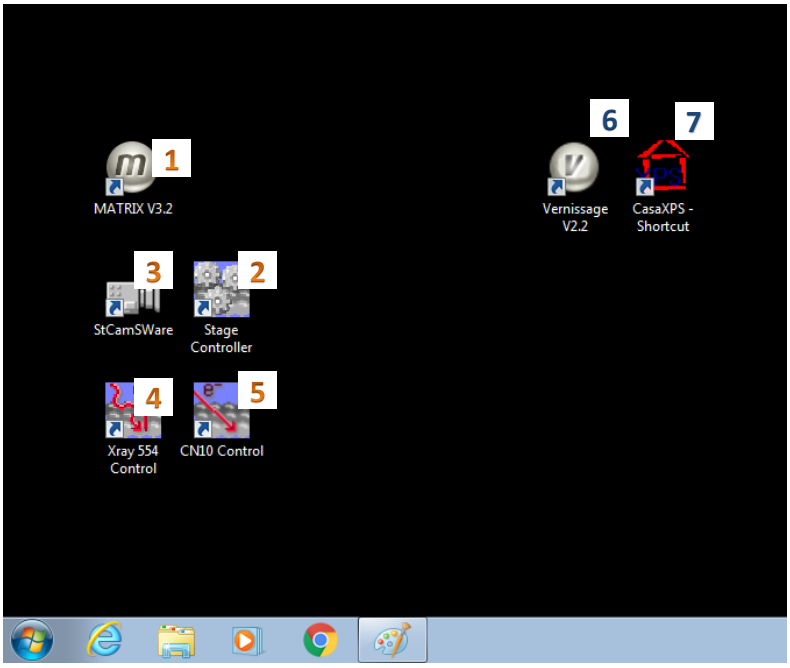
Load your sample:

1. Check: The analysis chamber pressure is <5E-9 and turbo 100% button is lit up.
2. Press **Close Valve** then **Turbo on** to isolate turbo pump and wait for the turbo pump to slow down and vent. **Wait until the vent indicator** (the amber LED by the turbo pump under bench) **stays on**. (~25 minutes)
3. Wipe clean the sample holder by IPA or acetone then attach your sample.
4. Immerse the vent tubine in liquid nitrogen and open the venting valve.
5. Unlock the sample loading door and wait until it can be opened
6. **Close venting valve and take away the liquid nitrogen**
7. Open the sample loading door and load your sample to the sample holder
8. Wipe clean the inside of transfer chamber and door seal. . Close and lock the sample loading door.
9. Check the turbo vent indicator is on (the amber LED by the turbo pump under bench). Press **Turbo on** to turn on the turbo pump then press **Open Valve** immediately to open turbo valve (if you wait too long, the pressure difference may make the valve not to open, then you need to press **Close Valve** and **Turbo on** and wait for ~20min for turbo pump to vent again so you can restart)
10. The turbo vent indicator should be off now. **Turbo setpt** on control panel should be on in several minutes. If not, check whether vent valve is closed and door seal is good.
11. Wait for the turbo to reach full speed then Press 'cycle' button to turn on the pressure gauge for the transfer chamber
12. Wait until transfer chamber pressure (the gauge on bench) reaches <7E-7 (usually ~30min). If it takes too long to pump down, the sample must be outgassing and cannot be transferred into analysis chamber. (To lower analysis chamber pressure, you may wait until the transfer chamber pressure reaches <6E-7)

Sample transfer

1. Open the gate valve. Monitor the analysis chamber pressure. **Close the valve if you see a sudden pressure increase**. Chamber pressure shouldn't be higher than **2E-7** when the gate valve is fully opened.
2. Transfer the sample holder into analysis chamber. There should be no rotation to the transfer arm or sample puck will fall off.
3. Lock the transfer arm in position
4. Move the sample to desired place by folk (Always **firmly hold** the folk or it can be forced into chamber and bump sample holder)
5. The folk is always placed on the column holder when not in use
6. Pull the transfer arm back and **close the gate valve**

