> 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 Box, 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://ywcmatsci.yale.edu/publications

> The core reserves the right to use the data for core promotion
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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
   - 810 West Campus Dr
   - West Haven, CT 06516

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. **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 \textbf{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. 

\textbf{Warning}: the \textbf{higher pressure} indicates either \textbf{stage outgassing} or \textbf{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 \textbf{11-425 E-BEAM SUPPLY} box as highlighted below and make sure is \textbf{ON in green}. If the LED light is off, stop sample preparation and report to manager immediately.

3) Check the building supply \textbf{N$_2$} and \textbf{Compressed Air (CA)} pressure gauges as labelled on the back wall and make sure the pressure reads \textbf{~100 psi} on both gauges. Report to manager immediately if any of those gauges reads \textbf{zero}.

\textbf{Warning}: \textbf{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 mm² 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

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 ºC – 600 ºC

3.3 Sample mounting

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

![Sample height diagram]

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:**
   a) Fix sample using removable double-sided Scotch tape provided in the lab. The sample cannot be heated in vacuum. The analyzer will be hit by higher samples if the active measurement is on lower samples.
   b) Fix sample with copper clips or metal cover on the sample holder. The sample can be heated in vacuum, or
   c) After sample mounting, shake and tilt sample holder to make sure samples are fixed on the holder.
   d) **Blow** the sample surface with dry N\textsubscript{2} inside fume hood

5) **For powder samples:**
   a) 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\textsubscript{2} inside fume hood
   b) 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 mm²)
> 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 N₂ inside fume hood

4 XPS Computer Login

1) Login FOM Program: click on Click here to login with NetID to unlock the 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.
      > Electron Neutralizer (ENeut) is at Off or Standby.
      > Ion gun (IGun) is at Off or Standby.
      
      If a red border appears around any of these areas, contact Core manager immediately
      before proceeding.

      | XRay Off | UPS Off | Dual Off | ENeut Off [A] | C60 Off | IGun Off [A] |
      |----------|--------|----------|---------------|---------|--------------|

   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.
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.

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 **Create Sample...** 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.
   a) Select 25 mm for regular 25 mm sample holder.
   b) Select 60 mm for regular 60 mm sample holder.
   c) 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

1) 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 in the Main Chamber, and then turn off Main Chamber light
   b) Check the **system diagram** below and make sure that the **Stage is empty** as highlighted. Report to lab manager immediately if not.

   **Warning:** Never leave samples in Main Chamber. The sample can be left inside the intro chamber after finish, which will be discarded by next user.

4) 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:
5) **Right after** venting started, put on gloves and hover on the glass cover. **Do not** touch cover.

6) Remove the glass cover and **slide** the sample holder along the bottom groove onto the transfer arm fork.

**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.

7) Check the pictures below. With the sample on the fork, make sure the **dent** on sample holder **touches** the **screw** on the fork.

8) Check from side window flange if the holder stays **flat** on the fork as shown below:

9) From the **top view** below, check if the sample holder is **symmetrically placed** on the fork and the **small circular part** (see in dashed box below) of the holder is facing the Main Chamber.
10) **Fully** retract the transfer arm so that the bullet on magnet **touches** the stopper ring on the transfer arm:

11) Check to make sure no particles/fibers inside the groove of the loading port. If found, clean with Kimwipes and IPA.

12) 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.

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

14) Pay attention to the camera shutter sound **Wait for several seconds** for picture loading into the program. Be patient, **NEVER** double click camera button.

15) Click the **Sample** tab next to **System** 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.

16) 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. **Warning:** The Intro Chamber might be leaking if the cover is not pressed during pumping.

17) Click and monitor the Intro Chamber pressure change on the system diagram:
   a) The Intro pressure gauge reading as highlighted below should drop from atmosphere pressure to ~3.0E+001 Pa in ~5 minutes;
   b) The Cold Cathode gauge should start reading from ~1E-002 Pa:
   c) Wait until the pressure drops to 4.0E-004 Pa before sample transfer can be started. Typically, it takes ~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

1) Check and make sure the Cold Cathode gauge pressure is **below 4.0E-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**.
7) 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.

8) Watch the sample holder in main chamber, and retract thumb and **move very slowly** until the magnet touches the **stopper ring** (see picture below). If feels resistance before touching the stopper ring, contact Core manager immediately. **DO NOT** force sample holder into stage to damage parts on the stage.

9) **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.

10) Wait the following **window** to appear. **DO NOT click OK** to damage parts on the stage.

11) **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.
12) **Go back to the computer** and click **OK** on the popup window in **Step 9**.

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

14) Turn off Main Chamber light.

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

16) Consider **liquid nitrogen** cooling on the stage with under following circumstances:
   a) **If the pressure stays in the 10E-6 Pa range for more than 30 mins,**
   b) **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:
   > 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) Click button as highlighted on top of SXI image window below to start Z-Align

9) 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.

10) Wait for the following Z Height window to appear and watch the Z height change in the space next to button. The typical height after alignment should be ~ 16-17 mm. The Z Align will fail if the number reaches above 18 mm. Report to Core Manager immediately if it happens.
8.2 Create multiple sample positions

1) After Z-Align, right click on all interested areas/samples on the Sample Photo and select Create Point on the dropdown list. An active ID will be assigned to each position as shown in the sample position list below:

2) Put sample/position names in the Name column and choose Point in the Type column as highlighted below:

8.3 XPS Survey (Full Range) Scan

1) Click $\text{XPS}$ 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 µm, 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 Su 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:
   a) 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.
   b) Input pass energy value in the Pass column, typically 187.850 eV; 117.4 eV can also be used to improve spectral resolution but more sweeps is needed for smooth peaks.
   c) Input scan steps in the eV Step, typically 0.8000 eV; 0.4 eV can also be used for higher resolution.
   d) Close the popup XPS Regions window.

