Does Your SEM Really Tell the Truth? How Would You Know? Part 2

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Summary: The scanning electron microscope (SEM) has gone through a tremendous evolution to become indispensable for many and diverse scientific and industrial applications. The improvements have significantly enriched and augmented the overall SEM performance and have made the instrument far easier to operate. But, the ease of operation also might lead, through operator complacency, to poor results. In addition, the user friendliness has seemingly reduced the need for thorough operator training for using these complex instruments. One might then conclude that the SEM is just a very expensive digital camera or another peripheral device for a computer. Hence, a person using the instrument may be lulled into thinking that all of the potential pitfalls have been eliminated and they believe everything they see on the micrograph is always correct. But, this may not be the case. An earlier paper (Part 1), discussed some of the potential issues related to signal generation in the SEM, instrument calibration, electron beam interactions and the need for physics-based modeling to understand the actual image formation mechanisms. All these were summed together in a discussion of how these issues effect measurements made with the instrument. This second paper discusses another major issue confronting the microscopist: electron-beam-induced specimen contamination. Over the years, NIST has done a great deal of research into the issue of sample contamination and its removal and elimination and some of this work is reviewed and discussed here. SCANNING 36:347–355, 2014.

Introduction

An earlier paper (Part 1, Postek and Vladár, in press), discussed some of the potential issues related to signal generation in the SEM, instrument calibration, electron beam interactions, and the need for modeling to understand the actual image and its formation. All these were summed together in a discussion of how these issues effect measurements made with the instrument. This second paper, discusses electron-beam-induced specimen contamination. This form of contamination and its detrimental effects on imaging and measurements made with the SEM is something that every user should know, and understand before any critical quantitative work is attempted. Over the years, NIST has carried out a great deal of research into electron-beam-induced sample contamination, its source, removal and elimination. Sample contamination contributes to the uncertainty of any measurement and must be considered in any uncertainty statement about the accuracy of a measurement. Furthermore, especially for nanometer-scale imaging and measurements, electron-beam-induced contamination can seriously hamper or prevent work, as the few-nm size objects get obscured under a layer of carbonaceous material. Therefore, NIST has been interested in eliminating or diminishing this component of measurement uncertainty and some of this work is reviewed and discussed here. Due to the work carried out in cooperation by NIST and two small US companies, contamination-free imaging and measurements are now possible and the procedures are described in this paper. These methods rely on the use of low-energy and low-power (5–100 W) of oxygen, hydrogen, or helium plasma cleaning of the instrument and the sample. The choice of plasma type depends on the materials in the instrument and the sample. For example,

for eliminating contamination from a gold-on-carbon "resolution" sample one should not use oxygen plasma; hydrogen was found to work efficiently in restoring the usefulness of the sample.

The situation with electron and ion beam-induced contamination is that there are now reliable procedures (low-energy plasma cleaning for the instrument and for almost any sample and chemical cleaning for the reference sample), which essentially eliminate this problem. At present very little is known scientifically about the exact chemistry on the surface, or the mobility and the composition of the materials that under electron irradiation end up as the so-called "contamination." Surface chemistry at the nanometer-scale, in vacuum, under electron or ion beam bombardment is far from a settled science. Unfortunately, exploratory efforts with residual gas analysis (RGA) proved to be inconclusive, partly due to the surface nature of the problem. Work is continuing on exploring what exactly happens, why and how contamination is deposited.

Charged Beam-Induced Sample Contamination

Contamination induced by electron beam bombardment of a surface and its detrimental effects has been known for some time (Stewart, '34; Marton et al., '46; Watson, '47). The same kind of contamination may appear with ion beam bombardment, e.g. in scanning helium ion microscopes. In both cases low energy secondary electrons play key roles in altering carbonaceous molecules that then adhere to the surface of the sample. Early diffusion pump-type vacuum systems were especially prone to specimen contamination due to both diffusion pump and fore (roughing) pump fluid backstreaming. Early SEMs were essentially "oil immersion" microscopes. With the poor pumping stations and handling techniques, hydrocarbon contamination was common and reluctantly accepted as a "fact of life." With the use of high accelerating voltage for SEM work, contamination was less bothersome; it was present, but many times less noticeable. The need for cleaner instruments by the semiconductor industry led to the replacement of diffusion pumps with turbomolecular pumps backed by dry backing pumps which began to reduce instrument-related contamination. However, without coupling the clean pumping with instrument cleaning and clean sample preparation procedures, contamination was only reduced, but still remained a problem. Cleanliness is not just something that is desirable, but it is indispensable, especially for nanometer-scale imaging and metrology. Without clean instruments and samples it is impossible to achieve the best resolution and measurement repeatability.

