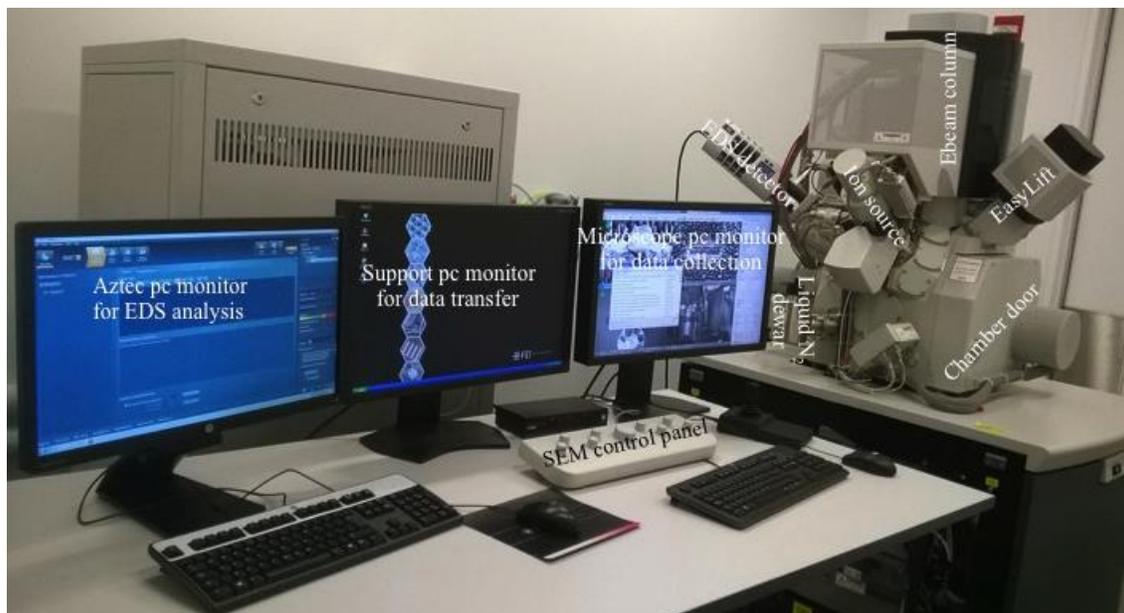


# FEI Helios NanoLab 660 Dualbeam FIB-SEM Operation

## Instruments Layout



## Computers Layout



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# Introduction

1. Check the status of FIB-SEM: the green light in front of the Helios is on (Figure 1).



Figure 1

2. On the SEM server computer, check whether xT microscope server and xT User Interface (xT UI) are running: if not double click the “FEI System Control” on the desktop to



Figure 2

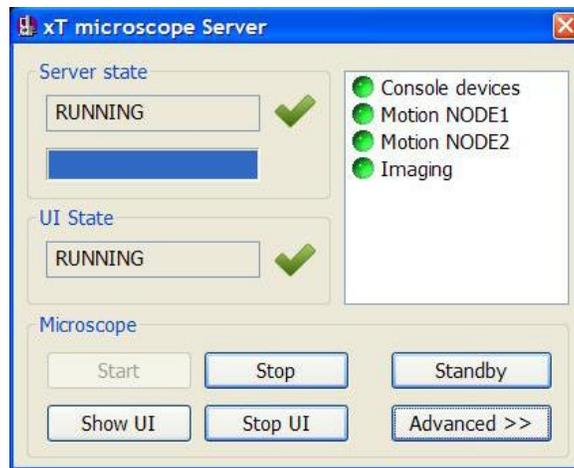


Figure 3

launch xT microscope server (Figure 2). Double click the blue area to show full view (Figure 3): Make sure that indicators for **Console devices**, **Motion NODE1**, **Motion NODE2** and **Imaging** are green in the right panel. Click “Start UI/Stop UI” button to start/stop UI. During starting UI a log-in window will pop-up for you to log in. Username and password are both “supervisor”. Full view of xT UI (also called xT microscope control) is shown in Figure 4.

