Quick User Guide for XPS Analysis

Please refer to "General User Guide" and its table of contents to find details on how to execute the following tasks. When in doubt, discuss your procedure with Dr. Lozovyy before proceeding.

1. Your sample and sample plate should be UHV compatible and prepared according to UHV requirements (refer to PHI manuals).
2. Sign in into lab logbook.
3. Create new platen in software prior to experiment (p. 17).
4. Load platen (with properly prepared sample(s)) to intro station and follow procedure to move platen to the analytical chamber (p. 18).
5. Navigate to and set up positions for sample analysis (p. 20).
6. Setup data acquisition protocol (i.e., charge neutralization, Z-alignment, sputter treatments, XPS acquisition type, scan properties, positions sequence, queue; p. 23,42)
7. After data acquisition is complete, remove platen from analytical chamber followed by intro station (p. 18 – reverse procedure from step 2).
8. The XPS apparatus, vacuum condition should be at the same condition as prior to your experiment.
9. Transfer data for analysis with MultiPak from primary computer. Facility is not responsible for data management.
10. Sign out in the lab logbook.
11. Any data published in a manuscript/presented are to acknowledge support from NSF DMR 1126394 and Indiana University's Nanoscale Characterization Facility. Scholarly standards are also to be observed when acknowledging contributions from research staff.

General User Guide

This overwview is provided to help you start your XPS experiment, collect data and reduce them using SmartSoftware or MultiPack software.

Contents

Introduction to SmartSoft-VersaProbe 3

Create New Platen 17

Attaching/Removing a Platen(Sample) at the Intro Station 18

Move Platen(Sample) into Analytical Chamber 19

Navigate: Overview 20

1 Navigate: Navigate using the Platen Viewer Images 21

2 Navigate: Navigate using the Platen Viewer Google Bar 21

3 Navigate: Navigate using the Position List 22

4 Navigate: Navigate using the Live SXI Viewer 22

5 Navigate: Navigate using the Stage Tab 22

Positions: Add Analysis Positions on Platen Viewer images 23

XPS: Setup Charge Neutralization for Acquisitions 23

Z-Align at a Position Before Acquisitions 24

Presputter at a Position Before Acquisitions 24

XPS Survey: Acquire Data 25

XPS Survey: Acquire More 27

XPS Survey: Data Reduction in SmartSoft-VersaProbe 28

XPS Survey: Data Reduction in MultiPak 28

Multiplex: Acquire Data 29

XPS Multiplex: Acquire More 32

XPS Multiplex: Data Reduction in SmartSoft-VersaProbe 33

XPS Multiplex: Data Reduction in MultiPak 34

XPS Test Acquire 36

Queue Acquisitions Overview 38

Queue: Open User-1 Acquisition Queue .....

Queue: Open and Load a Setting .....
Queue: Clear the User-1 Acquisition Queue .....
Queue: Add to the User-1 Acquisition Queue .....
Queue: Edit (Add, Delete, Change Order) 39
Queue: Edit the Queue [During an Acquisition] .....
Queue: Stop the Current Job and Continue at the Next Job 40
Queue: Stop the Queue Completely 40
Queue: Abort the Acquisition and Continue at the Next Position 41
Queue: Abort the Current Job and Continue at the Next Job 41
Queue: Abort the Queue Completely 41

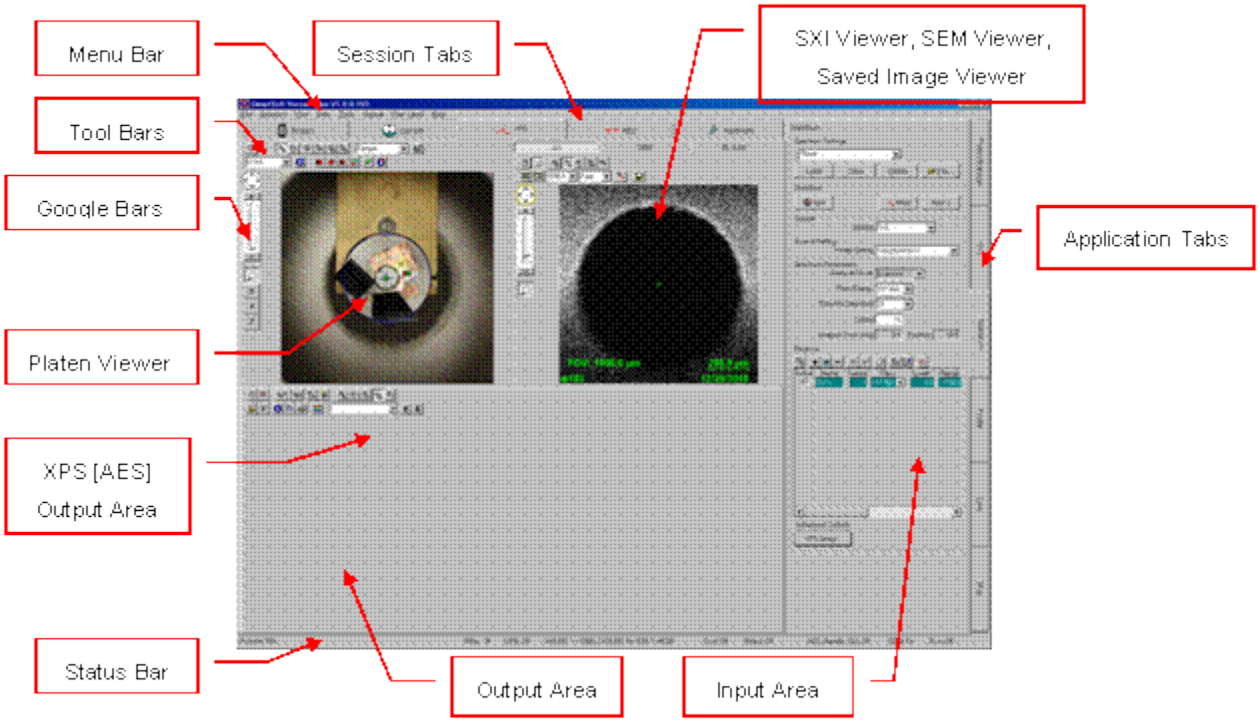
Some Analysis Considerations **46**

Introduction to SmartSoft-VersaProbe

SmartSoft-VersaProbe is a Windows-based interface with five primary "**Sessions**" devoted to different tasks:

- **System**, for loading and unloading samples or platens;
  This section also includes system control including vacuum and bake functionality;
- **Sample**, for locating and selecting analysis positions and **SXI** imaging;
- **XPS**, for acquiring **XPS** surveys, depth profiles, angle resolved profiles, line scans, and maps;
- **Hardware**, for Xray-Gun setup, analyzer and detector optimization, depth profile and general sample sputtering and charge neutralization.

SmartSoft-VersaProbe Operator Interface



SmartSoft-VersaProbe software's user interface consists of major interactive areas; the **Session** tabs [each consisting of an **Output** area and **Input** area], the **Menu** bar and the **Status** bar. Flow across the user interface is from left-right across the **Sessions** and top-bottom down the **Application** tabs. Each**Session** will reveal corresponding viewers in the **Output** area and unique **Application** tabs in the **Input** area.

Session Tabs

There is a dedicated section of the manual for each **Session** with detailed description of each **Session's Output** and **Input** areas. Each section will contain user tasks related to this **Session**. **Sessions** can be accessed across the top of the **SmartSoft-VersaProbe** window or through the **Menu** bar.

Output Areas

The **Output** area consists of the viewers listed below with the corresponding **Sessions** that use these viewers [refer to the corresponding "**Session**" section of the manual for detailed information regarding each viewer]:

Camera Viewer	**System**
Chamber Viewer	**System**
Vacuum Viewer	**System**
Platen Viewer	**Sample**
SXI Live Viewer	**Sample**
SEM Live Viewer	**Sample**
Saved Image Viewer	**Sample**
Position List Table	**Sample**
Spectral Viewer	**XPS [AES]**
Profile Viewer [Line]	**XPS [AES]**
Image Viewer [Map]	**XPS [AES]**

Tile/Fullscreen:

Each viewer in the **Output** area typically can be toggled between a tiled viewer and a fullscreen viewer. Use the **Tile/Fullscreen** icon in the upper left corner of each viewer for this operation.

Toolbar:

Each viewer contains a **Toolbar** area across the top that either will launch a unique task or define modes for other interactive operations. These **Toolbars** are discussed in each "**Session**" section of the manual.

Tooltip:

Hover the cursor over icons [without clicking the icon] to display a **Tooltip.** These **Tooltips** reveal either names of icons or information related to that icon.

Context:

Right-clicking within a viewer will reveal **Context** menus giving access to other operations or **Properties** windows.

Input Areas [Application Tabs]

Each **Session** contains its own unique set of **Application** tabs providing control parameters related to the tasks. The **Platen Manager Application** tab is involved with most **Sessions** and will be located at the top of each **Input** area. The **Platen Manager** is discussed in detail in its own section of the manual. The remaining **Application** tabs are described in detail in the corresponding "**Session**" sections of the manual.

Each **Application Tab** will provide access to commonly used parameters. Other less frequently used parameters or parameters that define default modes of operation can be found in the **Advanced Properties** window. A button is available at the bottom of each **Application** tab to open these windows.

Editing Values in Entry Fields:

Clicking on a field will "insert the cursor" and double-clicking on a field will "select the value in the field" allowing the user to perform one of the following editing techniques.

*Note: hardware will change when **Return** is selected with the following*

- Insert numbers to the current value and hit **Return**
- Replace the value and hit **Return**

Note: hardware will change instantly with the following

- Slew/step the value with the mouse wheel
- Slew/step the value with the keyboard up/down arrows
- SPECIAL case paired fields such as **X,Y** can be slewed/stepped using the left-right keyboard arrows for the 1st pair and using the up/down keyboard arrows for the 2nd pair.