XPS software



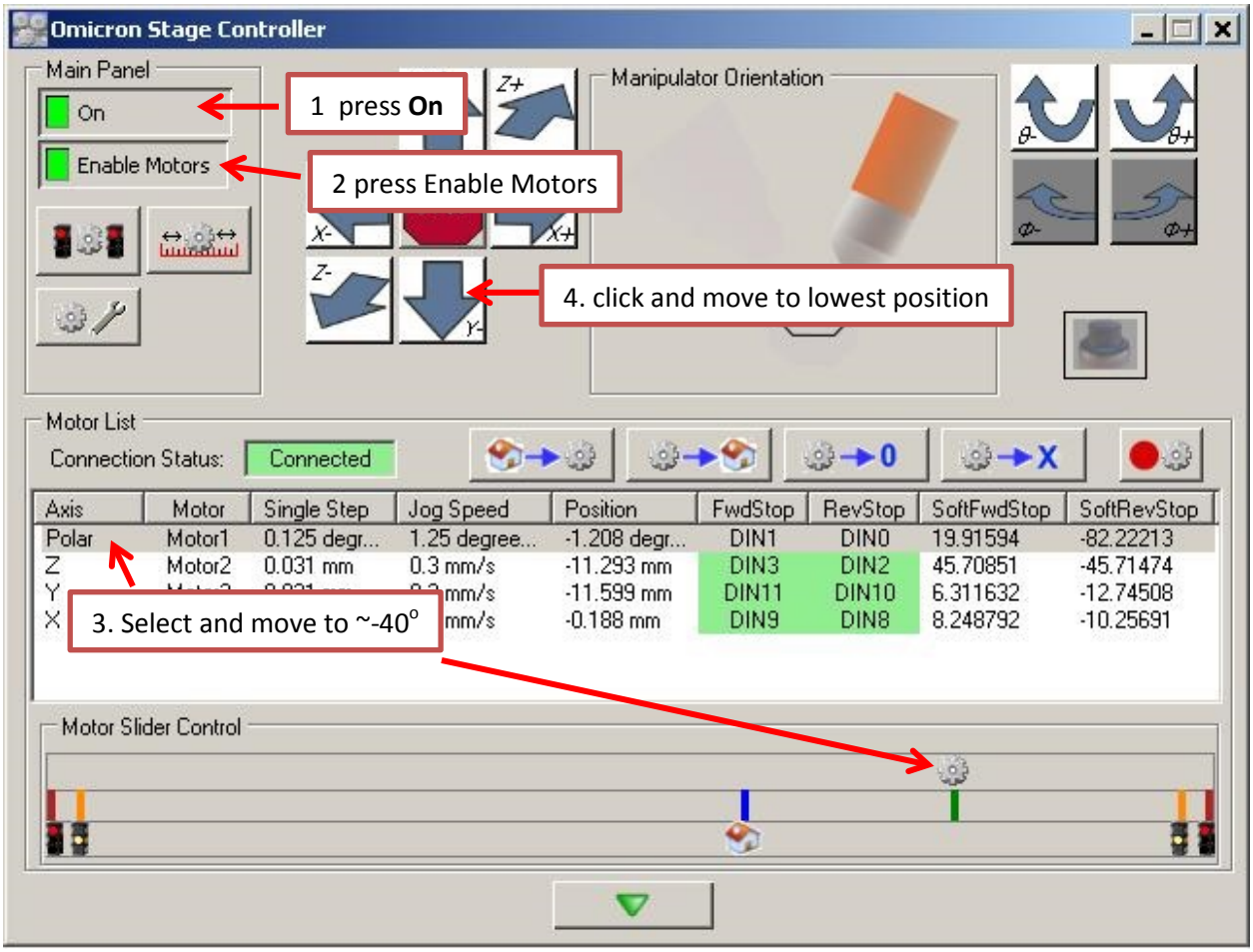
Softwares for XPS data acquisition

1. Matrix (if you see an alert window, close it and restart the computer)
2. Stage controller
3. StCamSWare (camera)
4. Xray control
5. CN10 (neutralizer for nonconductive materials)

Softwares for XPS data processing

6. Vernissage for data export
7. CasaXPS for data processing

Start Stage controller



* You can rotate the stage to any angle you want but need to check the sampling area.

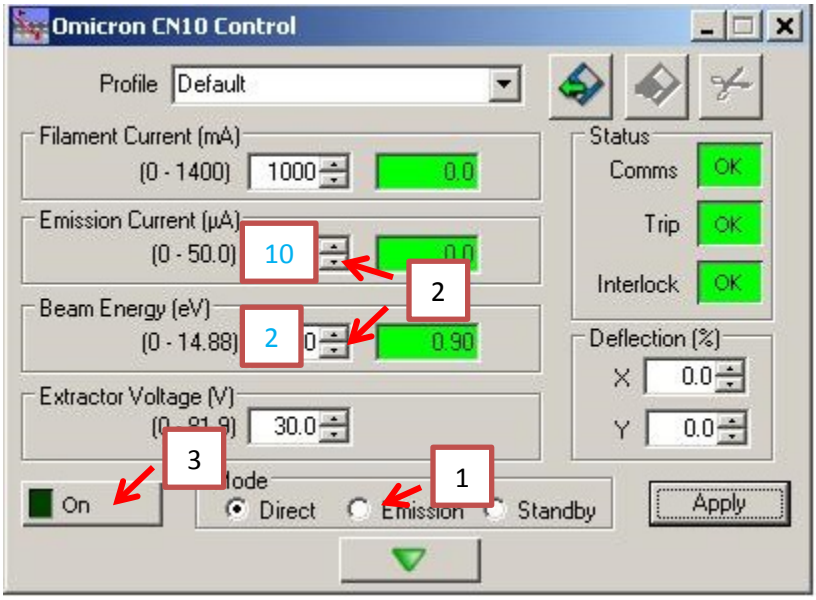
Start StCamSWare:

1. Click the + button on tool bar. Move the cursor to ~(**650, 320**) where the sampling area is centered and click to make a mark(or use laser) , move x+/x- , z+/z- until the center of your sample matches the mark

Turn off the stage controller to record current stage position for easy recovery from system crash.

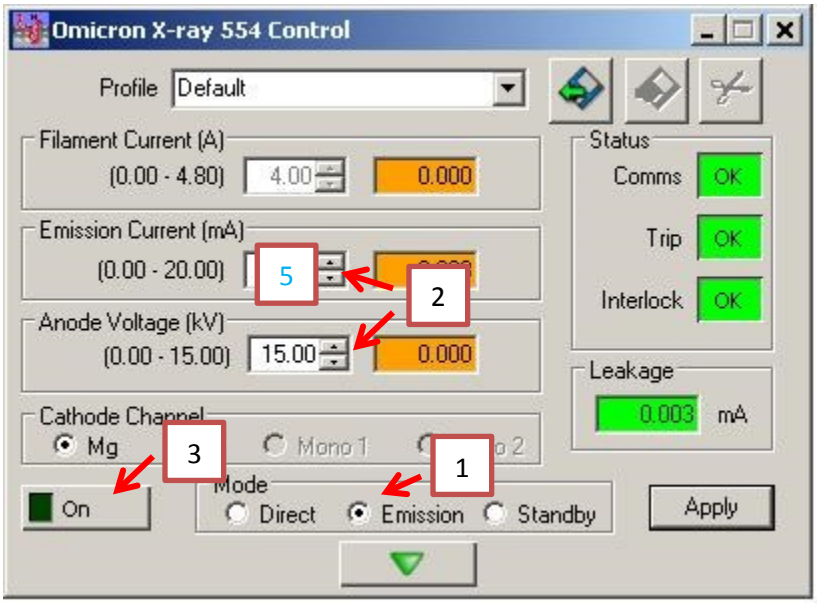
2. Click the + button on tool bar. Then move the cursor to ~(**770, 350**) and click to make a mark , lower the Xray gun until it matches the mark. The Xray gun needs to be at the same height to get comparable intensity.