3. Check the vacuum: all three parts of microscope icon in the lower right corner of the main xT UI are green (Figure 4).
4. Check the CCD quadrant (must be live): the stage is 0° tilt; there is enough distance clearance between stage (no tilt) and the pole piece (stage must be below the 4mm line); retractable parts (GIS needle and Easylift probe) should be retracted (Figure 4).

Note: GIS control is in Patterning  page and Easylift control is in Navigation  page.



Figure 4

5. Check both Ebeam and Ibeam quadrants, both Ebeam and Ibeam should be off: **Beam On** button in grey indicates that beam is off; **Beam On** button in yellow indicates that beam is on (Figure 5).

Note: Orbital icon  indicates Ebeam and the ball cluster icon  indicates Ibeam; The green bar above **Beam On** button indicates that beam is ready and white bar indicates that beam is not ready.

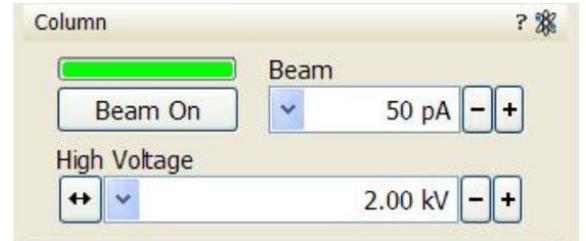


Figure 5

6. Check and make sure there are no unresolved errors in xTm Application Status message window in the CCD quadrant (Figure 6). The message window can be open through **Tools > Application Status** if it does not pop-up automatically. Note: Click **Hide** in Application Status window to bring CCD live view to front.

Note: DO NOT click **Clear** in Application Status window.



Figure 6

## SEM Operation:

### *Loading/Unloading Samples*

1. Click **Vent** button in Beam Control  page of UI to vent the chamber (Figure 7). Click **Yes** on the xTm: Vacuum Message dialog.



Figure 7

2. Venting the chamber for ~3 minutes, the chamber door is ready for open.
3. Open the chamber door slowly, and locate the sample stage. Sometimes moving stage to 60 mm in both X and Y in Navigation page  > **Stage** > **Coordinates** > **X/Y** will facilitate sample loading/unloading. Attach sample tightly to proper pin stab and fasten pin stab to the stage (Do NOT overtight the screw). Adjust the height of sample to make sure the sample surface is between 4 and Max markers of the gauge (Figure 8).

Note: Watch the live CCD quadrant during opening and closing the chamber door and be prepare to stop for unexpected incidents.



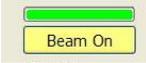
Figure 8

4. Close chamber door fully and click **Pump** button in Beam Control  page of UI. Wait until all three parts of microscope icon in the lower right corner of the main UI turn green (Figure 4). It is recommended to wait until the chamber pressure is below **4e-5** mBar (Figure 4). Park mouse cursor over microscope icon to read chamber pressure if it is not displayed.

### *Ebeam Imaging*

1. Go to **Stage** > **Take Nav-Cam Photo** to take a photo of sample layout. Nav-Cam photo allows user to quickly move to area of interest by double-click. The green cross centers at area of interest that is currently viewed by Ebeam.

Note: Always watch live CCD quadrant when moving sample stage.

2. Click Ebeam quadrant, select proper HV (start with 2kv or 5kv), current (start with 25 pA), dwell time (300 ns for searching), resolution (start with 768 x 512) and filter mode (average 4 frames). Click **Beam On**  button to turn it in yellow. Click Pause button  in Toolbar (Hotkey: F6) to start scanning and watch live image in Ebeam quadrant (Hotkey F5 switches selected quadrant to full screen view mode; Hotkey F7 activates reduced area view mode). Reduce magnification if needed and locate a visible feature on the sample surface. Adjust **Brightness/Contrast** knobs using SEM control panel or use auto contrast and brightness button  in Toolbar (Figure 4) to obtain clear image.

3. Try the best to focus on sample surface feature until a mag > 2000 x, then click Link Z to Working Distance button  in Toolbar. Go to **Navigation** page , change Z coordinate of the stage to 6 mm, focus on the sample again, and click Link Z to Working Distance button  again, then change Z to 4 mm, and focus on the sample.

