

# Model 1040 NanoMill<sup>®</sup>

## TEM specimen preparation system

*When not in use, the NanoMill<sup>®</sup> TEM specimen preparation system should be in vacuum status with the filament off. The NanoMill system is never turned off except for maintenance purposes. The base vacuum reading should be below  $\leq 3 \times 10^{-6}$ . The argon gas pressure should be 25 psi  $\pm$  5.*

### Stage cooling

1. If stage cooling is desired, verify that the base vacuum is in the low  $10^{-6}$  torr range or better before filling the dewar.
2. Fill the dewar with liquid nitrogen.
3. Wait 15 to 20 minutes for the stage to cool before milling and verify that the temperature is  $< -150$  °C for at least 10 minutes before proceeding.

### Prepare the specimen

1. Load the specimen into the NanoMill system specimen holder.
2. Plasma clean the loaded holder in the Fischione Model 1020 Plasma Cleaner or Model 1070 NanoClean for 5 minutes; if the specimen contains organic materials, plasma clean for 2 minutes.

### Insert the specimen holder

On the NanoMill system's **Main** tab:

1. Click Gate Valve **Close**.
2. Click Specimen Position **Exchange** to place the stage at 0° tilt angle.
3. Click Load Lock **Vent**.
4. Open the load lock and thread the specimen holder clockwise onto the end of the insertion rod.
5. Close the load lock.
6. Click Load Lock **Pump**.
7. Click Gate Valve **Open** (the gate valve will not open until load lock vacuum pressure is below  $10^{-1}$  torr).
8. Insert the rod until the specimen holder is engaged with the stage.
9. Turn the rod counterclockwise to disengage the specimen holder from the rod.

10. Fully retract the rod into the load lock; the rod must be fully retracted to prevent the gate valve from closing on the rod.
11. Click Gate Valve **Close**. A dialog box opens and asks you to confirm that the rod is fully retracted.
12. Confirm that the rod is fully retracted.
13. Click **OK** to close the dialog box.

### Set the Ion Source controls

1. Set the Ion Source Desired **Emission** current; a typical value is 150  $\mu$ A.
2. Set the Ion Source **Energy**; a typical value is 900 eV.
3. Click Filament **Start**.

### Set the Imaging controls

1. Set the Scan Speed to **Fast**.
2. Press Scanning **Start**.
3. Adjust the image. Use the Imaging:
  - **Contrast** control to adjust the image contrast.
  - **Brightness** control to adjust the image brightness.
  - **Focus** control to adjust the image focus.
  - **Magnification** control to adjust the image magnification.
4. Select Scan Speed **Slow**.

### Set the Targeting controls

1. Use the Targeting **Pan** arrows to center the specimen's region of interest in the image. (Press the center of the **Pan** control to reset the image to the original position.)  
Alternatively, click on the region of interest; the image will be centered at the location where you clicked.

2. Use the Specimen Position Tilt controls to adjust the tilt. The tilt range is  $-10^{\circ}$  to  $+30^{\circ}$ . If you intend to perform both positive and negative tilt milling, for best results do the positive tilt first.
3. Use the Specimen Position **Rotation** controls to change the specimen's rotation angle to align the specimen with the ion beam.
4. Set the Targeting mode to **Point** for point milling or **Area** for area (raster) milling. In most instances, use **Area** mode.
  - **Point** milling. Position the target using the mouse or by entering  $X_0$  and  $Y_0$  values.
  - **Area** milling. Position and size the raster box using the:
    - *Mouse*. Drag the raster box to the desired position. Drag a corner of the raster box to resize it.
    - *Value fields*. Enter the values for the top left corner of the raster box in the  $X_0$  and  $Y_0$  fields and the size of the raster box in the  $\Delta X$  and  $\Delta Y$  fields.

### Mill the specimen

1. Click Milling **Start**. A dialog box presents three milling options:
  - *Run set parameters indefinitely*  
If you select this option, the NanoMill system will perform milling operations according to the parameters you set until you click Milling **Stop**.
  - *Run set parameters with a timed endpoint*  
If you select this option, a timer control is displayed in the dialog box. Enter the milling time in hours, minutes, and seconds (hh:mm:ss).
  - *Run purge with a timed endpoint*  
If you select this option, a timer control is displayed in the dialog box. Enter the

purge time in hours, minutes, and seconds (hh:mm:ss).

2. Click **OK** to close the dialog box.  
The milling (or purging) operation begins. The image that was displayed when you started milling operations remains on screen. Status messages are displayed on the information bar at the bottom of the screen, including the elapsed milling time.

### Remove the specimen

1. After milling is complete, click Specimen Position **Exchange** to return the specimen to home position.
2. Click Gate Valve **Open**.
3. Insert the rod and then turn it clockwise to engage the specimen holder thread.
4. Retract the rod into the load lock.
5. Click Gate Valve **Close**. A dialog box opens and asks you to confirm that the rod is fully retracted.
6. Confirm that the rod is fully retracted.
7. Click **OK** to close the dialog box.
8. Click Load Lock **Vent**. If specimen is cold, leave it in the load lock for at least 15 minutes prior to venting to prevent condensation on the specimen.
9. Open the load lock door.
10. Remove the specimen holder from the rod by turning the rod counterclockwise.
11. Close the load lock door.
12. Click Load Lock **Pump**.
13. Transfer the specimen to the transmission electron microscope holder.

