

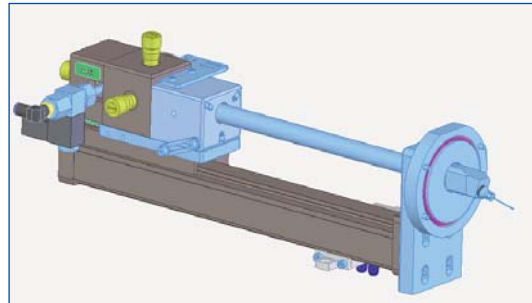
In-situ Sample Cleaning

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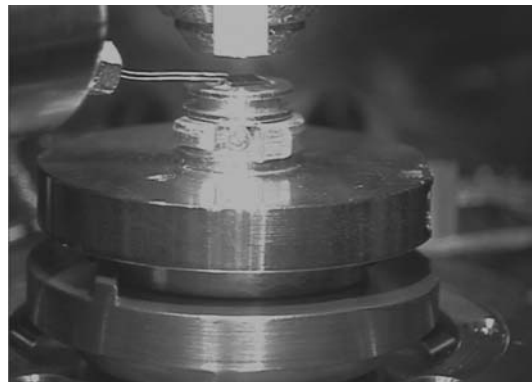
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Introduction

With ongoing miniaturization and optimization of materials, high resolution imaging with field emission scanning electron microscopes (FE-SEM) becomes of increasing importance. The integration of a charge compensation system expands investigations to all kind of nonconductive samples. In electron microscopy a main limiting factor for sufficient image quality is contamination of the sample with hydrocarbons. Volatile hydrocarbons in the residual gas can adsorb on the surface and get activated by the incident electron beam resulting in a deposition on the surface. As a result, a constant darkening of the scanned area becomes visible due to an increase of carbon deposition on the surface. The electron dispersive x-ray (EDX) spectrum shows over 90at% (atomic percent) carbon content and also 10at% oxygen content in the deposited structures. Deterioration of image quality increases with the reduction of energy used for imaging. This is due to a larger cross section of interaction at lower energies and the reduced penetration depth of low energy electrons that are more sensitive to the sample surface. Hence, contamination is a crucial problem for investigation of sensitive samples such as polymers that melt when high energy electrons are injected and therefore have to be imaged at very low energies. Furthermore, electron beam induced processes such as electron beam induced deposition (EBID) and electron beam induced etching (EBIE) will be negatively affected by the undesired carbon contribution originating from the contaminants available in the residual gas: For EBID, the already high amount of carbon in the deposits



The Carl Zeiss gas injection system is pneumatically inserted into the chamber. Alignment in x, y, z-directions is performed with the help of micrometer screws.



The stainless steel needle of the Carl Zeiss gas injection system can be individually moved as close as possible to the sample surface.

will increase further resulting in a diminished purity of the final deposit. EBIE processes may be prevented at all by a passivating carbon layer that gets instantly deposited on the sample surface before the actual etching process can even start. Finally, the deposition of hydrocarbon contaminants taints EDX spectra by adding carbon and oxygen contributions to the spectra originating from the growing residual gas deposition, which will increase during the EDX measurement. Using Carl Zeiss NTS' charge compensation system with oxygen instead of



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nitrogen offers the unique opportunity to clean the sample surface in-situ reducing sample preparation efforts to a minimum. Imaging can then be done after cleaning or even while an oxygen flow constantly prevents contamination.

