

# FEI Helios DualBeam System Operation Manual

Helios DualBeam system is a charged particle microscope that integrates both electron and Ge ion beams in it. The electron beam is primarily used for imaging and the ion beam is primarily used for top-down fabrication tasks such as TEM lamella preparation, cross-section surface preparation, and circuit editing. The Helios system at IAC comes with FEI EasyLift Nanomanipulate system which facilitates sample lift-out process. This manual describes (1) SEM imaging modes and their applications, (2) FIB operation, and (3) general guidelines of TEM lamella preparation workflow. The operation steps in the manual are organized in chronological order. In addition, basic description of the microscopy user interface (UI) is provided in the end of the manual.

## General precautions

- Always wear gloves when handle samples and parts such as sample stub and holder that are used in the chamber in order to prevent chamber contamination. The ion beam operation requires good vacuum condition ( $\leq 3 \times 10^{-6}$  torr).
- Always align the ion beam in the beginning of a session that involves the use of ion beam. Align each current/voltage to be used in the session.
- Load appropriate sample holder(s) for your session. Both user sample and the grid row holder can be fitted on FEI's universal mounting base (UMB). The row holder is required for TEM lamella preparation work.
- The working distance of the tool is 4 mm. This is the eucentric position for the dual beam system.
- Magnetic materials can be imaged only in Mode 1 (regular magnetic field free mode).

## Operation summary

1. e-beam imaging and EDS
  - mount the sample to an appropriate sample holder
  - pump down the chamber
  - turn on the beam(s)
  - choose a desired beam energy to work with (2 kV works best for imaging)
  - focus the sample at reasonable magnification (~5000 X) and link WD to stage z-height
  - align the beam by using the direct alignment tool
  - choose a desired imaging mode and detector and start the imaging session
  - insert the EDS detector and start the chemical analysis work
  - when the work is finished, retract the EDS detector, vent the chamber, take out the sample, and pump down the chamber
2. dual beam operation (TEM lamella preparation and cross-section preparation)
  - mount the sample to an appropriate sample holder, pump down the chamber, and turn on the beams
  - choose a desired e-beam energy to work with (2 kV works best for imaging)
  - focus the sample at some reasonable magnification (~5000 X) and link WD to stage z-height
  - align the e-beam by using the corresponding direct alignment tool
  - set the stage to its eucentric height
  - align the ion-beam at each current to be used
  - use ion beam shift to align the area of interest such that both e- and ion-beams look at the same area
  - start your sample preparation session
  - when the work is finished, vent the chamber, take out the sample, and pump down the chamber

## SEM operation – imaging modes

During the chamber pumping step, take a NaviCam image under ‘Scan’ tab for easy stage manipulation before turning on the beam(s). The optimal acceleration voltage for SEM imaging is 2 kV at 50 pA. When the vacuum is ready (chamber schematic turns green, or vacuum is at least under  $10^{-6}$  Torr) click ‘Wake Up’. Check that the screen quadrant you’ve selected is in e<sup>-</sup>-beam mode (bottom-left corner of quadrant should display an ‘atom’ graphic) and click ‘Beam On’. You can now begin imaging your sample.

The microscope has three main SEM imaging modes:

- Mode 1 (Field-Free) is a low-resolution mode used for sample navigation.
- Mode 2 (Immersion) is used for high-resolution imaging. Note that it uses a strong magnetic field, so should not be selected when dealing with samples with loose magnetic parts. The lowest magnification in this mode is 2000 X.
- Mode 3 (Weak-Immersion) is the EDX mode. It has improved resolution at higher currents, as prevents backscattered electrons reaching the detector, but requires a higher voltage (~20 kV). It also uses a magnetic field, so the same concerns apply as with Mode 2.

