

# Standard Operating Procedure I: XPS Spectral Measurement



Yale West Campus  
Materials Characterization Core  
[ywmatsci.yale.edu](http://ywmatsci.yale.edu)

ESC II, Room A119E  
810 West Campus Drive

Version 2.01, April 2024



- > **FOLLOW** the SOP strictly to keep the instrument in good condition. **No** explorations allowed on software unless permitted by lab manager.
- > **NEVER** use your own USB drive on the XPS computer. Data can be either uploaded to Yale OneDrive or copied to the Jump Drive provided by the Core.
- > **NEVER** surf the web on the XPS computer to minimize the risk of the computer being hacked.
- > Users should **acknowledge** MCC in their publications. Please check the following link for details:  
*<http://ywc matsci.yale.edu/publications>*
- > The core reserves the right to use the data for core promotion.



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## PHI VersaProbe III AD XPS/UPS Standard Operating Procedure (Basic XPS Measurement)

### 1 Introduction

#### 1) Instrument Features:

- > 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 A119E  
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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. **No** explorations on program allowed unless approved by core manager.

## 2 Initial System Status Check

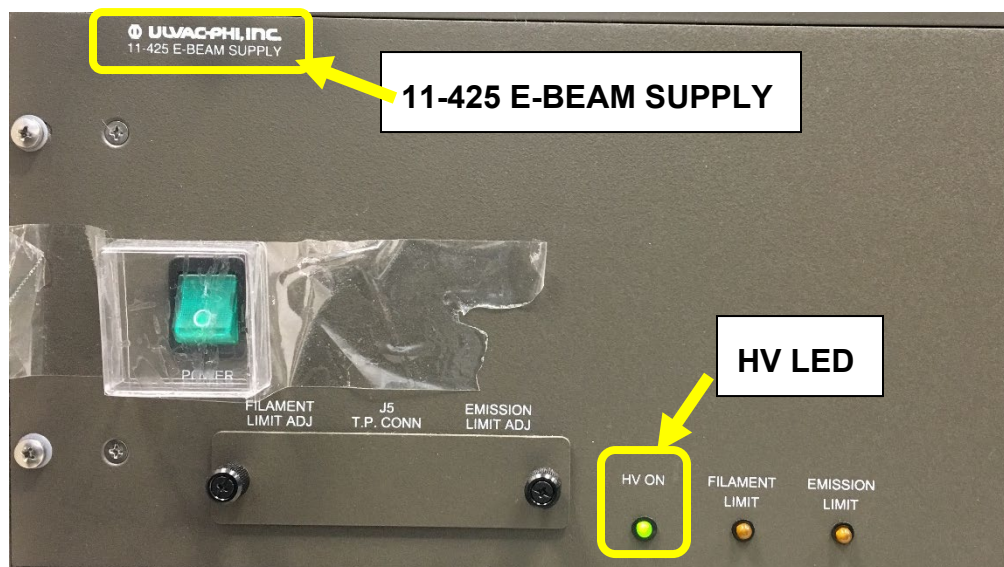
**Warning:** Before sample preparation and loading, the users are required to check the XPS system status following the steps listed below:

- 1) Check on the highlighted panel on the XPS control rack and make sure the main chamber pressure is **below  $5 \times 10^{-7}$  Pa** before getting started with sample preparation. Report to Core manager immediately if the pressure is in high  $10^{-7}$  or in  $10^{-6}$  Pa range.

**Warning:** the **higher pressure** indicates either **stage outgassing** or **chamber leaking** which will lead to x-ray source and detector damage during operation.



- 2) Check the “HV” high voltage LED light as highlighted in the picture below on the **11-425 E-BEAM SUPPLY** box as highlighted below and make sure is **ON in green**. If the LED light is off, stop sample preparation and report to manager immediately.



- 3) Check the building supply  $N_2$  and **Compressed Air (CA)** pressure gauges as labelled on the back wall and make sure the pressure reads  **$\sim 100$  psi** on both gauges. Report to manager immediately if any of those gauges reads **zero**.

**Warning:** Do not adjust the regulators on top of those gauges.





### 3 Sample Preparation

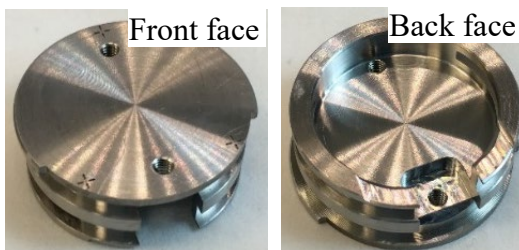
#### 3.1 Sample preparation in user lab

Make sure the samples are **dried** completely before coming. The following methods are recommended for sample preparation in your lab:

- 1) Always wear **gloves** when handling XPS samples,
- 2) Leave samples inside vacuum overnight,
- 3) Bring samples with a portable desiccator if possible to isolate samples from ambient moisture.

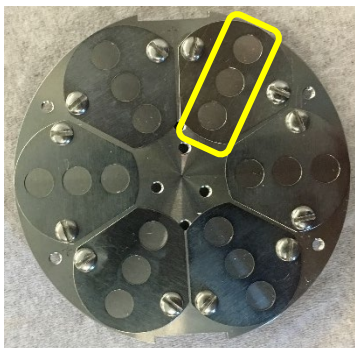
#### 3.2 Sample holder types

- 1) **25 mm** regular sample holder for **as-is** sample analysis. This is most used sample holder which can hold more than 15 samples of 3 x 3 mm<sup>2</sup> size



- 2) **60 mm** large sample holder for **as-is** sample analysis. This holder is good for large samples or more small samples. The three-hole metal mask can be used to ground and fix samples.

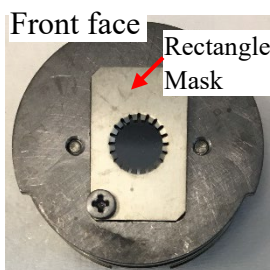
**Warning:** Never use 60 mm holder for XPS scan at tilted angles. The analyzer input lens will be hit by the holder when tilted.



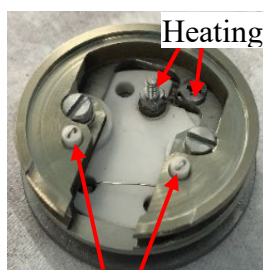
3) **Angle-4 position** sample holder. This holder is for **Angle Resolved (AR) XPS/UPS**.



4) **25 mm hot** sample holder used for *in situ* heating at the range of RT – 800 °C



Rectangle Heater

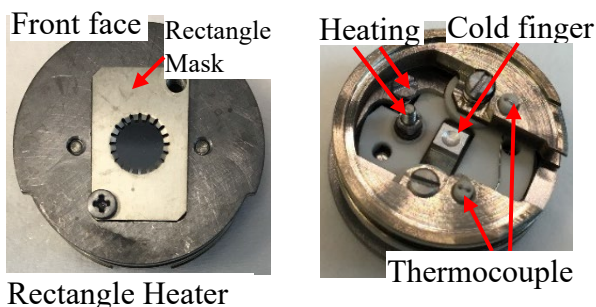


Thermocouple

**Warning:**

- > Never put any sticky tapes on the holder. Use the **rectangle mask** only with **ceramic screws**.
- > Choose the right size cross screwdriver for sample mounting. The screws can be easily damaged.
- > Choose the right type of holder in **HC Control** program and keep Reading Heater Power below **75%**. **Otherwise the stage will be damaged/overheated in main chamber.** User's account will be suspended, and repair cost will be charged on PI's account.

- 5) **25 mm hot/cold** sample holder for *in situ* cooling and heating required in the range of -140 °C – 600 °C



### 3.3 Sample mounting

- 1) Make sure the **maximum** sample height is **below 5 mm** above the holder surface as shown below



- 2) Make sure all sample heights are **within 1 mm difference** once mounted on the holder. You can put substrates under lower samples. **The analyzer will be hit by higher samples if the active measurement is on lower samples.**
- 3) The **minimal** sample size should be **~3 x 3 mm** for XPS and **5 x 5 mm** for UPS (UV beam size on the stage is ~ 4 x 4 mm).
- 4) **For solid samples:**
- Fix sample using removable double-sided Scotch tape provided in the lab. The Sample cannot be heated in vacuum, or
  - Fix sample with copper clips or metal cover on the sample holder. The sample can be heated in vacuum, or
  - After sample mounting, **shake and tilt** sample holder to make sure samples are fixed on the holder.
  - Blow** the sample surface with dry N<sub>2</sub> inside fume hood
- 5) **For powder samples:**
- Use **Scotch removable double-sided tapes** provided by lab:
    - > Cut and paste a slightly larger tape square (~5mm) onto holder,
    - > Apply enough powders on the tape, cover the sample surface with weighing paper and press and turn firmly the surface with glass slide
    - > **Blow** the sample surface with dry N<sub>2</sub> inside fume hood
  - Use **Si chip** as substrate:
    - > Add powder in solvents such as ethanol, isopropanol (IPA) or Dichloromethane (DCM) and mix the solution using ultrasonicator.

- > Drip small amount of suspension onto Si chip (typically 3x3 ~ 5x5 mm<sup>2</sup>)
- > Dry sample about 10 minutes under infrared lamp
- > Repeat above steps until enough samples can be seen on Si,
- > Sample mounting:
  - > Fix sample on the holder either with Scotch tape, or
  - > If sample surface needs to be grounded, use provided metal cover to fix the sample.
- c) **Blow** the sample surface with dry N<sub>2</sub> inside fume hood
- d) **Warning: DO NOT use the holder if dropped to the ground.** Use a new holder and inform manager immediately for repair.
- e) **Warning: DO NOT label samples on the holder with mark pen.** Instead, make a sample holder drawing on the paper. Volatile inks from mark pens will cause severe contamination to Ultra High Vacuum chamber.
- f) **Warning: DO NOT use carbon or copper tapes on the sample holder.** It will take hours to reach the required vacuum in the main chamber.

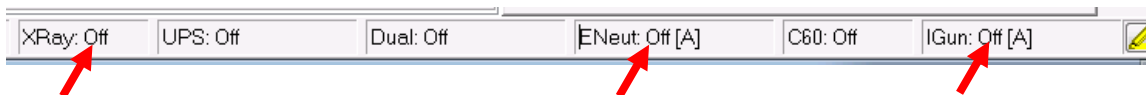
#### 4 XPS Computer Login

- 1) Login FOM Program: click on **Login with Yale NetID** for **internal users only** to unlock the computer screen.

