Nanomanufacturing Concerns about Measurements made in the SEM II: Specimen Contamination

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ABSTRACT

The scanning electron microscope (SEM) has gone through a tremendous evolution to become a critical tool for many and diverse scientific and industrial applications. The improvements that have been made have significantly improved the overall SEM performance and have made the instrument far easier to operate. But, ease of operation also fosters operator complacency. In addition, the user friendliness has reduced the "apparent" need for more thorough operator training for using of these instruments. Therefore, this overall attitude has fostered the concept that the SEM is just another 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. The first paper in this series, discussed some of the issues related to signal generation in the SEM, instrument calibration, electron beam interactions and the need for physics-based modelling to accurately 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: 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.

Keywords: calibration, measurements, metrology, modelling, contamination, scanning electron microscope, SEM, standards, reference material

1.0 INTRODUCTION

The first paper in this series, discussed some of the issues related to signal generation in the SEM, instrument calibration, electron beam interactions and the need for modelling to understand the actual image formation process (Postek and Vladár, 2013). All these were summed together in a discussion of how these issues effect measurements made with the instrument. This second paper, discusses specimen contamination. Contamination and its detrimental effects on imaging and measurements made with the SEM is something that every user should understand before any critical quantitative work is attempted. Over the years, NIST has carried out a great deal of research into sample contamination, its source, removal and elimination. Sample contamination contributes to the measurement uncertainty of any measurement and must be considered in any uncertainty statement about the accuracy of a measurement.

2.0 CHARGED BEAM-INDUCED SAMPLE CONTAMINATION

Specimen contamination has been an unfortunate consequence associated with the particle beam (electron or ion) irradiation of samples under examination. Early diffusion pump-type vacuum systems were especially prone to specimen con-
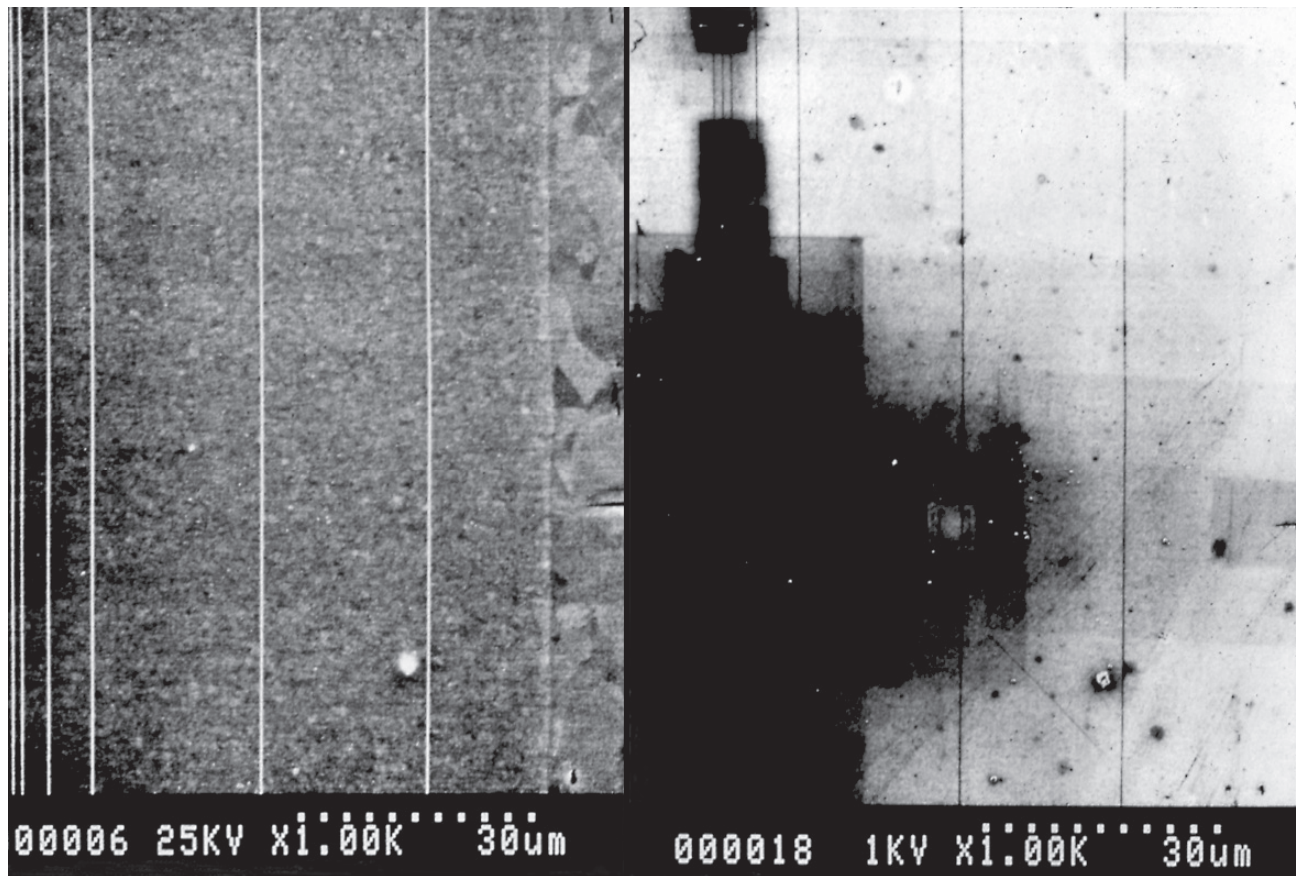
Figure 1. NIST SRM 474 scanning electron microscope magnification standard. (left) high landing energy (25 keV) image showing no obvious contamination observed. Magnification calibration can be easily done under these conditionssince the gold lines are readily observed, in good contrast to the lower atomic number nickel. (right) Low landing energy image (1 keV) where the contamination, deposited buy many calibration cycles readily obscures the gold lines. Note that the particle in the lower right side is the same in both images, hence these are two micrographs of the same area. (FOV = 81 μm)

