

PHI5000 VersaProbe Operation Notes

Standby Condition

- Recover the specimen then pump down the intro chamber. Confirm that V3 is open and the intro is evacuated.
- Confirm X-ray, neutralizer, sputter guns (Ar and C60), monitors (three of them), bias power supply and optical light **OFF**; thermo-valve at **LIMIT**; C60 isolation valve, both UPS pumping valve **COLSED**. Turn off the power extension socket at right-hand side under the table will turn off all the monitors and optical light.
- Record and sign the usage check list. Note problems if any.

Introduce Specimen

1. Move the intro-arm all the way out then vent the intro chamber by clicking “backfill intro” in *Watcher*.
2. Open the intro chamber and mount the specimen holder on the fork. Make sure that the fork is at the lower groove and the notch fits the screw on the fork and the holder should not be able to freely rotate or move on the fork. The cut at the under-side of holder needs to be parallel to the intro-arm and the holder sits in the round cut of the fork.
3. Move the intro-arm all the way out and pump the intro chamber by pressing “pump intro” in *Watcher*. A 20 minutes count-down will be started and the vacuum gauge should reach its minimum (shown as 0.00E0 Pa) within 0.5 minutes. If vacuum cannot reach 0E0 within 1 minutes, remove and clean the specimen before retry. If it always takes more than 1 minutes, the sample is not compatible with UHV and cannot be used. Record the time on the log book.
4. A 5 min count-down will be started once minimum reading (0E0) is reached. This is a safety measure and cannot be by-passed. While waiting this count-down, press “intro” on the stage control of *Summitt*. This will move the stage to the position for accepting specimen holder.

Double check the stage position as follows to ensure the stage is ready for accept sample.

X= 7.000 mm, Y= 0.800 mm, Z= 9.080 mm, R= 0.0 °, T=45°

If need to abort the transfer procedure, press “Abort”.

5. Once the vacuum is ready and stage moved to its home position, press “transfer sample” in *Watcher*. V1 will open and the intro-arm can be moved into the main chamber. Record the chamber pressure on the log book at this point.
6. Carefully move the intro-arm into the main chamber while observing the chamber from the view port. The copper-ceramic piece in the center of stage should go through the cut under the holder and never touch each other. The bottom groove of the holder should slide smoothly between the cover plate and the holding piece. If the holder tilts due to the friction, pull the intro-arm out a little to allow the holder to be flat then push back in

again.

7. Click “up” in the *Summitt* while observing the movement of the stage. The stage will move up to release the holder (Z= 10.480 mm).
8. After the stage movement is finished, carefully move the intro-arm all the way out. V1 should be closed and V3 should be opened automatically.

Transfer Sample from Protective Environment

1. Load the sample into the transfer vessel and seal it. This process can be done in glove box so that the sample will not be exposed to air. **Mind the orientation of the holder for the notch on holder to fit to the screw on the fork.**
2. Vent the intro chamber and unscrew the two thumb-screws then remove the cover (two pieces). Move the fork all the way out.
3. Put the adaptor from the kit of transfer vessel and firmly screw it in place then put the transfer vessel on the adaptor.
4. Pump the intro chamber and wait for good vacuum. Repeat pump-vent for three times to ensure the cleanness of the environment.
 - If the vessel is under vacuum already, open the vessel and lower the sample.
 - If the vessel is under protective gas, vent the intro chamber then pump it again and open the vessel while V6 is opened (first stage of pumping that usually lasts for less than 30 sec). **Never open the vessel full of gas when V3 is open as the sudden change in pressure WILL damage the turbo pump.**
5. Lower the sample all the way down then pull it slightly up by about 1.75 turns. At this height, the fork should be able to grab the lower groove of the sample stage.
6. Move the vessel all the way down carefully and retract the fork.
7. Close the vessel and make sure it will not hit the sample.
8. Send the sample to the main chamber as usual.

Extract Specimen

1. **Make sure that thermo-valve control is at “limit” and V3 is opened for more than 5 minutes. If it is not opened, click “pump intro” in *Watcher* and wait for 5 minutes.**
2. In *Summitt*, press “extract” to move the stage to the position for removing holder.
3. After stage stopped moving, open V1 by clicking “transfer sample” in *Watcher*.
Double check the stage position as follows to ensure the stage is ready for remove sample.
X= 7.000 mm, Y= 0.800 mm, Z= 10.48 mm, R= 0.0 °, T=45°
If need to abort the procedure, press “Abort”.
4. Slowly insert the intro-arm into the chamber. The fork should be parallel to the cover plates that hold the holder.
5. Click “down” in the *Summitt*. The stage will be lowered and the lower groove of holder sits on the fork (Z= 9.080 mm).
6. After the stage movement is finished, move the intro-arm all the way out. V1 should be

closed automatically.

