PHI5000 VersaProbe Operation Notes

Standby Condition

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

Introduce Specimen

- Move the intro-rod all the way out then vent the intro chamber by clicking "backfill intro" in *Watcher*.
- Open the intro chamber and mount the specimen holder on the fork. Make sure that the fork is at the lower ring.
- Move the intro-rod 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 1 minutes. If vacuum cannot reach 0E0 within 3 minutes, remove and clean the specimen before retry. If it always takes more than 3 minutes, the sample is not compatible with UHV and cannot be used. Record the time on the log book.
- A 5min 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.
- Once the vacuum is ready and stage moved to its home position, press "transfer sample" in *Watcher*. V1 will open and the intro-rod can be moved into the main chamber. Record the chamber pressure on the log book at this point.
- Carefully move the intro-rod all the way into the main chamber while observing the chamber from the view port. The holder should stop on top of the stage.
- Click "*up*" in the *Summitt* while observing the movement of the stage. The stage will move up to support the holder.
- After the stage movement is finished, move the intro-rod all the way out. V1 should be closed automatically.
- Click "pump intro" in *Watcher* to open V3. This is to ensure that the intro chamber is under UHV.

Extract Specimen

- Make sure that V3 is opened. If it is not opened, click "pump intro" in *Watcher* and wait for 5 minutes.
- In *Summitt*, press "*extract*" to move the stage to the position for removing holder.
- After stage stopped moving, open V1 by clicking "transfer sample" in *Watcher*.

- Slowly insert the intro-rod all the way into the chamber. The fork should grab the lower ring of the specimen holder.
- Click "*down*" in the *Summitt*. The stage will be lowered and release the holder.
- After the stage movement is finished, move the intro-rod all the way out. V1 should be closed automatically.
- Vent the intro chamber and recover the specimen holder.
- Move the intro-rod all the way out and pump the intro chamber.

Operating Ar Ion Gun (for sputtering and neutralization)

- In *Watcher*, press "diff vlv open" to open V4 (differential pumping for Ar gun).
- Switch the thermo-valve control box to "*set point*" and set control program to *standby* mode.
- Check the Ar ion pressure at view->extractor pressure. The pressure should be about 14mPa. If it is too low, open then close the green valve on the ion gun to allow more Ar to get into the ion gun.
- 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".

Start-up of C60 Ion Gun

- Open the isolation valve manually. Set the sputtering mode as "*startup*" and start sputtering. Press control-D to bring up the diagnostic window.
- Wait till the temperature stabilizes at about 400˚C. This will take about an hour.
  Do NOT operate either Ar or C60 ion gun at this period.
- Before put it at "*10kV Typical*" for operation, make sure the pressure is below 1E-4 Pa.
- Following next section to setup the communication between programs and set it back when you are done if automatically sputter depth-profiling is required.
- Close the isolation valve and set sputter mode to "*statup*" before turn it off.

Switching between Ion Guns for Automatic Sputtering

- To use different ion gun for depth-profiling, one needs to setup the DDE command. The setting can be found in view->property of the ion gun control. With selecting different guns in the main screen, different guns can be set.
- To do depth-profile with Ar sputtering, put Ar *standby* and C60 *off*, DDE setting

	Ar	C60
On Command	Sputter	Keep State
Neutralize Command	Neutralize	Keep State
Standby Command	Standby	Keep State

- To do depth-profile with C60 sputtering, put Ar and C60 *standby*, DDE setting

	Ar	C60
On Command	Keep State	Sputter
Neutralize Command	Neutralize	Standby
Standby Command	Standby	Standby

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

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 then 2mm 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, following this section to manually control the neutralizer. Switch it back when it is done.
    - Set SXI neutralize to Pulse.
    - In Neutralizer control (standby mode), view->properties->in emission control menu, change mode from Auto to Manual->manually set filament current to 1.1A.
    - When done with SXI, set the mode back to Auto and SXI neutralize OFF.
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).