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The Effects of Evactron® Plasma Cleaning on Moxtek® X-ray Windows

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The Evactron family of plasma cleaners has proven to be very effective in removing adventitious hydrocarbon contamination from electron microscope chambers. Evactron plasma cleaning has also been shown to improve throughput with fast pump down to obtain the best analytical data with decreased noise [1]. Over 14 years of use there are no reports from operators who have EDS detectors on their SEM systems of X-ray window failure or damage due to oxygen radicals generated by the Evactron plasma cleaner.

The oxygen radicals created in the plasma oxidize carbon compounds, producing CO, CO_2 and H_2O, which are evacuated from the instrument. Quantum chemistry rules regarding energy loss state that these oxygen atoms do not react with diatomic molecules in two body collisions, but require a third body to kinetically carry away excess energy. Oxygen radicals also react on solid surfaces such as metals where they can react or recombine with hydrocarbons. Numerous studies performed at XEI Scientific using previously contaminated quartz crystal microbalances to measure cleaning rates have shown that Evactron plasma cleaning is very effective at removing hydrocarbons [2]. Faster decontamination rates were documented in SEMS and FIBS equipped with turbo molecular pumps.

As an update to an earlier study [3], Moxtek and XEI Scientific performed window exposure tests to show that Evactron plasma cleaning will not damage the thinnest Moxtek window, the AP3.3 EDS window. The tests consisted of exposing several AP3.3 windows to the oxygen radical flow from an Evactron Model E50 in an experimental vacuum chamber in 20 hour continuous exposures for multiple run periods to total over 200 hours.

Experimental Procedure: Moxtek windows were placed on a platform in a 20 L vacuum chamber in the path of oxygen radical flow. The placement of the Moxtek windows was the same during each plasma exposure test. Testing was done in high vacuum with the turbo molecular pump on. Interaction of the back side of the Moxtek window with the oxygen radicals was minimized by a fine mesh on the platform on which the windows were placed. In practice, the back side of the window faces the x-ray detector and will never experience plasma cleaning within the SEM. Only the front side of the window is exposed to plasma cleaning.

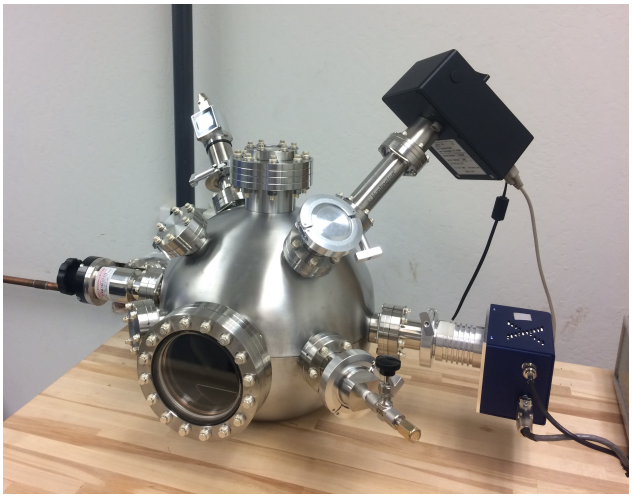
The Evactron Plasma Cleaner was operated at turbo molecular pressures (~3 mTorr operating pressure) at two RF power levels of 20 Watts and 50 Watts at 13.56 MHz with room air as the process gas. Two sets of windows were used with a set of two windows for each power level. Moxtek windows were exposed to a continuous flow of oxygen plasma at 20 Watts for 20 hours at a time. After the first 20 hour long plasma exposure, one of the windows was removed from the 20 L chamber and returned to Moxtek for evaluation (helium leak test and visual inspection of any surface damage). The second Moxtek window was exposed for another period of 20 hours of continuous plasma, then sent back to Moxtek for evaluation. This process (involving Moxtek windows being exposed to plasma, sent for evaluation and then sent back to XEI Scientific for another plasma cleaning cycle) was repeated multiple

times for up to 200 hours of cumulative plasma exposure. The experiment was repeated with a second pair of Moxtek windows using exactly the same set-up but a higher power of 50 Watts.

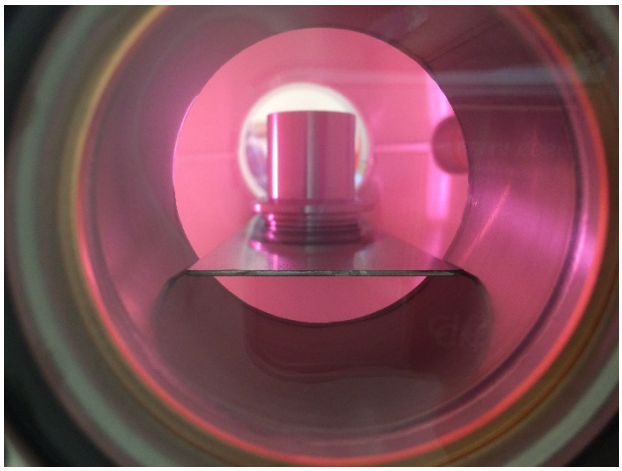
Evactron plasma cleaning does not damage Moxtek AP3.3 windows. Under usual operating conditions the Evactron E50 cleaning cycles are 5 to 10 minutes long, and each microscopy lab follows a tested protocol. Using cleaning periods of 5 minutes, 160 hours exposure is equivalent to 1,920 cleaning cycles, or 8.7 years of daily SEM cleaning. Inspection of the Moxtek windows after 40 hours of plasma exposure did not reveal any damage to the windows. Results from prolonged exposure will be presented.

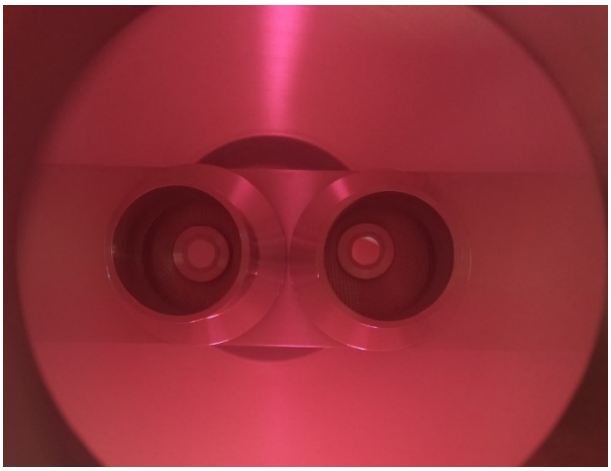
References:

[1] E. Kosmowska *et al*, Microsc. Microanal. **23 (Suppl. 1)** (2017), p. 74.
[2] R. Vane and C.A. Moore, Microsc. Microanal **20 (Suppl. 3)** (2014), p. 2014.
[3] R. Vane *et al*, Microsc. Microanal **10 (Suppl. 2)** (2004), p. 966.



Figures 1 and 2. Moxtek AP3.3 windows and experimental apparatus at XEI Scientific.





Figures 3 and 4. View of the Moxtek AP3.3 windows in the vacuum chamber during plasma operation.

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