Using a Focused Ion Beam (FIB) System to Extract TEM-Ready Samples from Complex Metallic and Ceramic Structures

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Abstract

Driven by the analytical needs of microelectronics, magnetic media and micro-fabrication industries, focused ion beam (FIB) systems are now capable of milling and manipulating samples for the analysis of microstructure features having dimensions of 180 nm or less. A technique for locating and extracting site specific specimens for examination by transmission electron microscopy (TEM) has been developed. An identified feature can be located and precisely milled with an FIB system from two sides to prepare an ultra-thin sample, and then extracted from the region with a glass rod micro-manipulator onto a grid for TEM analysis. This specimen preparation method has been applied to semiconductor failure analysis and to the study of metallic and ceramic microstructures with irregular topographies and complex multi-layered components.

Introduction

Transmission electron microscopes are commonly used to study biological, metallurgical, ceramic, and crystalline structures. By viewing a specimen with a TEM, one can determine elemental composition within a spatial resolution of <1 nm, analyze crystal lattice structures, and study complex microstructures. Specimens prepared for TEM analysis must be small enough (typically 3 mm in diameter or less) to fit into the microscope specimen holder, and thin enough (< ~ 100 nm) to be electron transparent.

Preparing a suitable TEM specimen is often the limiting factor in performing TEM analysis. Many specimen preparation techniques may require "artistic" skills for success. In some instances, recipes that are successful in one laboratory may not work in another. Using a focused ion beam (FIB) instrument, one can produce TEM specimens routinely, quickly, and precisely from a wide range of materials[1].

The Lift-Out Technique

The "lift-out" technique was developed initially for microelectronics device applications. A TEM specimen can be extracted while the high-value semiconductor wafer remains intact. The method was first described by a group at Philips[2] and further developed at Cirent Semiconductor and the University of Central Florida[3]. The specimen preparation technique described here was developed on integrated circuits and other metallic and ceramic microstructures utilizing a FEI FIB ion milling system. TEM analysis was performed on a Philips EM430 operating at 300 kV. Only the outline of the experimental technique is presented; a more detailed description has been published previously[3].

A bulk sample, such as a 200 mm diameter silicon wafer, is placed directly into the vacuum chamber of the FIB for TEM specimen preparation. Using control and navigation software provided with the FIB tool, the site of interest, such as a particular circuit, interface layer, metal bonding site, or potential defect area, is located on the sample surface. The local area to be cross-sectioned is coated with a protective layer of tungsten or platinum using ion beam induced deposition. Bulk material is removed by milling trenches parallel to each side of the protective film until a film thickness of approximately 300 nm is reached. Figure 1 is a view of the specimen after the platinum film and bulk material removal steps have been completed. The FIB system can also use reactive gas-assisted etching that accelerates the milling rate.

A high ion beam current is used for the initial bulk milling, while a lower beam current is used for the final thinning of the specimen to an electron transparent membrane. The diameter of the gallium ion sputtering beam can be below 10 nm and can be positioned with accuracy of less than 50 nm. The operator can visually monitor the thinning process and make adjustments as necessary. High-resolution secondary-electron images can be obtained during the process if desired. The wafer is tilted to 60 degrees, exposing the base and sides of the film. Subsequent FIB cuts partially separate the specimen section from the bulk material. Figure 2 shows the 300 nm thick sample severed at the bottom and sides. The sample is tilted back to 0 degrees and the film is further thinned to the desired thickness, usually ~ 100 nm for conventional TEM analysis. A final FIB release cut is made and the membrane is freed from the bulk sample, ready for extraction (Figure 3).

Sample Extraction

Once the membrane has been cut away, the entire bulk sample is then removed from the FIB and placed under an optical microscope. A glass rod or tube drawn to a small tip and attached to a micromanipulator arm is used to transfer the TEM thin membrane from the bulk sample to a TEM grid. It is believed that electrostatic forces attract the membrane to the glass pipette for safe transport to a carbon or Formvar-coated copper TEM analysis grid. Figure 4 shows an optical microscope and micromanipulator for extracting the specimen from the intact wafer. Figure 5 is a scanning electron micrograph of a lift-out specimen positioned on a TEM grid.