Backstreaming occurs when some of the pumping fluids stream back through the instrument manifolding into the specimen chamber. Under vacuum conditions, the surface mobility of oily residues is much higher than in air, and soon a thin layer of oil will cover every surface in the SEM specimen chamber and the sample. As the beam scans the sample, hydrocarbon molecules remaining in the specimen chamber or from the surface of the sample are broken and "pinned" to the sample by the beam. Liquid nitrogen cryotraps and cold fingers helped to reduce the problem. Other methods of reducing backstreaming were also used (Postek, '96), as well as, the implementation of clean cryopumps (Postek and Keery, '91). Some of the other methods employed over the years are shown in list below. Non-carbonaceous contamination in practical clean vacuum instruments so far was found to be less common, which is the reason for not dealing with it in this paper. Unwanted particles that may interfere with good quality imaging can be largely avoided with proper sample handling and storage.

Partial List of Methods Previously used to Fight Electron Beam Induced Contamination

- Dry vacuum system, with magnetically levitated turbo-molecular and oil-free fore pumps.
- Continuous bleeding of clean N_2 at low pressure, either into the sample chamber or the roughing pump line.
- The use of clean, dry N_2 during sample exchange and for venting and periodic purging of the sample chamber.
- Cryo-pump, cryo-shield or cold finger above the sample, and/or cryo-trap on the chamber.
- Sample heating; sample cooling; soaking and gentle washing of the sample.
- Irradiating the specimen chamber and sample surfaces with UV light.
- Application of a mild, clean, low-pressure gas jet over the imaged area of the sample.
- Variable pressure scanning electron microscopes (SEMs).

All these remedies work more or less effectively, but none of them eliminates the contamination completely. This is a very complex problem because both the sample and the SEM contribute to it to a varying extent. The fact is that the origins of contamination and the best methods to fight them are not obvious. Depending on the root cause(s) of the problem, different cleaning methods may also have to be pursued. One thing is clear: it is essential to separate the sample-related sources of the contamination from those of the SEM. As described below, this can be done with a sample that is known to be made so clean that it cannot be the source of the contamination.
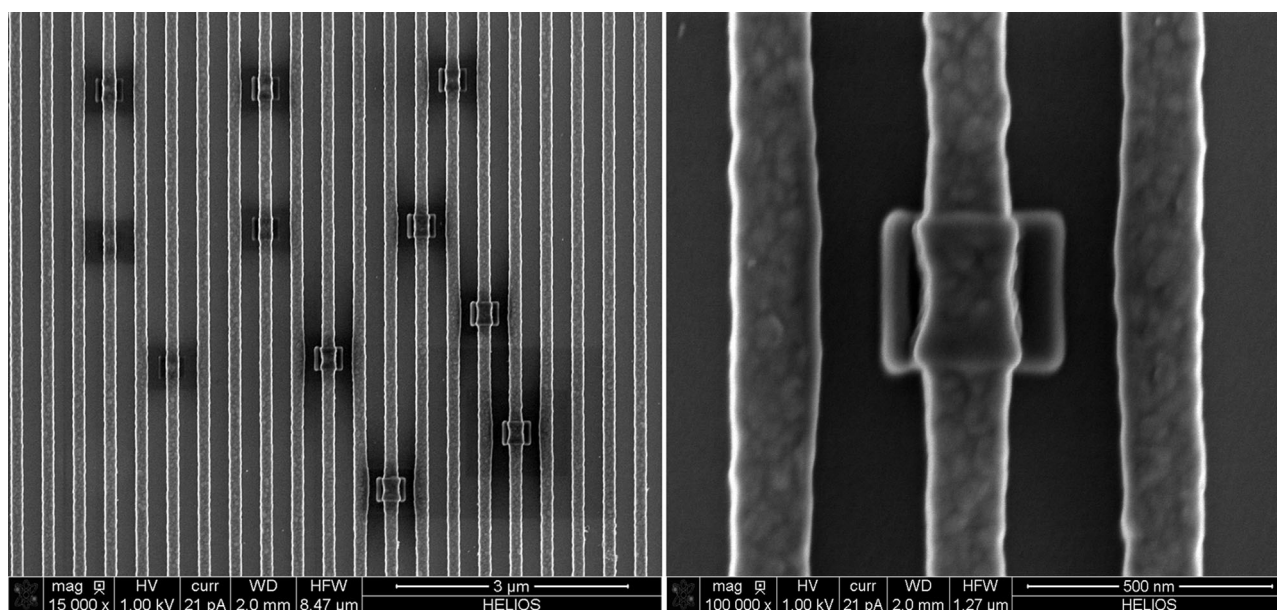
Fig 1. Example of a contamination test undertaken to show how rapidly beam-induced specimen contamination can be deposited on a sample: (**left**) silicon test sample showing the rapid build-up of contamination during exposures spanning from 1 to 10 min (in 1 min increments), in the case when both the sample and the instrument had not been cleaned (HFW = 8.47 μm). (**Right**) Enlarged area irradiated for 10 min showing the carbonaceous contamination obscuring fine sample details and making it useless for imaging or measurements (HFW = 2.54 μm).

