Operation Procedure for the DLD Axis Ultra XPS

Version 2.0*

Note

- This user handbook was a modification of the Kratos technician offered version
- Users are suggested to mount samples that can be analyzed in one session. Please bring your samples to EMAL at least one day before your session.
- Please have a copy of your collected data. EMAL will not take responsible of any data loss.
- Any problems please write in the logbook and report to EMAL staffs instantly.
Typical Experiment Sequence

1. Load sample(s) one day before your session.
2. Check conditions (Start up system and verify vacuum parameters) and sign-in in the logbook.
3. Transfer samples from STC (Sample Transfer Chamber) to SAC (Sample Analysis Chamber) if not performed.
4. Perform a survey scan and identify peaks of interest.
5. Sputter clean sample surface (if desired).
6. Perform narrow scan around peak(s) of interest to get additional chemical information (if desired).
7. Perform imaging to identify areas on sample of particular elemental or chemical states (if desired).
8. Perform small area or multipoint spectroscopy in areas of interest on sample (if desired).
10. Unload sample.
11. Return system to proper state for next user.
12. Sign-out in the logbook.

Each of these procedures is explained in more detail on the following pages.
**Startup Procedure (Typical)**

1. Check if the instrument is available. If OK, Sign in on the logbook.

2. Turn on the computer display screen if it is not.

3. Verify that the XPS software (Instrument Vision Manager) is open: Four Zones are all open: *Manager, Manual, Processing* and *Sys Administrator*; The “Controller Processor Unit” in the instrument displays “d3”.

4. The “Instrument Manual Control” window should be displayed on the screen. If it is not, click on the “Manual” zone in the top task bar.

   *Never close the “Instrument Vision Manager” window, as this will shut down the program and require you to re-start the software and re-calibrate the stage. If the software is closed by accident, please follow the procedure in the section “Start up procedure for when computer or software has been shut down” in this manual to re-start the software.*

5. Verify that the SAC pressure is $< 1 \times 10^{-8}$ and STC (or SEC) pressure is $< 5 \times 10^{-7}$ on the “Vacuum Control Unit” display (Figure 1A in standby condition) or in the Vacuum Control section in the “Instrument Manual Control” window (Figure 1B). *If vacuum levels are higher than these values, notify instrument staff and do not proceed unless a sample exchange or transfer has just been performed.*

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*Figure 1. (A upper and B lower) Vacuum meters for both STC and SAC chambers.*
Load/Unload Sample to/from the STC (Better for EMAL staff to do it for you)

Always wear gloves when handling samples and sample holders. Failure to do so will compromise the chamber vacuum and produce bad measurement results.

1. Click the “Manual” bar to let the “Instrument Manual Control” window show up if not.
2. Scroll down to the “Vacuum Control” section in the “Instrument Manual Control” window.
3. Click “Automatic Sequence” (Figure 2). Make sure the Green light at the end of the transfer arm barrel is illuminated not the Red light.

![Figure 2. Chamber vent and pump control unit.](image)

4. Click on “Vent STC”. This will vent the STC to allow sample loading.
5. Partially loosen the three brass screws on the end of the STC chamber and wait for chamber to completely vent (about 2 minutes).
6. Press the “Advance” button on the “Vacuum Control Unit” to stop the N₂ purge.
7. Completely loosen the three brass screws on the end of the STC chamber and rotate the chamber door open.
8. Rotate the translation knob to move the transfer arm forward for sample loaded on a Stub loading (When a Bar used, just load it by hand to a bar position in the STC).
9. Attach the sample Stub to the end of the transfer arm.
10. Rotate the translation knob to move the transfer arm backward so that the sample stub does not collide with the chamber opening when closing the chamber door.
11. Close the chamber door and partially tighten the three brass screws.
12. Rotate the translation knob to move the transfer arm forward and rotate the wheel underneath the STC to switch a Stub position on the way of the transfer arm. Once the Stub has reached the end of the Stub position, rotate the wheel and make the STC stage move toward you and release the Stub from the transfer arm. Rotate the transfer arm back to the end until the Green light on (view through the window when doing so).
13. Click on “Pump” STC and hold STC chamber door closed while pump down initiates.
14. After pump-down is initiated, tighten the three brass screws on the STC chamber door and wait for pump down to complete (typically about 15 minutes).
15. When completed, “Automatic Sequence Successfully Completed” will be displayed in the Vacuum Control Section.
16. Verify that the STC pressure is getting better before leaving.
Load Sample to the SAC (Only EMAL staffs are allowed to do)

DO NOT open the STC-SAC valve and transfer the sample if the STC pressure is $> 5 \times 10^{-7}$!!

1. Check if there is any Bar or Stub loaded in the SAC. If there is one, unload the Bar or Stub first see “Unload sample from the SAC session”.
2. Click the “Manual” bar to let the “Instrument Manual Control” window show up if not.
3. Scroll down to the “Vacuum Control” section in the window.
4. Click “Automatic Sequence” (Figure 2). Make sure the Green light at the end of the transfer arm barrel (Figure 3) is illuminated not the Red light.
5. Click on “Open STC-SAC Valve” and wait to see valve open in the vacuum diagram.
6. Verify that the Dual Anode X-ray source is backed out from the sample area.
7. Scroll to Manipulator section in the “Instrument Manual Control” window and click on “Position” box if the position table rows are not displayed (Figure 3).