## SEM operation – e-beam alignment

The SEM performance can be fine-tuned by using ‘Direct Alignment’ located on the commonly used function list or the tab on the right of the UI. The alignment steps are:

1. Crossover – Activate and then center the cross that appears in the bright spot.
2. HV Modulator – Using a fast scanning speed (25ns) minimize image movement with the grid next to the HV Modulator button. Don’t really need to use Lens Modulator.
3. Stigmator Centering – Again, move to minimize ‘wobble’. If the movement is too extreme, right-click on the grid and turn on ‘Adaptive Sensitivity’.
4. Contrast/Brightness – Although the auto function is serviceable, to optimize Contrast/Brightness it is necessary to do it manually. Turn on the ‘Scope’ (oscilloscope display graphic) button on the tab at the top of the screen and put contrast to zero. Adjust brightness until the signal hovers just above the baseline on the screen. Finally, increase contrast until the peaks of the signal are just within the screen and turn off the ‘Scope’.
5. Stigmation – Adjust this using a small window to optimize image quality.

## Dual beam operation – setting the stage to its eucentric height

For ion beam operation, the sample needs to be set at the eucentric height (e-beam WD ~4 mm). The stage information can be found in the ‘Navigation’ side tab. Using the following steps to set the eucentric height:

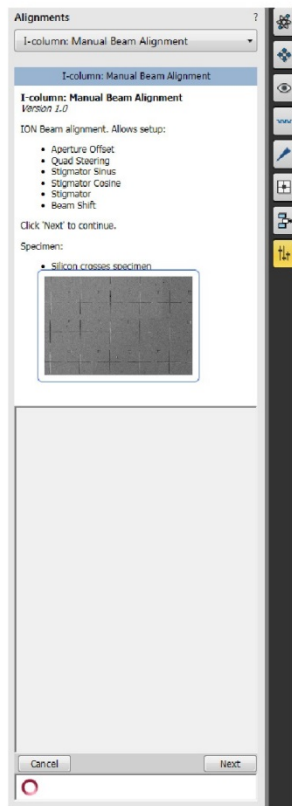
1. Use SEM to locate and move your feature of interest (FOI) to the center cross position of SEM image window (Quad1).
2. Tilt the stage to 15° and watch the shift of FOI. If it moves away from the center cross, adjust Z-height appropriately to bring the FOI back to the center. Do not move the stage x or y.
3. Tilt the stage back to 0° to check if the FOI moves away from the center cross. If yes, double click to move it back when at 0° tilt.
4. Tilt the stage to 52° and watch the shift of FOI. If it moves away from the center cross, use Z height adjust bar to move it up or down. (Usually only needs fine adjustment at this step).

5. Assign Quad 2 as ion imaging display. Choose ion beam energy and current to be 30 kV and 7.9 pA for imaging. Turn on the ion beam, click on Auto Contrast and Brightness button if needed.
6. Using the SEM image as your reference, align the ion beam image to the reference image by using ion beam shift X and Y.

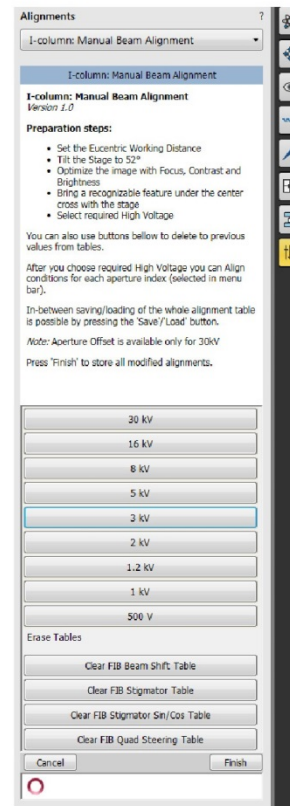
### Ion beam operation – beam alignment

1. Go to the ‘Alignment’ side tab on the right of UI and select ‘I-Column: Manual Beam Alignment’ as shown in **Fig. 1**.
2. Start the alignment procedure by following the screen prompts. Skip ‘Aperture Alignment’ step.
3. Execute ‘Quad Steering’ and ‘Stigmator’, refocus the image and save the setting for every current/voltage to be used in your session.

(a)



(b)



**Fig. 1** Ion-beam alignment is done by (a) I-Column Manual Beam Alignment. (b) The alignment is required for every beam condition to be use in the session.

### TEM lamella preparation

At the eucentric height of the area of interest, proceed to lamella preparation after aligning both e- and ion-beams. Please note that the actual lamella milling and thinning parameters vary with materials, and the parameters used here are based on Si.