Discussion

Figure 6 is a bright field TEM image of an integrated circuit cross section

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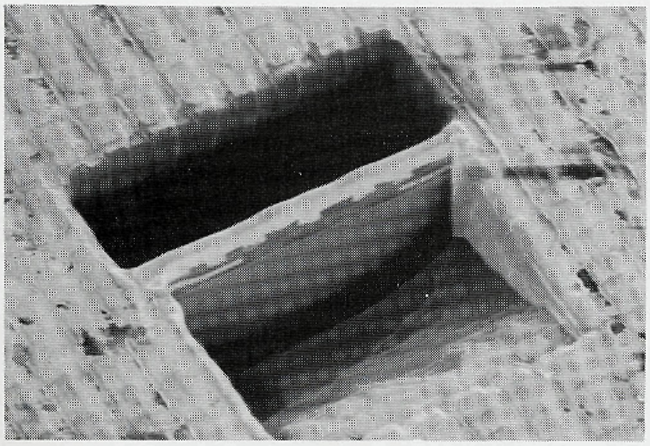

Figure 1: FIB image of specimen after the bulk material removal steps are completed.

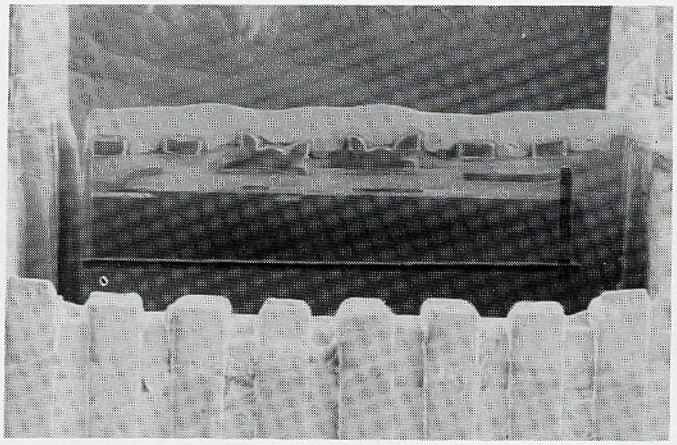

Figure 2: FIB image of specimen thinned to 300 nm and severed at the bottom and sides.

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that was prepared by the FIB lift-out technique.

A major benefit in performing an entire specimen preparation in a FIB tool is the time savings. Prior methods, which required movement of the bulk sample from machine to machine for different stages of the process, could take several hours and much longer for difficult specimens. The development of TEM specimen preparation techniques for new materials may consume days, weeks, or even years of research. In contrast, the majority of specimen preparations conducted to date with a FIB tool with the technique outlined here have taken at most 5 hours. Recent developments in FIB instrumentation allow much of the process to be automated and run unattended[4]. The vacuum environment of the FIB tool also minimizes the potential contamination of samples; this is important for the examination of sub-micrometer features.

Applications

Advances in focused ion beam technologies and their incorporation in commercially available systems is expanding the realm of microstructures that can be manipulated and analyzed. The FIB-based specimen preparation and extraction technique provides scientists and researchers with a new method for extracting TEM specimens from materials and from layered metal or ceramic structures with complex atomic interfaces or irregular topographies. This technique can be applied to preparing TEM specimens for quality control of manufacturing processes, failure analysis, or research and development of new materials for a wide range of industries.

Summary

Modern FIB tools are ideal for TEM specimen preparation for microelectronics and other difficult materials. The increased accuracy of on-wafer navigation, beam placement for FIB cuts, and finer beam resolution enables the precision identification and extraction of higher quality specimens having dimensions of 100 nm or less with consistent uniformity. TEM specimens may be produced without destructive sample preparation of the bulk material. These advances in FIB tool capabilities also expand the use of the TEM for the exploration of new materials and microstructures.