Figure 3. Manipulator sessions for stage control.
8. If “loadbar” or “load_stub” positions are not displayed in the position table rows, then perform the following:
   a. Click on “Read Positions”, a window will appear to ask to make a choice.
   b. Choose “loadbar.dset” or “load_stub.dset” depending on which one you want to load.
5. Click on “Go to Position”. This will move the stage to the proper position for transferring the sample holder.
6. Click on the “Manual” icon in the Manipulator section (Fig. 3).
7. While looking through the viewing window, rotate the translation knob to move the sample holder into the SAC chamber until it interlinks with the stage and stops.
8. Use the Autostage manual controller (Fig. 4) to move the stage away from the transfer arm (until stop), releasing the sample holder from the transfer arm:
   a) While jiggling the translator knob, press the left button on the Autostage manual controller to move the stage away from the transfer arm.
   b) Holding down the right button after pressing and holding the left button will speed up the stage movement. This works same to other direction movements.

![Figure 4. Autostage manual controller.](image)

9. When the stage and sample holder have cleared the transfer arm, rotate the translation knob to move the transfer arm completely out of the SAC chamber – until the green light at the end of the transfer arm barrel is illuminated.
10. Scroll to the Vacuum Control section in the “Instrument Manual Control” window and click on “Close STC-SAC Valve”.
11. Position sample using the “Autostage Manual Controller” while watching Video Monitor (Note: Magnification on monitor can be varied by adjusting zoom control on video camera).
    a) Use **Left, Right, In, Out** buttons to position sample area of interest in center area of video monitor.
    b) Use **Up, Down** buttons to adjust height of sample so that center region of sample is in focus on the monitor. The top and bottom regions on the monitor should be slightly out of focus.

*Note: For the majority of spectroscopy acquisitions, this accuracy of setting the sample height will be sufficient to achieve an acceptable number of counts. If further Z height optimization is needed, follow the procedure found in the section “Optimizing Z for Spectroscopy” and Optimizing Z for imaging” is explained in the following procedure section.*
Unload Sample From the SAC (only email staffs are allowed to do)

**DO NOT open the STC-SAC valve and transfer the sample if the STC pressure is > 5 x 10⁻⁷!!**

1. Make sure the “Instrument Manual Control” window is open.
2. Scroll to Manipulator section (Figure 3) in the Window and click on “Position” box if the position table rows are not displayed.
3. Click on the “Read positions” icon and choose the “unloadbar or unloadstub”.
4. Click on “Go to Position”. This will move the stage to the proper position for transferring the sample holder.
5. Click on “Manual” in the Manipulator section.
7. Choose *Automatic Sequence* box.
8. Click on “Open STC-SAC Valve” and wait to see valve open in the vacuum diagram.
9. While looking through the viewing window, rotate the translation knob to move the sample holder into the SAC chamber until it interlinks with the stage and stops.
10. Use the “*Autostage manual controller (Fig. 4)*” to move the stage toward the transfer arm, releasing the sample holder from the stage:
   a. Press the right button on the autostage manual controller to move the stage toward the transfer arm.
   b. Holding down the left button after pressing and holding the right button will speed up the stage movement.
   c. Drive stage in right direction while jiggling the translation knob until sample holder hooks onto the transfer arm, and then drive back to the left to center the manipulator and relieve tension between the sample and fork. When completely transferred, the movement of the translation knob should loosen up considerably.
11. When the sample holder has transferred, rotate the translation knob to move the transfer arm completely out of the SAC chamber – until the green light at the end of the transfer arm barrel is illuminated.
12. Scroll to the Vacuum Control Section, and click on “Close STC-SAC Valve” and watch display for valve to close.
**Conditioning the X-ray Source**

1. Make sure the “Instrument Manual Control” window is open. Scroll to X-Ray PSU (Power Supply Unit) session.
2. Choose a filament (Mono Al or Dual source (Al and Mg)*). Mono Al is good for spectrum while dual sources are good for imaging.
3. Make sure the anode HT is at 6 kV and emission is 1 mA.
4. Click “Standby” until the Green light is on then click “On” till the green light is on. Make sure the monitor light On and the two lights indicating the condition of chiller and pressure are On.
5. Watch up the vacuum of the SAC. Normally it will drop a little bit. Continue when the vacuum has been recovered.
6. Bring up anode HT (from 6 – 15 kV) at a 1 kV step and emission (from 1 - 10 mA) at a 1 mA step by inputting numbers and press the Enter button. Never go above 20 mA, as it will degrade source life.
7. Leave at “On” condition.

* Dual source:

- If the dual sources used they needs to be rotated in to approach the sample. The current tilt position was set for the Mg source only (if the Al source to be used please check the settings with the EMAL staffs).
- Rotate in the Dual source from the back of the instrument until the marked position has been reached. Watch it through the SAC chamber window (Never go further otherwise you will damage the dual source!).
- When the dual source has been inserted, special care should be taken when move the stage either manually or auto.
- Once finished using the dual source, retract it instantly completely. Otherwise it may be damaged.

