

Standard Operating Procedure II

PHI 5000 *VersaProbe* II XPS/UPS

(UPS, XPS Depth Profile, Angle Profile, Line and Map)



Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room E119E
810 West Campus Drive
West Haven, CT 06516

Version 1.0, May 2016

- > Please **FOLLOW the SOP strictly** to keep the facility in good condition. Any **explorations are strongly prohibited** unless permitted by lab manager
- > **NEVER** use your own USB drive on the **XPS computer**. Data can be retrieved from Yale data server
- > **NEVER** surf the web on the **XPS computer** in order to minimize the risk of the computer being hacked

- > Yale West Campus MCC facility users must acknowledge MCC in their publications that rely significantly on MCC resources. The general acknowledgement for SEM should read:
“The spectra were taken using the PHI VersaProbe II at Yale West Campus Materials Characterization Core (MCC).”
- > The core reserves the right to use the data for core promotion

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PHI 5000 VersaProbe II XPS/UPS Standard Operating Procedure (UPS, XPS Depth Profile, Angle Profile, Line and Map)

1 Introduction

1) Features of this Instrument:

- > Micro-area element composition and chemical state determination on material surfaces
- > Analysis of insulating samples with dual beam charge neutralization method
- > Depth profile analysis of structures and interfaces

2) Location

Materials Characterization Core
Room E119
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3) Primary Staff Contact

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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. We **DO NOT** recommend user explorations on program unless endorsed by core manager.

2 UPS Spectral Scan

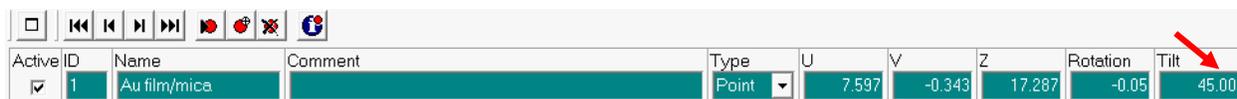
- 1) Perform **XPS** scan on samples **FIRST** before **UPS** setup to examine the sample surface. Follow the steps in **SOP Basic** for sample preparation, transfer and position definition

Warning: make sure the main chamber pressure is kept below 5×10^{-7} Pa before **UPS**

Notice:

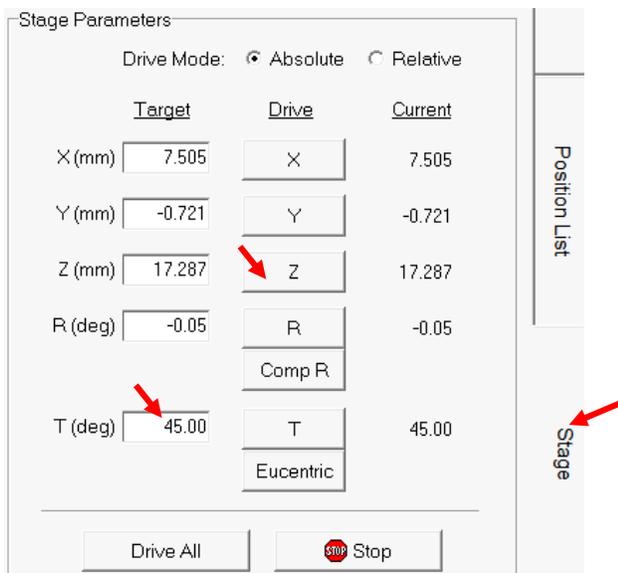
- > **XPS** scan **CAN** be performed at **UPS** positions (90 degree tilted sample holder), Keep in mind that using a **90 take off angle** for the photoelectrons is more sensitive to the bulk material than **lower take-off angles**.
 - > **UV source** need to be turned to **standby** during **XPS** scan at
 - > **Ar** sputtering can be performed on **UPS** positions (90 degree tilted sample holder). However, the sputtering is **NOT** as efficient as on flat positions because of incident **Ar⁺** beam at grazing angles.
 - > **UV source** can be kept **ON** during **Ar** sputtering
- 2) **UPS** position definition

- a) Click  Sample on top tab menu to enter **Sample** window, define and **Z-Align** the position ready for **XPS first** with **flat** stage (**Tilt = 45** degree)



Active	ID	Name	Comment	Type	U	V	Z	Rotation	Tilt
<input checked="" type="checkbox"/>	1	Au film/mica		Point	7.597	-0.343	17.287	-0.05	45.00

- b) Click **Stage** tab on the right side of **Sample** window to enter **Stage Parameters** window and change stage tilt angle **T(deg)** from **45.0** (flat stage) to **90.0** (tilted stage perpendicular to analyzer column), hit **Enter** and then hit  tab below to tilt stage to entered angle



Stage Parameters

Drive Mode: Absolute Relative

	Target	Drive	Current
X (mm)	7.505	X	7.505
Y (mm)	-0.721	Y	-0.721
Z (mm)	17.287	Z	17.287
R (deg)	-0.05	R	-0.05
		Comp R	
T (deg)	45.00	T	45.00
		Eucentric	

