Concentrated argon ion beam milling
An alternative to broad argon ion beam milling for post-FIB polishing of TEM specimens

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Background

**Improve** ion milled TEM specimens by **lowering** the ion **accelerating voltage**


Broad vs. concentrated Ar ion milling

Broad ion beam (BIB) milling

Concentrated ion beam (CIB) milling
Model 1040
NanoMill®
TEM specimen preparation system

Ion beam image
500 eV

Raster box;
1 μm Ar ion beam

Ion beam image
300 eV

excellence...magnified
Model 1080
PicoMill®
TEM specimen preparation system

Secondary electron detector image

STEM detector image

Raster box; 600 nm Ar ion beam

Ion beam image
300 eV
Broad vs. concentrated Ar ion milling

Ion milling conditions

**BIB milling**
- 2.5 mm beam size
- 1000 eV for 8 min
- 500 eV for 8 min

**CIB milling**
- 1 μm beam size
- 10 x 10 μm milling area
- 900 eV for 45 min
- 500 eV for 30 min
Specimen and analyses

- Si specimens prepared in the FIB at 30 kV and 5 kV
- Energy filtered TEM (EFTEM) analysis for thickness measurements
- High resolution TEM (HRTEM) imaging and fast Fourier transform (FFT) analysis; FFT image processing
- Energy dispersive X-ray spectroscopy (EDS) at 300 kV using a low-background TEM holder
- Electron energy loss spectroscopy (EELS) at 300 kV
Thickness measurements

- After FIB milling at 30 kV, specimen thickness of 175 nm
- Material removed
  - BIB milling: 100 nm
  - CIB milling: 90 nm
- Amorphous damage of 25 nm removed
Thickness measurements

- After FIB milling at 5 kV, specimen thickness of 125 nm
- Material removed
  - BIB milling: 57 nm
  - CIB milling: 65 nm
- Amorphous damage of 5 nm removed
HRTEM images: Specimen quality

Specimen after 5 kV FIB milling. Image shows non-uniform contrast across the specimen and FFT shows a diffused halo in the background (amorphization).

Specimen after 500 eV BIB milling. Image shows moiré fringes and FFT still shows a diffused halo in the background, as well as additional spots.

Specimen after 500 eV CIB milling. Image shows homogeneous contrast and diffused halo is absent in FFT.
Image processing of FFTs

- FFT after BIB milling shows additional spots that are not from Si
- Image processing was performed:
  - Thresholding to highlight the diffraction spots
  - FFT after FIB milling was subtracted from the FFT after BIB milling
- Additional spots from Cu and Cu oxide due to BIB milling
EDS results

- Ga was removed by BIB and CIB milling
- Cu signal was significant after BIB milling

Cu redeposition occurred during BIB milling, while none occurred during CIB milling
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EELS results

- Normalized zero loss peak resulting in decreasing plasmon intensities, i.e. thickness reduction by BIB and CIB milling.

- Cu L edge observed after BIB milling, while signal is within the noise level after CIB milling.

This verifies Cu redeposition after BIB milling.
Key factors in BIB redeposition

- **Beam size.** Broad ion beam is significantly larger than the FIB specimen

- **Beam current density.** Broad ion beam current is $10^6$ times larger than condensed ion beam current ($\mu A$ vs. pA)

- **Beam dose.** Specimen is continuously exposed during BIB milling
Conclusions

- CIB milling of FIB prepared specimens is preferred over BIB milling because it results in:
  - High quality specimen with a homogeneous surface
  - Consistent specimen thickness through controlled and precise milling

- Both BIB and CIB milling removed amorphization and Ga damage from the 30 kV and 5 kV FIB milled specimen

- BIB milling can cause Cu redeposition as evidenced by:
  - Moiré fringes on the HRTEM images that correspond to Cu and Cu oxide spots on the FFTs
  - Significant Cu EDS signal as compared to baseline Cu signal from the FIB milled specimen
  - Presence of Cu L$_{2,3}$ edge in EELS data