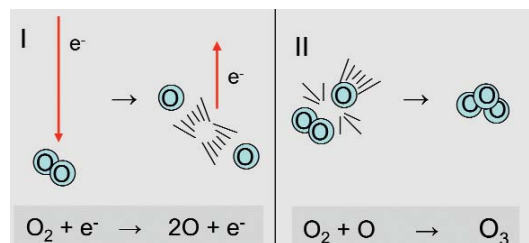
Instrumentation

Carl Zeiss NTS provides a wide range of FE-SEM systems designed to meet a whole variety of customer demands. For maximum versatility the ULTRA PLUS and MERLIN® FE-SEM, as well as the AURIGA® CrossBeam® system can be equipped with a pneumatically insertable gas injection system. A bendable stainless steel needle locally injects gas to the region of interest (ROI). The spatial limitation of the low vacuum area allows the use of all detectors that are normally used at high vacuum conditions only. Furthermore skirt effect issues, e.g. significant deterioration of image quality and reduction of resolution by beam broadening due to scattering, are drastically reduced in comparison to environmental systems where the chamber is at low vacuum entirely and, hence, the interaction volume is much larger. The needle can be adjusted in all directions. With its bayonet coupling it can easily be exchanged for alternative shapes that can be used for special applications such as EBSD. Gas flow is software controlled by slider from 0 to 100% that is equivalent to a system vacuum from approx. 6×10^{-7} mbar up to 7×10^{-3} mbar.

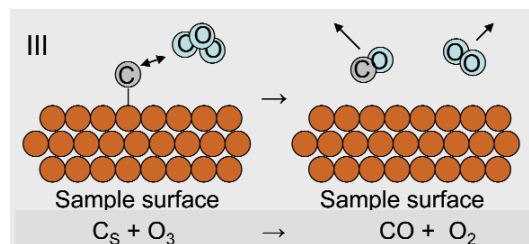
Basic Principles

Hydrocarbon contamination is a well known problem in electron microscopy. It is difficult to prevent because all surfaces exposed to room air at atmospheric pressure accumulate hydrocarbons. The trace elements are then dissociated by the electron beam and carbon is deposited on the sample surface. This carbon contamination is normally visible as a dark rectangle or frame, depending on the scan speed and contamination level of the chamber and sample. The result is a blurred image with reduced resolution and less, if any, detailed information.

Two possible cleaning mechanisms are described in the following images:



Ionization of oxygen molecules. Ozone generation.



Carbon contamination on sample generated by dissociation of hydrocarbons via electron beam is removed by oxygen radicals or by ozone, forming CO and O₂.

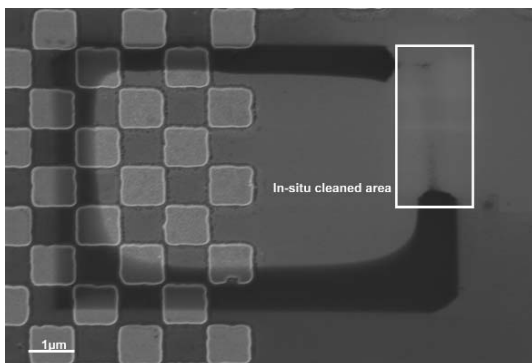
For better imaging results every effort has to be made to clean the sample prior to loading. Depending on the degree of contamination the specimen has to be wet and/or dry cleaned and baked-out. This way of preparation can be very time consuming while its success is uncertain as some samples getter hydrocarbons so quickly that it is hardly possible to prevent a monolayer of hydrocarbons. In other cases the chamber of the microscope itself is contaminated due to investigations of polluted samples whose impurities evaporated into the vacuum. With the Carl Zeiss NTS gas injection system the sample is cleaned locally with oxygen radicals within the chamber.

With the delivery of a constant oxygen gas flow from the Charge Compensation with in-situ cleaning, the secondary electrons and low energy back-scattered electrons fragment oxygen molecules into oxygen radicals. Partly, the free single oxygen radicals react with other oxygen molecules to form highly reactive ozone, but this reaction is believed to be secondary. Oxygen radicals and ozone are highly-reactive agents capable of efficiently oxidizing hydrocarbon species to volatile species such as carbon dioxide (CO₂) or carbon monoxide (CO), which can easily be removed by the pumping system of the vacuum

chamber, leaving a clean surface. The amount of radicals created, which directly corresponds to the intensity of the cleaning process, is controlled by gas flow, electron energy and current. Hence, at low electron energies the cross section of interaction between oxygen and electrons is large, facilitating the formation of radicals. Increasing the effect can simply be realized by changing the probe current, reducing the scan speed or, alternatively, using a reduced raster. The distance between sample and orifice of the needle should be kept small in order to most efficiently use the small volume of oxygen gas at higher pressure.

