Experience with a Dicing Saw for Rapid Pre-FIB TEM Sample Preparation

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Introduction

Since the publication of the use of a dicing saw for TEM sample preparation [1], several analytical labs have adopted this method as standard practice for site-specific cross section and plan view samples. In this article, we would like to provide additional practical details of these procedures, and describe several extensions, including useful notes on batch processing, preparing samples with an area of interest very close to the sample edge, and a Focused Ion Beam (FIB)-compatible sample holder. We present an unusual amount of detail in these processes to show some of the evolution of the method since its introduction and to allow others to easily reproduce these results.

The complete processing time for a single pre-FIB sample using this preparation method is less than 20 minutes. Batch processing of samples, described below, can easily yield four samples per hour. When comparing this processing time with that of competing sample preparation methods, we have found it important to specify the initial and final conditions of the sample.

- Initial condition: Area of interest and cut direction are identified and marked (preferably with a laser) on the original sample; if necessary, a thin protective coating of epoxy or photoresist has been applied.
- Final condition: The cut and cleaned pre-FIB sample is ready for coating and FIB milling; laser marks are documented to assist in FIB milling and TEM observation.

Few restrictions exist on the input sample for the dicing saw process. Although our own work focuses almost exclusively on semiconductor integrated circuits, we emphasize that the dicing saw process is extremely versatile. The input sample need be only a small slab of material typically 5-15 mm on a side with thickness roughly in the range 0.3-1.0 mm. No restrictions on sample crystallography or cleaved edge quality exist, since the process relies on cutting, not cleaving. For the standard process, the area of interest must be at least 1 mm from the edge of the input sample, but even this is relaxed in one of our modified processes. The output sample, shown in Figure 1, is a 3 mm long sliver with a thin ridge containing the area of interest. We do not mount this sliver on a grid; instead, by design the dimensions are such that, after FIB milling, the adhesive-free sliver can be loaded directly into a standard 3 mm TEM holder.

The precision dicing saw used for this work [2] has a single water-cooled blade 200 µm wide mounted on a spindle rotating at 30,000 rpm. The work piece is held on a 150 mm diameter vacuum chuck that is capable of *X*, *Y*, and theta (rotation) motions; cutting height is controlled by *Z* motion of the blade and spindle. The saw is also equipped with an optical microscope and camera system for accurate positioning (~1 µm in practice) of the work piece. A simple computer interface allows programming of multiple steps. The primary consumable of this tool is the blade; we use a relatively slow cutting speed of 1 mm/s on our silicon samples to reduce sample chipping and to extend blade life.

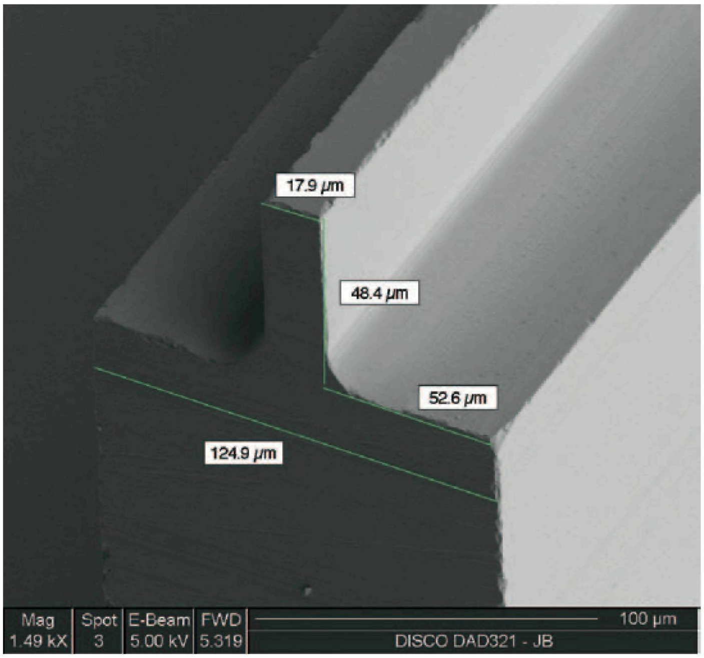

Figure 1. Detail of cross section sliver with typical dimensions of ridge and surroundings.

