Contamination-Free Scanning Electron and Helium Ion Microscopy

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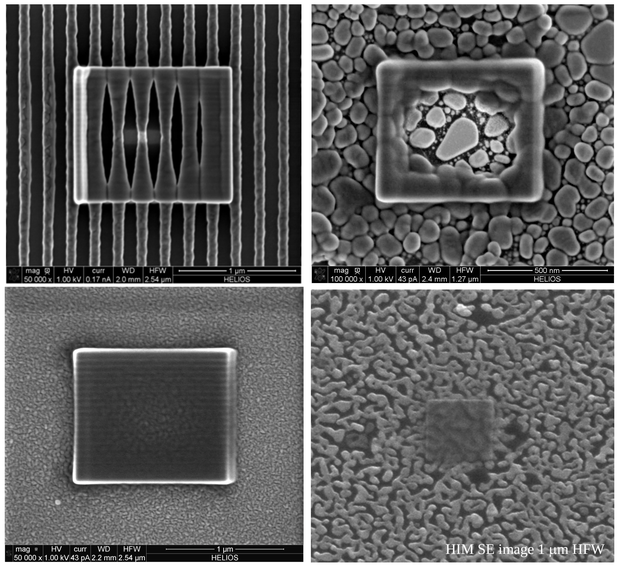
Charged particle beam-induced contamination has been one of the most persistent problems since the beginnings of electron and ion microscopy. Contamination manifests itself as a gradual buildup of carbonaceous material on the surface of the sample in the vicinity where the electron or ion probe excites the sample, which results in characteristic dark patterns. Contamination changes the sample itself and the number, trajectory, and energy of the electrons or ions leaving the sample, and consequently it makes repeatable quantitative measurements and achieving the best spatial resolution difficult or impossible. Fortunately today, with wet and dry cleaning processes, obvious charged particle beam-induced contamination can largely be eliminated[1-7].

We are reporting here on the various processes that are effective in cleaning both samples and the instrument. The cleaning processes followed by several minutes of electron bombardment leads to even better, possibly ultimate sample cleanliness. The procedure not only allows for more repeatable quantitative electron and ion microscopy measurements and material deposition and milling, but also makes it possible to achieve higher imaging resolution than otherwise would be possible.