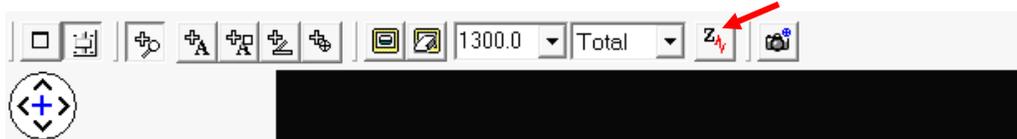
Position List

Stage

Drive All 

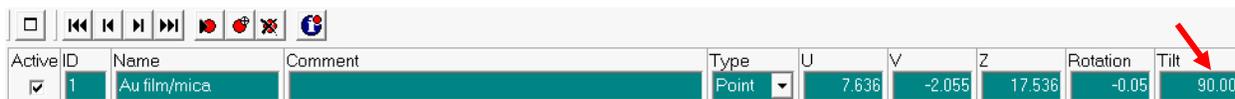
Warning: the optics configuration (UV source vs. Analyzer) inside the chamber **requires** sample stage to be **perpendicular** to the analyzer for **optimized UPS** signals.

- c) Hit  button on top of **SXI** image window to start **Z-Align**



- d) Click  above **Position List Table** to **Update Position** after **Z-Align**, check through chamber window to see the stage **perpendicular** to the analyzer column (**Tilt = 90 degree**, if **NOT**, report Core staff immediately.)

Warning: this step is **critical**, or the sample will stay at **FLAT**



Active	ID	Name	Comment	Type	U	V	Z	Rotation	Tilt
<input checked="" type="checkbox"/>	1	Au film/mica		Point	7.636	-2.055	17.536	-0.05	90.00

3) UV light source ignition

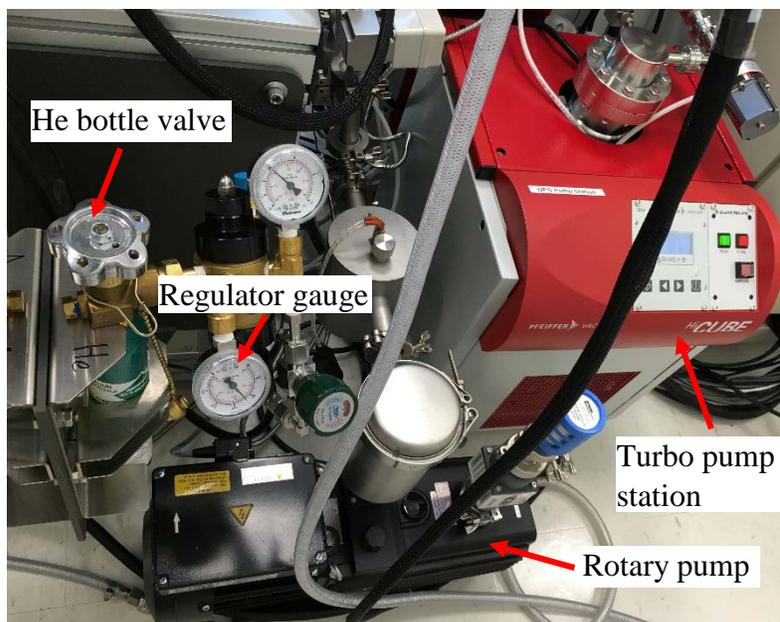
- a) Turn **ON** the **UV** source controller (**UV40A-PS**) on the rack.



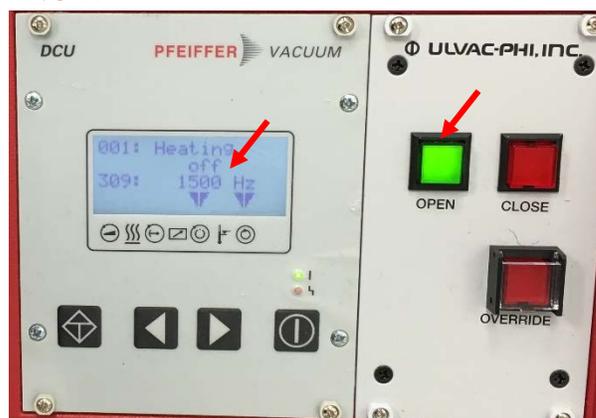
- b) Go to the back of the vacuum chamber and check to **MAKE SURE UPS differential pumping system ON**.

Warning:

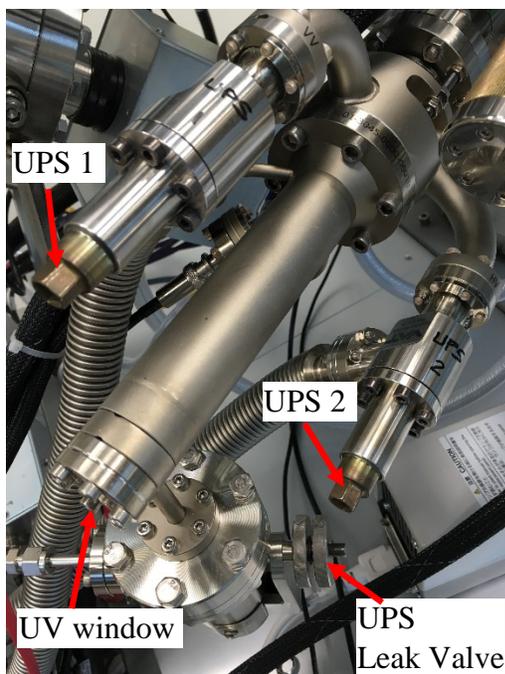
- This step is **CRUCIAL**, violation will lead to **accident venting of the main chamber** and **damage of XPS optics system** inside. Violated user account will be **suspended** and repair charges will be on **user's PI account!**
 - The differential pumping system needs to be turned **ON 12 hours before UPS**. **DO NOT** run **UPS** if the system is **OFF**. Contact Core staff instead.
- > **Touch** the **rotary pump** on the ground, **MAKE SURE** it is **RUNNING!**



- > Make sure the **OPEN** button on turbo molecular pump is **ON** (lit in **GREEN**), and check the **rotating speed** on the status panel, make sure it is running at **full speed (1500 Hz)**, **REPORT** to Core staff immediately if **NOT**.



