APPLICATION NOTE

Semiconductor sample preparation for scanning electron microscopy

Proper mechanical preparation of samples for ion milling increases milling process speed and improves the quality of the resulting scanning electron microscope (SEM) images. Both cross section and plan view sample preparation is discussed.

Fischione Model 1060 SEM Mill is a state-of-the-art ion milling and polishing system. It is compact, precise, and consistently produces high-quality SEM samples.

This application note describes the preparation of semiconductor samples for ion milling. Ion milling is used in the physical sciences to enhance the sample's surface characteristics. Inert gas, typically argon, is ionized and then accelerated toward the sample surface. By means of momentum transfer, the impinging ions sputter material from the sample at a controlled rate.

Materials

- Fischione Model 1060 SEM Mill
- Low-speed saw with diamond wafering blade
- Grinder/polisher with rotating platen
- Allied MultiPrep™ System or TEM Wedge Polisher with Cross-Sectioning Paddle
- Hot plate
- Crystalbond™ adhesive
- Carbon tape
- Heat-cured epoxy adhesive (EPO-TEK® 353ND)
- Silicon carbide abrasive disks (600, 800, and 1200 grit)
- Diamond lapping films (3 and 1 µm)
- Glass cover slip (150 µm thick)

For cross-section samples:

- Fischione Loading Station, part number 034-0351

Figure 1. Fischione Model 1060 SEM Mill offers tabletop precision sample preparation. Premium Edition model shown.

- Fischione SEM Cross-Section Holder, part number 041-0578
- Fischione SEM Cross-Section Holder Adapter Plate (included with the SEM Cross-Section Holder)

For plan view samples:

- Fischione Pin Stub Mount, part number 041-0558
- Fischione Stub Storage Mount, part number 500-2980
- Fischione Pin Stub Mount Gripper, part number 500-2959

Preparing SEM samples

Cross-section samples

To cut and polish a cross-section sample:

1. Either cleave or use a low-speed saw with diamond wafering blade to cut a sample that includes the area of interest.
2. To protect integrated circuits on the sample, use a heat-cure epoxy (e.g., EPO-TEK 353ND) to attach a glass cover slip to the circuitry side of the chip, if desired.
3. To locate the proper point of interest for milling and to reduce milling time, use Silicon carbide abrasive disks (600, 800, and 1200 grit) and diamond lapping films to mechanically polish the sample to 1 µm. In the case of a highly accurate cleave, polishing may not be necessary.

If you do polish the sample, use Crystalbond adhesive to attach the sample to a cross-sectioning paddle. To achieve a 90° edge on the cross section, polish the sample using one of the following methods:

- **Polishing with a stationary arm.** Mount the cross-sectioning paddle to a polisher with a stationary arm, such as the Allied MultiPrep System (Figure 2). Ensure that the stationary arm is properly aligned.
- **Polishing with the hand-held, tripod-type polisher.** Mount the cross-sectioning paddle to a hand-held, tripod-type polisher, such as the Allied TEM Wedge Polisher (Figure 3).

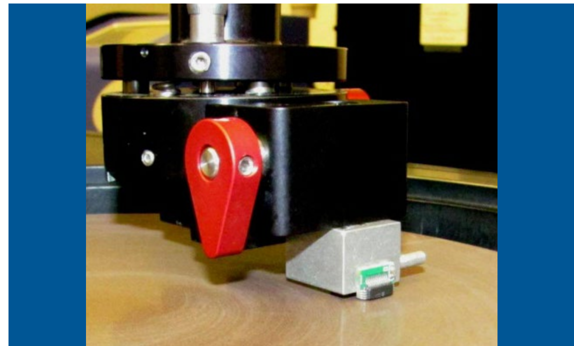

Figure 2. Sample mounted on a cross-sectioning paddle and then suspended on the polisher's arm.

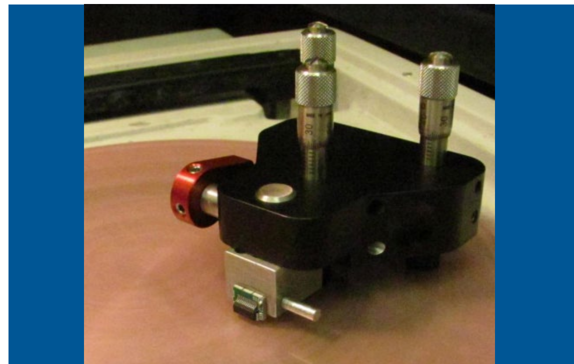

Figure 3. Sample mounted on a cross-sectioning paddle and then on a hand-held, tripod-type polisher.

