

TEM Sample Preparation Tips

Brennan L. Peterson, Ph.D., Process/Applications Development

When preparing samples for the transmission electron microscope (TEM), obtaining samples of uniform thickness (from 10–200 nm) is critical. There are a few different sample geometries, the most common of which is thin foils, or lamellae.

A broad literature exists on the exact methods and some of the tradeoffs around creating lamellae. Mayer, Giannuzi, et al. (2007, MRS Bulletin) detail many of the more intricate decisions. Extensive literature and application notes exist, and it is not the goal of this work to describe this literature in detail.

Rather than a detailed primer on step-by-step lamella creation, this document offers an overview of tips, tricks, and standards for the advanced practitioner.

Some critical requirements and restrictions on TEM preparation are:

- Positioning accuracy
- Lamella thickness
- Preparation speed
- The amount of amorphous material left from the FIB process
- Removal and placing on TEM grid
- Tools setup

Focused ion beam (FIB) methods are generally faster than manual preparation, with exceedingly high placement accuracy. Using the best methods and tools, placement accuracy of <25 nm (1 sigma) can be achieved, with placement of <10 nm possible in fully automated environments. For perspective, consider placing a 90 nm feature in a 120 nm lamella. To place the feature correctly, you would need placement accuracy of <30 nm. To place a 45 nm feature in a 60 nm lamella, the placement accuracy needs to be <15 nm.

In a similar trend for thickness control, demand from the semiconductor industry is driving significant increases in tool requirements. On non-aberration corrected TEMs, lamella thickness often needs to be <40nm to avoid edge and

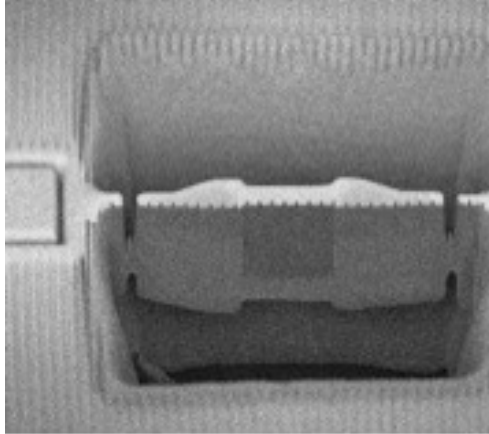


Figure 1 – Example of TEM lamella.

interface effects. For S/TEMs (scanning/transmission electron microscopes), this requirement is somewhat relaxed, but sample restrictions often result in the need for <30nm preparation.

Most critically for the lab manager, total TEM requests continue to increase, and total productivity (measured in samples/capital dollar or samples/lab area) needs to increase equivalently. While new tools can fill part of that gap, best methods and enhanced automation can dramatically improve the productivity for many labs.

Preparation details

Although this list is not absolute, Figure 2 gives a good indication of the major steps involved in lamella preparation and the rough time associated with each.

Step	Minutes	Notes
1. Locate	2-200	Sample location may include multiple cross-sections to find a site.
2. Protect	5	Coat with metal cap layer, add fiducials
3. Rough mill	5	Create 2 mu lamella
4. Medium mill	3	Thin to 250-400nm
5. Undercut	2	Release sample from substrate
6. Fine mill	2	Thin to final thickness
7. Endpoint	2-60	Thin to target location and thickness
8. Clean	3	Low kV cleaning of lamella
9. Sample transfer	3-30	30 min: Omniprobe <i>in situ</i> 3 min: <i>Ex situ</i> liftout

Table 1 – Lamella preparation steps.

The critical steps for timing are the highly manual steps to target the location of the lamella—either as part of manual thinning, or as an initial step. These steps can take as much as three hours, dwarfing any gains from automating the remainder of the process.

Step 2 (protective deposition and fiducials) generally involves adding a tungsten or platinum cap, as well as surface and buried fiducials (fixed reference points). An effective fiducial creation strategy can have a large impact on the robustness of an automated process. Different shapes have very different behaviors.