The following examples show the use of in-situ cleaning in its two working modes.

1.) Cleaning the ROI of the sample prior to image acquisition is applicable if only the sample and not the chamber is contaminated. Example A shows a contamination frame on a previously uncleaned silicon/gold calibration sample, obtained by slow scanning for 30 mins. Parts of the carbon were removed by in-situ cleaning in less than 1min using a reduced raster at high current and slow scan speed. For high resolution imaging it is now possible to zoom into the cleaned area without observing further deterioration of image quality. This working mode is highly useful for time-efficient high resolution investigations where only a certain part of the sample has to be cleaned for better imaging results. For low energy investigations the needle can be retracted after cleaning.



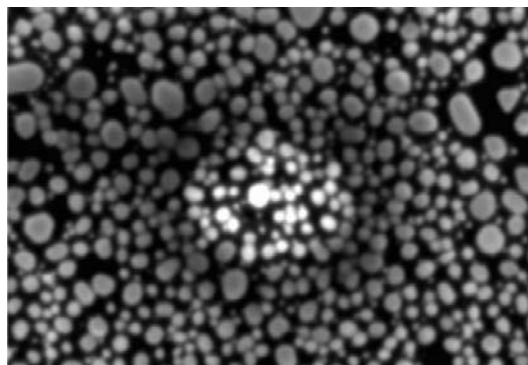
A: Contamination frame on previously un-cleaned calibration sample, obtained by slow scanning for 30 mins, parts were removed by in-situ cleaning in less than 1 min (reduced raster, slow scan speed, high current).

Image acquired at 1 kV, in-lens SE detector.

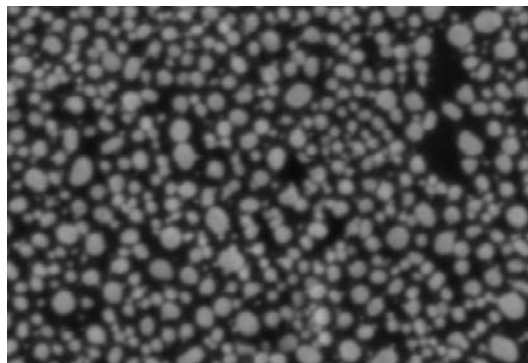
2.) Cleaning the ROI of the sample while imaging is recommended if contamination is predominant in the chamber (hence, a cleaning step before imaging would not help) or if the sample is too sensitive for high doses of electrons. In this mode, depending on the degree of contamination, intensity of the cleaning can be reduced as the generation of carbon is mitigated immediately and no existing residues have to be removed.

A gold on carbon sample, deliberately contaminated prior to loading, was used as an example.

In-lens image B was acquired without oxygen flow with 1 kV landing energy at 150kX magnification. Prior to image acquisition the same ROI was scanned at 600 kX for 1 minute. Dark and bright areas of contamination are clearly visible. In-lens image C shows the same sample but was now acquired with oxygen flow with the same set of parameters. No ablation of carbon can be seen.



B: In-lens image of Au on C sample, deliberately contaminated prior to loading, image acquired without oxygen flow, 1 kV, WD 5 mm at 150kX mag after 1 min scanning at 600kX: contamination visible.



C: In-lens image of same Au on C sample, now acquired with oxygen flow, 1 kV, WD 5 mm at 150kX mag after 1 min scanning at 600kX: no generation of contamination visible.

Further Potential

With oxygen radicals as highly reactive components it is also possible to modify the surface of a suitable sample. This can be done – depending on the sample – by using a high dose of oxygen (100 % gas pressure) in combination with slow scanning, large current and a small ROI. Due to the electron-induced generation of ozone etching is limited to scanned regions only offering a whole new range of application possibilities.

Summary

Using the Carl Zeiss NTS gas injection system with oxygen instead of nitrogen offers the unique opportunity to clean the sample surface in-situ, reducing sample preparation efforts to a minimum. Image acquisition can then be done after cleaning or even while an oxygen flow constantly mitigates contamination. In-situ cleaning contributes to the versatility of the ULTRA PLUS, MERLIN® and AURIGA® systems, making sample investigations more time-efficient and easy.

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