Sources of Contamination

Several published studies (Black, '74; Harada *et al.*, '79; Hren, '79; Akishige, '86; Hirsch *et al.*, '94; Bruenger *et al.*, '97) documented well that the origins of the contamination are both the sample itself and the vacuum system of the SEM. Although the pumping system is a major contributor to this problem, the history of the specimen prior to entering the vacuum system is also important. It is common that instruments with "good" vacuum will still exhibit unacceptable rates of electron beam-induced contamination because of residual organic materials in the instrument. This is especially true at low landing energies where the carbonaceous layer becomes obvious. Reimer ('93) described the formation of contamination as a process of drift and eventual dissociation of large molecular weight molecules under electron bombardment. The deposition of the material forming the contamination layer is a dynamic process. Molecules arrive at and leave the sample surface at the same time. The amount of contamination "pinned" down, i.e. deposited or leaving the irradiated surface of the sample depends on the electron dose (i.e. the length of time the beam dwells on the sample and the beam current and beam energy), the amount of available hydrocarbons, and the sample material. The longer the dwell time, higher the beam current, the thicker the contamination becomes (Fig. 1, left). Deposition rates of a few tens of nanometers per second have been observed, but smaller rates are more common especially with more "modern" instruments.[1] In many cases, the presence of

contamination is not always obvious; it may only lead to a measurable change in the amount and energy of electrons leaving the sample, hence a drop of signal. In other cases, surface contamination will lead to serious measurement errors because it obscures the fine details of the sample and thus, will rule out collection of any useful data (Fig. 1, right).

Working at high accelerating voltages or in more appropriate words high "landing energies"[2] masked, to some extent, the effects of beam induced contamination for a relatively long time because the electron beam was able to easily penetrate the low atomic number carbonaceous surface contamination, thus making all but the heaviest contamination "transparent" to the electron beam. Changing from high landing energies to a low landing energy readily demonstrates this problem, as shown in Figure 2. Note that the specimen contamination, so obvious in the low landing energy micrograph, is not at all visible in the high landing micrograph.

Particle beam instruments are considered to be "parfocal" since the beam can be focused and corrected

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[1] The performance of the instruments used in this study to exemplify the problems associated with electron beam induced contamination was typical for the industry at the time of the study and should not be associated with any single manufacturer.

[2] Low landing energy has replaced the term low accelerating voltage because in newer instruments the electron source can emit electrons at high accelerating voltage, but they are decelerated to a lower landing energy in the column and/or at the sample stage. This technique allows the electron source to constantly operate optimally.
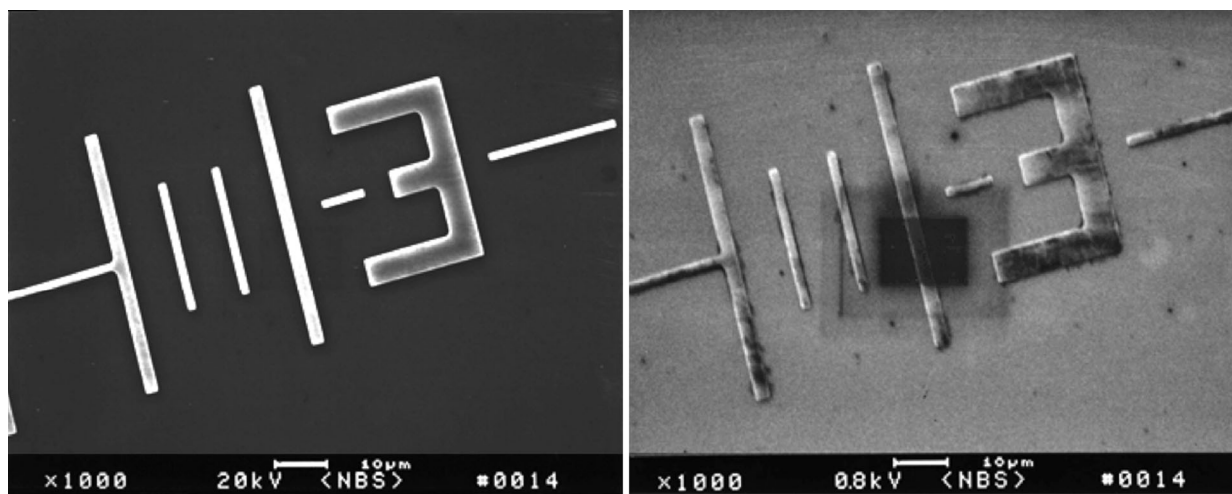
Fig 2. Example of the effects of landing energy on the visibility of sample contamination: (**left**) high landing energy image of a patterned silicon wafer sample (20 keV landing energy, HFW = 108 μm); (**right**) low landing energy image showing the same area of the sample and revealing the sample contamination that was not observed at high landing energy (0.8 keV landing energy, HFW = 108 μm) (figure originally from Postek, '94).