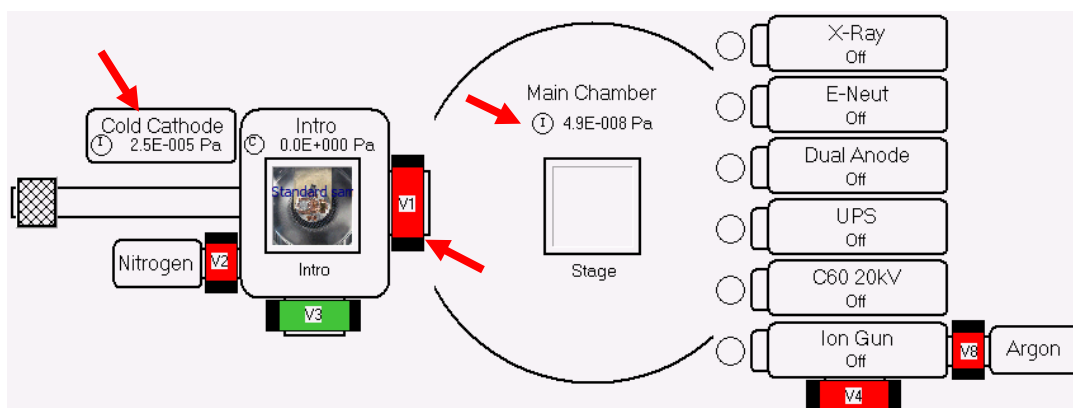


- 2) Check system status:
  - a) Check the bottom right status bar on the **SmartSoft** program window below and make sure:
    - > X-ray source (**XRay**) is at **Off** or **Park** (if < 60mins from previous session).
    - > Electron Neutralizer (**ENeut**) is at **Off** or **Standby** (if < 60mins from previous session).
    - > Ion gun (**IGun**) is at **Off**.

**Warning:** If **IGun** is at **ON**, or a **red border** appears around any of these areas, **Inform** manager immediately before proceeding. You may restart the software if permitted by manager. Never use the Task Manager to terminate the software.



- b) In system diagram below, check if "V1" valve is **closed** (in **red background**) between **Intro** and **Main Chambers**. If not, **contact** lab manager immediately. You may restart the software if had experience before.

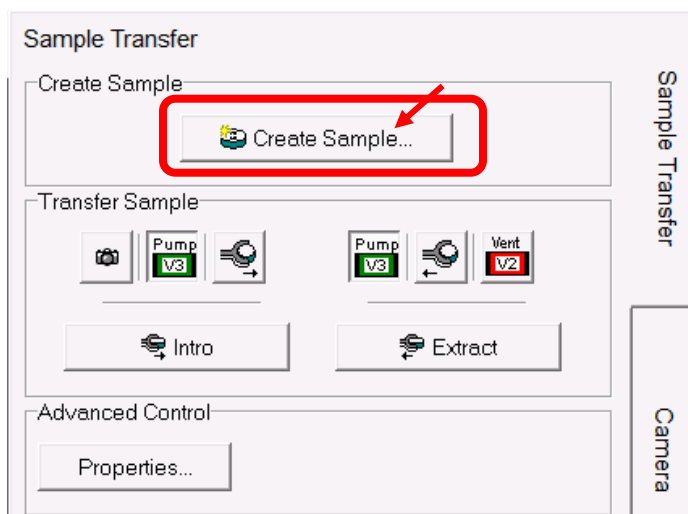


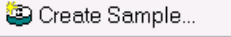
- 3) Sign in on the logbook and write down the **Main Chamber** and **Intro Chamber** initial pressure on Cold Cathode gauge as highlighted in above diagram before sample loading.
- 4) **Report to manager immediately** if the Cold Cathode pressure gauge reads above 4E-4 Pa.

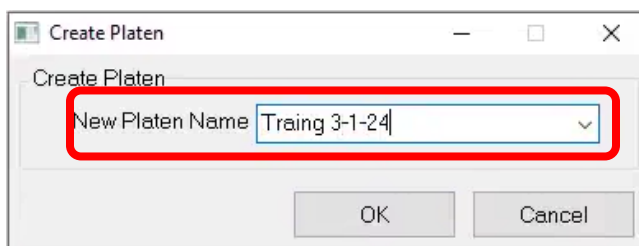
## 5 Create Sample in SmartSoft Program

- 1) Click  **System** tab on top of **SmartSoft-VersaProbe** program to enter the **System** window, then click the **Sample Transfer** tab on the right side to enter **Sample Transfer** tab as shown below.

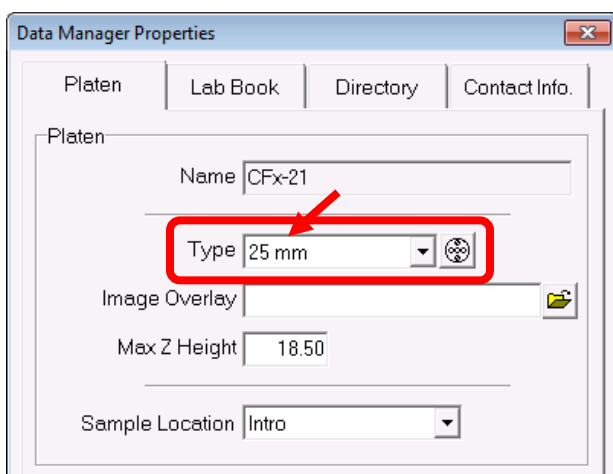
**Warning:** the software should never be closed after use. If software restart is required, click the icon on the desktop and click **OK** to open. No password required.



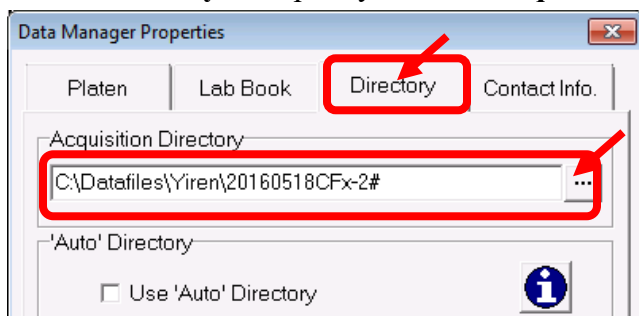
- 2) Click  button on above window to open **Create Platen** (sample) window below. Enter a new platen name and hit **OK**. **Do not** use previous platen name which was affiliated with previous sampling position info.  
**Warning:** **NEVER** change or create a new platen name in the middle of data collection. This will **lead to hardware damages** inside vacuum.



- 3) In the popup **Data Manager Properties** window below, choose the right type of sample holders.
- Select **25 mm** for regular **25 mm** sample holder.
  - Select **60 mm** for regular **60 mm** sample holder.
  - Select **Angle-4 position** for **Angle Resolved** sample holder.



- 4) Click **Directory** and specify the data **Acquisition Directory**



- 5) Hit **OK** and **Close** above window.

## 6 Sample Loading into Intro Chamber

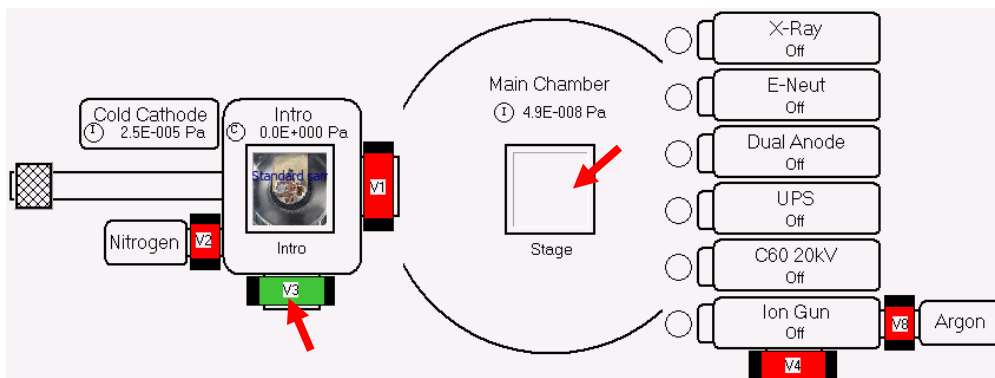
**Warning: DO NOT use the holder if dropped to the ground.** Use a new holder and inform manager immediately for repair.



- The sample holder must be **cleaned** with nitrogen gas to remove any loose particles, hair or fibers that may exist on the holder.

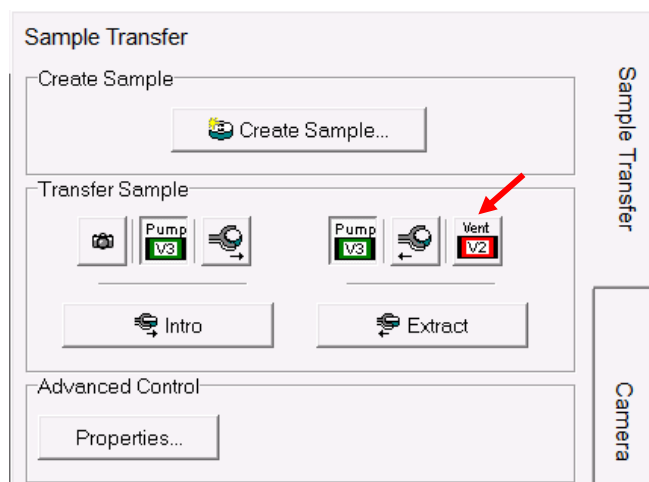
- 2) Turn **ON** the Main Chamber light.



- 3) Confirm **no samples** inside Main Chamber:
  - a) Look through the view port to confirm no samples on the stage in the Main Chamber, and then turn off Main Chamber light. **If samples found on the stage**, go to **Page 29, Section 9: Closing XPS measurement** to extract the samples to the intro chamber.
  - b) Check the **system diagram** below and make sure that the **Stage is empty** as highlighted and the sample photo is inside the Intro chamber as shown below:  
**Warning: Never** leave samples in Main Chamber unless after overnight scans. The sample can be left inside the intro chamber after finish, which will be discarded by next user.



- 4) Turn **OFF** the Main Chamber light.
- 5) In above system diagram in, check if  (V3) valve is in green indicating the **Intro Chamber** being pumped, then click  in the **Sample Transfer** window below:



- 6) **Right after** venting started, put on gloves and hover on the glass cover. **Do not** touch cover.
- 7) Remove the glass cover and place the backside on the XPS table with a clean Kimwipe underneath.

**Warning:**

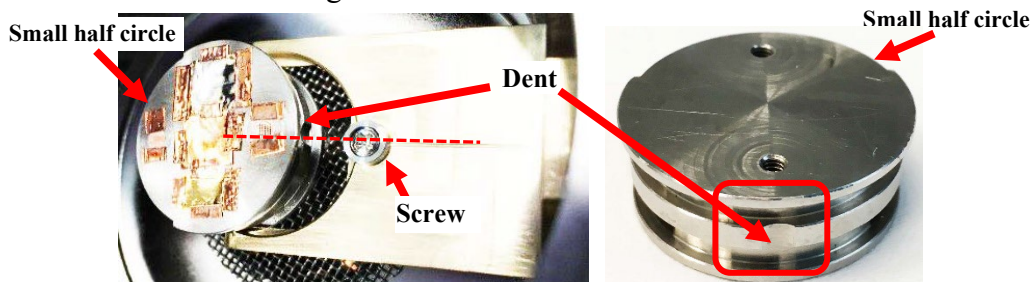
- > **Never** touch the sample loading port with exposed skins,
- > **Never** touch the **camera** atop the loading port,
- > The sample holder must have been cleaned with **nitrogen gas**.

**Serious chamber leaking or pump damage** might happen if any fibers or particles are sucked into vacuum.

- 8) Sample loading into the Intro chamber:

**For 25 mm holder:**

- a) Finger grab the holder with the small half circle facing toward the Main Chamber and slide the bottom groove onto the transfer arm fork.

