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Robotic Fabrication of High-quality Lamellae for Aberration-corrected Transmission Electron Microscopy

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Aberration-corrected scanning electron transmission microscopy (STEM) is widely used for atomic-level imaging of materials but requires damage-free and thin lamellae. So far, the preparation of such high-quality lamellae from a bulk material largely depends on manual processes by a skilled operator. This limits the throughput and repeatability of aberration-corrected STEM.

Focused-ion-beam (FIB) is a common method to prepare high-quality STEM lamellae [1,2]. Automation of FIB operation for preparing high-quality lamellae has become highly desirable in this era of data-driven discovery of materials [3]. However, there has been no experimental demonstration of “full automation” combined with the “high-quality” needed for aberration-corrected STEM. Here, we show that the latest automatic FIB can prepare 50-nm-thick lamellae from Si, $SrTiO_3$, and Al_2O_3 without the attendance of a human operator.

Figure 1 summarizes the automation processes of rough-milling, lift-out, and final-thinning at 30 kV, 5 kV, and 2 kV. Before the automation starts, the user sets FIB parameters, such as the chunking position, the chunk size, the grid position, the target thickness, the acceleration voltages / beam current of the final polishing, and the over-tilt angle. Once these parameters are given, the latest automation software (Thermo Scientific AutoTEM 5) operates a FIB system (Thermo Scientific Helios 5 UX) without the attendance of an operator. The throughput of preparing a lamella is typically 1 hour. In total, 52 lamellae were prepared and 51 lamellae were successfully completed. Thus, the automation system is very robust even when the material is non-conductive.

The quality and overall repeatability of the resulting lamellae was evaluated by aberration-corrected STEM (Thermo Scientific Spectra 300). The thickness at the bottom edge ranges from 40 nm to 50 nm in the three samples (Figure 2). In atomic-level STEM imaging, all samples showed dumbbell structures of Si atoms with 55 pm spatial resolution (Figure 3). Automatic FIB was also used to successfully prepare high-quality lamellae from $SrTiO_3$ and Al_2O_3.

This study has demonstrated reliable fully-robotic FIB preparation of STEM samples from crystalline materials. Such advanced automation technology will pave the way for operator-free, high-throughput, and repeatable preparation of high-quality lamellae for aberration-corrected STEM.

Note: The full article is available in [4].

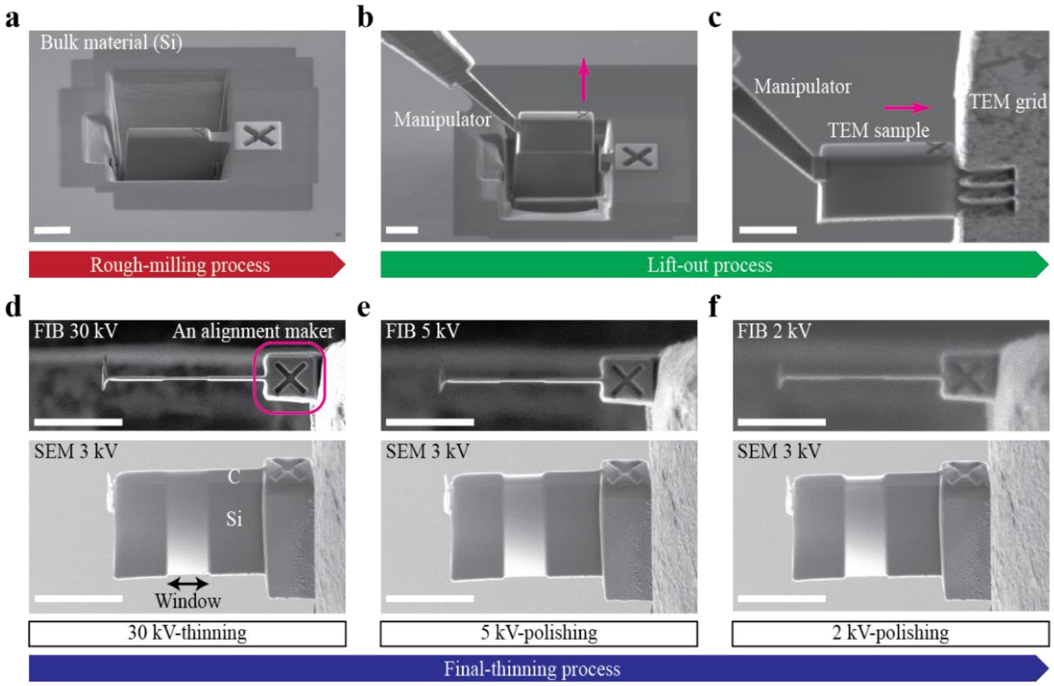

Figure 1. The main steps of AutoTEM 5 automated sample preparation: (a) rough-milling, (b) and (c) lift-out, (d) – (f) final-thinning. All scale bars are 5 µm.