**Charge Neutralizer (Spectra From Insulator)**

- When collect XPS spectra from insulator (non-conductive) materials, the Charger neutralizer is needed to be On. Otherwise ghost peaks may show up in the spectra.
- Click on the parameter button in the Charger Neutralizer session in the “Instrument Manual Control” window, three parameters can be found: Filament current (A) (1.8 ~ 2.1 A), Charge balance (v) (2.5 ~ 3.6 v) and the Filament bias (v) (1~ 1.6 v).
- Aim of the charge neutralizer is to maximize the peak intensity while minimize its width.
- The three parameters are strongly interdependent and changing one value might require the optimization of the others. The general rule is that the “Charge balance” is the most critical one in the determining the efficiency of the neutralizer process.
- The optimum charge neutralizer conditions (e.g., the default setting) tend to a lower BE than would be obtained if the sample were an conductor e.g. C 1s peak (carbon on metal) approximately 284.8 eV, C 1s peak hydrocarbon polymer) approximately 282.5 eV.
Optimizing Z for Spectroscopy

* The aim of “optimizing Z” is to adjust the Z height of the sample to get maximized counts for a spectrum. For spectroscopy, manually optimizing Z should be enough that is described here even

1. Turn on the TV monitor if not on. Change the camera Zoom and find an interest area of the sample and move it to the marked point in the TV screen.
2. Focus the image of the sample by adjusting the height of the stage until a clear image has been seen in the screen.
4. In the Analyzer section, enter the following parameters:
   b. Lens Mode: Hybrid.
   d. Aperture: Slot.
5. In the acquisition section, choose the type “Snapshot”.
6. Choose an energy peak for example C 1s and use it for the sample height optimization. Make sure this scan is pasted into the “Real-time window”.

Enter the region settings as follows:
   a. Click the “energy region”.
   b. Input the peak for the selected element and the scan settings for that element will be automatically loaded in the acquisition region settings.
   c. Modify the region settings if needed (e.g., Peak value, width eV, dwell, and sweeps etc.)
7. In the “acquisition” control section, click on the “On” button. If it can not be turned on, switch to the “Vision Instrument Manager” window and click the “Manual now” icon.
8. In the “Manipulator” section, click the Manual icon and adjust Z by click the Z increasing or decreasing icon in the “manipulator” session until a maximum CPS has been obtained of the interested peak.
Performing a Survey Scan

1. Click on the “Manager” zone to open the “Vision Instrument Manager” window.
2. Click “Resume” (a in Figure 5) button to set to Automatic mode.
3. Choose the “Dataset” button, Enter a filename for storing your data in the Name field
4. Click the Browse and open your own folder and enter a name in the filename field.
5. Click on the middle mouse button to paste the “Filename” sequence in the flow chart section.
6. Choose the “Acquisition” button and set up the parameters for the survey scan. The following are typical values (See also Acquisition Conditions Reference Sheet):
   - Name: Survey
   - Standby Control: Leave On (leaves X-rays on after scan completed)
   - Analyzer:
     - Mode: Spectrum.
     - Lens Mode: Hybrid (only works together with the Slot).
     - Resolution: Pass Energy 160.
     - Aperture: Slot (700 × 300 um).
   - Number of Detectors: 115.
   - Check the X-ray PSU parameters. They should be same to those in the Manual window.
   - Scan Control:
     - Region Name: Type in “Survey”.
     - Start eV: 1200 (right click on header to change from Centre eV to Start eV).
     - End eV: -5.
     - Dwell ms: 200.
     - # Sweeps: 1.
     - Click on “Active” box.
7. Click on the middle mouse button to paste the “Survey” sequence in the flow chart.
8. Click on “Submit” to start the flow chart job (Survey scan).
9. Choose “Acquiring” in the view window to see the scan data being collected in the “Real-Time Display” window. The icon in the Viewing Acquiring Session shows pink color when the acquiring is performing and turns to be grey color when finished.
10. When completed, the scan may be viewed in the “Real-Time Display” by highlighting the item in the acquiring window and middle mouse button “pasting” into the right-hand section of the real-time display window.
11. Using mouse control menus you may zoom in on regions of the scan, change the scan label, etc.
12. Choose element list from Windows pull down menu to open up an element list for identifying peaks.
13. Clicking on peaks of interest in the Real-Time Window will cause associated element to display in the element list window.
14. Save the collected data one by one or in a dataset by group them together.
Performing Region Scan Around Peak(s) of Interest

This procedure assumes that a survey scan has been performed as described previously.

1. Click on the “Manager” zone to open the “Vision Instrument Manager” window.
2. As was done to create a survey scan, create an acquisition flow chart with a descriptive name such as “Core Scan”.
3. Choose the “Acquisition” button and set up the parameters for the scan (Refer the Acquisition Conditions Reference Sheet).
4. Scroll to Scan Control section and enter in scan settings using one of the following methods:
   a. Use Element List
      i. Choose “Element list” from the pull down Windows menu in the Manager window.
      ii. Click on a peak of interest in the Real-Time Display window (paste it there).
      iii. Click on the associate element in the element list, and the scan settings for that element will be automatically loaded in the scan control settings.
   b. Enter region name manually (e.g., O 1s) and settings will automatically load. (Note: region name must match exact spelling and case of entry contained in database).
5. Click on “Active” box beside each row in the Scan Control section for which you want a scan to be made.
6. Click on the middle mouse button to paste the “Core Scan” sequence in the flow chart.
7. Click on “Submit” to start the flowchart job (Core Scan) if still in the auto mode.
8. Choose “Acquiring” in the view window to see the scan data being collected in the Real-Time Display window.
9. When scans are completed, highlight them in the viewing window and paste into the Real-Time Display window to view all scans.
10. Save the collected data one by one or in a dataset.
**Imaging: Elemental/Chemical State Mapping**

The operation mode allows users to pick specific peak energies and make a spatial map (~ 4 micron spatial resolution) of element or chemical state of the sample (dual source better).