- > Open the He gas bottle by turning the **highlighted He bottle valve** counterclockwise **~45 degree**
- > Check to make sure the pressure is **1000 ~ 1500 psi** on the **Regulator Gauge**. Report to Core staff if dropped **below 100 psi** and have the He bottle replaced.
- c) **TOUCH** the high voltage (**HV**) window on the UV source controller to see the kV number **flashing in WHITE** and **THEN** turn the knob on the right side to **1.00 kV**
- d) Open UPS differential pumping valves
 - > Turn the **UPS 1** valve (connected to **rotary pump**) **counterclockwise** to **fully open** the valve; the main chamber pressure should drop slightly.
 - > Turn the **UPS 2** valve (connected to **turbo pumping unit**) **counterclockwise** to **fully open** the valve; the main chamber pressure should improve.



- e) Turn **SLIGHTLY** the **UPS leak valve counterclockwise** to **leak** helium gas into the UV source

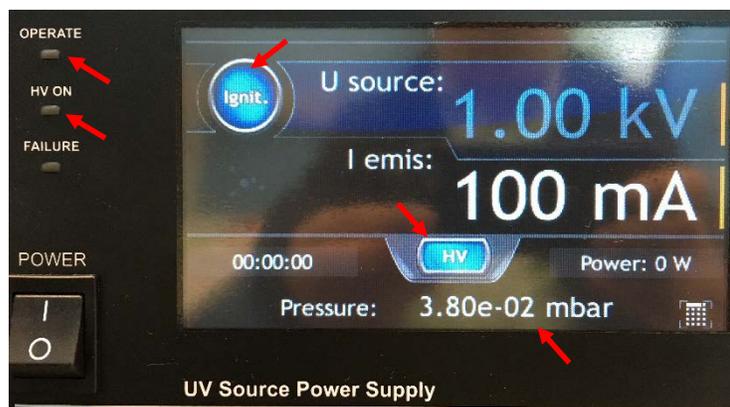
Warning:

- **This step is CRUCIAL, violation will lead to high pressure helium backfill of the main chamber and XPS system shutdown!** Violated user account will be **suspended** and possible repair charges will be on **user's PI account!**
- **Always press and turn** the **leak valve knob** to introduce helium manually.

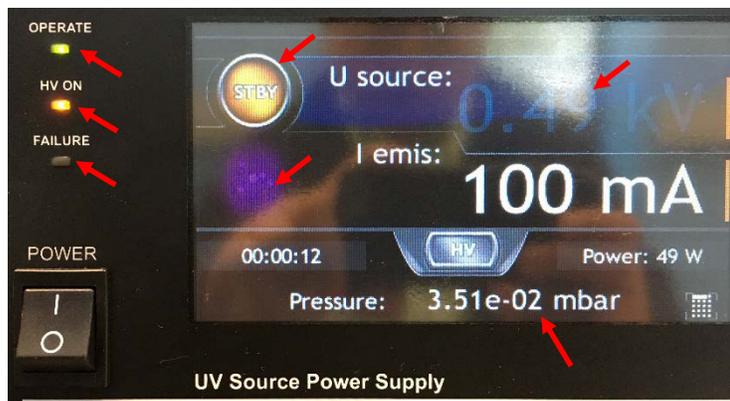
- > **Watch main chamber pressure VERY CLOSELY** while **slowly turning** the leak valve knob and **stop turning** when a **little pressure increase** is seen.

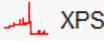


- > **Then, watch the pressure window on UV source controller VERY CLOSELY** and **continue slowly turning** the leak valve knob and **stop turning** when the pressure increases to $\sim 4 \times 10^{-2}$ mbar. The main chamber pressure should increase from **high 10^{-8} Pa** to **mid or lower 10^{-7} Pa**.



- > Press **HV** button on the controller, the **OPERATE** and **HV ON** LED should be **ON**.
- > Press **Ignit.** ignition icon on the controller to ignite the UV light. If **FAILURE** light is not turned **ON**, then the **purple light spot** appears on the controller and the **HV** drops to **0.5 kV**. The UV light should be visible through the **UV window** on the UV source.



- > **VERY SLOWLY** turn the leak valve **clockwise** to lower the He pressure reading on the controller from **$\sim 4\text{e-}02$ mbar** to **$2\text{e-}02$ mbar**, and then wait for **15 minutes** to **stabilize** the UV light.
Warning: For **He I UPS**, it is important to keep the helium pressure at **$2\text{e-}02$ mbar** in order to maximize the He I emission line intensity.
- 4) In **SmartSoft** window, click  **XPS** tab on top tab menu to enter **XPS** analysis window and click **Spectrum** tab on the right side. In Source window, choose **UPS** (Ultraviolet Photoelectron Spectroscopy) and in **Source** window.
- 5) Click  on top left of **Regions** window to enlarge the **Regions (Spectrum)** window:



- 6) Click on  button on the Regions window to open the Periodic Table, and click on  on the table to start a Spectral Survey (**Su**) Scan, then close the table.