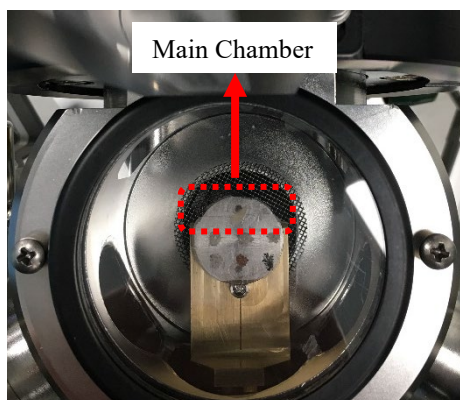


- b) Check the pictures above. With the sample on the fork, make sure the **dent** on sample holder **touches** the **screw** on the fork.
- c) Check the holder from side window and make sure the holder stays **flat** on the fork as shown below:



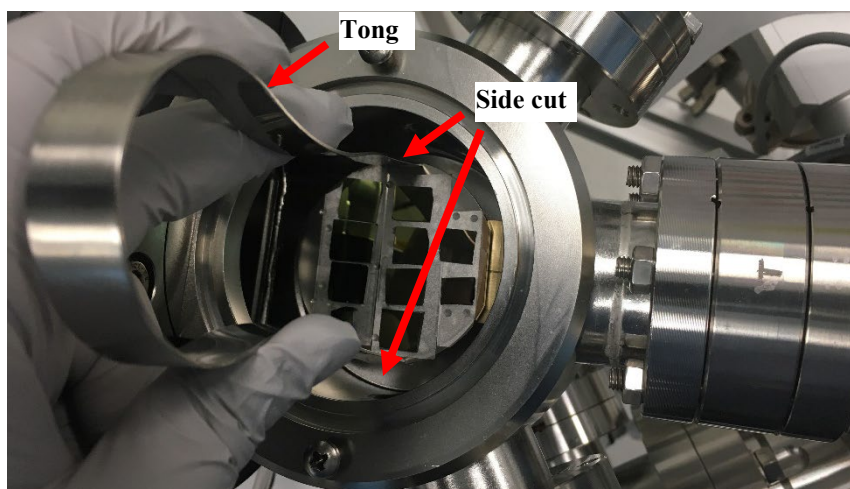
- d) Check the **top view** below, make sure the sample holder is **symmetrically placed** on the fork and the **small half circle** (dashed box below) is close to the Main Chamber.



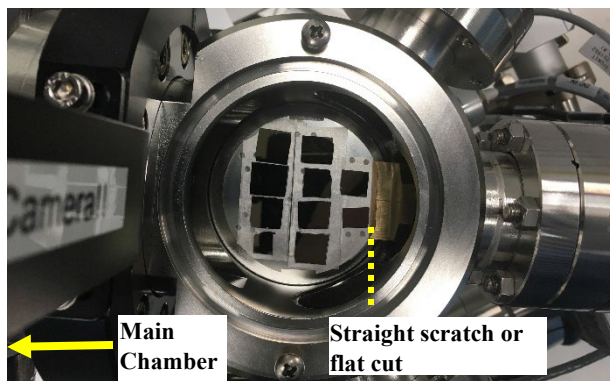


**For 60 mm holder:**

- a) Use the tong to grab the side cut of the holder and slide the bottom groove onto the transfer arm fork as shown below:



- b) Make sure from the top-view below that the **scratched straight line** or the **flat cut** (new 60 mm sample holder) on the sample holder is on the right side (away from the

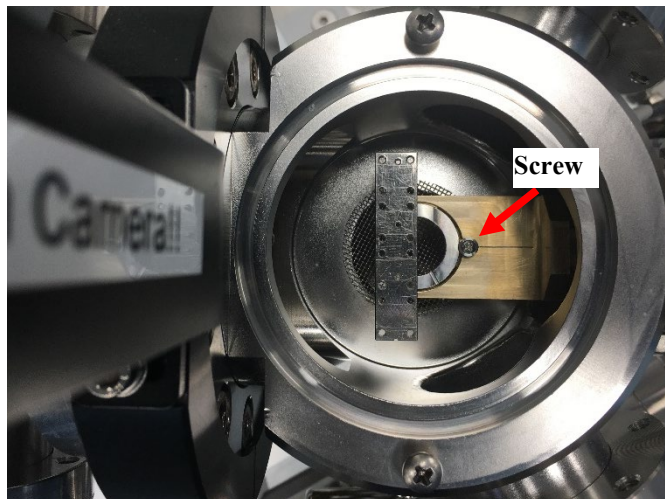


main chamber).

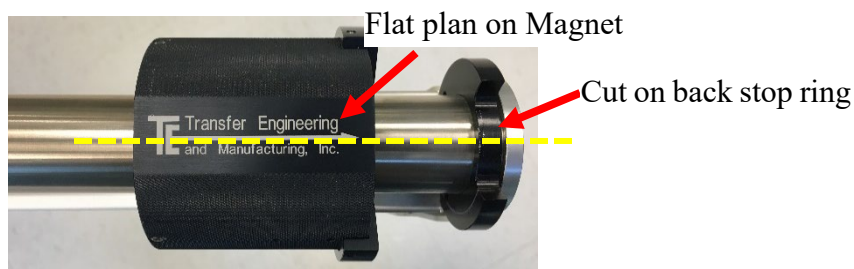
- c) Hand adjust/turn the sample holder slightly and make sure the holder can be stopped by the screw on the transfer rod.

**For 4-position rectangle holder:**

Insert the holder with the dent on the bottom touching the screw on the transfer rod. The rectangle holder should be on the left side (close to the main chamber) as shown below:





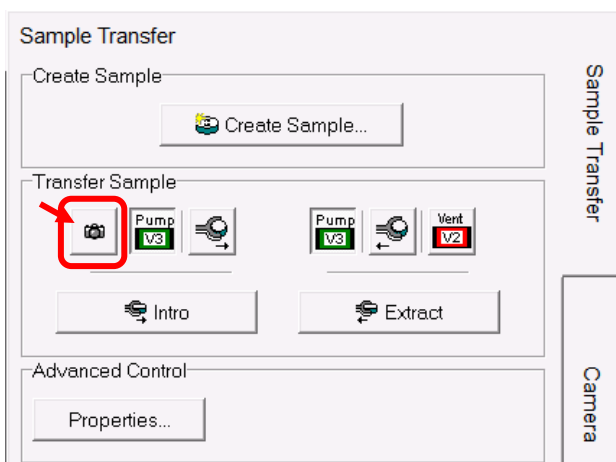
- 9) **Fully** retract the transfer arm with the magnet and let the flat plane **face upward**. Make sure the flat surface aligns the open cut on the back stop ring as highlighted by the dashed line below:





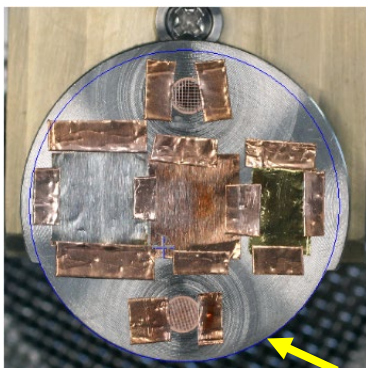
- 10) Check to make sure no particles/fibers inside the groove of the loading port. If found, clean with Kimwipes and IPA.
- 11) Blow the inner side of glass cover (see picture below) with nitrogen and place back to the loading port. **Make sure** the cover can be pressed into the groove smoothly.





- 12) In the **SmartSoft** program  **System** window below, click  button on the **Transfer Sample** window to take a picture of the sample holder:

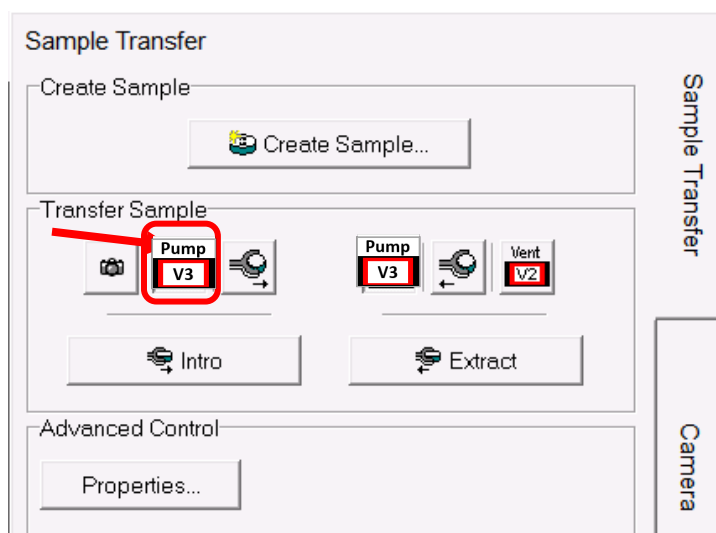


- 13) Pay attention to the camera shutter sound **Wait for several seconds** for picture loading into the program. Be patient, **NEVER** double click camera button.
- 14) Click the  **Sample** tab next to  to enter the **Sample** window. Check the sample



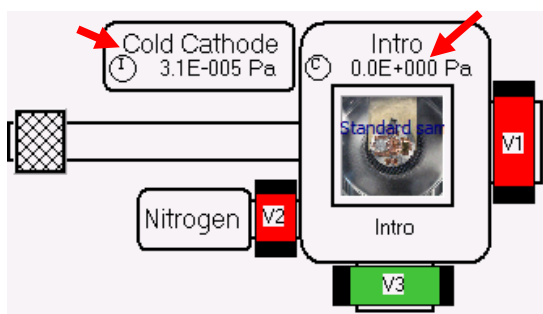
holder photo and make sure the **blue circle** aligns well with the **bottom half** (smaller circular part) of the **25 mm** sample holder. If not, contact lab manager. For **60mm** and **Angle-4 position** holder, the blue circle/rectangle should match the entire holder.

- 15) Click  **System** and click  button on the **Sample Transfer** window below and **quickly go back** to the loading port; **hold and press** the glass cover until pumping starts after air hissing sound.
- Warning:** The **Intro Chamber** might be leaking if the cover is not pressed during pumping.



16) Monitor the **Intro Chamber** pressure change on the system diagram:

- The **Intro** pressure gauge reading as highlighted below should **drop** from atmosphere pressure to  $\sim 3.0\text{E}+001$  Pa in  $\sim 5$  minutes;
- The **Cold Cathode** gauge should start reading from  $\sim 1\text{E}-002$  Pa;
- Wait until the pressure drops to  $5.0\text{E}-004$  Pa before sample transfer can be started. Typically, it takes  $\sim 15$  minutes for dry samples. If it takes longer than **one hour**, consider removing samples from intro chamber for further drying treatment.




## 7 Sample Transfer into Main Chamber

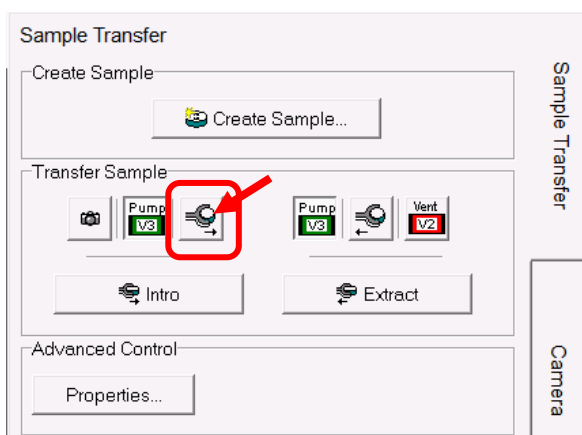
- Check and make sure the **Cold Cathode** gauge pressure is **below  $5.0\text{E}-004$  Pa**.