tamination due to 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." Early SEM was done at high accelerating voltage. Because of beam penetration, contamination was less bothersome and not immediately observed, but not reduced or eliminated (Figure 1, left). It was not until the need for cleaner instruments by the semiconductor industry that clean turbomolecular pumps with dry pump backing began to reduce the instrument-induced contamination. However, without coupling the clean pumping with instrument cleaning and clean sample preparation procedures, contamination was only reduced, but still remained a problem.

Backstreaming occurs when some of the pumping fluids flow back 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, 1996) as well as the implementation of clean cryopumps (Postek and Keery, 1991). All these remedies work more or less effectively, but none of them eliminated the contamination completely. Specimen contamination 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 instrument. 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 and an appropriate instrument cleaning procedure.

2.1 Sources of Contamination. Several published studies (Akishige, 1986; Black, 1974; Bruenger et al., 1997; Harada, et al., 1979; Hirsh et al., 1997; Hren et al., 1979) 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, especially at low landing energies where the carbonaceous layer becomes especially obvious. Reimer (1993) describes this 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 leave the irradiated surface of the sample depending on the electron dose, (i.e. the length of time the beam dwells on the sample and the beam current) and the amount of available hydrocarbons, and the sample material. The longer the dwell time, higher the beam current, the thicker the contamination becomes (Figure 1, right). Deposition rates of a few tens of nanometers per second have been observed, but smaller rates are more common especially with more "modern" instruments. 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 completely obscuring the fine details of the sample and serious measurement errors and will rule out collection of any useful data (Figure 1, right).

2.2 Semiconductor Manufacturing as a Driving Force. 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, extremely small horizontal fields of view imaging at high resolution are possible even at low lending energies. Low landing energy electrons generate more secondary (low-energy) electrons, which are good for imaging, but are also particularly effective in dissociating oily molecules and causing contamination. As the raster pattern is reduced (higher magnification/reduced horizontal field width), the greater the chance and the more rapid is the contamination deposition. This results because the current density of the electron beam at the sample increases within that area.

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

Divide and Conquer. Solving the complex specimen contamination problem is not simple. 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. 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 other key requirement is to have an effective sample chamber and vacuum cleaning method (described below). The sample and sample chamber and stage cleaning procedures developed at NIST and described below offer a comprehensive and effective solution.

3.0 Materials and Methods

3.1 Plasma Cleaners. The plasma cleaners used in this work were the Evactron and the GV10x Downstream Asher. These 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 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 controller can be configured to automatically control the entire cleaning process. The cleaning procedure is performed at higher pressure (from 270 Pa to 5 Pa (Evactron) or to 0.1 Pa (GV10x)) than the normal operating pressure (10^{-3} - 10^{-4} Pa) of the specimen chamber. 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 (filtered, clean, room air or hydrogen) into the specimen chamber and then stabilize the pressure at its starting value of 10 to 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 initial 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 effect. This procedure provides gentle plasma cleaning. The ionized oxygen or hydrogen introduced into the chamber reacts with the oily residues in the vacuum, and on the surfaces of the specimen chamber, and the resulting volatile products are pumped out. The use of argon gas mixture for generating plasma is not recommended, because it can be detrimental to some of the structures of the specimen chamber or the specimen itself. Argon-oxygen mixture was found to be useful for cleaning of small parts and sample holder fixtures. This 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.