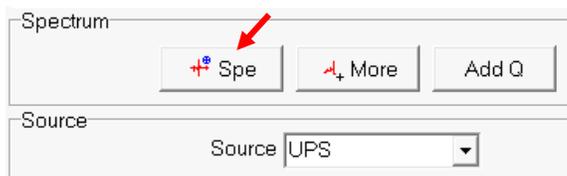


- 7) In **XPS Regions** window, click **Pass** to change **Pass Energy** to **typically 2.950 eV**, and **eV Step** to **typically 0.0250 eV**. **Close** the window. Set **Lower** energy to **-5 eV** and **Range** to **21 eV**
Notice: for **UPS** scan, typically **smaller Pass Energy** (**2.95 – 5.85 eV**), **smaller Step** size (**0.025 – 0.05 eV**), and **20 ms Time Per Step** should be chosen to increase **spectral energy resolution**.

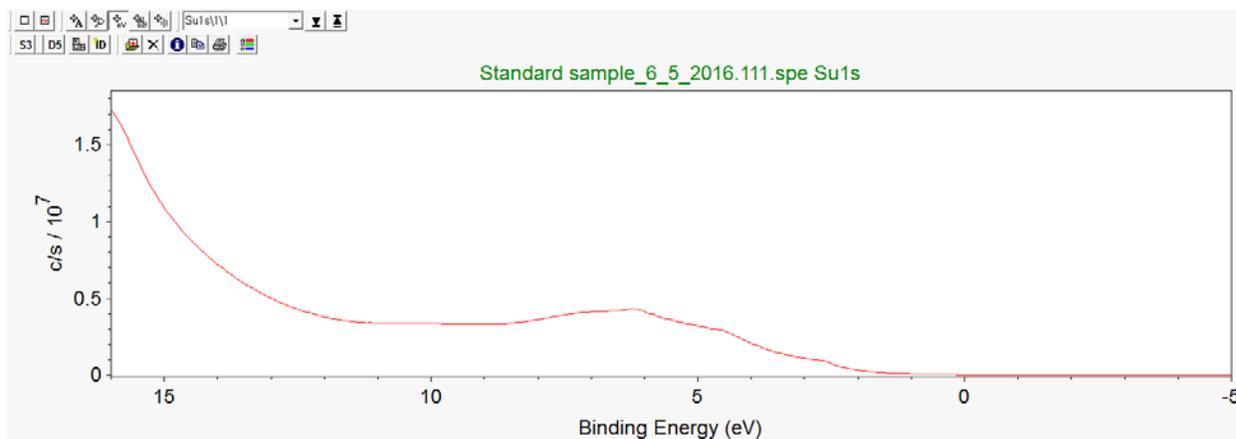


- 8) Click  button, in **XPS Acquisition Setup** window:
 - a) In **Setup** tab, choose **OFF** in XPS: E-Neut Neutralization Auto [A] Off
 - b) In **Setup** tab, choose **OFF** in XPS: Ion Gun Neut Auto [A] Off
 - c) In **Region** tab, choose **OFF** in **-5V Bias** Enabled Disabled

9) Hit  Spe in Spectrum window to **start UPS** scan



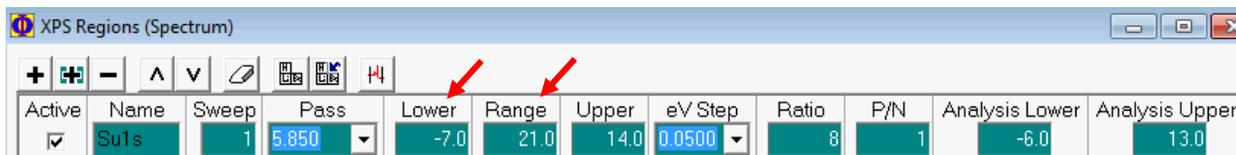
10) The Survey spectrum will appear in the **Spectral Window**.



11) To measure **Work Function** using **UPS**

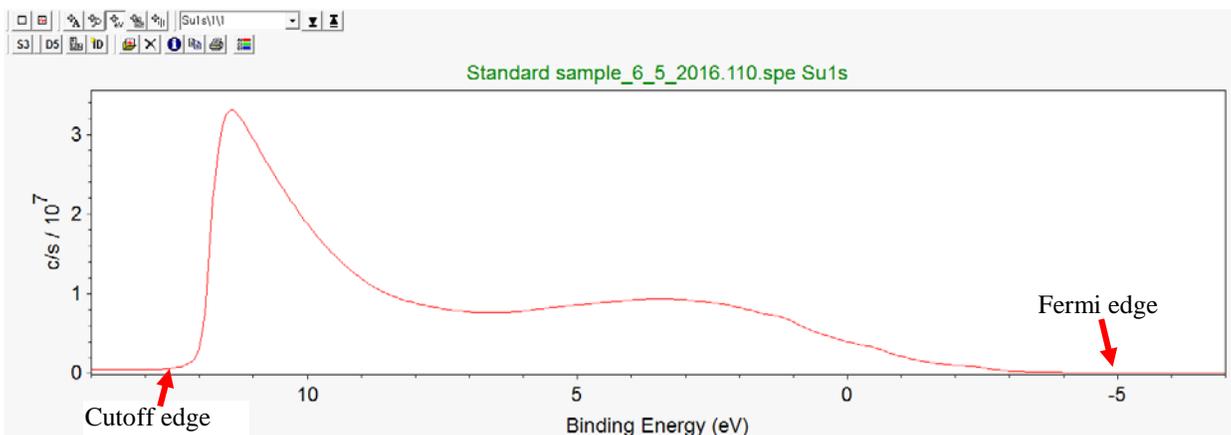
a) Click  button, in **XPS Acquisition Setup** window:

- > In **Setup** tab, choose **OFF** in XPS: E-Neut Neutralization Auto [A] Off
- > In **Setup** tab, choose **OFF** in XPS: Ion Gun Neut Auto [A] Off
- > In **Region** tab, choose **Enabled** in -5V Bias Enabled Disabled
- > In **XPS Regions** window, set **Lower** energy at **-7 eV**, **Range to 21 eV**, **smaller Step** size (**0.025 – 0.05 eV**), and **20 ms Time Per Step** should be chosen to increase **spectral energy resolution**.



a)

12) Hit  Spe in Spectrum window to **start UPS** scan, and the UPS spectrum will appear in the **Spectral Window** with both the cutoff edge on the left side and Fermi energy edge collected:



13) Closing UPS measurement

- Press  ignit/STBY icon **ONCE** on the controller to turn **OFF** the UV light
- Turn the controller **knob** on the right side to decrease the HV to **0 kV**
- Hit** the **Power button** on the left side to **turn OFF** the **UV** source controller.
- Turn the **UPS leak valve clockwise** to **close the valve**
Warning: it is **CRUCIAL** to perform this step **before** the following steps, otherwise the **main chamber** will be filled with **high pressure** He gas and the **SmartSoft** software **will fail for sample transfer!** (Stage “**Initialize**” may need to be performed for sample extraction, **contact** Core stuff if happened.)
- Turn the **UPS 1 valve clockwise** to **fully close** the valve; the main chamber pressure should increase slightly to **higher 10⁻⁷ Pa**
Warning: DO NOT overtighten both **UPS 1** and **2** valves to cause mechanic damage. To judge if the valves are closed properly, the **main chamber pressure** should be improved after a brief increase.
- Turn the **UPS 2 valve clockwise** to **fully close** the valve; the main chamber pressure should increase up to **higher 10⁻⁷ Pa**
- Go to the back of the vacuum chamber, turn the **He bottle valve clockwise** to **fully close** the valve
- On SmartSoft window, click **Stage** tab on the right side of **Sample** window to enter **Stage Parameters** window and change stage tilt angle **T(deg)** from **90.0** (tilted stage perpendicular to analyzer column) **BACK** to **45.0** (flat stage) to, hit **Enter** and then hit  tab below to tilt stage to entered angle
- Check to make sure the sample is **flat through the vacuum chamber window**.
- Then**, follow the steps in **SOP Basic** to finish the **sample extraction**.

3 XPS Depth Profile

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.

NOTE: The sputtering process may cause some chemical damage and broaden the peaks.

NOTE: Before depth profile analysis, the **Survey** or **Multiplex (Region)** acquisitions should have been performed to identify Regions of interest and that **analysis areas** have been defined.

- 1) Select the desired **Active** analysis positions in the  Sample > **Position List Table**.
- 2) Enable the **Auto Z** before **Acquire (XPS>Profile> XPS Setup...)** if the analysis positions have **NOT** been previously aligned
- 3) Enable the **Automated Neutralization (XPS>Profile> XPS Setup... or Status Bar)** if the sample is insulating
- 4) Open the **XPS Profile Application** window (**XPS>Profile tab**).
- 5) *Optional use of Profile Settings*
 - > Select a setting from the **Profile Settings** combo box.
 - > Click the **Load** button.
- 6) *Optional use of Previous Acquisition file from Profile Settings*
 - > Click the  File... box.
 - > Choose a **Profile** file with desired settings.
 - > Click **Open**. The **Profile 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 the desired **UnScanned** or **Scanned Analyzer Mode**.
- 10) Select a **Pass Energy** (typically **187.85 eV**) and **Time Per Step (ms) (Scanned mode) or Time Per Region (ms) (UnScanned mode)** (typically **20 ms**) that meets your needs.

