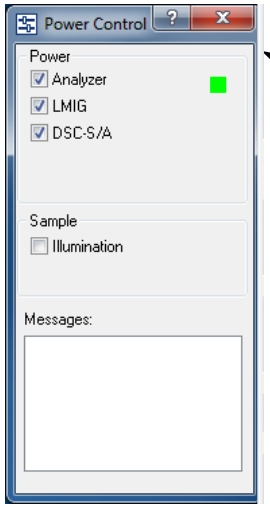
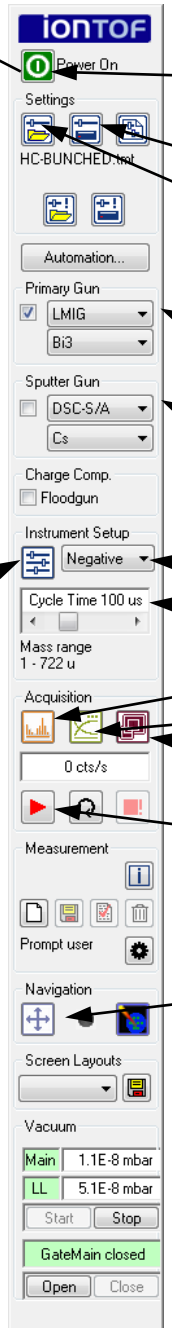
TOF SIMS Procedure

TOF SIMS Lab

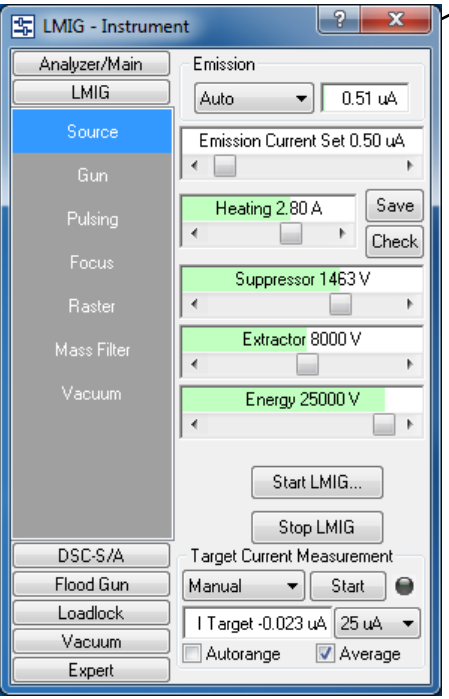
AIF NCSU

ION TOF panel orientation





Instrument window



1. Click the green icon and show the power control window
Power for Analyzer, LMIG and 2nd Gun should be checked.
Close Power control window

2. Button to save setting

3. Button to load setting files for LMIG, Analyzer, sputtering gun (Cs or C60) or flood gun

4. Primary ion Gun: LMIG, Cs or C60 (normally LMIG)

5. Sputter Gun Cs or C60 species
If cycle time < 100us, the square check box before sputter gun will not show.

6. Change positive or negative ion mode

7. Cycle time for one pulse (at one pixel) of data collection.

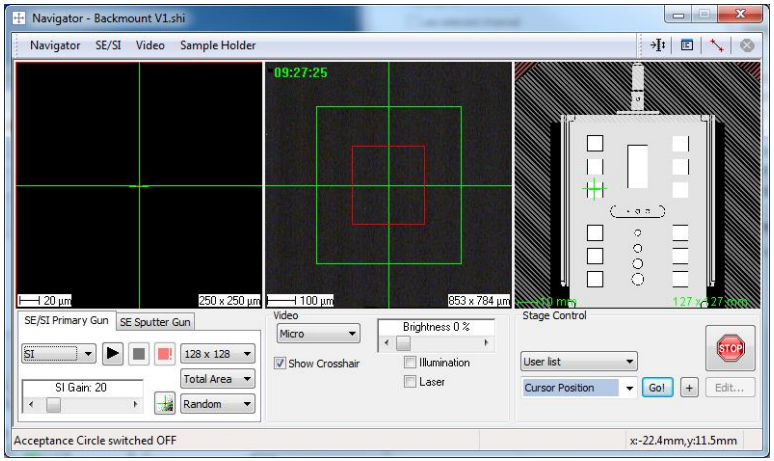
Spectrum window

Depth profile window

Imaging window

Start data acquisition

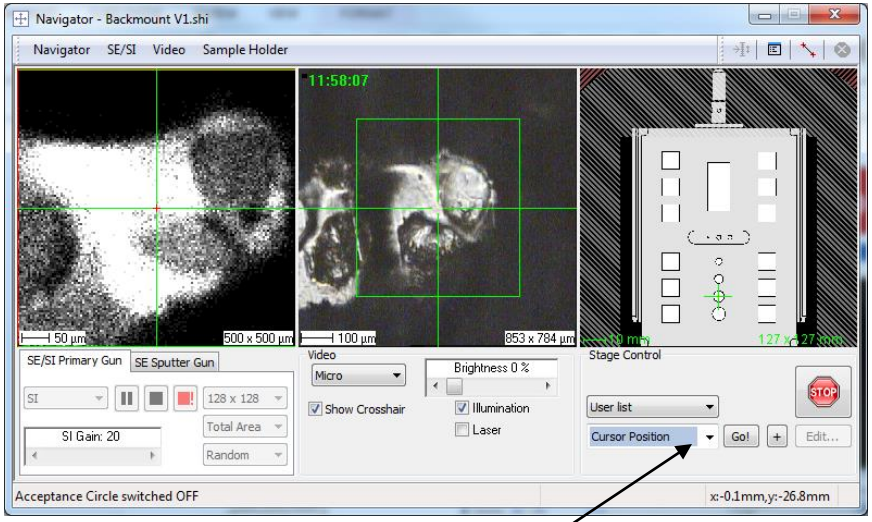
Navigation window

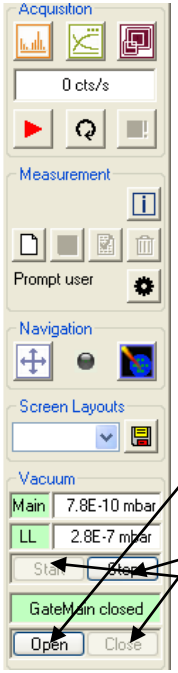


Daily procedure:

1. Record pressure
2. Start LMIG source
3. Data acquisition
4. Shut down
5. Data interpretation

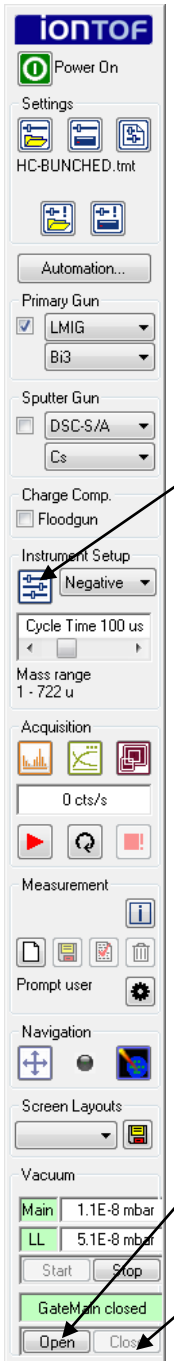
Loading Samples into Loadlock



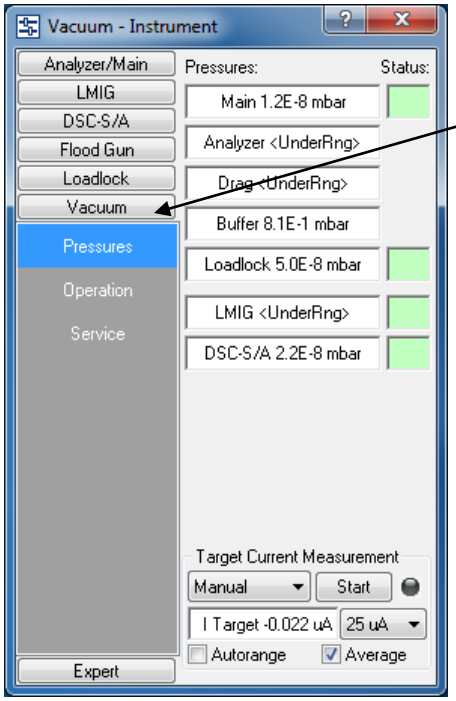


1. If sample holder is in the main chamber, click "exchange position" from the drop down menu in Navigator window and press Go button.
2. Click Open button to open the gate between main chamber and loadlock.
3. Move transfer rod to the main chamber, turn the rod and lock the sample holder and move transfer rod back to loadlock.
4. "Close" button is black when the gate is open, which allows you to close the gate. Click close to close the gate.
5. A stage control window pops up on top of Navigation window, Click OK.
6. Click stop to vent the loadlock chamber. It will take about 2 min.
7. Take the holder out and mount samples. Make sure there is no loose powder. Check that screws on the back of the holder are all tightened.
8. Insert the sample holder in the transfer rod and close the loadlock door.
9. Click Start to pump down the loadlock chamber.

