

# FIB Standard Operating Procedure:

## Basic FIB Operation



**Yale** WEST CAMPUS  
*Material Characterization Core*  
[ywcmatsci.yale.edu](http://ywcmatsci.yale.edu)

ESC II, Room A119F  
810 West Campus Drive  
West Haven, CT 06516

Version 1.03, January 2023



**Core Policy:**

- > **FOLLOW** the SOP strictly to keep the instrument in good condition. **No** explorations allowed on software unless permitted by lab manager
- > **NEVER** use your own USB drive on instrument computer. Data can be either uploaded to Yale Box, or copied to the USB Drive provided by the Core.
- > **NEVER** surf the web on the XPS computer to minimize the risk of the computer being hacked
- > Users should **acknowledge** MCC in their publications. Please check the following link for details:  
*<http://ywcmatsci.yale.edu/publications>*
- > The core reserves the right to use the data for core promotion



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# FEI Helios G4 Dual Beam Standard Operating Procedure

## 1 Introduction

### 1) Instrument features:

- > High quality ultra-thin TEM sample preparation: automated sample preparation using AutoTEM allows multiple lamella preparation for final polishing.
- > Slice and view: automated sequential mill and view to collect series of slices images. Allow sample 3D reconstruction for further analysis such as 3D segmentation.
- > EDS: allows simultaneous EDS mapping for 3D reconstruction
- > EBSD: provides grain orientation maps, grain boundary maps, phase maps and pole figures.
- > FIB patterning: creates small structures with feature size down to tens of nanometers via 3D ion milling and deposition.
- > E-beam lithography: e-beam writing on resist coated surface for device fabrication with feature size smaller than 10 nm.

### 2) Location

Materials Characterization Core  
Room A119G  
810 West Campus Drive  
West Haven, CT 06516

### 3) Primary Staff Contact

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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

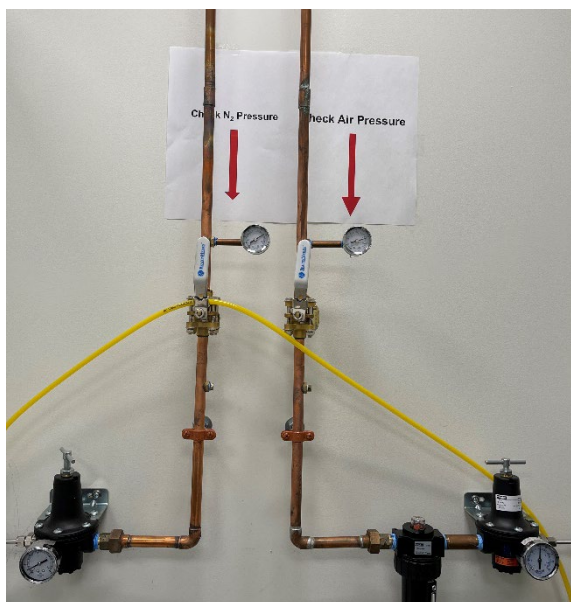
**Notice:** Please **follow** strictly the **SOP** to keep the facility under good condition. **No** explorations on program allowed unless approved by core manager.

## 2 Initial System Status Check

**Warning:** Before sample preparation and loading, user should check the system status following the steps listed below:

- 1) Check the building supply **N<sub>2</sub>** and **Compressed Air (CA)** pressure gauges as labelled on the back wall and make sure the pressure reads **~100 psi** on both gauges. Report to manager immediately if any of those gauges reads **zero**.

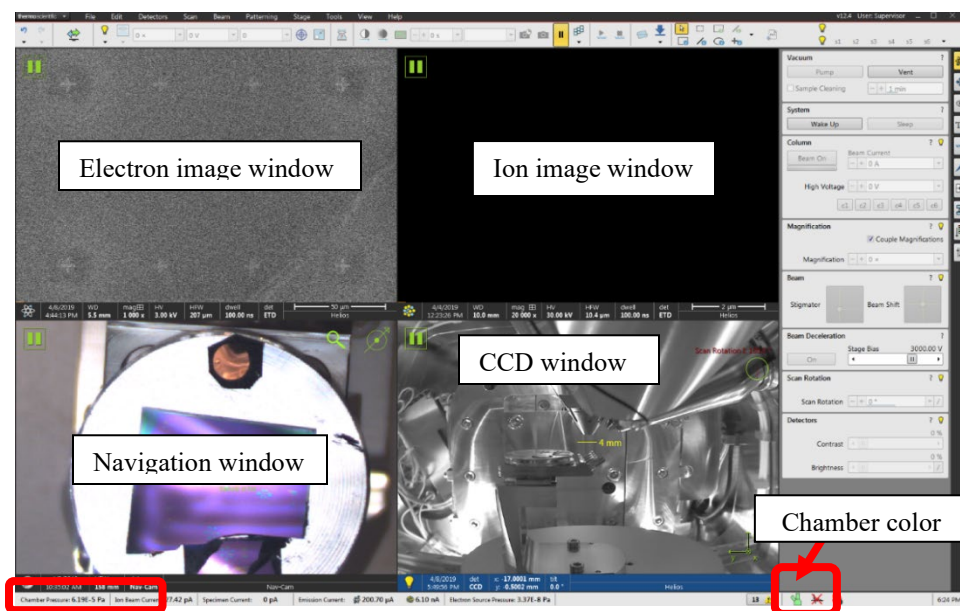
**Warning:** Do not adjust the regulators on top of those gauges.



- 2) **Log into** the FIB computer using **FOM calendar** or **FOM Screen Lock** window below by clicking **Click here to login with NetID**:



- 3) Sign in on the logbook and fill required info. The **chamber pressure** can be found on the **bottom left** corner of the **Microscope Control XT** window as highlighted below.

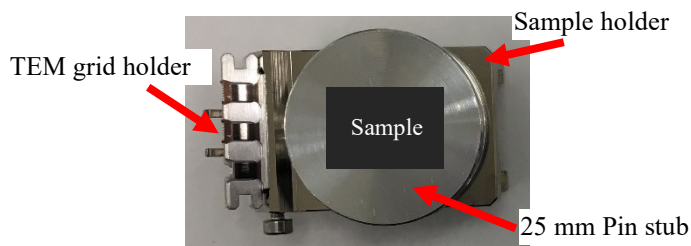


- 4) Check the **bottom right** corner of XT window above and make sure the chamber color is indicated in **green** and below **1.5E-4 Pa**. **DO NOT** proceed if the chamber color is in **orange**. This may indicate chamber leaking or outgassing. **Report** to manager immediately.
- 5) Check inside the CCD window (the bottom right quarter of above window) to **make sure no samples inside the chamber**.

### 3 Sample preparation

**Note:** the steps below are **ONLY** for sample transfer using the **Quick Loader**. For large sample loading which needs to vent the main chamber please see separate SOP.

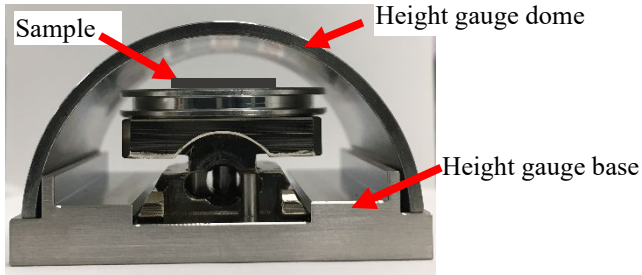
- 1) **Always wear gloves** for vacuum sample preparation!! **Change gloves** if touched computer keyboard and mouse.
- 2) The sample for FIB should be completely **dried** before mounting.
- 3) The sample can be fixed using double sided carbon tape directly onto the pin stub as shown below:



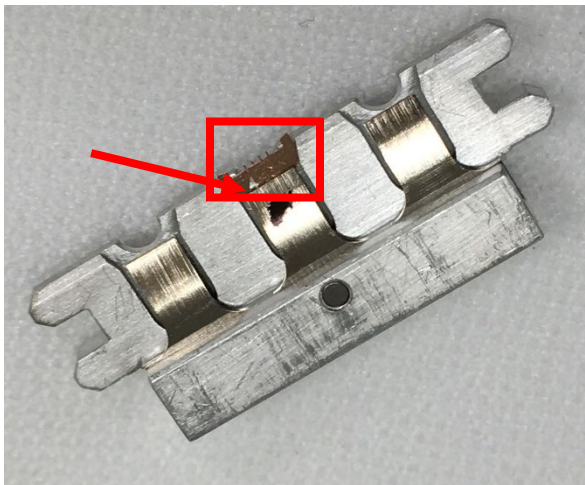
- 4) Insert the pin stub into the hole in the middle of the sample holder and fix the stub by turning the set screw on the side of the holder. **Warning: Hand Tight** using the Ellen key screwdriver. **DO NOT overtighten** the set screw to damage the thread.

