown in **Figure 9** will appear. Perform a “Survey Spectrum” scan.
 - ii. Select which individual elements of interest for high-resolution scans. You can right-click on the element to select the specific transition peaks.
 1. Under “**Spectrum Type**” select “**Scanned**”. This option is for the best possible resolution. Snap shot collects more quickly and is used best for multi-level data sets (lines or areas).
 - iii. Click “**OK**” and the Tree will populate with the selected scans.
 1. You can easily move the scans in a specific order by dragging the scan to that position.
 2. You can also copy a series of scans to create another set of runs by selecting the “**Point**,” right click, and select “**Copy Selected Item**.” Then right click and “**Paste Selected**” Item.
 3. To delete a scan, highlight that scan and click the delete key.

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4. You can disable (without deleting) a scan (or series of scans) by highlighting the scan and push the space bar.

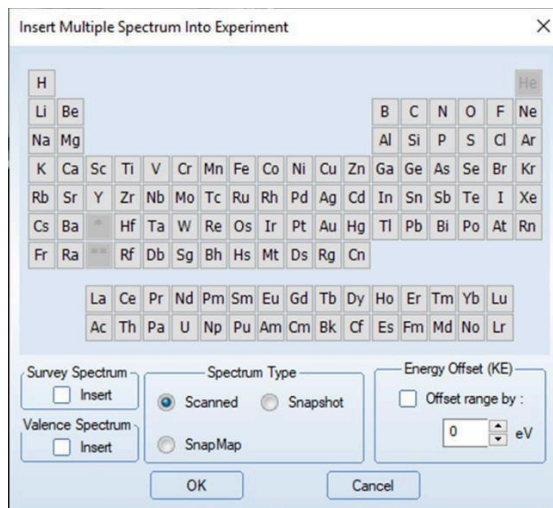


Figure 9: Multiple spectrum menu. Select “Survey Spectrum” to run a fast scan from 0 to 1350 eV. Click on elements to select them for high-resolution scans. Right-click on the element to select a peak different from the most intense core line.

- iv. Highlight each element or survey scan in the Experiment Tree to see the “Scanned Spectrum Object” tabs (**Figure 9**). The “Pass energy” determines the energy resolution. 50 eV is the recommended pass energy with 0.1 eV step size for narrow scan spectra. For a survey spectrum, 200 eV pass energy with 1 eV step size is recommended.
- v. The Binding/Kinetic energy range can also be adjusted, which is particularly useful if multiple peaks overlap in the similar range.

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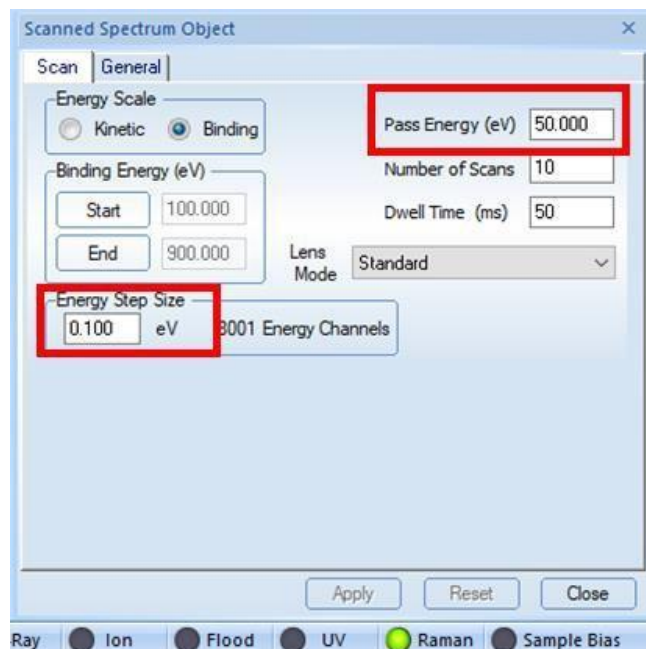


Figure 10: Scanned Spectrum Object menu of options. Under the “Scan” tab, this controls the options for each element or survey scan for Pass Energy, Energy Step Size, and Binding/Kinetic Energy Range.

- d. For Profiling measurements (labeled **d** in **Figure 7**),
 - i. Ion Beam Etch Object is for cleaning a surface to free it from contaminants, like carbon. About 10 seconds Etch Time at 500 kV.
 - ii. Depth Profile is for running multi-layer measurements
 - 1. Generally, set up a Survey Scan before the Depth Profile
 - 2. Under “**Profile**” the options to change will be sample dependent but follow **Figure 11** for a starting point.



