doi:10.1017/S1431927611004521
Microsc. Microanal. 17 (Suppl 2), 2011
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Importance of the Protective Layers and the Specimen Preparation for Reproducible APT Results

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The specimen preparation and the use of protective layers are rarely discussed in the literature, but are crucial to achieve reproducible results in the atom probe tomography (APT).

If the region of interest (ROI) is at the surface of the sample, cap layers have to be deposited to protect the ROI during the specimen preparation in the focused ion beam (FIB). Chosen cap layer materials have to fulfill several requirements: good adhesion, no intermixing with the ROI, no mass peak overlaps with the ROI and good gallium stopping during the FIB milling. Good adhesion is crucial, as the APT measurement puts the analyzed material under a high stress. The cap layers deposited with electron beam deposition have besides the high purity and good reproducibility very low deposition energy and therefore the intermixing with the ROI is avoided. Each cap layer material has a specific isotope ratio, which can help to identify the detected ions, but can also lead to mass peak overlaps. By choosing the right cap layers, these mass peak overlaps can be avoided. Different cap layers have different sputtering rates and a different gallium stopping power. The cap materials silicon, chromium, nickel and cobalt are compared to show the influence of different cap layers on the tip shape, the summary is displayed in fig. 1.

After depositing the protective cap layers, the specimen preparation in the FIB is important to obtain reproducible tips. High reproducibility in the tip shape can be achieved if the parameters used for the FIB milling are reproduced very accurately. The tip shape itself plays an important role in achieving high measurement yield. Blunt tips require high applied voltage and tend to fracture more often than sharper tips. If the tip is too sharp, then the mass resolving power deteriorates dramatically. The critical dimensions will be discussed in detail. In the specimen preparation the material lift-out is attached to a silicon post with platinum in the FIB, using the standard specimen preparation procedure, described in [1]. This platinum weld plays an important role in increasing the measurement yield, as the lift-out tips begin to fracture at the applied voltage higher than 8 kV. During the tip sharpening a step formation may occur due to different sputtering rates of the material stack. In fig. 2 two different cap layers on the same ROI (high-k material) were deposited and tips were sharpened using the same FIB settings. In a) silicon / nickel material stack leads to a smooth tip shape. In b) an additional chromium layer is inserted to improve the adhesion to the ROI. This results, however, in a step formation, which leads to a higher fracture probability due to an irregular tip shape. Choosing lower ion beam currents and better focus reduces this step formation and increases the measurement yield.

https://doi.org/10.1017/S1431927611004521 Published online by Cambridge University Press

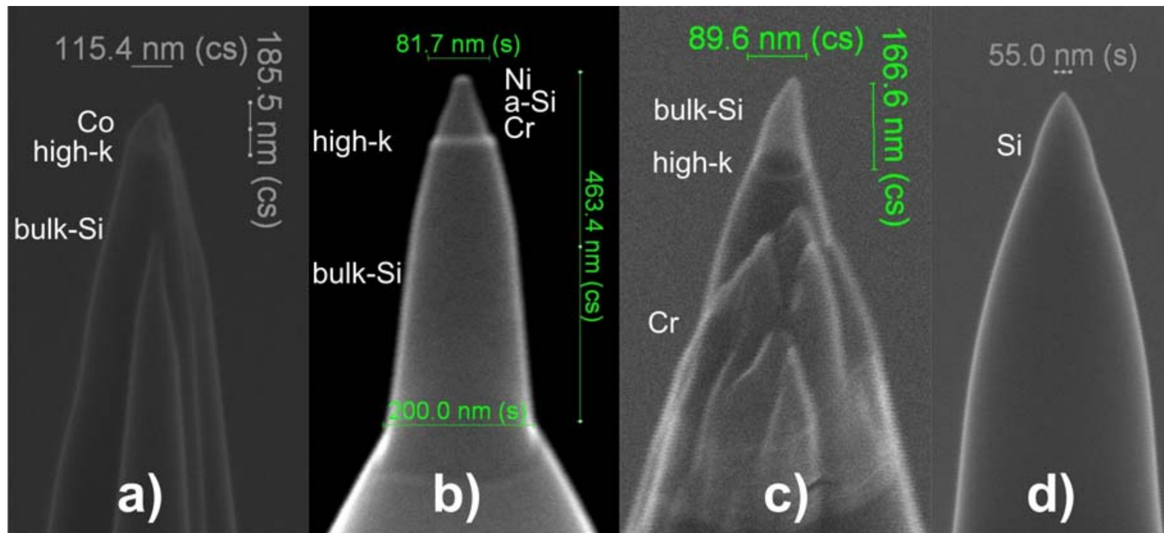

FIG. 1. The influence of the cap layer on the tip shape is summarized: a) cobalt and c) chromium lead to irregular tip shapes due to the grain formation, b) stack of nickel / amorphous silicon / chromium and d) crystalline silicon lead to reproducible and smooth tip shapes.

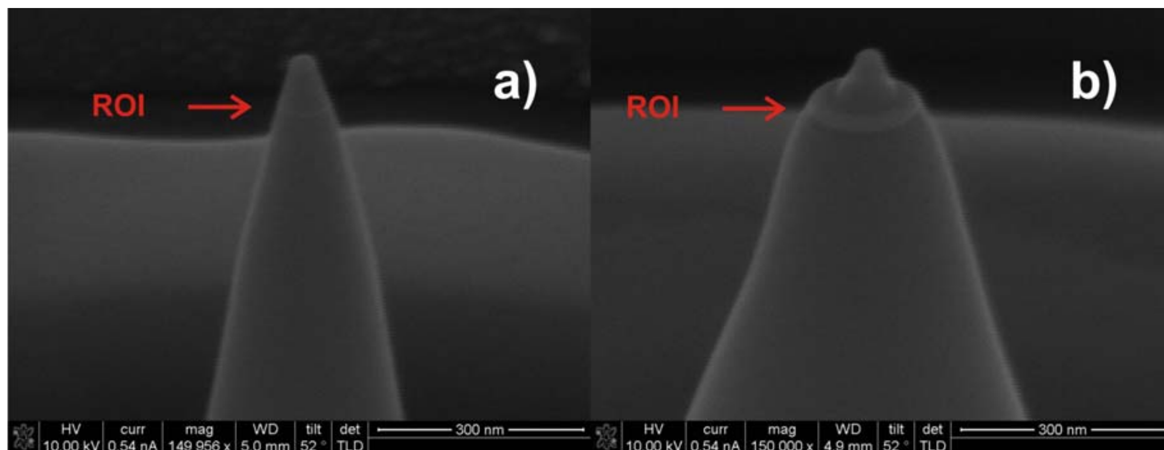

FIG. 2. Different cap layer stacks are deposited to protect the ROI (high-k): a) nickel / silicon results in a regular tip shape, b) nickel / silicon / chromium improves the adhesion to the ROI, but leads to a step formation due to different sputtering rates.

References

[1] K. Thompson et al., Ultramicroscopy 107, 131 (2007)

[2] The project described in this publication has been funded in line with the technology funding for regional development (ERDF) of the European Union and by funds of the Free State of Saxony.

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