- 5) Sample height alignment: place the sample holder into the gauge as shown below and **make sure** the sample is **not touching** the inner wall of the height gauge dome.

**Warning:** misaligned/misplaced sample will hit the electron column and cause severe damage! Charges will be applied on your PI's account.




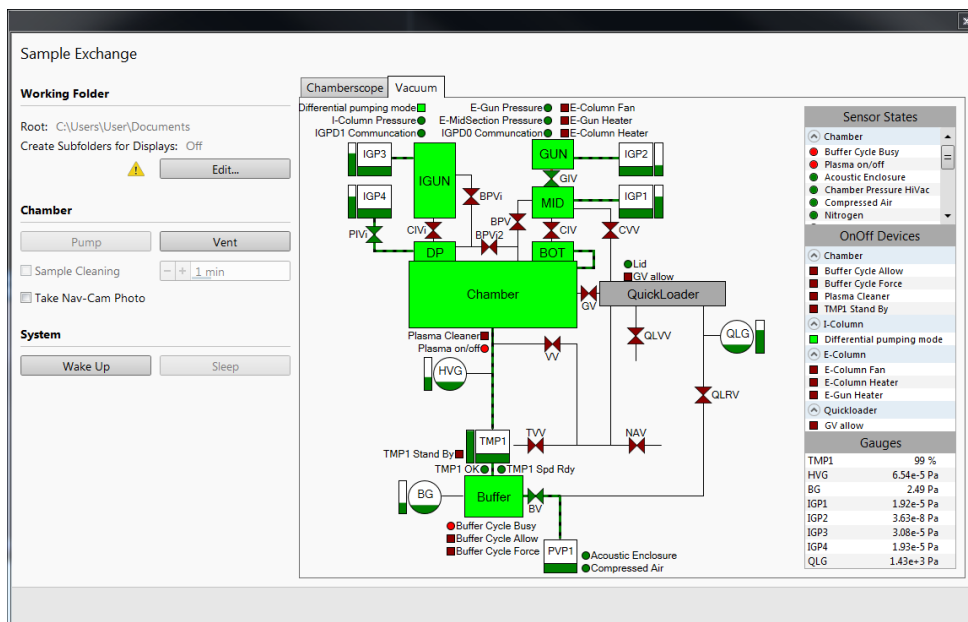
- 6) **Clean sample holder:** bring the specimen stub inside the fume hood and **blow off** loose particles on the sample surface (**not on the grid**) using the **N<sub>2</sub> gun**.
- 7) For TEM users, make sure the grid was mounted correctly on the holder with the number **“4”** on the left side of the grid as shown below. **Do not** blow grid with nitrogen.




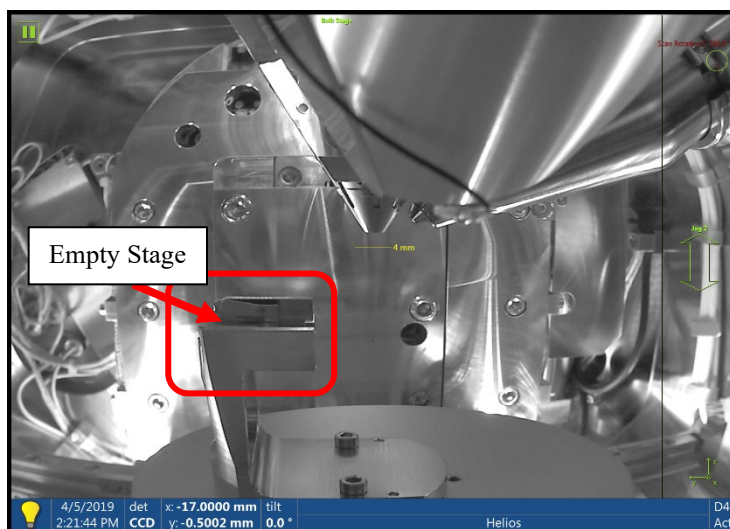
#### 4 Sample Loading through Quick Loader


**Note:** if not familiar with sample loading steps, read the entire section before start.

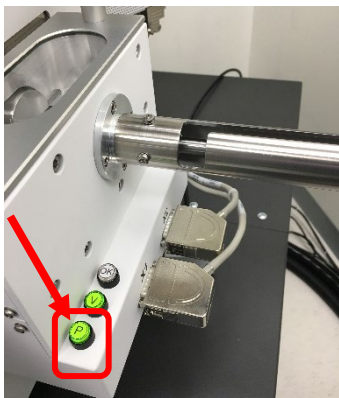
- 1) Click  button on XT window tool bar to open the **Sample Exchange** window below:



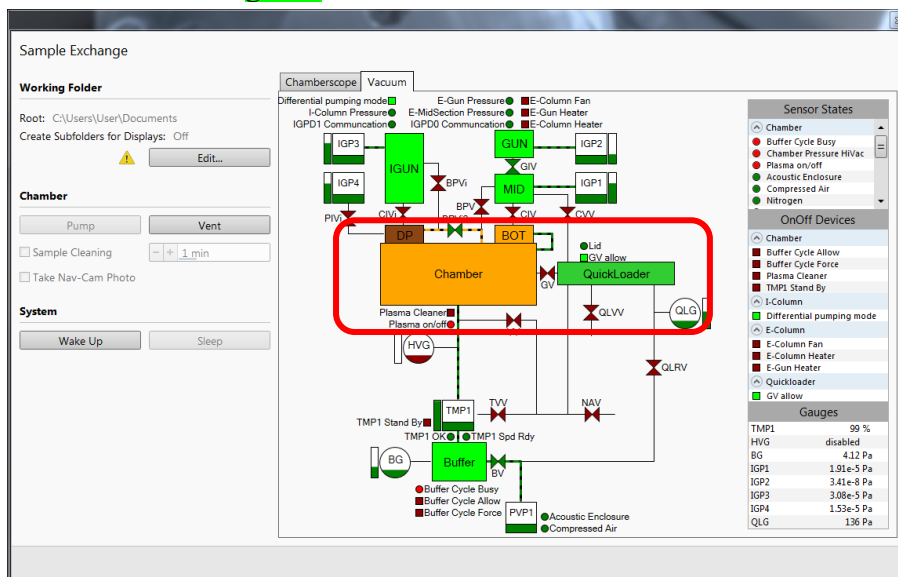
- 2) Click  inside **CCD** window (bottom right window) to unpause/activate **CCD** view inside main chamber.
- 3) Click top menu **View > Large Image Window** to amplify the **CCD** window onto the computer monitor on the right side. **Make sure** there is no sample holder on the stage.