Toolbar:

Some **Application** tabs contain a **Toolbar** area across the top that either will launch a unique task or define modes for other interactive operations. These **Toolbars** are discussed in each "**Session**" section of the manual.

Tooltip:

Hover the cursor over icons [without clicking the icon] to display a **Tooltip,** revealing either names of icons or information related to that icon.

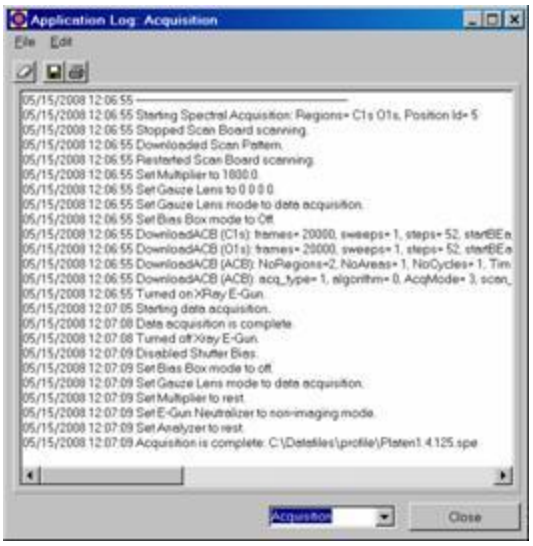
Hover the cursor over entry fields[without clicking the entry field] to display a **Tooltip,** revealing parameter ranges for that field.

Context:

Right-clicking within an **Application Tab** will reveal **Context** menus giving access to **Diagnostics** windows and **Application Log** windows.

The **Diagnostics** windows provide a further level of control and/or hardware read-backs typically for use by service engineers.

The **Application Log** windows provide a history of events that are related to each **Application**. A file will be saved during the current session of **SmartSoft-VersaProbe** and four backup copies saved during the last four sessions of running **SmartSoft-VersaProbe**. All high-level-tasks listed in the**Application Logs** will be separated by a dashed line. The combo box at the bottom of the window will allow selection of other **Logs**.



The **File** and **Edit** menus provide typical MS editing capabilities related to the text in the log. The combo box on the bottom will provide access to other **Application Logs**. The window can be minimized or closed.

Acquisition
This tab will record all pre-acquisition, acquisition and post-acquisition events

AutoTool
This tab will record all Queue related events

E-Neut
This tab will record any related hardware errors

GCIB
This tab will record any related hardware errors

Image Registration
This tab will record of Image Registration progress and results

Intro Camera
This tab will record any related hardware errors

Ion Gun
This tab will record any related hardware errors

Platen
This tab will record all platen transfer events

SEM
This tab will record all SEM process events

Stage
This tab will record all stage, arm, anode and monochromator motor events

Status Line
Vacuum

This tab will record all vacuum events including subtask operations and conditional operations

X-Ray

This tab will record emission status and any related hardware errors

Z-Align

This tab will record all Z-Align steps and results

Status Bar

The **Status Bar** is located below the **Input/Output** area. A yellow box will appear around the **Stage** area when the stage is in motion. . A red box will appear around the **Gun** areas when a communication error has occurred with that device. The **Status Bar** provides real-time display of the following fields.

- Last *acquisition, vacuum, platen or stage* control status
- Current **X**,**Y**,**Z**,**R**,**T** positions
- **Xray-Electron** gun state (***Park***, ***On***, ***Off***)
- **E-Neut** state *(**Neutralize**, **Standby**, **Off**) (**Auto** Neutralization state)*
- **Ion Gun** state *(**Neutralize**, **Sputter**, **Blank**, **Standby**, **Off**) (**Auto** Neutralization state)*

The **Status Bar** also provides real-time display for the following options.

- **AES Gun** state *(**On**, **Standby**, **Off**)*
- **UPS** state *(**On**, **Off**)*
- **Dual Anode** state *(**On**, **Off**)*
- **C60 20kV Ion Gun** state *(**Sputter**, **Standby**, **Off**)*
- **C60 10kV Ion Gun** state *(**Sputter**, **Standby**, **Off**)*
- **GCIB** state *(**Sputter**, **Blank**, **Off**)*

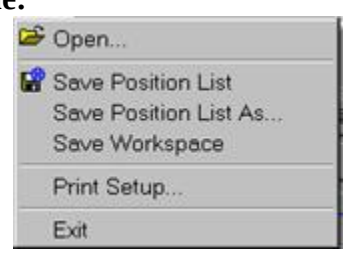
Context:

Right-clicking within the "gun" zones of the **Status Bar** will reveal **Context** menus giving access to re-initialization operations, states of each gun and Auto Neutralization states.

Menu Bar

The following functions are included in the menu bar:

File:



Open…:

This provides an **Open** dialog box allowing the user to open a datafile.

Save Position List and **Save Position List As...:**

The **Position List Table** is automatically saved when the "**current**" platen is changed or when **SmartSoft-VersaProbe** is exited. These provide an option to save this list at any time.

Save Workspace:

The **Platen Manager** is automatically saved when the "**current**" platen is changed or when **SmartSoft-VersaProbe** is exited. These provide an option to save this at any time.

Print Setup...:

This provides a **Print** dialog box allowing the user to define the default printer for all **SmartSoft-VersaProbe** print operations.

Exit:

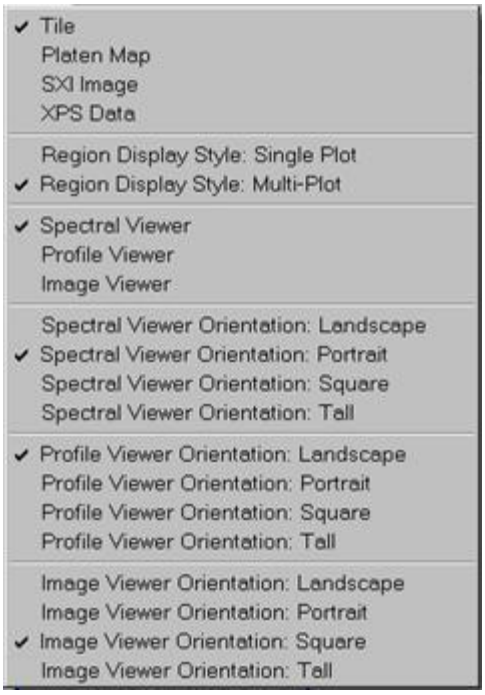
Selecting this will safely shut-down the hardware and exit **SmartSoft-VersaProbe**.

Sessions:

This reveals an extended pull-down list providing access to any **Output Viewer**; **Application Tab**; and **Properties Windows** within each **Session**.

Views:

This reveals an extended pull-down list providing access to **Output Viewers** and **Viewer Properties**; customized as a function of which **Session** is currently open**.**



Data:

This reveals an extended pull-down list providing the ability to close data files within the three data viewers**.**

Additional selections include:

Data>Spectrum>Smooth…:

Smooth Properties provides options related to the **Smooth** tool in the **XPS>Spectral Viewer**. Changes will be reflected in the **Smooth** icon label in the **XPS>Spectral Viewer** and in future smooth operations performed.

Data>Spectrum>Deconvolution…:

Target Pass Energy (eV): [combo]

The region data in the **Spectral Viewer** will be deconvolved to this **Target Pass Energy**.

[Typically 11.75]

Peak to Noise Limit:

The minimum **Peak To Noise** ratio required to perform the deconvolution process.

[Typically set to 100]

Iterations:

The number of **Iterations** performed in the deconvolution process.

[Typically set to 50]

*[Choose **Iterations * Rate ~ 1** to optimize]*

Rate:

The **Rate** of approach to the solution in the deconvolution process. A larger number increases the magnitude of the change in the estimated spectrum (at each iteration).
[Typically set to 0.02]
*[Choose **Iterations * Rate ~ 1** to optimize]*

Stop Criteria:
The relative improvement in the resolution (for each iteration) is examined. When the resolution changes are less than this value; the iterations are stopped.
[Typically set to 1]

Deconvolve: [button]
Click [depress] or click [un-depress] the **Deconvolve** button to "use" or "not use" this process on subsequent **Spectral Viewer** displays of region data.

Data>Image>Properties:

Opening this **Image Properties** while an image is loaded in the **XPS>Image Viewer** will provide information related to that data file.

Tools:

System Colors...:
This opens the **Color Configuration** window providing the ability to select color options for the **Data Viewers** and the **Platen Viewer**. Editing these selections will take immediate affect.

Background:

This provides a selection for the **Data Viewer's** background color.

Curve 1-20:

This provides a selection for the **Data Viewer's** data color.

Platen Colors: Platen Caption:

This provides a selection for the **Platen Viewer's** annotation color.

Position Colors: Position Active:

This provides a selection for the **Platen Viewer's** Active Position color.

Position Colors: Position Inactive:

This provides a selection for the **Platen Viewer's** Inactive Position color.

Queue:

Clear Queue:

This will clear all **Queue Jobs** from the **Queue**

Queue:

This will open the **Queue** window

Help

SmartSoft-VersaProbe Help:

Selecting this will provide access to the .htm **User Manual**. The home page for this manual provides icon links to the "**How To**" Procedures, **Introduction and Overview** section; five **Session** sections; the **Platen Manager** section and two support documents.

About SmartSoft- VersaProbe …:

Selecting this will open the **About SmartSoft-VersaProbe** window.