Standard cross section

The process for creating a site-specific, pre-FIB, TEM cross-section sample consists of six cuts. Two shallow cuts (referred to as cuts 1 and 2) that define a narrow ridge 15-20 µm thick at the top of the sample in which the area of interest is centered. Four deep cuts (cuts 3, 4, 5, and 6) define the width and length of the bulk of the sample. Between cuts 3 and 4 is a "squish" step that helps to support the final sliver. Figure 2 (left side) illustrates critical steps of the process.

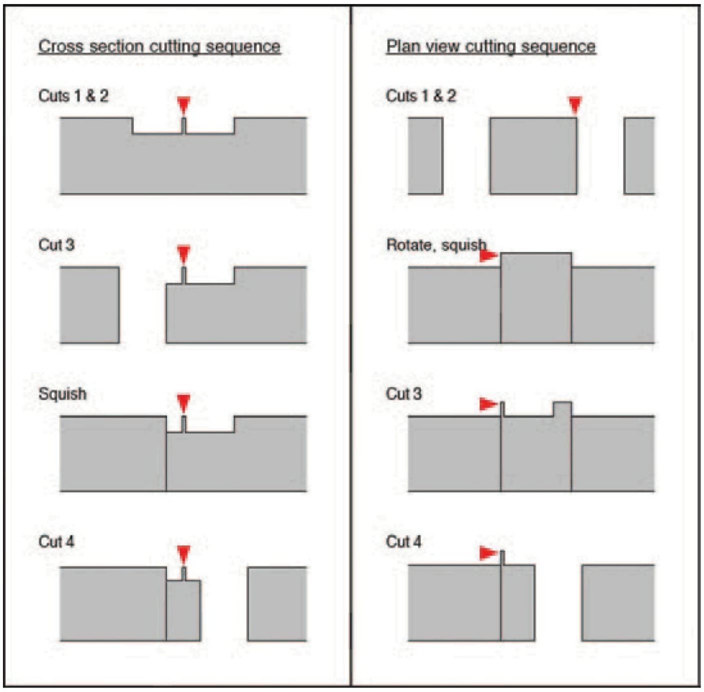

Figure 2. Steps for creating cross section and plan view TEM pre-FIB samples. Red arrow indicates area of interest on each sample.


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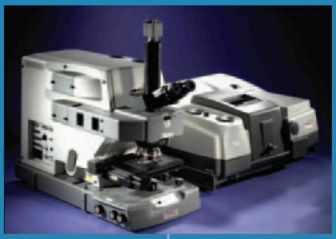

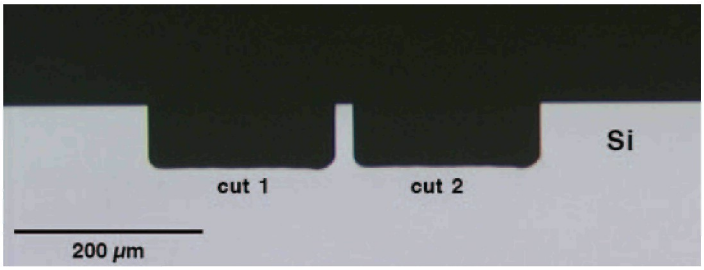

Figure 3. Cleaved silicon sample showing effective blade profile in a pair of shallow 200 µm wide cuts.