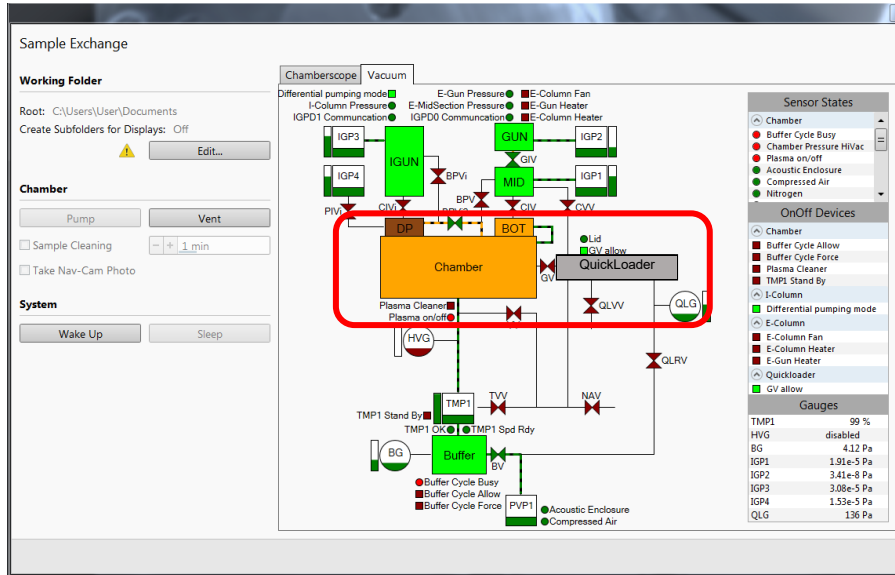
- 4) **Put on gloves.** Press and hold  button for **one second** on **Quick Loader** as shown below:



- 5) Watch the **Sample Exchange** window till the **Chamber** color changes to **orange** and **Quick Loader** to **green** as shown below:

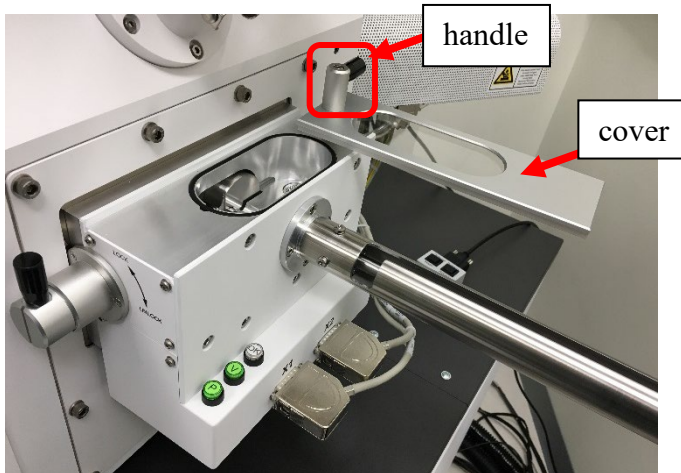


- 6) As soon as the **Quick Loader** turns into **green** while **Chamber** stays in **orange**, press and hold  button for one second on **Quick Loader** and wait to hear an air sucking sound, indicating the **Quick Loader** is being vented.
- 7) Watch the Quick Loader changing into **dark grey** as shown below, indicating **Quick Loader** venting is finished.

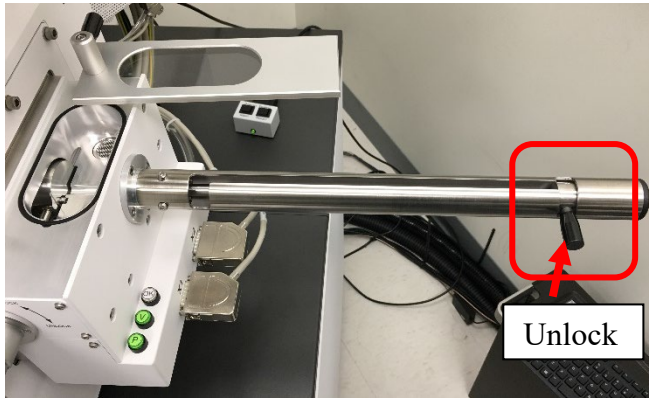


8) Grab the handle on top of the **Quick Loader** and **SLOWLY** lift the cover upward and turn it **90 degrees** to expose loading area as shown below.

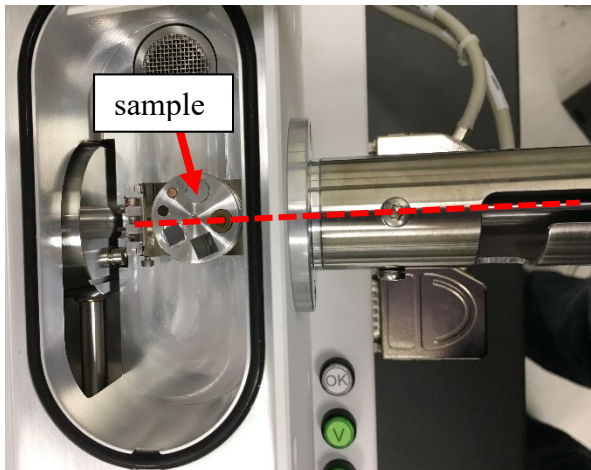
**Warning:** **Never force to break the vacuum** if the **Quick Loader** is not fully vented.



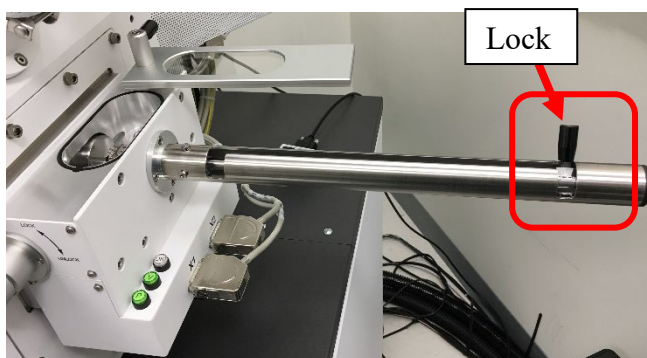
- 9) Pull the handle on the transfer rod downward to **Unlock** position as shown below:



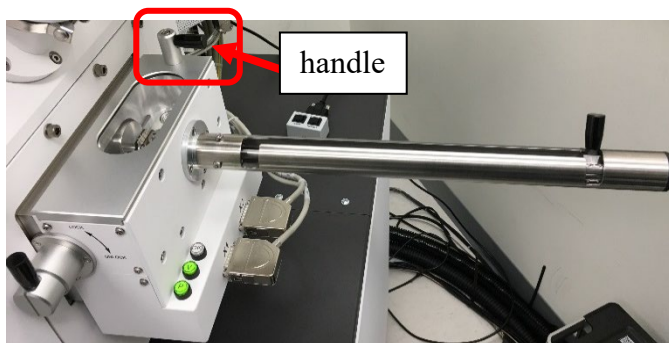
- 10) Pull the rod backward and meanwhile place the sample holder inside **Quick Loader** and align with the transfer rod as shown below:



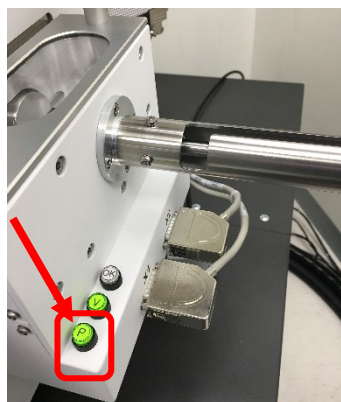
- 11) **Left hand** holds and pushes the sample holder into the transfer rod meanwhile **right hand SLOWLY** turning the **Quick Loader** handle upward to the **Lock** position as shown below. **Never force the handle to break parts.**