Start Neutralizer (**CN10**) (required for non conductive samples)



1. Select Emission
2. Input Emission current and Beam Energy (recommend: 10 uA and 2eV) (you may need to check your reference peak for a more appropriate setting)
3. Click On

Start X-Ray

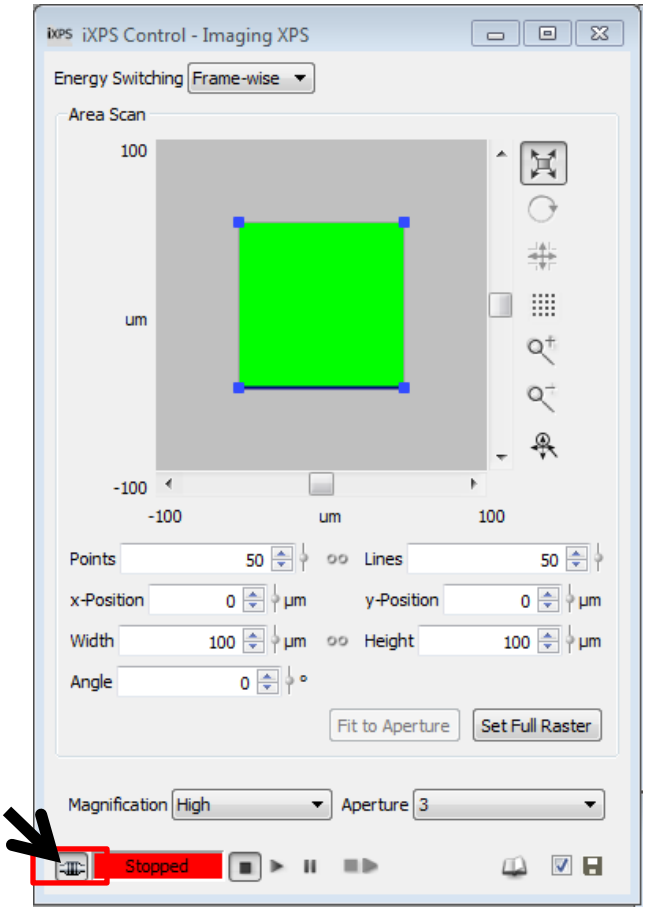


1. Select Emission
2. Input Emission current and Anode Voltage (for samples may outgas, start from current=5mA)
3. Click On
4. Monitor the analysis chamber pressure when the **Emission Current** starts to increase. Xray gun shuts off at 1E-7 mbar.
5. Wait until Voltage and current turn green
6. If you start from lower current setting, slowly increase the current to the maxima allowed by the sample or analysis chamber pressure

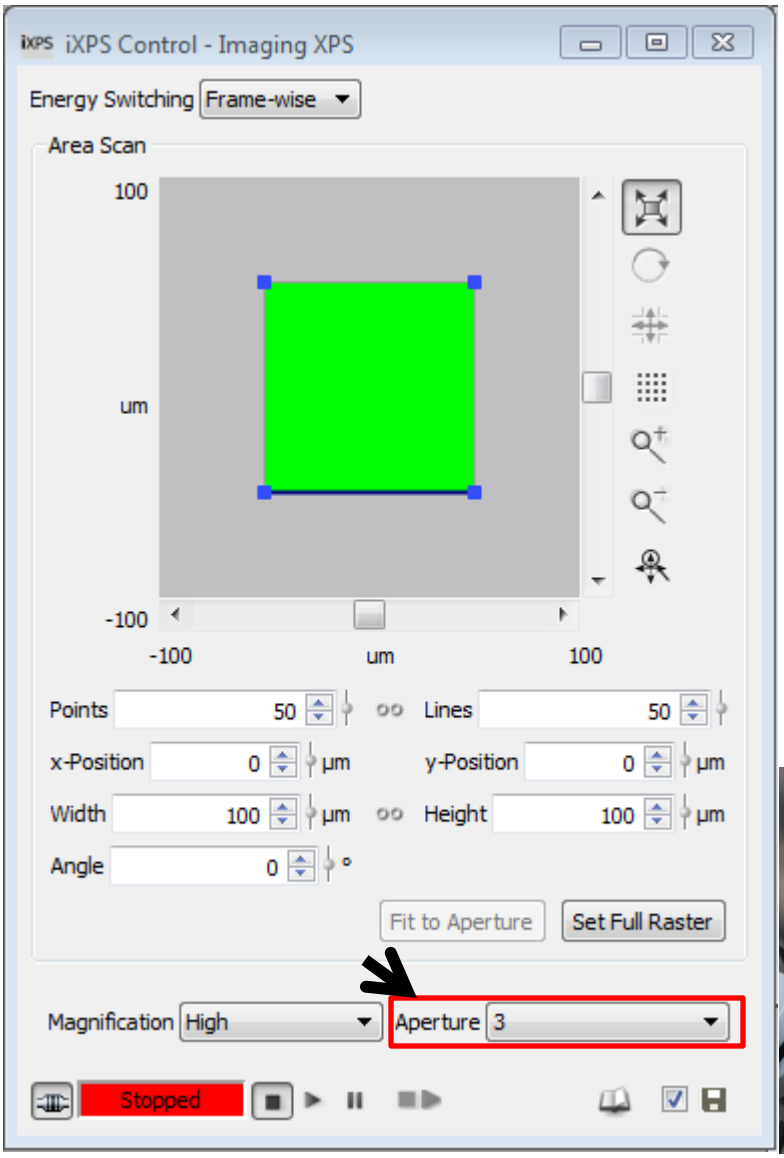
* You can switch X-ray to **standby** when not doing data acquisition. Then switch back to **Emission** right before data acquisition to minimize sample outgasing.