Contamination Ruins the Sample and Resolution

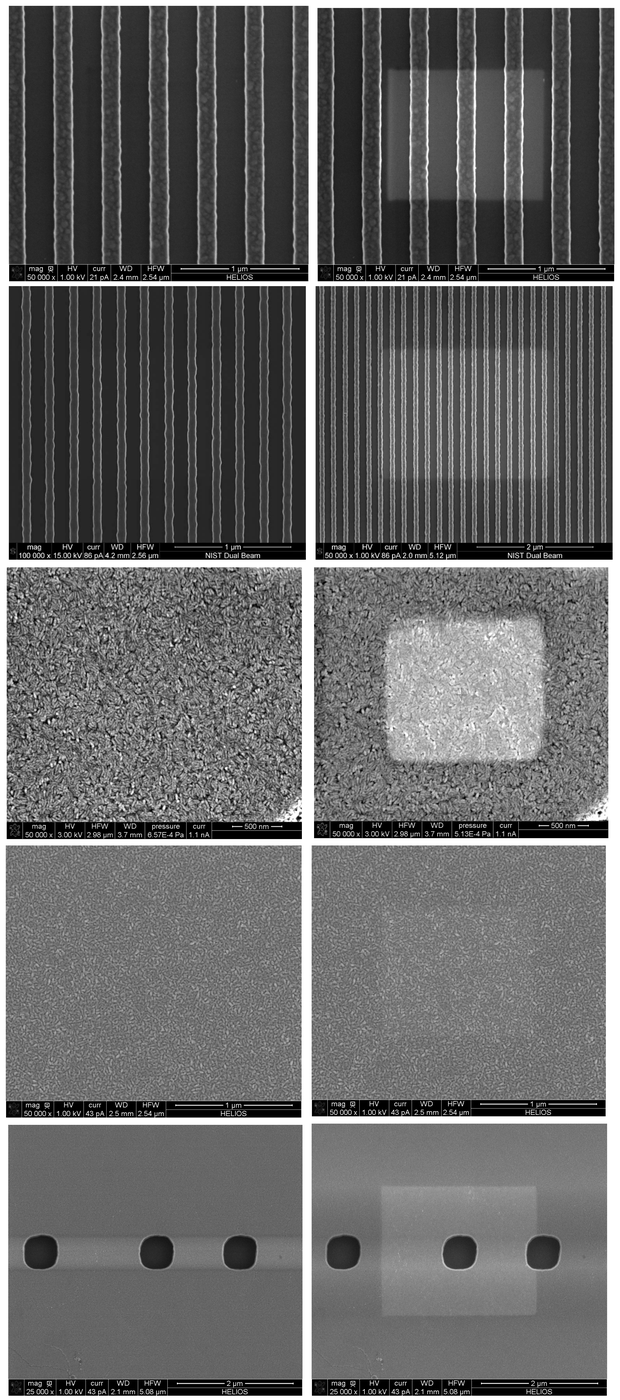


Dirty SEM/HIM and dirty sample leave imaged areas useless, and prevent high-resolution imaging.

NIST Contamination Specification

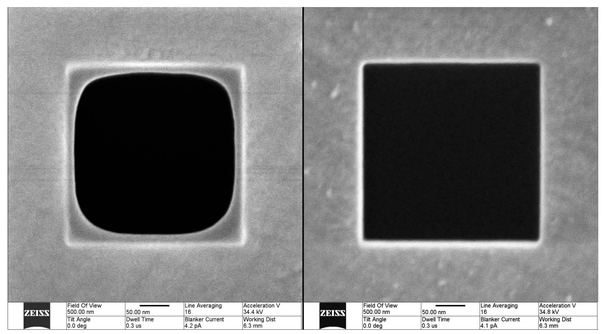
1. On amorphous Si patterns of a Si chip, using the best resolution imaging parameters take one image at 100,000 times magnification. Save the image.
2. Go up to 200,000 times magnification and continuously image for 10 minutes
3. Go back to 100,000 times magnification and take another image.
4. If there is any visible darkening, frame, any structure beyond the sample itself in the middle of the 2nd image, the instrument fails to meet this specification.
5. If the specification was not met, first clean the sample in the mixture of 3:1 ratio of 30 % hydrogen peroxide solution added to concentrated sulphuric acid (acid piranha solution -CAUTION! When preparing the piranha solution keep in mind that the reaction is exothermic. The solution will become hot. The peroxide must be added to the acid, not the other way around). This ferocious oxidizer will clean all hydrocarbon residues from the sample in less then 30 minutes.
6. If the instrument -with the clean sample- fails the test again, it needs to get cleaned with a low-energy plasma cleaning process. (Ibss GV10x* or XIE Scientific Evactron or others)

Contamination-free SEM



Using piranha solution, low-power oxygen plasma, hydrogen plasma, helium plasma and laser beam cleaning, contamination can be essentially eliminated. Note the increased secondary electron yield after electron bombardment

Ion-beam Nano Milling



Milling in dirty HIM leaves the milled areas of the sample unusable. Much better pattern can be obtained after cleaning the HIM with oxygen by Evactron Model 45 20 W plasma cleaner

Meeting the NIST contamination specification means the instrument is clean. On clean samples a few minutes of electron bombardment results in ultimate secondary electron yield and in the highest attainable spatial resolution. Both these are indispensable for nanometer-scale imaging and measurements.

Refrences

1. Harada, Y., Tomita, T., Watabe, T., Watanabe, H. & Etoh, T.. Reduction of Contamination in Analytical Electron Microscopy, Scan. Elect. Microsc., 103 (1979)/II
2. Hren, J. J. Specimen Contamination in Analytical Electron Microscopy: Sources and Solutions, Ultramicroscopy, 3, 375 (1979).
3. Bruenger, W. H., Kleinschmidt, H., Hassler-Grohne, W. & Bosse, H. Contamination Reduction in Low Voltage Electron-Beam Microscopy for Dimensional Metrology, J. Sci. Technol. B 15(6), 2181 (Nov/Dec 1997).
4. Akishige Ono. Influence of Incident Electron Energy on Specimen Contamination in SEM Mode, Proc. XIth Int. Cong. On Electron Microscopy, Kyoto, 417 (1986).
5. Ura, K. Electron beam Interaction with Specimen, 175, Electron beam testing Technology, Ed. Thong J T, NY. (1993).
6. Hirsch, P., Kassens, M., Puttmann, M. & Reimer, L. Contamination in a Scanning Electron Microscope and the Influence of Specimen Cooling, Scanning Vol. 16, 101 (1994).
7. Black, J. T. The Scanning Electron Microscope: Operating Principles, Principles and Techniques of Scanning Electron Microscopy, Vol. 1, 1 (1974).

*Certain commercial equipment is identified here to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment identified is necessarily the best available for the purpose.