Operation Overview

Typical operation is performed within the **System**, **Sample**, **XPS,** and/or **AES** Sessions and does not require the user to startup or setup hardware. Most of this is automated; leaving the user with the following tasks:

- Sample introduction, platen manager setup and capturing photos performed in the **System Session**
- Analysis area definition, SXI imaging and Z-Alignment performed in the **SAMPLE Session**
- Acquisition setup, data acquisitions, data review and Queue operations performed in the **XPS Session** and **AES Session**

Navigating on the Platen is accomplished using the **Platen Viewer** and **SXI Viewer** on the **Sample Session**. See this section of the manual for detailed description of the following:

- Coarse **XY** moves are accomplished dragging the **Stage Marker** within the **Platen Viewer**.
- Fine **XY** moves are accomplished with the **Point-n-Click** feature in the **SXI Viewer**.
- **Z-Height** adjustment is accomplished with the **Z-Align** tool in the **SXI Viewer** and with the pre-acquisition **Z-Align** option in the **Acquisition Properties**.
- **Compucentric Rotation** is accomplished with the **Rotate Horizontal** tool in the **SXI Viewer**.
- The "**Stage**" **Application** is only needed for manual **Tilt** changes.

Vacuum tasks such as **Pump/Vent Intro** or **Pump/Vent Chamber** pumping are performed in the **System**>**Valves** tabs:

Vacuum tasks such as **sublimation** or **Baking** are performed in **Watcher**:

Gun performance tuning or creating new gun settings are performed in the **Hardware** tabs:

Analyzer/Detector performance tuning is performed in the **Hardware** tabs:

Automated setup is found in the following locations:

Automated **Intro** Photo	**System**>**SampleTransfer**>**Properties**
Automated **Sputter** setup	**XPS**>**Profile**>**SputterTable** **XPS**>"**Application**">**AcquisitionProperties**>**Setup**>**Pre-Sputter** **XPS**>"**Application**">**AcquisitionProperties**>**Setup**>**Pre-Sputter**
Automated **X-Ray** setup during **SXI** & **SXI Z-Align**	**XPS**>**SXI**>**SXIProperties**>**Setup** **XPS**>**OutputArea**>**SXIViewer**>**ContextMenu**>**Setup**
Automated **X-Ray** setup during Acquisition	**XPS**>**AcquisitionApplicationTabs**
Automated **E-NeutNeutralization** setup	**XPS**>"**Application**">**AcquisitionProperties**>**Setup** **XPS**>**SXI**>**SXIProperties**>**Setup** **XPS**>**SXIViewer**>**Toolbar**>**Context**>**Properties**>**Setup** **System**>**OutputArea**>**ChamberViewer**>**E-Neut** **StatusBar**>**E-Neut**>**ContextMenu**
Automated **Ion GunNeutralization** setup	**XPS**>"**Application**">**AcquisitionProperties**>**Setup** **AES**>"**Application**">**AcquisitionProperties**>**Setup** **XPS**>**SXI**>**SXIProperties**>**Setup** **XPS**>**SXIViewer**>**Toolbar**>**Context**>**Properties**>**Setup** **System**>**OutputArea**>**ChamberViewer**>**IonGun** **StatusBar**>**IonGun**>**ContextMenu**
Automated **Analyzer**settings [**SXI** ,**Z-Align** & **Detector**]	**Hardware**>**Analyzer**>**Properties**>**Lens**
Automated **Ion Gun Differential Pumping** &**Argon** Gas Admission	**Hardware**>**IonGun**>**IonGun** **Hardware**>**IonGun**>**IonGun**>**Properties**>**Service**
Automated **X-Ray Beam Parking**	**XPS**>"**Application**">**AcquisitionProperties**>**Setup** **Hardware**>**Source**>**X-Ray**>**Properties**>**Service**
Automated	**AES**>"**Application**">**AcquisitionProperties**>**Setup**

Beam Current Reading Before **AES** Acquisition	

Settings

Many of the **Application** tabs include a "settings" area. A setting is a group of parameter values related to the displayed application tab. The settings area allows the operator to save and retrieve parameters as settings for future use.

NOTE: Not all related parameters are stored when a setting is created. Some parameters will have the same value regardless of the setting, but can be changed individually by the operator.

Special Settings:

The **PREVIOUS** setting is found in all the setting areas, and will load the setting that was active prior to the last system shutdown. The **PREVIOUS** setting is updated whenever the operator moves to a new session tab or exits **SmartSoft-VersaProbe**.

The **INITIAL** setting is found in settings areas that save hardware parameters. The **INITIAL** setting is automatically selected when the system is first started as it sets the system hardware to safe default parameters.

Load Settings:

To select a setting, use the text box to highlight the desired setting, then click **Load**.
*Note: Selecting and loading a setting with the **E-Neut**, **Ion Gun**, **C60 20kV Ion Gun, C60 10kV Ion Gun** or **GCIB** in an OFF state; allows the user to view parameters without voltages being applied to that gun.*

Alternatively, you can restore a setting that was used during the acquisition of a specific data file. Data files (**Surveys**, **SXI** images, **Maps**, etc.) generated in **SmartSoft-VersaProbe** include information on the settings used during their acquisition. Click the **File** button in the settings area and then select the desired data file. The file itself will not open, but the settings stored with it will be loaded into the active **Application Tab**.

Save Settings:

To create a new setting, set the parameters as desired. Type a setting name in the text box and click **Save**.

Delete Settings:

To delete a setting, select it and then click **Delete**.

Note: some settings are **RESERVED** and cannot be deleted.

There are several types of XPS data you can collect:

1 Survey

A **Survey** is one spectrum acquired from a quick, high-sensitivity scan of a wide energy range (typically 0 to 1100 eV in 1 eV steps) to survey the elements present at a point on the sample or over an area. In *point* analysis, a stationary x-ray beam is positioned on a specific point. In *area* analysis, the beam is rapidly scanned, or rastered, over an area or multiple areas of the surface.

2 Multiplex

A **Multiplex** is a set of spectra acquired from a series of high-resolution surveys of narrow binding energy ranges (typically 4-31 eV wide in 0.25-1 eV steps). This type of acquisition yields great sensitivity and spectral detail in a short analysis time, because only selected energy regions expected to contain **XPS**peaks of interest are scanned.

The more commonly used multiplex acquisition routines are the line and profile.

3 Depth Profiles

An **XPS Depth Profile** provides compositional data as a function of depth. A sputter depth profile consists of a series of spectra that have been collected at different depths as material was removed from the sample. An **XPS** depth profile is generally made by alternating sputtering with data acquisition. The purpose of a depth profile is usually to verify or determine the construction of a thin film structure. Other applications include studying inter-diffusion of layers, looking for impurities in layers or at interfaces, and general characterization of surfaces and thin films. Depth profile data can be used to extract chemical state information as well as atomic concentration data.

4 Angle Profiles

An **XPS Angle Profile** provides compositional data as a function of depth. **Angle-Resolved** (angle-dependent) profiles are used to probe the near surface region of a sample in a non-destructive manner. **Angle Profiles** are possible, because the input lens of the analyzer **(SCA)** can operate with a small acceptance angle, and it is possible to tilt the sample in front of the analyzer. The analysis depth at a given angle is defined by the equation:

$$d = \alpha \sin\theta$$

where ***d*** is the effective analysis depth,
α is inelastic mean free path (escape depth) and
θ is the angle between the sample surface and the analyzer input lens.

Angle Profiles are used typically to measure the thickness or explore the chemistry of very thin layers. Applications include the study of chemically modified surfaces, lubrication, cleaning processes, and such.

5 Maps

A **Map** is a set of intensity-value arrays acquired over the area of the sample to show the surface distribution of specific elements. Each array of intensity values corresponds to an element, and each value in the array corresponds to a point in the map area. The intensity value is obtained by measuring the intensity at a specific **XPS** peak energy, then subtracting the background intensity.

Acquiring map data for every element identified in the survey will completely characterize the distribution of elements in the analysis area. These maps are then compared to the **SXI** image or platen view.

6 Line Scans

An **XPS Line** scan is acquisition of data along a single line across a sample. The intensity value is obtained by measuring the intensity at a specific **XPS** peak energy, then subtracting the background intensity.

Here we provide and overview on first two of XPS data types: **Survey** and **Multiplex.** We refer to **VersaProbeII** manulas if you need other data type to collect, but any of these starts, as described below, from **New Platen** creation before introducing your sample into analitical (main) chamber.

Create New Platen

Creating a Platen is the first step of data analysis. It should be performed **before** placing the platen(sample) in the intro chamber. Refer to the **Platen Manager** section for information related to the **Create Platen** window within the **System** session.

1. Click the **Create Platen** button on the **System>Platen Manager>Platen** tab.
   Or Right-click on the **Intro Station** within the **Intro>SystemGlyph** and select **Create Platen**
   *Note: if at anytime you would like to cancel the **Create Platen** operation, simply select the **Cancel** button and the dialog box will close. No platen will be created.*

2. Enter a **New Platen Name**

3. Click **OK**
   *The **Create Platen** window will automatically close and a new platen(sample) will now appear at the **Intro Station** within the **System** Session.*

4. View the **System>PlatenManager>Platen** application window.

5. Select the platen(sample) **Type** [**25mm, 60mm, AR**]

6. *Optional: Select an image overlay if a new **Intro** station **Navigation Photo** will not be captured for this sample.*
   *Templates are found in the **C:\SmartSoft-VersaProbe\Platen** folder*
   *Other **Intro** station **Navigation Photos** are found in **C:\Datafiles** folders*

7. Use the default **Max Z Height** for this type
   or enter a lower value if your samples are raised above the sample holder surface.

8. Select a **Directory** where data will be saved for this sample.
   If a new folder is desired, then click on the root directory and add a **\foldername** to the end of the root directory path in the **Directory Name**: field

9. Click the **System>PlatenManager>Platen>AdvancedControl Properties** button to edit the Filenaming format or the Comment format

10. Select the desired automated **Filenames** and **Comments** options in the **Filename** tab.

11. Enter desired information in the **Contact Info** tab.

12. Click the **Close** button when finished with the **Platen Manager Properties** window

Attaching/Removing a Platen(Sample) at the Intro Station

Warning: This task is to be performed during **Loading** and **Unloading** a platen(sample) at the **Intro** station and should be performed **when** the user is prompted to**.**

Installing a Platen(Sample)

1. Lift the Intro lid.

2. Pick up the platen(sample) with a gloved hand or intro tool. The platen(sample) should be oriented with the alignment marker towards the **Intro Rod**.