Record Pressure and Load Samples for Analysis



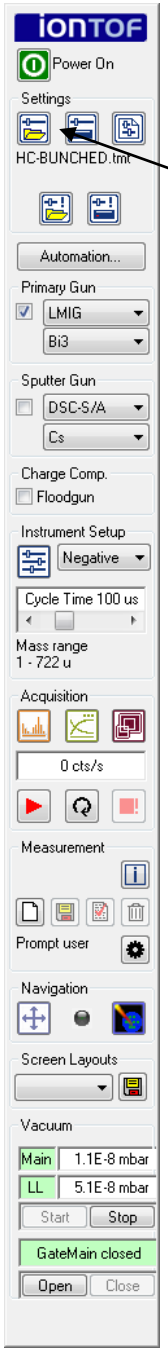
1. To show vacuum, click Instrument panel. You will see window shown in the right.



2. Click on Vacuum on the left and record pressures. The values displayed are pressures in normal range.

3. Click open to open the gate between Loadlock and Main chamber.
4. Transfer sample holder to the holder stage in main chamber.
5. Loose transfer rod from the sample holder and move transfer rod back to loadlock.
6. Click close to close the gate.
7. Select sample holder window pops up on top of Navigation window, Click OK if this is correct holder

Load Sputtering Gun

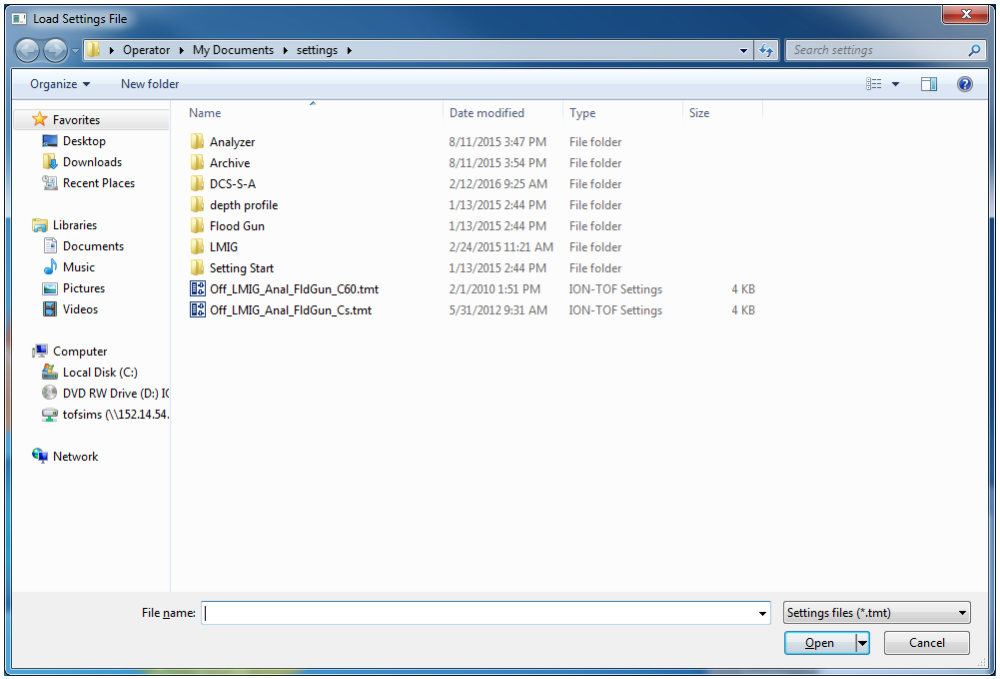


1. This is only needed if acquiring depth profile. Do this step before starting LMIG.

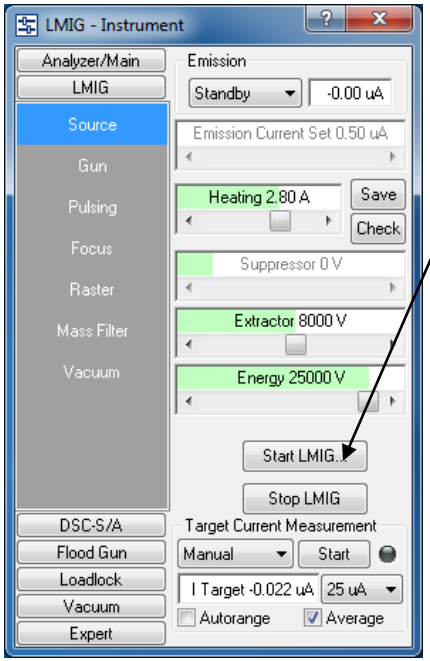
2. Select Load setting

Select DSC-S-A folder: Cs-1Kev.tmt, Cs-3Kev.tmt , Cs-10Kev.tmt

3. Wait 1 hr for Cs source to stabilize.



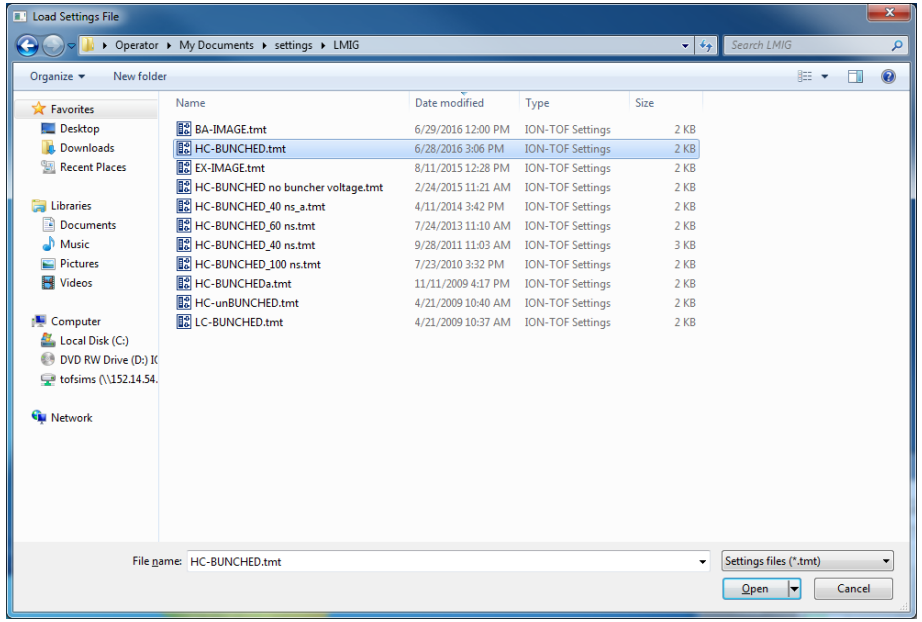
Start the LMIG Bi Source

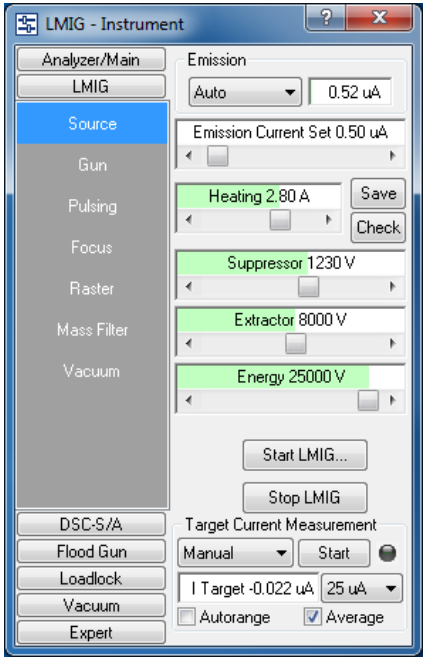


1. Click Start LMIG (Liquid Metal Ion Gun)

2. Then window shown on the right pops up. Select appropriate file and click Open to start the LMIG source:
HC-bunched (high mass resolution);
BA-image (high spatial resolution imaging).

