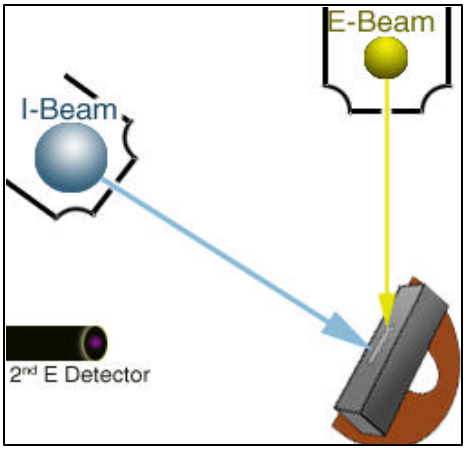
1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499
e-mail: info@southbaytech.com
www.southbaytech.com

A Method for Pre-FIB Specimen Preparation

Introduction

Within the past few years Moore's Law relating to transistor density on integrated circuits has prevailed. This exponential growth and ever-shrinking transistor size has resulted in the need for analytical techniques that can resolve these sub-micron devices. The invention of the Focused Ion Beam (FIB) instrument has allowed this analytical process to evolve. FIB systems have been produced commercially for approximately ten years and were initially used solely by semiconductor manufacturing companies. In recent years academic institutes and smaller research labs have utilized these FIB systems. FIB systems operate in a similar fashion to a scanning electron microscope (SEM) except, rather than a beam of electrons; FIB systems use a finely focused beam of gallium ions for site specific milling. Imaging is done with the ion beam in 'standard' systems. Dual beam systems are also available which contain both an ion beam source and electron beam source for viewing of specimens nondestructively.

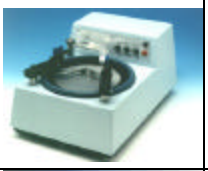
Below is a general procedure for preparing pre-thinned specimens for introduction into a FIB system.



Schematic illustration of the dual beam FIB used for TEM sample preparation.

Materials

The following equipment and consumable items were used for the preparation:

Equipment	Description
Model 920 8" Lapping and Polishing Machine with Glass plate 	The Model 920 is a multi purpose grinding and lapping machine designed for accurately lapping and polishing a wide range of materials.
Model 650 Low speed diamond wheel saw with 4", .012" thickness, fine diamond cut off wheel 	The Model 650 Low Speed Diamond Wheel Saw is a compact, multipurpose, precision saw designed to cut a wide variety of materials with minimal subsurface damage.
Model 590 Tripod Polisher® 	The Model 590 Tripod Polisher® can be used to prepare a sample for both SEM and TEM analysis, in both plan-view and cross section view.
30?m, 15?m, 3?m and 1?m diamond abrasive films	1?m is used dependant on material, surface features
MWH135 Quickstick 135 Mounting wax	135 degree crystal clear wax, high hardness and strength. Widely used in semiconductor and optical materials
Cu slotted or Mo slotted grids	The use of halved-slotted grids has become the norm for pre-thinned FIB specimens.
EPOTEK epoxy	A stable two part epoxy used for adhering polished specimen to grid.

1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499

e-mail: info@southbaytech.com
www.southbaytech.com

Method

Initial site marking

For the purposes of this application we will use a silicon integrated circuit semiconductor device. We first begin by marking the area of interest. Recent advances into the .13µm copper technology have pushed the limits of optical light microscopes seeing specific device areas, because of this either the FIB or a variable frequency laser marker are the norm employed to mark the specific areas of interest (AOI).

Cleaving and Mounting

1. The area of interest (AOI) is cleaved out to roughly 3mm by 5 mm section. Ensure orientation and plan out cleave lines so that a cleaved face is ~1.5 mm parallel with the AOI along the 3mm length of the section. The closer the cleave face is to the AOI then the less amount of material that needs polishing off which mean less time involved. (Figure 1).
2. Briefly clean the cleaved section off with acetone to remove any residuals from the cleaving process.
3. Heat the cross section stub to 135-200 C and apply a small amount of Quickstick 135 mounting wax to the surface of the cross section stub and mount the section.
4. Orient the AOI just barely over the edge of the stub so that it is parallel to the line made by the stub edge (Figure 2&3).
5. Press firmly on the section using a tweezers away from the AOI.
6. After waiting for the stub to cool off attach it on the Model 590 Tripod Polisher® and use a bubble level to generally level the polisher.

