

## Two Methods for Measuring Lamellae Thicknesses *In situ* for Improved FIB Specimen Preparation

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Scanning/transmission electron microscopy (S/TEM) has demonstrated remarkable capabilities in elucidating fundamental understanding in a range of fields.[1, 2] Advances in S/TEM instrumentation, have made recording atomic resolution images routine if not trivial. To fully exploit the increased imaging and spectroscopy capabilities of current S/TEM instruments, requires a very thin and clean, undamaged specimen that is representative of the original material. A wide range of techniques are available to prepare S/TEM specimens, with the optimal method determined by the material and desired viewing orientation. The focused ion beam (FIB) is a highly versatile instrument for the preparation of thin S/TEM lamellae with unrivaled site specificity.[3]

One of the most challenging aspects of FIB preparation is determining whether the thickness of the lamellae is appropriate for S/TEM analysis, typically 20 – 80 nm[4]. Accurate and precise lamellae thickness determination is not only crucial for preparation of high quality specimens, but also for subsequent interpretation and analysis of the collected data. Currently this assessment is heavily reliant on operator experience, interpreting changes in specimen morphology and contrast observed in scanning electron microscope (SEM) images. This seriously limits the reproducibility of the specimen preparation, especially the accuracy of lamellae thickness. Previous attempts to measure specimen thickness *in-situ* in the FIB SEM are experimentally challenging, of low accuracy or require the addition of expensive equipment.[5-7]

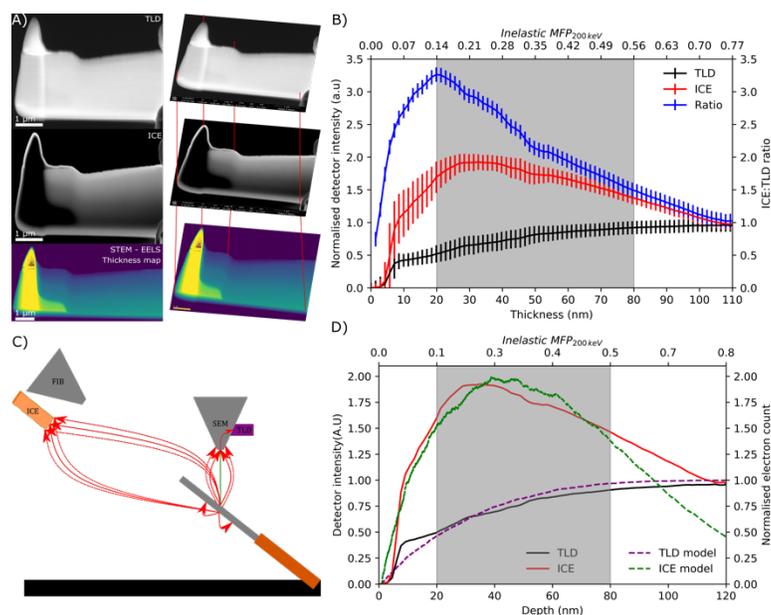
Here we present two methods for the accurate and near real-time *in-situ* measurement of S/TEM lamellae thickness inside the FIB-SEM. We first demonstrate a calibration method which allows for the accurate assignment of lamellae thickness based on detector intensity values. This was achieved by acquiring secondary electron (SE) SEM images of a silicon wedge lamellae using both the through lens detector (TLD) and in chamber electron (ICE) detectors. Subsequently, electron energy loss spectroscopy (EELS) was used in the STEM to gain a log-ratio thickness map of the lamellae, and the two data sets were aligned in order to assign thicknesses to the TLD and ICE detector values (Fig 1A). The EELS data was collected using a FEI Titan 80-200 fitted with a Gatan Quantum-ER imaging filter operated at 200 kV, 180 pA, with  $\alpha$  and  $\beta$  angles of 21 and 62 mrad respectively. The SEM ICE and TLD SE images were collected using a FEI Helios 660, operated at 5 kV. The TLD and ICE detectors exhibit strikingly different responses as a function of lamellae thickness (Fig 1B). This difference in behavior is due to the detector geometry which changes the availability of SEs (FIG 1C). The TLD displays the ideal monotonic response, such that a detector intensity value corresponds to a single thickness value. Whereas the ICE detector has a non-monotonic response, where a single intensity value may correspond to multiple thickness values. This is further complicated by a spread of grayscale values, such that a single thickness value may be mapped to a spread of detector intensities rather than a single value, introducing an uncertainty in the estimated lamellae thickness. The ICE response has smaller errors when compared to the TLD, s (errors of  $\pm 8.73$  and  $\pm 19.32$  nm for ICE and TLD respectively), except for thicknesses around 25 – 40 nm (the turning point in the ICE data), where the assigned thickness error become large ( $>20$ nm). We demonstrate that errors can be reduced to  $\pm 4.12$  nm, in the region of 25 – 80 nm, by taking the ratio of ICE:TLD.

While this calibration method offers several advantages over existing methods, it is specific to the individual material-instrument pair, and requires the lengthy process of preparing and measuring a calibration lamellae. Our second approach, uses novel Monte Carlo simulations[8] to model the detector responses as a function of lamellae thickness, thus eliminating the requirement for a calibration sample. We successfully model the TLD by considering the SE electrons produced from the top surface, while the ICE is modeled by both the top and bottom surface SEs (Fig 1D). We show that the top surface signal is proportional to the number of back scattered electrons, and the bottom surface SE signal is proportional to the number of electrons transmitting through the specimen. We outline a framework for incorporating these techniques into the workflow of lamellae production, through leveraging the increasing availability of python scripting APIs on FIB instruments.

In summary, we have demonstrated two methods for the *in-situ* measurement of lamellae thickness in the FIB-SEM. The first approach is based on the calibration of the ICE and TLD signals and the second is based on Monte Carlo simulations of the SE signals. We show that these methods can be used to estimate lamellae thickness *in situ* and hence improve the accuracy and efficiency of S/TEM sample preparation.

#### References:

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**Figure 1.** A) Illustration of the alignment of TLD and ICE SE images to the EELS thickness map. B) TLD, ICE and ICE:TLD signals as a function of silicon lamellae thickness. C) Illustrative overview of the geometry in the FIB instrument. D) Comparison of experimental and modelled data for TLD and ICE detector signals as a function of silicon lamellae thickness.