The rough, medium, and fine milling steps use decreasing FIB beam sizes to improve control. As in all applications, there is a tradeoff between slow etch time (which causes increased drift sensitivity) and beam resolution. In general,

it is a good rule of thumb to target no less than five seconds for a mill, and to target beam size using the largest appropriate aperture.

A typical recipe or program uses 13 nA for bulk milling and undercutting, 1 nA for medium thinning, 30–100 pA for fine milling, and 3kV–120 pA (using 1000pA aperture) for final cleaning. Variations (3 nA instead of 1 nA for medium thinning) are possible, depending on user preference and the details of the available aperture strip.

For the timings listed above, fully automated processing in arrays should use the lowest timings and skip the endpointing stop. This results in ~20 minute preparation times. Faster is possible, again depending on the exact application and targets.

Positioning accuracy

The position of the TEM lamella is critical to find an isolated feature, or to fully encapsulate a reference feature (for instance, a row of contacts). In cases where only a single feature in an array is needed, the lamella can easily be prepared at a slight angle to the feature.

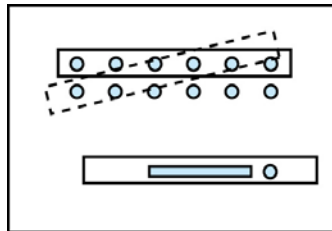


Figure 2 – Positioning the lamella at an angle to more easily capture a single feature.

With FIB systems, the lamella can be positioned relatively accurately within the instrument's mean drift (including both sample drift, due to imaging or charging, and stage drift).

Through fiducials can be used to improve location of a particular feature during cross-sectioning. During final thinning, the user can thin until the precut (and filled) fiducials are seen in a cross-section image. These lines are usually milled at ~100 nm width, which forms a good basis for judging final thickness. To form these sorts of through fiducials, small (<100 pA) beams and relatively short dwell times are used. These fiducials can be conveniently scripted.

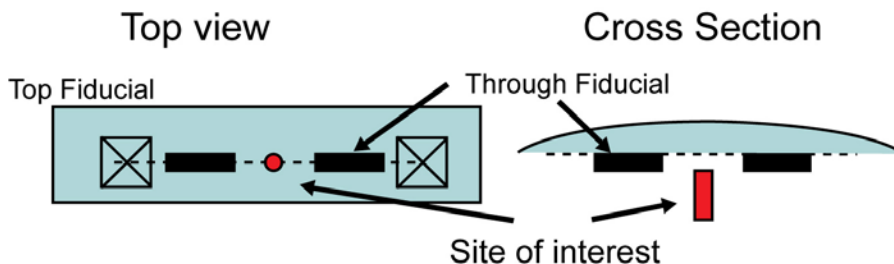


Figure 3 – Top fiducials and through fiducials for easy registration.

With dual-beam systems, the SEM or S/TEM (scanning transmission electron microscope) can be used to improve the registration by stopping the thinning at a particular image location. Without a dual beam for final location, the measured accuracy is on the order of 50 nm (3 sigma) for top-down FIB-prepared systems. Refinement in a small dual beam can allow a practiced operator nm-level placement.

To measure placement accuracy, there are a few different metrics: the placement of the fiducial relative to the feature, as well as the placement of the final lamella as compared to the initial fiducial. Overall placement can also be judged by measuring the final placement of a known reference feature within a lamella.