7. Backfill the intro chamber and recover the specimen holder.
8. Move the intro-arm all the way out and pump the intro chamber.

Operating Ar Ion Gun (for sputtering and neutralization)

1. In *Watcher*, press “diff vlv open” to open V4 (differential pumping for Ar gun).
2. Switch the thermo-valve control box to “*set point*” and set to *standby* mode.
3. Check the Ar ion pressure by clicking the checkbox of “extractor pressure”. **The pressure should be about 15 mPa.**
4. Before removing sample, switch the thermo-valve control back to “*limit*” then press “pump intro” in the *Watcher*. **Wait for at least 5 minutes before press “transfer sample”.**

Operating C60 Ion Gun

1. Open the isolation valve manually. Set the sputtering mode as “*startup*”, press “load” and start sputtering. After a couple of minutes, the temperature should start to increase slowly. If it does not, press “load” again.
2. **Wait until the temperature stabilizes at 368 °C.** This will take about an hour.
Do NOT operate either Ar or C60 ion gun at this period.
3. **Before put it at “10kV10nA” for operation, make sure the pressure is below 1E-4 Pa. The Imeter should be around 20 nA when sputtering at 10kV10nA.** If the Imeter shows negative value in sputtering mode, press “load” again.
4. When finished, put it at “*idle*” and sputtering for 30 minutes.
5. **Close the isolation valve and set sputter mode to “*statup*” before turn it off.**

Suggested Acquisition Parameters

- In general, small pass energy will yield better energy resolution. However, the signal intensity will be weaker and need longer time to achieve enough signal-to-noise ratio.
- For fast survey scan, pass energy: 117.4eV; energy step: 1eV
- For regular elemental range, pass energy: 58.7eV; energy step: 0.5eV
- For high-energy resolution, pass energy: 23.5eV; energy step: 0.2eV
- For mapping or fast profiling, unscan the spectrometer and set pass energy for desire energy range.

General Operation Procedure

- **For quickly set default acquisition parameters, load "z:\default.pas" from the top of Acquisition Setting window. This setting file will set for the e+i neutralization during auto-Z and acquisition, XPS mode with energy scanned, 100um25W beam for acquisition, 10um1.25W for SXI, 0-1400 eV range with 187.85 eV pass energy and 1 eV step, no sputtering and no angle resolve.**
1. Prepare the sample by sonicate in appropriate solvents for at least three minutes.

2. Mount the sample on the holder and confirm that the height is uniform and the highest point is at the center.
3. Introduce the sample (see above).
4. For insulating sample or sputter is required, put neutralizer standby and follow above procedure to make desired ion gun standby. Note that C60 will require about 1h to startup.
5. Move stage to X=0, Y=0 position while not changing Z, R, and T.
6. For insulator, set both Auto-Z Neutralize ON and Auto-Z Ion Neutralize On.
7. Set Auto-Z viewer on to observe counts.
8. Run Auto-Z for the first time. **Keep an eye on the stage movement and prepare to stop Auto-Z by clicking Auto-Z again when it might hit analyzer lens. *Hint: the usual analysis position is just a couple of mm away from the lens. If it is less than 2 mm away and the Auto-Z shows no counts, it WILL hit the lens.***
9. Move the stage to desired analysis position and run Auto-Z again.
10. For small area analysis, one could use SXI button to image the sample (X-ray induced SE image) and set the analysis spot at exact locations. For SXI of insulators, set SXI neutralize to Pulse.
11. Define point, area analysis, or line/map acquisition at desired locations.
12. Choosing X-ray settings in X-ray menu (probe size and output power).
13. Define a survey to get the spectrum. Usual wide scan is from 1000eV to 0eV. **Refer to the handbook to make sure that all the peaks are included.**
14. Define narrow scan on each elements if needed and set appropriate pass energy.
15. Extract the sample (see above).

UPS Operation

A. Purge He Line

1. Make sure UPS pump station is running and TMP at 1500 Hz.
2. Make sure that the stage 2 pumping valve (between turbo pump and UV lamp) is closed with 10 mm wrench.
3. Close the valve on the He regulator.
4. Open the black valve near the TMP controller on the pump station slowly. If the TMP shuts off because the valve is opened too fast, turn the TMP back on immediately.
5. Wait until the turbo is back to 1500 Hz and current below 0.1 A.
6. Close the valve on the pump station, open the valve on He regulator (slightly over 0.1 MPa is enough for normal running) and stage 2 pumping valve on the UV lamp.

