Operating Instructions for

Helios NanoLab 600

Dualbeam Focused Ion Beam & Scanning Electron Microscope

RULES OF USE

1) DO NOT TOUCH A CONTROL IF YOU DON'T KNOW EXACTLY WHAT IT DOES.

2) NEVER, EVER FORCE ANYTHING BEYOND FINGER STRENGTH.

3) IF IN DOUBT - ASK FOR HELP.

Please note: This instruction can only be used by trained users as a reference. It is not intended to be the textbook on DIY purposes for the unauthorized personnel to use the machine without training and direct supervision.

ANFF@ANU

Front note

- The right hand side computer is used to run operating software of Helios, which should be setup and ready to use
- The left hand side computer has the software for Genesis EDS system and TSL EBSD data collection and analysis system
- The computer in the middle is the node connected to the ANFF server. All the data collected on Helios are saved in local director "SharedData/users" at first, and then uploaded to the server through the node by the user
- Usage will be monitored with both login information and system log files

Task 1: Start a FIB session

- Check list:
- > The sample size is suitable for the machine
- > The sample is flat and locked onto a pin stub
- The sample is dry and clean
- > The surface is conductive and well earthed
- > The silver paint is fully solidified, if applied
- The sitemap is available for multiple samples mounted on the same stub
- Double click system control software
 - Advanced 197
- Login your user account

Click on Start UI

• If the user interface is hided off the scene on the right hand side computer, click on Show UI on the screen,

Start	Stop	Show UI	Stop UI	×
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Task 2: Load sample and get E beam image

- Vent the chamber
- Go to page 1, ^{*}
- Click on vent and confirm the action in pop-up window
- Wait for ~ 4 min, then try to open the chamber by pulling the handle bar gently
- Lock the stub onto the stage with the Allen key, and check the overall height with the gauge, make sure the height lower than the max line.
- Pump the chamber
- Gently close the door
- Click on Pump
- Wait for ~ 5 min, until the vacuum icon ¹ turn green ¹
- Select both electron and ion beam images in serial, and click on Beam On for both columns. If the ion source is not "green", click on Wake Up .



- Locate region of interest (ROI)
- Select E beam image, e.g. top left quad window
- Click on pause button to start E beam scan
- Reduce the magnification to <100x, and have an overview of whole sample</p>
- Align the working area if necessary
- > Locate a small feature, e.g. dust, near ROI as the reference
- Achieve a focused image
- Link Z to WD
- Go to page 2,
- > Click on the link icon $\boxed{\mathbf{A}}$ on the top of the window

Task 3: Setup Eucentric height and beam coincidence

- Standard working conditions are all based on the Eucentric height (~4.0 mm)
- Pt deposition can only be done at the Eucentric height



- Move stage upwards
 - Increase magnification of E beam image to above 1000x
 - Achieve a fine focus and update Z measurement by click on the icon
 - Input Z of 8 mm, check focus and update Z measurement with the button
 - > Input Z of 6 mm, check focus and update Z with
 - Input Z of 4.0 mm, check focus and update Z with
- Title stage
- Select "Zero beam shift" from the drop down menu of "stage" for both E beam and I beam image respectively
- > Set a small feature in the centre cross of the E beam image.
- Input title, T of 15 degrees.
- Make the feature back to the centre with Z adjustment by the mouse.
- Input T of 30 degrees, and re-adjust Z to centre the feature.
- ➢ Input T of 52 degrees, and re-adjust Z, if necessary.
- Setup beam coincidence
- Centre a reference feature on E beam image at 3500×
- Select I beam image in another window, e.g. top right quad window
- Check if the I beam current is small, then get a snapshot with the icon
- Centre the feature with beam shift knobs if necessary, and update the image with snapshot

Task 4: Cover ROI with Pt deposition

- Select an appropriate ion beam current
- > 2-6 pA/um² is recommended for general application
- Update the image after 10 seconds if changing the aperture sleep time to stabilise the beam
- Insert GIS needle
- Make sure that eucentric height has been set up correctly before doing this!
- * Make sure there is nothing higher than ROI nearby!!
- Go to page 3,
- Locate the "Pt dep" line, double click on the word "cold" in "Heat" column
- Wait until the yellow timing bar turn to the word "Warm"



- Insert the needle by tick the box "in"
- Confirm the action within the pop up window by click "Yes"



- Define a pattern
- Select rectangular pattern
- Draw a box on the I beam image
 - Green color box for deposition
 - Yellow color box for milling
- Change the Application to "Pt dep"
- Start/stop deposition
- Click on the triangle button
 - to start deposition
- The same set of buttons will allow the user to pause and stop
- When finish, remove the tick within the box under "in" to retreat the GIS needle