- 2) Turn on the **Main Chamber** light.

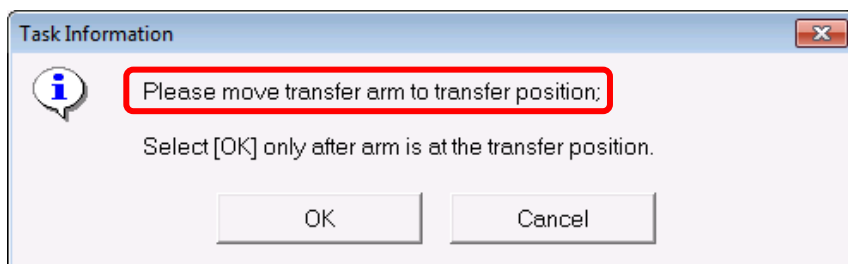


- 3) **Wear glove** on right hand for sample transfer.  
 4) Read through **Step 5) to 12)** below before proceeding.  
 5) Click  in the **Transfer Sample** window below to start sample transfer from Intro Chamber to Main Chamber

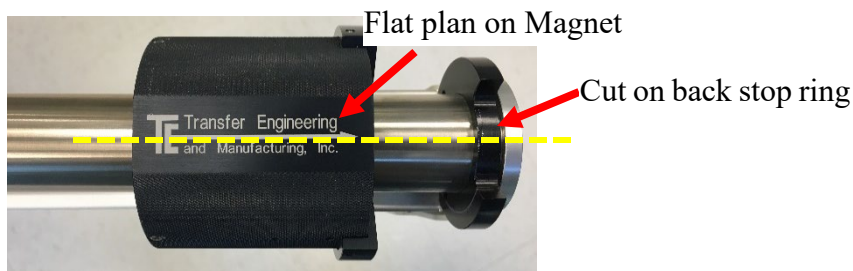
**Warning:** NEVER stop the transfer process in the middle and **force to close** the **SmartSoft** program in **Task Manager**. Contact Core manager immediately if did by accident.



- 6) Wait for the following window to appear. **DO NOT click OK button** on the window below:



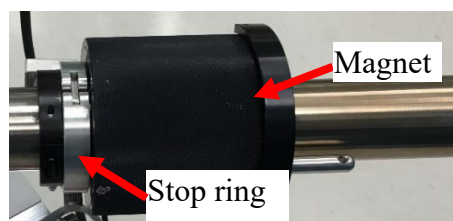
- 7) Check and make sure the flat surface aligns the open cut on the back stop ring as highlighted by the dashed line below:



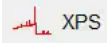
- 8) Grab the magnet with the right-hand thumb placed on the left side of magnet and **slowly** move toward main chamber until finger touches the stopper ring as show below.

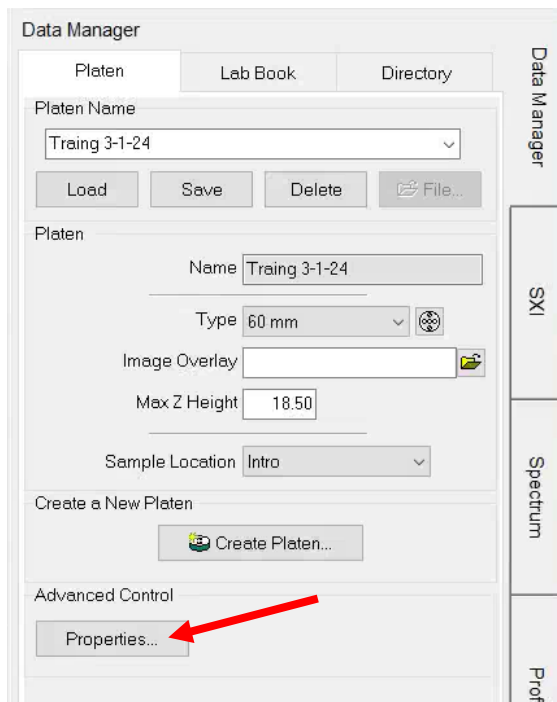


- 9) Watch the sample holder in main chamber, retract the thumb and **move very slowly** until the magnet touches the front **stop ring** (see picture below).

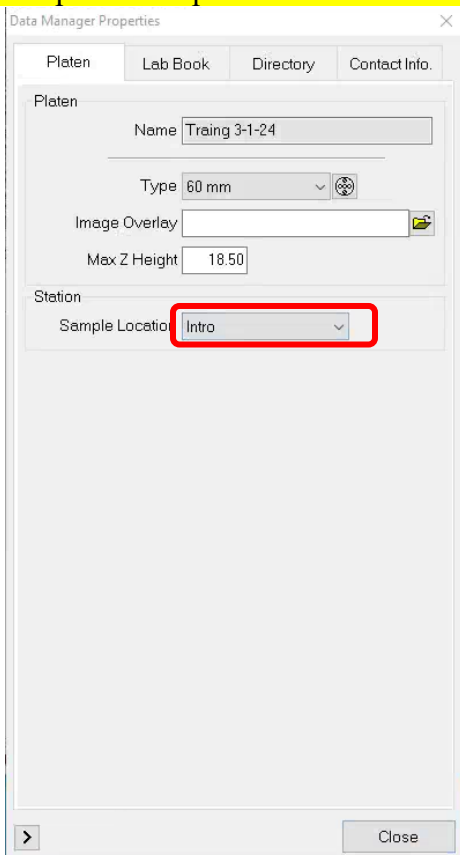



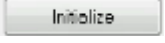
- 10) **Watch closely the main chamber stage and a popping sound** should be heard from underneath the sample holder while the holder is locked by a pushup part from the stage. **If not**, perform the steps a) - e) below:

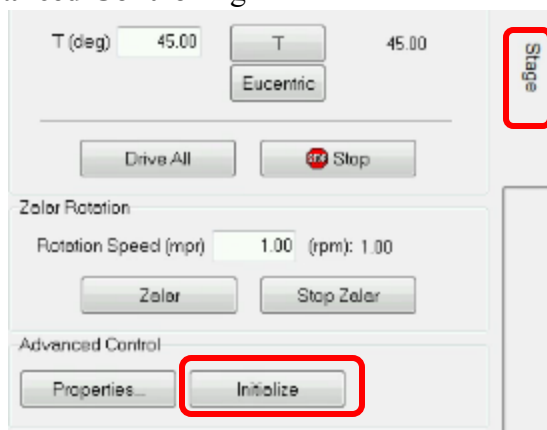
- a) **Check if there is still a larger gap (>1 mm) between magnet and front ring** in the image above:
  - > **DO NOT force** the transfer rod into the main chamber stage to **DAMAGE** parts.
  - > **Retract** the rod slowly **with sample holder** back into Intro chamber and click OK on popup windows to close the V1 valve.
  - > **Inform** manager immediately for help.
- b) **Under the manager's approval and instruction:**
  - > Click the top  tab and click the **Data Manager** tap on the right side to open the **Data Manager** side window as shown below. Click the highlighted **Properties** button. **Note:** the sample holder picture has been moved to Main Chamber after initial transfer failure as shown below:



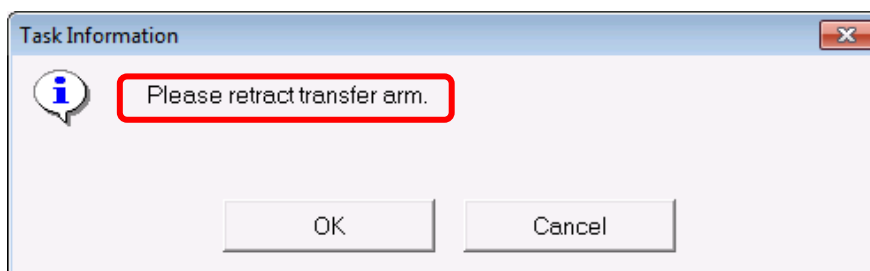
- > In the popup **Data Manager Properties** window below, choose **Intro** in the dropdown list for **Sample Location**. Then click Close button at the bottom. The **Sample holder** picture will be moved into **Intro Chamber**.



- c) Click  **Sample** tab to enter the side **Stage** menu and click  in the **Advanced Control** region as shown below.



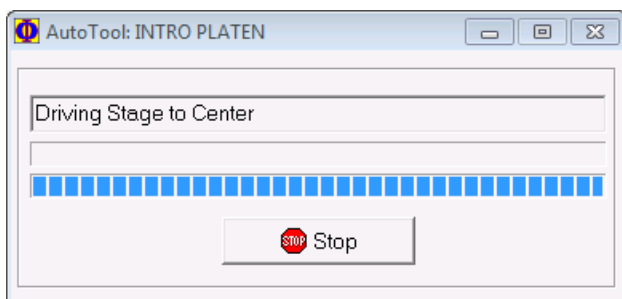
- d) Wait for “The stage initialize complete” window to appear and click OK button to finish. It should take **< 5 minutes**.
- e) Try transfer again. **If still feels a slight resistance** before hearing a popping sound, retract the rod with the sample holder, close the **V1** valve and **Contact** manager immediately for help.
- 11) **Keep** the transfer arm at the transfer position with magnet touching stopper ring, **go back** to the computer and click **OK** button on the popup window in **Step 6)** above.
- 12) Wait the following **window** to appear. **DO NOT click OK!** on the window below:



- 13) **Go back** to the chamber, watch the holder in main chamber. **Rest right hand** on the transfer arm and **very slowly** pull back the arm from the stage to **avoid** sample holder being dragged away/dropped from the stage. Once the front fork leaves the stage move faster to fully retract the transfer arm.
- 14) **Go back to the computer** and click **OK** on the popup window in **Step 12)** above.



15) **Wait until** the stage is driven to the center.



16) **Note** that the sample holder picture should appear inside the main chamber after finish.

17) **Turn off** Main Chamber light.

18) Wait ~ **5 - 30 mins** for the main chamber pressure to reach **5E-007 Pa** before proceeding to **XPS Scan**.


19) Consider **liquid nitrogen** cooling on the stage with under following circumstances:

- If the pressure stays in the **10E-6 Pa** range for more than **30 mins**,
- If the pressure rises to **higher E-6 Pa** right after transfer.

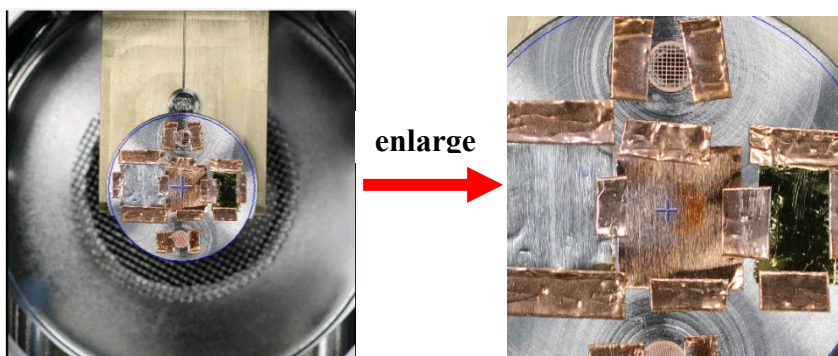
## 8 XPS Scan

### 8.1 Stage Z-Alignment

1) Check and make sure the **Main Chamber** pressure reaches below **5E-007 Pa**.