Acknowledgments

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References

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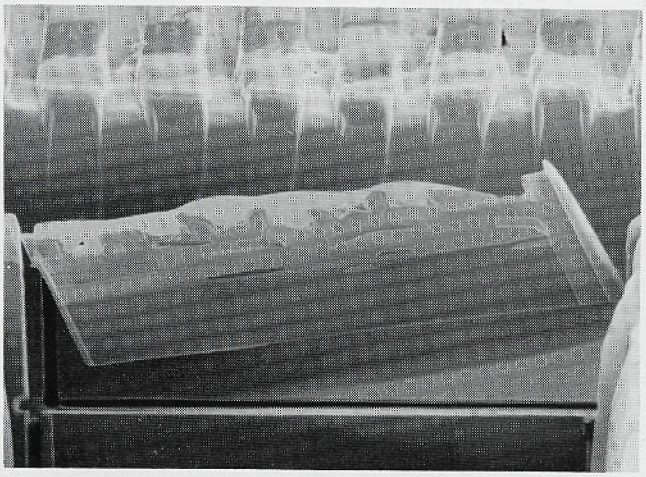

Figure 3: FIB image of a specimen ready for extraction after release from the whole wafer.

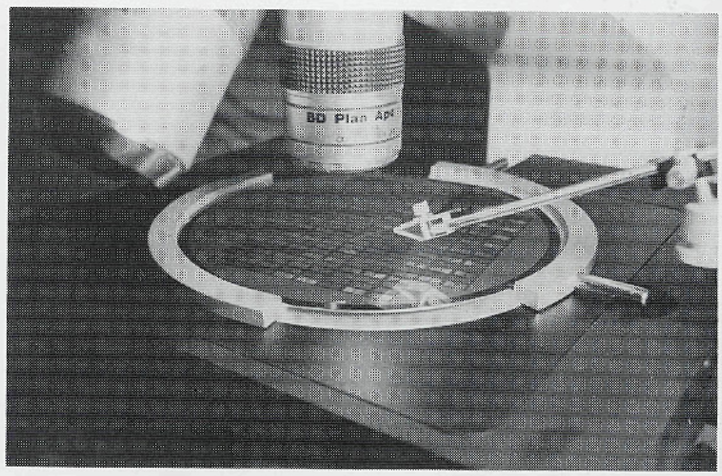

Figure 4: Extraction of sample from intact wafer by micromanipulator.

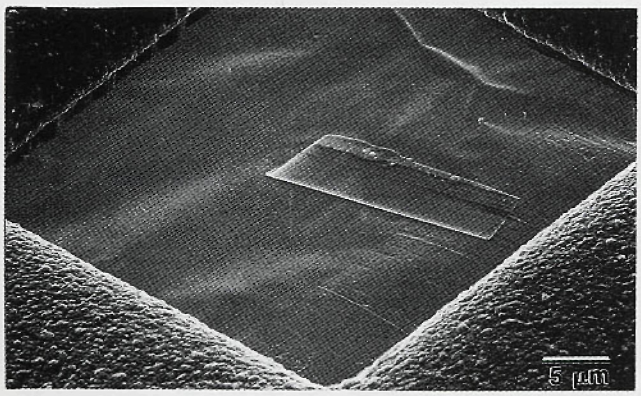

Figure 5: SEM image of a lift-out specimen positioned on a TEM grid.

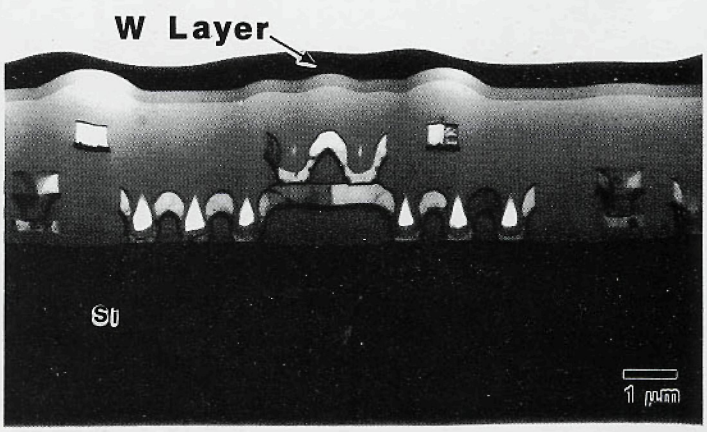

Figure 6: TEM image of an integrated circuit prepared by the FIB lift-out technique.


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