Note: it is a good practice to link Z to working distance if Z height and/or focus have changed.

4. Check stigmatism by changing focus back and forth to see if the image shows any stretch. If so, adjust focus until no stretch showing, and adjust **Stigmator X** and **Y** knobs on SEM control panel to minimize the astigmatism (showing sharpest image).

5. Take SEM images by using **Photo** button in **Image Acquisition** . You can change the setting of **Photo** in **Scan > Preferences > Photo Preset**.

6. High resolution imaging in mode 2 (Immersion): switch SEM mode to Mode 2 (Figure 9). Repeat step 4 and step 5 to achieve sharpest image.

Note: Response of image movement is much more sensitive in mode 2 than that in mode 1, so stay focused and be patient.

Note: TLD detector for secondary electron (SE) will be automatically chosen to replace ETD detector in mode 2.



Figure 9

7. When finishing, Click **Beam On** button to turn it to grey. Tilt the stage back to 0° if it's not at 0°. Click **Vent** and wait for ~3 minutes to unload the samples, after that, close the chamber door and click **Pump**.

Note: the following step 8, 9, and 10 are optional.

8. Direct alignment:

- a. Click the icon  and direct alignment will show up as a pop-up window (Figure 10 left or right panel).

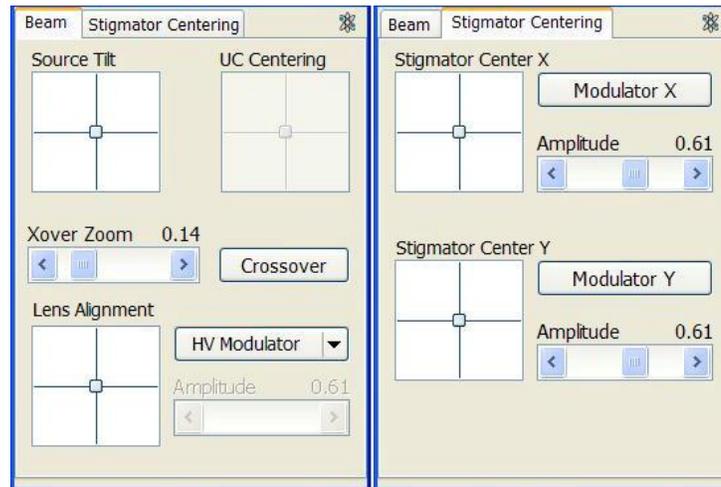


Figure 10

- b. Choose **Beam** page and click **Crossover** button, the Ebeam quadrant will show the beam crossover image. Use **Xover Zoom** sliding bar to zoom in/out crossover if needed. Move mouse cursor to **Source Tilt** square, click either lines or center icon and hold mouse left button to center the crossover in Ebeam quadrant. After you are done click the **Crossover** button again to return to normal Ebeam image. Adjust focus and stigmatism to get sharp view.
- c. Next click **HV Modulator** in **Beam** page, Move mouse cursor to **Lens Alignment** square, click either lines or center icon and hold mouse left button to minimize image movement in Ebeam quadrant. Use **Amplitude** sliding bar to change the amplitude of image movement.
- d. Now choose **Stigmator Centering** page and activate **Modulator X**, move mouse cursor over **Stigmator Center X** square, click either lines or center icon and hold mouse left button to minimize image movement in Ebeam quadrant, repeat this step for **Modulator Y**. Use **Amplitude** sliding bar to change the amplitude of image movement. Close direct alignment window after you are done.

Note: it is recommended to do direct alignment again if any one of Z height, HV, beam current has changed.

