On Clean Samples and High-Resolution Quantitative Charged Particle Microscopy

A. E. Vladár, K. P. Purushotham, and M. T. Postek
*National Institute of Standards and Technology (NIST)**
100 Bureau Dr., Stop 8212, Gaithersburg, MD 20899 andras@nist.gov

*Certain commercial equipment is identified in this report 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.

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 low-power oxygen or hydrogen plasma cleaning systems, obvious charged particle beam-induced contamination can largely be eliminated[1].

We are reporting here on a new finding: oxygen or hydrogen plasma cleaning 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 microscope measurements and material deposition, but also makes it possible to achieve higher imaging resolution than otherwise would be possible. Figure 1 shows a gold-on-carbon sample that is contaminated so severely that it fails the NIST contamination specification.[2] This specification requires continuous electron beam irradiation for 10 minutes while imaging at twice as high magnification than what is used for images to prove the resolution performance of the instrument. The sample, the instrument, or both can be contaminated. In this case the SEM was known to be clean and passed the test. Figure 2 shows the same sample in the same instrument after 2 hours of hydrogen plasma cleaning of the sample only. The sample now meets the specification, because there is no visible sign of contamination after the same test procedure. Figure 3 reveals something beyond this. The region that was bombarded by the electron beam the fine, shallow surface details on the top of the larger gold grains became better resolved and the area got noticeably cleaner (brighter). Similar improvement was observed on many other types of samples as well. It was also observed that the areas cleaned with both plasma and electron beam stayed clean for a long period of time. Further improvement was not observed in the secondary electron yield or in the achievable spatial resolution, even after longer times of hydrogen or oxygen plasma cleaning and or electron beam bombardment.

[1] A. E. Vladár, M. Postek and R. Vane Proc. of SPIE Vol. **4344** (2001)
[2] A. E. Vladár, K. Purushotham, and M. Postek Proc. of SPIE Vol. **6922** (2008)

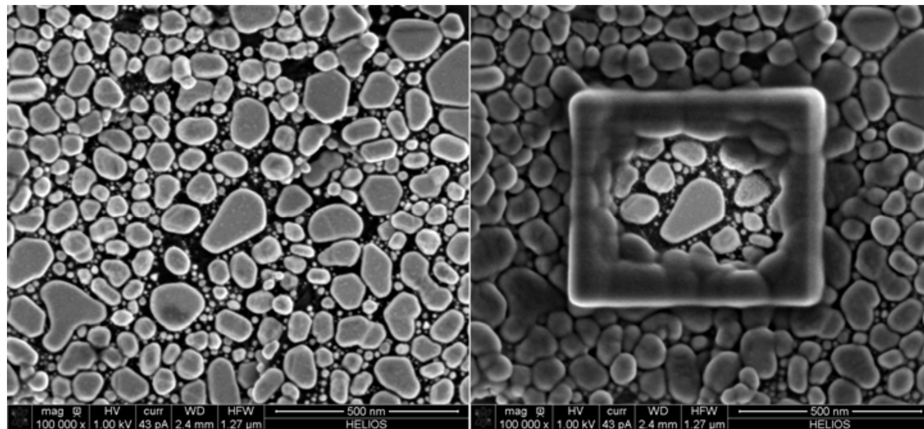

Figure 1. Contaminated sample: A contaminated gold-on-carbon sample allows for one relatively contamination-free image (left), but 10 minutes continuous imaging at twice as high magnification leaves large amount of contamination (right) in a clean SEM.

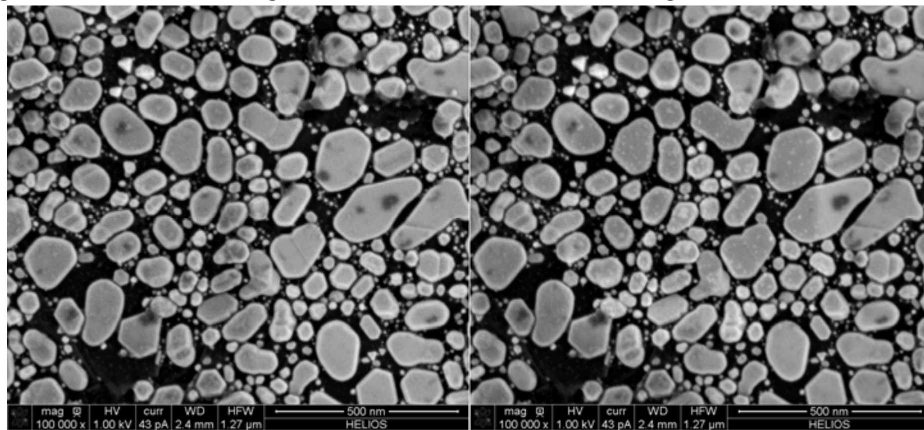

Figure 2. Plasma-cleaned sample: The same sample after 2 hours of hydrogen plasma cleaning is a bit brighter (left), and allows for more than 10 minutes of contamination-free imaging (right) even at twice as high magnification.

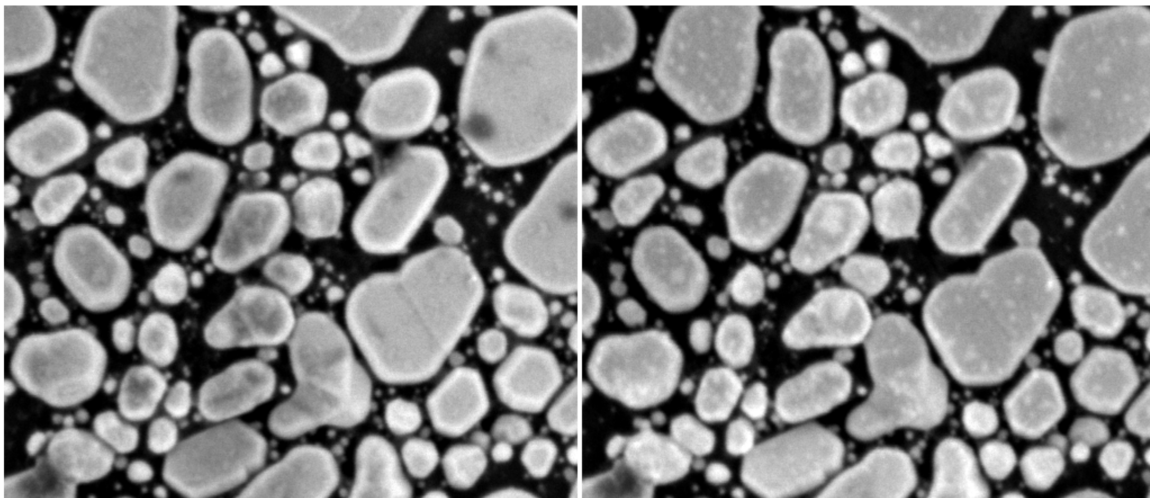

Figure 3. Comparison of center portions: The 10 minutes continuous electron beam bombardment made the sample even cleaner, which allows for better resolution.