- **Pick one element or chemical state energy peak value of interest.**
- **Auto Z for imaging:** Manually optimizing Z is always a start point for auto Z (see page 9).

1. Adjust Z height of the sample manually until a clear image is obtained in the screen.
2. Open the “Instrument Manual Control” window.
3. In the acquisition section, choose the type “Snapshot”.
4. Choose an energy peak for example C 1s and use it for the sample height optimization.
5. Scroll to Manipulator section in the Window and click on “Position” box if the position table rows are not displayed.
6. Click the “Update position” icon. The current position will be inserted in the table.
7. Scroll to the Auto Z section (Z range: -4 ~ + 4 mm), set Number increments to an Odd number (for example 21), Increment size to 0.02~0.05mm; Ordinate choice to “area”; Acquisition setting to “Current Manual”. Click “Crab Scan” to load the current active snapshot setting into the Auto Z routine. A2 in the position table will show “req”. Then click “Optimize Position” icon.
8. Open the “Vision Instrument Manager” window.
9. Choose the “Dataset” button and enter a filename for storing your data in the name field.
10. Browse the folder where the data will be saved. Enter a name for this flow chart item in the name field. (e.g., Filename).
11. Click on the middle mouse button to paste the “Filename” sequence in the flow chart.
12. Choose the “State Change” button and enter a name for this flow chart item in the name field (e.g., Position).
13. Choose the Sample Position button and load the position table.
14. Click on the middle mouse button to paste the “Position” sequence in the flow chart.
15. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Map).
16. Set up the parameters for general alignment imaging (referring the **Acquisition Condition Reference Sheet**): (check also the pixel size to 256 × 256).
   a. Analyzer:
      i. **Analyzer mode:** Image.
      ii. **Lens Mode:** FOV1.
      iii. **Resolution:** 160.
      iv. **Aperture:** Low Res.
   b. Scan Control:
      i. **Choose Map.
      ii. **Enter region name or energy value based on element or chemical state energy peak value of interest.
      iii. **Dwell (time per sweep):** 20 (or longer if a low intensity peak).
      iv. **# Sweeps:** 1.
17. Click on the middle mouse button to paste the “Map” sequence in the flow chart.
18. Choose the “State Change” button and enter a name for the counting flow chart item in the name field (e.g., Count).
19. Choose the Counter button and enter the number of cycles. This should be the same as the number of Z increments that were entered in the position table.
20. Click on the middle mouse button and paste the “Count” sequence in the flow chart.
21. In the flowchart area, highlight the “Position”, “Map” and “Count” items. Then right click and choose “Loop Back”.
22. Click on “Submit” to start the flowchart job. An image map will be taken and a file created for each Z height setting that was entered.
23. If desired, choose “Acquiring” in the view window to see the image data being collected in the Real-time Display window.
24. When run is completed, highlight the files in the viewing window and paste into the Real Time Display window to view all maps. (You may also use the functions in the Process zone to view all maps simultaneously).
25. Choose the optimum image and note which Z position produced that image.
27. Scroll to Manipulator section in the Window and highlight the row in the position table which contains the optimum Z value.
28. Click on “Go to Position”. This will move the stage to the optimum Z height.

**Setting up an image mapping flowchart**

1. Open the “Vision Instrument Manager” window and clear all flowchart items from the Z optimization flowchart.
2. Choose the “Dataset” button.
3. Enter a filename for storing your data in the name field.
4. Browse the folder where the data will be saved. Enter a name for this flow chart item in the name field. (e.g., Filename).
5. Click on the middle mouse button to paste the “Filename” sequence in the flow chart.
6. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Map).
7. Set up the parameters for elemental or chemical state imaging (see *Acquisition Conditions Reference Sheet*):
   8. Analyzer
      i. Analyzer mode: Imaging.
      ii. Lens Mode: Typically FOV2
         
         *Note: FOV2 is the only field of view that is referenced to the various apertures to give the spot size shown in the Acquisition Conditions Reference Sheet.*
      iii. Resolution: 160 for elemental imaging, 40 or 80 for chemical state imaging
      iv. Aperture:
         • FOV2: Medium Res or High Res
         • FOV3: High Res
   9. Scan Control
      i. Choose Map
      ii. Enter region names or energy values based on element or chemical state energy peak values of interest.
      iii. Dwell: 20 (or even much longer for a low intensity signal or at lower pass energy).
      iv. # Sweeps: 1 (or more if desired).
   10. Click on the middle mouse button to paste the “Map” sequence in the flow chart.
11. Click on “Submit” to start the flowchart job. An image map will be taken and a file created for each energy value that was entered if still in auto mode otherwise click the “Resume” first.

12. If desired, choose “Acquiring” in the view window to see the image data being collected in the Real Time Window.

13. When run is completed, highlight the files in the viewing window and paste into the Real Time Display window to view all maps. (You may also use the functions in the Process zone to view all maps simultaneously or overlap maps, etc.).

