Immobilization and Removal of Hydrocarbon Contamination Using the Evactron® De-Contaminator

Ronald Vane

XEI Scientific Inc., 1755 E. Bayshore Rd. Suite 17, Redwood City, CA 94063

Background

The Evactron® De-Contaminator is well known for its ability to reduce and remove Hydrocarbon contamination artifacts in Electron Microscopes. The primary mechanism for this control is the oxidation of HC deposits to form short chain volatile compounds and oxides that can be pumped out the vacuum pumps. Comparison of RGA results on the removal of volatile components and visual observance of the removal of Hydrocarbon films indicates that the immobilization of Hydrocarbons on surfaces by Polymerization is also an important mechanism for reducing contamination interference with imaging in electron microscopy.

Figure 1 Evactron De-Contaminator Model 10

Experimental

XEI uses a vacuum chamber equipped with a large window to study Hydrocarbon removal using the Evactron De-Contaminator. The Evactron De-contaminator uses RF plasma in an Oxygen Radical Source (ORS) connected to a chamber port to make O radicals from air, and then uses the pumping differential to flow the Oxygen radicals though the specimen chamber and oxidize the HC on all surfaces. Removal involves forming short chain molecules that can be volatilized and carried to the roughing pump such CO, CO2, H2O, and short chain organic oxides such as alcohols, ketones, and esters. Polymerization involves cross linking the Hydrocarbon molecules under the influence of the oxidizing Oxygen radicals. The Oxygen radicals are most apt to attack by Hydride extraction, the removal of Hydrogen. If the resulting free radical site is not attacked quickly by another Oxygen radical, polymerization with adjacent molecules can occur. Polymerization creates longer chain molecules that are less volatile and are more resistant to oxidation due to the higher energy of the Carbon – Carbon single bond.

Three tools were available to study Hydrocarbon removal. The simple method was to observe the removal of deposits from mirrored surfaces though the window on the chamber. Quantitative results on the removal of thin layers of hydrocarbons can be done using a quartz crystal thin film thickness monitor. A Residual Gas Analyzer can be used to study the reduction of the hydrocarbons partial pressure in the chamber. The RGA is pumped with a turbo molecular pump inside a Pfeiffer Vacuum dry pump package. For Evactron operation a rotary vane pump is used to maintain a pressure of 300 -1000 miliTorr and viscous gas flow inside the chamber. To the use the RGA, the Pfeiffer TMP is started and rotary vane pump is valved off when the pressure reaches 10 milliTorr. Once the TMP reaches full rotation of 1500 RPM and the pressure is below 10-4 Torr the RGA can be turned. If Evactron cleaning use been done before hand, water vapor pressure will be low enough for useful RGA spectra to be taken almost immediately.