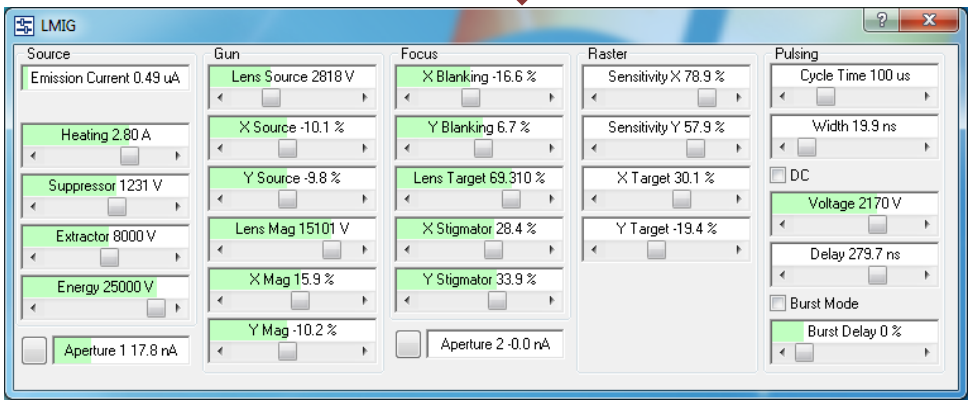
3. The start LMIG recipe will fire the LMIG. It takes about 5 min.



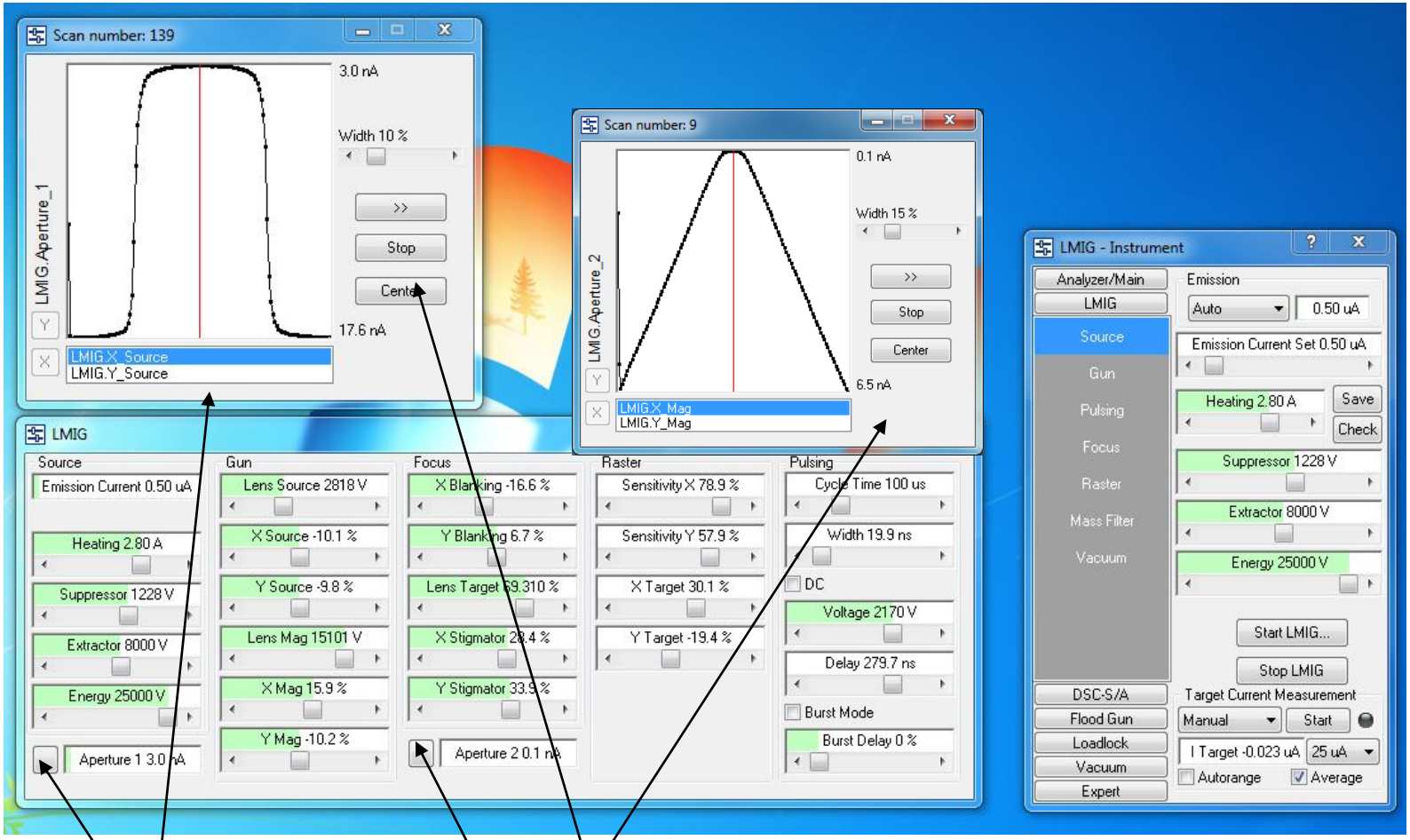


4. LMIG window after turning on LMIG Bi source.

5. On Instrument window, click LMIG + SHIFT together to expand LMIG parameter window



Bi Beam Alignment for HC_bunching



Emission current set to 0.5 µA when it is selected as Auto: Suppressor voltage will automatically control emission to the set value.

1. Emission current should be around 0.5 µA but can be up to 1µA. The current is set by emission current set (select auto).

2. Click Aperture 1, center the beam by clicking center for both X source and Y source (shape should be plateau + sharp slope). Close the scan window after centering. Same for Aperture 2 (trapezoid).

3. Apt1 should have current > 18 nA ideally at 0.5 µA current. The worst case should be > 16 nA. If current in Apt 1 is below <16 nA, see note in next slide or look for Elaine

4. Record the current in Apertures 1 and 2. Need aperture 2 window open to read aperture 2.

5. Note: values of heating, suppressor, extractor, lens source displayed in the manual may be different from current setting, which is fine.

If current in Apt 1 is low <16 nA, try following steps:

1. Uncheck emission control in LMIG-Instrument window

2. Drag Suppressor bar to maximum voltage 2000V. Normally you will see the emission current increase to its maximal current 12.5 µA. The current in Apt 1 is 62.5 nA. If you do not see the current goes to its maximum, increase Extractor little by little until the maximal current is reached.

3. Decrease Suppressor voltage until the current is around 1 µA.

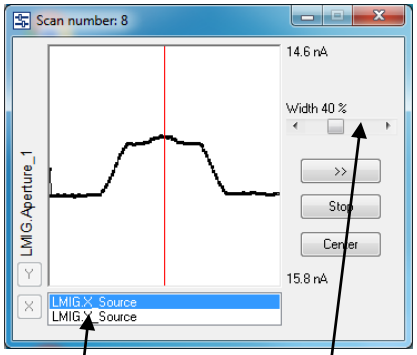
4. Check the Apt 1, Apt 2 current and center the beam.

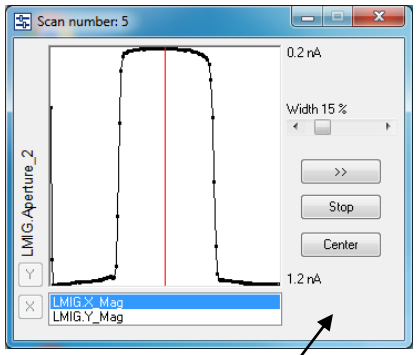
5. If you change the Extractor voltage, you also need to change the lens source voltage. The rule is: the higher the extractor, the higher the lens source.

