

Ultra-fast in situ S/TEM sample preparation

Complete sample preparation in under 30 minutes



The DualBeam[™] (FIB/SEM) offers the capability of both highly localized sample preparation and high resolution sample analysis. Site specific cross-sections and S/TEM samples through disparate materials can be prepared using FIB milling, while simultaneous SEM imaging can be used to monitor the sample preparation, allowing the section to be precisely positioned.

With decreasing feature sizes in all areas of nanotechnology, the need for S/TEM analysis is dramatically increasing. Also, as the variety and complexity of specimen increases, so too does the difficulty for traditional S/TEM sample preparation methods.

The use of DualBeam therefore is of great importance to enable production of high quality S/TEM specimen, but it also enables high throughput.

There are several different ways to produce S/TEM specimen using a FIB, but the most reliable and popular method is the in situ lift out method. In this method, the area of interest is initially cut out with the FIB, before attaching it to a micromanipulator, and then transferring it to the S/TEM grid.

Method

By utilizing the high beam current density provided by the FEI Sidewinder[™] and Tomahawk[™] ion columns, the entire sample preparation (from selecting the area of interest, to finalizing the low energy cleaning step of the sample on the grid) can be performed in under 30 minutes (times given apply for a Si based sample; samples with lower sputter rates will take longer to process).

Key benefits

- Complete sample preparation in under 30 minutes (non-site-specific in Si).
- HR TEM/STEM is attainable with final sample quality.
- High reproducibility and success rate.



Step 1: Initial sample preparation

- Once the area of interest is selected, a protective layer of platinum is deposited.
- Two large trenches are milled on either side of the region of interest and a cleaning cross-section is applied while applying sample tilt into the beam to maintain parallel side walls (total time to this step ~ 7.5 minutes).





Step 2: Lift out procedur

- A micromanipulator is inserted and attached to the sample by using a small Pt deposition. The lamella can then be released and lifted free from the bulk sample.
- The S/TEM grid is then centered and the lamella is attached to it by using two small platinum depositions. A final releasing cut completes the lift out procedure and the sample is now ready for final thinning (total time to this step ~ 18.5 minutes).



Step 3: Final thinning

- Final cleaning cross-sections are applied to both sides of the lamella using 30 kV and tilting the sample into the beam.
- To complete the sample, a low energy cleaning step is applied to both sides of the lamella to remove any residual amorphous material (total time to this step ~ 30 minutes).





TEM results (obtained using FEI Titan[™] 80-300 microscope)







 $\rm C_s$ corrected TEM image of FIB prepared Si sample. Total preparation time including 2 kV low energy cleaning step was below 30 minutes.

Thin sample was measured to be approximately 30 nm thick using energy filtered TEM. Image shows Si lattice using zero-loss filtered HR-TEM.

HR-STEM image shows the Si dumbbells. The line profile confirms the dumbbells are 0.136 nm apart.



Conclusion

- Complete S/TEM sample preparation possible in under 30 minutes (non-site specific in Si).
- Experience gained from the process described here can also be applied to understand the limiting factors on preparation time on more demanding materials. This will enable a faster throughput than otherwise achievable.
- HR TEM/STEM is attainable with final sample quality.
- High reproducibility and success rate.





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