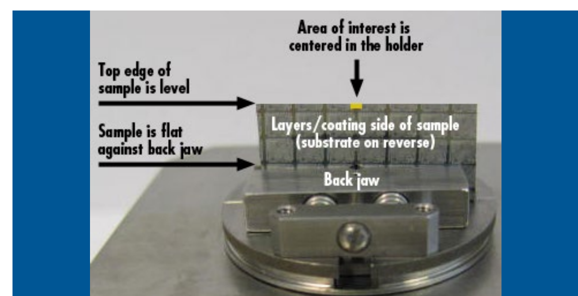

Figure 4. The top edge of the sample should be level and the sample should sit flat against the back jaw. The area of interest should be centered in the holder. The ion beam will be directed at the substrate side of the sample during milling operations.

Do not grind away the point of interest during this step.

To mount the sample on the SEM cross-section holder:

1. Place the SEM cross-section holder into the loading station. Follow the instructions, *Quick Guide: Mounting Cross Sections Using the SEM Cross-Section Holder* (document number 041-0647).

   Place the sample directly in the jaws of the holder (Figure 4). Be careful to align the point or feature of interest in the geometric center of the assembly. The sum of the adapter plate, holder, and sample height should not exceed ~ 15 mm total.
2. For better grounding and imaging, secure the cross section in place in the holder with double-sided carbon tape.

Plan view samples

To mount the sample on the pin stub mount:

1. Insert the pin stub mount into the stub storage mount.
2. Center the sample's area of interest over the cross hairs of the pin stub mount.
3. Adhere the sample to the stub with adhesive (e.g., Crystalbond) or double-sided carbon tape; keep the sample as flat as possible.

Performing ion milling operations

The SEM Mill is an excellent tool for removing surface artifacts from SEM samples leading to improved imaging and analysis.

In many instances, SEM samples are prepared by metallographic polishing. Often, an unwanted topography remains. With today's advanced SEMs, even a minimal amount of damage may limit the ability to fully resolve or analyze the sample surface.

Sputtering material from the sample surface with inert gas ion beam milling is an ideal method to remove prior damage. This is the basis for the SEM Mill.

Follow the instructions below to load the sample into the SEM Mill and to set the milling parameters for either cross-section samples or plan view samples.

Cross-section samples

To load a cross-section sample into the SEM Mill:

1. Press **Vent** on the **Main** tab of the touch screen or the Operator Console application. This releases the load lock vacuum.
2. Lift the load lock cover and then rotate it to expose the stage (Figure 5).

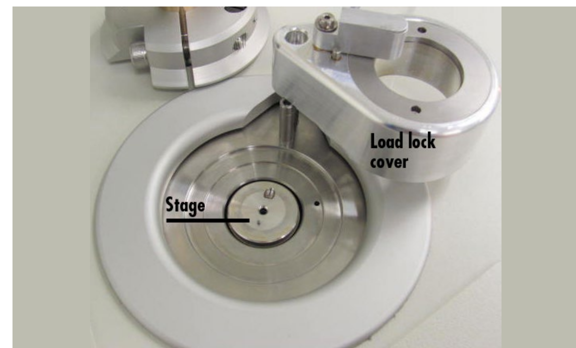

Figure 5. Lift the load lock cover and then rotate it to expose the stage.

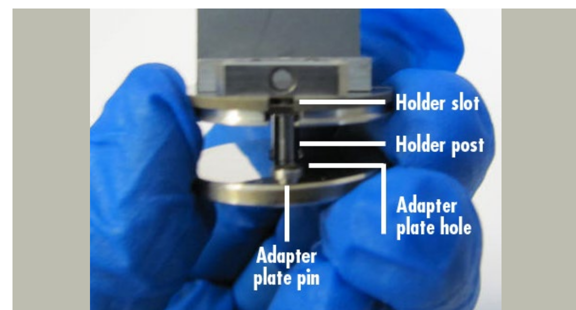

Figure 6. Insert the holder into the adapter plate.

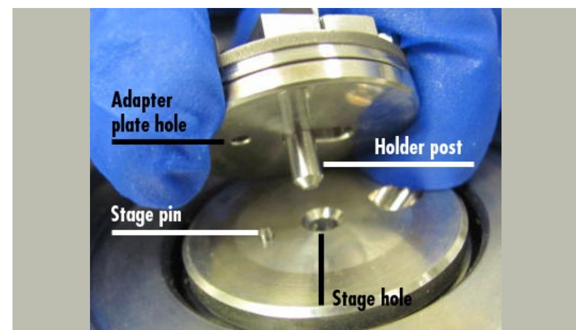

Figure 7. Insert the holder post into the stage hole.