1. Deposition of sample protection layers

Deposit a thin layer of C or Pt with the e-beam (2 kV and 3.2 nA) at 0° tilt. This e-beam deposition is necessary for ion-beam sensitive materials.

To deposit on the sample, warm up the appropriate GIS needle (warm up Pt even if using C, as it will be required for subsequent steps). Wait a few minutes after the needle says 'Warm', to maximize performance. Ensure e-beam image is focused and aligned at the current/voltage selected. Draw a 'Rectangle' box of necessary size on your sample, and then go to the side-panel and select parameters:

- Type: select either e-beam C-dep or Pt-dep.
- Depth: a few (2-300nm) is sufficient.
- Overlap: this determines the overlap distance of successive spots, expressed as a percentage of the 'Pitch'.
- Pitch: the distance between the centers of successive spots.
- Blur and Defocus: these two settings defocus the beam. 'Blur' specifically increases the beam diameter.

After e-beam deposition, tilt to 52° to deposit an appropriate amount (2µm is standard) of Pt in a 'Rectangle' box pattern using the ion beam at 30kV. To determine current, the rule is to calculate the area of the deposition box (in µm × µm) and multiply by 10, to give the current in pA. Focus/contrast the ion beam image by pausing/unpausing image acquisition and incrementally altering focus or contrast. You can watch the deposition process using the (2 kV) e-beam at a current 3x as large as the ion beam current.

## 2. Milling (ion-beam at 30 kV and 21 nA)

After finishing Pt-dep, wait for vacuum to reach 10<sup>-6</sup> Torr range. This will prevent re-deposition when you begin milling the sample. Then use 30 kV and 21 nA. Focus/contrast the ion-beam at this voltage/current combination as above. Use REGULAR CROSS-SECTION, and 'Si-multipass' setting to mill.

Draw two 'Regular Cross-Section' boxes, one above the sample and one below. The upper box should be longer (in y-direction), and the lower box should be deeper. Make the trench about 15µ long in the x-direction – this will prevent the Pt concentration in the trench being too high when you begin removal of the sample later on. Set mill direction so that the beam mills towards the sample (so the deeper ends of each box are closest to the sample). This means you mill 'Bottom to Top' for the lower box, and 'Top to Bottom' for the upper box.

## 3. Cleaning (ion-beam at 30 kV and 9.3 nA)

Use 'Cleaning Cross-Section' patterning tool with 'Si-Invert CCS' setting to clean the sides of the lamella. Make a small box very close to the Pt-dep area. Tilt 1.5° towards the deeper side (more if sample is harder) and mill to same depth. Tilt to 0°, rotate 180°, and then tilt to 50.5°

## 4. U-Cut (ion-beam at 30 kV and 2.5 nA)

- Tilt to 0°, but do not rotate – you need to cut from the shallower side. Use 2.5nA, REGULAR box (not cross-sections), and again, focus/contrast by pausing/unpausing image acquisition. Set up 4 boxes as in the picture, and select 'Parallel Mill' in the side-panel to mill the boxes simultaneously. Note that on the left-hand side, the box does not cut all the way through. You can also click 'Select All' to enable you to drag all the boxes at once. Set a depth (2-3µm) that will be certain to mill through to other side, and take periodic snapshots with the ion beam to see if the cut has been made.
- You may want to tilt the sample a small amount (-5°) to check the sample has cut cleanly, and increase current to 9.3nA if necessary, particularly if your trench was not wide enough, and the Pt re-deposits when cutting through the sample. It is also possible to check on the SEM to view from a

different angle – however, use snapshots so that the ion beam blocks. This means that you can choose any current for the e-beam.