Note: The **Pass Energy** in the **Profile 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
- 11) 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 **Multiplex**, **Angle**, **Line** or **Map** acquisitions. Selecting the **Import Spectral Display** will import elements identified in the “highlighted” **Spectral Viewer** [elements identified with **Peak ID**]
- 12) 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.
- 13) Customize the setup of each **Region** if desired. Modify the energy range of an element or change the **Pass Energy** of a specific element.
- Notice:**
- > **Larger Pass Energy (117.4 or 187.85 eV)** should be chosen to increase signal sensitivity as **no need** to examine **sputter-damaged chemical states** by improving energy resolution (smaller **Pass Energy**).
 - > **Step** size can be set **smaller (0.125 or 0.2 eV)** to improve **S/N**.
 - > To see the entire table without using the scroll bar, click the **Show Table Details** icon in the table toolbar; this brings up the **Profile Regions** window.
- 14) Select the desired **Sputter Mode** [**Alternate**; **Alternate w/Zalar**; **Continuous** or **No Sputter**].
- 15) Open the **XPS>Properties>Profile** window.
- > If **Zalar** is desired; Select **Zalar Accuracy**; **Zalar Interval (cycle)** and **Rotation Speed (rpm)**
 - > Click **XPS>Profile>** Profile... button to select desired **PreSputter Cycles** in
 - > Close the **XPS>Properties>Profile** window
- 16) Setup **Sputter** parameters and **Cycles** depending on the **Sputter Mode**
- Note:** the related **Sputter Rate**; **Sputter Depth**; **# of Cycles**; **Total Cycles** fields will be updated as parameters are entered.
- Note:** the **Total Cycles** will include the Pre-Sputter Cycles

Alternate	Continuous	No Sputter
Open the Sputter Rate Table window	Select the desired Sputter Setting	Select the # of Cycles
Use the edit keys to create the desired number of sputter Layers and assign Names if desired	Select the # of Cycles	
Select the desired Gun Type for each Layer Ar, C60 or GCIB		
Select the desired Time for each Layer <i>Total time sputtered for this layer for this Gun Type. This time will be broken up into sub Cycles resulting in each sputter cycle etching a duration of the Interval time</i>		
Select the desired Interval (sputter time at each Cycle) The # of Cycles will be update to full-fill the Time (min) requested		
Select the desired Sputter Setting for each Layer		
Select the desired total Time (min) at each Sputter Layer		
Select the desired Interval (sputter time at each Cycle) The # of Cycles will be update to full-fill the Time (min) requested		
Close the Sputter Rate Table window		

- 17) Click the  Profile button in the **Profile** tab.
- > The **Queue: Acquire Profile-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 for each analysis area with multiple traces [a set of traces for each region (or energy)]
- > Each file will contain **Spectral Data** and **Depth Profile Intensity Data** for each **Region**

- > The Filename will be:
<filename>. <file number>. <platen name>. <position #>. <position list name>_n. pro with the **_n** indexed corresponding to each analysis area (e.g. **sample.101_1.pro** and **sample.101_2.pro**)
<filename>. <file number>. <platen name>. <position #>. <position list name> will remain the same for each analysis area file.
- > The **SmartSoft-VersaProbe** Spectral data will be displayed in the Spectral Viewer. Each Region will be displayed separately within the viewer. There will be a **Spectral Viewer** for each analysis area.
- > The **Profile** intensity plot will be displayed in the **Profile Viewer**. There will be a **Profile Viewer** for each analysis area.
- > 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 Profile-1** window.

18) Click the  **More** button in the Profile tab

If a Depth Profile had Stopped or completed; the file will be loaded in the Profile Settings and the acquisition parameters will be updated.

- > *Continue below by modifying desired parameters*

If the last Depth Profile had been aborted,

- > An **Open File** window will appear.
- > Select the aborted **Profile** file and click **Open**
Note: the filename for an aborted Profile will appear with the following format:
PlatenName.121 aborted at May 13 2008 14 7_1.pro
- > The **Profile Acq Warning** window will appear with a message
- > Follow the instructions to re-select the analysis position in the **Sample>Position List Table** and Drive the stage to this position if the stage has been moved
- > *Continue below by modifying desired parameters*

19) Modify desired parameters.

20) 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>.pro
Subsequent **More** acquisitions will preserve previous files; acquire new data and save a new file with the **Filename**:
<"previous name"><+2>.pro

4 XPS Angle Profile

Angle-Resolved (angle-dependent) profiles are used to probe the near surface region of a platen (sample) in a non-destructive manner. **Survey** or **Multiplex** acquisition should have been performed and analysis areas have been defined (**Sample Session**) before Map acquisition.

- 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 Angle Application** window.
- 5) *Optional use of Angle Settings*
 - > Select a setting from the **Angle Settings** combo box.
 - > Click the **Load** button.
- 6) *Optional use of Previous Acquisition file Angle Settings*
 - > Click the **File...** box.
 - > Choose a **Angle** file with desired settings.
 - > Click **Open**. The **Angle Parameters** will update with the acquisition file settings.
- 7) Select the **Source** type from the combo box.
- 8) 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 the desired **UnScanned** or **Scanned** Analyzer Mode.
- 10) Select a **Pass Energy** and **Time Per Step (ms)/Time Per Region (ms)** combination that meets your needs.

*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*
- 11) 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 **Multiplex**, **Profile**, **Line** or **Map** acquisitions. Selecting the **Import Spectral Display** will import elements identified in the “highlighted” **Spectral Viewer** [elements identified with **Peak ID**]
- 12) 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.

- 13) 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 **Angle Regions** window.*

- 14) Select **Standard** or **Narrow Acceptance Angle** in the **XPS>Properties>Profile** window.

- 15) Select the **XPS>Angle>Angle Table** button to open the **Angle Table** window.

- > Use the edit keys to add the desired number of angles
- > Enter the desired **Angle** and **Cycles** for each angle

Note: The intensity response of the Versa Probe II varies with the sine of the platen (sample) tilt angle. For this reason, you should collect data for a longer time (more Cycles) at low angles.