9. Multiple detectors set up: It is possible to use up to three detectors simultaneously in Ebeam imaging (quadrant 1-3, always keep quadrant 4 for chamber CCD). Most frequently used combination is ETD for SE, Mirror Detector (MD) for Backscattered Electron (BSE), In-column Detector (ICD) for BSE. If sample is not tilted, a two detector combination is often used: ETD for SE and retractable CBS detector for BSE. Shift + left click  to control scanning in all detectors simultaneously.
10. SmartScan can be setup by right click any one of 1 through 6 button in Scanning Preset (Figure 4). Right click any number (**1 through 6**) and choose **edit** to bring up smartscan setting window (Figure 11). Choose shorter dwell time (< 500 ns) and integrate 2<sup>n</sup> frame (normally 32 to 256) to generate the final image. This is an efficient strategy to avoid charge build-up for non-conductive samples. Another one is to choose low HV(1 kV, 500V, 350V) and beam current (25 pA, 7.7 pA, 1.1pA). Press **Ctrl** button and click on selected Smartscan button to acquire an image.

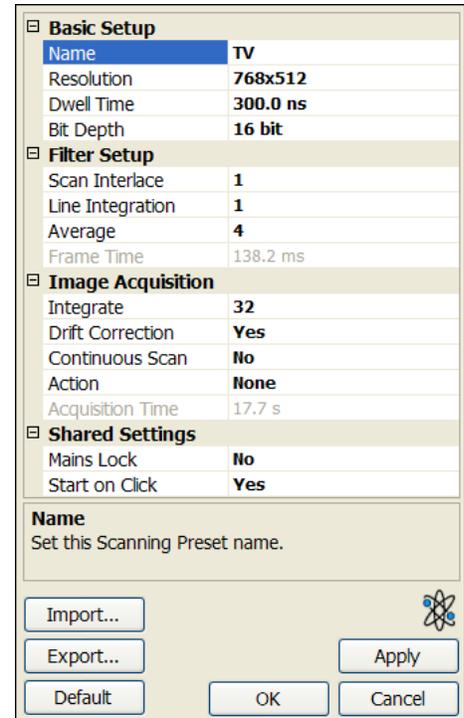


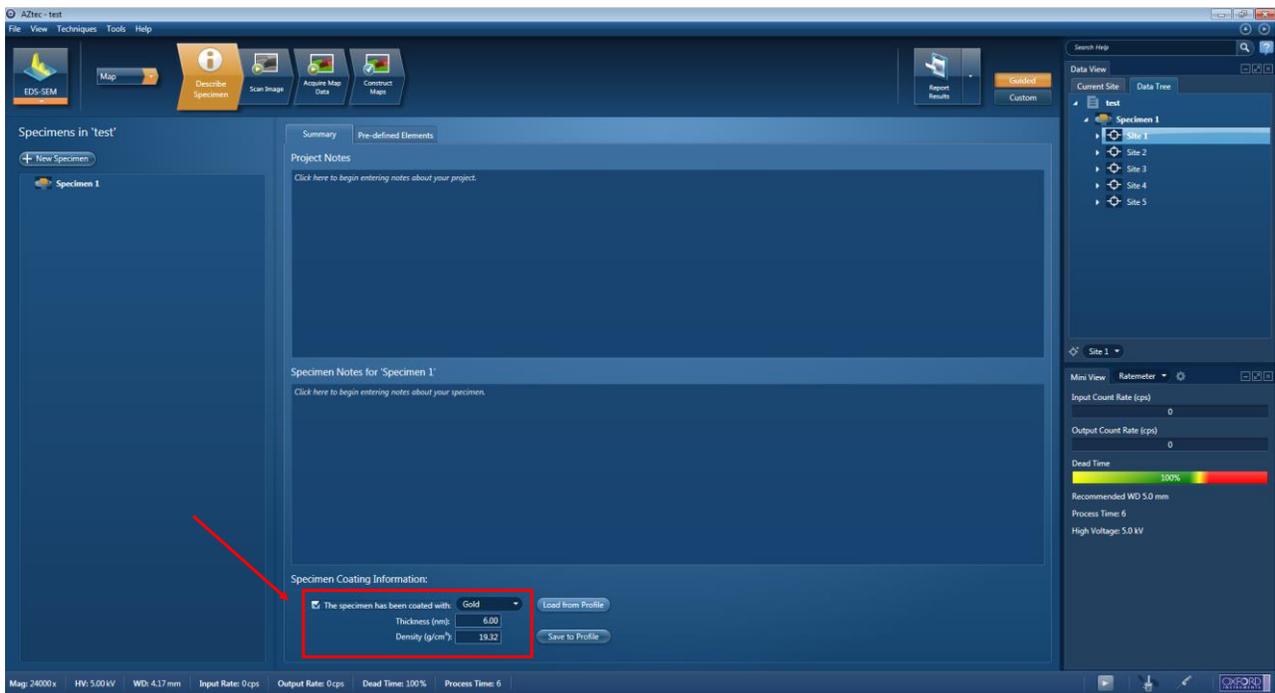
Figure 11

## EDS operation:

1. Log in the EDS computer, password “supervisor”, start Aztec program.
2. Click on the New Project button and select the folder to save the project.

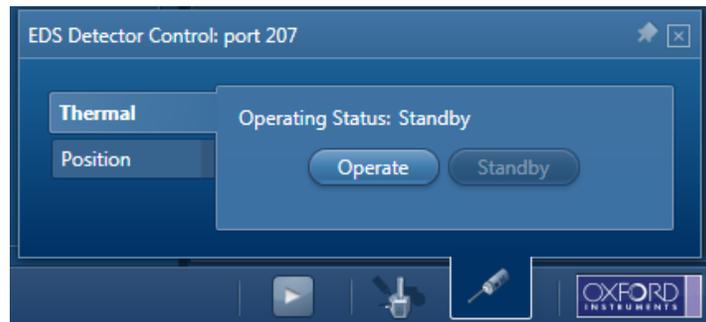