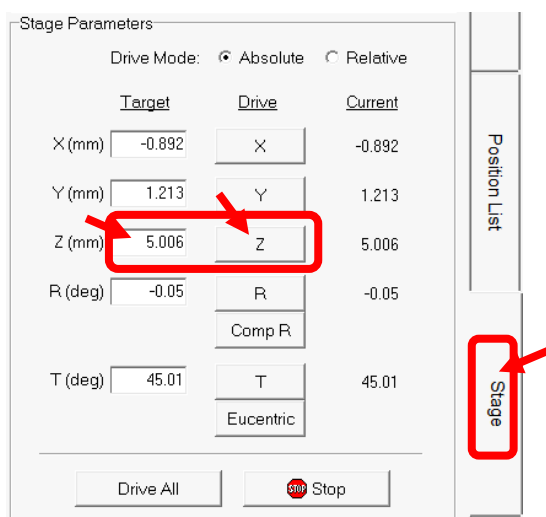
2) Click  **Sample** tab on top of program to enter **Sample** window.

3) **Hover** mouse cursor over the sample photo window on the left, **scroll** mouse wheel to enlarge photo and find the sample to be analyzed.

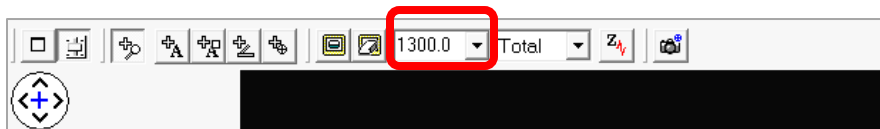


4) **Right click** mouse on the first intended analysis area and select **Drive To Click** on the dropdown menu, the **yellow box** should move to the selected area. **Do Not** drag yellow box.

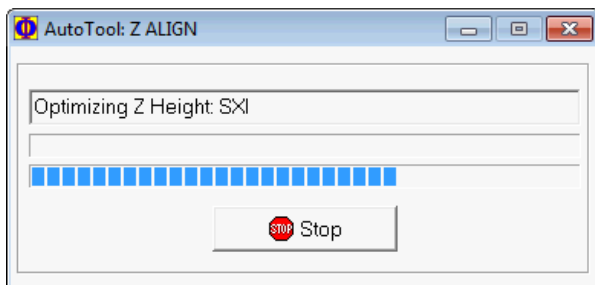
- 5) Click **Stage** tab on the right side of **Sample** window to enter **Stage Parameters** window as show below:



- 6) Input initial stage Z height in above window:
- > For **25mm** and **60mm** holder:
    - > For thin samples (lower than 1 mm), put **15** inside **Z(mm)** space in above window and hit **Enter** on keyboard.
    - > For thick samples, put **12 mm** instead.
  - > For **4-position** rectangle holder or thick samples on **25mm** and **60mm** holders, put **12 mm** instead.
- 7) Click  button next to **Z(mm)** space and wait for the sample stage moving to entered heights next to  button.
- 8) Choose **350** in the dropdown list of the window below.



- 9) Click  button as highlighted on top of **SXI** image window above to start Z-Align
- 10) Watch closely several windows popping up including **Ion Neutralization Standby**, **Turning E-Neut On**, **Ion Neutralization On** and **Filament Startup**. Report to Core manager immediately if the Z-Align stops at any of those windows for more than **5 minutes**.
- 11) Wait for the following **Z Height** window to appear and watch the Z height change in the space next to  button. The typical stage Z heights after alignment should be ~ **16-18.5 mm**, sample thickness dependent. The **sum** of **sample thickness** and aligned **stage Z height** should be slightly less than **18 mm**. If the stage Z height dropped back to the initial values (15, 12 mm), it indicates the failure of Z Align. Please repeat and contact manager if needed.



## 8.2 Create multiple sample positions

- 1) After Z-Align, **right** click inside the **yellow box** on the **Sample Photo** and select **Create Point at Stage** to create the first sampling position as shown in the position list below:


Active ID	Name	Comment	Type	U	V	Z	Rotation	Tilt
<input checked="" type="checkbox"/> 1			Point	-24.420	6.088	5.000	0.00	45.00

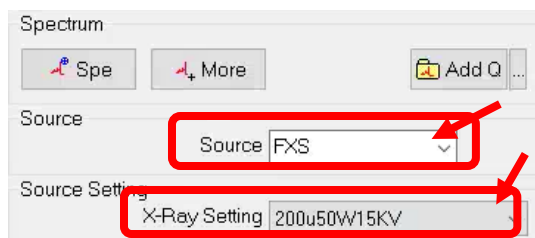
- 2) Input the position name in the **Name** column and choose **Point** in the **Type** column as highlighted below:

Active ID	Name	Comment	Type	U	V	Z	Rotation	Tilt
<input checked="" type="checkbox"/> 1	Sample 1		Point	-24.420	6.088	5.000	0.00	45.00


- 3) **Right** click on other samples or interested areas and select **Create Point** to define all other scan positions.

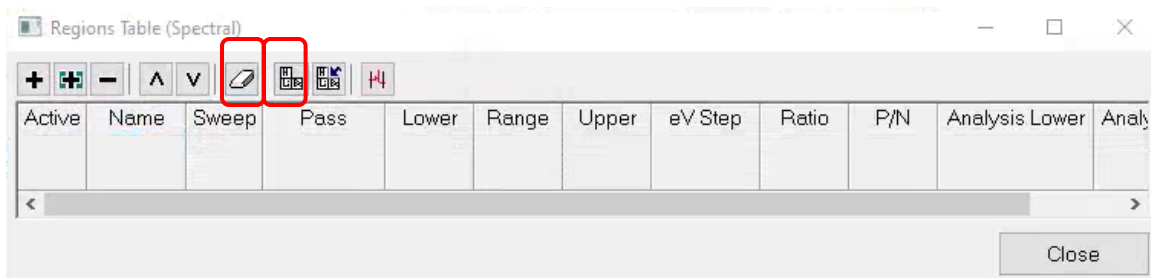
## 8.3 XPS Survey (Full Range) Scan




- 1) Click  XPS tab on top tab menu to enter XPS analysis window and click **Spectrum** tab on the right side.
- 2) In **Source** window below, choose **FXS** (Focused X-ray Source), and in **Source Setting** window choose **200u50W15kV** (X-ray beam size: 200  $\mu$ m, power: 50 W, and e-beam energy: 15 kV).



- 3) If sample is sensitive to X-ray radiation, e.g. XPS peaks shift or widen during scan, choose **100u100W20kV\_HP** to minimize sample damage and go back to **Step 2)** in **Section 8.2** to choose **HP** in the **Type** column in the sample Position List.

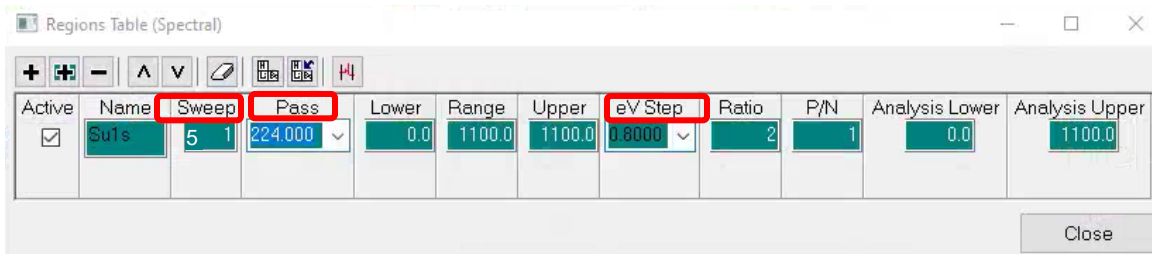
- 4) Click  on top left of **Regions** window to enlarge the **XPS Regions (Spectrum)** window as shown below:



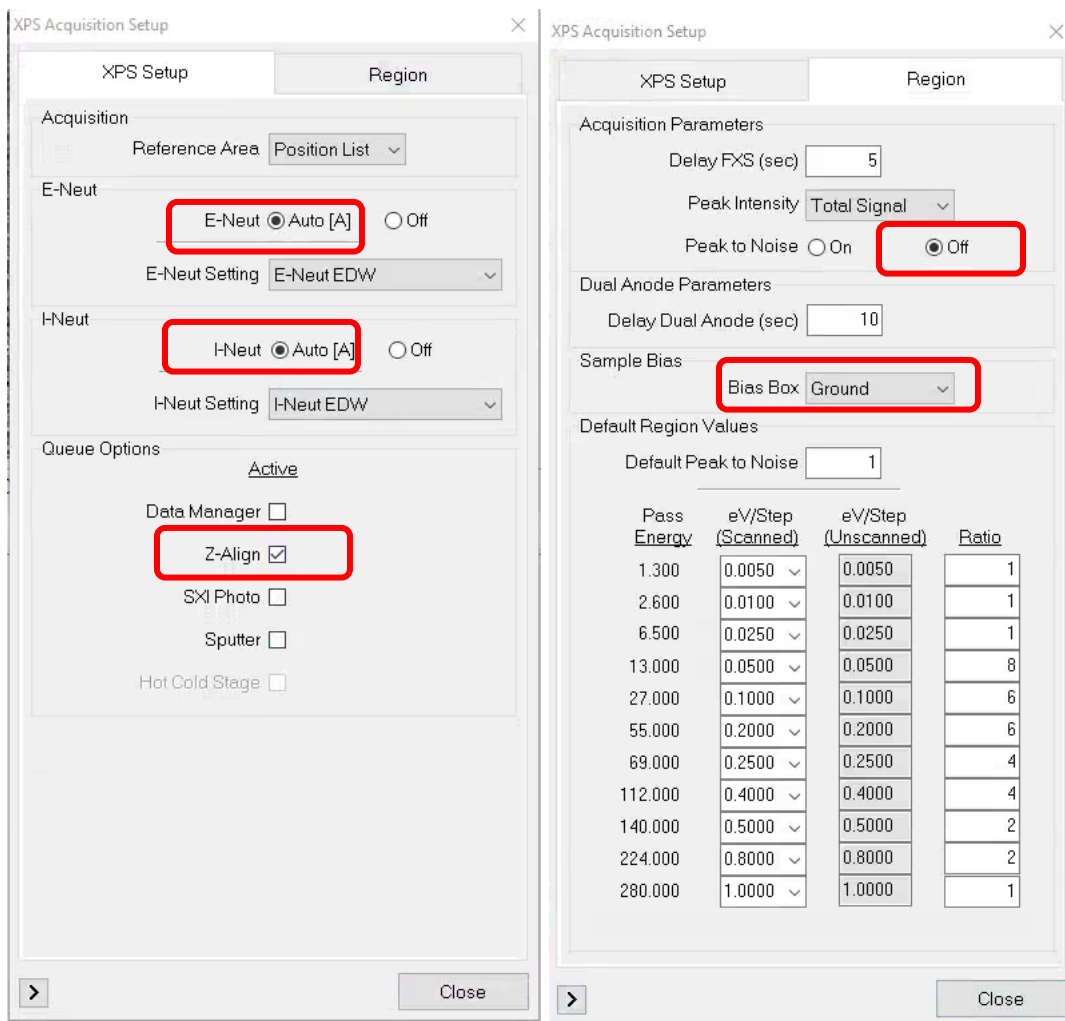
- 5) Click  in above window to erase previous setup parameters.
- 6) Click on  button on the window above to open the Periodic Table below and click  to start a **Spectral Survey (Su)** Scan.