**Multipoint Small Area Spectroscopy and Line Scan**

**Multipoint Small Area Scans**

1. Click on the “Manager” zone to open the Vision Instrument Manager window.
2. Choose the “Dataset” button.
   a. Enter a filename for storing your data in the name field.
   b. Enter a name for this flow chart item in the name field. (e.g., Filename).
3. Click on the middle mouse button to paste the “Filename” sequence in the flow chart.
4. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Small Spot).
5. Set up the parameters for a small area scan.
   a. See the Acquisition Conditions Reference Sheet to set values for desired spot size. *Note that FOV2 is the only field of view that is referenced to the various apertures to give the spot size shown in the Acquisition Conditions Reference Sheet.*
   b. Analyzer mode should be “Spectrum”.
   c. Enter Scan Control parameters for desired energy and spectra settings.
      
      **Typical parameters are:**
      
      - *Energy width of 15eV around the energy value of interest*
      - *Step size of 0.1*
      - *Dwell time of 200*
      - *# Sweeps = 1*
6. Bring up the image map and point the mouse to the area where you want the scan to be performed, and then left click on the mouse button in the Real-time Display window.
7. In the Analysis Position section of the Manager window, click on “Import Position”. The x,y position of the point identified in the image map will be input.
8. Click on the middle mouse button to paste the “Small Spot” sequence in the flow chart.
9. To perform small area spectroscopy on multiple points in an image map:
   a. Copy and paste the “Small Spot” sequence in the flow chart. Paste as many copies as desired points
   b. Go into each small spot sequence, choose the position on the image map and import the position (as in Steps 6 and 7).
10. Click on “Submit” to start the flowchart job
11. Choose “Acquiring” in the view window to see the scan data collection in the Real Time Display window.
12. When completed, the scan may be viewed in the Real Time Window (You may also use the functions in the Process zone to view all scans simultaneously, etc.).
**Line Scans** (*Line scan can also be performed by draw a line in a map*)

1. Set up a filename and flowchart in the Manager window (See steps 1-3 in the Multipoint Small Scans Procedure).
2. Choose the “Acquisition” button and enter a name for this flow chart item in the name field (e.g., Line Scan)
3. Set up the parameters for a small area scan.
   a. See the Acquisition Conditions Reference Sheet to set values for desired spot size.
   b. Analyzer mode should be “Spectrum”
   c. Enter “Line Scan” in the Scan Control Sections
   d. Enter other Scan Control parameters for desired energy and spectra settings. (as in Step 5 of Multipoint Small Scans Procedure)
4. Bring up the image map and click and drag the mouse to form a rectangle in the area where you want the scan to be performed. The line scan will proceed from the upper left corner of the rectangle to the lower right corner.
5. In the Analysis Position section of the Manager window, click on “Import Position”. The coordinates of the line identified in the image map will be input.
6. Click on the middle mouse button to paste the “Line Scan” sequence in the flow chart
7. Click on “Submit” to start the flowchart job
8. If desired, choose “Acquiring” in the view window to see the scan data being collected in the Real Time Window
9. When completed, the scan may be viewed in the Real Time Window (You may also use the functions in the Process zone to view all scans simultaneously, etc.)

**Other Spectrum Collection Modes:**

**Angle-resolved XPS:**

*The AXIS can be used for ARXPS using the standard strip or the “constant height bar” to rotate the sample about the horizontal X-axis. Users are encouraged to read published articles on ARXPS to gain further information on the determination of ARXPS concentration depth profiles.*

   - Use the “Constant height bar” and optimize Z for spectroscopy ($p9$).
   - Using the auto stage software control rotate the sample towards the Mono Al source by – 15 degree, optimize Y axis and then another 15 degree to -30 degree, then -45 degree, -60 degree and -75 degree. Insert all the position in the general position table. (Be care: make sure the stage will not collide with the top of the magnetic lens.

2. Define the ARXPS experiment in the “Instrument Manager Control” window.*

**Automated spectrum acquisition from several samples:**

**Acquiring spectra from similar samples:**

(* Refer the Instructions in the Lab.*)
**Ion Gun Sputtering and Depth Profiling**

*It is suggested that you acquire a spectra on peaks of interest before and after performing a sputter clean so that a comparison can be made and the effect of the clean determined. For example, you may want to look at the oxygen and carbon peaks before and after sputtering to verify that they have decreased sufficiently the sample peak of interest to determine that it has increased sufficiently.*

