

PHI690 Auger NanoProbe Operation Notes

Standby Condition

- Load *ZERO* setting in **SEM** session → **SEM Hardware** tab and **Hardware** session → **Ion Gun Startup** tab.
- **Turn off E-beam** then close the manual isolation valve between upper and main chamber then recover the specimen. Pump down the intro chamber then confirm that V3 is open and the intro is evacuated.
- Set the field emitter to *Standby* state if no one scheduled to use in the same day.
- Confirm manual isolation valve, V4 and V8 **COLSED**,
SED Multiplier (SEM session → SEM Hardware tab) **OFF**,
Beam State (SEM session → SEM Hardware tab or right-click AES databar) **OFF**,
Ion Gun (Hardware session or right-click **Ion Gun** databar) **OFF**,
AES Multiplier (Tools menu → Analyzer and Detector → Channel Plates tab) **OFF**.
Turn off the power extension socket at right-hand side under the table to turn off all the monitors and optical light.
- Record and sign the usage check list. Note problems if any.

Change State of Field Emitter

Confirm *Beam State* is off and the manual isolation valve between upper and main chamber is closed.

A. *Standby* → *Running* state

1. Change user level to *Administrator* in **System** menubar → **Change User Level**.
2. **Confirm the vacuum of upper chamber is $< 2 \times 10^{-7}$ Pa.**
3. In **SEM** session → **Properties** → **Source** tab, slowly increase **Extraction Voltage** to **3.980** kV in 0.1 kV/min and **make sure the upper chamber is $< 2 \times 10^{-7}$ Pa in each step.**
DO NOT CHANGE EXTRACTION MAXIMUM.

B. *Running* → *Standby* state

1. Change user level to *Administrator* in **System** menubar → **Change User Level**.
2. In **SEM** session → **Properties** → **Source** tab, slowly decrease **Extraction Voltage** to 3.000 kV. **DO NOT CHANGE EXTRACTION MAXIMUM.**

C. *Shutdown* Emitter

- **SEM** session → **Properties** → **Source** tab → *FE Shutdown*.

D. *Shutdown* → *Running* state

1. Close *SmartSoft-AES* software then start *FEStartup* software.
2. Set **Filament Current (A)** to **2.315** (1750 K from data sheet) and **Extractor Voltage (kV)** to **3.980** (250 μ A/sr from data sheet or to obtain 10 nA target current at 10 kV).
3. Confirm the **18-19x** is set to *COM2* and **SVC** is set to *COM1* then press *Connect*.
4. Confirm the vacuum of upper chamber is $< 2 \times 10^{-7}$ Pa then press *FE Startup*.

5. When the sequence finished and parameters reached required values, close *FESStartup* software then start *SmartSoft-AES* for normal operation.

Introduce Specimen

Confirm *Beam State* is off and the manual isolation valve between upper and main chamber is closed.

1. Move the intro-arm all the way out then vent the intro chamber by clicking *V2: Backfill Intro* icon in **System** session → **Sample Transfer** page. The V3 icon should turn red (closed) and V2 turn green (opened) automatically.
2. Open the intro chamber and mount the specimen holder on the fork. **Make sure that the fork is at the lower groove.**
The top side of the image at 0° rotation will be at the opposite side of V1 gate.
3. Move the intro-arm all the way out and pump the intro chamber by click *V3: Pump Intro* icon in **System** session → **Sample Transfer** page. 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.**
4. A 3 min count-down will be started once minimum reading (0E0) is reached. This is a safety measure and cannot be by-passed. **During this count-down, the mouse cursor on the message window will become a circle (Windows way of telling the software is busy) then turn back to standard arrow after it is ready.** Although not necessary, it is possible to switch to *Watcher* software behind to see the count-down but do not operate *Watcher* directly.
5. **Take Intro Photo** by right-click the stage icon in *Intro* area of **System** session or press *Photo* in **System** session → **Camera** page.
 - If the stage icon is not in *Intro* area, confirm there is no sample on the stage (main chamber) then set **Sample Location** to *Intro* in its **Properties** page (right-click the icon in **System** session) or in **AES** session → **Data Manager** page.
6. After pumping sequence is finished (mouse cursor returned to standard arrow), press *Transfer Sample (Intro)* icon in **System** session → **Sample Transfer** page to move the stage to the position for accepting sample holder. V3 would close and V1 would open automatically. **Record the chamber pressure on the log book at this point.**
7. **Before following the on-screen instruction and insert intro-arm, double check the stage position as follows to ensure the stage is ready to accept sample holder. The fork should align with the smaller sample retaining piece.**
X= 0.296 mm, Y= 0.981 mm, Z= 0.000 mm, R= 210°, T=0°
8. Follow the on-screen instruction to carefully insert intro-arm into the main chamber while observing the chamber from the view port. The holder should stop on top of the stage.
9. Click *OK* in the on-screen instruction while observing the movement of the stage. The stage will move up to support the holder (Z= 8.000 mm).