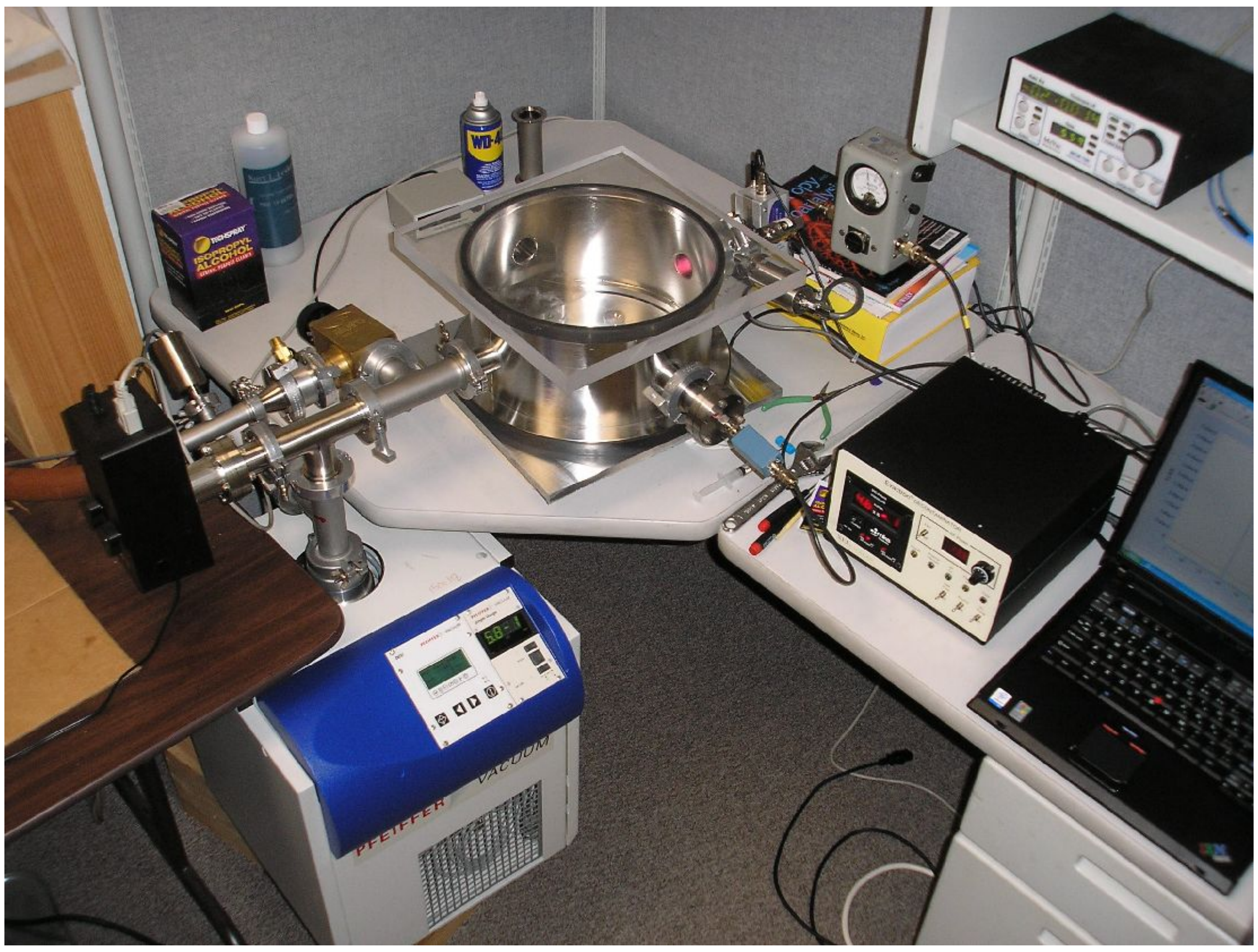


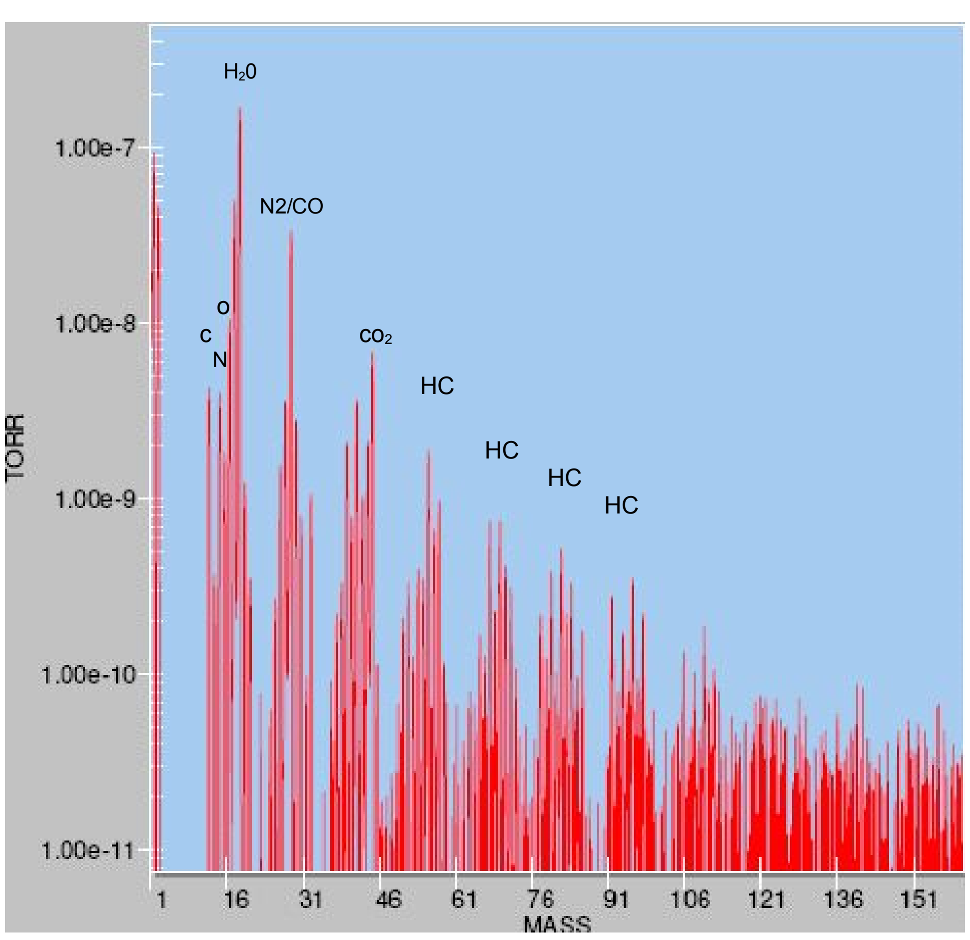
Figure 2 Evactron mounted on test chamber with RF power meter, RGA and quartz crystal monitor attached. Pink glow is Evactron plasma.