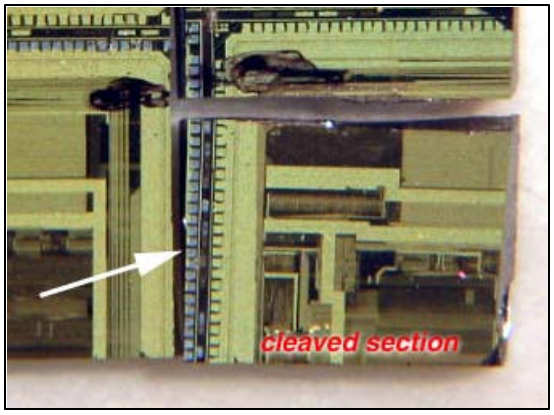
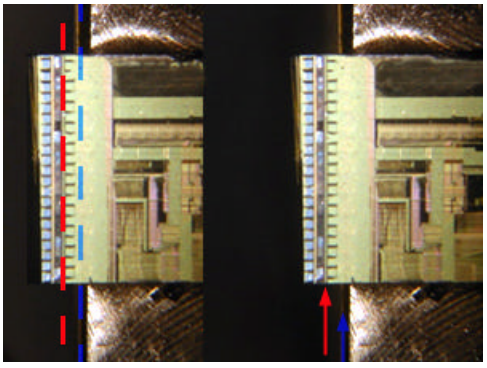


Figure 1: A cleaved section with arrow pointing to the area of interest face.



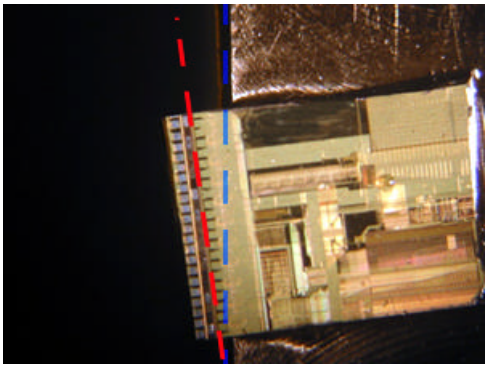


Figure 2 and 3: At left, the blue dotted line/arrow denotes stub edge while red dotted line/arrow denotes the area of interest cross section plane. Notice how both lines are parallel; this contributes to less adjustments during the polishing process. At right image shows a poorly placed section on a stub this would cause excessive adjustments and loss of time during the process.

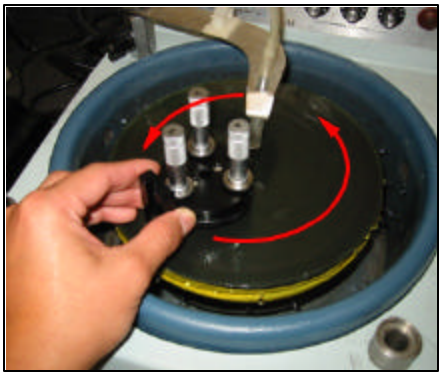
1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499

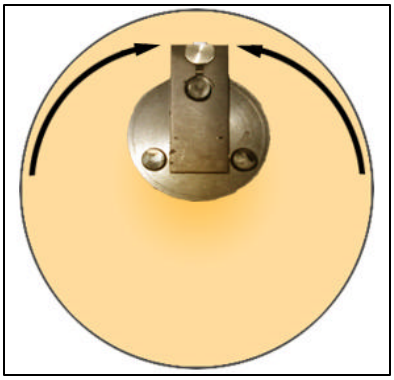
e-mail: info@southbaytech.com
www.southbaytech.com

1st Side Polishing

Polishing requires two stages: first and second side polishing. The Model 920 is best suited for polishing by providing stability and high torque at the low speeds required for mechanical thinning a pre thinned FIB specimen. Both stages of polishing are completed using a hard glass lapping plate with plain backed diamond lapping films. Diamond films of 30 µm, 15 µm, 3 µm and an optional 1µm are used for polishing of the specimen to the desired area. The use of a 1 µm polishing step is dependent on the sample type in most cases. In the case of integrated circuits, copper interconnects can start to smear over the top surface during polishing. Using a 1µm diamond film cleanup the top edge and polishing interface can remain sharp and crisply detailed when viewed in the FIB system.