6. Adjust lens source with Apt 1 scan window open. Adjust lens source until a plateau + sharp slope is obtained.

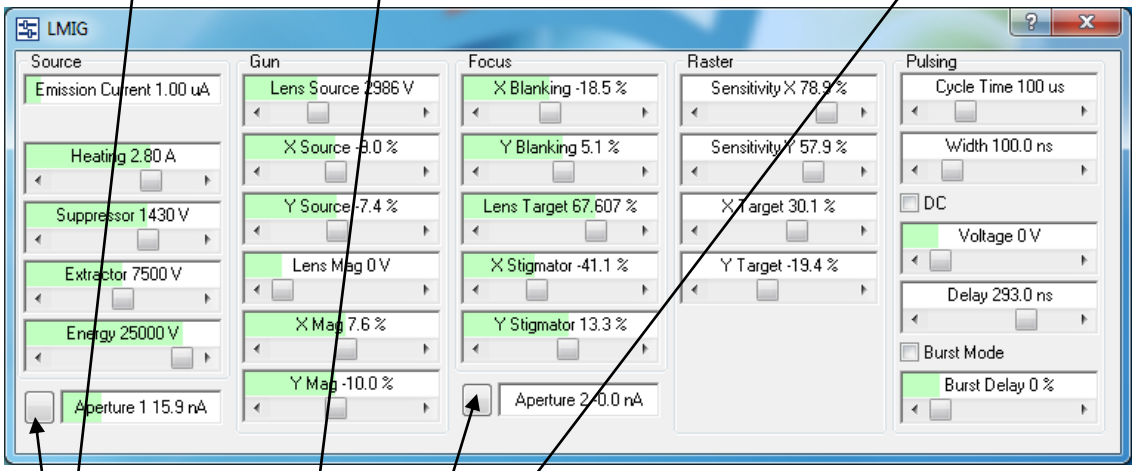
7. If this still does not solve the problem, please contact Elaine for further assistance.

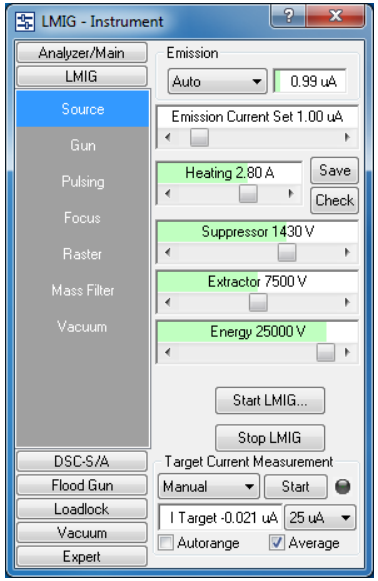
Bi Beam Alignment for BA_image





This is only needed when acquiring high spatial resolution images





1. Emission current should be around 1.0 µA. The current is set by emission control (check box is checked).

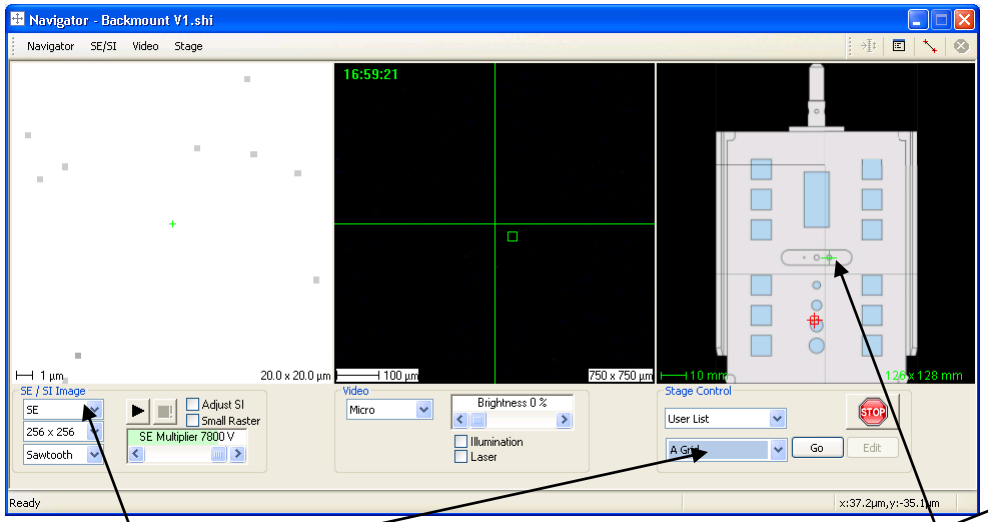
2. Click Apt 1, increase width to 40%, center the beam by clicking center for both X source and Y source (plateau + slope). Close the scan window after centering. Same to Apt 2 (plateau + sharp slope).

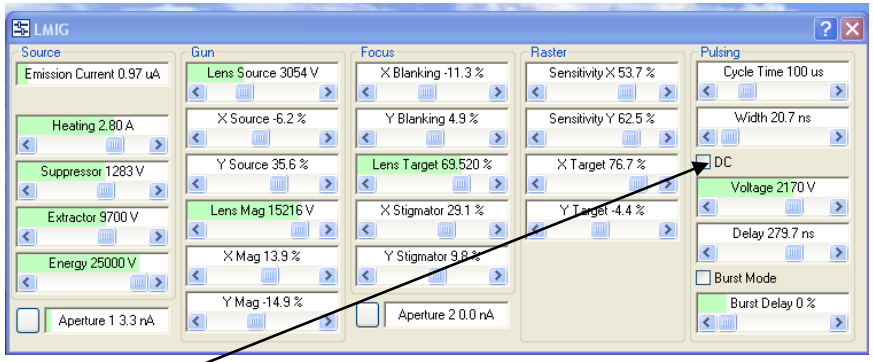
3. Apt1 should have current > 17 nA ideally at 1.0 µA current. The worst case should be > 15 nA. If current in Apt 1 is below <16 nA, see note in next slide or look for Elaine)

4. Record the current in Apt 1 and 2.

5. Note the value of heating, suppressor, extractor, lens source displayed in the manual is different from current setting, which is fine.

