TEM Sample Preparation for the Semiconductor Industry — Part 3

John F. Walker, FEI Europe Ltd., Cambridge, England

Part 1 of this series described how focused ion beam (FIB) microsurgery is used to successfully cross-section and prepare *material-specific* samples for SEM and TEM analysis. In Part 2, we detailed how FIB is also the tool of choice to prepare *site-specific* samples, particularly for transmission electron microscopy (TEM) analysis. In this final article of this series, we describe actual sample preparation, cutting a selected area to size and mounting it on a grid for FIB preparation. Focused ion beams are very useful in preparing TEM specimens that have unique characteristics. In particular, the ability of such systems to image submicron features within a structure has allowed accurate identification of the precise place to make a membrane.

Prior to the FIB preparation process, the approximate area must be selected, ground to a suitable size, and attached to a TEM grid.

The equipment needed for TEM sample preparation includes a good optical microscope, a polishing jig, polishing pads of appropriate grades, a micrometer or calipers, a scribe, tweezers, and scalpels. Glue—cyanoacrylic or epoxy—or clear wax, solvents, and filter papers are needed. Also useful are rotary or wire saws, dicing saws, ultrasonic cutters, and tripod polishers.

To prepare a sample for TEM with FIB, start by cleaving the wafer with a diamond-tipped scribe, nicking the back of the wafer at the edge to start the cleave. Continue cleaving until the sample reaches 2-3 mm square (Figure 1), when it becomes too small to cleave further and must be ground to final size. Cleaving is considerably easier when the substrate is first thinned to 200-300 microns, which can be done with 20-50 micron Carborundum paper—be sure to check regularly with a micrometer.

Next, wax or cement the cleaved square to the edge of a glass slide (Figure 2). Use an optical microscope, if available, to orient the cross-sectioned membrane more precisely. Grind the face of the specimen to within 100 microns of the feature using a medium grade of abrasive (Figure 3).

At this point, a finer grade of abrasive can be used. When a thickness of about 50 microns is reached, remove the specimen from the jig and clean it in acetone. An optical microscope and micrometer screw on the jig can be used to check the progress of the grinding—use the micrometer to determine when to stop.

Mounting on a TEM grid is the final step in pre-FIB sample preparation. The fragile specimen is vulnerable to handling, so a copper TEM grid needs to be prepared by cutting out a segment from a hole or slot grid. A pie-shaped piece allows unimpeded access for the ion beam, even when the sample is tilted in the FIB system. The grid can be cut with a scalpel or scissors. When the grid is ready, attach the specimen with adhesive to the rough side of the grid. Silver-loaded epoxy is a good choice. When the epoxy is cured, the sample is ready for insertion into the FIB system. Figure 4 shows the grid with the TEM sample inserted into the sample holding vice prior to putting it into the FIB system.

A variation is possible for making a membrane for plan-view observation of a feature. Plan-view TEM is useful for looking at vertical features, like trench capacitors and dislocation networks.

This article is condensed from an application note entitled, "TEM Sample Preparation: Cutting a Selected Area to Size and Mounting It on a Grid for FIB Preparation." For a copy of the complete note and additional figures, please contact FEI Company, 7451 NE Evergreen Parkway, Hillsboro, OR 97124-9931.

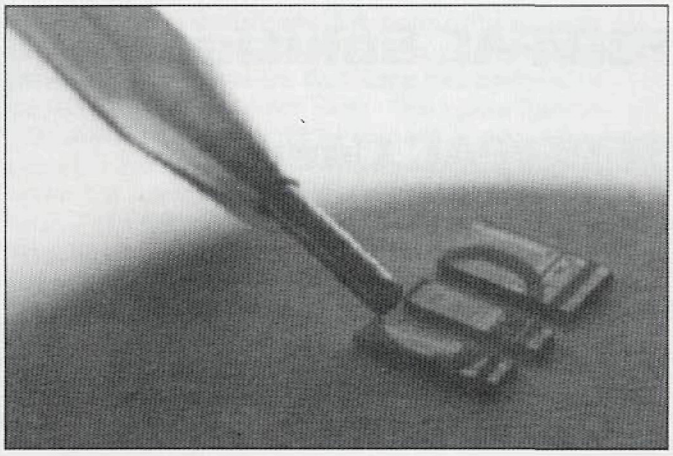


Figure 1: Continue cleaving until the correct size is reached.

