# A Study of the Effects of Evactron® Plasma Cleaning on X-ray Windows

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Paper presented at Microscopy and Microanalysis 2004, Savannah, GA Aug. 2-5, 2004

The Evactron® Anti-Contaminator has proven to be very effective in removing labile hydrocarbon contamination from Electron Microscope chambers [1]. Evactron Plasma cleaning has also been shown to improve Carbon analysis by X-ray spectrometry [2]. There are no reports of X-ray window failure or damage from Oxygen Radicals developed in the Evactron A-C by operators who have EDS detectors on their SEM systems.

To show that the Evactron A-C cleaning will not damage the thinnest MOXTEK™ Ultra Thin window, (the AP3.3 EDS window), MOXTEK™ and XEI Scientific performed window exposure tests on the effects of Evactron A-C cleaning. The tests consisted of exposing several AP3.3 to the oxygen radical flow in 20-80 hour continuous exposures for multiple run periods of a total of over 160 hours. During the tests, the windows were examined for leaks with a helium leak detector and 500X and were also examined for visible damage using light microscopy.

The Evactron A-C is a compact RF plasma device that creates oxygen radicals from an air bleed into the Oxygen Radical Source. The Oxygen Radical Source (ORS) is mounted on an SEM chamber accessory port and creates localized plasma. The ORS plasma does not bombard surfaces with ions, but rather cleans by chemical reaction. The free radicals ash hydrocarbons and organics into CO, $CO_2$, and $H_2O$ molecules that are readily pumped by the roughing pump. Oxygen radicals flow towards the roughing pump from the ORS through the chamber by the pumping differential. The Evactron process is gentle in order to meet the requirements of cleaning the insides of delicate vacuum instruments. Cleaning with oxygen radicals is very effective for removing organics in the gas phase and those adsorbed on the walls and other chamber surfaces, including specimens. The cleaning process very slowly attacks heavy and polymerized contamination deposits or polymer films. The low level of polymer attack results from a low concentration of oxygen radicals and a high concentration of C-C single bonds in polymers. The mechanism of the oxygen radical attack on organic molecules involves a first step hydride extraction. This results in the creation of reactive sites on the carbon skeleton that are susceptible to further oxidation. Without hydride extraction, the oxidation of the C-C single bonds in polymers has a very low reaction rate.

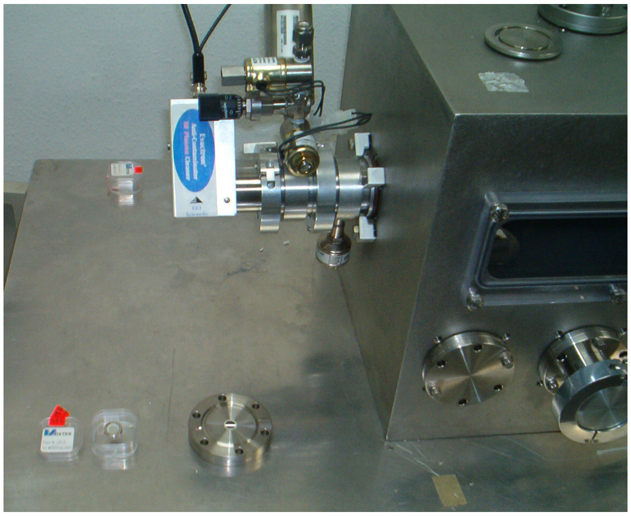

**Figure 1.** MOXTEK™ test windows # 3 & #4 shown beside test chamber # 2. Window #3 is in the CF 275 flange and window #4 is in the MOXTEK™ engineering mount. **Both have been exposed to 160 hours of Evactron cleaning and both are He leak tight.** The Evactron ORS oxygen Radical Source) is mounted to the side port of the test chamber.

On Aluminum previous tests with XPS by Walck et al (3) show no growth of the $Al_2O_3$ oxide layer over short periods of 10-20 minutes. The MOXTEK™ windows are coated with Al film. Any attack on the windows would have to begin with an attack on the Al film. The resulting $Al_2O_3$ can powder off and create a hole. This can be detected by the presence of light leaks seen by transmission optical microscopy.

MOKTEK tests windows by optical microscopy and He leak detection. The windows all left the factory with He Leak rates below the baseline rate of 2.2x10-10 milli bar Lit/sec.

**Experimental**: The first three MOXTEK™ AP3.3 EDS Windows were mounted onto a CF 2.75" flange with a hole in center. The flange was placed in Evactron test vacuum chamber, at the same location each testing period, in the oxygen radical flow. The AP3.3 is the thinnest MOXTEK™ window and provides best light element x-ray transmission. The AP3.3 window front surface is 30 nm Al film. It acts as a secondary gas barrier and blocks photons.