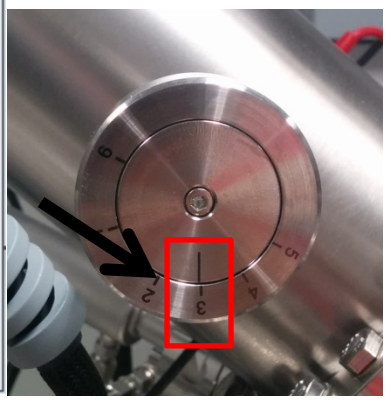
Matrix Quick start:

1. Start Matrix and select analyzer (the middle ‘S’ button in the toolbar)
2. Select iXPS
3. Connect electronics and input sample information

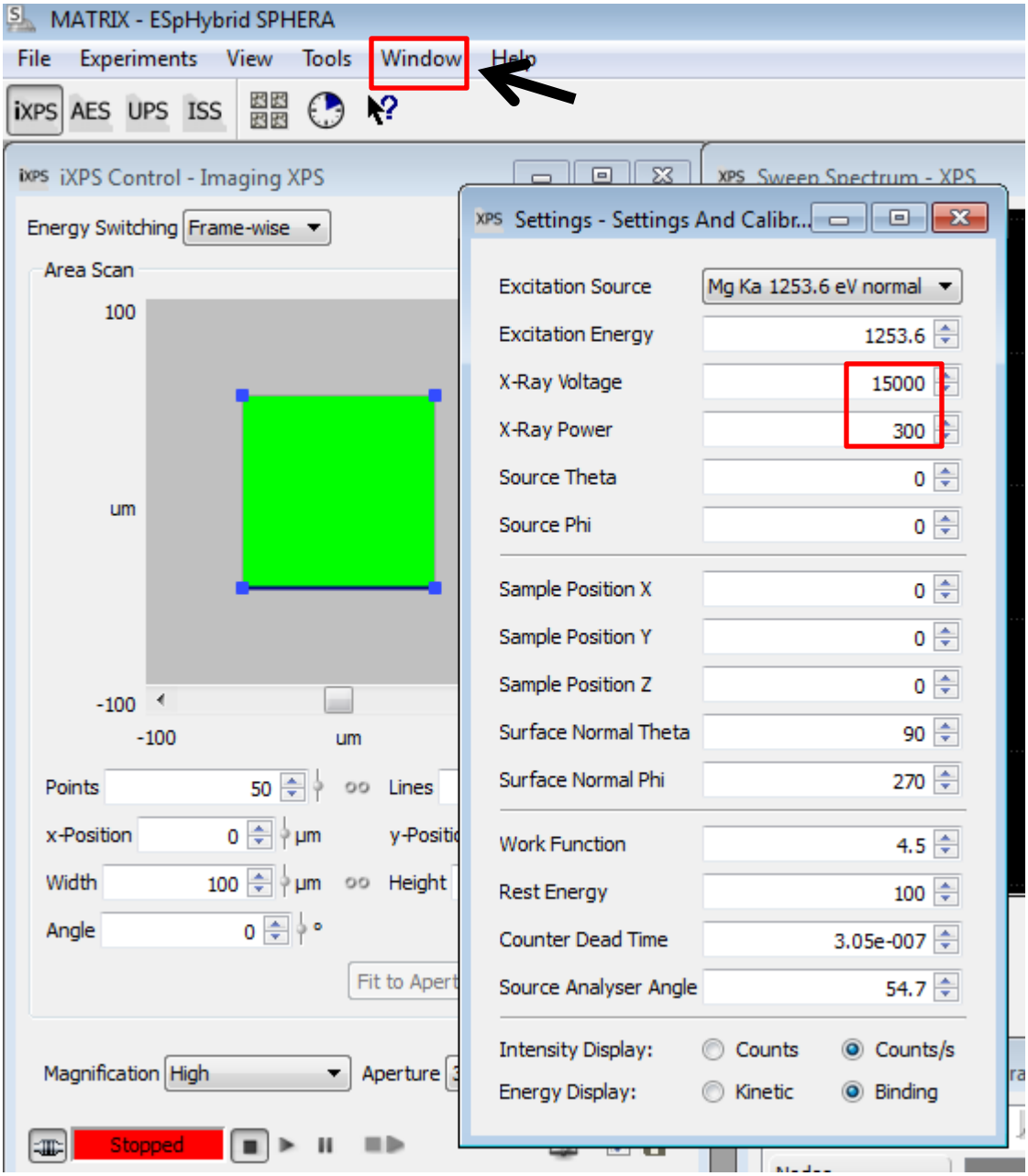