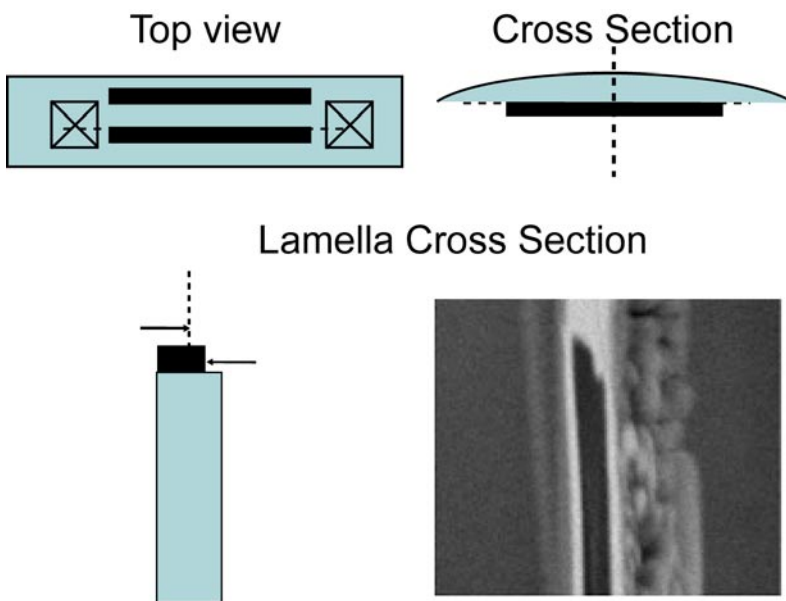


Figure 4 – Cut placement measurement via cross-section of a line.

Lamella thickness

Lamella thickness is critical for accurate TEM metrology, and to a lesser extent, for S/TEM imaging. In transmission techniques, analysis is greatly simplified for single scattering events. In addition, the sample attenuates the beam. Roughly speaking, S/TEM samples generally target approximately 80 nm. For TEM, samples generally range from 20–80nm, depending on the specific sample and TEM tool.

Thickness is usually measured either by cross-sectioning the thin lamella, or by measuring the electron beam attenuation (this requires an accurate calibration standard). Quoted values in 1/e lengths center roughly on 100 nm +/- 20%.

Top-down measurement of TEM sample thickness is heavily influenced by the protective overcoat, and is only approximately accurate (30%). For accurate and reliable measurement, cross-sections of lamella (see Figure 5) are much more accurate and useful.

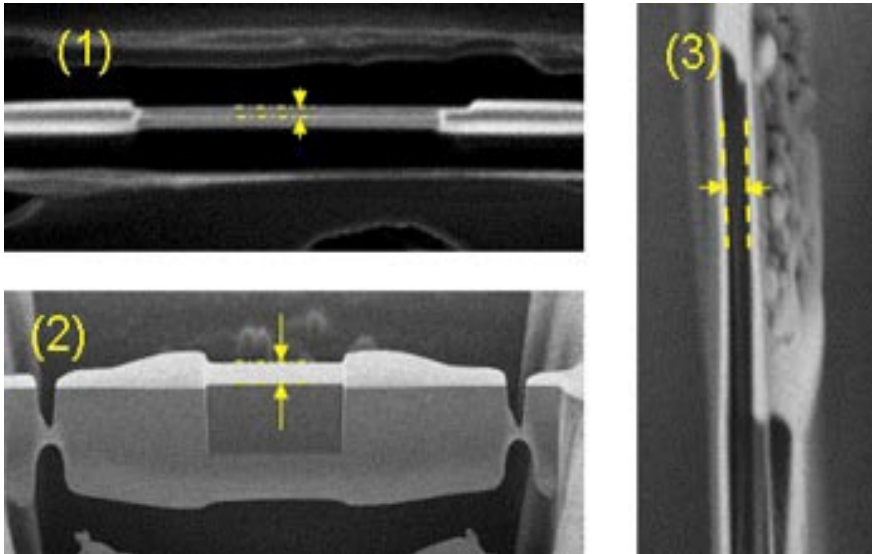


Figure 5 – 1) Top-down measurements. 2) Heuristic protective cap measurements. 3) Cross-sectional measurements (most accurate).

Uniformity of thickness is also important, although sometimes samples are intentionally made with varying thickness. This can simplify preparation (it is easy to make a wedge, vastly harder to make a uniform 10 nm sample) but must be taken into account when interpreting data.

Preparation speed

For large-scale lab implementation, preparation speed is a useful quantifier of the process. However, TEM preparation speed varies depending on the nature of the sample. In particular, overall preparation time is affected by how long it takes to remove the sample and find the area of interest. Depending on the exact use case and desired thickness, different preparation speeds are possible.

To measure preparation speed, it is important to specify and understand the removal method (plucking with a glass rod is fast, in situ transfer to a TEM grid is slow) as well as any special conditions necessary for preparation.