3. Aztec defaults to a sputter coating of Gold for your sample. Make sure you uncheck this if you did not coat your sample.



4. Make sure CCD quadrant is paused  ; it provides a high noise to the EDS detector.

5. Click on icon "EDS Detector Control"  in bottom right of software and click on **Operate** in "Thermal", wait until the detector fully cooled, which will show "Cold" and the blue light on the detector stops flashing. Then insert the detector by clicking **In** in "Position".



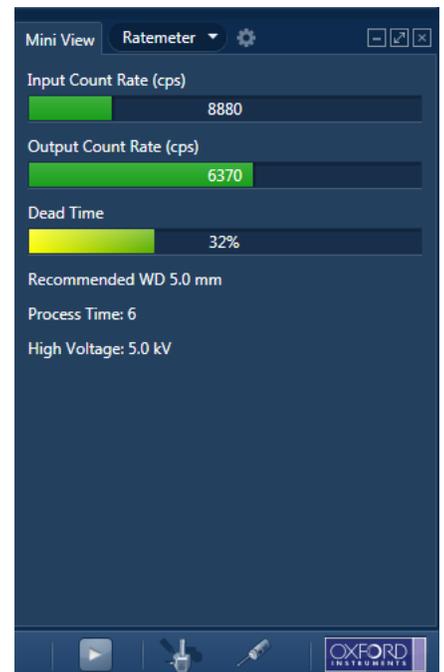
6. In SEM, use a high enough kV to excite the x-ray peaks you are interested in (typically 2x peak energy). For example, if you want to see a peak that appears at 6 keV, use 10 kV or 15 kV accelerating voltage (not 5 kV).

7. Ideally, you want to increase your current so that the dead time is in between 20% to 60% when SEM is live (such as 1.6 nA or 3.2 nA). DO NOT let the dead time exceed 60%.

8. Adjust brightness, contrast, focus, stigmator alignment in SEM.

9. In AZtec, choose what type of EDS scan you want to perform from the list in the top left ("Map" is recommended for general use):

- a. Analyzer - This will provide you with a general spectrum encompassing all the sample in the SEM

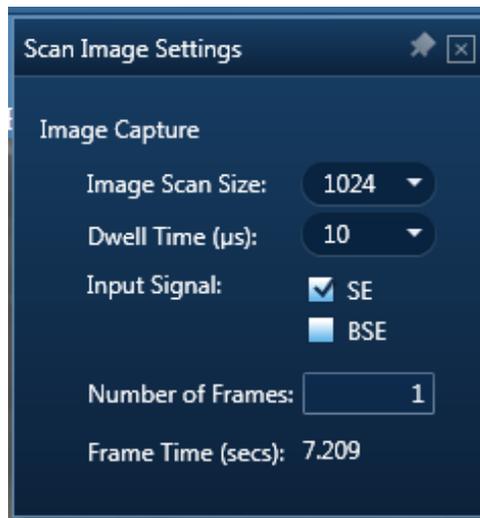


window (collect with the SEM live, not paused). This is ideal if you have a uniform sample and you are trying to identify what elements are present.