- Tilt back to 52° once the cuts have finished. It may be necessary to utilise a CLEANING CROSS-SECTION (Si-invert ccs) to ensure cut is flat, and isn't curved at one end. Tilt to 53.5° to check. A good figure for the final width of the lamella is 1.5µm.
5. Welding the sample to the EasyLift needle and lift out
- Lower the stage to 6mm. Tilt back to 0° and insert Easylift needle to 'Park Position'. Use 'Ctrl' + 'j' to overlay the Easylift graphic on the screen, and move the needle tip to eucentric height (yellow cross), by jogging Z. Use the ion beam (30kV, 7.7pA) to zoom out from sample and find the needle. Insert the GIS Pt needle now, so that if the Easylift moves as a result, it will not knock your sample. Use the e-beam to x-y position the Easylift needle point so that it sits above the end of the sample that has been fully cut through, and ion beam to drag down in the z-direction to bring it close to the sample surface – you should be able to see a shadow cast by the Easylift.
  - Use REGULAR rectangle pattern to deposit a Pt box, attaching the Easylift to the sample. 500nm in depth is frequently enough, but more may be required, depending on the size of the sample and the orientation of the Easylift and sample. Because of the small area, 24pA should be sufficient. As ever, remember to re-focus/contrast every time you change the current.
  - Now, cut through the joining piece that connects the lamella to the rest of the sample; watch the image in the box using the ion beam, and stop it once the piece has been cut through. If the cut is not being made, it is likely that Pt is re-depositing and your trench is too small, and/or you need to wait longer for the Pt to dissipate after having attached the Easylift to the lamella. Once the cut has been made, switch to 7.7pA, turn on image acquisition and lift out the lamella with the Easylift. Retract the GIS, move the Easylift up (in z-direction – use ion beam image) and a reasonable distance from the sample surface. Retract the Easylift needle.
6. Affixing the sample to a TEM grid
- Locate TEM grid using SEM (you should be at 0° tilt) and use 'Align Feature' to make the TEM holder horizontal on the screen. Insert the Easylift, and move it towards one of the grid 'teeth', using ion beam and e-beam. Note that it may be necessary to tilt the stage, particularly if the lamella is at an angle, in order to approach the tooth correctly. Insert GIS needle when the Easylift is still some distance away.
  - Move the lamella close to the tooth, and choose an appropriate place to affix it, depending on size, then use Pt-deposition to affix it. Usually 500nm is sufficient, but bear in mind that the Pt-layer should look smooth – striation is a sign of weak bonding. In such cases, 1µm may be required. Wait 1-2mins after depositing to allow Pt to disperse, and then cut off the needle by milling through the Pt-layer connecting the Easylift to the lamella.
  - Rotate stage 180° and use Pt-deposition on the other side to fully affix the lamella. It may be necessary to rotate the stage a little further to get a better view of the deposition site.
7. Thinning the lamella – 1
- Tilt the sample such that the lighter sections on either side of the lamella appear to be of the same width on the e-beam (starting at 0° tilt). Pause image acquisition on the e-beam, and then switch to the ion beam. Tilt the ion beam (7.7pA, 30kV) to achieve the equal widths again. Correct the beam shift on the ion beam, relative to the e-beam image, until the images on either beam appear the same, and there is no relative shift in the ion beam image when you move on the e-beam image. Save position (use one of the side panels).