for stigmatism at high magnification and as the magnification is decreased it remains in sharp focus. This is one of the great advantages of these instruments. However, this ability is one of the notable contributors to specimen contamination since the small areas of the sample used for optimizing focus and astigmatism often appear as dark squares in lower magnification images, due to contamination build-up. With a dirty instrument or sample it is very difficult to focus, correct for astigmatism and decrease the magnification rapidly enough to avoid contamination deposition; even the shortest periods of exposure show some effect. This problem, depending on the severity of contamination, can be avoided if the recorded field is shifted enough before the final image is taken to exclude the focusing area. Contamination deposition in this manner should not be confused with a darkening due to the build-up of a positive charge on a sample. Positive charging can be dissipated, but contamination deposited on a sample will remain.

On-line semiconductor inspection and process control at low and ultra-low landing energies spurred an even greater interest by the industry to eliminate the specimen contamination problem. SEMs have always been capable of low accelerating voltage operation, but it was not until about the mid-1980s that this operational mode became more prevalent and manufacturers began to improve instrument performance for that mode of operation. Today, imaging and measurements with very small horizontal field widths (HFW) at high resolution are possible even at low landing energies. Low landing energy electrons generate more secondary (low-energy) electrons, which are particularly effective in dissociating oily molecules and causing contamination. Reducing the raster pattern (i.e. going to higher magnification/

reduced horizontal field width), increases the chance for interaction with adhering molecules and speed of contamination deposition. This occurs because the total current density of the electron beam at the sample increases within that area. Figure 3 shows the effect of continuous imaging on a chromium-on-quartz photomask sample. Figure 3 (left) shows an initial micrograph of a sample taken quickly enough that no contamination is obviously visible and the details of the chromium structure are sharp and well defined. Figure 3 (right) shows the same sample after 10 min of continuous irradiation at twice the magnification. In this case, gross contamination has been deposited and the overall sharpness of the surrounding chromium has also been reduced.

In 2008, NIST in collaboration with SEMATECH's Advanced Metrology Advisory Group (AMAG) proposed a specification for cleanliness for the SEM (Vladár *et al.*, 2008). It became abundantly clear that specimen contamination by the "clean" particle beam instruments, referred to as "carry-over" in integrated circuit feature size measurements, was a significant problem. Contamination deposition increased the size of the structures being measured, well beyond the industry's acceptable uncertainties.

