

Contamination Specification for Dimensional Metrology SEMs

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ABSTRACT

Electron beam-induced contamination is one of the most bothersome problems encountered in the use of the scanning electron microscope (SEM). Even in “clean-vacuum” instruments it is possible that the image gradually darkens because a polymerized hydrocarbon layer with low secondary electron yield is deposited. This contamination layer can get so thick that it noticeably changes the size and shape of the small structures of current and future state-of-the art integrated circuits (ICs). Contamination greatly disturbs or hinders the measurement process and the erroneous results can lead to wrong process control decisions. NIST has developed cleaning procedures and a contamination specification that offer an effective and viable solution for this problem. By the acceptance, implementation and regular use of these methods it is possible to get rid of electron beam induced contamination.

Keywords: electron beam, contamination, plasma cleaner, piranha solution, scanning electron microscope, CD-SEM

1. INTRODUCTION

It is inevitable that all SEMs measurements influence the sample to some extent. As electrons of the primary electron beam hit the sample, an excited volume is formed, where high flux of primary, secondary and backscattered electrons, x-ray and light photons and phonons is present. This can have profound effects on the irradiated regions of the sample. These effects to some degree change the sample itself and its top surface.

The sample surface is never entirely clean, i.e. beyond the sample material, there are other materials on the surface. Typically a thin layer of molecules containing water and carbon is on the surface of samples that were exposed to room air. Other molecules from storage or from the processes the sample has gone through can also adhere to the surface. The vacuum in the specimen chamber and the various parts of the sample stage are not perfectly clean either. Typically there are oily residues that cover the surfaces with a thin layer. These molecules may have long mean free path and quite high mobility in vacuum conditions, and could end up in the vicinity of the region hit by the electron beam. In a dynamic process of adsorption and desorption, these molecules can be deposited on the surface of the sample. The amount of these molecules can be very large, so much so, that it literally makes various sample features grow. Beyond this, it changes the very top of surface from the original sample surface to a carbonaceous material, which generally leads to a significant change in the secondary electron emission. Both these have detrimental effects: the sample changes its size, which is a critical parameter for many industrial applications, especially for IC process control, and the changes in electron emission lead to erroneous results, because the measurement is reporting results not on the sample but on the sample with an uncharacterized layer of different material on the top of it. For accurate dimensional measurements and sufficient process control, it is indispensable to minimize electron beam induced contamination to levels where it is negligible.

2. ELECTRON BEAM-INDUCED CONTAMINATION

Figure 1 shows an extreme example for carbonaceous contamination formed on an etched polysilicon wafer sample during 2 hours of continuous electron beam bombardment. The sample was continuously imaged at a smaller field of view (higher magnification). After finishing this, two images were taken. The top-down view image shows that very large amount of contamination was deposited, especially at the outer frame of the higher magnification image. This happens because in the center the desorption rate was higher than at the sides of the area irradiated. The contamination depends on the dose, therefore the contamination is worse where the primary electron beam waits for the horizontal line and vertical frame scans to be synchronized with 60 (or 50) Hz frequency of the line voltage. The beam stayed for the longest time at the upper left corner, because it was waiting there a bit longer for the frame scan to start. During the long

time of higher magnification imaging the sample stage has moved (drifted), which lead to the formation of a smeared double-frame contamination pattern. The center remained deceptively relatively clean. Nevertheless, there are two noticeable problems: the square pattern in the center of the higher magnification image got significantly smaller, i.e., the size of that sample feature has changed, and the electron emission has changed also as it is demonstrated by the altered secondary electron intensity of the edges and the bottom of the square structures. Other areas as far as 3 μm from the irradiated regions also got some amount of contamination, especially in the vicinity of the upper left corner. This again is due to the different dose and to the adsorption and desorption processes at work.