- > Close the **Angle Table** window

*Note: the **Analysis Time**; **Runtime** and **Cycles** will be calculated as a function of the **Region** parameters, **Angle Table** parameters and **Angle Application** tab parameters.*

- 16) Click the **Start** button in the **Angle** tab.

- > The **Queue: Acquire Angle-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 for each analysis area with multiple traces [a set of traces for each region (or energy)]
- > Each file will contain **Spectral Data** and **Angle Intensity Data** for each **Region**
- > The Filename will be:
<filename>.<file number>.<platen name>.<position #>.<position list name>_n.ang
 with the **_n** indexed corresponding to each analysis area (e.g. **sample.101_1.ang** and **sample.101_2.ang**)
<filename>.<file number>.<platen name>.<position #>.<position list name>
 will remain the same for each analysis area file
- > The **SmartSoft-VersaProbe Spectral** data will be displayed in the **Spectral Viewer**. Each **Region** will be displayed separately within the viewer. There will be a **Spectral Viewer** for each analysis area.
- > The **Angle** intensity plot will be displayed in the **Profile Viewer**. There will be a **Profile Viewer** for each analysis area.

- > 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 Angle-1** window.

5 XPS Line

Lines provide a **two**-dimensional display of elemental or chemical state information. Typically, **Lines** are used in situations where it would be too time consuming either to collect a **Map** or to determine the detailed shape of an edge or interface. A **Line** is a single row of pixels or data points. A complete spectrum is collected at each pixel. These spectra are collected in what is called an “**UnScanned**” mode. That is, the entire spectrum is collected in one step, like a snapshot, which greatly reduces the time required to collect a line. **Survey** or **Multiplex** acquisition should have been performed and that analysis positions and analysis areas have been defined (**Sample Session**) before **Line** acquisition.

- 1) Select the desired **Active** analysis positions in the **Sample>Position List Table**.

Note: there must be at least one “line” analysis area selected

- 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 Line Application** window.
- 5) *Optional use of Line Settings*
 - > Select a setting from the **Line Settings** combo box.
 - > Click the **Load** button.
- 6) *Optional use of Previous Acquisition file Line Settings*
 - > Click the **File...** box.
 - > Choose a **Line** file with desired settings.
 - > Click **Open**. The **Line Parameters** will update with the acquisition file settings.
- 7) Select an **X-Ray Setting** picking a spot size that is similar in size to the features you expect to see in the **Line**.
- 8) Set the Pass Energy to **117.4 eV**
- 9) Select the desired **Time Per Pixel (ms)**
- 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 **Multiplex**, **Profile**, **Angle** or **Map** acquisitions. Selecting the **Import Spectral Display** will import elements identified in the “highlighted” **Spectral Viewer** [elements identified with **Peak ID**]

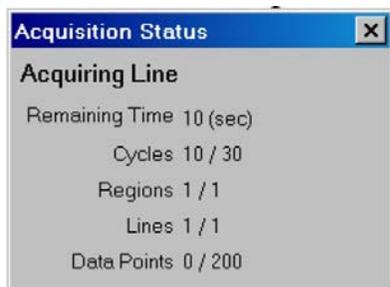
- 11) Select a number of **Sweeps** for each **Region**. Define a higher number of **Sweeps** for elements that are present at lower concentrations to optimize the acquisition.
- 12) Customize the setup of each Region if desired. Modify the energy range of an element or change the **Pass Energy** of a specific element.

Note: Unique **Pass Energy** settings can be applied for each **Region** using the **Pass Energy** in the **Region Table** area

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 **Line Regions** window.

13) Click the **Start** button in the Line tab:

- > The **Queue: Acquire Line-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 for each analysis area with multiple traces [a set of trace for each region (or energy)]
- > Each file will contain **Spectral Data**, **Line Intensity Data** and a memory map file for each **Region**
- > The Filename will be:
 - <filename>. <file number>.<platen name>. <position #>. <position list name>_n. lin**
 - with the **_n** indexed corresponding to each analysis area (e.g. **sample.101_1.lin** and **sample.101_2.lin**)
 - <filename>. <file number>.<platen name>. <position #>. <position list name>** will remain the same for each analysis area file
- > The **SmartSoft-VersaProbe Spectral** data will be displayed in the **Spectral Viewer**. Each **Region** will be displayed separately within the viewer. There will be a **Spectral Viewer** for each analysis area.
- > The **Line** intensity plot will be displayed in the **Image Viewer**. There will be an **Image Viewer** for each **Region** of each analysis area.
- > All **Active Line** analysis positions in the **Position List Table** will be visited sequentially.
- > To **Stop** or **Abort** the acquisition before all Cycles are completed, use the **Stop** button within the **Queue: Acquire Line-1** window.
- > Acquire **More Line**:
 - > Click the **More** button in the **Line** tab to add additional data to the **Stopped** or completed **Line** acquisition
 - > Add the more **Cycles** to increase the Analysis Time
 - Note:** If the **Line** acquisition was stopped; the current number of **Cycles** can be used to continue the acquisition*

- > Click the **Start** button
 - > The original acquisition file will be preserved with its previous name.
 - > One new file will be saved
 - > The new Filename will be
<"previous name"><+1>. lin
 - > Subsequent More acquisitions will preserve previous files; acquire new data and save a new file with the Filename:

- > **<"previous name"><+2>. Lin**

6 XPS Map

Maps provide a two-dimensional display of elemental or chemical state information. Maps consist of an array of individual pixels. A complete spectrum is collected at each pixel. These spectra are collected in an “**UnScanned**” mode. That is, the entire spectrum is collected in one step, like a snapshot, which greatly reduces the time required to collect a Map. **Survey** or **Multiplex** acquisition should have been performed and that analysis positions and analysis areas have been defined (**Sample Session**) before **Map** acquisition.