3. Hold the platen(sample) in front of the intro fork and slide the platen(sample) onto the end of the intro fork.

Removing a Platen(Sample)

1. Lift the Intro lid.

2. Grasp the platen(sample) with a gloved hand or intro tool.

3. Slide the platen(sample) off the end of the intro fork and remove from the Intro chamber.

4. When the platen(sample) is removed from the **Intro**, place the lid on the intro chamber to keep this area clean.

Move Platen(Sample) into Analytical Chamber

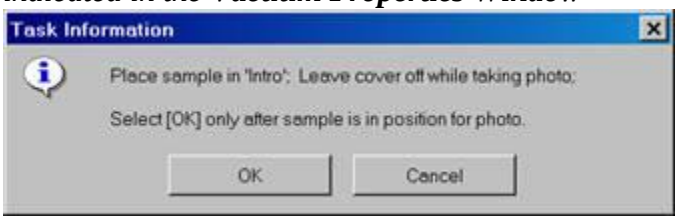
The new platen(sample) should be at the **Intro** station before performing this task.

1. Drag-n-Drop the platen(sample) from the **Intro** Station to the **Stage** Station.

Or click the

Intro Sample [button] on the **System>SampleTransfer** application window.

*The **Intro** chamber will be automatically backfilled to the "atmospheric pressure" indicated in the **Vacuum Properties Window***



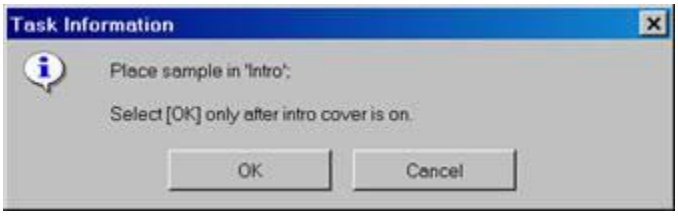
2. Place the platen(sample) on the intro fork

3. With the **Intro Camera** option:

    a. Leave the lid off

4. Click **Ok**.

*The **Intro** station **Navigation Photo** will automatically be captured and saved*



5. Place the lid on the intro chamber

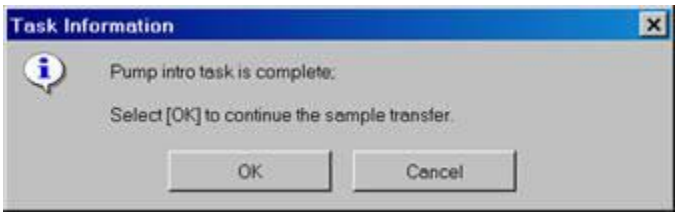
6. Click **Ok**

*The **Intro** chamber will automatically be pumped-down to the "intro pressure setpoint" defined by **Watcher.***

*TheIntro procedure will then wait for the pressure indicated in the **System>SampleTransfer>SampleTransferProperties** window to be met if the **Cold Cathode** is installed on the intro chamber .*

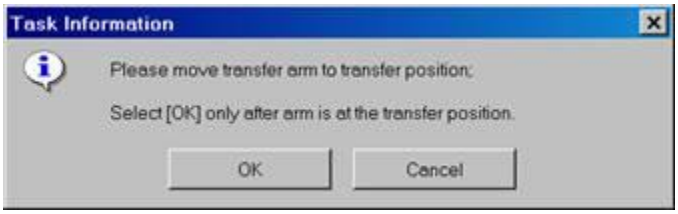
*Another delay will occur if the **Additional Delay (min)** box is checked.*

Then the following prompt will appear



7. Wait if more pumping time is still desired and then click **Ok** otherwise click **OK**

*The stage will automatically be moved to the transfer position, the **Intro Gate Valve V1** will automatically be opened and the following prompt will appear.*



8. Slide the intro rod to engage the platen(sample) with the stage and click **Ok**

*The stage will automatically raise the Z axis by the amount indicated in the **System>SampleTransfer>SampleTransferProperties** window to engage the platen(sample) with the stage and the following prompt will appear.*

9. Slide the intro rod out and click **Ok**

The Intro Gate Valve V1 will automatically close when the intro rod is completely retracted.

The Stage will move to a centered orientation with the analyzer

Once your sample is in analytical chamber you can navigate.

Navigate: Overview

Stage moves within the **Platen Viewer** will **not** always lower the **Z** axis or level the **T** axis
Caution should be taken if a platen(sample) is on the **Stage** with raised samples.

Stage moves within the **Sample>Stage** tab using the **Drive All** button will offer this protection if the radio button for **Protection Drive All** is set to **On** within **Sample>Stage>StageProperties>Properties>StageProtection.**

Stage moves during an acquisition will offer this protection if the radio button for **Protection Acquisition** is set to **On** within **Sample>Stage>StageProperties>Properties>StageProtection.**

The following are preferred and optimized modes of Stage axis navigation:

- Lateral navigation should be performed using the **Stage Marker** within the **Platen Viewer** and **Point-n-Click** operation with the **SXI Live Viewer**.
- **Z** adjustments should be performed using the **Z-Align** operation.
- **Rotation** adjustments should be performed using the **Rotate Horizontal** icon within the **SXI Live Viewer**.

The following navigation methods are available:

- Multiple axis moves can be performed by editing the **X**,**Y**,**Z**,**R**,**T** target fields followed by a **Drive All** using the **Sample>Stage** tab
- **Z, R** or **T** adjustments can be performed by editing the corresponding target field followed by a **Drive Z, Drive R** or **Drive T** using the **Sample>Stage** tab.

*See the **Sample** session discussion of the manual for further information*

1 Navigate: Navigate using the Platen Viewer Images

The platen(sample) should be on the Stage.

Method 1-move stage marker location

- Grab the **Stage Marker** and drag-n-drop to a new location on the image

Method 2-move stage to anywhere in the image

- Right-click at a desired location on the image and select **Drive to Click** to move to this location

Method 3-move stage to a Position in the image

1. Right-click on the desired **Position** on the image and select **Drive To Position**

or

1. Left-click on the desired **Position** on the image
2. Click the **Drive To Position** icon in the **Platen Viewer** toolbar

2 Navigate: Navigate using the Platen Viewer Google Bar

The platen(sample) should be on the Stage.

1. Select the **X**, **Y** or **Z** button within the **Platen Viewer Google** bar
2. Grab the **Slidebar** and drag-n-drop up/down to a new axis position
3. Left-click the **Increment Up** or **Increment Down** buttons on the slidebar to step the axis
4. Use the Up/Down keyboard arrows to step the axis

5. Return the button selection to **Zoom**

3 Navigate: Navigate using the Position List

The platen(sample) should be on the Stage.

Method 1

1. Right-click within the **ID** column on a position in the **Position List** and select **Drive to Position** to move to this position

Method 2-Use the "Drive To" icon in the **Position List** toolbar

1. L-Click on the desired **Position** in the **Position List** to select that position
2. Click the **Drive To Position** icon in the **Position List** toolbar

4 Navigate: Navigate using the Live SXI Viewer

The platen(sample) should be on the Stage.

Point-n-click Method

1. Start **SXI** imaging
2. Select the **Zoom** icon in the **Live SXI Viewer** toolbar
3. L-Click within the SXI image to move the stage XY to that location
   Drag-n-drop within the SXI image will also restart the SXI imaging with a new field-of-view defined by the drag-n-drop box and will move the stage XY to align to the center of the drag-n-drop box

Rotate Horizontal Method

1. Start SXI imaging
2. Select the **Rotate Horizontal** icon in the **Live SXI Viewer** toolbar
3. L-Click within the SXI image to define the new "centered" location
   Drag-n-drop a line within the SXI image defining a desired horizontal orientation
   Note: L-click only will perform a point-n-click operation

5 Navigate: Navigate using the Stage Tab

The platen(sample) should be on the Stage.

Single Axis Moves

1. Edit the **Target Position** of a single axis
2. Click the **Drive** button for that axis

or

1. Select the **Target Position** field for a single axis
2. use the mouse wheel to edit and instantaneously move that axis

Two to Five axis moves

1. Edit the **Target Position**(s) of any axis of interest
2. Click the **Drive All** button to move to the new 5 axis position

To be able collect data you need to choose the position on your sample.

Positions: Add Analysis Positions on Platen Viewer images

The platen(sample) should be on the Stage. Analysis positions added to a **Navigation Drawing** or to an **Intro** station **Navigation Photo** will be assigned an analysis area at the time of they are created.

1. Create a new analysis position on the **Navigation Drawing** or **Intro** station **Navigation Photo** in the **Platen Viewer**

   Choose from the following options:

   - Right-click at the desired position location on the image and select **Create** [**Point**]
   - Click on the **Define Point** icon at the top of the **Platen Viewer** L-Click at the desired position location on the **image**
     *Note: Multiple positions can be created while one of the "**Define**" icons is selected*
     *Note: Return control to the **Zoom** icon in the **Platen Viewer** toolbar when this task is completed*

3. Create a new analysis position at the "Current Stage Position"

   - With the **Stage Marker** on the **Platen Viewer** at the desired location; Right-click over the **Stage Marker** and select **Create Stage** [**Point, Area, Line, HP**]

*Note: A position will be added to the **Platen Viewer** and to the **Position List Table***

Refer to **VersaProbeII** manuals if you need to analyze area with preset dimensions such as **Area**, **Line** or **HP.**

XPS: Setup Charge Neutralization for Acquisitions

Charge neutralization will be used for any new acquisition if the **Auto Neutralization** is setup in the **XPS>Application>AcquisitionProperties>Setup** window.