- Rotate stage 180° and repeat the above. Save position.
8. Thinning the lamella – 2
- Depending on the sample hardness, there are two parameters that will be varied. The first is current; the second is overtilt. The standard values, which are optimal for Si, to start with are 0.79nA and 1.2°. If the milling is too slow, adjust the overtilt in 0.3° increments, and watch the lamella to monitor mill speed. Depending on whether you are milling from the front- or back-side of the lamella, the tilt value will be increased or decreased by 1.2° to obtain the desired overtilt. In any case, use a CLEANING CROSS-SECTION, Si-invert ccs mode, and start at 30kV, 0.79nA, with 1.2° of overtilt. Make the y-pitch small with a large overlap (85%), to ensure a smooth surface.
  - Mill at 30kV, towards the sample, until you are about 200nm away from your feature of interest. You can watch SEM images as you mill using the iSPI feature, which can be activate at the bottom of the milling side-panel. This pauses milling while it captures an SEM snapshot.
  - Once you are 200nm away from your feature on one side, switch to the other side and mill until you are 200nm away on that side too. The aim is to create a ‘wedge-like’ sample which cuts off just below you feature, with the top with the Pt cap wider than the bottom.
9. Polishing the lamella
- Now that your sample is quite thin, mill at 30kV, 80pA, and reduce the overtilt, which is less necessary at lower currents. Polish the sample, stopping when the SEM image looks good. Then, switch to the other side and repeat. Note that it is only recommended to mill at 30kV until the Pt cap starts to erode; then it is necessary to switch to 5kV. Be watchful for this, and reduce voltage if necessary; the figures above are ultimately guidelines.
  - To get closer to sample and do final polishing, use 5kV, 0.12nA, REGULAR box patterning and a large overtilt. Because of the size of the TEM holder stage, it may not be possible to tilt as much as desired on one side. However, still tilt to the larger value on the other side of the lamella, as symmetry isn’t important here. Also, as the 5kV beam is not very ‘tight’, be sure to be careful placing the milling box to account for this, and have quite a small overlap region between the box and the lamella.
  - For final polishing, switch to 2kV, 72pA. Focus, stigmat etc. to get a good ion-beam view using the ICE detector in SE mode (used for low-voltage ion beam imaging; also works well at 5kV). Also use an even larger overtilt for the lower voltage. Use a REGULAR box pattern and polish either side. Only about 30s on each side is required.

# Helios User Interface

The screenshot displays the Helios User Interface with four numbered callouts:

- 1**: Points to the top menu bar containing File, Edit, Detectors, Scan, Beam, Patterning, Stage, Tools, View, Help, Start, Stop, Hide UI, and Stop UI.
- 2**: Points to the main Quad display area, which is divided into four quadrants labeled Quad 1, Quad 2, Quad 3, and Quad 4. Each quadrant shows a different view of the sample or instrument components.
- 3**: Points to the side panel on the right, which contains various control options such as Pump, Vent, System (Wake Up, Sleep), Column (Beam On, Beam Current, High Voltage), Magnification (Simple Magnifications, Couple Magnifications), Beam (Stigmator, Beam Shift), Beam Deceleration (Stage Bias), Sun Rotation, and Detectors (Contrast, Brightness).
- 4**: Points to the bottom status bar, which displays various parameters including WD (4.0 mm), det (ETD), mode (SE), mag (150 x), dwell (200.00 ns), HV (10.00 kV), HFW (1.38 mm), and system pressure (3.76e-6 Torr).

1. List of commonly used functions 2. Quad display area 3. Side panel 4. System status

## Commonly used functions



### Display Source

Display Electron/Ion/NaviCam/IR image to the selected Quad



### Imaging Mode

Mode 1: magnetic field free mode (ordinary mode)

Mode 2: high resolution imaging mode

Mode 3: high resolution imaging/EDS mode



### Magnification

This box display the current magnification. Desired magnification cen be set here.



### Beam Energy

Desired beam energy can be set here.

Helios e-beam operates the best at 2 kV for imaging.



### Beam Current

Desired beam current can be set here.



### Direct Alignment

Beam: Crossover and HV Modulator

Stigmator Centering: Stig. Center X and Y



### Reduced Area Focus

Select a smaller scan are to facilitate focus



### Link WD to Z

Set working distance (WD) to the stage z-height after focusing the sample area of interest.

This step is required if your session involves any ion beam operation.



### Auto contrast and brightness

The system automatically sets the contrast and brightness for you.



### Scope

Manual adjustment of Iimage contrast and brightness



### Scan time

The longer the scan time, the cleaner the image. Set an appropriate time for your sample.



### Image resolution

Desired resolution can be set here.



### Snapshot

Acquire an image with 'Snapshot' preset.



### Photo

Acquire an image with 'Photo' preset.

## Emergency Information:

Medical Emergencies: **Contact 911 and Public Safety (609) 258-1000**

Room / facility emergencies: **Contact Public Safety (609) 258-1000**

Issues related to the instrument:

1. **Contact IAC Staff.**
2. **Leave system as is, Do Not disable vacuum system.**
3. **Try to shut off the High Tension.**

Audible/Siren Emergency Alerts:

**Follow previous steps 2 & 3 and leave the building.**

## Emergency Contact Information:

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