Table 2 shows a breakdown of preparation times. More details can be found in Table 1. Because preparation needs differ dramatically between sites, these times offer at best a guide.

It is worth noting the significant differences between the different preparation times. The most fundamental difficulty and time-determining step is site-specificity.

The fundamental rate limit is easily calculated. To carefully mill and image takes ~15 seconds per step. These mill steps need to be ~2–5 nm. If placement is within 50 nm and a bit of safety margin is added, between 10 and 30 minutes of milling will be needed per side to locate a feature.

This is much more important when the features (as in post-package defect analysis) are not easily visible top-down. In this case, as much as 2 microns of material may need to be investigated. This can easily require 2–3 hours of dedicated searching for defects in cross-section. Cut placement really is key, and it is critical to place the initial cross-section of lamella accurately in order to maximize productivity.

Preparation type	Notes or examples	Time
Non-location specific	TEM prep along trench	~20 minutes, fully automated
Electrical defect (isolate)	VC defect isolation, thermal failure, post-package failure analysis. Post-etest analysis. Particularly difficult to find line defects.	>4 hours, depending on difficulty in defect location
Site-specific	Single contact/gate isolation	~1-2 hours
<30 nm	Higher difficulty	2 additional hours, increased failure rates

Table 2 – Approximate times for different preparation types. Note the wide gaps in potential preparation times.

Amorphization

FIB-based preparation systems leave behind a damaged surface after milling. The thickness of this damaged layer depends on the orientation of the FIB beam to the sample surface (typically a few degrees) as well as the beam energy. The damage is amorphized material, caused by the gallium beam, and the damage depth in nm is approximately the same as the FIB beam energy in kV. For instance, a 30 kV sample will give approximately 30 nm of gallium implantation and amorphization. At lower angles (normal milling) this value is smaller but still significant.

For perspective, a 50 nm sample will be almost completely amorphized by a normal 30 kV beam (20 nm per side). With the FIB used to clean the sample at 5 kV, this is reduced to 5 nm of total damage (or 10%). For 2 kV cleaning, this is further reduced to ~2 nm. See extensive work by Mayer, Gianuzzi, et al. (MRS Bulletin 2007).

For reference, compare Figure 6, which shows the effects of 5 kV deposition and SEM deposition (SEM leaves no damage). This is a proxy for in-plane damage to a FIB sample, not a direct image. It does show the same key effects—namely, that the FIB does damage surfaces and interfaces, which is one of the key challenges in sample preparation.

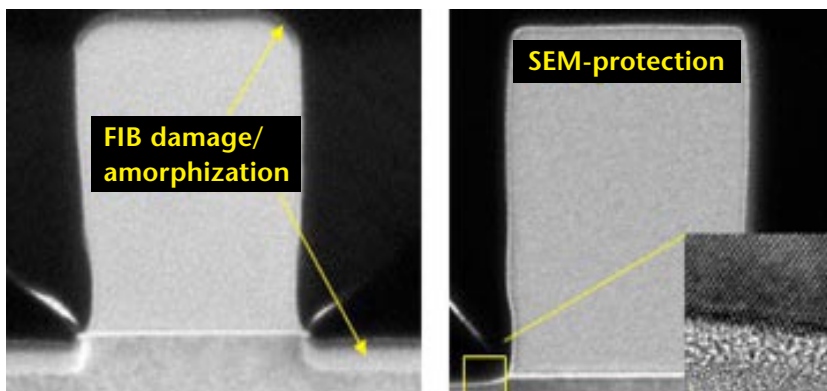


Figure 6 – Top-down view of FIB damage, showing ~1.5 nm of amorphization per kV of energy at normal incidence, and ~.5nm/kV at 88 degrees.

To compare success, it's useful to use the TEM's capability to look at the silicon "dumbbell." This feature is fairly indicative of both good TEM alignment and good preparation. If the sample is prepared well, the Si dumbbell is easily visible in a TEM with <1.4 nm resolution.