1. Cover the glass plate area about ¼ with water. Run your hand over the surface of the plate to ensure that there are no particles or contaminants on the surface, which may damage the specimen during polishing.
2. Float a sheet of 30 µm diamond lapping film and squeegee it to the glass plate with the abrasive side up. Surface tension holds the film in place.
3. Turn on the lapping machine and set the wheel speed at around 150 rpm. The Tripod Polisher® should be placed with the specimen cross-section face, facing parallel to the direction of the lapping wheel (Figure 5). With the vast amount of metal layers this prevents any delamination, chipping, and smearing at the top surface. Start polishing from the center of the wheel and work towards the outer edge. (See Figure 4, 5 &6)





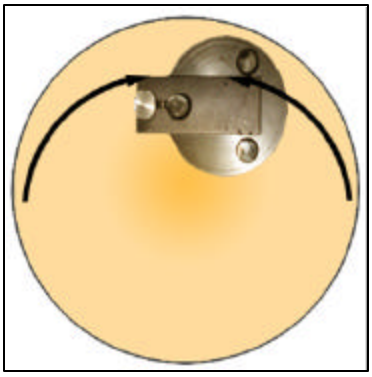


Figure 4,5, and 6: At left, Fig. 4 showing orientation of Tripod with respect to wheel rotation. Figure 5 (center) shows a view seeing through the wheel up to the bottom of the Tripod. Figure 6 (right) shows incorrect direction of polishing which can cause delamination, chip outs and smearing in certain samples.

4. Once polishing has reduced the specimen to within 200-250 µm of the desired area of interest, the diamond abrasive film should be changed to 15 µm film. Using the same procedure as listed in steps 1-2, polish the specimen until the area of interest is within +/-100 µm.
5. Change lapping film to the 3 µm grit and polish until the area of interest is about 10-15 µm away.
6. (**Optional**) Again change the abrasive film to 1 µm and polish until the area of interest is 10 µm or less away. 1st side polishing is complete! Remove stub from Tripod Polisher® and place on hot plate.

1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499

e-mail: info@southbaytech.com
www.southbaytech.com

Diamond Sawing

1. Remove the section from the stub and place it centered on aluminum/graphite mounting plate, press firmly to seat the section within the wax on the mount. Allow to it cool.
2. Using the alignment microscope of the Model 650, maneuver the section so it's 400µm to 500µm from the AOI edge. Start the sawing with a wheel speed of 2-3. Always monitor the sawing action, if the section was mounted improperly then there's risk of premature break off of the small section being cut.
3. After cutting is complete use dry air or nitrogen to carefully blow off any water remaining from the cutting process, then return mounting plate to hot plate.

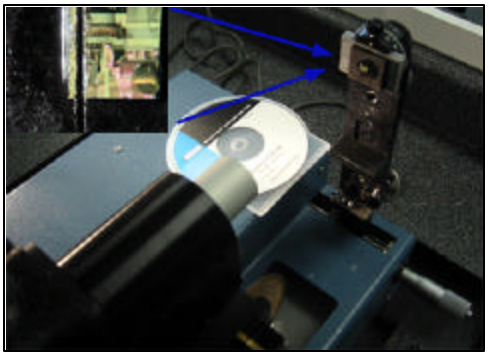


Figure 7: Model 650 Low Speed Diamond Wheel saw with sample mounted for cutting. The cut is approximately 500µm from the AOI.

Tripod Polisher® Planarization

Before the cut section can be mounted to the Tripod Polisher® , it is necessary to establish a flat, planar surface to start from. There are several ways to level the Tripod Polisher®, listed below are a few common techniques.

Method #1: The Tripod Polisher® is placed onto a hard, flat surface (such as the glass leveling slide) with the pyrex polishing mount in place. The two rear micrometers are then adjusted such that the Pyrex and the back feet are coplanar. Using a business card to monitor the angle of tilt and pitch is a quick and easy method for leveling the tool. Once the tool has been leveled, the entire assembly is ground on 15 micron diamond lapping film to planarize the feet and Pyrex. (See Figure 8 below)