In the XEI chamber, we study the removal of Hydrocarbon deposits from mirrored surfaces because these results are easily observed end point for cleaning. A simple test is to use skin oil on a IPA wipe that is passed across the surface of mirrored surface such as a silicon wafer or stainless steel mirror. The resulting grey film is easily observed during Evactron cleaning and usually quickly disappears. Other types of oil have also been used in this test. One observes that position and thickness are variables that effect removal rate. On thick deposits, Evactron cleaning creates dark areas and these dark deposits then are removed more slowly. If there is liquid oil present on the surface, Evactron cleaning makes these deposits become tacky and sticky. These black or tacky areas are attributed to be oxidation induced polymers. Studies with the RGA showed that these black or sticky deposits showed very low HC partial pressures. Thus polymerization by Evactron cleaning immobilizes the HC deposits by polymerization that reduces the HC vapor pressure and prevents the migration of the HC to region being scanned. Contamination on the specimen volatile HC is prevented.

RGA Results

An Extorr XT 300 RGA was connected to the chamber with a Turbo pump pumping station so that the chamber could be pumped down low enough for the RGA to operate. The Extorr RGA was selected because it contains a pirani gauge as well as high vacuum ion gauge for total pressure measurements. It is designed to be tolerant of medium vacuum (10-4 Torr) operation and can be turned on early in the pump down process. The data showed that the black polymer deposits were not incompatible with clean RGA spectra. This finding shows that immobilization of Hydrocarbons (HC) by Evactron cleaning may be an important mechanism for preventing electron beam induced contamination in images.

The Evactron cleaning significantly improves pump down times on vacuum chamber exposed to air. On our test chamber the pump down time to turn on pressure for the RGA is dropped from over 10 minutes to less than two minutes after Evactron use . The accumulation of Water vapor on vacuum surfaces is the main contributor to long pump down times. The Evactron ORS plasma generates UV light which desorbs water vapor from the chamber walls. The water vapor is then efficiently carried to the pumps by the viscous flow transport mechanism. In addition we observed AMC hydrocarbon peaks in chambers left open to room air. Evactron cleaning during pump down was found to remove these AMC peaks from the RGA spectrum.