Method 1

1. Open the **XPS>Application>AcquisitionProperties>Setup** window.
2. Select **Auto[A]** for **E-Neut Auto Neutralization** and/or **Ion Gun Auto Neutralization**
3. Select desired **E-Neut Setting** from the corresponding combo box and/or the desired **Ion Gun Setting** from the corresponding combo box

4. Select **Source Tolerance Required** (**Enabled)** for the **E-Neut** if the acquisition starting should be dependant on whether the E-Neut's emission is in tolerance
5. Close the **XPS>Application>AcquisitionProperties>Setup** window

*All subsequent acquisitions will perform an automated neutralization of the platen(sample) until the **Auto Neutralization** is set to **Off***

Method 2

1. Right-click on the **E-Neut Status** box at the bottom of **SmartSoft**

2. Select **E-Neut Auto Neutralization: Auto[A]**
3. Right-click on the **Ion Gun Status** box at the bottom of **SmartSoft**

4. Select **Ion Gun Auto Neutralization: Auto[A]**

*All subsequent acquisitions will perform an automated neutralization of the platen(sample) until the **Auto Neutralization** is set to **Off***

1 XPS: Z-Align at a Position Before Acquisitions

Z-Align can be performed automatically at each position in the **Position List** before each acquisition. Each position's **Z** height will be updated with the Z-Align's calculated height.

1. Open the **XPS>Application>AcquisitionProperties>Setup** window.
2. Select **Z-Align Enabled** in the **Z-Align Before Acquire** section
3. Select the desired **X-Ray Setting** and desired **X-Ray Setting (High Power)** from the corresponding combo box
4. Close the **XPS>Application>AcquisitionProperties>Setup** window

*All subsequent acquisitions will perform an automated Z-Align of the platen(sample) until the **Auto Z-Align** is set to **Disabled***

2 XPS: Presputter at a Position Before Acquisitions

Presputter can be performed automatically or manually with the **Ar Ion Gun** or with the optional **C60** or **GCIB** ion guns.

Automated Method

1. Open the **XPS>Application>AcquisitionProperties>Setup** window.
2. Select **Presputter Enabled** in the **Presputter Before Acquire** section
3. Select the desired **Ion Gun Type** from the combo box
4. Select the desired **Ion Gun Setting** from the **Presputter Setting** combo box
5. Set **Zalar** to the desired **Enabled** or **Disabled** setting
6. Select the desired **Presputter Time(min)**
   *Note: The **Presputter Depth Angstrom** field will indicate the corresponding sputter depth if this Ion Gun setting has a calibrated sputter rate parameter*
7. Close the **XPS>Application>AcquisitionProperties>Setup** window.

*All subsequent acquisitions will perform an automated presputter of the platen(sample) until the **Presputter** is set to **Disabled***

Manual Method

1. Move the platen(sample) to align the area to be sputtered and perform a **SXI-Z-Align**
2. Open the **Hardware>"Desired"Ion Gun** window.
3. Load a desired sputter setting
4. Select the desired **Sputter Time(min)** in the **Timed Sputter** section
   *Note: The **Sputter Depth Angstrom** field will indicate the corresponding sputter depth if this Ion Gun setting has a calibrated sputter rate parameter*
5. Click the **Timed Sputter** button or the **Timed Sputter w/Zalar** button to start.
   *Note: Sputter time will be displayed within the **Timed Sputter** button.*
   Note: Click this button to abort the timed sputter

XPS Survey: Acquire Data

An **XPS Survey** is a quick, high-sensitivity acquisition of **XPS** data over a wide energy range. An **XPS Survey** scan is used to identify which elements are present in the analysis area. This is typically the first step in any sample analysis, providing an overview of elements present that will guide the design of the remaining analysis session.

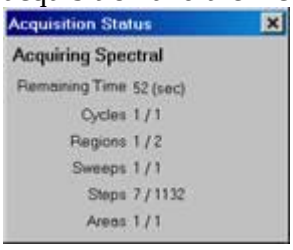
An **XPS Survey** involves acquisition of one spectrum from a quick, high-sensitivity scan of a wide energy range (typically 1100 to 0 eV in 1 eV steps, at 20 milliseconds/point) to survey the elements present at a point or area on the sample. The highest **Pass Energy** (187.85eV) is used to maximize elemental sensitivity and not chemical sensitivity (energy resolution).

Another part of the analysis strategy for survey spectra is to use the largest diameter **X-Ray** probe that is possible, while confining the analysis to the feature of interest. With the Monochromatic -Ray source, **X-Ray** power increases linearly as the diameter of the **X-Ray** spot increases. Time is also a variable in the survey experiment. However, the quality of the spectrum increases as the square root of time, so it is very desirable to optimize the other parameters first.

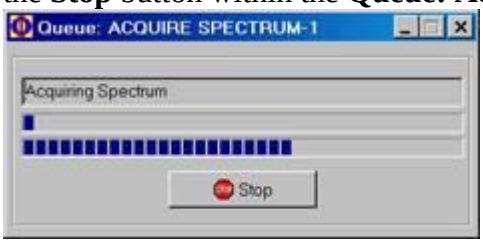
It is assumed that the platen(sample) is in the analysis chamber (**Intro Session**) and that analysis positions and analysis areas have been defined (**Sample Session**).

1. Select the desired **Active** analysis positions in the **Sample>Position List Table**.
2. Enable the **Auto Z Before Acquire** (**XPS>Properties>Setup**) if the analysis positions have not been previously aligned
3. Enable the **Automated Neutralization** (**XPS>Properties>Setup** or **Status Bar**) if the sample is insulating
4. Open the **XPS Spectrum Application** window.
5. *Optional use of **Spectrum Settings***
   a) *Select a setting from the **Spectrum Settings** combo box.*
   b) *Click the **Load** button.*
6. *Optional use of **Previous Acquisition** file **Spectrum Settings***
   a) *Click the **File…** box.*
   b) *Choose a **Spectrum** file with desired settings.*
   c) *Click **Open**. The **Spectrum Parameters** will update with the acquisition file settings.*
7. Select the **Source** type from the combo box.
8. Select an **X-Ray Setting** picking the largest diameter **X-Ray** probe that is possible, while confining the analysis to the feature of interest.
9. Set the **Pass Energy** to 117.4
10. **Add** the **Su1s Region** to the **Region List** and set the **Active** boxes so only this **Region** is **Active**
11. Use the recommended 20ms **Time Per Step (ms)**
12. Select the number of **Cycles** to provide a data collection time of 1-3 minutes [**Acquisition Tim (min)**] for larger diameter **X-Ray** spots or 5-15 minutes [**Acquisition Tim (min)**] for smaller diameter **X-Ray** spots.
    *For example, an acquisition range of 1100 to 0eV, 1 sweep, 117.4PassEnergy, 1 eV/step and 20 ms/point, set for 13 **Cycles**, will result in a data collection of ~10 minutes [**Acquisition Tim (min)**].*
13. Click the **Start** button in the **Spectrum** tab.

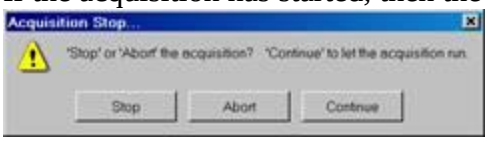
- The **Queue: Acquire Spectrum-1** box appears displaying progress of the job and the **Acquisition Status** box appears displaying progress of the acquisition and the **Remaining Time** in the acquisition.



- One file will be saved with multiple traces [one for each analysis area]
- The **Filename** will be:
  <filename>. <file number>.<platen name>. <position #>. <position list name>.spe
- The **SmartSoft-VersaProbe** data will be displayed in the **Spectral Viewer**. A **Su1s Region** will be displayed with a separate trace for multiple analysis areas.
- All **Active** analysis positions in the **Position List Table** that are assigned a valid analysis area for the current acquisition will be visited sequentially.
- To **Stop** or **Abort** the acquisition before all **Cycles** are completed, use the **Stop** button within the **Queue: Acquire Spectrum-1** window.

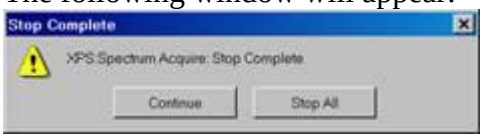


If the acquisition has started, then the following window will appear:



- **Stop** button terminates data acquisition at the completion of the current **Sweep** and saves the data file.
- **Abort** button terminates the acquisition immediately and eliminates the data file that was created.
- **Continue** button will return to the current acquisition

- The following window will appear:



  - **Continue** button will advance to the next **Position List** position of the current **Job** and repeat the acquisitions process.
  - **Stop All** button will advance to the end of the current **Job** and stop the acquisitions process.

XPS Survey: Acquire More

The platen(sample) must be at the same analysis position and no **XPS** parameters should have been changed. **Survey** should be the last acquisition that was **Stopped** or completed. Any **Inactive Regions** from the **Survey** acquisition will be removed from the **Region List** when the **More** acquisition is started. **Auto Neutralization** and **Beam Park** will be supported. **Pre-sputter** and **Z-Align** will not be repeated.

1. Click the **More** button in the **Spectrum** tab to add additional data to the **Stopped** or completed **Survey**.

2. Add more **Cycles** to increase the **Analysis Time**
   *Note: If the **Survey** was stopped; the current number of **Cycles** can be used to continue the **Survey***

3. Click the **Start** button

   - The original acquisition file will be preserved with its previous name.
   - One new file will be saved with multiple traces [one for each analysis area] The new **Filename** will be:
     <"previous name"><+1>. spe
   - Subsequent **More** acquisitions will preserve previous files; acquire new data and save a new file with the **Filename**:
     <"previous name"><+2>. spe

XPS Survey: Data Reduction in SmartSoft-VersaProbe

Refer to the **Multiplex** subsection for information on displaying data, smoothing data, identifying peaks, annotating peaks and copying/printing the depth profile using the toolbar buttons in the **XPS** output area.