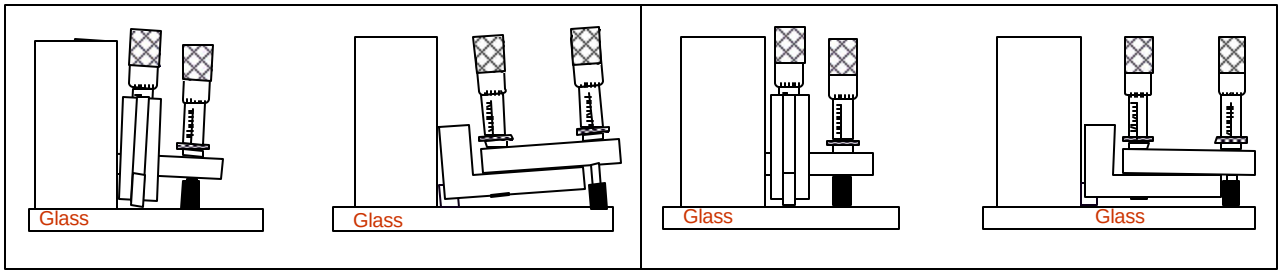


Figure 8: Illustration of the use of a business card to align the Tripod Polisher®. The card is used as a T-square to make the L-bracket level with the back feet. On the left the Tripod Polisher® is severely out of alignment and needs adjustment. On the right the tool has been properly setup.

Method #2: The Tripod Polisher® is placed onto a hard, flat, level surface and checked with a bubble level. With the pyrex stub in place and the bubble level on the Tripod, the two rear micrometers are then adjusted such that the pyrex and the back feet are coplanar. Once the tool has been leveled, the entire assembly is ground on 15 micron diamond lapping film to planarize the feet and Pyrex.

Method #3: The Tripod Polisher? is inverted and a measurement is taken from the bottom face of the pyrex stub to the bottom of the L-bracket. The Delrin feet are leveled to the bottom of the L-bracket using a glass leveling slide. The measurement from the pyrex stub to the bottom of the L-bracket is then used to adjust the micrometer feet position. Once the adjustments have been made to level, the entire assembly is ground on 15 micron diamond lapping film to planarize the feet and Pyrex.

1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499

e-mail: info@southbaytech.com
www.southbaytech.com

Remounting for 2nd Side Polishing

1. The pyrex stub is removed from the polisher and heated on the hot plate.
2. Using tweezers carefully lift the sliver from the graphite/aluminum mounting plate of the 650 and transfer to pyrex stub. The specimen should be placed onto the pyrex stub with the polished side down, area of interest facing towards the flat edge of the Pyrex stub and aligned such that the specimen is parallel with the flat edge.
3. Transfer heated stub to under microscope for alignment of sliver on stub edge.
4. Working quickly under the microscope, use tweezers and align the specimen top surface edge to the edge of the pyrex stub. If needed, transfer the stub back to the hot plate to remelt wax to proper viscosity.
5. Tilt the stub to check for edge-to-edge alignment, with no gap between the stub edge and specimen edge visible. Allow the stub to cool completely.
6. Carefully scrape away any excess wax that remains. A q-tip or optical tissue with acetone can be employed to gently wipe away wax. Any small deposits of wax will be removed in an acetone wash after second side polishing has been completed.

Figure 9: Cut specimen mounted up to pyrex stub edge

Figure 10: Using a retractable razor remove any excess amounts of wax.

2nd Side Polishing

1. Place a slight amount of water onto the surface of the glass plate. Run your hand over the surface of the plate to ensure that there are no particles or contaminants on the surface, which may damage the specimen during polishing. Float a sheet of 30 µm diamond lapping film and squeegee it to the glass plate with the abrasive side up. Surface tension holds the film in place.
2. Take an initial measurement of the specimen thickness. Turn on the lapping machine and set the wheel speed at around 150 rpm. The Tripod Polisher® should be placed with the specimen cross-section face, facing parallel to the direction of the lapping wheel (Figure 2). With the vast amount of metal layers this prevents any delamination, chipping, and smearing at the top surface. Start polishing from the center of the wheel and work towards the outer edge. (See Figure 4,5 &6).
3. Once polishing has reduced the specimen to within 200-250 µm thickness, then diamond abrasive film should be changed to 15 µm film. Using the same procedure as listed in steps 1-2, polish the specimen until thickness is <100 µm but not less than 75 µm.
4. Change lapping film to the 3 µm grit and polish until final thickness is +/- 20 µm.
5. (**Optional**) Again change the abrasive film to 1? m and polish until the surface edge is damage free.