The windows were positioned in the area of maximum cleaning oxygen radical concentration in two different XEI Scientific test chambers, pumped, by an oil rotary pump. Test chamber #1 was approximately 14"Ø x 16" high. Test chamber # 2 was an 11" cube with observation windows on the side. The windows were mounted on a circular flange with a hole behind the window to equalize the pressure. Oxygen radical interaction with the window backside was minimized by providing only small clearance to the vacuum relief passage. The Evactron A-C was operated under XEI Scientific's recommended operating conditions of 0.6 Torr and 10 Watts of RF power at 13.56 MHz with room air as the feed gas. The first test window was exposed to the oxygen radical flow in ~ 30 hour continuous exposures for five run periods, for a total of 160 hours. Additional windows were exposed for longer periods to search for a maximum exposure limit.

After each exposure to oxygen radical flow, the windows/flanges were tested for leaks and damage. The windows were tested for a detectable helium leak, with the limit of detection at 3x10-10 mbar L/sec. Pictures of the window were also taken and examined after each oxygen radical exposure run period to examine any degradation of the window.

**Results:** Window 1: After 40 hours of continuous exposure, some pin hole light leaks were noted (See Figures 1 and 2.) The size of these light leaks did not increase in subsequent examinations at 70, 100, 127, and 160 hours. After 100 hours of exposure, a small leak of 0.6x10-10 milli bar Lit/sec appeared in the first window due to handling damage as identified by microscopy at MOXTEK™. The leak was within MOXTEK™ quality standards, but it raised a question about the data. This leak did not worsen with two more exposures, but the testing was stopped on the first window after 160 hours of exposure in favor of testing a second window. The tests were done in test chamber 1.

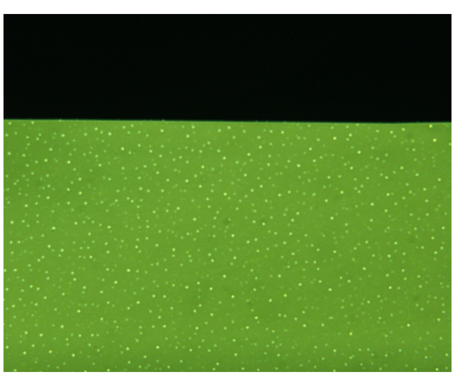

**Figure 2.** A transmission mode micrograph close up of window 1 shows a multitude of small light leaks after first 40 hours of exposure to Evactron cleaning. **No Vacuum leak was found.** The light leaks did not change with increasing exposure time and were not repeated on other windows. Dark area is the underlying support grid.

Window #2 was destroyed in shipping back to Moxtek after an initial exposure of 40 hours.

Window # 3 was exposed for three exposures: 60 hours, 24 Hours, and 62 Hours (146 hour total) in test chamber 2. After the last exposure, a small number of light leaks (65) appeared in the Al coating. No increase in He leak rate was detected. This was the highest amount of damage seen on any of the test windows, and was not repeated on the other windows.

The fourth and fifth windows were mounted in smaller MOXTEK™ "engineering" mounts rather than the CF 40 flange. XEI Scientific obtained its own optical microscope and He leak detection equipment to speed the testing. The exposures were done in test chamber #1.

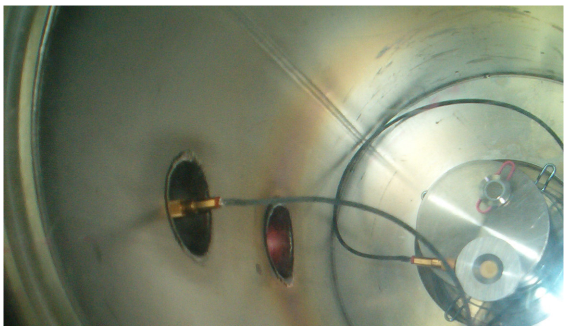

**Figure 3** Interior of Chamber #1 showing Evactron plasma pink glow during Evactron cleaning exposure of window # 5. A quartz thin film thickness monitor is also shown in chamber placed on pedestal beside window.

Window #4 was exposed to 20 hours of cleaning between each examination by light microscopy. No light leaks were noted until 160 hours of exposure, when one pinhole light leak was observed. No vacuum leak was found by He leak detection.

Window # 5 was exposed to three long period of continuous exposure to see if duration of exposure to Evactron Cleaning affected the results. No pin holes were seen at the end of the first exposure of 66 hours. After another 46 hours of continuous exposure, 112 hour total, no light leaks were seen - even after a second examination was done following rapping the mount rim to dislodge any loose Alumina from the surface. After another 48 Hours of exposure (160 hours total) no light leaks were seen.