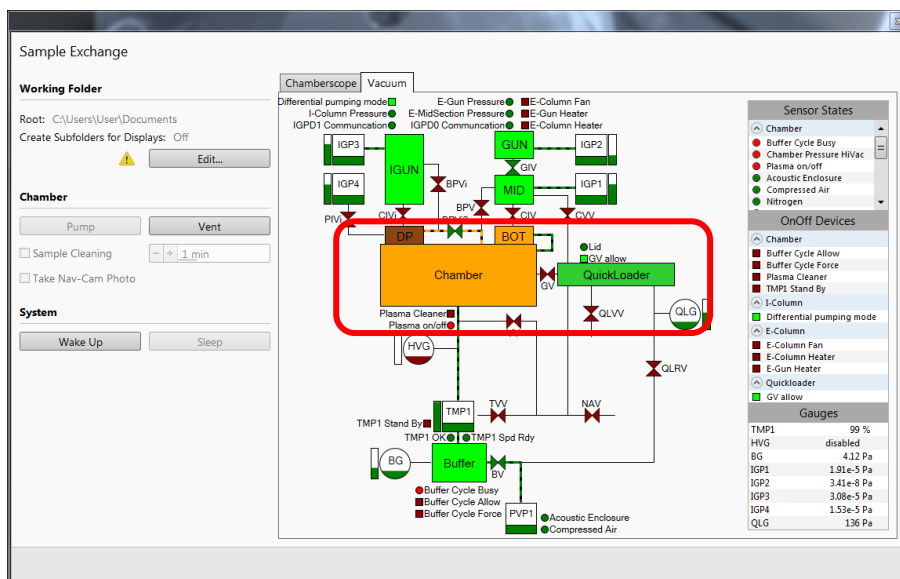
- 12) Rotate the cover handle back and press down to seal the **Quick Loader** chamber as shown below:



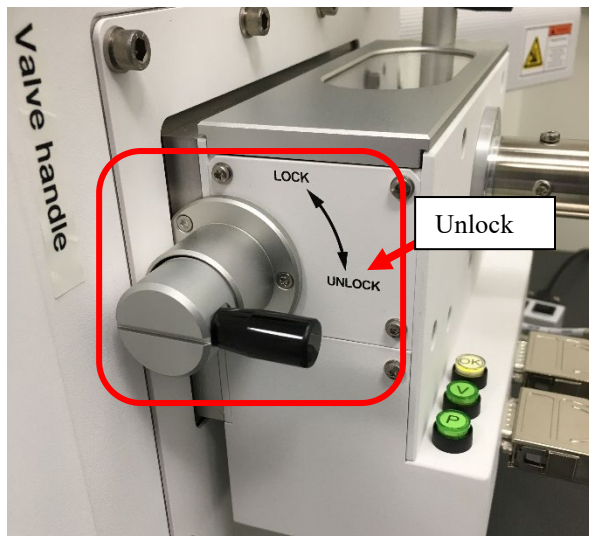
- 13) Press and hold  button for **one second** on **Quick Loader** as shown below:



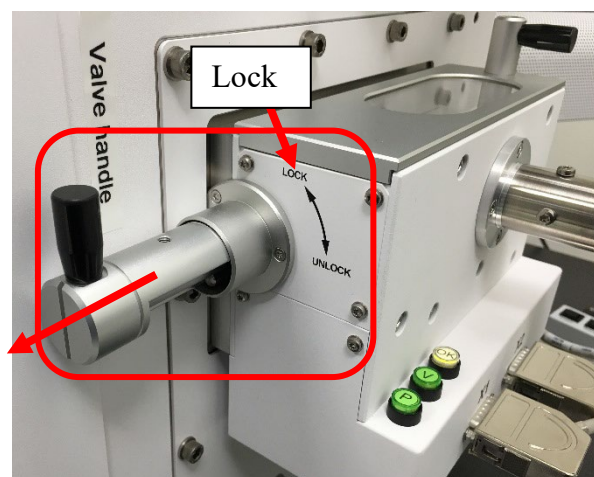
- 14) Watch the **Sample Exchange** window till the **Chamber** color changes into **orange** and **Quick Loader** in **green** as shown below:



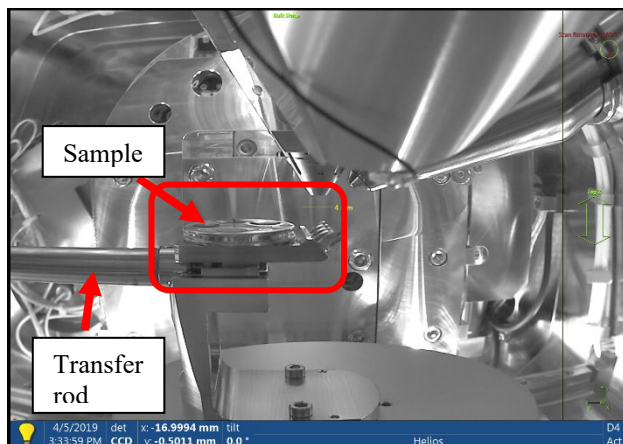
- 15) As soon as the **Quick Loader** changes into **green** while **Chamber** stays in **orange** turn the **Quick Loader Valve handle** to **UNLOCK** position as shown below. Hold the handle for next step.



- 16) Pull the **Valve handle** outward to open the valve and turn the handle back to **LOCK** position as shown below. The valve should be kept open for sample transfer.



- 17) Rotate and align the **Quick Loader** handle with the track on top of the transfer rod between **LOCK** and **Unlock** position and **SLOWLY** push the transfer rod toward the main chamber. Meanwhile watch the **active** **CCD** window when the sample holder is approaching the stage as shown below. **Note:** make sure the CCD camera is active.

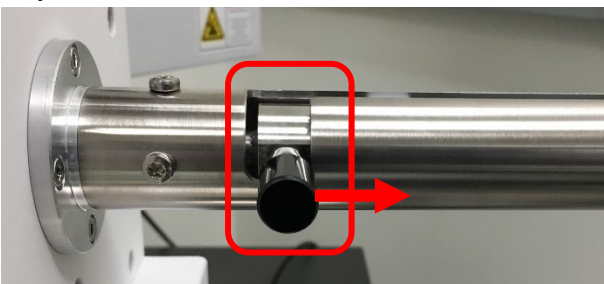


- 18) When the transfer rod is **stopped** rotate the transfer rod handle **SLOWLY downward** into the slot until **stopped** as shown below:

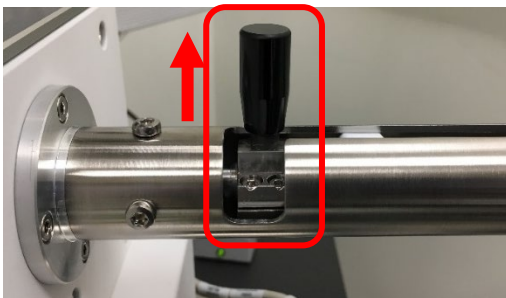


- 19) Pull **VERY SLOWLY** the handle **backward inside** the slot as shown below and meanwhile **watch** the transfer rod leaving the sample holder in the **active** CCD window. **Stop** retracting if the sample holder is being pulled out.

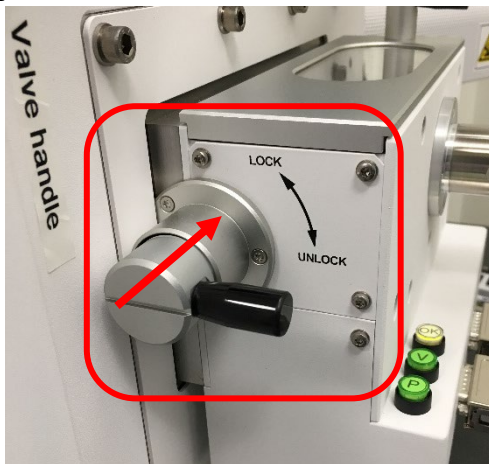
**Warning:** **Never force the handle to damage parts.** Report to manager immediately for any issues.



- 20) **Hold** the handle and **SLOWLY** turn **upward** as shown below and then pull the transfer rod all the way to the end and turn **upward** to the **LOCK** position.