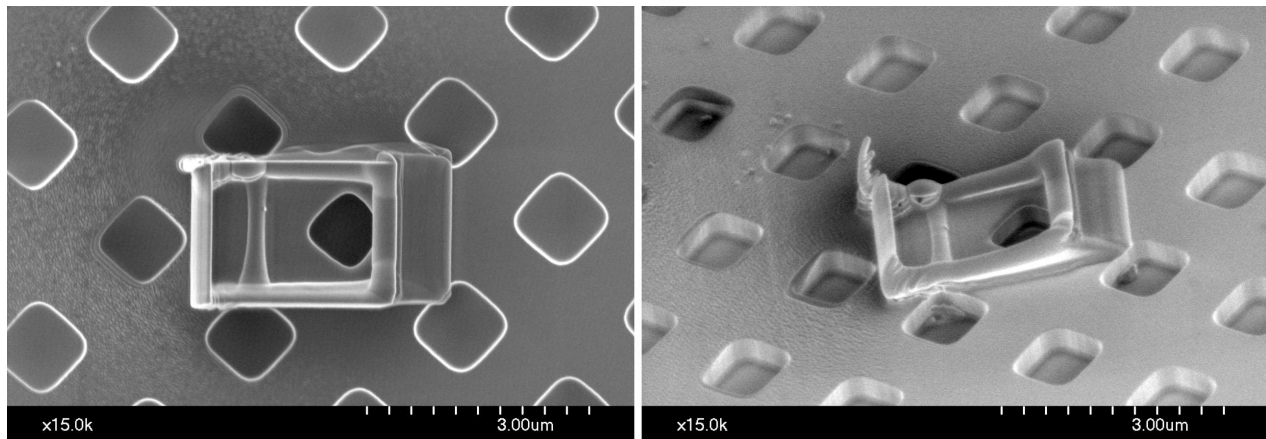


Figure 1. Contamination pattern formed on an etched silicon wafer sample during 2 hours of continuous electron beam bombardment. Due to the different dose and to the adsorption and desorption processes at work a contamination frame structure was formed. 10 μm field-of-view images, for the left image the sample was tilted by 35 degrees.

While only some measurements or imaging require longer than 10 minutes or so acquisition, it is not rare to have close to a nm changes in the size of the structures imaged at high magnifications by the end of the few ten seconds of imaging. The change in the secondary electron emission occurs even faster, within a few seconds. The secondary electrons play a major role in the deposition of carbonaceous materials on the sample surface, because they have the right energy to modify the otherwise more mobile molecules. As a consequence the balance of the adsorption and desorption processes changes so that deposition wins, and contamination forms. In the case on environmental SEMs, it is common to observe sample cleaning, especially when water vapor is used to maintain the pressure needed for the environmental mode of operation.

3. METHODS FOR FIGHTING ELECTRON BEAM-INDUCED CONTAMINATION

Over the decades of the existence of scanning electron microscopy there were many attempts to eliminate electron beam-induced contamination. Today one or the combination of the following methods is in general use [1 to 5]:

- Dry vacuum system, with magnetically levitated turbo-molecular and oil-free fore pumps
- Continuous bleeding in 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 above the sample, and cryo trap on the chamber
- Sample heating; sample cooling; soaking and gentle washing of the sample
- Irradiating the sample with UV light
- Application of a mild gas jet over the imaged area of the sample

All these work more or less effectively, (some almost as much as wishing that the contamination would just go away), but none of them eliminates the contamination completely. The problem is complex, because both the sample and the

SEM contribute to it to a varying extent, and due to the fact 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 have to be pursued. It is essential to separate sample-related sources of the contamination from that of the SEM. This can be done with a sample that is known to be so clean that it cannot be the source. One key step is to find a sample with a suitable cleaning method. The use of a clean sample allows the user to make a decision whether the cleaning of the SEM is necessary. The other key requirement is to have an effective sample chamber and vacuum cleaning method. The sample and sample chamber and stage cleaning procedures developed at NIST now offer an effective comprehensive solution.

4. NIST CONTAMINATION SPECIFICATION

Regular monitoring of the contamination performance of the SEM is indispensable. To separate sample-related and SEM-related contamination and thus avoid misleading results, it is necessary to have a clean sample, or a sample that can be cleaned. It was found that amorphous Si structures on 2 nm gate oxide on Si substrate work well, and if needed wet chemical cleaning and thorough rinsing will clean it if necessary.