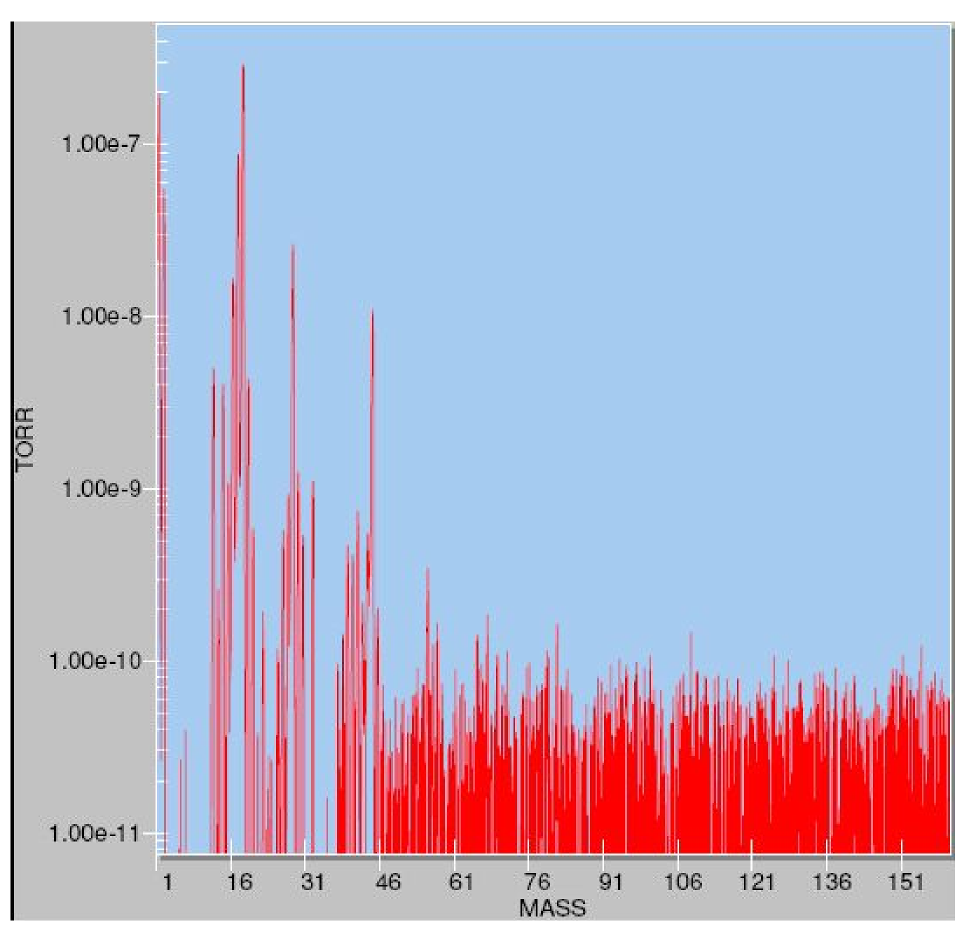


Fig. 3. Chamber before cleaning showing HC peaks with common air peaks. Fig. 4 Chamber after a 5 minute Evactron Cleaning. HC removed.

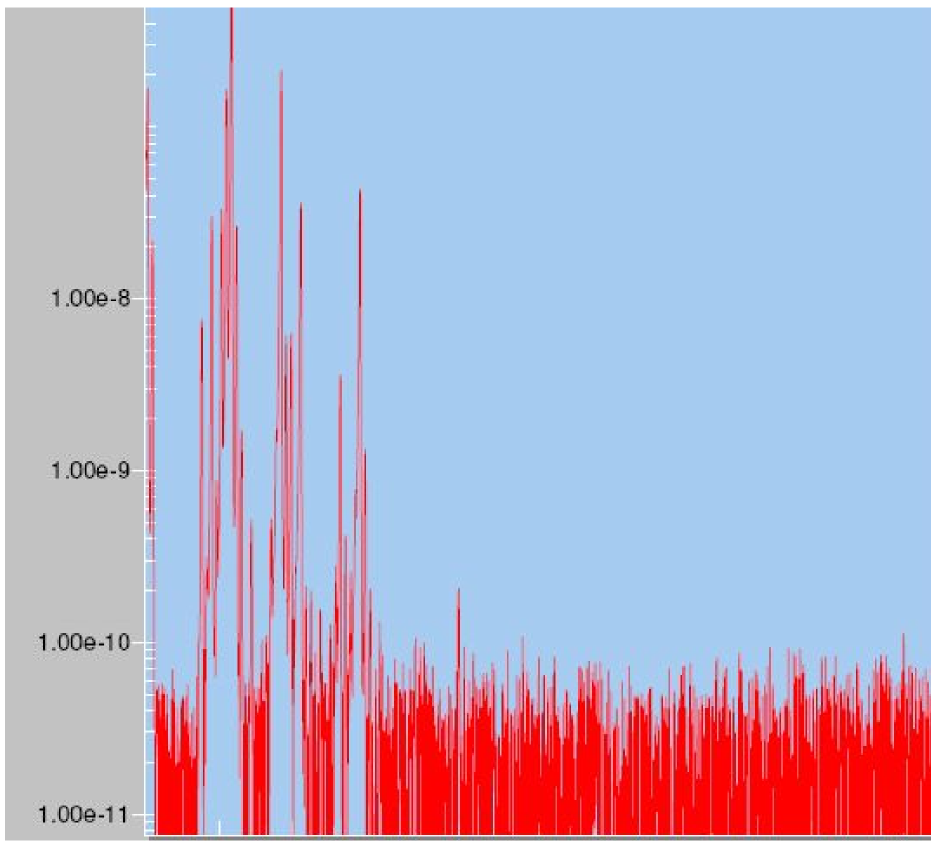


Fig. 5 Chamber after 2 hours more of Evactron cleaning showing complete removal of HC peaks.

Evactron cleaned chambers stay clean after cleaning if there are no sources of HC.