XPS Survey: Data Reduction in MultiPak

After collecting the survey spectra, open the **MultiPak** software to reduce the data. The **MultiPak Spectrum** window and **Periodic Table** will appear. For detailed information about the use of **MultiPak**, refer to the ***MultiPak Software Manual***.

1. Click the **LastAcq** button (which will open the file most recently acquired) or click the **File** button on the upper tool bar to open the file of choice from a **File** selection menu.

   - *The filename is displayed in green, indicating that this data is "selected." Any **MultiPak** functions activated will be performed on this data.*
   - *The header (displayed only when **Header** in the **View** menu is checked) lists the file's data acquisition parameters (date of acquisition, beam voltage of acquisition, etc.).*
   - *The third line of the header will be updated with a processing history as data reduction routines are applied to the data.*

2. Click the **PeakID** button to identify and label the peaks in the spectrum. This will:

   - Perform an automatic charge correction based on the position of the C1s peak.
   - Search its database to identify and label the peaks.
   - Create a push button for each element in the **Region** bar at the bottom of the **Spectrum** window.

3. Verify the accuracy of the labels using your experience and the **Handbook of X-ray Photoelectron Spectroscopy**, which is available from **Physical Electronics**. If mistakes were made, you can correct them with one or more of the software tools available.

   - To remove a label, click on it and drag it outside the window using the left mouse button or turn **OFF** the element in the **MultiPak Periodic Table** by toggling it off with the left mouse button.

- To label a peak or element that was missed by the **Auto PeakID** routine, you can turn that element **ON** in the **Periodic Table** by clicking the button for it. This method will also create a pushbutton at the bottom of the **Spectrum** window for that new element.
- If you do not know the identity of an unlabelled peak, you can manually search through the database of labels.

a) Using the middle or right mouse button, click on an open area of the spectrum. This will display a cursor and list **ESCA** transitions that occur at the current cursor location.

b) While still holding down the mouse button, you can drag the cursor and search around the area of the unknown peak to see what elements occur in that energy range.
Note: If you use the right mouse button for this operation, releasing the mouse button places the current label on the display at the location of the cursor.
Note: If using the middle mouse button, when you release the mouse button, no labeling will take place.
***Note**: When labeling peaks in this manner, no buttons for the associated elements will be created at the bottom of the window. If you want to add buttons for elements that were not identified by **PeakID**, you must create them by selecting the element from the **Periodic Table**.*

4. Quantify Data in MultiPak. Quantification of ESCA spectra is most accurate when **Peak Area** measurements are used. In most situations, it is recommended that you use a **Shirley Background Subtraction** for making the area measurement.

a) To view or change the end points that will be used to define the background subtraction for a specific elemental peak, click the button at the bottom of the **Spectrum** window for that element and the software will automatically expand the region for that element's default peak and display the cursors that are used for background subtraction. If it is necessary to adjust the cursors, click and drag the cursor using the left mouse button. While the cursor is being moved, a line depicting the background will be displayed to assist you in positioning the cursor.

***Note**: The default peak is defined in the **Transition Editor** menu and, usually, it is the main or major elemental peak. If you want to change the default peak, you can do so in this menu.*

Multiplex: Acquire Data

A multiplex is a set of spectra from a series of high-resolution acquisitions of narrow energy ranges (typically 30 eV in 0.5 eV steps at 20 ms/point). This type of acquisition yields great sensitivity and spectral detail in a short analysis time because only selected energy regions expected to contain **XPS** peaks of interest are scanned. A multiplex acquisition is useful primarily when looking for trace elements. As many as 20 elements can be acquired in one multiplex.

The more commonly used multiplex acquisition routines are the window **Line** scan and **Depth Profile (**refer to VersaProbeII manuals). This subsection describes the basic **Multiplex** acquisition routine.

A narrow energy range allows you to optimize either spectral detail or sensitivity, while keeping the data collection time short.

To optimize spectral detail, a low **Pass Energy** is chosen (typically, 23.5 – 58.7eV) and the number of data points is increased (0.05 - 0.2**eV/step**). There is a temptation to select a very low **Pass Energy** and a very small step size to obtain the "best" data. In practice, it is necessary to make a compromise choice between the experiment time, **Pass Energy**, and data step size. For an efficient, high-energy resolution experiment, you should use as high of a **pass energy** as possible, while still resolving the spectral features of interest. You may also want to include the fewest number of data points possible while recording an accurate shape of the spectrum.

To optimize elemental sensitivity, a high **Pass Energy** should be chosen (typically, 93.9 to 117.4eV) and the number of data points is reduced (0.2 to 0.4**eV/step**). Since sensitivity increases almost linearly with **Pass Energy**, it is most advantageous to use as high of a **Pass Energy** as possible.

Use this customization capability to optimize an experiment. For example, if you would like high-resolution spectra of a few major elements, you can select a **Pass Energy** of 23.5eV for those elements. For minor elements where the interest is sensitivity and quantification, you can select a **Pass Energy** of 117.4eV. The **MultiPak** data reduction software is capable of reducing data files that have mixed **Pass Energy** and providing correct quantitative results.

The required data acquisition time will depend on:

- The quantity of the element present in the analysis area
- The **Pass Energy**
- The **X-Ray** spot size.

The data acquisition time may vary from a few minutes per element to 30 minutes per element. The **Analysis Time (min)** displayed in the **Spectrum** menu is the total time for all elements selected.

The following procedure assumes that the **Multiplex** acquisition is following an initial **Survey** acquisition, and that analysis areas or points have been defined. Otherwise, verify that the platen(sample) is in the analysis chamber (**Intro Session**) and that analysis positions and analysis areas have been defined (**Sample Session**).

1. Select the desired **Active** analysis positions in the **Sample>Position List Table**.
2. Enable the **Auto Z Before Acquire** (**XPS>Properties>Setup**) if the analysis positions have not been previously aligned
3. Enable the **Automated Neutralization** (**XPS>Properties>Setup** or **Status Bar**) if the sample is insulating
4. Open the **XPS Spectrum Application** window.
5. *Optional use of **Spectrum Settings***
   *a) Select a setting from the **Spectrum Settings** combo box.*
   *b) Click the **Load** button.*

6. *Optional use of **Previous Acquisition** file **Spectrum Settings***

   *a) Click the **File…** box.*

   *b) Choose a **Spectrum** file with desired settings.*

   *c) Click **Open**. The **Spectrum Parameters** will update with the acquisition file settings.*

7. Select the **Source** type from the combo box.

8. Select an **X-Ray Setting** picking the largest diameter **X-Ray** probe that is possible, while confining the analysis to the feature of interest.

9. Select a **Pass Energy** and **Time Per Step (ms)** combination that meets your needs. (Refer to the **Pass Energy** discussion in the previous paragraphs.)
   *Note: The **Pass Energy [Default]** in the **Spectrum Parameters** area will set the **Pass Energy** for all current and new **Regions***
   *Note: Unique **Pass Energy** settings can be applied for each **Region** using the **Pass Energy** in the **Region Table** area*

10. Select **Regions** for analysis.

    Selecting Elements Using the Periodic Table

    Click the **Period Table** tool to select elements for analysis. Click the button for each element to be analyzed in the **Element Table**. This will define a narrow energy range for the most commonly used peak for each element

    Selecting Elements Using the Import Elements Tool

    Click the **Import Elements** tool to select elements for analysis. This brings up the **Import Element** box. Click the desired button to import elements used in previous **Profile, Angle, Line** or **Map** acquisitions. Selecting the **Import Spectral Display** will import elements identified in the "highlighted **"Spectral Viewer** [elements identified with **Peak ID**]

11. Select a number of **Sweeps** for each **Region**. Define a higher number of **Sweeps** for elements that are present at lower concentrations to optimize the acquisition.

12. Customize the setup of each **Region** if desired. Modify the energy range of an element or change the **Pass Energy** of a specific element.
    *Note: To see the entire table without using the scroll bar, click the **Show Table Details** icon in the table toolbar; this brings up the **Multiplex Regions** window.*
    *Note: See the **Region List Overview** discussion for more details if needed.*

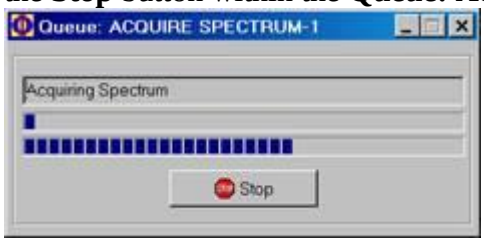
13. Select the number of **Cycles** to provide a data collection time of
    1-3 minutes [**Acquisition Tim (min)**] for larger diameter **X-Ray** spots
    or 5-15 minutes [**Acquisition Tim (min)**] for smaller diameter **X-Ray** spots.

14. Click the **Start** button in the **Spectrum** tab.

    - The **Queue: Acquire Spectrum-1** box appears displaying progress of the job and the **Acquisition Status** box appears displaying progress of the acquisition and

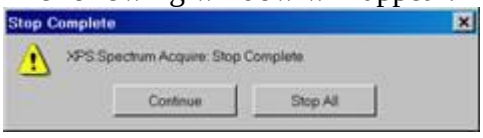
the **Remaining Time** in the acquisition.

- One file will be saved with multiple traces [one for each analysis area]
- The **Filename** will be:
  <filename>. <file number>.<platen name>. <position #>. <position list name>.spe
- The **SmartSoft-VersaProbe** data will be displayed in the **Spectral Viewer**. Each **Region** will be displayed within the viewer; with a separate trace for multiple analysis areas.
- All **Active** analysis positions in the **Position List Table** that are assigned a valid analysis area for the current acquisition will be visited sequentially.
- To **Stop** or **Abort** the acquisition before all **Cycles** are completed, use the **Stop** button within the **Queue: Acquire Spectrum-1** window.