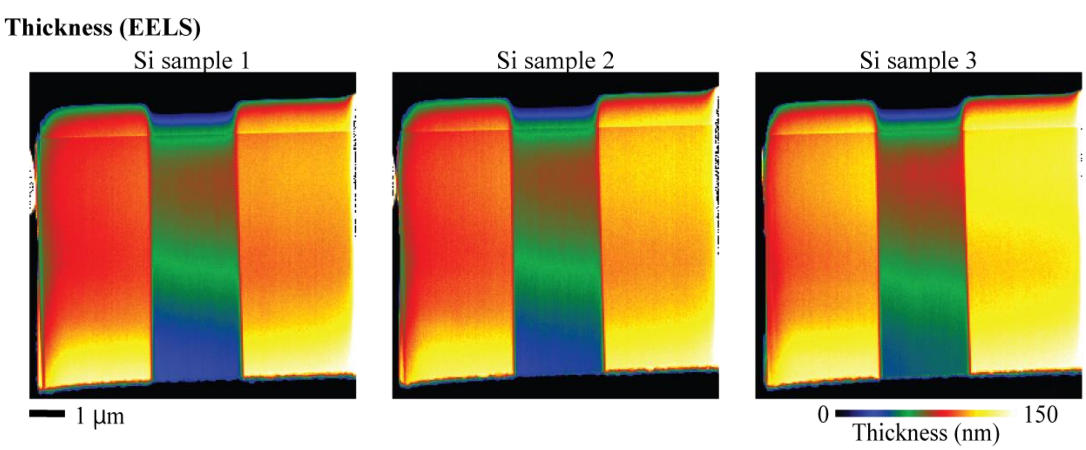

Figure 2. Shape and thickness of the three Si lamellae that were prepared by AutoTEM 5.

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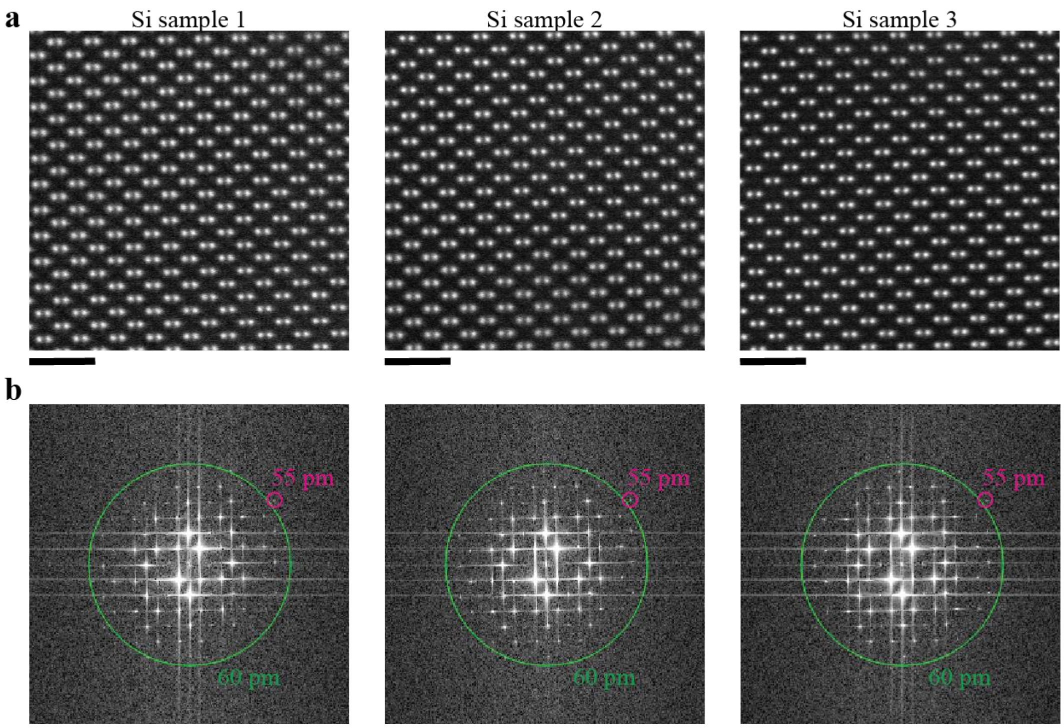

Figure 3. Aberration-corrected STEM results of three Si lamellae. (a) HAADF-STEM images and (b) their FFT patterns. All scale bars are 1 nm.

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