**Discussion**

Window failures due to vacuum leakage did not occur to the periods tested of about 160 hours on all the windows. The results on window 3 suggest that the duration of Evactron cleaning may affect the rate at which pinholes appear in the Al Coating of the window, but the test on window #5 did not confirm this result. Continuous Evactron cleaning may cause more window light leaks than shorter exposures, adding up to the same exposure. Continuous exposure may actually be more rigorous than multiple exposures for the following three reasons. 1) Typically, the cold window surface will trap hydrocarbon contamination in the chamber, thereby protecting the Al window from oxygen radicals during typical short cleaning periods. 2) Continuous exposure subjects test windows to higher oxygen radical concentrations. Therefore, there is the potential for more damage since surface hydrocarbon contamination entering the vacuum chamber between sample exchange cycles is not present. 3) Attacks on the Al crystal structure may depend on weak points or openings in the $Al_2O_3$ oxide coating. These weak spots may heal when exposure to Evactron cleaning stops.

Vacuum leaks are the usual mode of failure for MOXTEK™ UTW windows. Vacuum leak causes can be determined under the microscope by MOXTEK™ Inc.. Vacuum failures due to impacts, corrosion, materials failure, or workmanship errors are easily identified by microscopy. He leak detection is done on each new window as part of quality assurance by MOXTEK™ Inc. None of the Evactron cleaned windows- except window #1- showed any vacuum leakage increase. Window #1 showed evidence of mechanical damage after shipping that was blamed for the slight increase in the He leak rate. Further Evactron cleaning did not increase the leak on this window. It is not known how much continuous Evactron cleaning would be needed to crate a vacuum leak, but from the appearance of the windows and known polymer decay rates due to Evactron cleaning, XEI estimates it would be well over 200 hours and possibly over 500 hours of continuous exposure.

MOXTEK™ has no specification for Light Leaks. During Evactron cleaning, light leaks can cause a increase in background on EDS detectors and could possibly open up the underlying polymer film to corrosion. The multitude of small light leaks on window # 1, seen after the first cleaning, did not increase in size with further cleaning. Window #1 had a multitude of light leaks which were seen after initial cleaning. The other windows showed no such defect. The results suggest that if the UTW is subjected to excessive Evactron cleaning and light leaks do begin to appear, the first symptom would be a rise in the background levels in the EDS X-ray spectrum.

Under the usual and recommended operating conditions, the Evactron A-C cleaning cycles have a duration time of 2 to 5 minutes. The number of cleaning cycles varies greatly from SEM to SEM and from lab to lab. In most labs, Evactron cleaning is performed only when image quality begins to deteriorate. In some cases, Evactron cleaning is performed daily. Using cleaning periods of 5 minutes, 160 hours of exposure is equivalent to 1,920 cleaning cycles. If there is 1 cleaning cycle/day and 5 SEM operating days/week, then the total test period of **160 hours equals 8.7 years of 5-minute daily exposures.**

**Conclusions**: 160 hours of exposure did not adversely attack the mechanical structure and leak tightness of the four AP3.3 MOXTEK ™ windows. Evactron testing time periods were selected to extend far beyond the normal expected lifetime of an AP3.3 window at normal Evactron cleaning periods. On a typical analytical SEM, the Evactron A-C is operating 5 minutes a week to suppress contamination build-up and maintain image quality. In cases where contamination is introduced to the system daily Evactron A-C cleaning may be needed more frequently. In the extreme case of daily cleaning at 5 minutes/day, the total Evactron A-C use per year would be about 22 Hours. About 4 ½ years is needed to obtain 100 hours of exposure. MOXTEK™ windows are guaranteed against failures due to materials and workmanship for 16 months and typically last for several years before replacement. On four different Moxtex windows 160 hours of cleaning caused light leaks but did not cause the windows to fail vacuum leak tests. The total test time of 160 hours is well over the exposure time a window would see during its normal life and normal Evactron A-C cleaning periods. **The use of Evactron Cleaning is safe for MOXTEK™ UTW windows as long as XEI Scientific recommended cleaning practices are followed.**

**XEI Recommendations:**
These are the recommended cleaning practices of XEI Scientific:

- **On SEMs equipped with MOXTEK™ UTW equipped detectors, Evactron clean for 5 minutes periods or less and vent the chamber between cleanings.**
- **Evactron clean for no more the 30 minutes daily.**
- **Only Evactron clean when contamination effects image quality or light element x-ray sensitivity.**
- **Never use Evactron Cleaning overnight.**

Following these recommendations will result in the UTW window lasting its expected normal lifetime of more than 5 years without trouble.

**References:**
[1] N. Sullivan et al, A STUDY OF THE EFFECTIVENESS OF THE REMOVAL OF HYDROCARBON CONTAMINATION BY OXIDATIVE CLEANING INSIDE THE SEM. Microscopy and Microanalysis 8 (suppl. 2)(2002) 720CD
[2] P. Rolland et al, IMPROVED CARBON ANALYSIS WITH EVACTRON PLASMA CLEANING, Microscopy and Microanalysis 2004

[3] Walck et Al, XPS EVALUATION OF SAMPLES SURFACE CLEANED BY THE XEI EVACTRON®, Microscopy and Microanalysis 7 (suppl. 2)(2001)