If the acquisition has started, then the following window will appear:

  - **Stop** button terminates data acquisition at the completion of the current **Sweep** and saves the data file.
  - **Abort** button terminates the acquisition immediately and eliminates the data file that was created.
  - **Continue** button will return to the current acquisition
  - The following window will appear:



  - **Continue** button will advance to the next **Position List** position of the current **Job** and repeat the acquisitions process.
  - **Stop All** button will advance to the end of the current **Job** and stop the acquisitions process.

XPS Multiplex: Acquire More

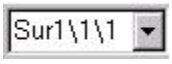
The platen(sample) must be at the same analysis position and no **XPS** parameters should have been changed. **Multiplex** should be the last acquisition that was **Stopped** or completed. Any **Inactive Regions** from the **Multiplex** acquisition will be removed from the **Region List** when the **More** acquisition is started. **Auto Neutralization** and **Beam Park** will be supported. **Pre-sputter** and **Z-Align** will not be repeated.

1. Click the **More** button in the **Spectrum** tab to add additional data to the **Stopped** or completed **Multiplex** acquisition.
2. Add more **Cycles** to increase the **Analysis Time**
   *Note: If the **Multiplex** acquisition was stopped; the current number of **Cycles** can be used to continue the acquisition*
3. Click the **Start** button
   - The original acquisition file will be preserved with its previous name.
   - One new file will be saved with multiple traces [one for each analysis area]
     The new **Filename** will be:
     <"previous name"><+1>. spe
   - Subsequent **More** acquisitions will preserve previous files; acquire new data and save a new file with the **Filename**:
     <"previous name"><+2>. spe

XPS Multiplex: Data Reduction in SmartSoft-VersaProbe

Data reduction and reporting can be performed in **SmartSoft-VersaProbe** or **MultiPak**. **MultiPak** allows for more advanced data reduction. In many cases the **SmartSoft-VersaProbe** procedures will be sufficient.

Spectral Display

- Use the **Zoom** to enlarge a region of interest and the Right-click Context menu; **Fullscreen** to revert back.
- Select the **Show One**. Use the 

Current Curve to view a single **Region**.
- Click the **Fullscreen** button for a full screen view of the **Spectral Viewer**.

*Note: With **Profile** or **Line** or **Map** data displayed; use the **Fullscreen Data/Tile** button to enlarge the highlighted file's **Spectral** or **Profile** Viewer.*

Annotate the Spectral Viewer

- Double-click within the viewer and type in text to annotate. Click outside the text box to set the text.
- To move the text, click and drag the text to the desired location.
- To edit text, double-click over the text box, then edit. Right-click over the text for more options (cut, copy, paste, delete, etc.).
- Right-click over the viewer for additional options, including delete all annotation. Click the **Properties** option to open the **Graph Annotation Properties** box. Use this box to select the text font, size, color, etc.

Data Reduction

- Use the **9 Point Savitsky-Golay Smooth** to smooth the data.
- Use the **Peak Identification** to identify peaks
- Use the **Energy Cursor** to identify peaks
- Use the **Element Cursor** to identify peaks

Output the Data

- Click the button to **Copy** the selected viewer to the clipboard. This allows the data to be imported as a graphic into any Microsoft Office application (Word, PowerPoint, etc.).
- Click the button to **Print** the selected viewer to the default printer.
    - *NOTE: Annotation changes made to the viewer are not saved as part of the data file.*

XPS Multiplex: Data Reduction in MultiPak

After collecting the **Multiplex** data, open the **MultiPak** software to reduce the data. The **MultiPak Spectrum** window and **Periodic Table** will appear. For detailed information about the use of **MultiPak**, refer to the ***MultiPak Software Manual***.

1. Click the **LastAcq** button (which will open the file most recently acquired) or click the File button on the upper tool bar to open the file of choice from a File selection menu.

    - *Initially, all the individual spectra will be displayed on one energy axis, and there will be a button for each element at the bottom of the **Spectrum** window (in the **Region** bar).*
    - *To view the spectrum of an individual element, click the button that represents the element.*
    - *The filename is displayed in green, indicating that this data is "selected." Any **MultiPak** functions activated will be performed on this data.*
    - *The header (displayed only when **Header** in the **View** menu is checked) lists the file's data acquisition parameters (date of acquisition, beam voltage of acquisition, etc.).*
    - *The third line of the header will be updated with a processing history as data reduction routines are applied to the data.*

2. **Quantify Data** in **MultiPak**. Quantification of **ESCA** spectra is most accurate when **Peak Area** measurements are used. In most situations, it is recommended that you use a **Shirley Background Subtraction** for making the area measurement.

    a) To view or change the end points that will be used to define the background subtraction for a specific elemental peak, click the button at the bottom of the **Spectrum** window for that element and the software will automatically expand the region for that element's default peak and display the cursors that are used for background subtraction. If it is necessary to adjust the cursors, click and drag the cursor using the left mouse button. While the cursor is being moved, a line depicting the background will be displayed to assist you in positioning the cursor.

***Note**: The default peak is defined in the **Transition Editor** menu and usually it is the main or major elemental peak. If you want to change the default peak, you can do so in this menu.*

b) Click the **%** button to view the results of the atomic concentration calculation.

c) To print the **AC Table**, open the **File** pull-down list on the **AC Summary** window and click **Print**

3. **Chemical Information** in **MultiPak**. If the focus of the spectrum is to examine the peak shape and position of the spectra to obtain chemical shift information, there are several software tools in **MultiPak** to help you with that task. Before reducing the data in a spectrum, a smoothing routine may be applied to the data set to improve the accuracy of peak position and peak area measurements. **A Savitzky-Golay Smooth** routine is recommended because it retains the original shape of the data curve during the smoothing process. Depending on the size of the data steps in your data and the width of the peaks, a 5-, 7-, or 9-point smoothing operation is typically used. To set up the preprogrammed **Smooth** button, which is displayed on the upper tool bar, click **Data>Smooth/DerivativeSetup.** The **Smooth/Derivative** setup options will be displayed below the spectrum. Using the left mouse button, select the**Savitzky-Golay Smooth** and the desired number of points to be used for smoothing. If you selected nine points, the Smooth button in the upper tool bar will read "**SG9**." The smoothing functions operate on the displayed data. If all of the data regions are displayed, all of the regions will be smoothed. If you click the **Smooth** button a second time, the data will be smoothed twice.

4. **Energy Scale Calibration** in **MultiPak**. The first step in extracting chemical information from a peak is to correct the energy scale for any shifts that are due to charging. Typically, this is done by looking at the position of the C1s peak from the hydrocarbon that is present on the surface of most samples. If the C1s peak is not at approximately 284.8eV, it should be adjusted using the **Shift** routine.

   a) To open the **Shift** routine, select **Shift Setup** from the **Data** pull-down list.

   b) When a yellow cursor appears on the screen, position it with the left mouse button on the center of the hydrocarbon portion of the C1s peak.
   *Note: When you release the mouse button, the C1s will shift to 284.8eV (or the position defined for C1s in your **MultiPak Periodic Table** database).*
   Note: If no carbon is present on the sample, a peak from a matrix element with a known chemistry and binding energy may be used in place of the C1s to correct the energy scale for charge-induced shifts.
   *Note: The energy can be read by positioning the cursor on the peak and reading the value on the right side of the lower **Spectral Shift** tool bar.*

5. **Binding Energy Identification** in **MultiPak**. The next step in extracting chemical information is to determine the binding energy of the peaks for each element. This can be done in several ways.

   a) With the peak displayed in the **Spectrum** window, position the cursor at the center of the peak, using the middle or right mouse button. The location of the cursor is displayed

in terms of the binding energy and intensity at the right side of the lower tool bar in the Spectrum window.

b) The **FWHM/Area Tool** may also be used to determine the binding energy of a peak. With the peak displayed in the **Spectrum** window, select the **FWHM/Area** function from the **Tool** pull-down list.
The **FWHM/Area Tool** routine will automatically search for the most intense peak in the displayed data and display a list of information that describes the peak including its binding energy. A yellow cursor is displayed where the software has determined the location of the peak to be. If you want to change this position, grab the cursor using the left mouse button and drag it to the desired location. When you have finished moving the cursor, the displayed information will be updated.

6. **Curve Fit** in **MultiPak**. If the peak of interest contains more than one chemical state, it may be necessary to use the **Curve Fit** routine to determine the binding energy of each of the chemical states. To perform a curve fit, use the following steps:

a) Make any necessary corrections to the energy scale using the **Spectral Shift** routine.

b) Click the **Curve Fit** button.

c) Deselect the **Setup** mode and adjust the energy cursors for background subtraction.

d) Select a **Gaussian-Lorentzian** peak shape. (This peak shape is appropriate for most **ESCA** peaks.)

e) Define initial bands by pointing and clicking at the desired band position with the right mouse button.

f) Readjust the initial band estimates by entering numerical values in the **Curve Fit Setup** menu, or drag the flags attached to the currently selected band, using the left mouse button.

g) Click **Fit** to start the fitting routine.

h) Evaluate the resulting peak positions as shapes using your experience, reference spectra, and database information.

i) Make any necessary adjustments to the band parameters and click **Fit** again.

j) When you are satisfied with the results, print the **Summary** information and the curve fit spectrum.

k) Click **Exit** to leave the **Curve Fit** routine.
Note: The error (in red) follows the baseline evenly, indicating a good fit.
*Note: The **Curve Fit Summary** table contains peak position information that can be used to determine the identity of the chemical state and peak area information to quantify those chemical states. For a more detailed discussion of the **Curve Fit software**, refer to the **MultiPak Software Manual**.*

XPS Test Acquire

Perform a **Test Acquire** for each selected element. **Test Acquire** provides an opportunity to determine whether the **Acquisition Window Setup** for each element is optimized.