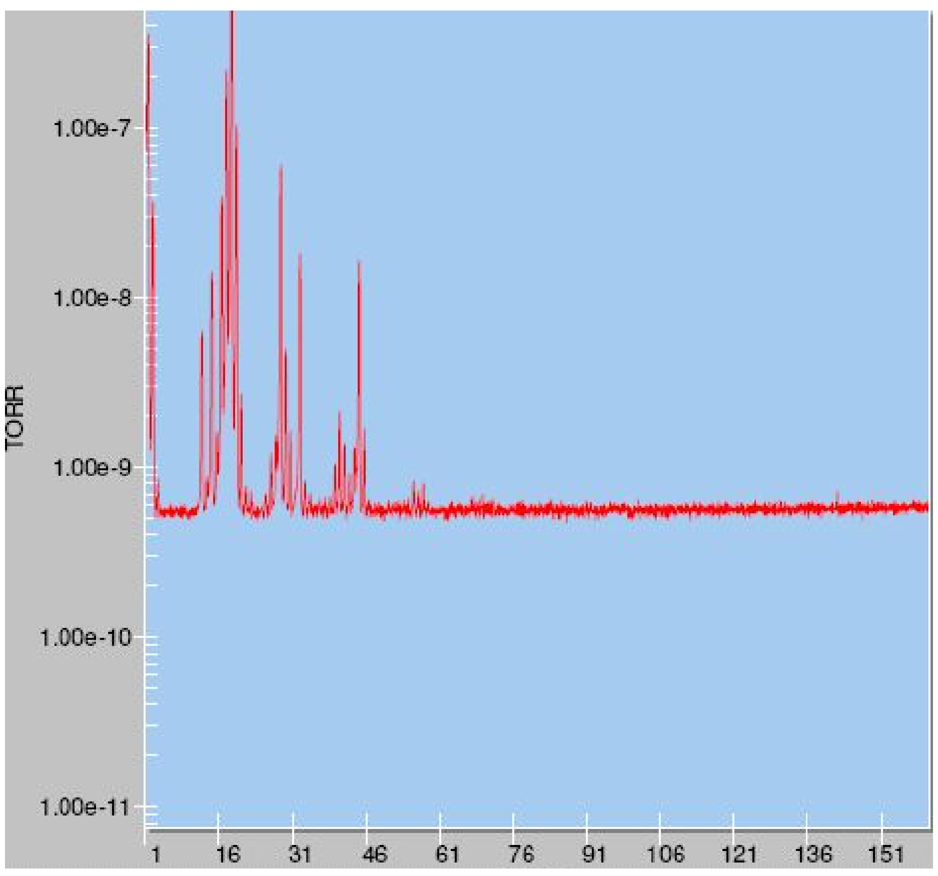


Fig. 6 After exposure to room air for 4 days, before cleaning (F424)