1120 Via Callejon ■ San Clemente ■ California ■ 92673 ■ USA
Telephone: (949) 492-2600 ■ Fax: (949) 492-1499

e-mail: info@southbaytech.com
www.southbaytech.com

Mounting the Grid to the Specimen

1. First prepare a slotted grid by cutting it in half on a hard surface. The hard surface ensures a fast cut without warping the grid. Remove pyrex stub from polisher and place under microscope.
2. Place two small dots of epoxy on the specimen, then with tweezers mount the halved slotted grid onto specimen (figure 11).
3. Place stub with grid attach onto hot plate for 10-20 minutes depending on epoxy used. After allocated time while the stub is still hot use tweezers to carefully and gently extract specimen/grid off the pyrex stub (Figure 12). Soak grid/specimen in acetone till any wax residue is removed.

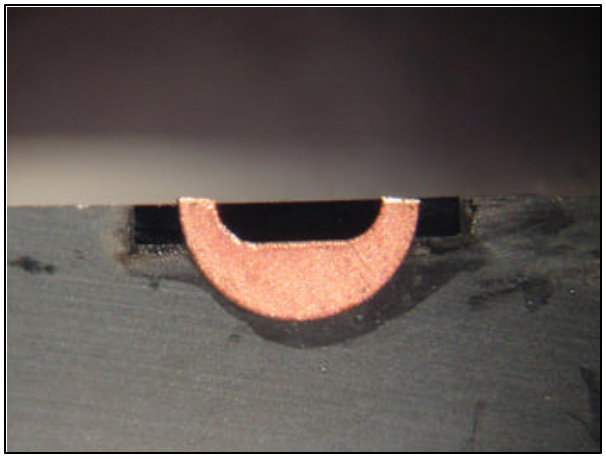


Figure 11: After placing grid onto polished specimen align carefully and place onto hot plate to cure epoxy.

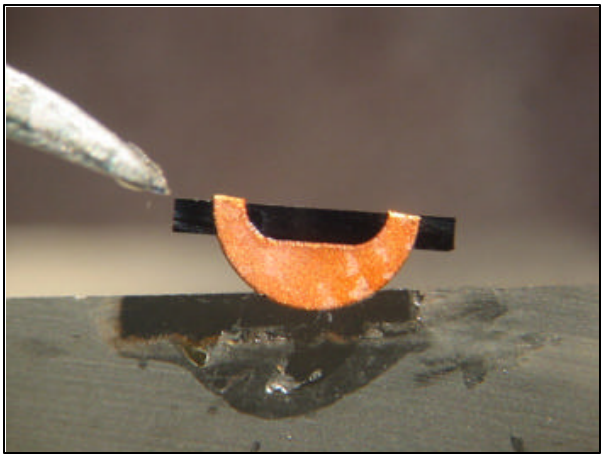


Figure 12: After curing the epoxy, remove specimen with tweezers while wax is still viscous and soak specimen in acetone.

Conclusion

As can be seen the Tripod Polisher® can be a valuable tool in preparing pre-thinned FIB specimens. While elaborate and expensive systems exist to automate the pre FIB sample preparation, the Tripod Polisher® can allow total preparation in less than an hours time at an expondentially less cost to the user. The ability of the tool to monitor the specimen thickness directly, to adapt the tool to prepare virtually any material type, and the high specimen throughput make the Model 590 Tripod Polisher® the ideal tool for pre-thinned FIB specimen preparation.

References

J. Benedict, R. Anderson, S. Klepeis, M. Chaker, A Procedure for Cross Sectioning Specific Semiconductor Devices for both SEM and TEM Analysis, ed. R. Anderson, Mat. Res. Soc. Proc. vol 199, Pittsburgh, PA USA. Pp. 189, 1990.

C. Liu, C.Chen, J.Chiou, D.Su, A Methodology to reduce ion beam damage in TEM specimens prepared by FIB, ISTFA Proc '02.

Yaguchi, T., Kamino, T. (2000) Cross-sectional specimen preparation and observation of plasma sprayed coating using the focused ion beam/transmission electron microscope. Microsc & Microanal: vol. 6, number 3, 218-223.

Cairney, J., Smith, R. (2000) Transmission electron microscope specimen preparation of metal matrix composites using the focused ion beam miller. Microsc & Microanal: vol. 6, number 5, 452-462.