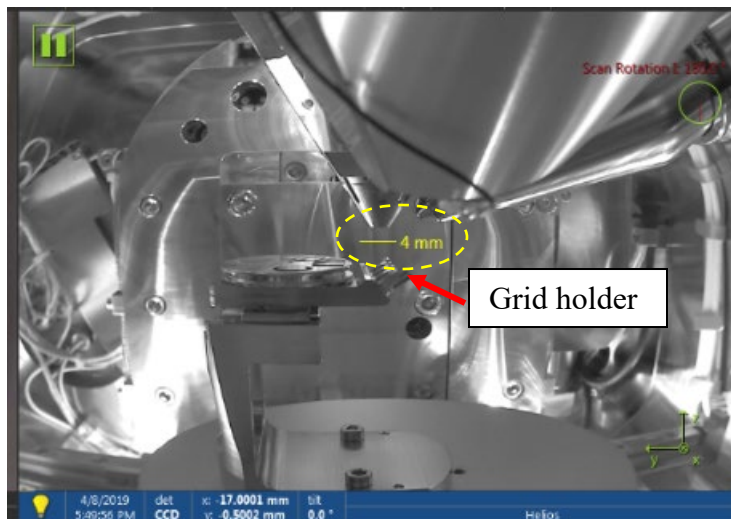


- 21) Turn the **Valve handle** downward to **Unlock** position and push the valve into sealing position as shown below, and turn the handle upward to **Lock** position.

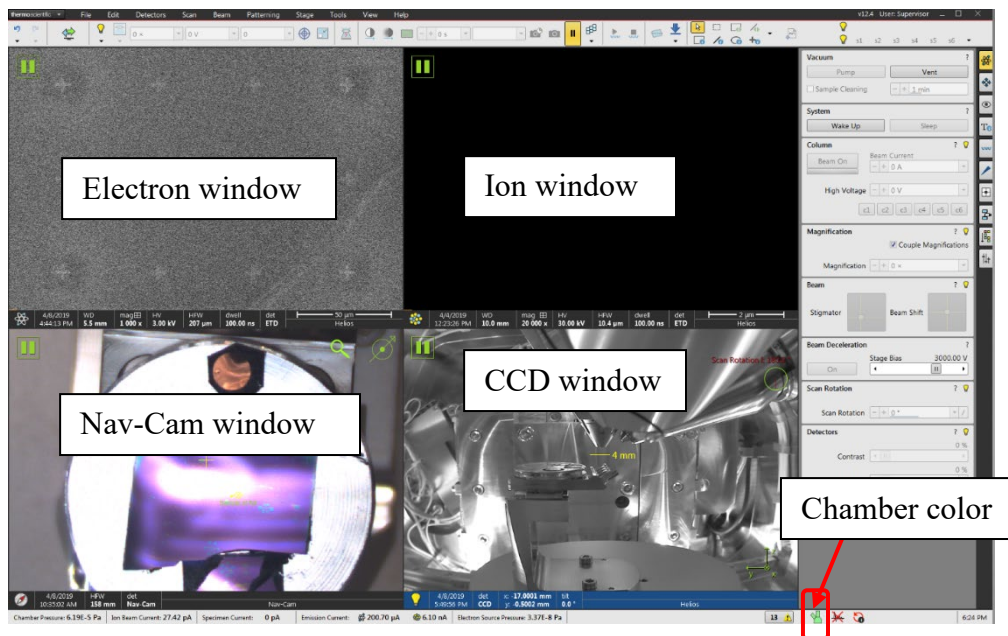


- 22) Check the **CCD** window below to **make sure**:


- > The sample surface is below the **yellow 4 mm line** underneath electron column. **Report to manager immediately** if the sample surface is above the yellow line to avoid sample crashing into the electron column.
- > The TEM grid holder is on **the right side** of the sample:

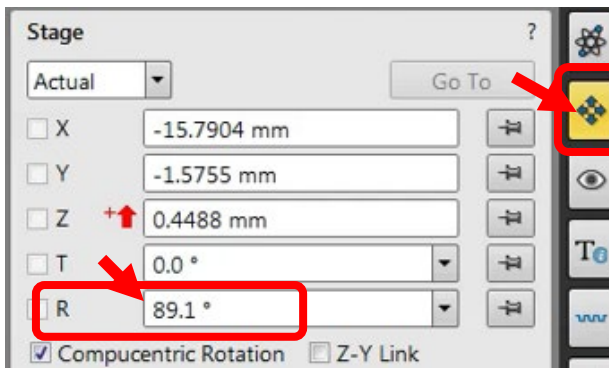


- 23) **Watch and wait** the Chamber color changes into **green** at the bottom right corner of the **XT** window as shown below and the pressure below **1.5E-4 Pa** from the bottom left corner.




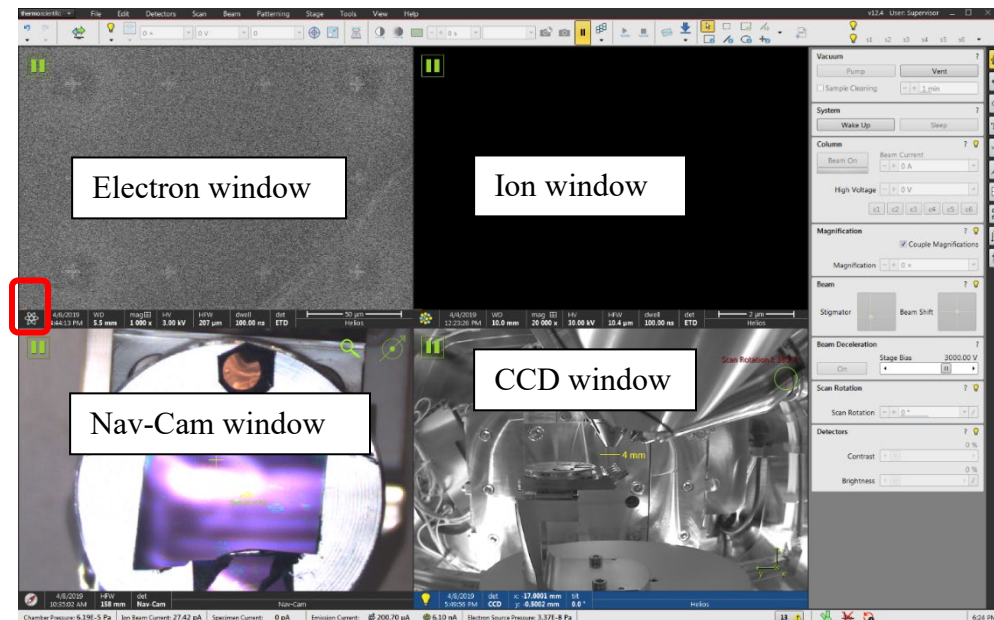
## 5 Sample Z link to Working Distance

- 1) Click on  Navigation button on the right side of the **XT** window and **make sure** the Rotation angle is around  $\sim 90.0^\circ$  as shown below, and the X, Y and Z coordinates right after sample loading should also be close to the numbers below.

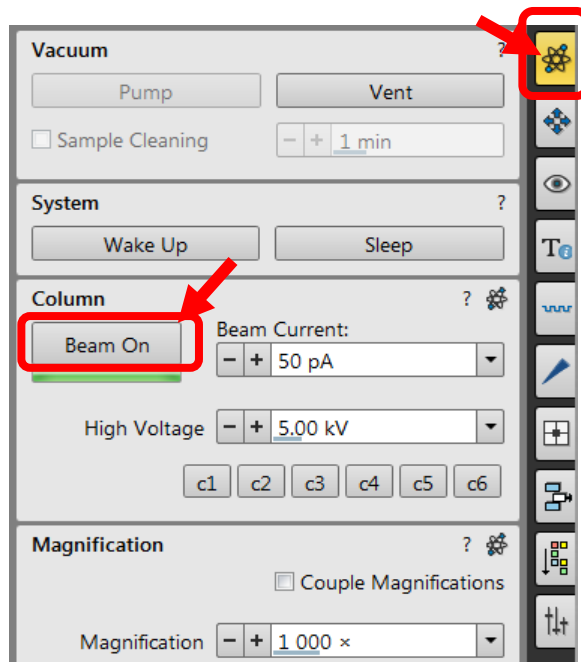


- 2) **For electron beam use only** (SEM, EBL, EDS and EBSD):

- a) Click on the top left electron imaging window below with an  icon on its bottom left corner to switch **Beam control** to electron beam.