10. After the stage movement is finished, carefully move the intro-arm all the way out. V1 should close and V3 should open automatically.

Specimen Parking

Only 25 mm holders can be parked. DO NOT attempt to park 60 mm holders.

While manual isolation valve between upper and main chamber must be closed before open V1 or backfill intro during sample exchange, it is possible to introduce multiple samples continuously into the main chamber while the manual valve is already closed. Exchanging samples between stage and parking within the main chamber can avoid the frequent need of opening and closing the manual valve.

A. Park (move holder from stage to empty parking space for storage)

1. Untighten the locking knob and slightly extend the parking device then rotate it to an empty fork and finger-tight the locking knob. **Make sure samples on the device do not hit anything inside and the rotation stop at notch so the fork is horizontal.**

2. **Confirm sample tilt is at 0°.**

Press *Park* in **System** session → **Sample Transfer** page → Properties. **Double check the stage position as follows to ensure the stage is ready for parking.**

X= 20.000 mm, Y= 9.600 mm, Z= 7.000 mm, R= 245°, T=1°

3. Follow the on-screen instruction to extend the parking device to grab the sample holder at upper groove and finger-tight the locking knob.
4. Click *OK* in the on-screen instruction while observing the movement of the stage. The stage will move down to release the holder (Z= 0.000 mm).
5. Untighten the locking knob then retract the parking device then finger-tight the locking knob.
6. Set **Sample Location** to *None* or *Intro* in its **Properties** page (right-click the icon in **System** session) or in **AES** session → **Data Manager** page.

B. Unpark (move holder from parking space to empty stage for analysis)

1. **Make sure there is no holder on the stage and sample tilt is at 0°.**

2. Untighten the locking knob and slightly extend the parking device then rotate it to move the desired sample holder to the top then finger-tight the locking knob. **Make sure the samples on the device do not hit anything inside and the rotation stop at notch so the holder is horizontal.**

3. Press *Unpark* in **System** session → **Sample Transfer** page → Properties. **Double check the stage position as follows to ensure the stage is ready for unparking.**

X= 0.296 mm, Y= 0.981 mm, Z= 0.000 mm, R= 245°, T=1°

4. Follow the on-screen instruction to extend the parking device. The holder should stop on top of the stage. Finger-tight the locking knob.
5. Click *OK* in the on-screen instruction while observing the movement of the stage. The stage will move up to support the holder (Z= 7.000 mm).

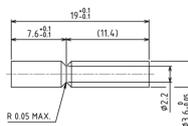
6. Untighten the locking knob then retract the parking device then finger-tight the locking

knob.

7. Set **Sample Location** to *Stage* in its **Properties** page (right-click the icon in **System** session) or in **AES** session → **Data Manager** page and load appropriate .pho file in *Image Overlap* to aid sample navigation.
 - After unpark, the stage will not rotate back to 0°. Although the sample photo is aware of this rotation, the XY of SEM image will differ from the photo because photo will not rotate.
 - With parking, the sample rotation may slightly changed with respect to the stored image so the actual position can be slightly different from that shown in photo.