The NIST specification for contamination test is this:

- On amorphous Si patterns on a Si wafer, using the best resolution imaging parameters (landing energy, beam current, dwell time) take one image at 100 000 times magnification. Save the image.
- Go up to 200 000 times magnification and continuously image for 10 minutes.
- Go back to 100 000 times magnification and take another image.

If there is any visible darkening, frame, any 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, first clean the sample in the mixture of 3:1 ratio of 30 % hydrogen peroxide solution added to concentrated sulphuric acid (vitriol), which form the so-called acidic piranha solution [6]. CAUTION! When preparing the acid 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. Before application the solution must be let cool down to room temperature. The piranha solution should be used fresh, because the hydrogen peroxide decomposes on its own. To prevent thermal shock, which can crack the sample, immersing the sample (wafer) into the solution should be done slowly. This ferocious oxidizer will clean all hydrocarbon residues from the sample in less than 30 minutes. It was found that the aforementioned sample can stay in the solution for many hours and endure repeated treatments without any perceptible damage. The test sample (SEMATECH/NIST AMAG 5 amorphous Si) was found to stay clean for months in a semiconductor grade plastic container.

If the instrument with the clean sample fails the test again, it needs to get cleaned with a low-energy oxygen plasma cleaning process using an Evactron* device [7]. Oxygen plasma cleaning very effectively cleans oily residues from the vacuum and 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, but, advantageously, the process is very effective on hydrocarbon residues. It is recommended to use the minimum, but sufficient time and plasma power (5 – 7 W). While there were no adverse, damaging effects to the SEM, unnecessary cleaning is not recommended. If the instrument meets the contamination specification there is no need for cleaning. It takes some time to go through the cleaning process, which involves airing and keeping the sample chamber at around 60 Pa pressure for the time of the plasma cleaning and then pumping it down. Excessive exposure of the sample chamber and sample stage to oxygen plasma might result in undesirable consequences.

Starting with instruments plagued with bad cases of contamination, at the beginning three hours of plasma cleaning, and in one case one overnight cleaning was needed to meet the contamination specification. Later, as the instruments gradually cleaned up, 1 hour or even only 10 minute-long cleaning procedures were sufficient. Regular, periodic checking of the contamination performance against the specification showed that the time between needed cleanings has increased. Now all four instruments meet the specification and three to four weeks could go by without needing plasma cleaning again.

* Certain commercial equipment is identified in this work 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. Contribution of the National Institute of Standards and Technology; not subject to copyright

Figure 3 for 15 kV accelerating voltage, Figure 4 for 1 kV accelerating show the results of a dimensional metrology SEM that meets the NIST specification. There is no visible contamination; actually sample cleaning is observable, especially pronounced way in the low energy case. Figure 5 was taken with a helium scanning ion microscope (SIM). This instrument also needed cleaning and after several treatments, it is now capable of secondary electron imaging without noticeable contamination.