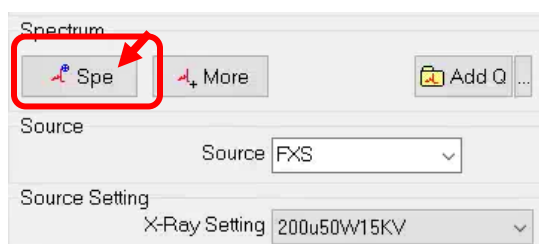
- 7) Click **OK** button on the **Periodic Table** to close the window.
- 8) Scan parameter setup in the popup **XPS Regions** window:
- Input the number of sweeps/scans in the Sweep column as highlighted below, typically choose **5 times**; consider 10 times or more sweeps for noisy spectrum.
  - Input pass energy value in the Pass column, typically **224.0 eV**; 140.0 eV and 112.0 eV can also be used to improve spectral resolution, but more sweeps is needed for smooth peaks. **Do Not use 280 eV** as it creates very wide peaks.
  - Input scan steps in the eV Step, typically **0.8000 eV**; **0.4 eV** can also be used for higher resolution.
  - Close** the popup **XPS Regions** window.



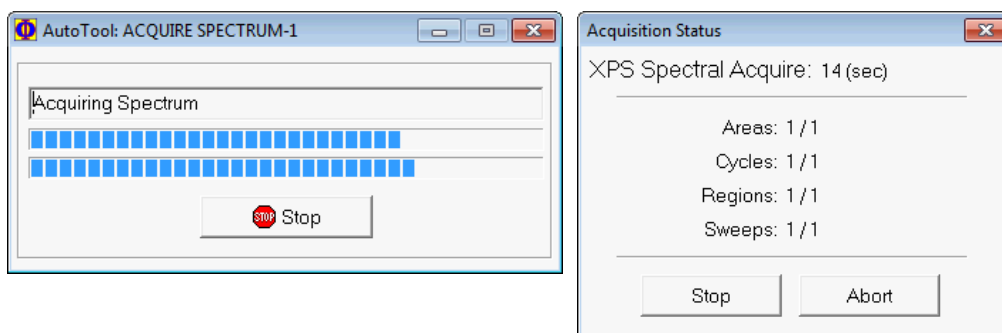
- 9) Click on **XPS Setup...** button on the bottom of the **Spectrum** window to open **XPS Acquisition Setup** window and **choose highlighted options** in the window below:



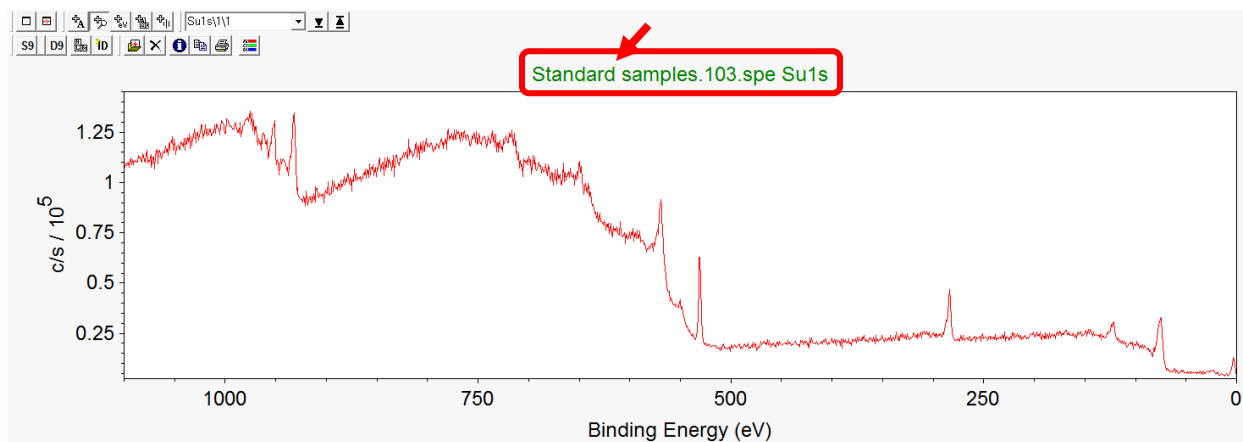
- 10) Hit **Spe** in Spectrum window below to start Survey scan



11) Wait the following windows to appear:



12) The **Survey** spectrum will appear in the **Spectral window** below. The filename as highlighted below is in the form of “**platen name.serial number.spe**” followed by scan type. Scan type is Su1s for survey.

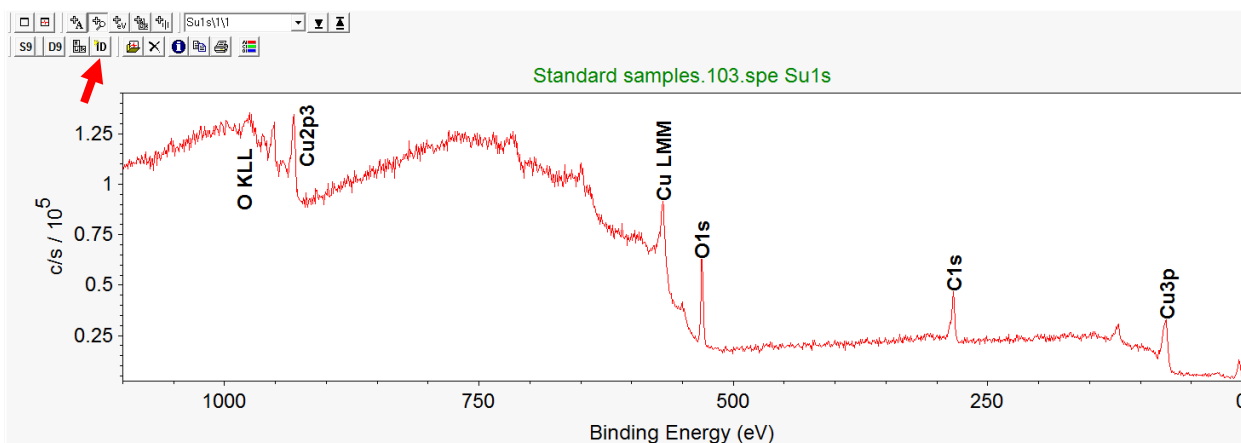


13) The data files will be automatically saved into specified folder which can be viewed using **MultiPak** program.

14) If the spectral acquisition fails due to Z-Align failure on specific samples, follow the instructions in **Section 8.1 Steps 6) to 10)** to perform manual Z-Align.


### 8.4 Regional (Elemental) Scan

1) Once survey scan is complete, click  on the Survey spectrum to start **Peak ID**



2) Click  on top left corner of **Regions** window to enlarge the **XPS Regions (Spectrum)** window below:

Active	Name	Sweep	Pass	Lower	Range	Upper	eV Step	Ratio	P/N	Analysis Lower	Analysis Upper

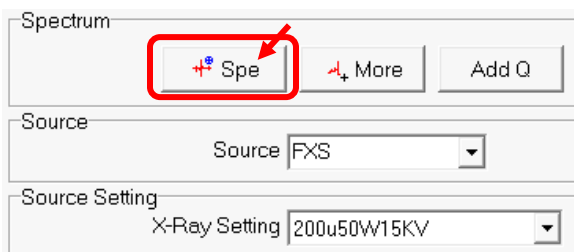
3) Click on  button on the window above to open the **Periodic Table** below and click on the table to add intended elements into **XPS Regions** window/list.

Peak ID		Acquisition Setup										Peak ID Filter					
Acquisition Setup																	
H	Su	2	3	4	5	6	7	8				He					
Li	Be							B	C	N	O	F	Ne				
Na	Mg							Al	Si	P	S	Cl	Ar				
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac															

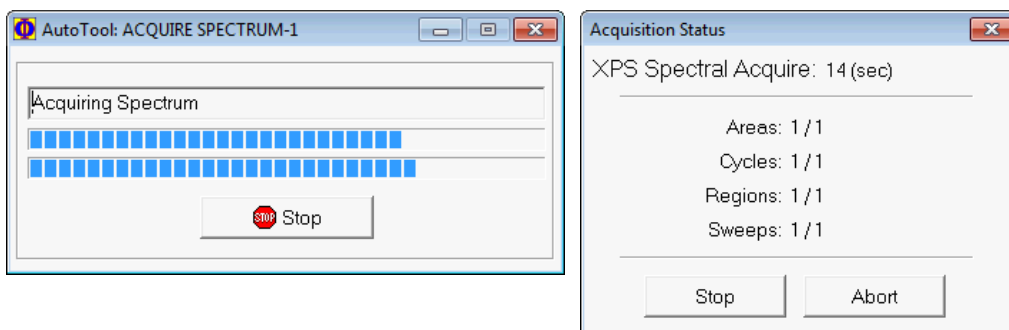
- 4) Scan parameter setup in the popup **XPS Regions** window below:
- Input the number sweeps/scans in the **Sweep** column below, typically **5 ~ 20** times depending on peak intensity collected in survey scan. Consider more sweeps for weak element peaks.
  - Input pass energy value in the **Pass** column, typically **27.0 eV** for relative higher peaks, and **13.0 eV** to improve the spectral resolution. If the peaks are small, choose higher pass energy from **55 eV** to **112 eV** to increase peak intensity. **Note:** this will lower the spectral resolution to make the peaks wider. For valence band scan, **13.0 eV** and **6.5 eV** should be chosen but more sweeps are needed.
  - Input scan steps in the **eV Step**, typically **0.05** or **0.1000** eV. For valence band scan, **0.05eV Step** or **0.025 eV Step** is good but more sweeps are needed.
  - Close** the popup **XPS Regions (Spectrum)** window.