1. Click on the “Manual” zone in the top task bar to open the “**Instrument Manual Control**” window.
2. Standby the X-ray source.
3. Scroll to the **Vacuum Control** section and choose **Automatic Sequence**
4. Open the valve of the Ar gas cylinder (normally it is always open).
5. Open the green valve (on the back of the instrument) of Ar for 2 seconds and then close it.
6. Click “**Ion Gas On**” and look for the valves to open. This opens the valves to allow the Ar gas in for sputtering (Monitor the SAC vacuum pressure using the Magnifier in the desktop or in Start/Accessories/Accessibility. Turn the Ar gas control knob (next to the ion gun) counter anti-clockwise slowly for close to two turns (we have another mark). Please watch the black mark on the knob, at the same time watch the SAV pressure. The pressure will gradually increase to $5 \times 10^{-8}$ torr when it is almost two turns. Wait a few minutes to let the pressure stabilize at ~$5 \times 10^{-8}$ torr.
7. Scroll to the **Ion Gun** section and choose Table button. The table contains pre-set setting for various ion sputtering conditions.
8. From the Table, choose a desired kV (1-5 kV) (for depth profiling, choose the 4kV medium spot and a raster of 2 mm should normally suffice. Consider also the possible damage of your sample).
9. Click “**Restore Row**” to enter these settings. You may click on the “**States**” button to watch the sputtering parameter values (you should see numbers other than Zero).
10. Choose desired Raster Size ($1 \times 1$, $2 \times 2$, $3 \times 3$, $4 \times 4$, or $6 \times 6$ mm$^2$) in Operation Settings
11. Click on “Standby” to ramp up voltage and currents.
12. Set the ion pump voltage to 7 kV. The manual Leak Valve should be set so that the pressure in the SEC is $3-4x10^7$ torr, the SAC ~ $3.3 \times 10^8$ torr, and the Ion Pump reading ~$7x10^{-5}$ amps on the 7kV setting. This is all with the gun on. The regulator takes a little time to respond once the line is opened, and the pressure is still somewhat dynamic early on in the process when the valves have just been opened.
13. After ramp-up complete, click on “Start” to start the sputter process.
14. Allow to run for desired time (Typical time is 10 minutes for a sputter clean).
15. Click on “Standby” after desired time has elapsed.
16. Perform spectra around the peaks of interest and compare post-sputter spectra with pre-sputter spectra. If additional sputtering is desired, click on “Start” and sputter for additional desired time.
17. Standby the Ion Gun and Click on “**Off**” after acceptable sputter cleaning has been achieved
18. Close the Ar gas control by turning it clockwise two turns and align the black marks on the knob.
19. In Vacuum Control section, click on “**Ion Gun Gas Off**” and look for valves to close.
20. Wait for the SAV vacuum to recover back to ~ $8 \times 10^{-9}$ torr.
21. Turn on the X-ray source to continue the collection of spectrum.
Returning System to a Proper State for Next User

1. **DO NOT** close any windows or exit the software.

2. Turn off the X-ray source (first click “Standby” then “Off”) and set Emission to 1 and HT to 6 in the X-ray PSU. Turn off the **Charge Neutralizer** if used.

3. **In the Vision Manager zone window:**
   a. Clear all flowcharts from the flowchart section.
   b. In the acquisition section, right click on the mouse and choose the “Close Dataset” option.

4. **In the Process zone window:**
   a. From the “File” pull down menu, choose “Close all Datasets and Clear Scratch”

5. **Click on the “Manual” zone in the top task bar to open the “Manual Control Window”, and leave this window open.**

6. **Turn off the computer monitor**

7. **Complete entries in the written log book**

Data Processing*

The data processing software allows you to change the way in which data is viewed (including overlaying of multiple spectrum scans or images), and also allows you to quantify the results (e.g., weight or atomic per cent of elements)

1. Click on the “Process” zone in the top task bar to open the data processing work area
2. From the “File” pull down menu, choose “Open Dataset for Processing”
3. Click on the “Update” button to load recent datasets
4. Choose the dataset filename(s) for which you want to process the data.
5. See the on-line manual in the “PDF” desktop folder for instructions and explanations of the various features in the Processing software.

*Details please refer the Instruction book in the Lab.*

Convert the vision manager dset to *vms type for the Casa Control software:

- Open a dset using "Vision Processing" and highlight it.
- In the "Vision Processing" "option" window, open the "Browser Action" window.
- Click "Describe" in the top right corner of the window.
- In "Destination" select "Vamas File" and then click "Apply". A save window will appear and ask you to save to *.vms format.
Acquisition Conditions Reference Sheet

Note that these parameters should only be considered as a reasonable starting point. Acquisition parameters may be optimized dependent on the instrument or overall experimental goal.

I. Charge Neutralizer Conditions – exact conditions may vary slightly between instruments.
   - Charge balance (2.6 – 3.2 V)
   - Filament current (1.6 – 2.1 A)
   - Filament Bias (0.9 – 1.3 V)

II. Spectroscopy (Based on use of monochromatic x-ray source).

III. A. Large area (300um x 700um) – slot

<table>
<thead>
<tr>
<th>Energy Range</th>
<th>Magnification</th>
<th>Pass Energy eV</th>
<th>Step Size eV</th>
<th>Time (min's)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Survey</td>
<td>hybrid</td>
<td>160</td>
<td>1</td>
<td>1 - 3</td>
</tr>
<tr>
<td>Region</td>
<td>hybrid</td>
<td>10 or 20</td>
<td>0.1</td>
<td>1 - 10</td>
</tr>
<tr>
<td>Valence Band</td>
<td>hybrid</td>
<td>20 or 40</td>
<td>0.2</td>
<td>5 - 20</td>
</tr>
</tbody>
</table>

B. Small area – small area spectroscopy must be referenced from medium magnification images for following spot sizes

<table>
<thead>
<tr>
<th>** Spot size (Physical size and name in software)</th>
<th>Magnification</th>
<th>* Pass Energy</th>
<th>Step Size eV</th>
<th>Time (min's)</th>
</tr>
</thead>
<tbody>
<tr>
<td>110 um (2mm Physical)</td>
<td>FOV2</td>
<td>10 – 40</td>
<td>0.1</td>
<td>1 - 4</td>
</tr>
<tr>
<td>55 um (1mm Physical)</td>
<td>FOV2</td>
<td>10 – 40</td>
<td>0.1</td>
<td>1 - 10</td>
</tr>
<tr>
<td>27 um (0.4mm Physical)</td>
<td>FOV2</td>
<td>20 – 80</td>
<td>0.1</td>
<td>3 - 15</td>
</tr>
<tr>
<td>15 um (0.15mm Physical)</td>
<td>FOV2</td>
<td>20 – 80</td>
<td>0.1 – 0.2</td>
<td>5 – 30</td>
</tr>
</tbody>
</table>