B. Ignition of UV lamp

1. Make sure UPS pump station is running and TMP at 1500 Hz.
2. Make sure that both the stage 2 pumping valve (between turbo pump and UV lamp) and stage 1 pumping valve (between rough pump and UV lamp) are fully opened.
3. Slowly open the He leaking valve on the lamp until the chamber pressure increased to around $1\text{E-}5$ Pa (do not exceed $5\text{E-}5$ Pa).
4. Set the voltage set point to 1000V and current set point to 100 mA.
5. Press "Ignition" button to start the discharge and the voltage should drop to about 500 V and status become ready.
6. If it did not ignite, slowly open the leaking valve until it ignites. Do not exceed $7\text{E-}5$ Pa in this process. If the source still does not ignite, purge the He line.
7. Look from the back of the lamp, one should see a bright pinkish spot in the center. If diffused light is observed, lower the He pressure by closing the leaking valve slowly.
8. If He II is desired, increase the current to 300 mA and lower the He pressure. The color will become purplish.

C. Obtain UPS Spectra

1. Load the specimen just as XPS mode and do the auto-Z at 45° and find desired location.
2. Carefully tilt the specimen to 90° and do auto-Z again.
3. Switch to UPS mode from FXS mode in "x-ray" page.
4. On the range definition window, click "hardware" and go to "analyzer" page. Select minimum energy step of 0.005 eV.
5. Make sure the neutralizer is set to off and collect the spectrum with a pass energy smaller than 2.95 eV.
6. The detector overload at $\sim 1\text{E}7$ cps. Use smaller pass energy if the count rate is too high.
7. Set the analyzer back to 0.025 eV and FXS mode before removing the sample.

D. Sample Bias

9/6/17

Sample bias is needed only when SE electron is to be collected. Because of the low kinetic energy of SE, a negative bias on sample is required to accelerate the SE to overcome the work function of detector.

When applying bias, charge neutralization should be turned off (turn off “Ion Gun” and “Neutralizer”, also set *Off* in the Neutralize box at the bottom of “Acquisition Setting”). After finished, set the Neutralize box back to *On* in “Acquisition Settings”.

1. Press +VSET(7) or -VSET(4) to dial the voltage desired. Usually, 5-10 V is enough.
2. Switch the bias box to UPS mode.
3. Press ±OUTPUT(ON/OFF) to turn on the voltage.
4. **Never apply bias while doing sample transfer. The intro-arm will create a short and overload the voltage supply.**
5. Turn off the power supply and switch the bias box back to XPS mode when finish.

E. Shutoff UV lamp

The UV light will not interfere with normal XPS operation (besides SXI). Therefore, there is no need to shut the UV lamp off during sample exchange. It can be difficult for the UV lamp to restart in a short time.

1. Slowly turn the current down until the UV light is off.
2. Close the He leaking valve then stage 1 pumping valve.
3. **Do not close stage 2 pumping valve until the chamber pressure is back to the order of E-7 Pa (usually overnight).**

SIMS Operation

1. Do not exceed 1E6 count rate to protect the detector.
2. The primary ion source is controlled by the VersaProbe main software so that prepare a *continuous* sputter depth profile using *0set* x-ray according to the instruction of XPS operation.
3. Extend the analyzer probe all the way in before SIMS operation and retract after finished.
4. Make sure that the UPS power supply is off then switch the target bias box from XPS to SIMS and switch back to XPS when finished.
5. Template for RGA (+SNMS), +SIMS, and –SIMS are stored in respective directories under c:\Templates\. These directories and files is read only so that use “save as” to save the data in your own working space (Y: and Z: is located on SIMS and XPS computer, respectively).
6. If you did not see resulting trace, select the channel you need and press “*new graph view*” or Ctrl-G.
 - I. Full Tune (this is time consuming and normally not required).
 1. Open “00 Full Tune” for the required operation mode.
 2. Set $m/z=28$ (N₂), 15 (CH₃), or 16 (O) and start the job for RGA, positive, or negative SIMS mode, respectively.
 3. Cycle 2 and Cycle 3 should yield similar results. If not, modify the job file manually for most sensible range and start again.
 - II. Quick Survey
 1. Open “01 Survey Bar Scan” file for the required operation mode.
 2. Set appropriate m/z range and start the scan.
 - III. Fine Tune
 1. Open “02 Fine Tune” file for the required operation mode.
 2. Set appropriate m/z that you want to optimize in the “Global”.
 3. Start the scan and look for the plateau or maximum intensity. Record the setting and input to “Global” then start again until consistent result is obtained.
 4. Note that different m/z may require different parameter. These parameters can be input in the *Set* function for each range of m/z .
 - IV. Pattern acquisition
 1. Create a new job or open “03 Spectrum” for the required operation mode.
 2. In the “profile” page, input desired scan range and step size.
 - V. Depth profile
 1. Create a new job or open “04 depth profile” for the required operation mode.
 2. In the “MID” page, input the desired m/z (as many as needed) for profiling.
 3. Because we are using optical gating right now, there might be some oscillation in the intensity. In this case, change the “Dwell Time” in “SEM” box.
 4. To change the color of each trace, use View/Trend view setup or Ctrl-T

SIMS Gating Setting

7. The gating is mainly useful for depth profiling. Following procedure is main for depth profile. Nevertheless, it can be used for pattern acquisition as well.