- 2) Select the desired **Active** analysis positions in the **Sample>Position List Table**.
Note: there must be at least one “area” analysis area selected
- 3) Enable the **Auto Z Before Acquire (XPS>Properties>Setup)** if the analysis positions have **NOT** been previously aligned
- 4) Enable the **Automated Neutralization (XPS>Properties>Setup or Status Bar)** if the sample is insulating
- 5) Open the **XPS Map Application** window.
- 6) *Optional use of Map Settings*
 - > Select a setting from the **Map Settings** combo box.
 - > Click the **Load** button.
- 7) *Optional use of Previous Acquisition file Map Settings*
 - > Click the **File...** box.
 - > Choose a **Map** file with desired settings.
 - > Click **Open**. The **Map Parameters** will update with the acquisition file settings.
- 8) Select an **X-Ray Setting** picking a spot size that is similar in size to the features you expect to see in the Map.
- 9) Set the Pass Energy to **117.4 eV**
- 10) Select the desired **Time Per Pixel (ms)**
- 11) 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 **Multiplex**, **Profile**, **Angle** or **Line** acquisitions. Selecting the **Import Spectral Display** will import elements identified in the “highlighted” **Spectral Viewer** [elements identified with **Peak ID**]

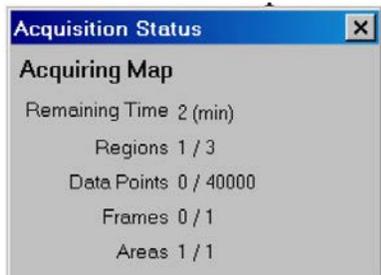
- 12) Select a number of **Frames** for each **Region**. Define a higher number of **Frames** for elements that are present at lower concentrations to optimize the acquisition.
- 13) Customize the setup of each Region if desired. Modify the energy range of an element or change the **Pass Energy** of a specific element.

Note: Unique Pass Energy settings can be applied for each Region using the Pass Energy in the Region Table area

*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 **Map Regions** window.*

14) Click the **Start** button in the Map tab:

- > The **Queue: Acquire Map-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.



- > Each **Region** will be acquired sequentially.
- > The Map acquisition begins. The incident X-Ray beam steps point by point along a line while “**unscanned**” spectral data is collected at each point. A pixel intensity is calculated using the **Peak Intensity** method that is selected in the **XPS>AcquisitionProperties>Region** tab. The spectral data and calculated intensity values are then saved for each pixel.
- > This is then repeated for the next line, and so on until all the lines have been acquired. This is one “**frame.**” This is then repeated for the next frame, at each point calculating and saving only the average of [**peak-minus-background** intensity at this point on this sweep] and [the value stored at this point during the preceding sweep]. When the specified number of frames/sweeps is completed, the acquisition for the next region (element) begins.
- > One file will be saved for each analysis area with multiple traces [a set of trace for each region (or energy)]
- > Each file will contain **Spectral Data, Map Intensity Data** and a memory map file for each **Region**
- > The Filename will be:
<filename>. <file number>.<platen name>. <position #>. <position list name>_n. map
 with the _n indexed corresponding to each analysis area (e.g. **sample.101_1.map and sample.101_2.map**)
<filename>. <file number>.<platen name>. <position #>. <position list name>
 will remain the same for each analysis area file
- > The **SmartSoft-VersaProbe Spectral** data will be displayed in the **Spectral Viewer**. Each **Region** will be displayed separately within the viewer. There will be a **Spectral Viewer** for each analysis area.
- > The **Map** intensity plot will be displayed in the **Image Viewer**. There will be an **Image Viewer** for each **Region** of each analysis area.

- > All **Active Area** analysis positions in the **Position List Table** will be visited sequentially.
- > To **Stop** or **Abort** the acquisition before all Maps are completed, use the **Stop** button within the **Queue: Acquire Map-1** window.
- > **Acquire More Map:**
 - > Click the **More** button in the **Map** tab to add additional data to the **Stopped** or completed **Map** acquisition
 - > Increase the **Sweeps Multiplier** to increase the Analysis Time
*Note: If the **Map** acquisition was stopped during the first **Region** of the first analysis area; the new Sweeps Multiplier will be used to continue the acquisition. This will result in the first **Region** completing with the new number of sweeps and the remaining **Regions** performed with the new number of sweeps*
 - > **Otherwise:**
*If the **Map** acquisition was **stopped** during the first **Region** of the first analysis area; the current Sweeps Multiplier will be used to continue the acquisition. This will result in the first **Region** completing and the remaining **Regions** performed.*
- > Click the **Start** button
 - > The original acquisition file will be preserved with its previous name.
 - > One new file will be saved
 - > The new Filename will be
<"previous name"><+1>.Map
 - > Subsequent More acquisitions will preserve previous files; acquire new data and save a new file with the Filename:
<"previous name"><+2>.map