* use 160 eV for surveys
** a predetermined iris setting must be used for each spot size

IV. Imaging (parallel)

A. Elemental

<table>
<thead>
<tr>
<th>Goal</th>
<th>Magnification</th>
<th>PE (eV)</th>
<th>Acq. Time (min's)</th>
</tr>
</thead>
<tbody>
<tr>
<td>General Alignment</td>
<td>FOV1</td>
<td>160</td>
<td>1</td>
</tr>
<tr>
<td>Small area spec. Position referencing</td>
<td>FOV2</td>
<td>160</td>
<td>1 – 5</td>
</tr>
<tr>
<td>Highest Lateral Res.</td>
<td>FOV3</td>
<td>160</td>
<td>2 – 8</td>
</tr>
</tbody>
</table>

B. Chemical State and generating spectra from images

<table>
<thead>
<tr>
<th>Goal</th>
<th>Magnification</th>
<th>PE (eV)</th>
<th>Acq. Time (min's)</th>
</tr>
</thead>
<tbody>
<tr>
<td>General Alignment</td>
<td>FOV1</td>
<td>40 or 80</td>
<td>1</td>
</tr>
<tr>
<td>Small area spectroscopy. Position referencing</td>
<td>FOV2</td>
<td>40 or 80</td>
<td>1 - 10</td>
</tr>
<tr>
<td>Highest Lateral Res.</td>
<td>FOV3</td>
<td>40 or 80</td>
<td>3 - 15</td>
</tr>
</tbody>
</table>
Start Up Procedure For When Computer or Software Has Been Shut Down

1. Turn on computer (You will not need to log into Windows)

2. Double click on the “Zones” icon on the desktop and click OK on the error message that appears. The Zones taskbar should appear at the top of the screen.

3. Double click on the “Shortcut to Manager” icon on the desktop to open the manager software

4. Click on the Window menu and:
   b. Open Real time display.

5. Left click on the Zones button in the top task bar and choose “Control”

6. Drag the manual window into the manual zone

7. Drag the real time display window into the manual zone and into the manager zone

8. Calibrate the stage as follows:
   a. Go to the manipulator section in the Instrument Manual Control window.
   b. Click on the “parameters” box.
   c. Click on “calibrate axes” (Verify that the dual anode gun located on the back of the SAC chamber is all the way out (away from the sample stage), and verify that nothing is sticking out from the sample stage.).
   d. Click on “confirm request”. The stage will go through a series of motions to reach the stage limits and calibrate the stage position.
   e. When completed, the calibrated light will illuminate green.
Start Up Procedure of XPS-Kratos After Power Failure

- Check cooling water.
- On the main unit, go to Vacuum Control Unit:
  1. Options.
  2. System.
  4. Set.
  5. Engineer.
  8. Gauges.
 10. On.
- Start computer.
- Open Instrument Manual Control.
- Scroll down to Vacuum Control session.
- Set to Safe Manual.
- Read the vacuum in Vacuum Control (P.G.).
  1. If the number is better then $2 \times 10^{-1} \tau$, open V3.
  2. Then turbo pump will be opened.
- Turn on CCG of STC, and check the vacuum, wait until it is better then $3 \times 10^{-7} \tau$.
- Turn on CCG of SAC, and check the vacuum.
  1. If the vacuum is better then $3 \times 10^{-8} \tau$, go to step A.
  2. If the vacuum is around $3 \times 10^{-7} \tau$, go to step B.

Step A:
  1. Turn on SAC Ion Pump on windows.
  2. Turn on the switch on the unit.
  3. Set Voltage to 5kV.
  4. Wait for vacuum to reach $10^{-9} \tau$.

Step B:
  1. Open Flat valve.
  2. Once the SAC vacuum is better then $10^{-8} \tau$, close Flat valve.
  3. Go to step A.
**Instructions to Vent the Entire System**

1) Open the “Instrument Manual Control” window.
2) Open STC/SAC Valve (aka flap valve).
3) Open V2 (Ion Gun Differential Pumping Valve).
4) Turn off Ion pump high voltage.
5) Turn of SAC gauge (CCG attached to SAC).
6) Close V3.
7) Turn off turbo pump.
8) Open V5 (vent valve) for 2-3 seconds. Then close the valve. Wait until turbo spins down (30-60 seconds), then continue venting by opening V5 again.
9) Loosen SEC door screws to allow overpressure to escape.
10) Leave the vent valve (V5) open to allow purging of the vacuum chamber until ready to pump. Close the Vent Valve (V5) on the Instrument Manual Control window, when vent operations are completed.

**Instructions to Pump the Entire System**

2) Close V5 (if not already done).
3) Turn on the Rough Pump (if not already on) - switch on side of pump.
4) Ensure that V2 (Differential Pumping Valve) and STC/SAC Valves are open.
5) Open V3 - tighten SEC door screws-allow system to rough until Pirani Gauge shows $10^{-2}$ range.
6) Start Turbo Pump.
7) Wait at least 20 minutes before turning on the CCG’s. Turn on SEC CCG gauge in software. Ensure range is in low $10^{-6}$ range before turning on SAC CCG.
8) Once vacuum is adequate (approx. $1E x 10^{-6}$) turn on power to ion pump in manual control screen. Turn on ion pump high voltage at the controller (be sure it is set at 3kV). Ensure that pump starts and that the base pressure is $10^{-8}$ range prior to changing to 5kV or 7kV settings.
The Instructions For Backout The Instrument

1) Remove the camera and illumination source.