Task 5: Mill cross section and record image

- Select an appropriate ion beam current
- > 6.5 21nA are recommended for rough milling
- For the same pattern, smaller beam current leads to sharper images but in longer milling time
- Define a pattern

 Select/activate "Regular cross section" pattern for rough milling
Draw a box on the ion beam image

 Confirm Application file as "Si"
Other material files are also available in the click/drop-down menu



- Update parameters and location of the pattern if necessary
- Start/stop milling process
- The same set of buttons will allow the user to pause and stop
- Clean surface
- > Select a smaller ion beam current, 9.7 93 pA are recommended
- Update the image
- Select/activate "Cleaning cross section" pattern
- Draw a narrow box on the ion beam image
- > Update parameters and location of the pattern if necessary
- The same set of buttons will allow the user to pause and stop
- Monitor the progress with snapshot
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- Take a photo
- Select/activate the quad desired E beam imaging is in common, but I beam imaging may show extra information for certain materials, such as channelling contrast for polycrystalline metals
- Check magnification, location and fine focus if necessary
- Press F2 -- pre-set scan speed for photo can be modified within Preferences/Scanning

Task 6: End a session

- Return to Page 1,
- Turn beam off by clicking on "beam on" for both E beam and lon beam
- Vent the chamber
- Click on Vent and confirm the action within the pop-up window
- Wait for ~4min, then try to open the chamber by pulling the handle bar gently
- Pump the empty chamber
- Gently close the door
- Click on Pump
- Wait for ~4min, the vacuum icon should turn green
- If you are the last user on the day, turn off the ion source
- Select/activate ion beam image
- Click on Sleep to turn off the ion source
- Logout your account

Advanced task: TEM lamella sample preparation

- AutoTEM G2 Software
- Preconditions:
- Stage Tilt: 52 °
- High voltage: 30 kV
- > Working distance: Eucentric (defined with E Column)
- Magnification: 1,000x
- Define the membrane
 - Start the AutoTEM G2 Software

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- Save the project
- > Select the following recipe '12x5 ex-situ lift-out'
- Verify the current parameters:
- Deselect 'skip fiducial milling'
- Select 'Mill Fiducials' to create fiducials for image recognition.
- > Select 'Refine' to enable accurate lamella positioning
- Select 'Finish' closing the recipe definition process.
- Run the sites
 - > Press the 'Run' button in the position list window.

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Advanced task: 3D imaging – Auto Slice & View

- Setup Eucentric height
- Locate ROI and cover it with Pt deposition (with the ion beam current of 2-6pA/um²) defined a thickness less than 0.5um (recommended)
- Make a regular cross section with high beam current, e.g. 9.3 21nA, at the leading edge of the ROI a bit deeper and wider than ROI
- Make trenches on both sides of ROI to avoid shading on images
- Clean up the side edges as the reference for 3D reconstruction
- Select an appropriate beam current for ion beam image, e.g. 93pA or higher
- Using cleaning cross section to clean the viewing area
- Tick the tilt correction in automatic mode
- Hide UI and create a folder in SharedData to store the image files
- Start AutoSlice&View G2 (click on the icon on the desktop)
- Direct image save location to the predefined folder, and change the prefix for data set



• Setup E beam image scan parameter, e.g. 9.05s

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• Select Focus Adjust from Setup menu

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- Beam Shift for Image Centre is optional
- Select E beam image within the same quad of I beam image by clicking

the icon 🗱 🏶 🖓 on the top left corner of the screen

• Optional step - If high resolution imaging is desired, switch to mode 2 with drop down menu

> a range of working conditions including limited kV, nA and WD required to activate mode 2



- From the menu bar, select stage/Zero beam shift
- Double click on the image to centre the viewing area for E beam image
- Return to I beam image, and use beam shift knobs to centre the ROI
- Input parameters for the slice (without Enter)
- Click on Show to see the overlay of the pattern on the ion beam image
- Modify the location with beam shift – do not move the patterns
- Capture/print screen or record important parameters for 3D reconstruction
- HFW for the final E beam image
- Image resolution for E beam image
- \succ Slice length y
- > Slice number



- Click on Run and work out the total time
- If there is a pop-up warning for over limitation of image shift, reduce the magnification of E beam image and retry
- If everything is OK, go home!

• Relationship of the Two Columns



PLEASE NOT REMOVE THIS INSTRUCTION FROM THE MACHINE ANYTIME!