Divide and Conquer

Solving the complex specimen contamination problem is not simple. First, it is essential to separate sample-related sources of the contamination from instrument-related sources. This can be done with a sample that is known to be *so clean* that it cannot be the source of the contamination (described below). In this way, the use of a known clean sample allows the user to make a decision whether the cleaning of the SEM is necessary or not. The
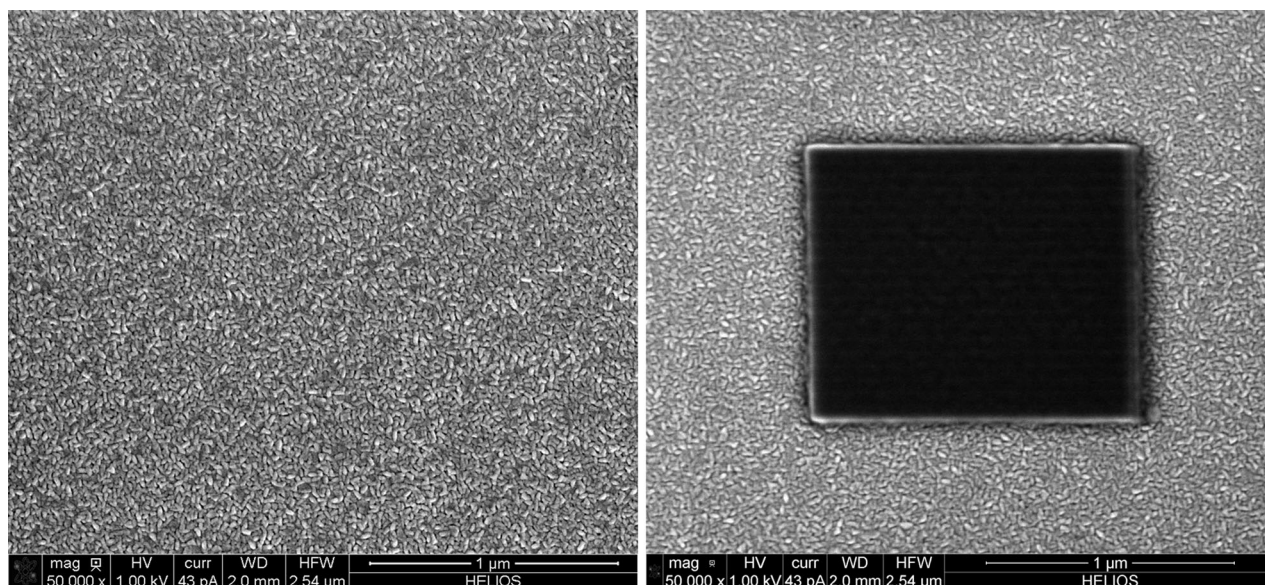
Fig 3.   Effect of beam induced contamination on a chromium on quartz photomask sample: (**left**) an initial micrograph of a sample taken quickly enough that no contamination is obviously visible and the details of the chromium structure are sharp and well defined. (**Right**) Micrograph taken of the same sample area following 10 min of continuous irradiation at twice the magnification. Note the contamination deposition and the loss of detail in the second micrograph (1 keV landing energy, HFW = 2.54 µm).

other key requirement is to have an effective cleaning method for the sample chamber and the vacuum (described below). The sample chamber and stage cleaning procedures developed at NIST and described here offer a comprehensive and effective solution.

Contamination Test Sample

As stated above, the use of a known clean sample allows the user to make a decision whether the cleaning of the SEM is necessary, or not. The NIST Reference Material (RM) 8820 (Postek *et al.*, 2010; Postek and Vladár, in press), is especially suitable both as a magnification calibration reference material and a contamination test sample. RM 8820 is a multi-use dimensional metrology calibration reference artifact; it was developed for calibrating the horizontal and vertical scales (magnification) of scanned particle beam, scanned probe, and optical microscopes all to the same standard (Postek *et al.*, 2010). There is also a huge set of other patterns designed for stage testing, optical overlay, and scatterometry.

Wet—Sample Cleaning

The RM 8820 sample can be routinely cleaned in a mixture of a 3:1 ratio of 30% hydrogen peroxide solution added to concentrated sulfuric acid (vitriol). This mixture forms the so-called acidic piranha solution.[3] The piranha solution should be used fresh, because over time the hydrogen peroxide decomposes

on its own, and the cleaning efficiency diminishes. *Warning! This solution is a ferocious oxidizer and care must be taken in its use*. The cleaning solution will readily clean all hydrocarbon residues from the sample in less than 30 min. It was found that the RM can stay in the solution for many hours and endure many repeated treatments without any perceptible damage or change of the calibration features. If treated carefully, RM 8820 will stay clean and ready to use for months in a semiconductor industry grade plastic container.

Plasma—Sample Cleaning

RM 8820, as well as other samples, can be cleaned with a low-energy and low-power plasma-cleaning device.[4] However, it should be noted that low-energy oxygen plasma could damage some samples. For samples that are sensitive to the oxygen plasma, hydrogen, or helium plasmas might be possible alternatives.[5] As shown in Figure 4 even heavily contaminated samples can be cleaned effectively. In this case, a contaminated gold-on-carbon resolution sample was used. Initially it was possible to record one image in about a minute, but longer work was severely limited by the quickly forming contamination. The same sample following hydrogen plasma cleaning even after 10 min irradiation at two times the initial magnification (landing energy 1 keV; HFW = 1.27 µm) showed no sign of contamination.

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[3] Piranha solution http://en.wikipedia.org/wiki/Piranha_solution.