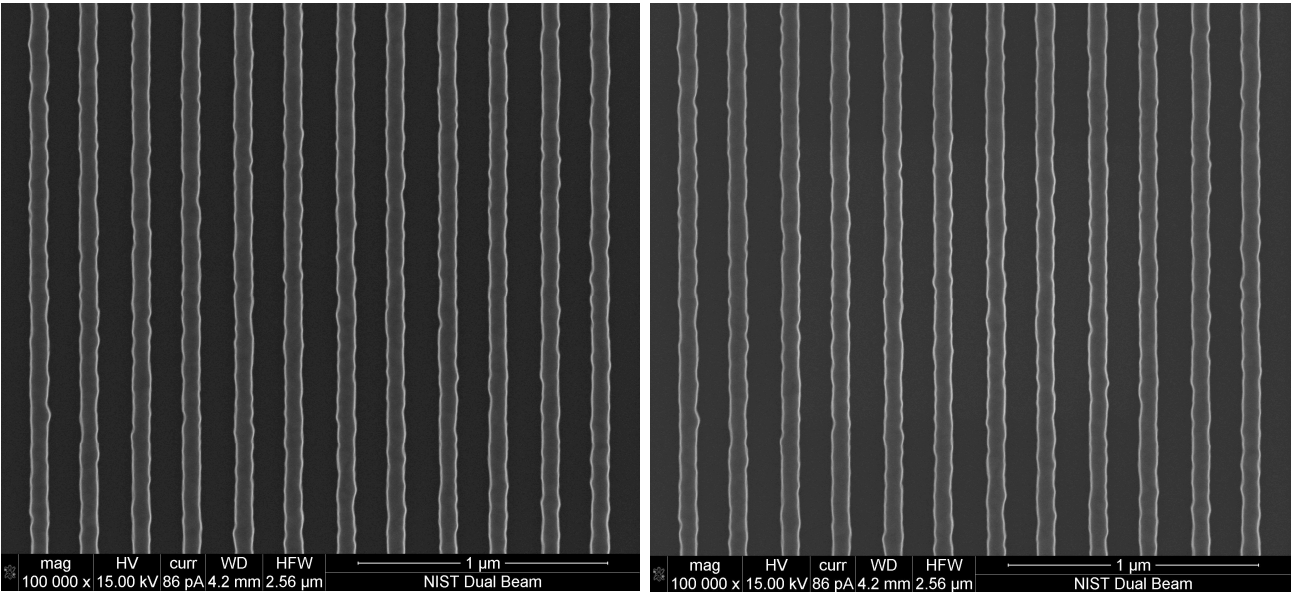


Figure 2. 15 kV, 86 pA SEM images of amorphous Si patterns at the beginning of the test (left) and after 10 minutes (left) of continuous electron beam bombardment. Essentially no contamination is observable. 2.5 µm field-of-view images.

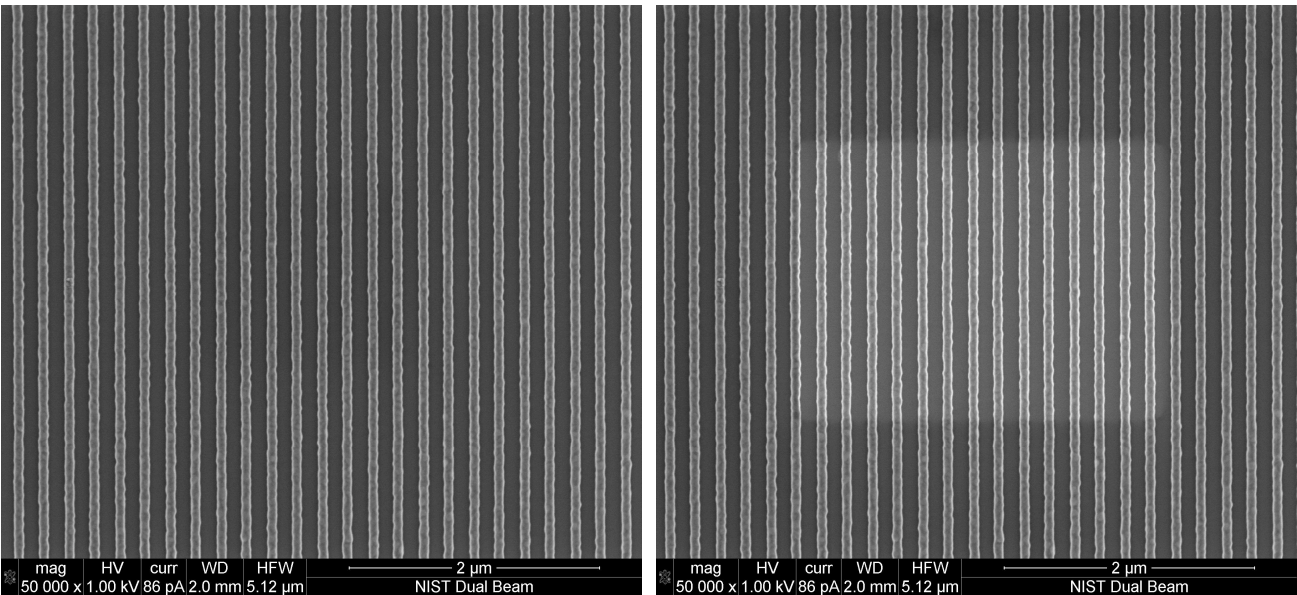


Figure 3. 1 kV, 86 pA SEM images of amorphous Si patterns at the beginning of the test (left) and after 10 minutes (left) of continuous electron beam bombardment. Actual cleaning is observable. 5 μ m field-of-view images.