- b) Click on  in **Column** control below to turn on the electron beam.

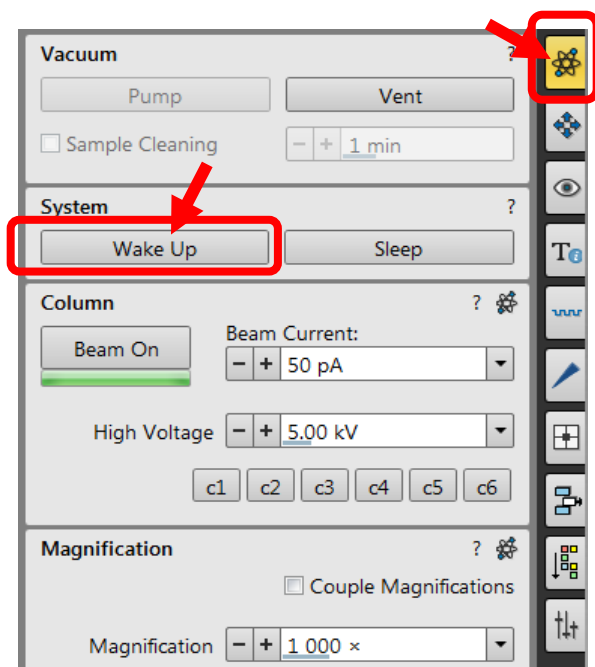


- 3) **For ion beam users** (TEM sample preparation, cross section, ion beam patterning): click

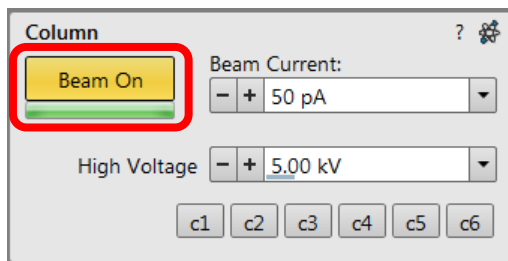


button on the top right corner of **XT** program window to enter **Beam control** window

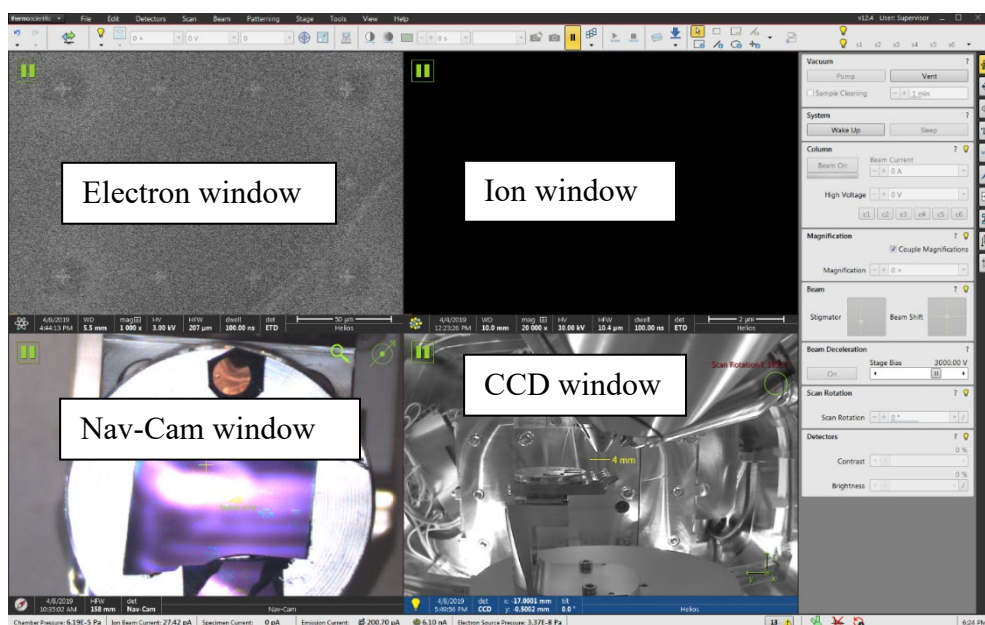
as shown below and click  to turn on both electron and ion beams.







- 4) It takes ~ **5 minutes** to warm up ion beam source (liquid gallium). Wait till the **Beam On** button turns into **orange** in **Column** control below:

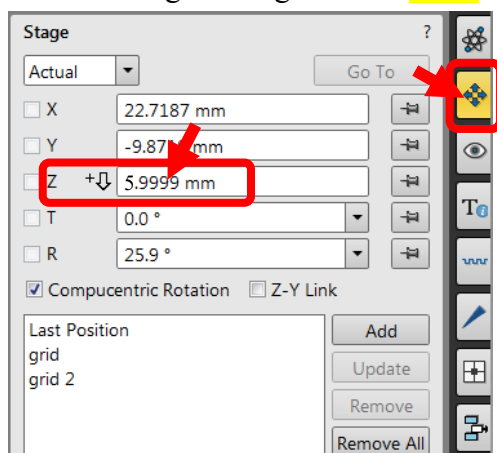




- 5) While the ion source is being warmed up, click **Stage** on top menu and select **Take-Nav Cam Photo** to take a sample picture in the bottom left Nav-Cam window:

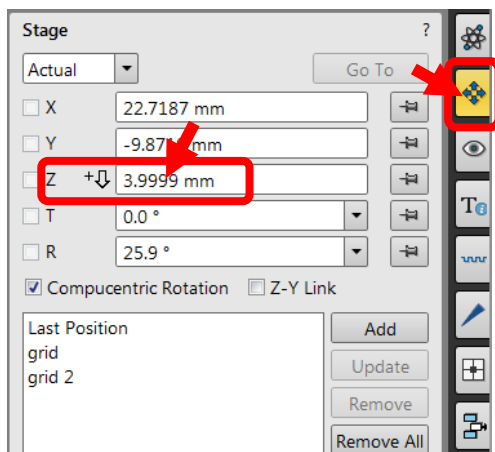


- 6) Wait until a **sharp** sample holder photo is taken and the stage is moved to the transfer position. **Warning: DO NOT** interrupt the photo taken process!
- 7) Check to make sure the sample surface is below the yellow **4 mm line**. **If not, report to manager immediately**. **Warning: DO NOT** proceed to cause sample holder crashing into the electron column.
- 8) Click  inside **electron** window (top left corner) to activate electron imaging.
- 9) Go back to **Nav-Cam** window and **Double Click** on the sample surface to drive stage to selected area. Zoom in and adjust focus, stigma to get a well-focused image. **Note:** try to avoid features too high above the surface.
- 10) **Stop at the step above for e-beam lithography users**, no need to do the Z-link blow.
- 11) Click  **Link Z to FWD** button on top tool bar to link **Stage Z** to working distance. The **Link Z to FWD** button changes into  once linked.

- 12) Click on  Navigation button on the right side of the **XT** window and check to make sure the **Z** height changes into ~ **6mm** by default as shown below:



- 13) **For FIB users only:** click on  Navigation button on the right side of the **XT** window and **input 4 and hit Enter key** on the keyboard to move the stage to 4 mm. Make sure the  appears next to the number, indicating increasing number brings the stage down.