- Mounting: Mount the sample piece on a sacrificial dummy piece of, preferably, the same material using a minimal amount of Crystalbond 509 wax. A typical sample for us is 10-15 mm on a side and is mounted on a scrap silicon piece roughly 40 mm on a side. The surface of the hotplate used in this method must be kept clean in order to avoid wax particles on the bottom of the dummy piece. Such particles can cause poor suction between the dummy piece and the vacuum chuck of the saw.
- Measuring: Use a digital thickness gauge to measure the thickness of the entire assembly (sample piece, wax layer, and dummy piece) and, separately, the thickness of the dummy piece. Measurement accuracy of 5 µm or better is recommended. This information is used to set the height of the blade, measured with respect to the chuck surface, for shallow and deep cuts.
- Cuts 1, 2, and 3: Make the shallow cuts 1 and 2 in the sample piece to define a ridge 15-20 µm wide such that the area of interest is centered in the ridge. Depth of these cuts is 50-70 µm, depending somewhat on sample material. Position cut 3 about 60 µm away from the ridge and cut through the sample piece and wax and slightly into the dummy piece. This cut defines one "shoulder" of the sliver. To increase accuracy and repeatability, cuts are always made "front to back" on the sample, and the final positioning of the blade is always made in the same direction to avoid backlash in the chuck motions.
- Squish: Remove the sample assembly from the dicing saw chuck and place it on the hotplate. When the wax has melted, use tweezers to gently push together the two pieces of the original sample to close the gap made by cut 3. Remove the assembly from the hot plate and return it to the dicing saw chuck.

This important "squish" step, not previously documented but first used by the authors of [1], allows some wax to flow by capillary action between the two vertical surfaces created by cut 3. The wax provides additional support for the long, thin sliver created by cut 4 (coming up next). Without this support, the sliver would be held by wax on its bottom surface only, and the risk of losing the sliver during cut 4 would be increased.

- Cuts 4, 5, and 6: Make cut 4 on the opposite side of the ridge from cut 3, again placing the cut about 60 µm from the ridge and cutting through to the dummy piece. This cut defines the second "shoulder" of the sliver. Finally, rotate the sample 90°, and make cuts 5 and 6 such that the sliver length is 3 mm and the area of interest is centered in this length. At this point, prior to unmounting the sliver, it is useful to capture an image of the area of interest and ridge to guide subsequent FIB milling.
- Unmounting and cleaning: Place the assembly on the hotplate and use fine tweezers to extract the 3 mm sliver. Remove residual wax from the sliver using acetone followed by propanol. A good technique is to place the sliver in folded filter paper, and soak this in a small beaker of acetone for about 5 minutes; repeat briefly with propanol; then, finally remove the sliver and filter paper, and place them on the hot plate for a moment to drive off residual propanol. Sample is ready for FIB milling.

Standard plan view

Preparation of a site-specific plan view pre-FIB TEM sample uses similar shallow and deep cut component steps as in a cross section, but in different order. Several steps -- mounting, measuring, cuts 5 and 6, unmounting, and cleaning -- are identical for plan view samples as cross sections. Figure 2 (right side) illustrates the following steps.

- Cuts 1 and 2: Make a pair of deep cuts on either side of the area of interest, with the first cut 5-10 µm from the area of interest. The position of the second cut is such that a bar of material is defined with a width about 70 µm greater than its depth.
- Rotate and squish: Place the assembly on a hot plate and carefully rotate the bar 90° about its long axis such that the area of interest is still exposed. Squish the three pieces together so that the bar will be supported by wax on the bottom and two sides. Note that after rotation about 70 µm of the bar, including the area of interest, is exposed above the original sample surface.
- Cuts 3 and 4: Make a shallow cut 3 to define a ridge 20-25 µm wide; note that one side of this ridge is the original top surface of the sample piece. Depth of this cut is 50-70 µm depending on material. Make a deep cut 4 about 100 µm from the ridge and through to the dummy piece. Proceed with cuts 5 and 6 as for cross section samples.

Extensions to the standard method

Batch processing of cross section samples is possible by waxing down several sample pieces in a diagonal line on a single dummy piece. The diagonal arrangement prevents any of the six cuts of one sample from damaging any other sample. Cuts 1, 2, and 3 are completed on all samples, and then the squish step is performed on all samples. Next, cuts 4, 5, and 6 are completed on all samples, after