- b. Point & ID - This technique allows you to grab an image of your sample and choose specific locations to acquire EDS spectra from. This is useful for verifying if certain locations contain elements that other locations do not
- c. Linescan - This technique will provide you with a linear elemental distribution across your sample
- d. Map - This technique will provide you with a 2-D elemental distribution of your sample
- e. Optimize - This is used for calibrating the detector and you should not use it.



10. In **Scan Image** Page, check the settings as following and press **Start** to capture an SEM image in the area of interest.



11. In **Acquire Map Data** Page, check the settings menu:

- a. Process time dictates data binning such that small process times lead to faster scans but broader peaks. So choose a higher process time (5 or 6) if you are looking for elements which have peaks close together.
- b. For maps and linescans, choose "Until stopped" or "Fixed duration" with adequate frame count for your needs.



12. Draw a rectangle, elliptical or freehand region from the left side menu to start acquiring EDS data in selected area. Note: pressing **Start** will collect EDS data on the whole SEM image region.



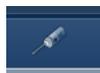
13. In **Construct Maps** Page, you can save the spectrum data by right click > Export > EMSA. It will save the x-ray energy (keV) and cps/eV in a text file.

14. Click **Report Results** on the top right and choose “**Site Report**” to export a Word file containing all the images and spectrums of selected site.



15. Repeat step 10 to 14 for any other area of interest on the samples. You can add new sites in the Data Tree on the right or add new Specimen.



16. When finished, click  to retract the detector (Position > **Out**) and set it to **Standby** in Operation.

## Focused Ion Beam (FIB) Application (imaging, milling, and patterning)

### FIB alignments and imaging:

1. If the Gallium ion source is not heated (The status bar above **Beam On** button is empty), click **Wake Up** button (Figure 4) in **Beam Control** page to start heating Gallium ion source until the status bar above **Beam On** button turns green.
2. Perform beam alignment in Ebeam (mode 1:field-free) and move stage Z to 4 mm..
3. Zero beam shift for both beams in **Beam Control** page: right click Beam Shift square in **Beam** tab and select **Zero** (Figure 12).

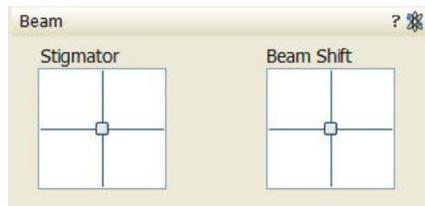


Figure 12

4. Set Eucentric height: Center a recognizable feature by double click it in Ebeam quadrant, fine tune focus and stigmatism to achieve sharpest image, link Z to working distance. Make sure Ebeam quadrant is live and tilt the stage to  $2^\circ$  while watch the centered feature in Ebeam quadrant. If the feature move out of the center of screen, adjust Z height in navigation page  to bring the feature back to the center of the screen. Then Tilt stage to larger angle (e.g.  $5^\circ$ ) and bring the feaure back to center if it moves. Repeat this step and each time choose a larger tilt angle ( $2^\circ$ ,  $5^\circ$ ,  $15^\circ$ ,  $40^\circ$ ,  $52^\circ$ ) to minimize the feature movement during tilting. The final tilt angle is  $52^\circ$ . At  $52^\circ$  tilt angle fine tune focus and stigmatism and link Z to working distance  .  
Note: do this step at relatively low mag first and increase mag to check; setting eucentric height is required for Ibeam application.
5. Choose HV(30 kV) and beam current (25/40 pA) for Ibeam imaging. Click **Beam On** button to switch on Ibeam (button turn yellow). Quickly adjust Brightness/contrast, focus, and stigmatism using reduced area  (HotKey: F7).
6. Make sure that both beams center on the same area of sample (Coincident Point): center a recognizable feature in Ebeam, adjust beam shift of Ibeam in Beam control page (Figure 4) to center the same feature in Ibeam imaging (HotKey: Shift + mouse left hold and drag).