[4] http://www.evactron.com/.
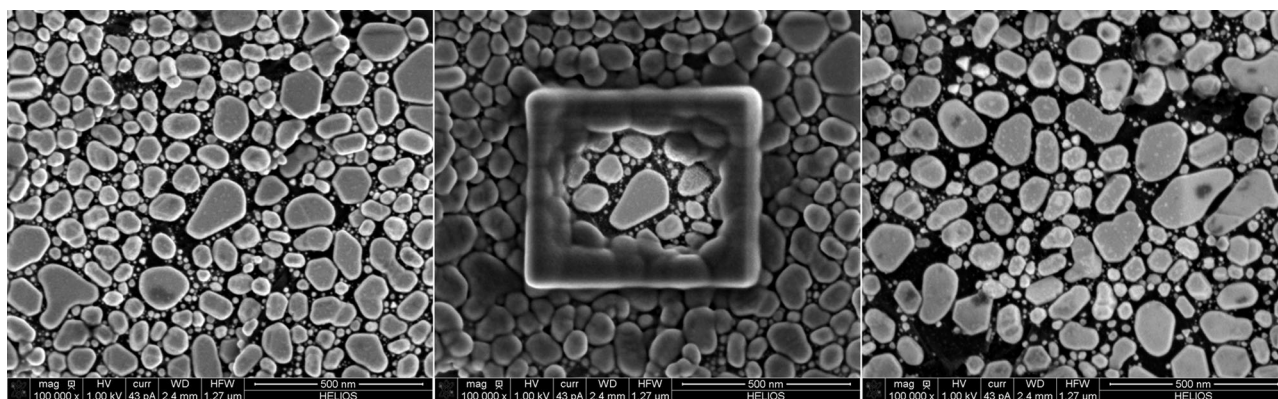[5] http://www.ibssgroup.com/.

Fig 4.   Plasma cleaning of a contaminated sample: (**left**) initial image of a gold-on-carbon resolution sample; (**center**) same sample following 10 min irradiation at two times the initial magnification that resulted in strong contamination; (**right**) same sample following hydrogen plasma cleaning after 10 min irradiation at two times the initial magnification (landing energy 1 keV; HFW = 1.27 μm) showing no sign of contamination.

Plasma Cleaning of the Specimen Chamber and Stage

Today, a number of low-energy and low-power plasma cleaning devices are available commercially and some have been designed for mounting directly on the chamber of particle beam instruments. The plasma generator can be mounted on the sample chamber of the instrument and then it can be used to periodically, effectively clean oily residues from the surfaces within the sample chamber (including the surfaces of the sample stage). It is important to point out that the ionized oxygen generated by the plasma cleaner oxidizes many materials, so care must be taken and the user should discuss the parameters and use of a plasma cleaner with the instrument manufacturer before its initial use.

The plasma cleaners used in this work were the Evactron[4,6] (Vladár et al., 2001) and the GV10x DS Asher[5,6] both are automatic plasma cleaning and vacuum monitoring systems. They can measure the vacuum level and by the use of valves, control the pressure of the gases introduced into the chamber needed for plasma cleaning. They also have built-in power supplies to drive a plasma-generating head. The cleaners can be mounted on the wall of the instrument's sample chamber and their electronics can be configured to automatically control the entire cleaning process. For regular cleaning low vacuum operation at 40–50 Pa is suitable. The cleaning procedure can be performed at low vacuum (for Evactron from 120 to 2 Pa or for GV10x from 240 to 9 Pa. GV10x is also capable of working in high vacuum mode, down to 4 mPa). The cleaning cycle starts with the closing of the necessary

valves to separate the specimen chamber from the electron optics. In some cases, the specimen chamber can also be separated from the turbomolecular or diffusion pump. In other cases, depending on the design of the vacuum system, the procedure will vary, for example, one might have to operate the turbomolecular pump at a lower than normal speed. The next step is to let the reactive, ionized gas (generated from filtered, clean, room air, or hydrogen) into the specimen chamber and then stabilize the pressure at its starting value of 40–50 Pa (depending upon manufacturers' recommendations). After reaching this point, the high frequency power is applied to the plasma head. The power applied and time duration depends on the size and cleanliness of the chamber. Lower pressures allow for longer mean free paths, and for more even distribution of the ionized gases, so gradually lowering the pressure while maintaining stable plasma might improve the efficacy of the cleaning. The ionized oxygen or hydrogen introduced into the chamber alters the oily residues on the surfaces of the specimen chamber, and/or the sample), and the resulting volatile products are pumped out. The use of argon-oxygen gas mixture for generating plasma is not recommended, because it can be too ferocious and detrimental to some of the structures of the specimen chamber or to the specimen itself.

The plasma cleaning procedure can easily be made fully automatic, thus the user only has to start the unit and wait until it has finished and the SEM is ready for its regular work schedule.

Contamination Specification Testing Procedure

The goal of the NIST specification is to provide a standard testing procedure to achieve a clean instrument (and a clean sample) where no hydrocarbons are available to be deposited (Vladár et al., 2008), see list below.

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[6]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.