3.2 Duration of Instrument Cleaning. Starting with an instrument exhibiting a very bad case of contamination, overnight or longer plasma cleaning may be needed to meet the contamination specification described below. Later, as the instrument gradually cleans up, 1 hour or even only a 10 minute-long cleaning procedure can be sufficient. Regular, periodic checking of the contamination performance against the specification showed that the time between needed cleanings is generally increased over time. It is important to point out that only the necessary, sufficient amount plasma cleaning is advised. Excessive plasma treatment might have harmful effects, and could lead to deterioration of the performance of various microscope components.

3.3 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 is an amprphous silicon sample and is especially suitable contamination test sample. RM 8820 is a multi-use dimensional metrology calibration reference artifact 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 are also many sets of other patterns designed for stage testing, optical overlay and scatterometry.

3.3.1 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 sulphuric acid (vitriol). This mixture forms the so-called acidic piranha solution. The piranha solution should be used fresh, because over time the hydrogen peroxide decomposes on its own. Care must be taken since this is a ferocious oxidizer and hence it can be dangerous - safety procedures must be followed. This solution will readily clean all hydrocarbon residues from the sample in less than 30 minutes (http://en.wikipedia.org/wiki/Piranha_solution). It was found that the RM can remain in the solution for many hours and endure repeated treatments without any perceptible damage or change of the features. RM 8820 was found to stay clean and ready to use for months in a semiconductor industry grade plastic container.

3.3.2 Plasma – Sample Cleaning. RM 8820 can also be cleaned with a low-energy oxygen plasma cleaning device. However, it should be noted that low-energy oxygen plasma can damage some samples. For samples that are sensitive to the oxygen plasma, hydrogen or helium plasma cleaning might be possible alternatives. Instruments of these types are now available (http://www.ibssgroup.com, http://www.evactron.com, and http://www.fischione.com).

3.4 Plasma-Cleaning of the Specimen Chamber and Stage. Today, a few low-energy plasma cleaner devices are available commercially (http://www.ibssgroup.com, http://www.evactron.com, and http://www.fischione.com). In some designs, the plasma generator can be mounted on the sample chamber of the instrument and it then it can be used to periodically clean oily residues from the surfaces within the sample chamber (including the surfaces of the sample stage). This can be very effective in eliminating sample contamination. 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 use of plasma cleaner with the instrument manufacturer before its use.

Table 1

NIST Contamination Testing Procedure

- Use an RM 8820 or an amorphous silicon sample
- Set up the instrument to achieve the best resolution imaging parameters (landing energy, beam current, dwell time, etc.)
- Take one image at 100 000 times magnification - Save the image.
- Increase magnification to 200 000 times and continuously scan the area for 10 minutes in live imaging mode
- Decrease the magnification back to 100 000 times magnification.
- Take another image and save it
- Compare the first and second images.[If there is any visible darkening, raster frame, or any added structure to the sample located in the middle of the second image, the instrument fails to meet this specification
- If the specification was not met, clean the sample in piranha solution
- If the instrument with the clean sample fails the test again, the instrument chamber needs to be cleaned with the low-energy plasma
- Retest as needed

3.5 NIST 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). This procedure is outlined in Table 1.

4.0 Results

Routinely following the instrument and sample cleaning procedures has shown that, if the procedures are carefully followed, an instrument can be cleaned to the extent that contamination will not be deposited. This has been tested at both high and low landing energies and in both scanned electron and scanned helium ion instruments. 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-minute continuous exposure to the beam at half the horizontal field width (HFW). The landing energy was 1 keV and the beam current was 86 pA and 15 keV and 86 pA for the SEM. Under these operating test conditions, specimen contamination would be expected in a "typical" instrument. Since this test sample has been properly cleaned, no hydrocarbon was deposited. But, further research has shown that instrument cleanliness is limited and must be periodically monitored. If contamination is found, it can be concluded that the specimen chamber of instrument was contaminated and hence needs additional cleaning by using the NIST methods of low-vacuum plasma cleaning using oxygen or hydrogen or helium ion plasma.

5.0 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 be used. The described procedures must be followed and periodic testing must be done. The use of an appropriately clean test sample is critical and the other key requirement is to have an effective sample chamber and vacuum cleaning method. But, the most important step is to use the procedures consistently. Meeting the NIST/SEMATECH contamination specification results in a clean instrument. With clean instruments and on clean sample,s a few minutes of electron bombardment results in ultimately clean samples with high and repeatable secondary electron yield and in the highest attainable spatial resolution. These both are indispensable for nanometer-scale imaging and measurements.

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