As a practical note, one of the primary challenges in cleaning samples is the difficulty of imaging and finding the sample at the very low voltages used to minimize sample damage. Depending on the geometry used, it is not critical to mill only the sample—the low-energy FIB can be treated as a broad beam source. This can significantly simplify some situations. There is some redeposition risk in this approach, which may actually be useful in many cases.

Automated preparation

Automated preparation follows the same steps as above, but uses fiducials to place mills on systems. In general, samples are prepared either for glass-rod extraction, or by creating chunks (3 μm wide thick lamella) followed by automated thinning.

At FEI, we use different automated preparation methods depending on the system in use.

- AutoTEM uses the AutoFIB scripting system on Windows XP and XT software systems.
- AutoTEM G2 is a simplified script-based system on Windows XT tools (Helios and Strata families), which is targeted at simple installation and application.
- IC3D is used on Certus/CLM tools, and is an alternate scripting system aimed at very high volume use (over 50 lamellae per day, with potential of four per hour).

Tool setup

One critical and often ignored component of good lamella preparation is accurate tool setup. Over the course of a four-hour lamella preparation, it is typical to spend 15 minutes or more refocusing and stigmating beams.

In TEM preparation, here are a few things that can make this simpler:

- Capacitance probe systems can automatically and accurately reset height. This keeps the FIB in focus at all times.
- Set up the tool at the FIB's eucentric point. Tilt stage systems have an option to set the eucentric point based on the coincident point, the FIB's eucentric, or the SEM's eucentric. TEM preparation is helped dramatically by setting the tool according to the FIB's eucentric point. This avoids beam shift and motion during sample preparation.
- Maintain tools well. It is imperative to make sure beams are well set. Less than nm variation in results is only possible if tools are kept in shape. Automated maintenance/PM routines are very useful in this regard—particularly FIB focus, stigmatation, and beam coincidence.

Sample transfer and storage

Sample transfer mechanisms fall into two general types: the *in situ* autoprobe, and *ex situ* plucking with a glass rod. Each method has benefits, depending on the application target. *Ex situ* liftout is typically significantly faster.

Endpointing

Endpointing in TEM preparation typically uses the SEM signal to look at either secondary or backscattered electrons from a sample, and correlates these to thickness. The easiest method is simply to use the brightness: very thin samples become either dark or bright (depending on detector layout and source) once thinned past ~100 nm. The exact value depends on the beam energy and imaging mode.

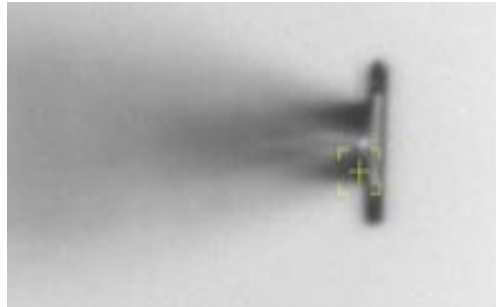


Figure 7 – Glass rod plucking of samples.

Similar techniques can be used with the integrated S/TEM detector system, with the added advantage of enhanced resolution over SEM-based techniques. The S/TEM system allows direct thickness measurements to be made from first principles, or contrast differential techniques can be used in darkfield.

Conclusion

Following these tips and tricks can greatly enhance productivity in TEM sample preparation by helping you achieve critical uniform thickness. For more on these methods and other helpful information from FEI, visit <http://wiki.fei.com>.

References

Mayer et al. 2007. TEM Sample Preparation and FIB Induced Damage. MRS Bulletin 07 V32.

© 5/2008
03WP-TE0111

FEI Company

World Headquarters and
North American Sales
5350 NE Dawson Creek Drive
Hillsboro, OR 97124-5793 USA
Tel: +1 503 726 7500
Fax: +1 503 726 2615

fei.com/sales

European Sales
Tel: +31 40 23 56110
Fax: +31 40 23 56634

Asia-Pacific Sales
Tel: +86 21 6122 5988
Fax: +86 21 6122 5999

Japan Sales
Tel: +81 3 3740 0970
Fax: +81 3 3740 0975