4. Select the Aperture in Matrix and the XPS **instrument part**

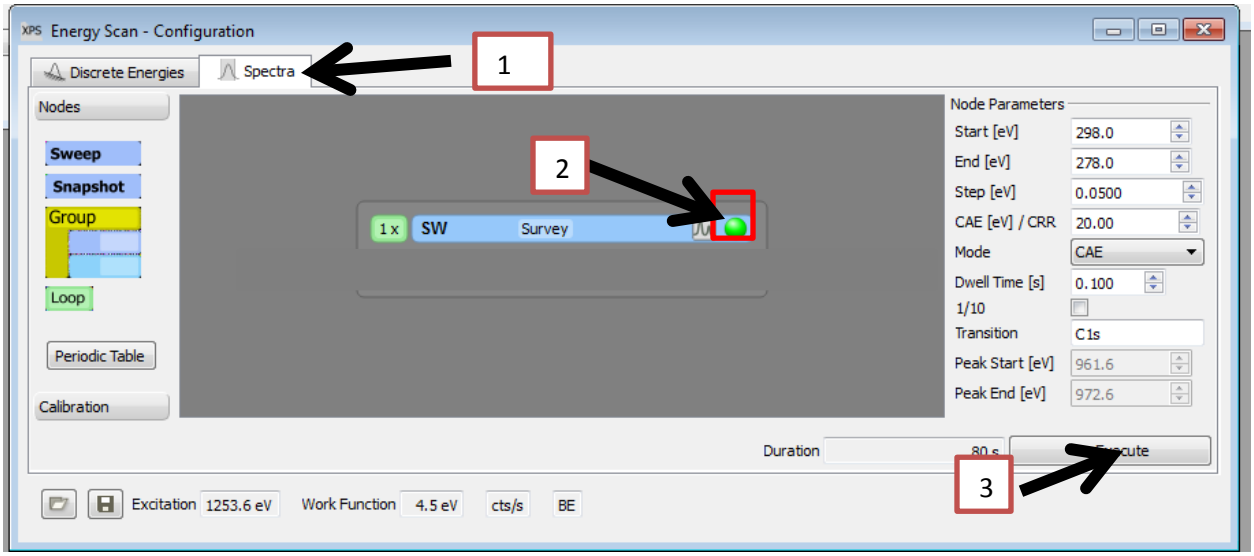




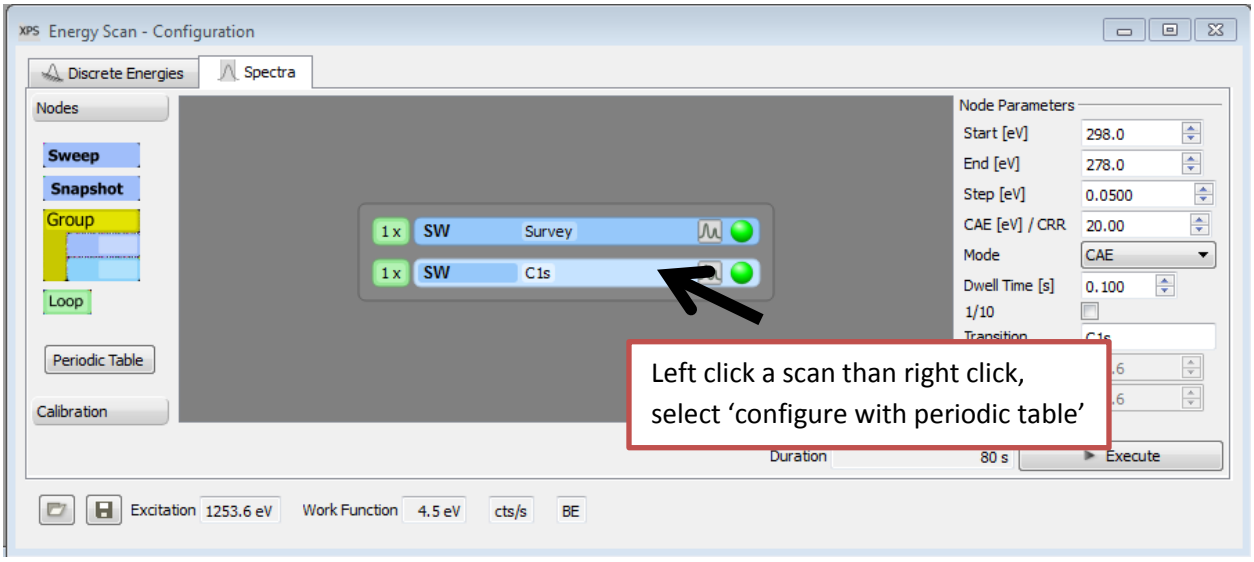
5. Go to window tab, select settings, then input Xray power in setting (anode voltage, power= anode voltage x emission current)