Note: Try checking **Dynamic focus** button in **Navigation>Stage>Tilt Correction** to see if the whole imaging area in Ebeam have the same focus.

7. In order to protect samples from being destroyed by Ibeam, choose smaller beam current (< **40 pA**), shorter dwell time (**50-100 us**), and low resolution (**768 x 512**) for Ibeam imaging. Smaller beam current higher the resolution.

Note: choose Ibeam current >100 pA for milling and patterning.

### **Ebeam Pt deposition:**

The beam (depending on whether you are doing Ebeam or Ibeam deposition) must be perpendicular to the sample surface during Pt deposition. Ibeam can easily destroy the top layer of samples of interests, it is necessary to add a thin layer of Pt layer by Ebeam before Ibeam deposition:

1. Ebeam deposition is to add a thin layer of Pt (0.1 um) to protect sample surface in Ibeam deposition process.
2. Return stage tilt to 0°.
3. Draw rectangular pattern in Ebeam quadrant and choose **Pt e-dep surface in Pattern>Basic>Application** (Figure 13), pattern should be in green color, make sure the pattern is slightly wider than area of interest. Choose depth to be 0.1 um. Make sure the GIS is in **Warm** status (bottom panel in Figure 13).
4. Choose HV (2kv) and current (1.6-2.4 nA). Adjust beam brightness/contrast, focus, and stigmatism after changing HV and/or current.
5. Insert GIS needle by checking green box in **Pattern>Gas Injection** and watch live CCD quadrant.

Note: After inserting GIS needle, take a quick Ebeam snapshot (double click F6) to make sure there is no stage drift.

6. Click **Play** button  in toolbar to start deposition.
7. After the Ebeam deposition is done, take a quick Ebeam snapshot to make sure the Ebeam deposition works. Retract GIS needle.

### **Ibeam Pt deposition:**

1. Tilt the stage to 52° and take a snapshot in Ibeam quadrant using low beam current (<40 pA). Find the Pt layer and adjust Ibeam brightness/contrast, focus, and stigmatism using reduced area if needed.

2. Draw rectangular pattern in Ibeam quadrant and choose **Pt Dep** in **Pattern>Basic>Application** (Figure 13), pattern should be in green color, make sure the pattern is slightly smaller than the existing Pt layer. Choose the depth to be 1-2  $\mu\text{m}$ .
3. Calculate the area of rectangular pattern in  $\mu\text{m}^2$  and choose proper Ibeam current to guarantee that the averaged beam density is  $6 \text{ pA}/\mu\text{m}^2$ . In addition, the total deposition time of 10-15 mins indicates the correct choice of beam current. After choosing Ibeam current do a quick Ibeam focus and stigmatism.

Note: DO NOT use Ibeam current that is larger than the calculated max current.

4. Insert GIS needle, take a quick Ibeam snapshot to make sure there is no image shift. Click **Play** button  in toolbar to start deposition. Activate iSPY to monitor the process of deposition if you prefer.

Note: iSPY is optional, you can turn on iSPY for the first several minutes and turn off iSPY after you have confirmed that the deposition is really happening.

5. After the Ibeam deposition is done, retract GIS needle and take a quick Ebeam snapshot to make sure the Ibeam deposition works.

### **Ibeam milling/patterning:**

If the top layer of sample need to be protected from Ibeam damage during milling/patterning, do the Ebeam and Ibeam deposition first before milling/patterning.

1. Select pattern shape in the pull-down menu of **Pattern** tab (left panel of Figure 14) and draw it in the Ibeam quadrant (pattern should be in yellow color); choose proper parameters in **Basic** and **Advanced** tab in **Pattern** tab (right panel of Figure 14): Application (**Si New** for rectangle pattern, **Si-multipass New** for regular cross-section and **Si-ccs New** for cleaning cross-section), X Y Z size, ScanDirection, etc.