NIST Contamination Testing Procedure

- Use an RM 8820 or another Si sample with amorphous silicon 300 nm or smaller size patterns.
- Set up the instrument to achieve the best resolution imaging parameters (landing energy, beam current, dwell time, focus, astigmatism).
- Take one image at 50,000 or 100,000 times magnification and save the image.
- Increase magnification to twice the initial magnification 100,000–200,000 times and continuously scan the area for 10 min in live imaging mode.
- Decrease the magnification back to the original magnification, and take another image.
- If there is any visible darkening, raster frame, or any additional structure beyond the sample itself in the middle of the second image, the instrument fails to meet this specification.
- If the specification was not met, clean the sample in the Piranha solution.
- If the instrument with the clean sample fails the test again, the instrument needs to be cleaned with the low-energy plasma.
- Retest as needed.

The results of following the instrument and sample cleaning procedures described above are shown in Figures 5 and 6. All high and low landing energy images show that clean samples in clean instruments allow for contamination-free work.

A clean RM 8820 silicon chip was used in the contamination test procedure to acquire images of a selected area before and after the 10-min continuous exposure to the beam at half the horizontal field width. The landing energy for the first pair was 15 keV and the beam current was measured at 86 pA and 1 keV and 86 pA for the second set of images. Under these operating test conditions, specimen contamination would be expected in a "typical" instrument (Fig. 5) and clearly observable at low landing energy (Fig. 6). Since this test sample has been properly cleaned, as shown in the micrograph no hydrocarbons were deposited. Figure 6 also shows and additional benefit, easily repeatable and stable secondary electron yield enhancement after a few minutes of electron irradiation was consistently observed on various samples (cleaned Si, Cr, on quartz and gold-on-carbon samples shown throughout the paper) to a more or less obvious extent. The enhancement is especially obvious in the 1 keV image. Research to find an explanation for this phenomenon and to exploit it further is ongoing and the results will be reported at a later date. The key is to recognize the existence of significant secondary electron yield enhancement; and when its makes sense, take advantage of it.

Instrument cleanliness is limited and must be periodically monitored. If contamination is found using the testing procedures described here, it can be concluded that the specimen chamber of the instrument has become contaminated and hence needs additional cleaning by using the NIST methods.

Duration of Instrument Cleaning

An instrument exhibiting a very bad case of contamination may need overnight or longer plasma
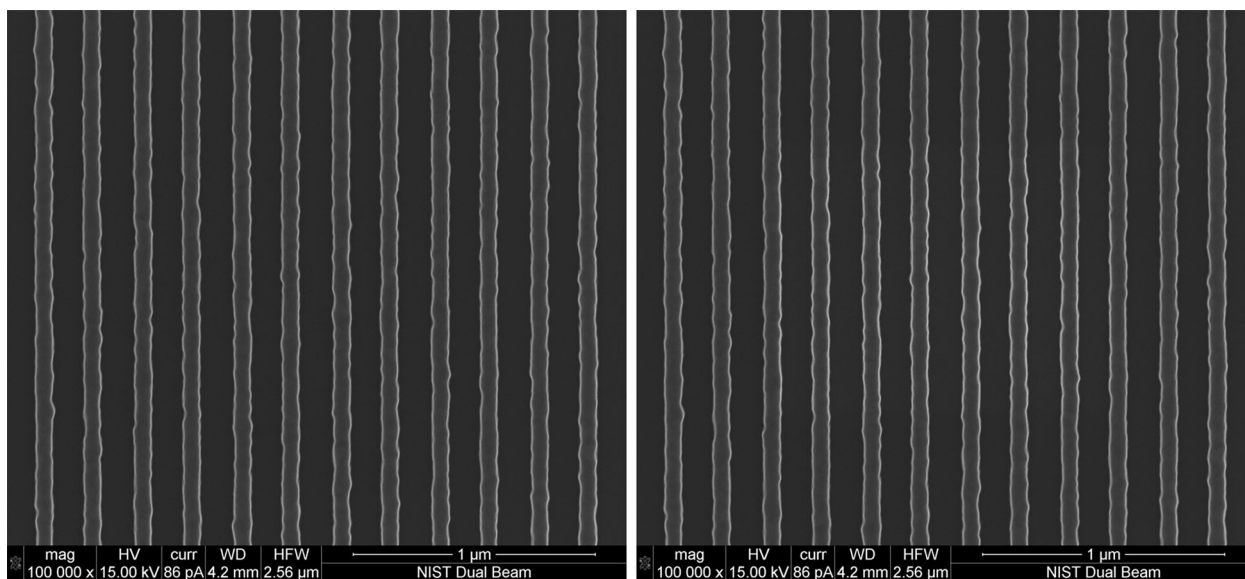