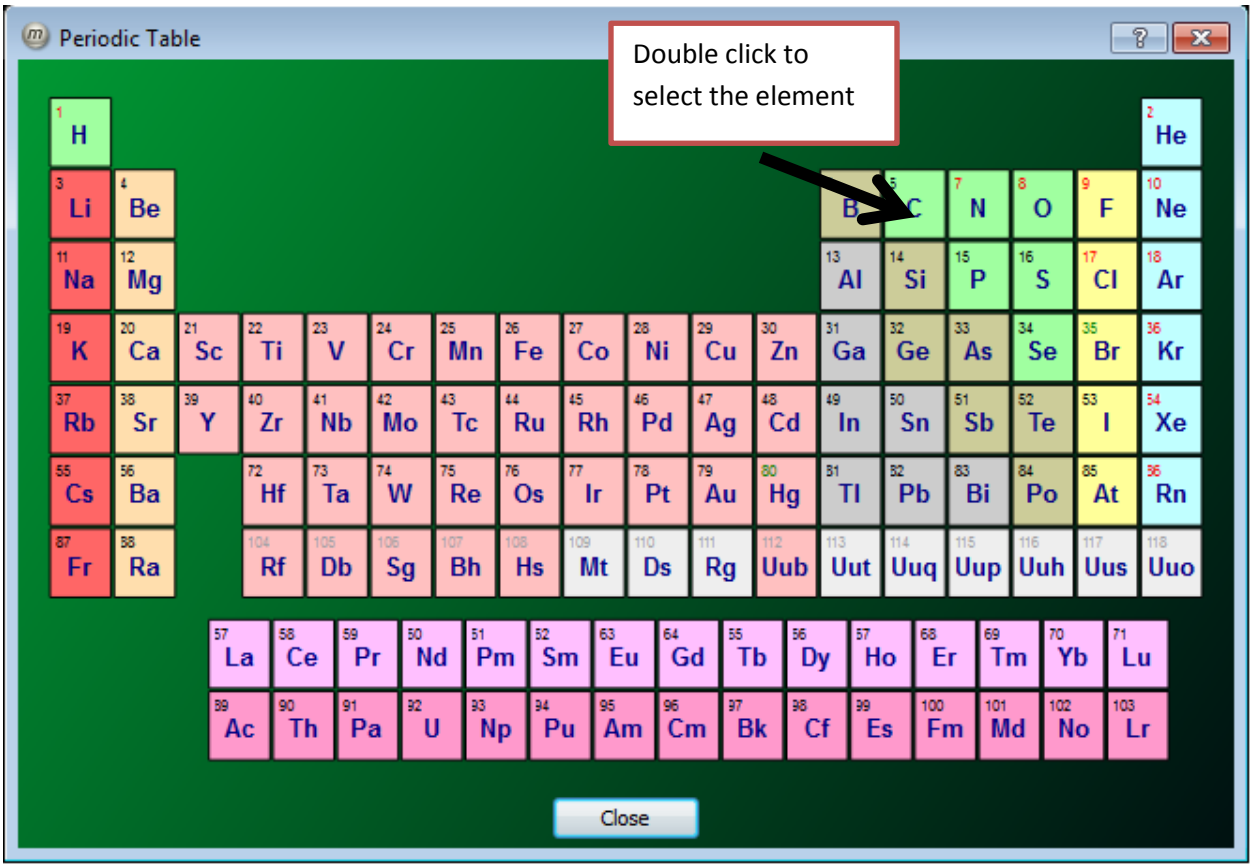


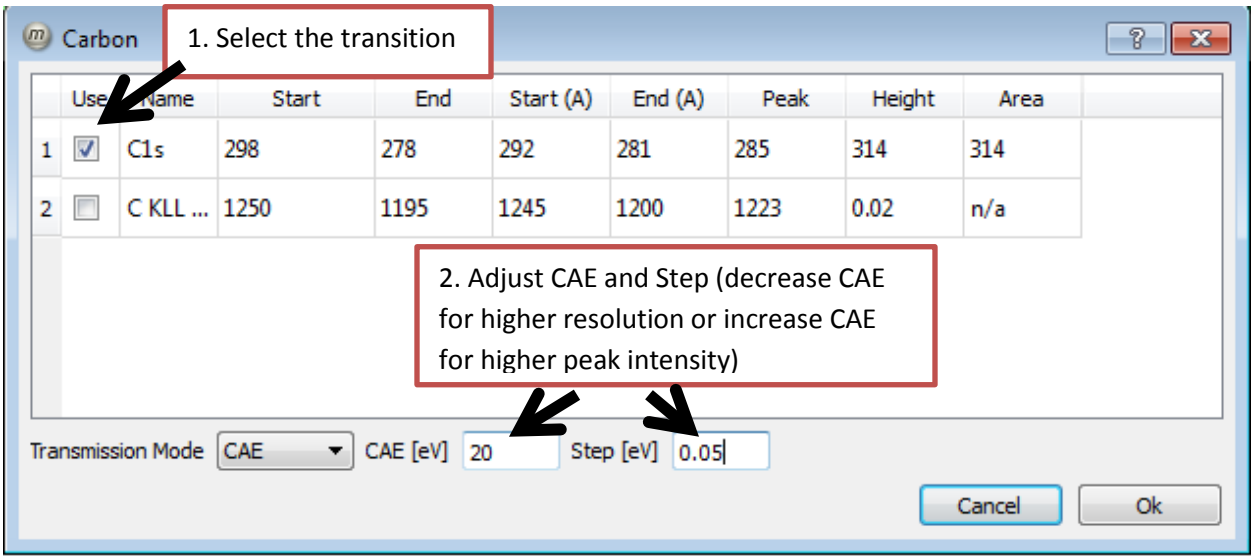
6. select a survey scan then Click **execute** to run



7. Live region alignment
8. Setup high resolution scans by periodic table (input CAE and step)




Periodic Table
Double click to select the element
Close


Carbon
1. Select the transition
Use
Name
Start
End
Start (A)
End (A)
Peak
Height
Area
1
C1s
298
278
292
281
285
314
314
2
C KLL ...
1250
1195
1245
1200
1223
0.02
n/a
2. Adjust CAE and Step (decrease CAE for higher resolution or increase CAE for higher peak intensity)
Transmission Mode
CAE
CAE [eV]
20
Step [eV]
0.05
Cancel
Ok

9. Adjust dwell time and scan number according to peak intensity in the survey scan

10. **Save the setting in matrix for easy recovery from system crash**

11. Click **execute** to run all the activated scans

Suggested parameters:

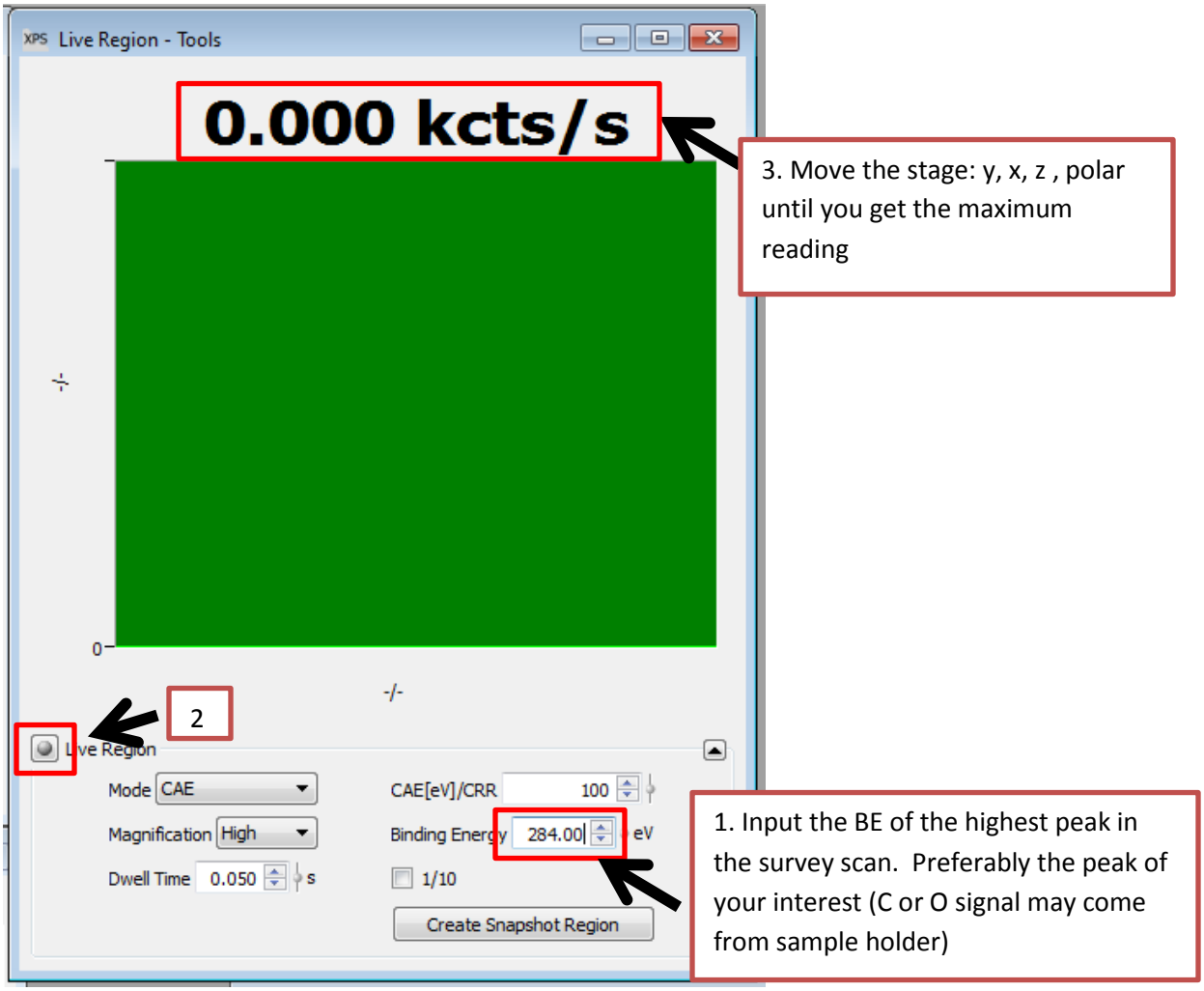
	start	End	Step (ev)	CAE	Dwell time(s)
survey	1100	-10	-1 (lower if you need to do quantitative analysis using survey scan)	100	0.05
High resolution			-0.05	20 (lower: lower intensity but sharper peak. Higher: higher intensity but broader peak)	0.05 (increase to improve S/N ratio for weak peaks)

For peaks of very low intensity:

1. Use aperture 5 (make sure your sample is large enough and lens is focused on your sample)
2. Increase pass energy (CAE 150, for example)
3. You may also need to increase dwell time and scan numbers.
4. If the Xray gun is at 300W, carefully lower the Xray gun further (the chamber pressure should be low enough)

Align the sample using Live region

1. Run a survey scan and pick the binding energy of the highest intensity peak or the peak of interest
2. Input the binding energy in the Live Region window



3. Click 'Live Region' to start the real time reading. If the reading is higher than 1M cps, pick a peak of lower intensity if applicable or don't keep the real time reading on for too long.
4. Adjust position of y axis first by stage controller until the reading reaches the highest intensity
5. Adjust x, z and polar if necessary until the reading reaches the highest intensity
6. Stop live region

Switch your samples

1. Switch X-ray from '**Emission**' to '**Standby**'
2. Lift the X-ray gun to higher position
3. Rotate the stage to ~-22 degree
4. Switch your sample with the one on the parking place
5. **To switch the sample with the ones in transfer chamber**,
    1) make sure the transfer chamber pressure is E-7 ~ E-8
    2) Open the gate valve. Monitor the analysis chamber pressure. **Close the valve if you see a sudden pressure increase** to midE-7
    3) Transfer and lock the transfer arm in position
    4) Move the sample to desired place by folk
    5) Move the transfer arm out and close gate valve
6. Turn the stage back to ~-40 degree
7. Move the cursor to ~(**650, 320**) in StCamSWare and center your sample
8. Move the cursor to ~(**770, 350**) in StCamSWare and lower the X-ray gun until it's close to the mark
9. Switch X-ray to '**Emission**'
10. If the X-ray is off, click **ON**
11. Align the sample by Live region
12. Change your sample information in Matrix
13. Start data acquisition

Finish:

1. Switch X-ray from '**Emission**' to '**Standby**'
2. Turn off neutralizer and close the window
3. Lift the X-ray gun to high position
4. **Disconnect the electronics** in Matrix then close the windows
5. Rotate the stage to ~-22 degree
6. Turn off stage controller and close the stage controller and StCamSWare windows
7. Turn off X-ray and close the window

Unload your samples

1. The transfer chamber pressure is E-7 ~ E-8
2. Open the gate valve. Monitor the analysis chamber pressure (the one on electronics rack). Close the valve if you see a sudden pressure increase to E-7
3. Transfer and lock the transfer arm in position
4. Move the sample to desired place by folk

5. Move the transfer arm out and **close gate valve**
6. Press 'cycle' button to turn off the pressure gauge for the transfer chamber
7. Turn off light
8. Press **Close Valve** then **Turbo on** to isolate turbo pump and wait for the turbo pump to slow down and vent. **Wait until you hear the vent sound**. (~25 minutes) (you can process your data while waiting)
9. Immerse the vent line in liquid nitrogen and open the venting valve.
10. Unlock the sample loading door and wait until it can be opened
11. **Close venting valve**
12. Open the sample loading door and unload your sample. Close and lock the sample loading door.
13. Check the turbo vent indicator is on (the amber LED by the turbo pump under bench). Press **Turbo on** to turn on the turbo pump then press **Open Valve** immediately to open turbo valve
14. Wait until turbo setpoint light is on in a couple of minutes. If not, check whether vent valve is closed and door seal is good.