***In Situ* Cyro-Fracture**

The shape of the sample should be made as following and can be fixed on the special Cu holder by the locking nut.



1. Turn the fracture knob all the way out and **make sure the upper and lower parts of the Cu block is separated**. **Because the Cu blocks are hold by two strong springs, it may require a lot force to turn the knob to separate them.**
2. After introduce the sample, transfer it to the parking device and rotate it to move the sample to the bottom position. **Make sure the samples on the device do not hit anything inside and the rotation stop at notch so the holder is horizontal.**
3. Retract the parking device so the sample holder **completely** inserts into the Cu block. Finger-tight the locking knobs.
4. Release the fracture knob slightly so it can rotate freely and the spring will close the Cu block firmly against the sample holder and provide good thermal contact.
5. Fill liquid nitrogen then wait for ~0.5 h to ensure the sample is cooled.
6. Turn the fracture knob all the way in. The head of the knob should remain extended and separated from the knob.
7. Pull the head of the fracture knob all the way out then quickly release it. The spring inside should provide enough force to fracture the sample and allow the head to be inserted completely. **In case the head still fail to insert completely (sample did not break) after a few attempts, carefully use hammer to force the mechanism to break the sample if necessary.**
8. Turn the fracture knob all the way out and **make sure the Cu block is separated** so that sample is released.
9. Untighten locking knobs then extend the parking device and rotate it for unparking the sample back to stage.

Extract Specimen

Confirm *Beam State* is off and the manual isolation valve between upper and main chamber is closed.

1. Make sure V3 is opened for more than 3 minutes. If it is not opened, click *V3: Pump Intro* icon in **System** session → **Sample Transfer** page and wait for 3 minutes.
2. Click *Transfer Sample (Extract)* icon in **System** session → **Sample Transfer** page to move the stage to the position for releasing sample holder. V3 would close and V1 would open automatically.

Before following the on-screen instruction and insert intro-arm, double check the stage position as follows to ensure the stage is ready to release sample holder. **The fork should align with the smaller sample retaining piece.**

X= 0.296 mm, Y= 0.981 mm, Z= 8.000 mm, R= 210°, T=0°

3. Follow the on-screen instruction to carefully insert intro-arm into the main chamber while observing the chamber from the view port. The fork should grab the lower groove of the sample holder.
4. Click *OK* in the on-screen instruction while observing the movement of the stage. The stage will move down to release the holder (Z= 0.000 mm).
5. After the stage movement is finished, carefully move the intro-arm all the way out. V1 should close and V3 should open automatically.
6. Backfill the intro chamber and recover the sample holder.
7. Move the intro-arm all the way out and pump the intro chamber.

Operating Electron Gun (SEM mode)

In most case, field emission gun is kept active and the lifetime is wasted even the system is not in use. To preserve its lifetime, the emitter is set to standby or turned off when the system is not used. When it is switched to running condition, it may require some time to stabilize.

1. Verify the state of the field emitter by reading the *EXT VOLTAGE* from the LCD panel of the controller. If it is 3.0 kV, it is in *Standby* mode and change it to *Running* as required.
2. Verify the *Main Chamber* pressure to be below 3×10^{-6} Pa and *Beam State* is off before open the manual isolation valve between upper and main chamber.
3. Load *Z Align* in **SEM Settings** in **SEM** session → **SEM** page → **SEM Hardware** tab then set *Beam State* to on and *SED Multiplier* to auto.
4. Set to lowest *Magnification* (depend on beam voltage) and adjust *SED Multiplier*, *Contrast* and *Brightness* (or use *AutoVideo*) to obtain initial image. Roughly adjust *Focus* if required.
 - The tuning knob can be pressed down then rotate to preform coarse adjustment.
5. In **Sample** session → **Stage** page, click *Joystick* for sample navigation and move the XYR position to roughly locate the region of interest.
 - XY can also be moved using the toolbar to the left of image or click on the image with grab mode.