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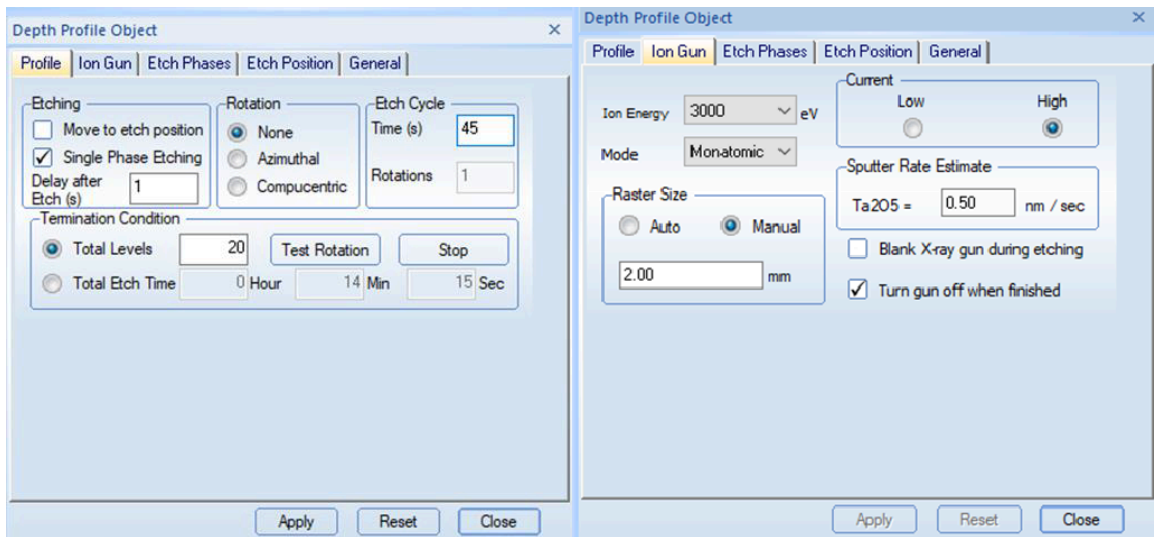


Figure 11: The Properties Panel for Depth Profile for multi-layer analysis. Under Profile, change the Etch Cycle time (s) based on how long to run the gun (how much material to remove between scans).

1. If “Auto” is selected under “**Raster Size**”, the ion beam sputtered area will be automatically optimized relative to the size of the X-ray spot (5× spot size). The raster area is rectangular in shape where it is elongated in the x direction.
- b. At the end of the Experiment Tree, insert the Gun Shutdown from the Source Icon

Figure 6.

- i. Click on the Gun Shutdown icon and in the Properties Panel, select “Transfer To Park under Sample Action: will shut down all guns”.
 - ii. This will turn off all sources and move the sample stage to the entry-lock chamber.
- e. Then click on the Play button to run the complete experiment (labelled e in

Figure 7).

3. When the experiment is over and the sample stage is in the entry-lock position, click on “Vent load entry”, labelled 1 in Figure 3. Click “Yes” to confirm this action.
4. Remove your samples from the stage and put the stage back into the entry-lock and click on “Pump and park in the entry lock” on the top left (which is labelled 1 in Figure 3).

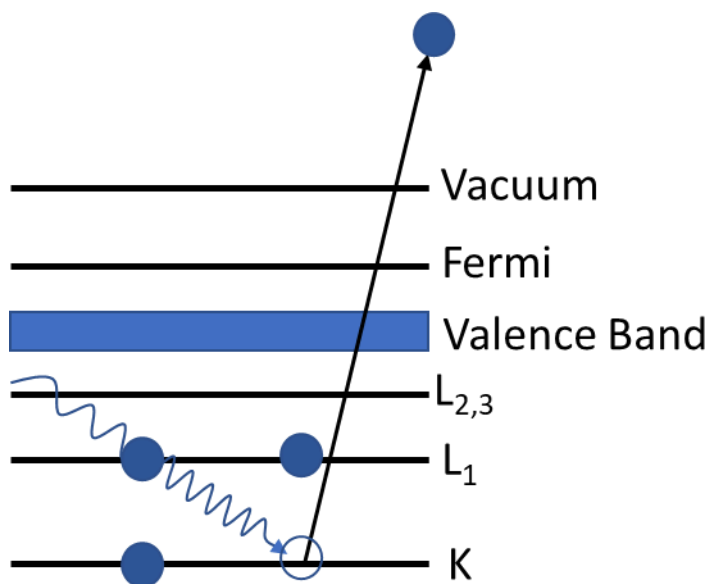
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Standard reference materials

1. Two standards are provided by the Crystallographic Laboratory.
2. Three inbuilt standard samples (i.e., Ag, Au, and Cu) are located inside the analysis chamber.

Basic principles of XPS

1. X-ray photoelectron spectroscopy (or electron spectroscopy for chemical analysis, ESCA) is based on the photoelectric effect where photons eject electrons when their energy is at the threshold frequency (ν_0).
2. The kinetic energy ($\frac{1}{2}mv^2$) is equal to the energy of the incident radiation ($h\nu$) minus the metal's work potential (Φ). The work potential is the minimum frequency that will eject an electron ($h\nu_0 = \Phi$).



3. The Nexsa uses a Al $\kappa\alpha$ source (1.487 eV) with a quartz monochromator



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References

1. ThermoScientific. (2018). *Nexsa Surface Analysis System Operator Manual*, Issue 2.
2. Donald A. McQuarrie, Quantum Chemistry, Sausalito, 1983.