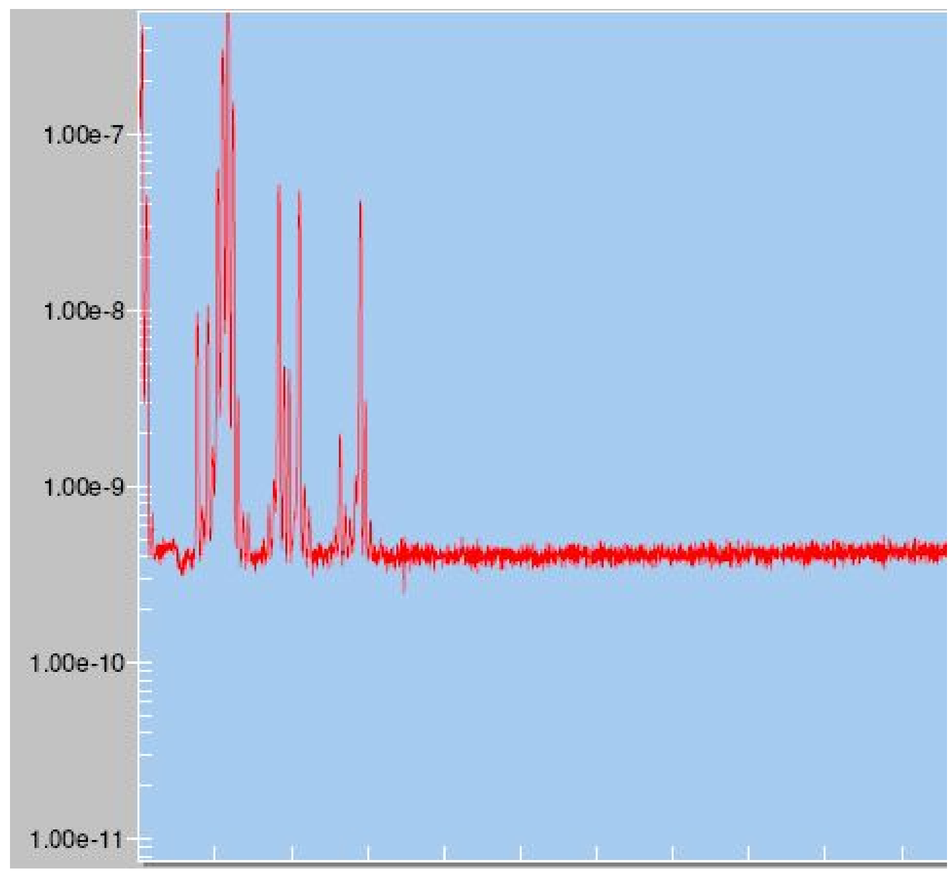
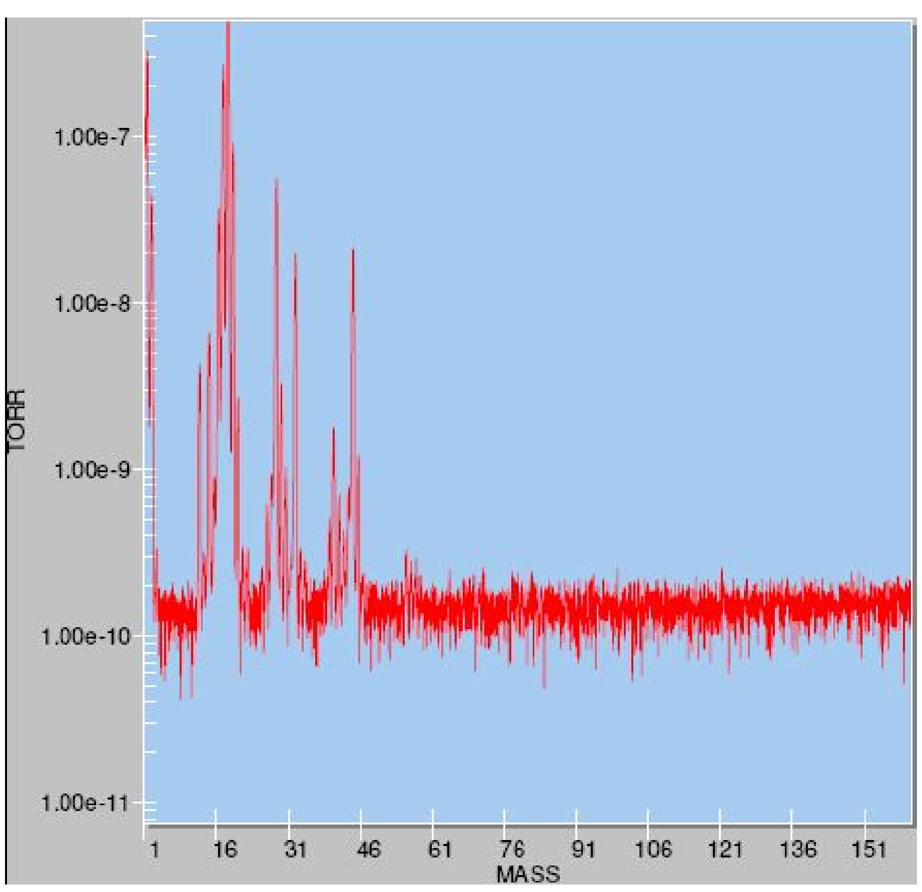