Fig 5.   Example demonstrating that a clean sample and clean instrument together can defeat specimen contamination at high landing energy: (**left**) Micrograph taken at high landing energy 15 keV with a clean sample in a clean instrument (HFW = 2.56 μm); (**right**) the same sample location after 10 min of continuous irradiation at twice as high of magnification. No sample contamination or degradation is observed (HFW = 2.56 μm).
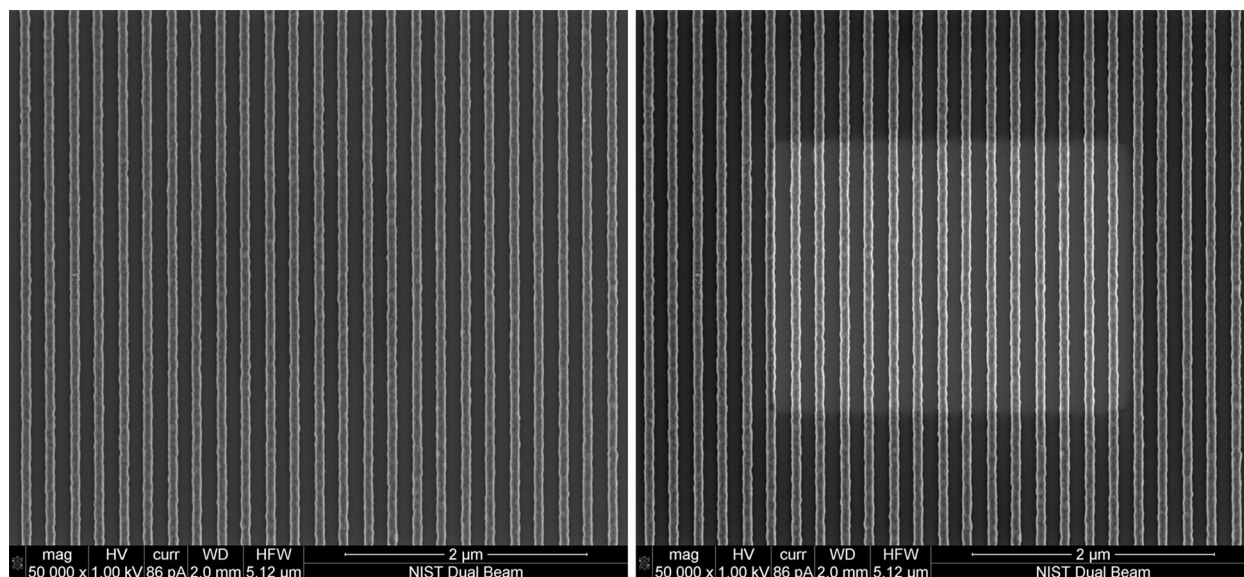
Fig 6.   Example demonstrating that a clean sample and clean instrument together can defeat specimen contamination, even at low landing energy. Here the same sample was used as Figure 4, but at low, 1 keV landing energy. (**Left**) image showing that no contamination has been deposited by the electron beam during the initial micrograph acquisition (HFW = 5.12 μm); (**right**) micrograph following 10 min of continuous irradiation at twice as high magnification as per the NIST specification (HFW = 5.12 μm). In many cases this results in increased secondary electron yield (discussed in the text). There is no sign of contamination in either case.

cleanings to meet the specification described. Later, as the instrument gradually gets clean, a 10 min to 1 h cleaning procedure may be sufficient. Periodic checking of the contamination performance against the specification showed that, as the instrument becomes clean, the time between needed cleanings increases. It is important to note that excessive plasma treatment may have harmful effects which could lead to deterioration in the performance of various components. Thus, plasma cleaning only when necessary and for times sufficient to meet the specifications is advised.

Conclusion

Contamination, induced in the particle beam instrument, masks fine regions of the sample, enlarges structures and compromises images and measurements made with the instrument. With the procedures described here, contamination can be correctly diagnosed by separating it into instrument-related and specimen-related components. Knowing where the contamination originates, directs the user to decide which cleaning procedure to use. The described procedures must be closely followed and periodic testing must be done. The use of an appropriately clean test sample is indispensable and the other key requirement is to have an effective sample chamber and vacuum cleaning method. Given these, the most important step is to use these procedures consistently. Meeting the NIST contamination specification results in

a clean instrument. With clean instruments and clean samples, highly repeatable secondary electron yield and highest spatial resolution can be attained. High resolution and high secondary electron yield are both indispensable for any nanometer-scale imaging and measurements.

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