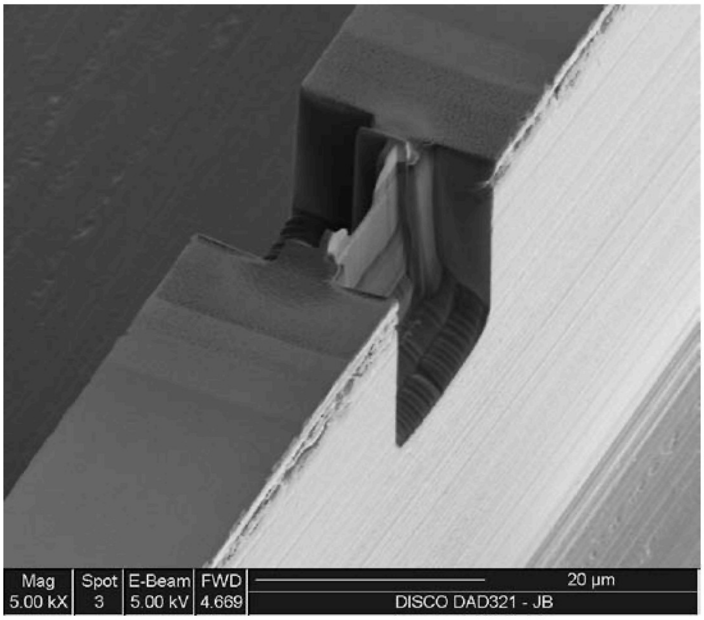

Figure 4. Final thinned sample ready for TEM imaging.

which each sample is demounted and cleaned in a separate beaker. We have achieved our fastest pre-FIB processing times with this method, often averaging less than 15 minutes per sample.

An area of interest arbitrarily close to the edge of a sample piece can be prepared using the dicing saw if the restriction on the use of adhesives is relaxed. In this case, cuts 5 and 6 are adjusted to produce a shorter (1.5-2.0 mm) sample sliver with the area of interest at one end; the other end is attached to a TEM half-grid using M-Bond 610 adhesive, thus placing the area of interest near the center of the half-grid. As an alternative, the original sample piece can be bonded with epoxy side-by-side to a second piece, after which this subassembly is prepared by the standard process.

A custom sample holder that we have developed for this type of pre-FIB sample has also helped us improve the speed and cleanliness of this process. Our current design holds up to six TEM slivers for FIB milling. Samples are loaded merely by pulling back a "soft" spring-loaded clamp, dropping the sliver into a slot, and gently releasing the clamp. The metal surfaces of the finely machined clamp are in direct contact with the sample sliver, thus providing excellent grounding of the sample during FIB milling. We had earlier used double-sided SEM adhesives to mount the sliver on the surface of a standard FIB holder, but our standard process is now completely free of adhesives.

Blade behavior and process limitations

The diamond resin composite blade used here has a nominal width of 200 μm, but we find that a new blade can give cuts as wide as 225 μm. The kerf stabilizes near 200 μm after cutting a few samples. The blade profile, shown in Figure 3, is easily determined by making shallow cuts in a silicon dummy piece, cleaving the piece, then examining the cuts edge-on in an optical microscope. As the blade wears, we observe that the sidewalls become tapered (non-vertical) and the bottom nonplanar: irregularities that may affect cutting accuracy. The profile can be "sharpened up" by a few passes of the blade on a dressing board that controllably erodes the blade to the optimum shape.

We also monitor blade exposure over the lifetime of a blade. New blade exposure in our saw starts at 1.30 mm; we replace the blade when this exposure falls to about 0.85 mm, since our samples are typically about 0.75 mm thick. With experience, we have found that we can extend the life of a single blade to over 1 000 cuts, or roughly 120-150 individual samples, after occasional kerf checks and blade dressing operations are included.

Multilayer samples with a weak interface may be a problem for the dicing saw technique. Delamination in such a sample will be evident in the cross section process after completing cuts 1 and 2; if this occurs, the sample is usually best made by FIB in situ lift-out. Materials affected by water (from the cutting blade) or by cleaning solvents may require alternate processing. Over 95% of the TEM samples (Figure 4) in our lab are prepared using the dicing saw, with most of the remainder fabricated by *in situ* lift-out or by the more traditional grinding, polishing, dimpling, and milling process. ■

References

1. Tsung, L., A. Anciso, B. Davidson, R. Turner, T. Alqaq, and A. Skloss. 2000. FIB/TEM Sample Preparation Using a Wafer Dicing Saw. Microscopy and Microanalysis Proceedings, vol. 6, supp. 2.
2. Model DAD321 from Disco Corporation, <http://www.discousa.com/>.