6. In **Sample** session → **Z Align** page, click *Start Z Align* to acquire elastic peak at 1 kV. While looking through the viewport and ensure the sample will not hit anything inside, carefully move the Z position to maximize the intensity and center the elastic peak at the calibrated green marker (~1 kV). The working distance is 8.61 mm.
 - The Z align should be performed or checked when sample is significantly moved or before spectrum acquisition. Unlike dedicated SEM where the system tracks the working distance of objective lens and corrects magnification factor, the scale is correct only at correct Z position on this system. The Z Align function will also reset the beam voltage to 0 then ramp up to 1 kV so it is also useful when lost communication.
7. Load desired **SEM Settings** in **SEM** session → **SEM** page → **SEM Hardware** tab
8. Click *Read Beam I* in **SEM** session (switch box set to *COMPUTER/BIASED*) to measure the beam current. Adjust **Gun Lens** (condenser lens) for desired current. Re-align the **Extractor Steering** to put the beam on optical axis by maximize the current after changing **Gun Lens**.
 - The reference setting is the beam current measured on Cu at 0° tilt with 90 V bias.
9. At desired region and magnification, fine tune *Focus*, *Stigmation*, *SED Multiplier*, *Brightness* and *Contrast* to optimize the image.
 - For optimal image resolution, *Focus Steering* should also be tuned to put the beam on optical axis by rocking objective lens in **SEM** session → **Properties** → **Source** tab if rocking was not started automatically when click in *Focus Steering* box.
- It is possible to rotate the image by rotating the raster direction in **SEM** session → **Properties** → **Image** tab instead of really rotate the sample.
- If sample tilt is used, the aspect ratio of resulting image will change. It is possible to compensate the tilt in **SEM** session → **Properties** → **Image** tab. To avoid collision, the analysis region and highest point should be arranged (with stage rotation) at an edge that is as far away from ion gun as possible.

Operating Ar Ion Gun (for profile and neutralization)

1. Verify the *SED Multiplier* is set to auto in **SEM** session → **SEM** page → **SEM Hardware** tab so SED will be off automatically during sputtering.
2. In **Hardware** session, load desired setting and set to *Blank* mode. Verify *Diff Pump (V4)* and *Argon (V8)* is opened.
3. Check the Ar ion pressure by clicking *Extractor Pressure* in **Hardware** session → **Ion Gun Startup** tab. The pressure should be about 10-15 mPa at *Blank* mode (~25 mPa at *Standby* mode).
4. Measure the target current of ion beam by switching the switch box to *DIRECT/UNBIASED* mode, set the second switch to *Target* (for beam current) or *Faraday Cup* (for beam position if Faraday cup is already correctly positioned) and read the value from picometer directly if necessary. **DO NOT use the Read Beam I function in**

the software because it will apply 90 V bias on the sample.

- Before removing sample, click *V3: Pump Intro* icon in **System** session → **Sample Transfer** page. Wait for at least 5 minutes before click *Transfer Sample (Extract)* icon in **System** session → **Sample Transfer** page.
- The default raster size of Ar is 2 mm at 30° tilt (effectively 45° incident where depth profiles normally preformed). While maximum raster size of E-beam can be ~5 mm, make sure the raster size of E-beam is smaller than Ar when doing depth profile and is centered correctly.
 - To ensure the beams overlapped at desired sample tilt, it is possible to image the sputter crater in SEM mode and move E-beam to center the feature.
 - It is possible to obtain ion beam image in **Hardware** session → **Ion Gun** page → **Properties** → **Raster** tab with ~100 μm resolution on large features.