2) Turn off Stage Control Unit and remove the white DB-25 cables to Iris/Aperture and Z-Axis motors and grey DB-9 cable between Iris and Aperture drives.

3) Remove Intro Rod Sensor from shaft on load lock apparatus after ensuring that the STC/SAC valve is open.

4) Turn off SEC CCG and remove rom the STC/SEC chamber (STC CCG can be left on if it is outside of the baking blanket, check it periodically to prevent overheating). Leave CCG to SAC connected. System must have this gauge on to monitor pressure during bake-out and maintain Vacuum Interlock switch.

5) Install blanket.

6) Flip ‘Baking Supply’ breaker to the ‘on’ position, if not already there.

7) Set up VCU for system bake out. Select ‘Main Menu’ then select the ‘Auto’ button. Push ‘Baking’. Select ‘Set Bake’. Bake time is the total time for the system bake including cool time. Set total bake time and add the amount of cool time desired to the bake time or it will subtract from your bake time. Select ‘Set Cool’ and set cool time. (Consult VCU manual - procedure is tedious.)

8) Press ‘Start’, if o.k. to bake you will see a message saying ‘System Status is O.K.’. Press ‘Start’ again. Bake Time will begin its countdown and display any error messages. It will also open the Ion Gun differential pump valve and the STC/SAC (flap) valve. VCU set up requires that we turn off the STAGE CONTROL UNIT. You will be prompted to do this. (The message will continue to scroll even after you turn the Stage Control Unit off, this is normal).

NOTE: All cables to the analyzer are bakeable.

- Reminder: Bake out begins by selecting START in the VCU (see page 28 in VCU manual). In the main menu of the VCU select AUTO. Select BAKING. Select START. Then press START again.
- Error messages will be displayed both in the VCU display window and in the Instrument Manual Control window on the computer.
- If X-ray water flow to the Monochromater Crystal falls below 1 litre per minute of water flow the bake will shut off. Check water pressure increase if necessary, pressure controls are located inside the chiller chassis. Also check for blockage in water lines.
- The Vacuum Interlock signal is provided by the SAC CCG, not the Ion Pump Controller.

- A word of caution: if you leave the baking menu during a bake-out, you will lose the sequence status display. Although the bake will continue, you will not be able to tell where you are.
Outgassing New/Dirty TSP Filaments

- When out-gassing new or particularly dirty TSP filaments here are some helpful hints:

1) The Varian controller will not let you begin out-gassing below 30 amps. It will also not allow you to ‘fire’ the TSP filament for less than 1 minute. This condition will shut off the controller as it is located on the Protected Mains.

2) The Ion Pump will also be shut off during the first attempts to out-gas the filaments under these conditions. So, during the first few attempts to out-gas, turn the Ion Pump off. Turn it back on after the filament times out to assist in returning to proper vacuum pressure if this is a problem.

3) Recommended initial time to out-gas is the minimum 1 minute to not overshoot the pressure. Slowly increase this time up to 2 minutes at 30 amps or until the filament begins to act normally.

4) Do this for all three filaments.

5) Choose a filament for full operation. Begin increasing amperage to a max of 45 - 50 amps with the Ion Pump turned on as this becomes possible. You may have to keep the Ion Pump voltage at 3kv initially until the TSP filaments become clean as this requires the lowest Ion Pump current.

6) Once 45 amps can be reached for 30 seconds without the pressure reaching $1 \times 10^{-6}$ Torr, then set the filament current to 48amps, and the timer to 2 minutes with a 10 minute cycle. Leave this way for the first hour or two. Then set to 30 minute cycles for the next couple of hours. After this, switch to one hour cycles for the next 24 hours. If heading into a weekend, set to 4 hour cycles for the period, instead of 1 hour cycles.

7) Base pressure should be reached after this. The TSP can then be manually cycled one time, once or twice a week as necessary to keep good vacuum in the SAC.
**Outgassing X-ray Sources and Ion Gun**

The software will do initial degas of all x-ray sources automatically over a slow ramp with no high voltage.

See the figure below. In the Manual Control Window, in the X-ray PSU section, select the degas check box. Move between the filament choices (Mg, Al, Al Mono), and select ramp times and maximum currents. Default values for the Al and Mg sources are shown in the figure below. For the mono, which runs at a higher current, values of 10 minutes to attain 4.0 amps and 20 minutes to attain 4.5 amps should be sufficient.

Once the values are set for each filament, the On button for degas (Green in the figure below) should be selected.

This is also true for the ion gun filament. Values shown in the figure below are for the MiniBeam I ion gun. Values of 10 minutes to attain 1.7amps and 20 minutes to attain 2.2 amps should work for a MiniBeam III ion gun.

Once the values for the filament have been selected, the degas button should be selected. Ensure that the differential pumping valve (V2) is open. This will require full manual control of the vacuum control section in the Manual Control Window to be opened by clicking on the valve. If this is not accessible or acceptable, please ensure that the manual leak valve is closed, then use the "Ion Gun Gas On" button in the Automatic Sequences. Using the automatic sequences will also purge the argon gas line once the "Ion Gun Gas Off" is used at the end to close V2.

Once the degassing of these filaments is complete, the SAC should be isolated from the SEC and the sublimation process continued.