Improved Carbon Analysis with Evactron Plasma Cleaning

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Analyzing carbon by EDS in an electron microscope is difficult for a number of reasons. The Carbon x-ray K line is of low energy and is easily absorbed. Its low energy means that only the Carbon x-rays from the surface of the specimen can be counted. Carbon x-rays are also easily absorbed by the x-ray detector windows. Furthermore, there can be a significant Carbon background signal, since hydrocarbon [HC] contamination can be responsible for a fast growing C peak when the beam is positioned in the spot mode. Hydrocarbons from the chamber surfaces, vacuum pumps, and sample surface migrate and react with the electron beam to form a black spot rich that is in carbon. This is where the EM analysis is to be conducted. The Hydrocarbon background also interferes with the detection of the Carbon x-rays, because an oil film can collect on the surface of the detector window. Figure 1 shows condensed oil on a detector snout and collimator. This oil file on a UTW (Ultra Thin Window) can significantly reduce the transmission of C and N rays. The analytical difficulty extends to N, whose x-ray peak is partially overlapped by the growing adjacent C peak and strongly absorbed by the carbon layer that builds up at the window surface.

Figure 1: Oil condensed on EDS detector snout and collimator. Oil on EDS detector window adsorbs C X-rays and interferes with quantitative analysis. Evactron cleaning removes this oil without damage to the UTW window

The Evactron Anti-Contaminator [A-C] removes HC contamination from the SEM vacuum and from the sample surface [1]. The Evactron A-C consists of a small plasma device, Oxygen Radical Source, [ORS] which mounts onto a suitable and available chamber port and a RF Generator/Controller.   The ORS generates O radicals in a plasma from air metered through a control valve during system pump down. The O radicals are transported from the ORS through the chamber by pressure differential, and then they ash HC into CO, CO_2, and H_2O to be pumped away. After Evactron cleaning, the chamber is pumped down to high vacuum for sample analysis.

Evactron cleaning quickly removes light surface layers of contamination for the SEM chamber. Although a full clean-up of a long-used microscope (which has been contaminated over the years) will not be completely cleaned in a few minutes, the following results show that the Evactron A-C makes low level carbon analysis possible just after installation. Complete cleaning may require repeated, short Evactron cleanings to keep the HC away for long longer periods. Monitoring of HC levels is the recommended way of determining when Evactron cleaning needs to be repeated. Monitoring C peaks by EDS is one possible monitoring method.

To test the C contamination level improvement, a pure Cu polished sample was used. The microscope, a Gemini 982, was fitted with a Noran thin window EDS. The sample was prepared using the best standard procedure recognized by the operator to give the smallest pollution: polishing, ultrasonic cleaning and final cleaning with pure ethanol. Spectra were acquired at 15 kV, and the EDS system used to measure net intensity peaked on the elements found: C and Cu La. The spot was left static for 15 minutes, and spectra were run for 50 seconds. The first spectrum was acquired immediately, followed by three others after 3, 6 and 15 minutes.

An Evactron Model C Anti-Contaminator was used for the tests. It was mounted to a port level with the sample stage. It was operated at the manufacturer recommended operating conditions of 10 Watts of RF power and at 80 Pa pressure. These conditions optimize the production of O radicals and the cleaning mechanism.

Results: The first series of spectra were acquired before Evactron A-C cleaning. The spectrum showed a C peak immediately, and this carbon peak quickly grew up from a relative C/ CuLa intensity ratio of 0.76 % to 5.06 % after 15 minutes of static beam. This can be seen on the spectrum in Figure 2. The Evactron A-C was then run 4 minutes to remove hydrocarbons. The first spectrum acquired after cleaning still showed a small carbon peak ratio of 0.61 % that grew up to 1.39 % after 15 minutes. This improvement was impressive but not enough: we do not expect to clean a sample and a large chamber that had been used for several years in 4 minutes. Therefore, a second 4 minute cleaning was done, as was a third one two hours later. This allowed time for the trapped hydrocarbons to diffuse back and be converted and removed. After the third run, the EDS system did not find any carbon in the spectrum for the spectra acquired immediately and after 3 minutes. The spectrum acquired after 6 minutes and 15 minutes showed a small increase of the C intensity that allowed the software to detect C.
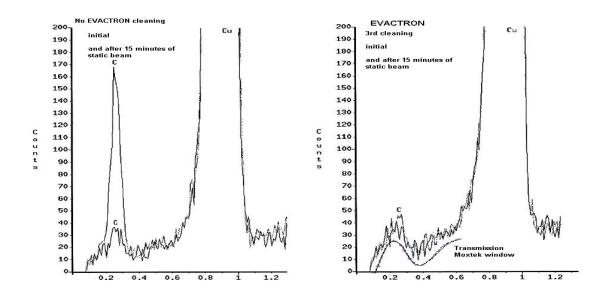
Figure 2. EDS spectra without cleaning and after third cleaning. The Moxtek, Inc. window transmission curve versus energy is added to the Evactron A-C cleaned spectrum.

Background hump in the C peak area: Although not identified as C, the spectrum showed a hump that was very close to a carbon peak shape. The Moxtek UT Window is constructed with a 3000 Å polymer with 300 Å Aluminum film [1]. The window transmission is not linear, and this hump strictly follows the window transmission curve as seen in Figure 2. When carbon was found, the software did not differentiate the C contribution from the background discontinuity. This explains the jump in the intensity that the software finds when C is found to be present, as seen in Figure 3.
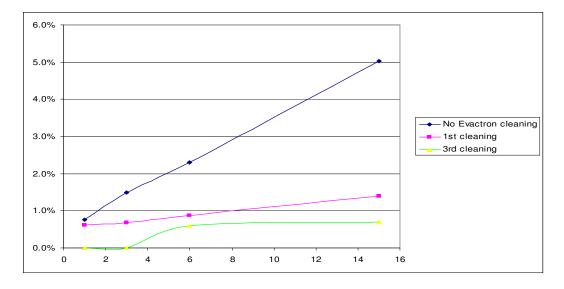

Figure 3 Measured C intensity versus time with no cleaning, after the first and the third cleaning. The measured intensity is zero if the peak is not found and integrates the background hump when C is found.

Conclusion: The Evactron A-C effectively cleaned the SEM chamber in a short time, improving carbon quantification by SEM-EDS analysis. Both sample and chamber were cleaned well enough in a total of 12 minutes by the Evactron A-C run to allow a stable, undetectable C peak for up to 3 minutes of static spot. After 15 minutes, the C intensity was smaller than the peak found immediately after positioning the beam on a sample prepared the classical way. The sample was clean enough to show the background hump that should not be confused with a small carbon peak. The Evactron A-C showed, in a few minutes, a significant improvement in cleanliness of a large chamber microscope in use for several years and sample surfaces. It was noticeable that no oxygen peak appears in the spectrum, although Cu is easily oxidized: Evactron A-C cleaning is efficient in removing labile hydrocarbons without being aggressive to the sample surface.

References:
[1] N. Sullivan et al, Microscopy and Microanalysis 8 (suppl. 2)(2002) 720CD
[2] C. Roberts, Moxtek, Inc. Application report: Improvements to Light element X-ray Windows for Si(Li) Detectors, Moxtek, Inc. Orem, Utah 2002