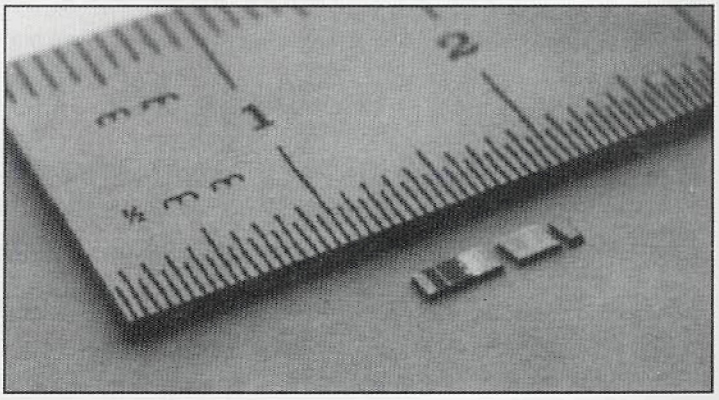


Figure 2: The center piece is ready for grinding when it is approximately 2-3 mm square.

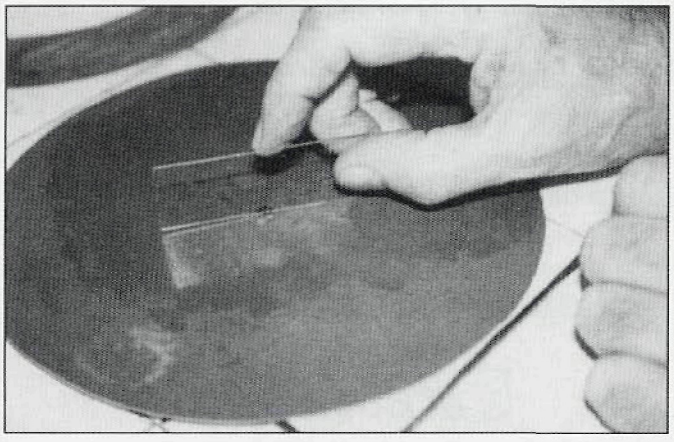


Figure 3:Grind until the bottom edge of the slide reaches the disk. Wax the sample to the edge of a glass slide for grinding.

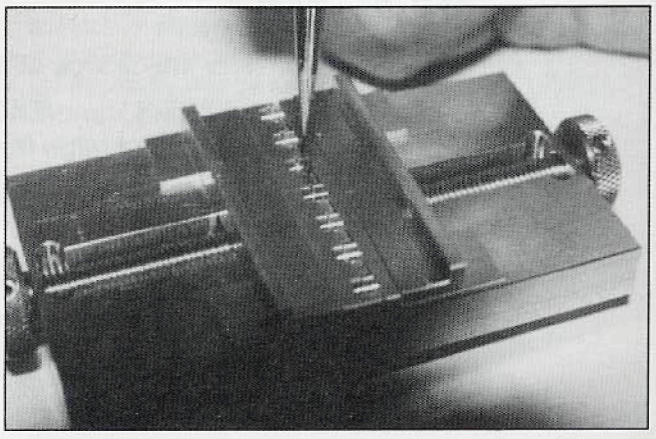
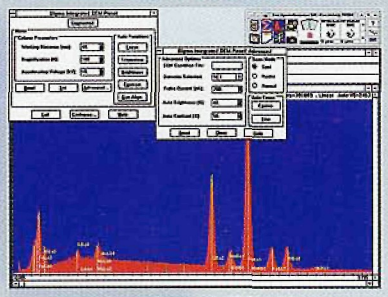


Figure 4: Insert the grid into the TEM sample-holding vice.

KEVEX

Automatic Return on Investment.... Integrated SEM/EDS Automation!

Automation using preassigned macro buttons...



Spectral Display with ISEM Menus.