3. Insert the cross-section holder into the adapter plate (Figure 6).
4. Load the cross-section holder/adapter plate into the SEM Mill:
    a. Insert the cross-section holder post into the stage hole (Figure 7).
    b. Align the adapter plate index hole with the stage index pin and ease the holder into place.
    c. Close the load lock cover.
5. Press **Vacuum** on the touch screen or the Operator Console application.
6. Set the milling parameters:
    a. Set one ion source (right or left) to 4 kV with 45% focus and a 7° beam angle; mill cross sections with the substrate side of the chip closest to the beam.
    b. Set a ±55° rocking angle.
    c. Set the milling time to 40 minutes.

During milling, look through the top window to confirm that the argon ion beam is illuminating the sample. If not, see *Troubleshooting ion beam issues, below.*

Plan view samples

To load a plan view sample into the SEM Mill:

1. Press **Vent** on the **Main** tab of the touch screen or the Operator Console application. This releases the load lock vacuum.
2. Lift the load lock cover and then rotate it to expose the stage (Figure 5).
3. Use the pin stub mount gripper to lift the pin stub mount from the stub storage mount.
4. Insert the pin stub mount into the SEM Mill:
    a. Insert the pin stub mount post into the stage hole.
    b. Align the stub index hole with the stage index pin (Figure 8) and ease the stub into place.
    c. Close the load lock cover.

Figure 8. Use the pin stub mount gripper to insert the pin stub mount into the stage. Align the stub index hole with the stage index pin.

5. Press **Vacuum** on the touch screen or the Operator Console application.
6. To remove about 1 µm of thickness of copper from a patterned device, set the following milling parameters:
    a. Set both left and right ion sources to 4 kV with 45% focus and a 4° beam angle.
    b. Select continuous rotation.
    c. Set the milling time to 30 minutes.

During milling, look through the top window to confirm that the argon ion beam is illuminating the sample. If not, see *Troubleshooting ion beam issues*, below.

Troubleshooting ion beam issues

If the ion beam is not illuminating the sample or is not hitting the area of interest, try the following suggestions:

Make certain the sample is opaque. The SEM Mill has a sample height detection feature that adjusts the stage height (the Z-axis position) so that the ion beam is centered on the sample. If the sample is not opaque (for example, the area of interest is covered with a glass cover slip or the sample is translucent), the height detection feature will not function correctly.

Make certain the sample is plumb and level. If the sample is not seated plumb and level in the

cross-section holder or on the pin stub mount, the sample height detection feature will not adjust the stage height correctly. Reseat the sample in the holder or on the stub, making certain that it is plumb and level.

Make certain the sample has a clean, straight surface. If the sample has uneven topography, the higher edge may impede the ion beam from reaching the area of interest. Or, the uneven surface may cause the sample height detection feature to set the stage height incorrectly.

Adjust the vertical offset. This is a user-controlled parameter that is used to shift the Z-axis position of the stage. In cases where the operator chooses not to use the sample height detection feature, set the vertical offset to the sample height:

- **Basic Edition instruments**
  To set the vertical offset on Basic Edition instruments, press **Motion** on the touch screen to display the **Motion** tab. Enter the vertical offset position of the stage with respect to the incidence of the ion source in the **Offset** control. This setting specifies an offset from the default position automatically determined when the sample is introduced into the chamber.

  The vertical offset functionality is affected by the **Ignore Sample Height** check box. If this check box is:

  Not checked (default), then the vertical offset allows you to incrementally move the sample up or down during the milling cycle to adjust the sample's vertical height.

  Checked, then the vertical offset is the measured height of the sample plus the holder (a negative value).

  The combination of the **Ignore Sample Height** option and the vertical **Offset** option is intended to be used for samples where the height cannot be readily detected with the laser, for example, when the sample is translucent or has gaps in the substrate.

- **Premium Edition instruments**
  On the Instrument Graphic, which is displayed on the left side of the **Main** tab of the Operator Console application, enter the sample height in the **Vertical Offset** field. This value sets the vertical position of the sample with respect to the incidence angle of the ion source. This setting specifies an offset from the default position automatically determined when the sample is introduced into the chamber.

  The vertical offset can only be changed in 0.1 mm increments (+ or -) in the Operator Console application. The **Ignore Specimen Height** option available on the touch screen is not available on the Premium Edition Operator Console application.

  If you set a vertical offset, remember to ***reset it to zero*** after you complete the milling operation. The vertical offset does not automatically reset.

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