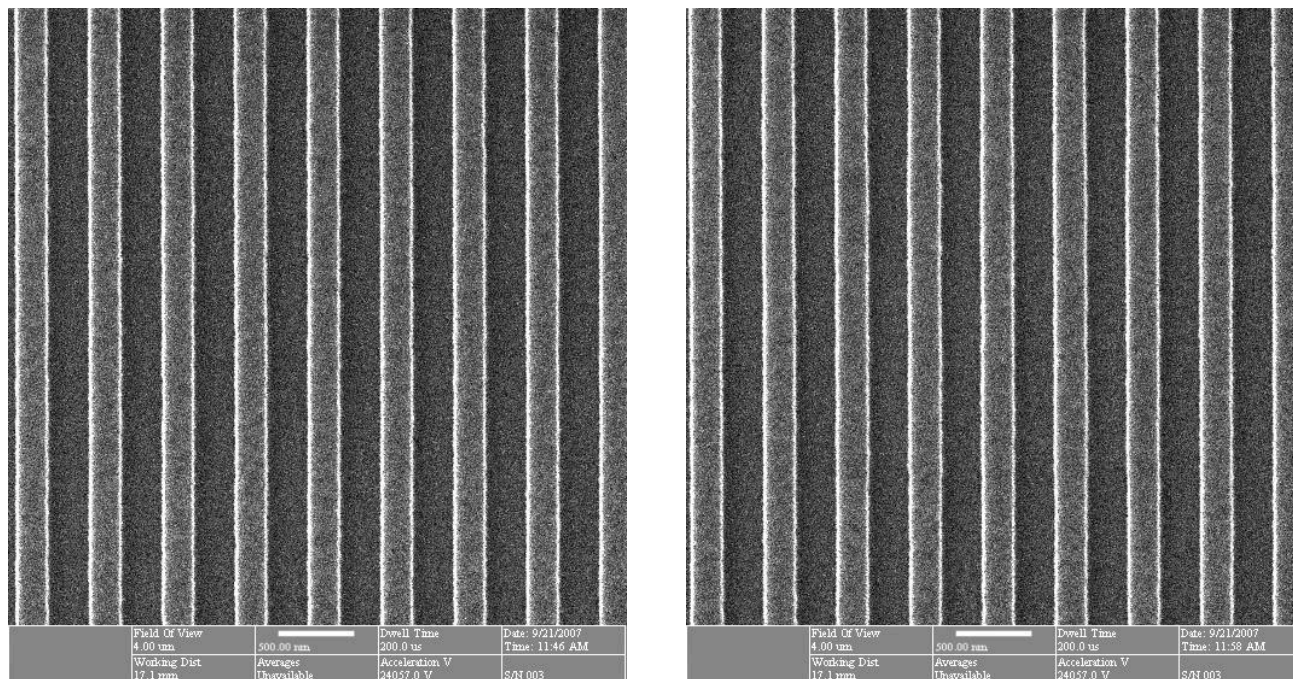


Figure 4. 24 kV, 3 pA SIM images of amorphous Si patterns at the beginning of the test (left) and after 10 minutes (left) of continuous electron beam bombardment. Essentially no contamination is observable. 4 μ m field-of-view images.

SUMMARY

Contamination is one of the worst problems of dimensional metrology SEMs, and it leads to erroneous results and decisions. The new procedures described in the paper offer an effective solution for monitoring and eliminating sample contamination. Low-energy oxygen plasma used with the minimum, but sufficient time and current is very effective to clean the instruments. Reliable and regular monitoring of the contamination performance of all SEMs is possible with clean amorphous Si wafers and chips. Essentially no contamination is acceptable, and now it is possible to fulfill this requirement.

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