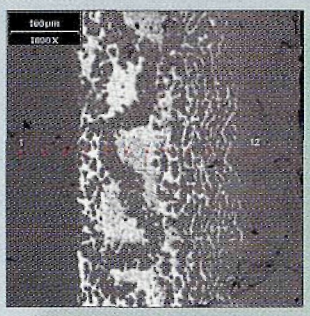
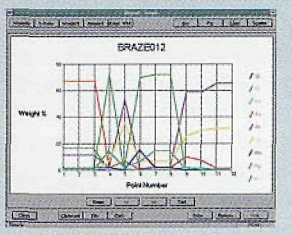
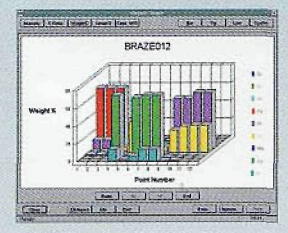


Image with DMA Line Profile.

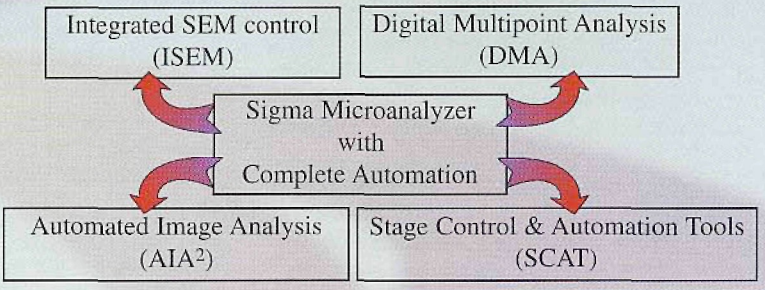


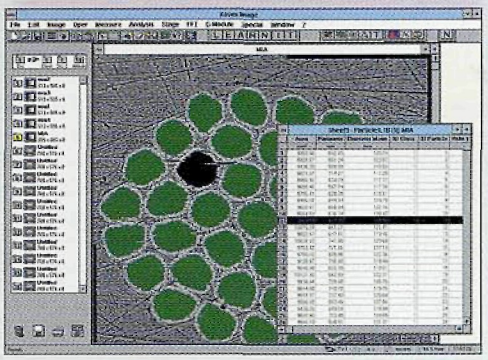
Resulting X-Ray Profiles.



3D Graph of Weight Percents.

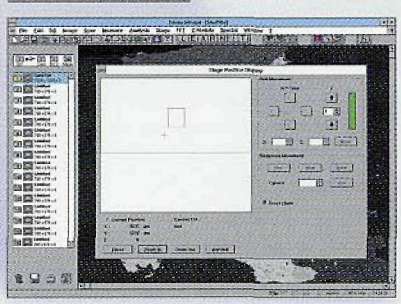
- Macro capability for Quantitative & Image Analysis
- Automated Beam Positioning
- EDS & Image Database
- Automated Reporting in Standard File Formats (Excel, Word...)



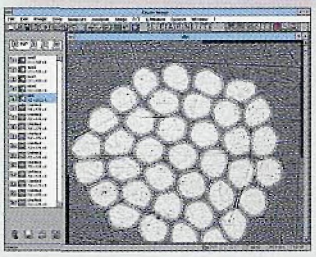


Processed Image with Results Table.

Automation using preassigned macro buttons... AIA² incorporates Stage Automation.



Stage Control Display and Imaging GUI.



Stitched Image within Imaging GUI.

- Image Stitching
- FFT Filtering (forward and reverse)
- Automated Morpho-Chemical Analysis
- Locked and Liberated Analysis
- Stage Automation for Unattended Operation

Kevex Introduces *NEW* Sigma Level 4: AIA²

The NEW Automated Image Analysis (AIA²) is more comprehensive than ordinary Image Analysis packages currently being offered for Microanalysis applications. AIA² offers high powered analysis tools such as Locked and Liberated analysis and Image Stitching, which can be implemented on Electron or X-Ray Images. AIA² supplemented by the NEW DMA, ISEM and SCAT offer today's Microanalyst a truely automated approach to Microanalysis. The Kevex Sigma offers an automation approach for every level of Microanalyzer, at affordable prices. Kevex has automated virtually every SEM on the market, including Leica/Zeiss, Topcon, Philips, Amray, Electroscan, Hitachi and JEOL.

Kevex Instruments • **24911 Avenue Stanford** • **Valencia, CA 91355** • **Tel. (805) 295-0019** • **Fax (805) 295-8714**

Circle Reader Inquiry #12