9) Click on button on the bottom of the Spectrum window to open XPS Acquisition Setup window and follow the instructions highlighted on the window:
Select Auto

- XPS: E-Neut Neutralization: Auto [A] or Off
- XPS: Ion Gun Neut: Auto [A] or Off
- XPS: Z-Align: Enabled or Disabled

Select Disabled

- SXI Photo: Enabled or Disabled
- XPS: Presputter: Enabled or Disabled

Select Enabled

- X-Ray Setting: 9ul 0.010kV
- Auto Shutdown: Enabled or Disabled
- Auto Shutdown Delay (hrs): 1.0

Select Standard

- Acceptance Angle: Standard or Narrow
10) Hit **Spe** in Spectrum window below to start Survey scan

![Spectrum window](image)

11) **Wait** the following windows to appear:

![Acquisition Status](image)

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.

![Survey spectrum](image)

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:

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.

4) Scan parameter setup in the popup XPS Regions window below:
a) 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.

b) Input pass energy value in the **Pass** column, typically 23.500 eV. For low intensity valence band scan, 11.75 eV can be chosen but more sweeps are needed.

c) Input scan steps in the **eV Step**, typically 0.1000 eV. Smaller step of 0.05 eV is good for higher resolution valence band scan but more sweeps needed.

d) Close the popup XPS Regions (Spectrum) window.

5) Click on **XPS Setup** to open XPS Acquisition Setup window as shown in Step 9) in Section 8.3 and select **OFF** on XPS: Z-Align in Z-Align 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:

   a) Click on **XPS Setup** to open XPS Acquisition Setup window as shown in **Step 9)** in **Section 8.3** and check **OFF** on **XPS: Z-Align** in Z-Align section.
   
   b) Set up a complete element list in the **Regions** window,

   c) Go back to **Sample** window, check **ONLY** intended sample positions on the list,

   d) Go to **XPS** window, select **ONLY** the group of elements for intended sample positions just chosen,

   e) Hit **Add Q** button in **Spectrum** window to see added selected queue to the **Add Q** window below. **Make sure clear queues added by previous users.**

   f) **Minimize** above window, go back to **Sample** window and repeat **Step b) – e)** until all sample positions are selected;

   g) Maximize the **Add Q** window and hit **Start** to start spectral scan.
9 Closing XPS Measurement

1) Put gloves on right hand and get ready for sample extraction.

2) **Extract** samples from Main Chamber: *(Never leave samples inside 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 2) below
   b) Click button on the **Transfer Sample** window, watch the Intro pressure reaches below **4.0E-004 Pa** on Cold Cathode gauge.
   c) Click below to start Sample Extraction
d) **Wait until** the following *window* appears. **DO NOT** click **OK** button.

![Task Information]

Select [OK] only after arm is at the transfer position.

e) 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.

f) Watch the sample holder in main chamber, retract thumb and **move very slowly** until the magnet touches the *stopper ring* (see picture below). If feels resistance before touching, contact Core manager immediately. **DO NOT** force sample holder into stage to damage parts on the stage.

![Stopper Ring Magnet](image)

i) **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.

j) **Go back to the computer** and click **OK** on the popup window in **Step h**.)
k) **Wait until** the stage is driven to the center.

3) Turn **OFF** chamber light.

4) **Check** liquid nitrogen dewar if used and clean any water left around on the table.

5) If your samples can be discarded, then **skip Step 6) – 8)** below. The next user should clear your samples on the holder.

6) **Otherwise** continue to the steps below to retrieve your samples from Intro Chamber.

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

8) 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.
9) Click the 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.

10) Watch the cold cathode gauge reads **4.0E-004 Pa** before logoff FOM. It should just take ~5-10 minutes.

11) Keep SmartSoft programs maximized on the screen. **Never close the program.**

12) Back up your data either through internet (box.yale.edu) or using Core USB drive. **Do not** use personal USB drive.

13) 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.

14) **Sign off** on the logbook.

15) **Remove** samples from the holder left by previously users and **clean** the surface with clean wipes and IPA.

16) **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 \( \sim \text{low E+001 Pa} \) longer than \( \sim 15 \text{ minutes} \), and turbo pump fails to start, or
     - Turbo pump can be started and it takes more than \( 60 \text{ minutes} \) to for intro chamber to reach \( 4.0E-004 \text{ Pa} \).
   - To retrieve good vacuum in Intro Chamber:
     - Vent the intro chamber through \textbf{SmartSoft} program.
     - Take the cover off and check the groove on the loading port. Clean the port with \textbf{Kimwipes} and \textbf{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 groves on the port during initial pumping. If the pressure is still not improved quickly, contact Core manager immediately.

2) Main Chamber leak:
   - Leak signs:
     - \textbf{Minor leak}: the main chamber pressure stays at \textbf{high E-007} or \textbf{low E-006 Pa} range without samples inside.
     - \textbf{Big leak}: the chamber pressure is above \textbf{high E-006 Pa}.
     - \textbf{Sample transfer related leak}: the main chamber pressure starts to increase right after the intro chamber is vented. The V1 isolation valve might leak.
   - Report the leak to Core manager immediately to avoid further optics damage inside main chamber.