Active	Name	Sweep	Pass	Lower	Range	Upper	eV Step	Ratio	P/N	Analysis Lower	Analysis Upper
<input type="checkbox"/>	Su1s	1	224.000	0.0	1100.0	1100.0	0.8000	2	1	0.0	1100.0
<input checked="" type="checkbox"/>	Cr2p3	1	27.000	569.0	20.0	589.0	0.1000	6	1	570.0	588.0
<input checked="" type="checkbox"/>	C1s	1	13.000	278.0	20.0	298.0	0.1000	8	1	279.0	297.0
<input checked="" type="checkbox"/>	O1s	1	13.000	523.0	20.0	543.0	0.0500	8	1	524.0	542.0

- 5) Click on **XPS Setup...** to open **XPS Acquisition Setup** window as shown in **Step 9)** in **Section 8.3** and uncheck **Z-Align** box in Queue Options section.
- 6) Hit **+ Spe** in Spectrum window to start **Region** scan

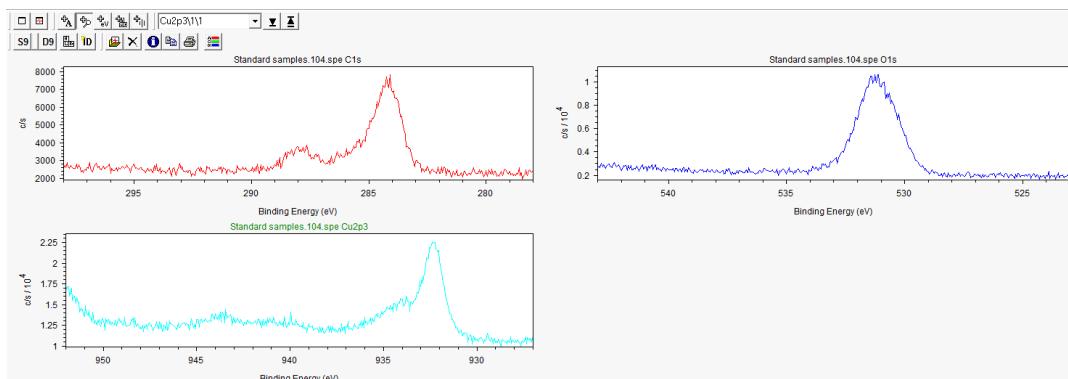


- 7) **Wait until** the following windows to appear



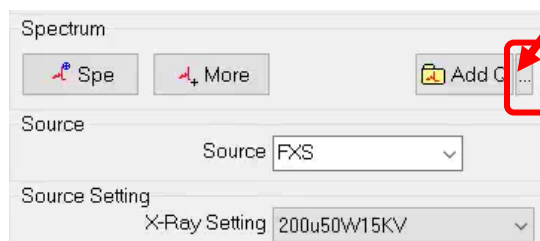


8) The **Region Scan** spectra will appear in the **Spectral Window** following the sequence in **Regions** table.

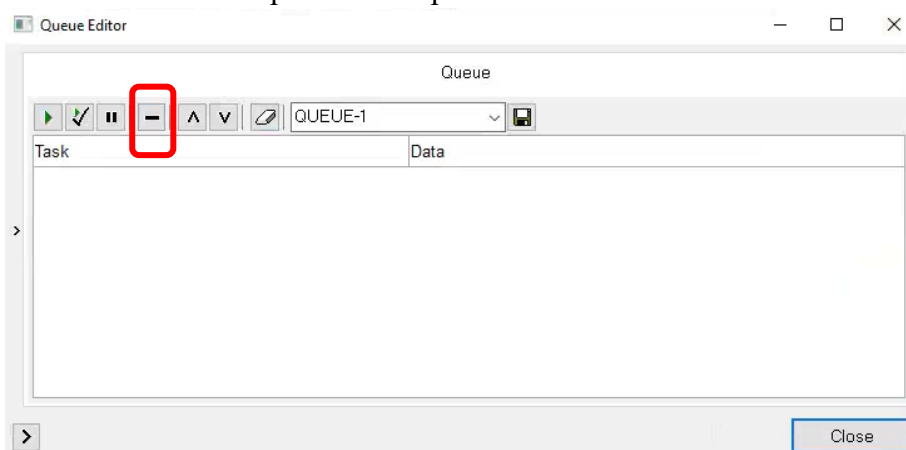


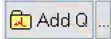

9) **Add Q** (Add to Queue mode) if interested elements are **different** on different samples:

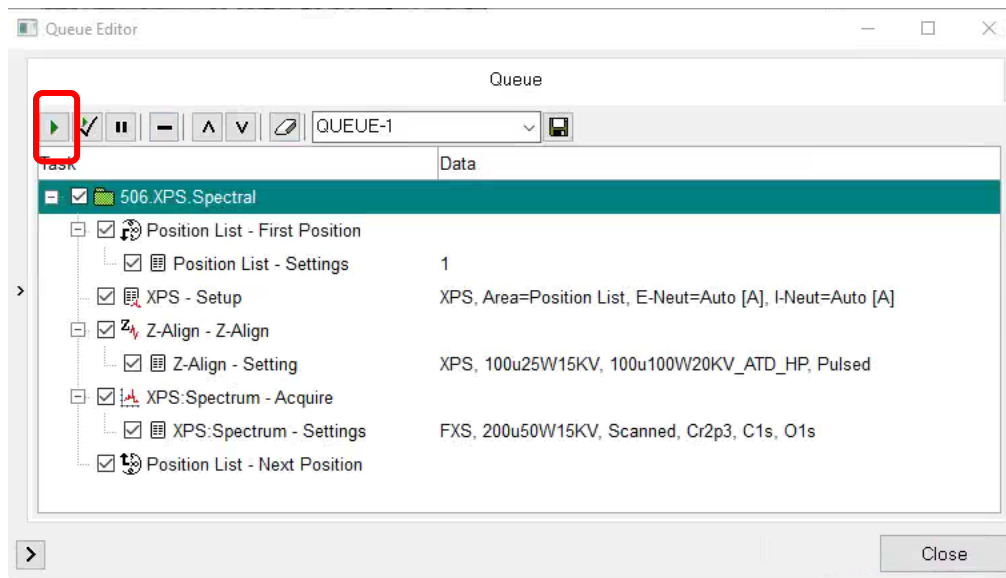
- Click on **XPS Setup...** to open XPS Acquisition Setup window as shown in **Step 9)** in **Section 8.3** and uncheck **Z-Align** box in Queue Options section.
- Set up a **complete element list** in the **Regions** window,
- Go back to **Sample** window, **check ONLY** intended sample positions on the list,
- Go to **XPS** window, **select ONLY** the group of elements for intended sample positions just chosen,
- Click the **right side** of the **Add Q** button in **Spectrum** window below:





f) The Queue Editor window with empty Task space will be opened as shown below. Click **[-]** on the top to remove previous tasks.

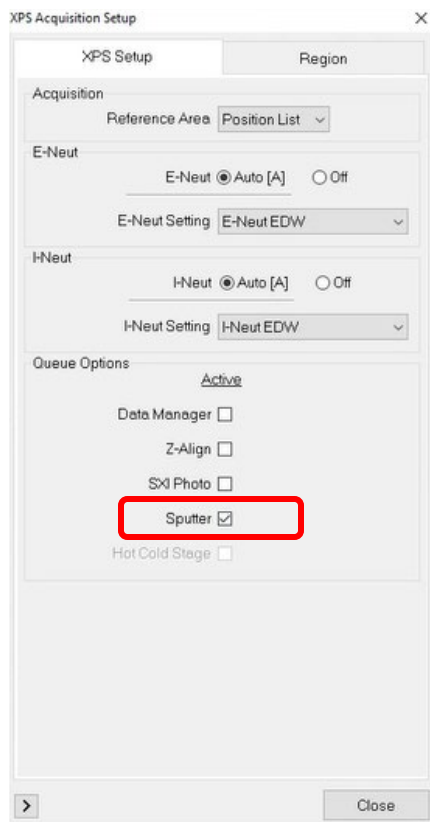



- g) Click the **left side** of the  button in **Spectrum** window to add tasks as shown below. Check to make sure all scan positions and parameters are correct and click  button on top to start scan.

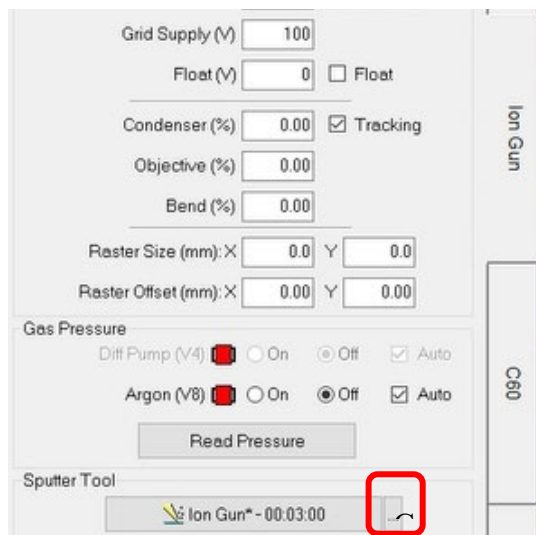


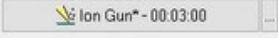
#### 10) Prespinner before scan:

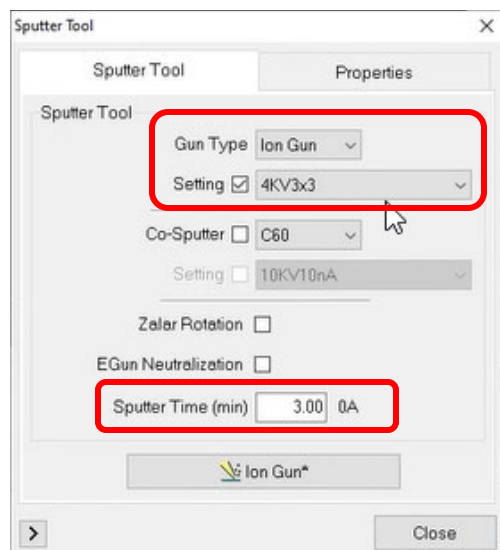
- a) Click  XPS tab on top tab menu to enter XPS analysis window and click **Spectrum** tab on the right side.
- b) Click on  button on the bottom of the **Spectrum** window to open **XPS Acquisition Setup** window and **choose highlighted options** in the window below:






- c) Click on  **Hardware** button top tab menu to enter **Hardware** window. Click on **Ion Gun** tab on the side menu to open the Ion Gun setting window below:

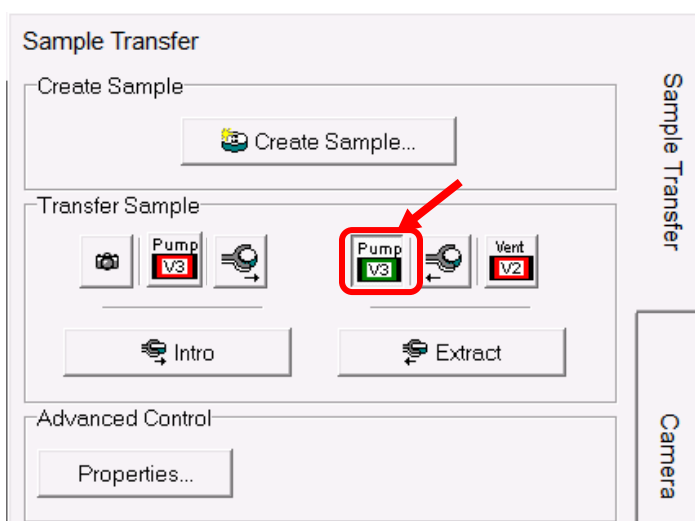



- d) Click on side of  highlighted above to open the **Sputter Tool** window below. Select the **Gun Type** as **Ion Gun** and specific **beam settings** and the **Sputter time (min)** in the highlighted regions. Close the window after finish.