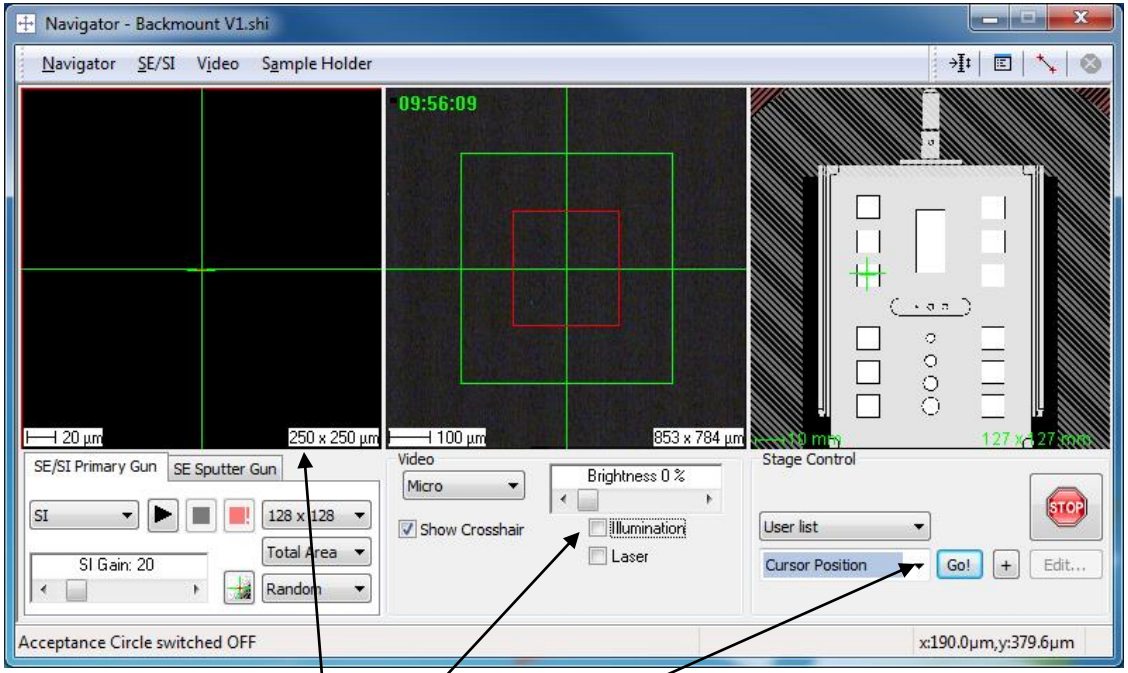
Bi Beam Focusing for BA_image

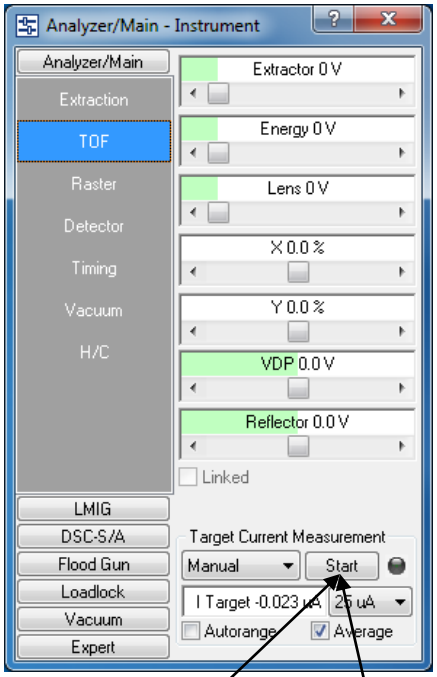




1. Find "A Grid" from the drop down list in stage control window and click go.
2. If holder contains samples higher than the surface, move Z handle UP a little bit, watch through the main chamber window to see the distance between holder and extraction cone. Click A grid position on stage control and then hit go. After the stage moves to "A grid", repeat step 1 (this will move to the right Z height).
3. Right click mouse, change field of view to 50 µm x 50 µm.
4. Check the box before DC in LIMG window.
5. In SE/SI image window, change SI to SE, pixel size to 256 x 256, scanning type to Sawtooth. Increase SE multiplier to 8000V.
6. Click black arrow to start SE image acquisition, tune Lens Target, X and Y stigmator for best image resolution.
7. Change filed of view to smaller area and repeat step 6.
8. Click square button (the same position as the black arrow). Uncheck DC, reduce SE multipler to 0V, change SE to SI.

Measure Current





1. Move the stage to Faraday cup: select faraday cup from the drop down menu and click go. (**Make sure no "protruding objects" on the holder otherwise the extraction cone will be destroyed)**
IMPORTANT: DO NOT MOVING THE HANDLE DOWN TOO FAST (Z direction) OR THE EXTRACTION CONE WILL HIT THE STAGE AND BE DESTROYED.

2. Turn off illumination. Illumination check box should be unchecked.

3. Make sure polarity in instrument panel is in positive.

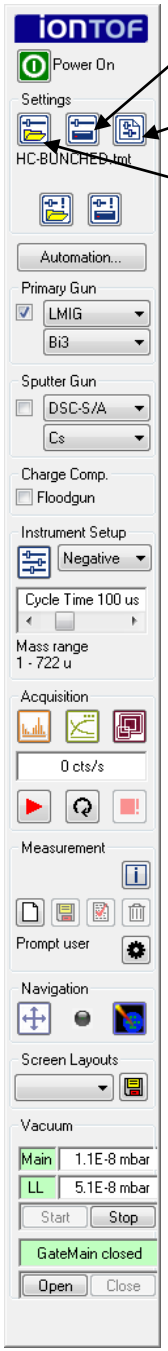
4. Right click mouse on SE/SI image window. Select specify raster field of view and change to 20 µm x 20 µm.

5. Autorange and Average boxes are checked.

6. The box in front of LMIG is checked if measure Bi current. Thebox in front of Cs should not be checked.

7. Click start from the instrument window. Right down the Target current and then click stop. (Start button changes to Stop when measuing the current.)

Load Setting files



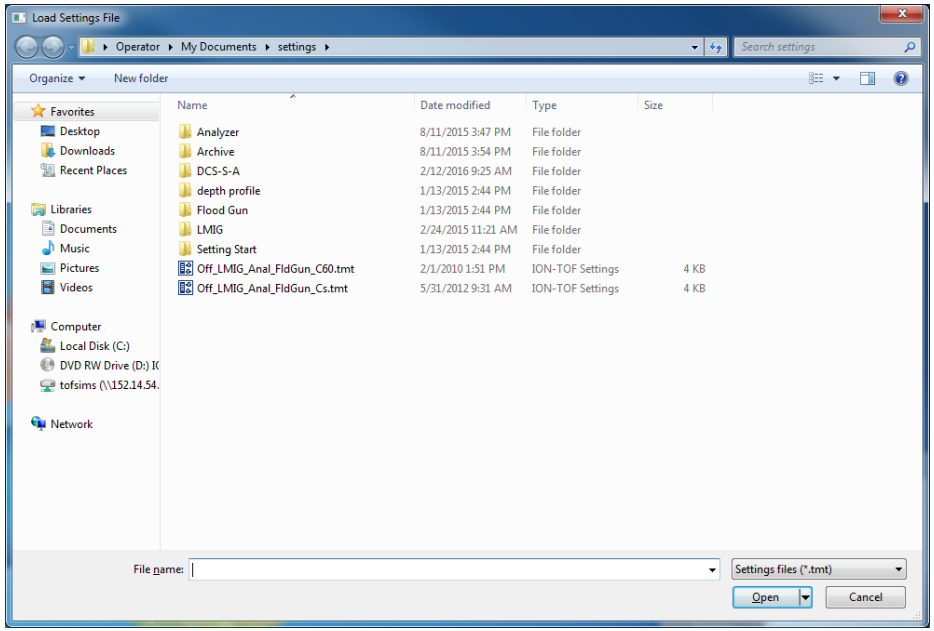
Save setting

View setting

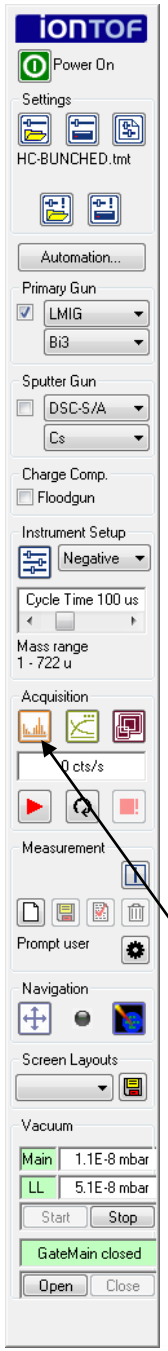
1. Select Load setting
2. Select appropriate folder for the respective species and open file

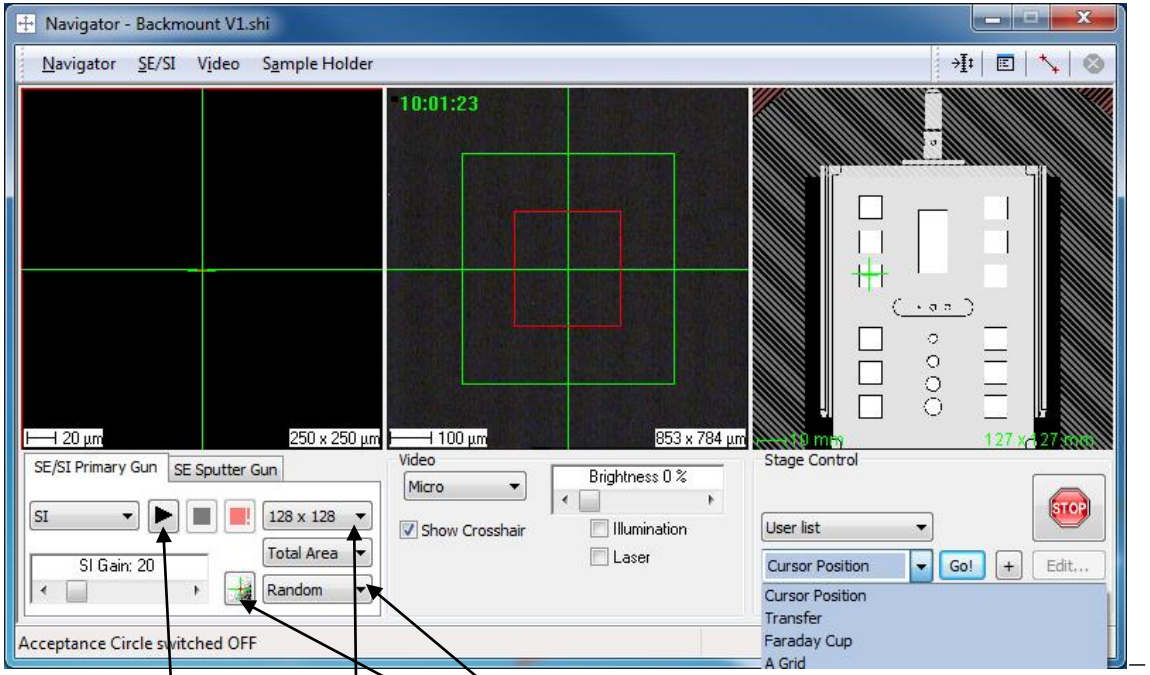
LMIG folder: BA_image.tmt (for High resolution image)
HC_bunched.tmt (for High mass resolution spectrum)
DSC-S-A folder: Cs-10Kev.tmt
Analyzer folder: Analyzer for HC.tmt
Flood Gun folder: Flood Gun.tmt

3. Flood gun is needed only if the sample is not conductive.
4. DSC-S-A is needed only if you want to acquire depth profile. Wait 1 hr for Cs source



Check Mass Resolution





Move the stage to Si to test beam performance and mass resolution.