Logout the computer

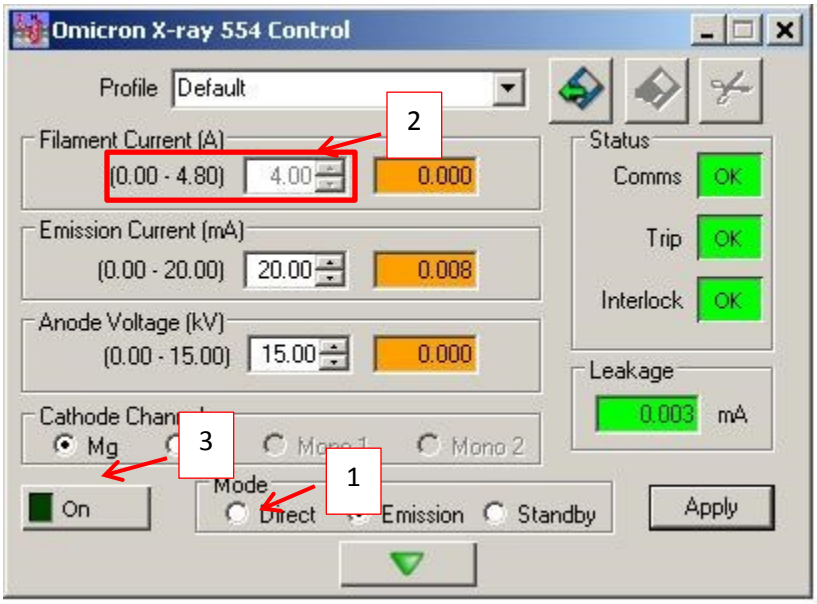
Ar sputter:

1. Turn off neutralizer (CN10)
2. Turn off X-ray gun
3. Lift the X-ray gun to high position
4. Turn on the ISE 5 gun controller (usually it displays 0.06)
5. Set the knob to **beam energy** and increase the beam energy to 5.05
6. To monitor the current during sputtering:
   1) Connect the blue&green leads to the red clip and grounding to black clip
   2) Set the knob to **current** and monitor current increase
7. Turn on the main Ar valve on the wall
8. Turn on then off the Ar in valve (Knob at the back of the XPS. The valve must be off during sputtering)
9. Slowly turn on the Ar leak valve (press the valve arm frequently to avoid sudden pressure change) until the chamber pressure starts to increase slowly to ~5E-6. You should start to see the current increase on the ISE 5 gun controller.

   (If you accidently put in too much Ar, the pressure gauge may be turned off. Close the Ar leak valve first then turn the pressure gauge on.)

10. Leave the setting on for 5-10 minutes
11. Set the knob to **beam energy** and Decrease the beam energy to minimum.
12. Turn off Ar leak valve immediately (don't over tighten it)
13. Turn on then off the Ar out valve (Knob at the back of the XPS. The valve must be off when you finish)
14. Turn off the main Ar valve on the wall
15. Connect the blue,green and ground leads
16. Wait for the pressure to decrease
17. Turn on the neutralizer (CN10)
18. Depending on your sample, it may be necessary to degas the X-ray gun first.
19. Turn off ISE 5 gun controller

X-ray gun degas:



1. Select '**Direct**'
2. Input 1.4 A to start
3. Press '**on**'
4. The analysis chamber may increase then decrease
5. Increase the filament slowly (every 0.2A at beginning) and wait for the chamber pressure to stabilize
6. Once the filament current reaches 4A, switch to **Emission**

UPS

1. Turn off and lift the Xray gun to high position if you used it
2. Turn on water chiller to UPS and He gas tank
3. Both valves to the UPS are closed
4. Turn on the UV source power supply. You should see 'Ignition locked' flashing.
5. Flash He gas line by turning on/off the gas inlet valve and purge valve alternatively
6. Turn on the He gas inlet
7. Slowly turn on the He leak valve until the chamber pressure starts to increase slowly
8. Turn on the stage 1 pump valve (you may need a wrench) immediately and stage 2 pump valve
9. Continue to increase He until the **He pressure** is 5E-2 .
10. Set current to 100mA and voltage to 900V by turning the current and voltage knob.
11. Press the **Ignition** button. The current should be on after a couple of ignition
12. If the current is not on, increase the He pressure slightly until the lamp is on. Don't exceed 8E-2.
13. Check the UV light right away. It should be on and inside of the tube. If the UV light is not visible or glowing outside of the tube. Turn off UV power supply right away and turn off He leak valve. Stop your experiment and Ask Dr Jing Wu for help.
14. Increase the He pressure if necessary. It's not advised to increase the He pressure too much.
15. Check the UV light is still on.
16. Set UV current to ~90mA

Start Matrix and choose UPS

1. Connect the electronics and select the right aperture.
2. Set the UPS acquisition parameter in energy scan window
3. Align the sample by live region
4. Run UPS acquisition

Finish UPS

1. Decrease the current to 0
2. Turn off He leak valve
3. Turn off Stage 1 valve. **Tighten with a wrench**
4. Turn off He gas inlet and He gas tank
5. Switch UV power source to 'standby'
6. Turn off chiller
7. Leave Stage2 valve on for 10 minutes or until there is no pressure increase when you turn it off
8. Turn off Stage 2 valve

Useful online resources:

CasaXPS manual: http://www.casaxps.com/ebooks/ebooks.htm

CaseXPS: http://www.casaxps.com/

ThermoXPS XPS knowledge base: http://xpssimplified.com/periodictable.php#

NIST XPS database: http://srdata.nist.gov/xps/main_search_menu.aspx

Aperture (high magnification):

	Analysis area (mm)	Lateral Resolution	a	b
A1	Round 0.066	54 um	1	0
A2	Round 0.198	136 um	1	0
A3	Rectangular 0.311x3.22 (recommended for high precision quantitative analysis)		304.3	0.91
A4	Round 1.93	1.11mm	24.17	0.24
A5	Rectangular 1.54x4.09		39.2	0.43
A6	Rectangular 1.23x4.66			