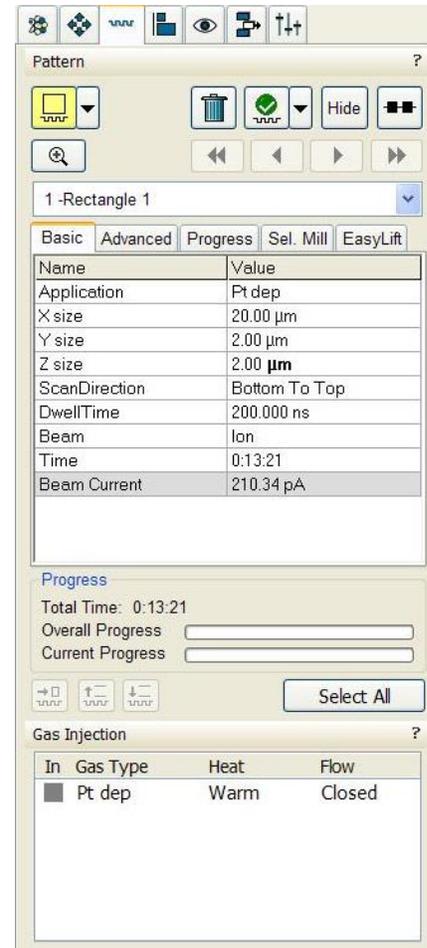


Figure 13

- Choose HV (30 kV), choose proper beam current (> **0.43 nA**), click **Play** button  in toolbar to start patterning.

Note: the Ibeam current choice depends on various patterning application. The following shows the most frequently used regular cross-section milling.

- Monitor patterning process: check **On** button in **Pattern>End Point Monitor>iSPY**, choose proper time interval. iSPY takes Ebeam snapshot for every time interval defined.
- Regular cross-section milling is most frequently used. Always draw regular cross section pattern wider than area of interest. Cleaning cross section is useful tool to make flat surface of cross section and is good for milling ball-like structures. Current selection for patterning:

Rough milling: 21, 47, 65 nA

Cleaning/regular cross section: 0.79, 2.5, 9.3 nA

TEM lamella polishing: 0.23, 0.43, 0.79 nA

Note: if choosing >21 nA to do rough milling, it is good practice to leave a safe distance (1-2 um) from the Pt deposition layer and then choose 2.5 nA for fine cleaning.

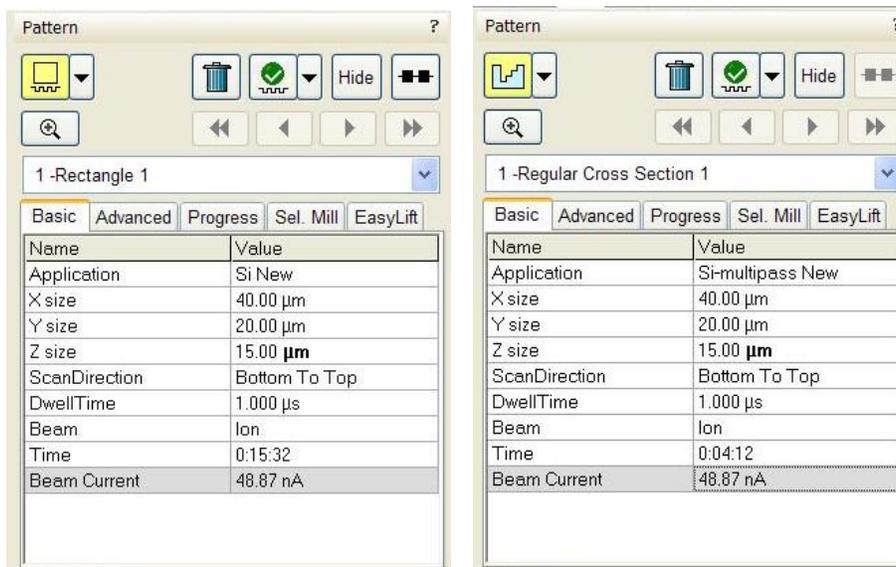


Figure 14