1. Find Si from the drop down menu and click go.

2. Right click mouse on SE/SI window and change Field of View to 500 um x 500 um.

3. Make sure LMIG in Fpanel is checked and sputtering gun is unchecked.

4. Change pixel size to 128 x128 and raster type to Random.

5. Start acquisition by clicking the black arrow and check Adjust SI.

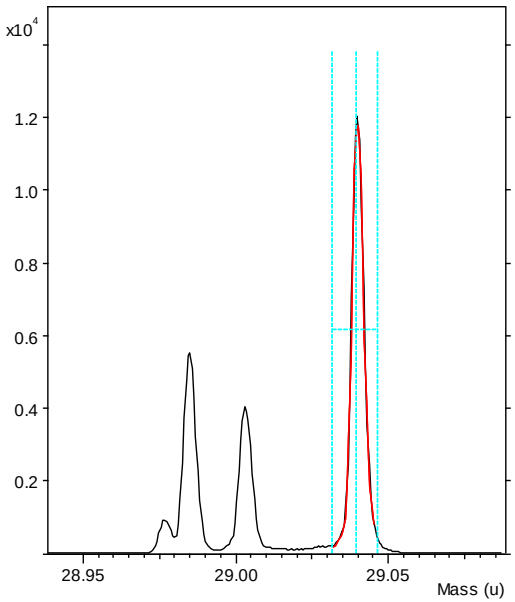
6. Adjust the sample holder height by moving the secondary ion circle to the middle. (Z handle up to move SI circle to the right and vice verse).

7. Uncheck Adjust SI

8. Right click mouse on SE/SI image view to change the field of view to 100 um x 100 um and repeat step **4-7**.

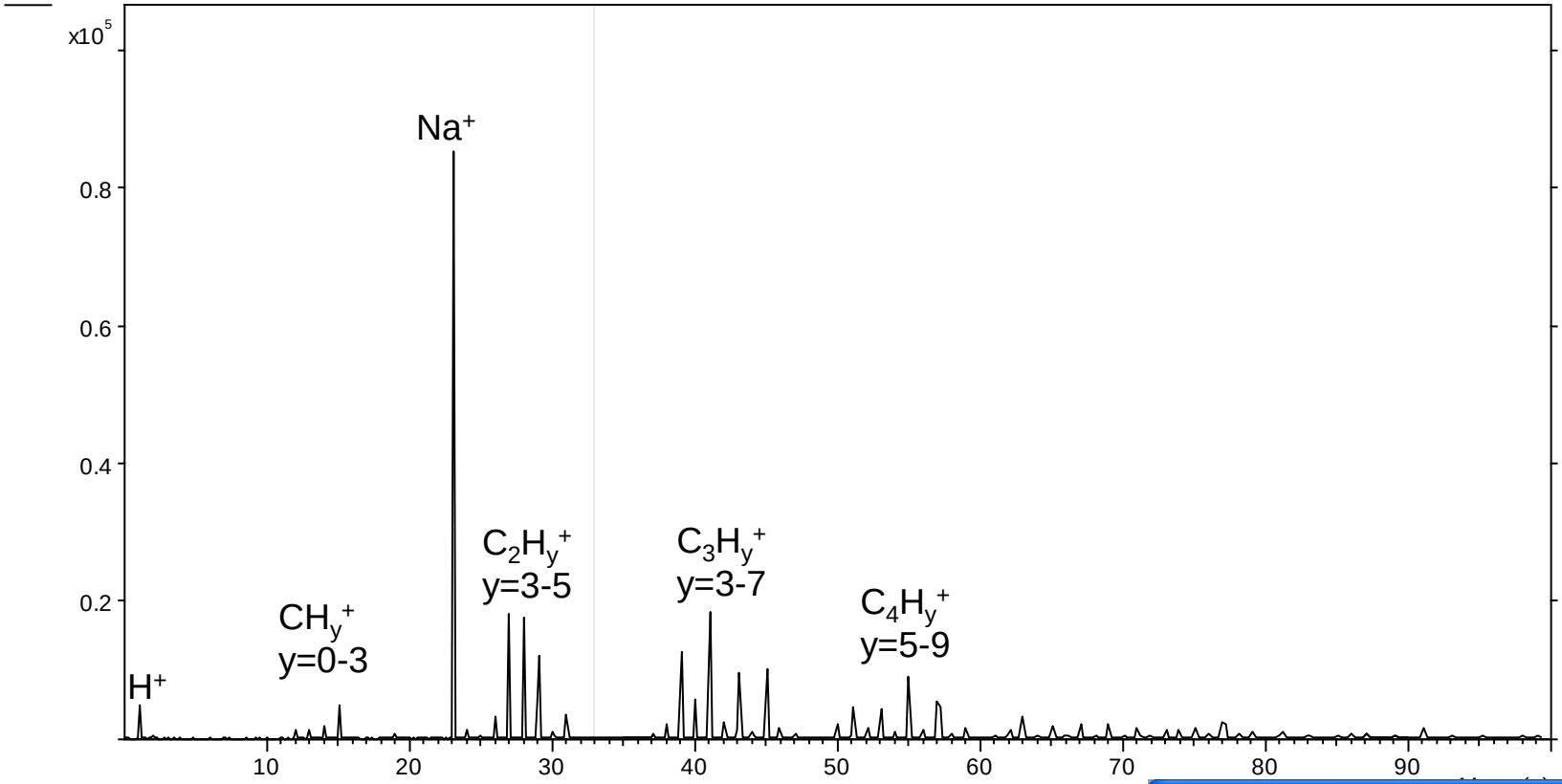
9. Open spectrum window, start acquisition by clicking the black arrow and hit the same button once a spectrum is shown in spectrum window.