1. Create a new job or open “05 depth profile (gating)” for the required operation mode.
2. Confirm the raster setting by double click the “Global” mode selection. Use the gating.xls on the desktop to aid the calculation of settings.

For C60 calibration in XPS software (subject to change and can be calculated by inserting current C60 setting in the Excel file).

X size (mm)	0.5	X offset (mm)	0.13
Y Size (mm)	2	Y offset (mm)	0.3

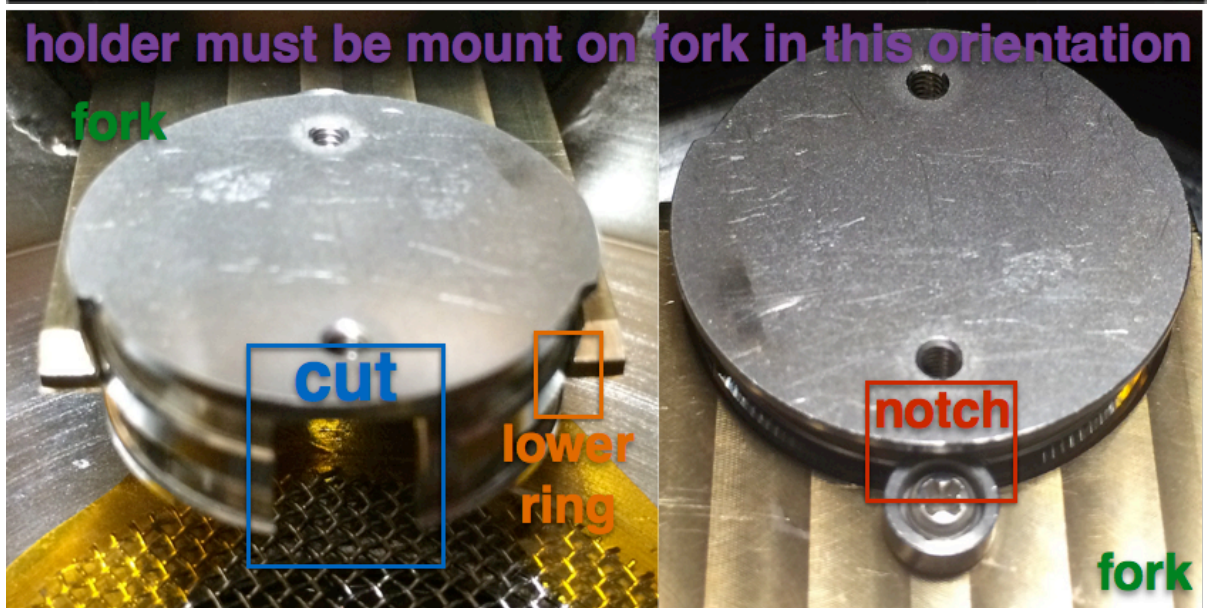
Suggested raster pattern of 50% area is

X1	-48	Y1	520
X2	-19	Y2	403
X3	123	Y3	-163
X4	152	Y4	-280
Dwell time (ms)	1	Step	20

3. Select “Rastered-SEM” as the input device
4. Switch the C60 deflection control from XPS to SIMS
5. In the “MID” page, input the desired m/z (as many as needed) for profiling.
6. After it is done, switch the deflection control back to XPS

Warning

- Do **NOT** touch anything that goes into the chamber with bare hand. Wear a glove and use clean tools.
- Powders are not allowed without permission.
- Samples need be at the same height on the holder.
- **Make sure the fork is at lower groove of holder and correctly oriented with the alignment groove.**
- **Intro pressure should reach 0E0 within 1 minute. If it takes more than 3 minutes, remove and clean the sample before retry.**
- Carefully move the intro-arm and make sure it cannot hit anything in the chamber and holder can slide into the stage smoothly.
- Do **NOT** change any valve, regulator, screw, and software setting (ion gun, neutralizer, X-ray)
- Do **NOT** close the computer program or shutdown.
- Do **NOT** save your data on C drive
 - Save files at Z drive and make your own backups. When the space is low, older files will be **DELETED** without notification.
- If anything should happen to the instrument or if you are not sure what to do, stop using the instrument then write a note describing the nature of the problem and what you were doing on the check list. Contact me at 0987173210.



The black magnet of transfer rod needs to be rotated to this orientation during sample transfer.

