Advanced specimen preparation using a low energy focused ion beam for atomic-scale characterization and analysis

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Focused ion beam (FIB) specimen preparation techniques have been successfully used to generate ultra-thin lamellae for TEM and STEM, and there are now many examples in the literature. This approach can also be used to create ultra-fine, conical specimens suitable for atom probe experiments. One example is the determination of the architecture of quantum well structures, where time-of-flight atom probe microcopy can be used to generate a 3-dimensional reconstruction at the atomic level [1].

FIB milling is often performed using high energy Ga^+ ions (i.e., 30 keV), which can impart damage up to ~20 nm at the surface of a specimen such as silicon [2]. Given that both the upper and lower surfaces of a lamellar specimen carry this damage, it can easily be sufficient to impede quantitative high resolution S/TEM imaging and is especially problematic for analytical techniques such as electron energy loss spectroscopy (EELS), where the total specimen thickness should ideally be on the order of 30 nm or so.

The advent of aberration-corrected S/TEM instruments capable of sub-Angström resolution [3, 4] makes it ever more imperative that specimens are prepared to the highest possible standards and, clearly, methods are needed to minimize both ion implantation and surface damage if we are to obtain meaningful results. Amorphization damage in silicon and gallium nitride has been shown to be removed by chemical wet polishing after FIB milling, but chemical polishing methods are material dependent and are difficult or impossible to use for complex multi-phase or multi-layered specimens. Broad beam ion milling is another approach to removing damage post-operatively [5-7].

However, perhaps a better solution would be to reduce the overall introduction of artefacts in the first instance, by FIB milling at lower ion beam energies. Recent advances in FIB column design allow us to go to beam energies as low as 500 eV whilst still maintaining a good beam profile. Hence, if a low energy ion beam is used for the final cleaning step in the specimen preparation process, any surface damage from high energy milling can be removed and replaced by a much smaller amount of damage.

A study of silicon cleaned with a focused 2 keV Ga^+ ion beam shows sidewall amorphization to be as little as ~ 1-2 nm or so [2]. Whilst not eliminating the damage completely, the results are within acceptable limits for high-resolution imaging and analysis. The concept is illustrated schematically in figure 1, while figures 2 and 3 demonstrate that ultra-high resolution can be achieved in the S/TEM using FIB-prepared specimens.

References

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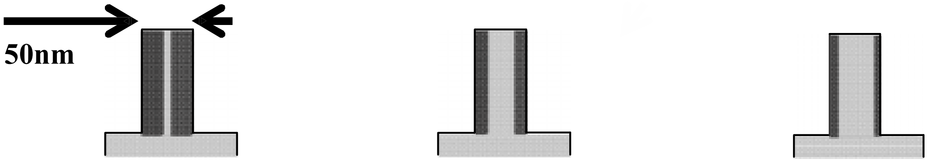

Little crystalline material left

>80% crystalline material left

>90% crystalline material left

Figure 1. Schematic diagrams to illustrate the effect of FIB milling a thin, lamellar specimen at successively lower ion beam energies. Left-to-right: 30 keV, 5 keV and 2 keV, respectively. Damage and ion implantation reduces with beam energy, to yield specimens suitable for high-resolution imaging and analysis.

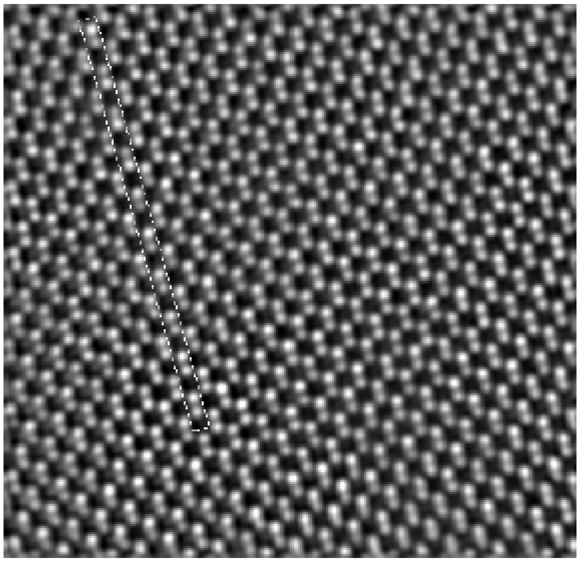

Figure 2. C_s-corrected TEM image of silicon, showing sub-Angström resolution. The lamellar specimen was prepared using a focused Ga^+ ion beam. For the final cleaning step, an energy of 2 keV was used, resulting in a very high-quality specimen.

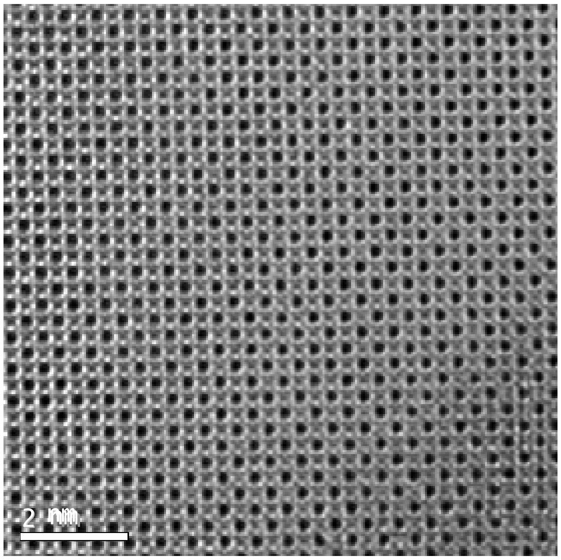

Figure 3. C_s-corrected TEM image of Lanthanum hexaboride, again prepared using a focused Ga^+ ion beam, with a final cleaning step at an energy of 2 keV.