10. Look at m/z 29 and check the mass resolution. m/Δm should be around 7000.



Mass Calibration

Positive ion spectrum on Contaminated Si

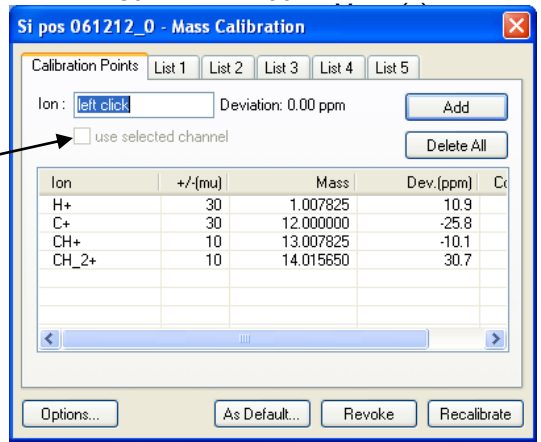


1. F3 to open calibration window.

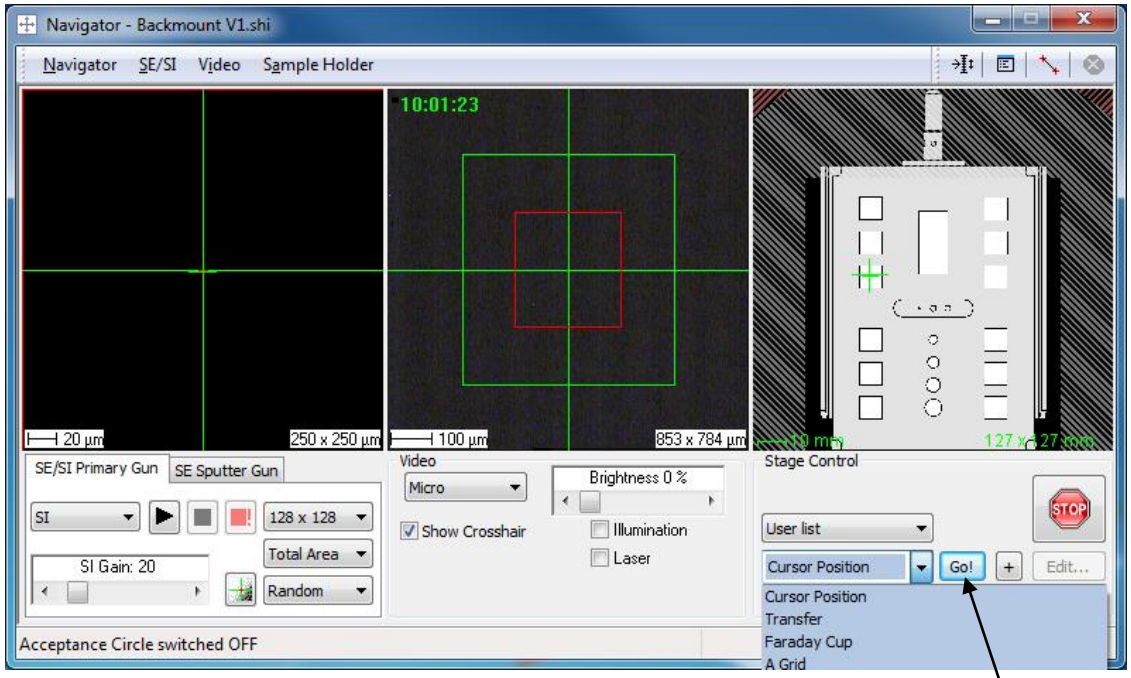
2. Find H, C, CH, CH2, C2H3, C3H5, C5H7 peaks for position ion spectrum calibration and H, C, CH, C2, C2H, C3, C4 for negative ions spectrum calibration.

3. Left click on the peak and then add it to the calibration list.

4. When calibrating spectrum for HR image, check the box before "use selected channel".



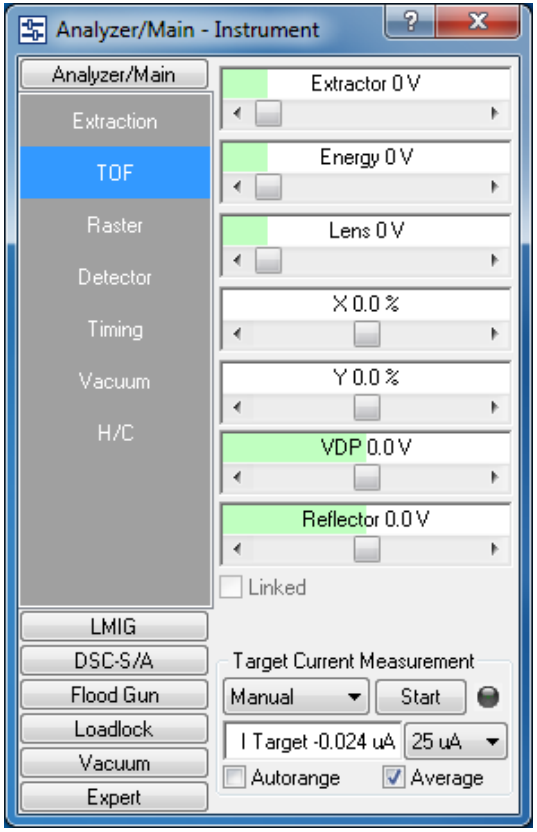
Data Acquisition



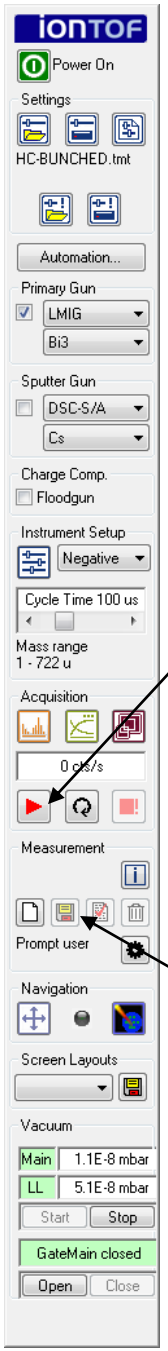
1. Move to the sample to be analyzed by clicking the sample location on the holder in stage control window and then hit go.
IF samples are top mounted, move handle Z UP (increase the distance between holder and TOF extraction cone) and do step 1.
2. Field of view: change to 100 µm x 100 µm or larger.
3. Start acquisition by clicking the black arrow and check Adjust SI.
4. Adjust the sample holder height by moving the secondary ion circle to the center. (Z handle up to move SI circle to the right and vice verse).
5. Uncheck Adjust SI
6. Change Field of view to desired value.
7. Start acquisition by clicking the black arrow and hit the same button once a spectrum is shown in spectrum window.
8. Calibration the mass spectrum.

Data Acquisition for Insulating Samples

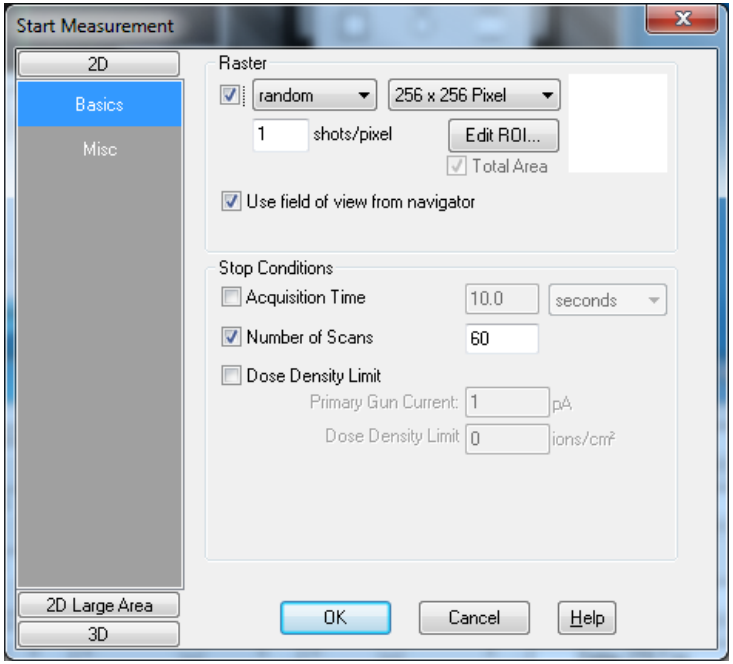
1. Select load setting in Fpanel and load flood gun setting.
2. Change field of view to 500 µm x 500 µm.
3. Start acquisition by clicking the black arrow and check Adjust SI.
4. Adjust the sample holder height by moving the secondary ion circle to the center. The secondary ion circle may appear to be fuzzy due to charging.
5. Go to Analyzer/Main in instrument window (shown right), click TOF on left colunm.
6. Check the box before Linked.
7. For positive ion mode, decrease reflector voltage (move to the left) until SI circle disappears on the SE/SI window.
8. From this point, increase 20V (move 20 V to the right).
9. Uncheck Adjust SI
10. Change Field of view to desired value and start acquisition by clicking the black arrow and hit the same button once a spectrum is shown in spectrum window
11. Calibration the mass spectrum. (note: calibration will be off if reflector voltage is changed).



Spectrum and Images:
Data Acquisition with Bi Single Beam



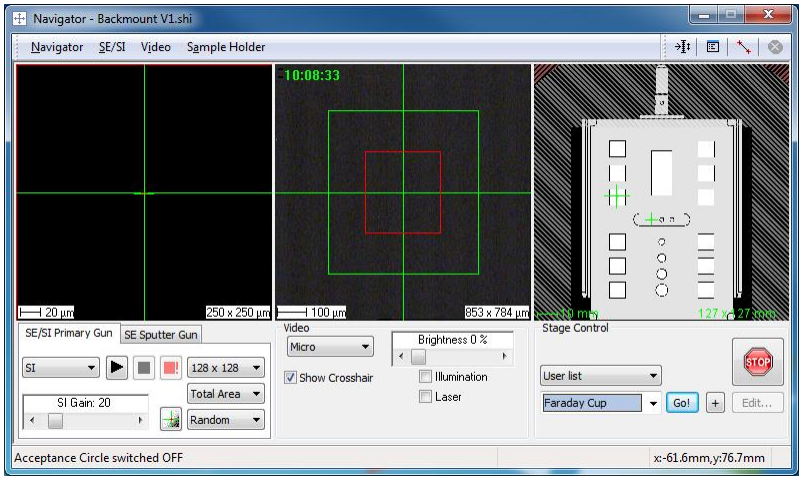
1. Start data acquisition by clicking the red arrow from the Fpanel.