It is important to make each window wide enough to include the entire **XPS** peak. Since **Test Acquires** can be performed only for the elements on the surface, the operator needs to consider the windows of the buried elements, especially on samples where there may be charging layers below the surface. The windows need to be wide enough so that, if the sample charges and the **XPS** peaks shift upward in energy, the peaks will not move outside the acquisition windows.

1. Select the desired **XPS Application** tab.

2. Left-click to select an **Element** to examine (highlighting the **Element** "green")

3. Click the **Test Acquire** icon to launch **the Test Acquisition**

   - The **Test Acquire** window will open.
   - **Test Acquire** will provide a real-time display of data for a single **Region** that will not be saved.
     - The highlighted Region in the **Region Table** will be acquired in either a "**Scanned**" or "**UnScanned**" collection mode depending on the **Analyzer Mode** selected in the current application tab
     - The **Sweeps** and **Ratio** will default to a temporary value=1
     - The "**Scanned**" **Time Per Step (ms)** will default to a temporary value=10ms The "**UnScanned**" **Time Per Region (s)** will default to a temporary value=1000ms
     - The **Persistence** can be used to integrate more counts to improve the signal-noise

   The **Test Acquire** acquisition will continue until the **Stop** button or the **Acquisition Status Stop** button is clicked.

4. Ensure that the full peak is contained within the analysis window. Adjust the width or energy of the window to ensure that the peak remains within the window.
   - Adjusting any of the following parameters will stop and restart a new **Test Acquire** acquisition:

     Scanned

     Lower; Range; Upper; Analysis Lower; Analysis Upper:

     The **Lower**, **Range** and **Upper** parameters define the acquisition parameters for data collection for each **Region**.

     The **Analysis Lower** and **Analysis Upper** parameters define the analysis window that will be used in calculating the intensity data for each **Region**.

     UnScanned

     Peak; Range; Analysis Peak; Analysis Range:

     The **Peak** and **Range** parameters define the acquisition parameters for data collection for each **Region**.

     The **Analysis Peak** and **Analysis Range** parameters define the analysis window that will be used in calculating the intensity data for each **Region**.

Pass Energy [Pass]:
The **Pass Energy** will define the analyzer band pass and resultant alignment of energies across the 16 channels.

eV Step:
The **eV Step** will define the energy resolution of the data file for each **Region**.

- Selecting a new **Element** in the **Region List** will stop and restart a new **Test Acquire** acquisition.
- Changing the **Test Acquire Marker** on the **SXI** viewer will stop and restart a new **Test Acquire** acquisition at the new scan position.
- Select the **Large Counts Display** icon to reveal larger font size to the **Max Counts** readings.

Queue Acquisitions Overview

The **Queue** is available to generate a list of **Jobs** and to perform them sequentially as a single task. These **Jobs** are added to the **Queue** from each of the five **XPS Application** tabs and **Run** from the **Queue** window. It is required to have all of the analysis positions defined in the **Sample>Position List Table** and **Add** each **Job** to the **Queue**.

Refer to each **XPS Application** instructions regarding acquisition setup. Refer to the **Acquire & Queue Overview (All XPS Applications)** for further information regarding **Queue** features.

1. Right-click on the **Add Q** button on any **XPS Application** tab and select **Clear Queue** to begin.

   a) Set the desired analysis positions to **Active** in the **Sample>Position List Table.**

   b) Setup the desired **XPS>Acquisition Properties** for pre-acquisition and post-acquisition parameters.

   c) Setup the desired **XPS Application** parameters

   d) Click the **Add Q** button

2. Repeat steps **a - d** to add additional **Jobs** to the **Queue**

3. Right-Click the **Add Q** button and select **Open Queue…** on any **XPS Application** tab
   *This will open the **Queue** window for viewing*

4. Click the

Start button

   - The **Queue: Acquisition Status** window appears displaying progress of the acquisition and displaying the **Remaining Time** in the acquisition.

- Refer to each **XPS Application** instruction regarding viewer updates and file saving operations.
- All **Summary** listed **Active** analysis positions in the **Position List Table** will be visited sequentially.

Stopping or Aborting Tasks

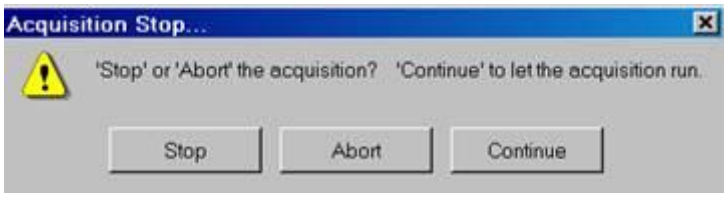
Use the Stop button to stop or abort acquisitions before they are complete.

Stop:

Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop or abort a current **Task** or **Job**.

Clicking the **Stop** button will stop non-acquisition tasks such as **SXI**, **Test Acquire**, **Platen Transfers**, etc.

Clicking the **Stop** button with an acquisition running will prompt the user to choose between **Stop** & save data, **Abort** & discard data, or **Continue** with the current **Job**.



Once the acquisition is stopped or aborted a prompt will appear giving the user the choice of **Continuing** with other **Positions**/**Jobs** remaining in the **Position List** or **Queue** or to **Stop All** Jobs.

Queue: Edit (Add, Delete, Change Order)

1. Open the Queue with the desired setting. **User-1** or customized setting.

Add

1. Verify that the desired **Acquisition Properties** are selected.
2. Verify that the desired acquisition parameters are selected.
3. Verify that the desired positions are **Active** in the **Position List.**
4. Click the **Add Q** button in an **Application** tab.
   *This **Job** will be added to the end of the **Job** list*

Delete

1. L-click to select the desired **Job** in the **Queue>Job>JobList**.
   Job will change to "green highlighted"

2. Click the **Delete** button in the **Jobs** toolbar.
   *This **Job** will be removed from the **Job** list*

Change the Order of the Job list

1. L-click to select the desired **Job** in the **Queue>Job>JobList**.
   Job will change to "green highlighted"

2. Click the **List Item Up** or **List Item Down** button in the **Jobs** toolbar.
   *This **Job** will be moved to a new order in the **Job** list*

Queue: Run the Queue

1. Open the Queue.
2. Click the **Start Queue** button to run the entire **Job** list

Queue: Run a Portion of the Queue

1. Open the Queue.
2. Right-click on a desired **Job** in the **Job** list and select **Run From Here**

Queue: Stop the Acquisition and Continue at the Next Position

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**
2. Click the **Stop** button in the **Acquisition Stop…** window
   The acquisition will stop at the end of a break point [sweep, cycle, frame]
3. When the Acquisition is complete
   Click the **Continue** button in the **Stop Complete** window
   *The current **Job** will continue at the next Position in the **Position List***

Queue: Stop the Current Job and Continue at the Next Job

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**
2. Click the **Stop** button in the **Acquisition Stop…** window
   The acquisition will stop at the end of a break point [sweep, cycle, frame]
3. When the Acquisition is complete
   Right-click the **Continue** button in the **Stop Complete** window
4. Select **Continue at Next Job** from the list of options
   *The **Queue** will continue at the next **Job** in the **Job** list*

Queue: Stop the Queue Completely

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**

2. Click the **Stop** button in the **Acquisition Stop…** window
The acquisition will stop at the end of a break point [sweep, cycle, frame]

3. When the Acquisition is complete
Click the **Stop All** button in the **Stop Complete** window
*The **Queue** will turn off any neutralization and stop*

Queue: Abort the Acquisition and Continue at the Next Position

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**

2. Click the **Abort** button in the **Acquisition Stop…** window
The acquisition will abort immediately

3. When the Acquisition is aborted
Click the **Continue** button in the **Stop Complete** window
*The current **Job** will continue at the next Position in the **Position List***

Queue: Abort the Current Job and Continue at the Next Job

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**

2. Click the **Abort** button in the **Acquisition Stop…** window
The acquisition will abort immediately

3. When the Acquisition is aborted
Right-click the **Continue** button in the **Stop Complete** window

4. Select **Continue at Next Job** from the list of options
*The **Queue** will continue at the next **Job** in the **Job** list*

Queue: Abort the Queue Completely

1. Click the **Stop** button within the **Queue:Acquisiton Status** window or within the **Queue** window to stop a current **Job**

2. Click the **Abort** button in the **Acquisition Stop…** window
The acquisition will abort immediately

3. When the Acquisition is complete
Click the **Stop All** button in the **Stop Complete** window
*The **Queue** will turn off any neutralization and stop*

Refer to VersaProbeII manuals about more Quene options.

Some Analysis Considerations

- Use as much current as possible for good signal-to-noise ratio while (1) still being able to resolve the features of interest (if the beam diameter is larger than the feature of interest, you will get substantial signal from the surrounding area) and (2) not causing electron beam damage to the sample surface.

- Differentiating data makes it easier to identify elements, because Auger peaks are riding on a high secondary electron background. Smoothing data will lessen the impact of noise on the spectrum, but peak resolution is lost as the smoothing function broadens.
- Survey spectra can be very noisy when rastering over an area that includes elements of drastically varying secondary electron yields or has rough surfaces with sharply changing angles.
- Longer acquisition times or multiplex spectra are usually needed to identify minor elements.
- Be aware of elemental sensitivity factors when choosing beam voltages. As a general trend, as the beam voltage is increased, the sensitivity to high-energy Auger peaks increases while the sensitivity to low-energy peaks decreases.
- Be aware of the four types of beam damage that the current selected can do to your sample:
- *Reduction*—Metal oxides and hydrocarbons can be reduced by the electron beam.
- *Desorption*—Adsorbed material can be removed.
- *Electron migration*—Mobile ionic material can be repelled or drawn toward the electron beam. For example, PSG glass contains phosphorus, which is attracted to the beam.
- *Diffusion*—The beam's heat can cause an intermixing of liquids, solids, and gases.