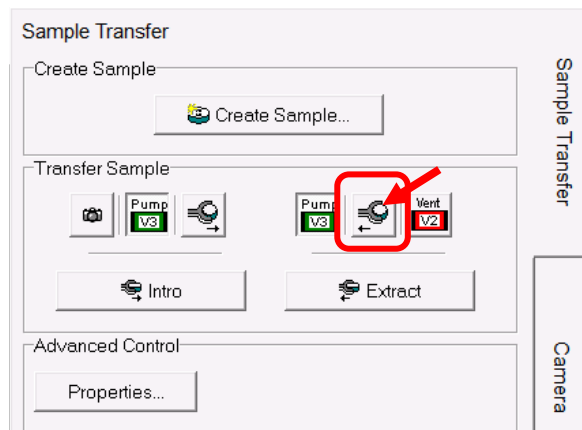


## 9 Closing XPS Measurement

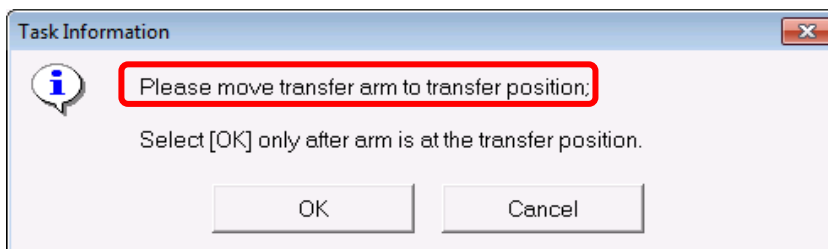
- 1) **Extract** samples from Main Chamber:
  - a) Check Intro Chamber status before sample extraction:
    - > Click **System** tab to enter the **Transfer Sample** window on the right side of the window, if the **V3** valve is in green , indicating the Intro Chamber is in **good** vacuum, then skip **Step b)** below.
    - > Else If **V3** valve is in red , continue **Step b)** below.
  - b) Click  button on the **Sample Transfer** window, watch the **Intro** pressure reaches below **5.0E-004 Pa** on Cold Cathode gauge.



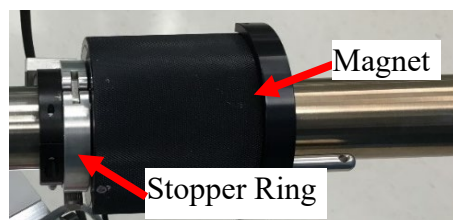
- 2) Turn **ON** the Main Chamber light.
- 3) Put gloves on right hand and get ready for sample extraction.
- 4) Click  below to start Sample Extraction



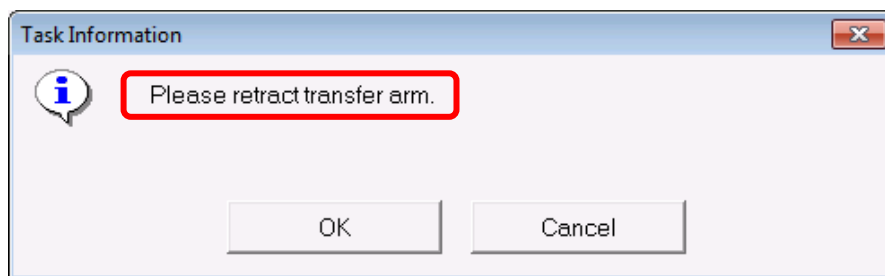
- a) Wait until the following window appears. **DO NOT** click OK button.



- b) Grab the magnet with the right-hand thumb placed on the left side of magnet and **slowly** move toward main chamber until finger touches the stop ring as shown below:

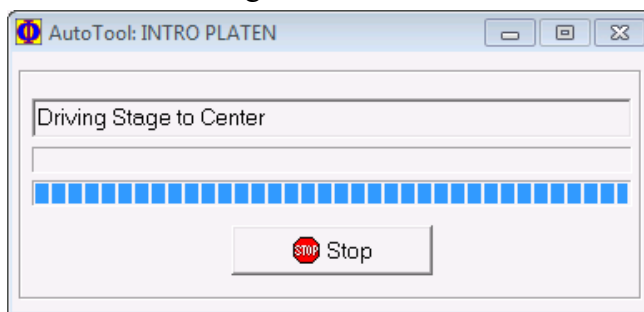


- c) There is no popping sound during extraction. If the gap between the magnet and the front stop ring is **> 1mm**:
- > **DO NOT force** the transfer rod into the main chamber stage to **DAMAGE** parts.
  - > **Retract** the rod slowly **without sample holder** back into Intro chamber and click OK on popup windows to close the V1 valve.
  - > **Inform** manager immediately for help
- d) **Keep** the transfer arm at the transfer position with magnet touching stopper ring, **go back** to the computer and click **OK** button on the popup window in **Step d)** above.
- e) Wait the following **window** to appear. **DO NOT** click **OK** button below:



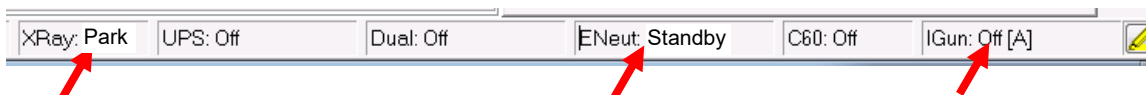
- f) **Go back** to the chamber, watch the holder in main chamber. Rest right hand on the transfer arm and **very slowly** pull back the arm from the stage to **make sure** sample holder stays on the fork during transfer. Once the front fork leaves the stage move faster to fully retract the transfer arm.
- g) **Go back to the computer** and click **OK** on the popup window in **Step h)**.

h) **Wait until** the stage is driven to the center.



- 5) Check the bottom right status bar on the **SmartSoft** program window below and make sure:
- > X-ray source (**XR**ay) is at **Park**. Right click on **Park** and select **Beam State: OFF**.
  - > Electron Neutralizer (**EN**eut) is at **Standby**. Right click on **Standby** and select **ENeut State: OFF**.
  - > Ion gun (**IG**un) is at **Standby**. Right click on **Standby** and select **Ion Gun State: OFF**.


**Warning:** If **IGun** is left **ON**, or a **red border** appears around any of these areas, **inform** manager immediately before proceeding. You may restart the software if permitted by manager. Never use the Task Manager to terminate the software.

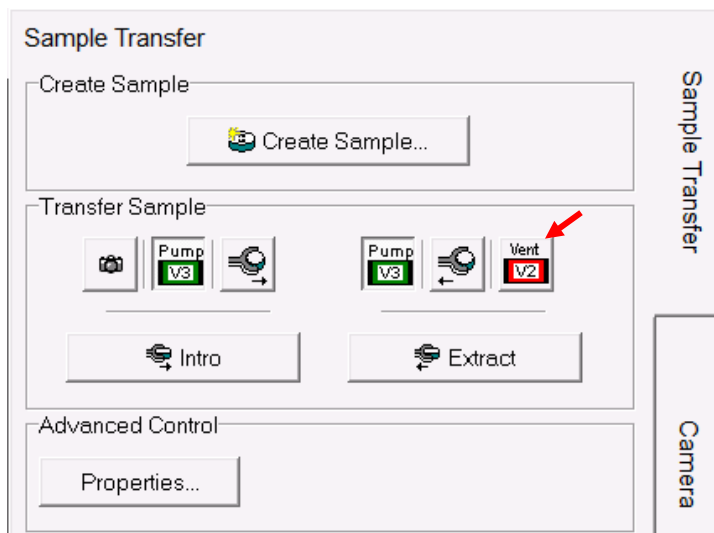



6) Turn **OFF** chamber light.

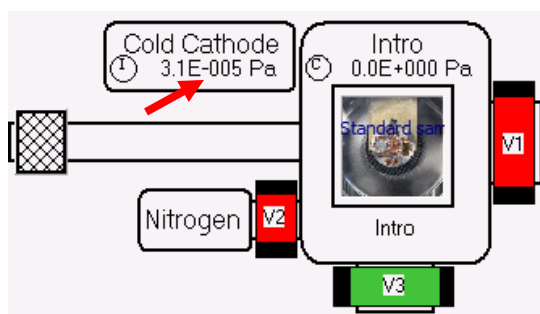



- 7) **Check** liquid nitrogen dewar if used and clean any water left around on the table.
- 8) If your samples can be discarded, then **skip Step 8) – 10)** below. The next user should clear your samples on the holder.
- 9) **Otherwise** continue to the steps below to retrieve your samples from Intro Chamber.

- 10) Click  on the **Sample Transfer** window below to vent the Intro Chamber



- 11) Right after venting started, put on gloves and hover on the glass cover. **Do not** touch cover. Slide the sample holder off from the transfer arm and put the cover back.
- 12) Click  button on the **Sample Transfer** window below and **quickly go back** to the loading port; **hold and press** the glass cover until pumping starts after sucking air sound.
- 13) Watch the Cold Cathode gauge reads **5.0E-004 Pa** before logoff FOM. It should just take ~ 5- 10 minutes.



- 14) **Resume the default settings in XPS Setup** window on Page 23, **Step 9)** in **Section 8.3**.
- 15) Keep **SmartSoft** programs maximized on the screen. **Never close the program**.
- 16) Back up your data either through internet (box.yale.edu) or using Core USB drive. **Do not** use personal USB drive.
- 17) Close **MultiPak** program, web browser and user folders.
- 18) Logoff **FOM** program: click the  icon on the taskbar below to activate the FOM program and click **Logoff** button in the FOM window. If any issues occurred during scan, check “**Something wrong**” and type message in the **Comments** space.





- 19) **Sign off** on the logbook.
- 20) **Remove** samples from the holder left by previously users and **clean** the surface with clean wipes and **IPA**.
- 21) **Store** the sample holder and other tools back into the tool box.

## 10 Some Analysis Concepts

- > High X-ray Power → High sensitivity but poor spatial resolution (beam size is bad)
- > Low X-ray Power → Low sensitivity but good spatial resolution (beam size is good)
- > High Pass energy → High sensitivity but poor energy resolution (peak is FAT)
- > Low Pass energy → Low sensitivity but good energy resolution (peak is THIN)
- > Big Step size → Acquire time is short but peak shape (resolution) is bad
- > Small Step size → Acquire time is long but peak shape (resolution) is good
- > High sensitivity → Signal-to-Noise is better so total acquire time can be shorter
- > Low sensitivity → Signal-to-Noise is worst so total acquire time needs to be longer
- > Sensitivity = Signal Intensity or counts

## 11 Chamber Leaking Emergency Operation Procedure

### 1) Intro Chamber leak:

- > Leak signs:
  - > Right after pumping, the intro chamber pressure stays ~ low **E+001 Pa** longer than ~ **15 minutes**, and turbo pump fails to start, or
  - > Turbo pump can be started and it takes more than **60 minutes** to for intro chamber to reach **4.0E-004 Pa**.
- > To retrieve good vacuum in Intro Chamber:
  - > **Vent** the intro chamber through **SmartSoft** program.
  - > Take the cover off and check the groove on the loading port. Clean the port with **Kimwipes** and **IPA**.
  - > Blow the inner side of glass cover with nitrogen gas.
  - > Put the cover back, **make sure** the cover is placed properly and start pumping.
  - > **Hold the cover firmly** into the grooves on the port during initial pumping. If the pressure is still not improved quickly, **contact Core manager immediately**.

### 2) Main Chamber leak:

- > Leak signs:
  - > **Minor leak**: the main chamber pressure stays at **high E-007** or **low E-006 Pa** range without samples inside.
  - > **Big leak**: the chamber pressure is above **high E-006 Pa**.
  - > **Sample transfer related leak**: the main chamber pressure starts to increase right after the intro chamber is vented. The V1 isolation valve might leak.

### 3) **Report the leak to Core manager immediately** to avoid further optics damage inside main chamber.