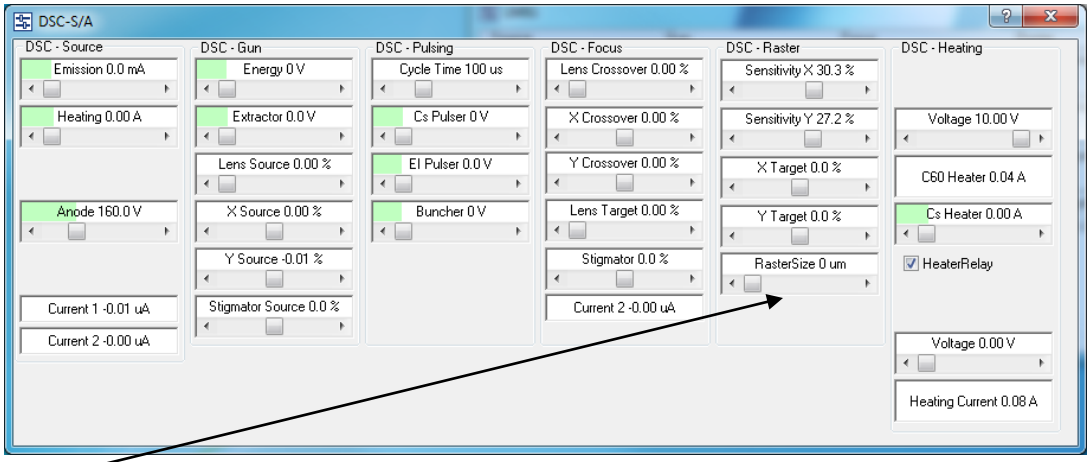


2. Select 2D for spectrum and image acquisition. Change raster to random (128 x 128 pixel) for high mass resolutions spectra acquisition and sawtooth (256 x 256 pixel) for high spatial resolution imaging acquisition.
3. Type a desired number in Number of scans and then click OK.

4. Save the measurement after the acquisition is finished.

Depth Profiles: Data Acquisition with Dual Beam





1. Check sample height
2. Right click mouse on SE/SI image window above and change Field of View to 500 µm x 500 µm.

3. Change Raster size in DSC-S-A window to 120 µm x 120 µm.

4. Align LMIG and sputtering gun: start a SI image of the sample in the navigator.

5. Enable the sputtering beam for a few seconds (Check the box before sputter gun and then uncheck in a few second), the modification of the SI yield by the sputter beam on the sample should become visible in the SI image. Typically, the sputter beam lowers the SI yield by cleaning the sample from surface contaminants and by reducing the oxygen content at the surface.

6. Move mouse to the middle of the crater generated by sputtering beam and drag to about 50 µm x 50 µm.

7. Right click mouse on SE/SI image window and change Field of View to 50 µm x 50 µm.

8. Start acquisition by clicking the black arrow , stop the acquisition by clicking the black stop button.

9. Calibrate the spectrum and add the ions of interests to peak lists.

10. Move to a new spot that is close to the alignment spot.

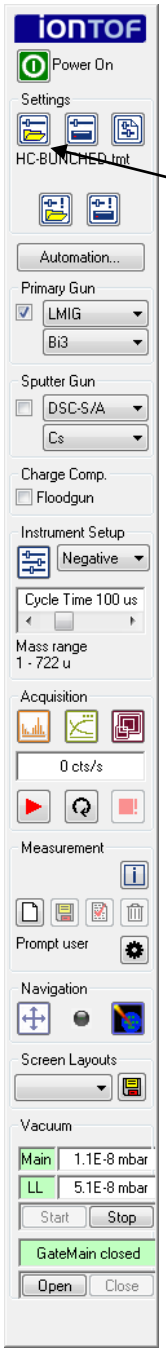
11. Open depth profile window.

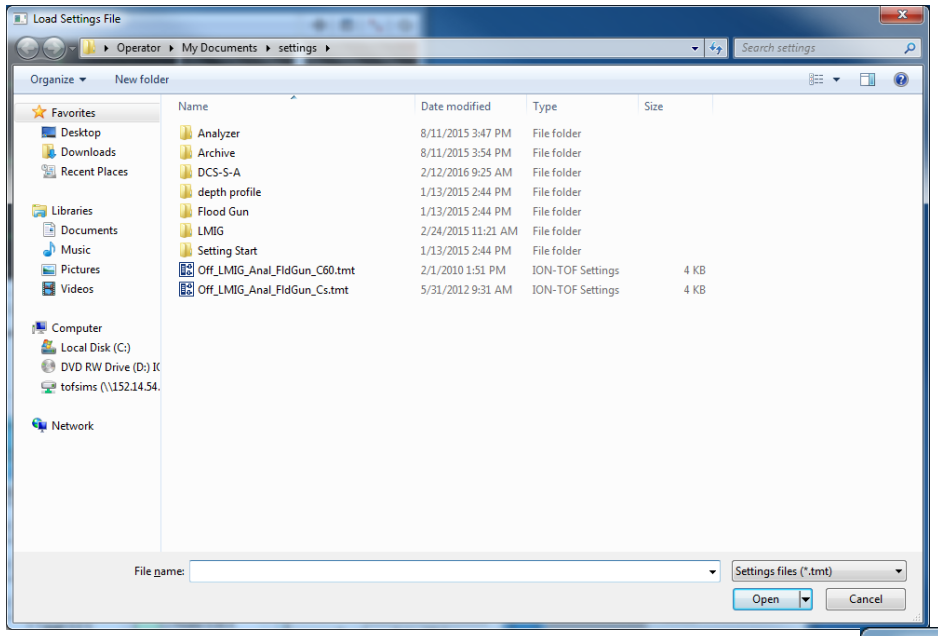
12. Click the red arrow in Fpanel to start the acquisition

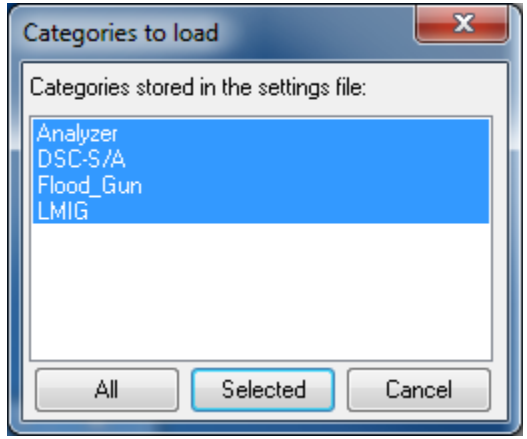
13. Select 3D for depth profile. Change raster to random (128 x 128 pixel) and type a desired number in Number of scans and then click OK.

14. Save the measurement after the acquisition is finished.

Shut Down Procedure

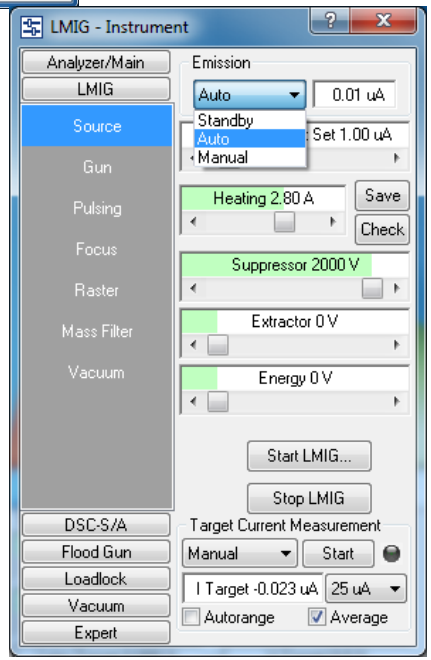






1. Click load setting button on Fpanel and then select off_LMIG_Anal_FldGun_Cs file.
Note: If Cs is on, reduce heating to 0A and then wait for 5 min before loading off Cs file.

2. Select the categories that need to shut down (ctrl+click for multiple entries) and then click selected



3. Change auto to standby and turn heating to 0A from LMIG window.
4. Turn off illuminator.
5. Select exchange position from drop down list and click go.
6. Open the gate between LL and Main,
7. Move the holder to LL.