Suggested Acquisition Parameters

- In general, smaller retard ratio will yield better energy resolution. However, the signal intensity will be weaker and need longer time to achieve enough signal-to-noise ratio.
 - For full survey scan and standard multiplex scan (elemental), fixed retard ratio of 0.5%; energy step: 1-0.5 eV. For multiples with better energy resolution, energy step 0.5-0.2 eV.
 - For high-energy resolution multiplex scan (HERO mode, special holder required and *Sample Bias enabled*), retard ratio: 0.25-0.1%; energy step: 0.25-0.1eV
Depends on the kinetic energy, the minimal retard ratio is different.
- 10 kV is most generally used and **quantification should only be done with 3, 5 or 10 kV**. Higher energy yields better spatial resolution and lower energy suppress sample charge up. Higher current yields stronger signal at expense of spatial resolution.

Voltage (kV)	3kV1nA	5kV1nA	10kV1nA	10kV10nA	20kV1nA
Beam size (nm)	50	30	17	22	11

- For insulating sample, it is very difficult to compensate for the charge-up.
 - In most case, lower beam energy leads to less charge-up. However, if the substrate is conductive and insulating layer is thin, higher beam energy allows the penetration of insulating layer and alleviate charge-up.
 - Tilt the stage to 30° can be helpful. If necessary, the use of 30° pre-tilt holder allows a total tilt of 60° and the lower incident angle would allow higher yield of SE and BSE to alleviate charge-up. **At high tilt angle, the analysis region and highest point should be arranged at an edge that is as far away from ion gun as possible to avoid collision.**
 - In some case, load appropriate low energy ion setting and enable it in **Hardware** session → **Ion Gun** page → *Neutralize Setting* can help. The actual ion energy can be easier optimized by tuning *Float* in **Ion Gun Hardware** tab to move know elemental peak to its theoretical position in *Test Acquire*.

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). The top side of the image at 0° rotation will be at the opposite side of V1 gate.
4. Use AES session → Data Manager page → Directory tab to navigate to your own folder in Z drive. Filename and comment can be set in the Lab Book tab in Properties of Data Manager. **Never save files in C drive.**
5. Use the SEM mode to find desired area and perform Z Align (see above).
6. Optimize SEM image with desired beam voltage.
7. Define point or area analysis in AES session → Survey/Profile page → Analysis Areas tab at desired locations. For Line or Map mode, define the analysis line or area in their respective pages.
8. Get full spectrum in AES session → Survey page → Survey tab. Refer to the handbook to make sure that all the peaks are included. The maximum scan range is 0 to 3200 eV but <20 eV and >(beam energy-500) eV regions are usually not collected.
9. Define narrow scan on each elements in AES session → Survey page → Multiplex tab if needed. Right click Element field to select desired element and related peak. Set desired retard ratio (HERO), Sweeps and eV Per Step for each element.
10. For Profile, Line and Map mode, in addition to the sputter setting and resolution setting, Acquisition Method can be changed to 1-, 2- or 3-Point to save acquisition time at the expense of no detailed spectrum as in standard Window mode.
 - To setup the 1-, 2- or 3-Point correctly, Test Acquire should be used to ensure the Background, Peak and Channels are set according to the spectrum feature.
 - If the signal-to-noise ratio is too low and peak cannot be identified, multiplex scan should be performed first with sufficient SN to set position correctly. For weak peak on asymmetrical background, 3-Point method should be used to interpolate the background under peak.
11. For long time acquisition, Image Registration can be setup in AES session → Image Registration page and enabled in each mode to compensate sample drift with beam shift.
 - Registration Area 1 is used for coarse compensation at a lower magnification and ensure the feature does not drift outside of the view.
 - Registration Area 2 is useful in higher magnification to ensure the drift is minimized.
12. Extract the sample (see above) and put the system to standby mode described above.

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.
- Do **NOT** operate the main isolation valve when the *Beam State* is not off.
- Make sure the main isolation valve between upper and main chamber is manually closed before open V1 or backfill intro.
- Make sure the fork is at lower groove of holder.
- 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** close the computer program or shutdown.
- Do **NOT** save/overwrite referencing settings. Add your name in front the setting name before save your own settings.
- Do **NOT** save your data on C drive.
 - Save files at Z drive and make your own backups. All the files can be removed without prior notice so make your own copy.
- 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.