

Removal of amorphous layer from nanoneedle specimens fabricated by focused ion beam

The nanoneedle sample configuration allows the use of multiple characterization techniques with a single sample. These techniques include nanoscale mechanical and electrical tests, transmission electron microscopy (including three-dimensional tomography) and atom probe/field ion microscopy. In each instance, residual amorphization and ion implantation must be minimized to obtain optimal results.

Producing the nanoneedle

The nanoneedle is produced by focused ion beam (FIB) milling with gallium (Ga) ions. A thin (> 100 μm) slice of the sample material should be mounted in plan view on an aperture grid.

Orient the material of interest so that the ion beam direction is perpendicular to the plane of the grid. Initial FIB cuts at 30 kV are made in a conical fashion until the needle geometry is created; the Ga ions should be parallel to the needle axis (Figure 1).

The sample diameter can be further reduced by additional milling at 5 kV with the ion beam directed perpendicular to this axis. The needle may need to be rotated and FIB milled in stages to produce a sample of uniform diameter. The tip radius should be approximately 50 nm for atom probe applications.

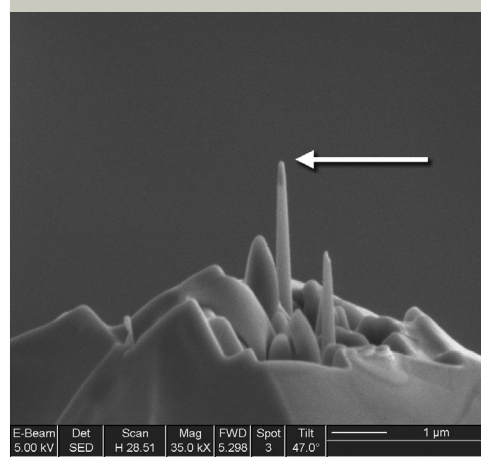
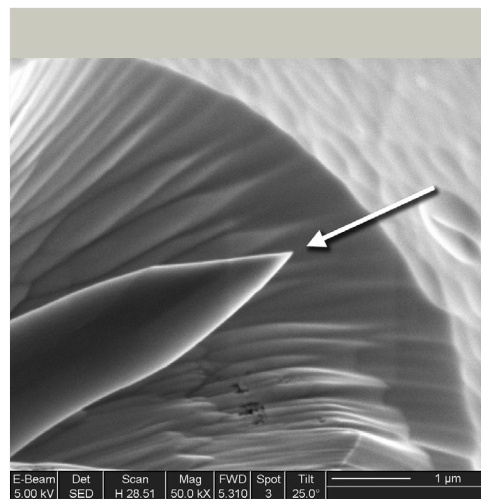
Final processing in the NanoMill® TEM specimen preparation system

The NanoMill system is ideally suited to removing Ga implantation and minimizing amorphous damage to the nanoneedle specimen.

Load the aperture grid supporting the sample material into the NanoMill system; the needle will be vertical with respect to the plane of rotation of the instrument's stage. The best practice is to ion mill at a low ion energy using a 0° tilt angle, rotate the specimen 60°, and repeat the ion milling step. This process should be repeated until all 360° of the specimen has been ion milled.

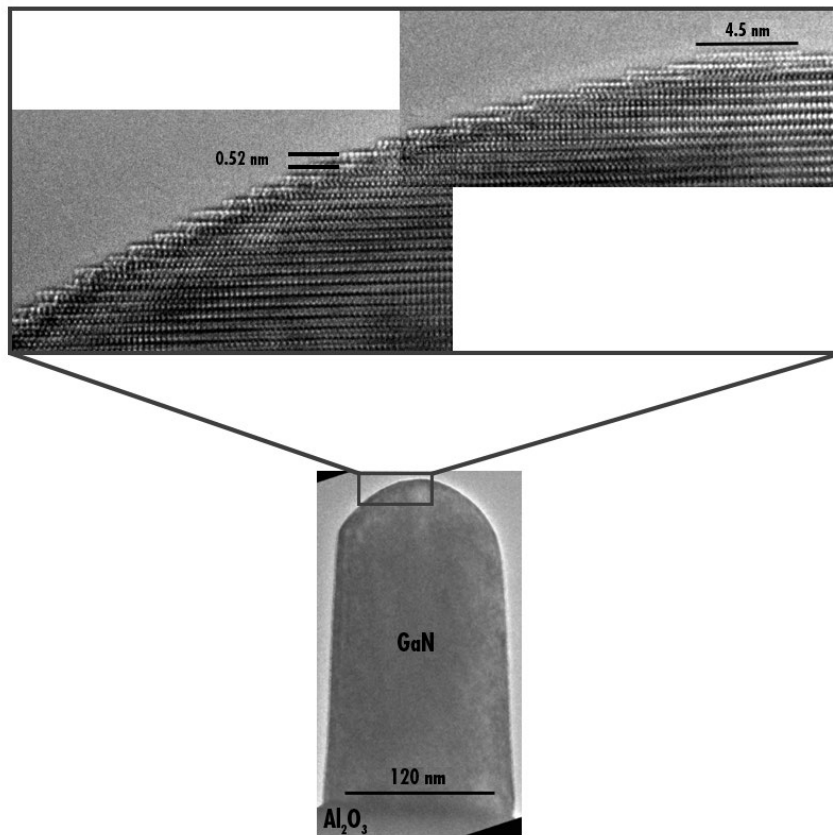
Ion milling parameters. The ion milling parameters vary by material, but a recommended starting point is:

Parameter	Setting
Energy	500 eV
	135 pA on Faraday cup
Raster box size	15 μm x 15 μm
Milling time	5 minutes per ion milling step



FIB MILLING GEOMETRIES

Figure 1. Initial FIB cut (top) and diameter reduction (bottom). Arrows indicate the direction of the Ga ions during FIB milling.



FIB-MADE NANONEEDLE

Figure 2. FIB-made nanoneedle (Pt/amorphous carbon/InN/GaN/AlN/Al₂O₃ base) prepared with the NanoMill system at 900 eV followed by 500 eV. The sample was imaged with the One Ångstrom Microscope at 300 kV at roughly Scherzer focus.

Image courtesy of D. Detert, P. Specht, O. Dubon, University of California, Berkeley, Berkeley, CA, USA; and C. Skelton, C. Song, P. Ercius, D. Alloyeau, C. Kisielowski, National Center for Electron Microscopy, Berkeley, CA, USA.



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The Model 1040 NanoMill® TEM specimen preparation system is the subject of United States Patent Nos. 7,132,673 and 7,504,623.

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