Figure 7 Evactron cleaning removed HC at M/E 54-58 (F424)



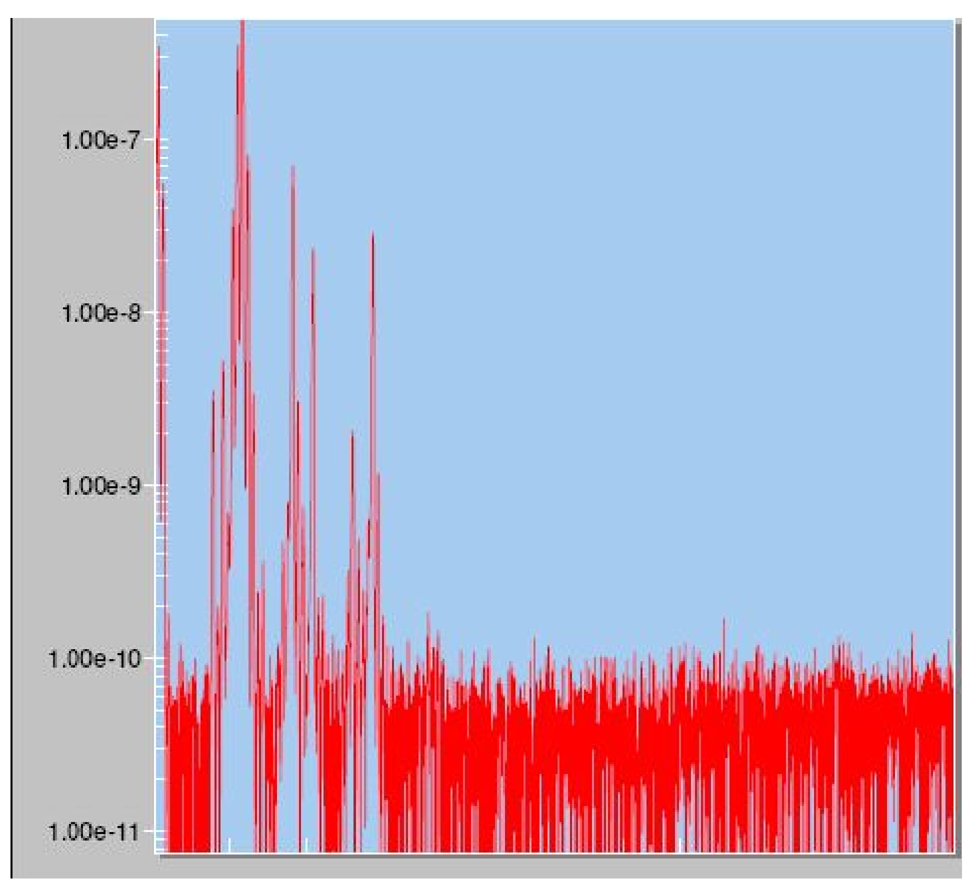


Figure 8 After 4 hours of pumping F0424 is still clean with lower baseline pressure. Figure 9 After overnight pumping with TMP baseline is lower. HC did not return.

Contamination return after cleaning:

Virtual leaks of HC into the chamber can cause the return of HC after Evactron cleaning. Test chamber GAO was built with outside welds and the inside joints are contaminated with machine oil. The chamber was Evactron cleaned until all HC peaks were removed from the RGA spectrum as shown in Fig. 10:

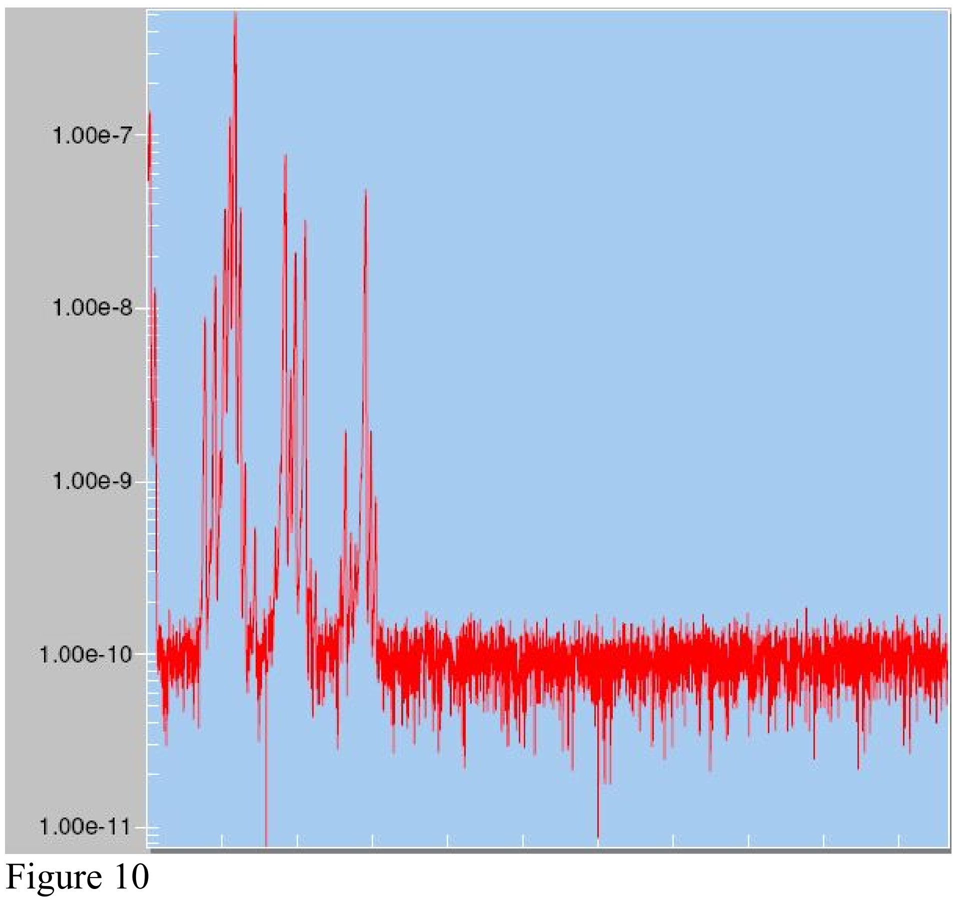


Figure 10

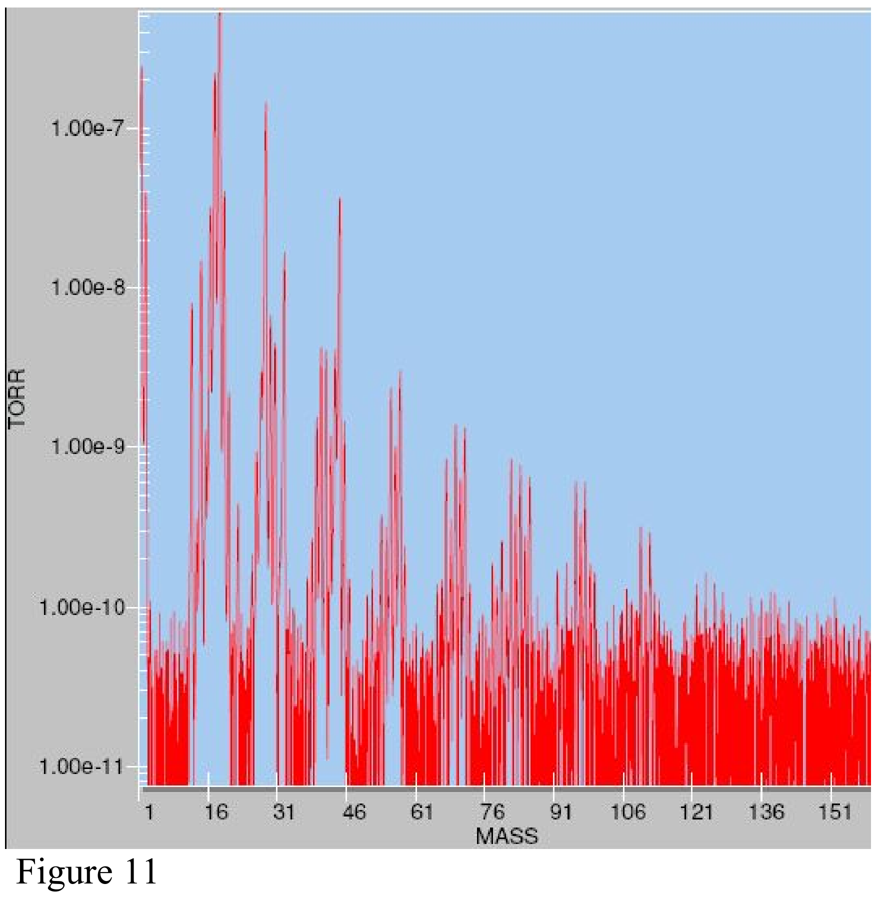


Figure 11

In Figure 11 after one hour continuous pumping with the TMP the RGA baseline is lower at 5 X 10-11 but HC peaks have reappeared. This shows that Evactron cleaning removed all of the HC on the exposed surfaces, but failed to clean HC in hidden areas. The HC is desorbed from its hiding places and redistributes about the chamber. Good vacuum design is needed to prevent the return of HC from virtual leaks inside the chamber.

Removal rates with Quartz Crystal Monitor

To measure removal rates with the quartz crystal monitor, a drop of IPA with a small amount of vacuum pump oil is placed on the quartz crystal face. The IPA is evaporated under vacuum leaving a deposit of oil. The thickness of the deposit is typically between 1000 -2000 Å. The evaporation of the IPA in the deposit can monitored. Once the thickness change is stable at < -.2 Å /sec then the Evactron ORS plasma is turned on and the rate of thickness change can be monitored. It is possible to then change the Evactron power levels and vacuum to monitor cleaning rates as expressed as thickness removal in various portions of the chamber.

The full results with quartz thickness monitor will be reported when a full data set is completed, and we are sure the results are reproducible. Preliminary results showed some the following trends.

Cleaning rate is position dependant with locations closer to the ORS being cleaned faster.

On light and thin deposits of oil the thickness of the deposit is quickly reduced and is removed by Evactron cleaning at rates of 0.4Å to 3.0 Å per minute depending on position in chamber

On thick oil films the thickness increases for 15 -30 minutes while the oil is oxidized by the Oxygen radicals, and an oxygen contain polymer is formed. The thickness then starts to decrease.

Better cleaning rates are achieved at 400 mTorr pressure and 14 Watts RF power.

Conclusions

Evactron cleaning reduces the partial pressures of Hydrocarbon molecules in vacuum chambers by both removal of Hydrocarbons by oxidation to volatile components and by polymerizing the Hydrocarbons to reduce their partial pressures. This correlates with Evactron Cleaning reducing contamination artifacts in electron microscopy by both oxidizing hydrocarbons and by inducing polymerization that reduces Hydrocarbon vapor pressure. Vapor phase transport of Hydrocarbon to specimen surfaces and into the electron beam is probably an important mechanism for the spread of contamination in a chamber to the imaged area. Data shows that reducing vapor phase Hydrocarbons by the use of Evactron cleaning to remove it or to immobilize it is the probable mechanism that improves imaging.