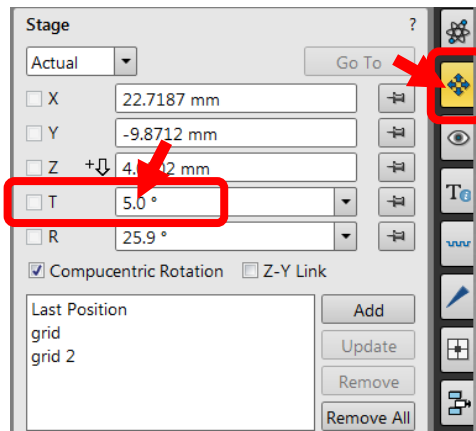


- 14) **For FIB users only.** Eucentric tilt adjustment:

- a) Double click on a reference feature on the sample to center the in the image indicated by a hair cross as shown below:



- b) **Input 5** and hit **Enter** key in **T** (tilt) space below:



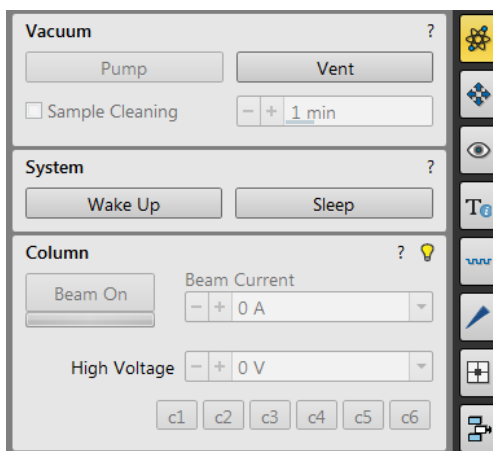
- c) Click **Up** or **Down** arrow in **Stage Z** control below or drag the slider square **SLOWLY** to change stage height and bring the reference feature back to the center of the image.



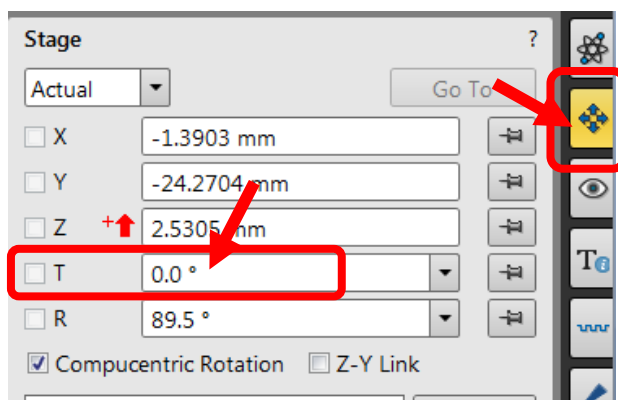
- d) Tilt stage to **20°** and finally to **52°** and repeat Step c) above at each angle to center the reference feature as shown in step a) above.

## 6 Closing measurement

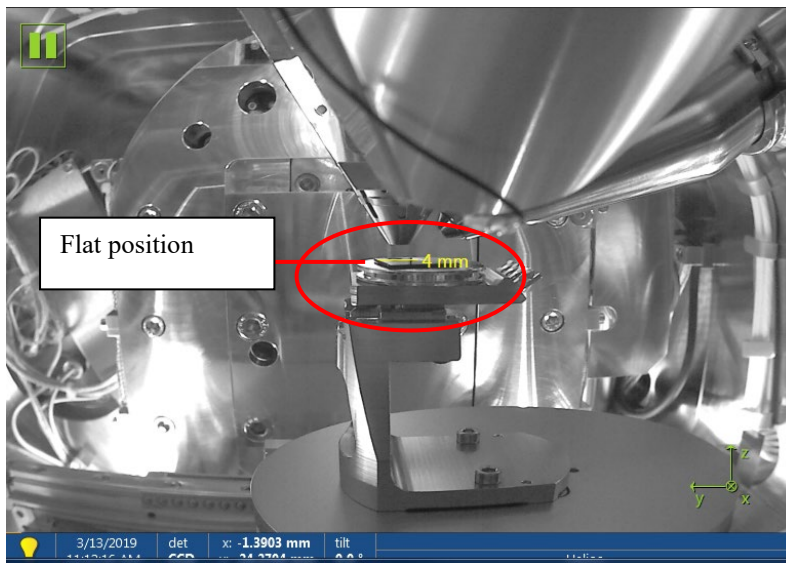
- 1) Click  in **Beam Control** > **System** panel below to **turn both electron and ion beam off**.




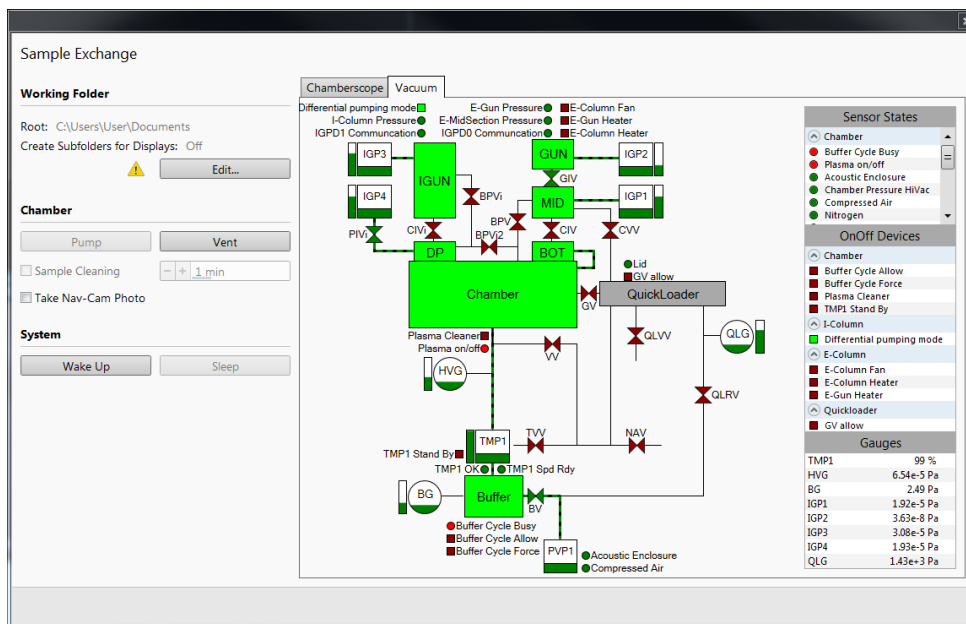
- 2) Tilt the sample stage down to  $0^\circ$  in **Navigation** > **Stage** control panel in the highlighted area below:



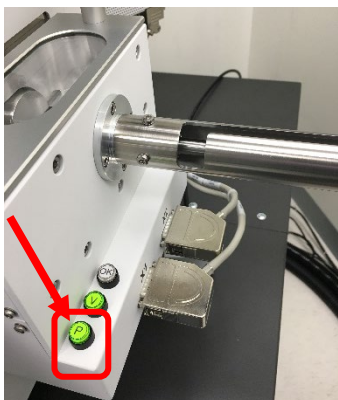
- 3) Activate **CCD** window and check the stage being tilt back to flat position as show below:  
**Note:** the **CCD** window was set to **OFF mode in every 10 minutes** to protect EDS and EBSD detectors.



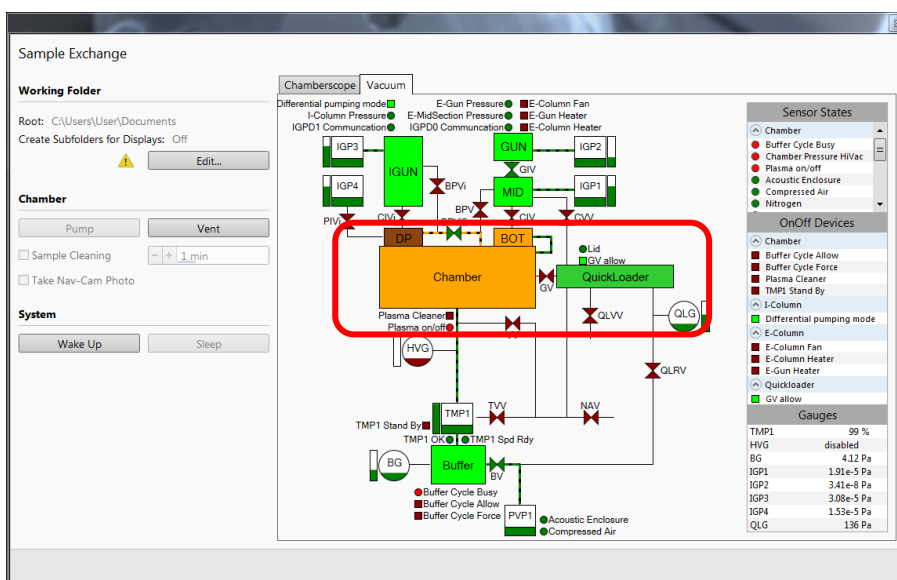
4) Click  button on tool bar to open the **Sample Exchange** window below:



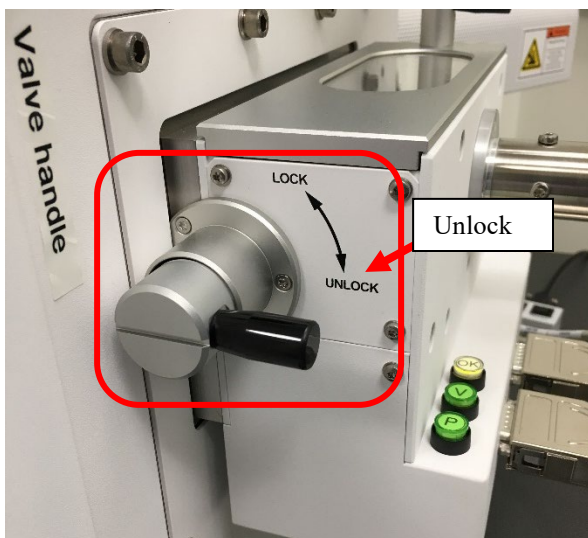
- 5) Press and hold  button for **one second** on **Quick Loader** as shown below:



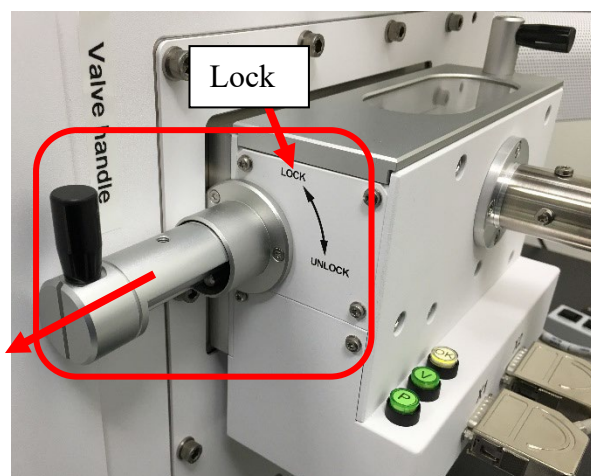
- 6) Watch the **Sample Exchange** window till the **Chamber** color changes into **orange** and **Quick Loader** in **green** as shown below:



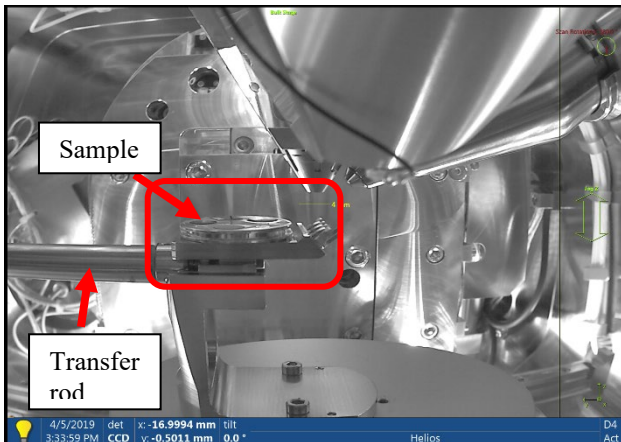
- 7) As soon as the **Quick Loader** changes into **green** while **Chamber** stays in **orange** turn the **Quick Loader Valve handle** to **UNLOCK** position as shown below. Hold the handle for next step.



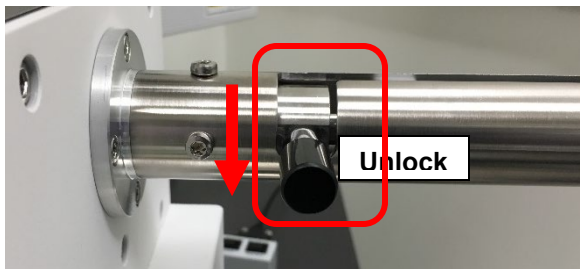
- 8) Pull the **Valve handle** outward to open the valve and turn the handle back to **LOCK** position as shown below. The valve should be kept open for sample transfer.



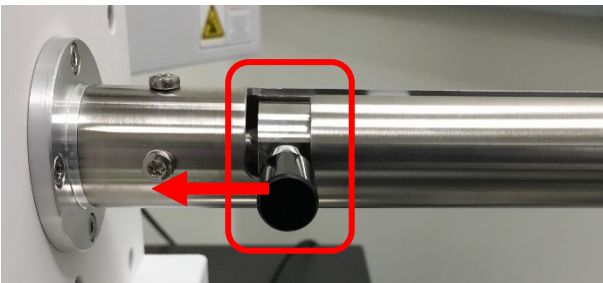
- 9) Make sure the CCD camera is active.
- 10) Rotate and align the **Quick Loader** handle with the track on top of the transfer rod between **LOCK** and **Unlock** position and **SLOWLY** push the transfer rod toward the main chamber. Meanwhile watch the **active** CCD window when the sample holder is approaching the stage as shown below.



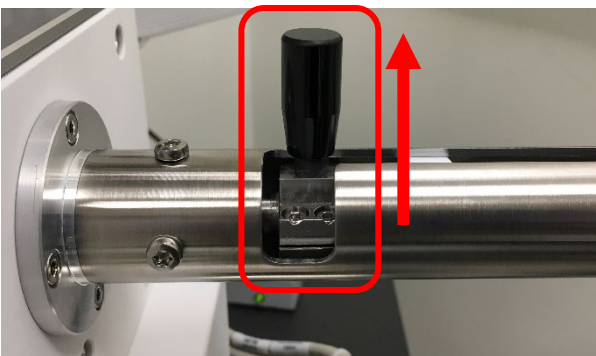
- 11) When the transfer rod is **stopped** rotate the transfer rod handle **SLOWLY downward** into the slot until **stopped** as shown below:



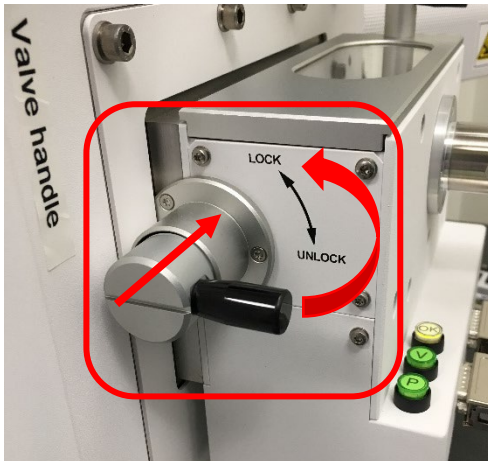
- 12) When the transfer rod handle is turned down to **Unlock** position, **VERY SLOWLY** push the handle **forward** in the slot as shown below:



- 13) **Hold** the handle and **SLOWLY** turn **upward** as shown below and pull the transfer rod all the way to the end and turn **upward** to the **LOCK** position.



- 14) Turn the **Valve handle** downward to **Unlock** position and push the valve into sealing position as shown below, and turn the handle upward to **Lock** position.



- 15) Follow steps in **Section 4 Sample Loading through Quick Loader** on page 4 to unload the sample holder from the Quick Loader and pump it back to vacuum.
- 16) **Wait** the **Chamber color** to reach **green** before log off **FOM**.
- 17) **DO NOT** close **XT** program
- 18) **Sign off** logbook.
- 19) Remove **ONLY** samples from the stub on the specimen holder and **clean** the holder with Kimwipes using **IPA**.
- 20) **Clear** the FIB work bench.