Remote Plasma Cleaning from a TEM Sample Holder with an Evactron® De-Contaminator

Christopher G. Morgan, David Varley, and Ronald Vane, XEI Scientific, Inc.,
1755 E. Bayshore Rd., Suite 17, Redwood City, CA 94063

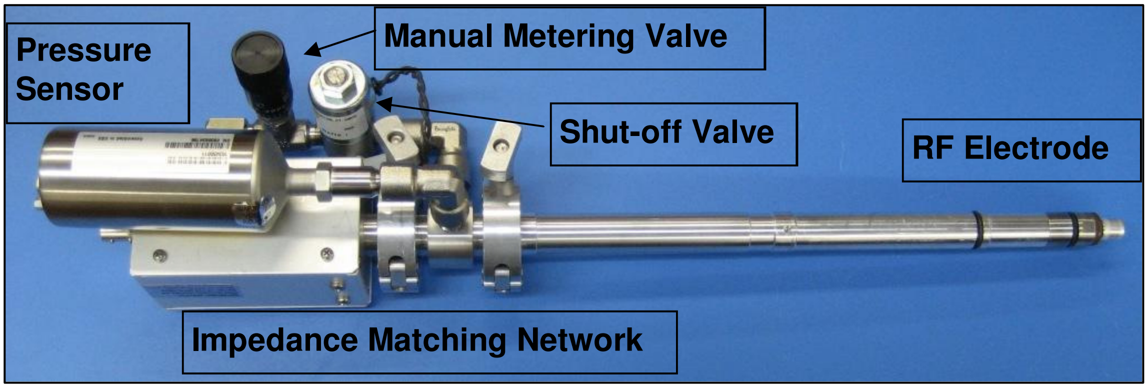
ABSTRACT

The Evactron® De-Contaminator (D-C), introduced in 1999, has been shown to be effective at removing hydrocarbon contamination from scanning electron microscopes (SEMs) [1]. Contamination results in poorer image quality, especially at high magnification and low beam voltage. The Evactron D-C works by placing an electrode in a small vacuum chamber and mounting this unit to the SEM chamber. Radio frequency (RF) plasma is generated at the electrode; room air or other oxygen containing gas is introduced into the SEM chamber and travels past the electrode, creating plasma and oxygen radicals. These radicals then travel through the SEM chamber and remove the hydrocarbons by chemical etch. The Evactron D-C operates between 0.05-1.0 Torr; this pressure range can be attained for limited periods by almost all SEMs. With minor changes to the vacuum system, longer clean times can be achieved. The Evactron D-C is effective because the open geometry inside the SEM chamber allows the radicals to be carried by convection from the plasma throughout the chamber. Significant improvement in image quality has been seen with cleaning times of 10 minutes.

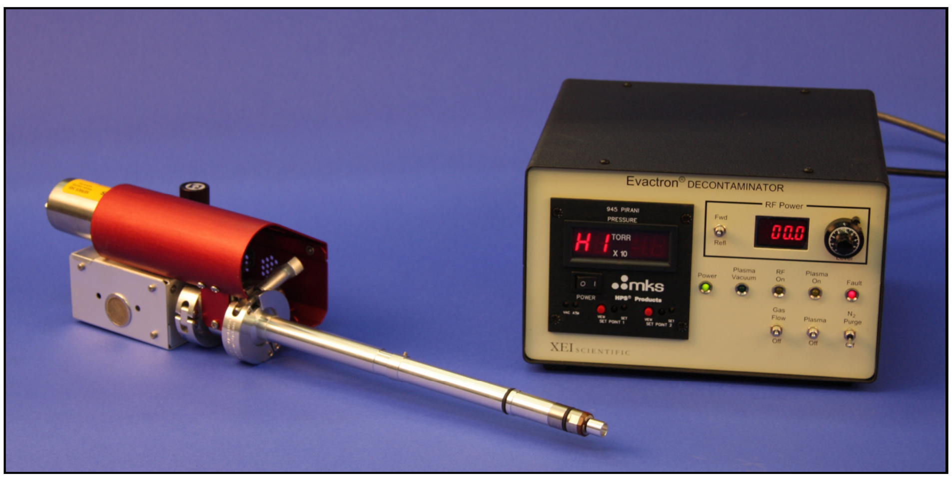
Transmission electron microscopes (TEMs) can also have their image quality affected by hydrocarbon contamination. However, when a conventional Evactron D-C is mounted on a TEM, the oxygen radicals are most likely produced a significant distance from the sample examination area most subject to contamination and requiring the most intensive cleaning. Also, the radicals will have to travel a more tortuous path than they would in the more open SEM chamber. These difficulties require that longer clean times be used, but the vacuum systems on TEMs often limit the ability to achieve this.

Horiuchi *et al.* reported a method to use an Evactron D-C in order to clean a TEM [2]. The authors mounted the Evactron D-C to a port on a TEM near sample examination region and an auxiliary pump to another port on the opposite the side of the TEM column. After 20 cycles of 3 minutes remote plasma cleaning and 3 minutes of nitrogen purge post cleaning, the authors were able to show that clearer TEM images could be obtained.

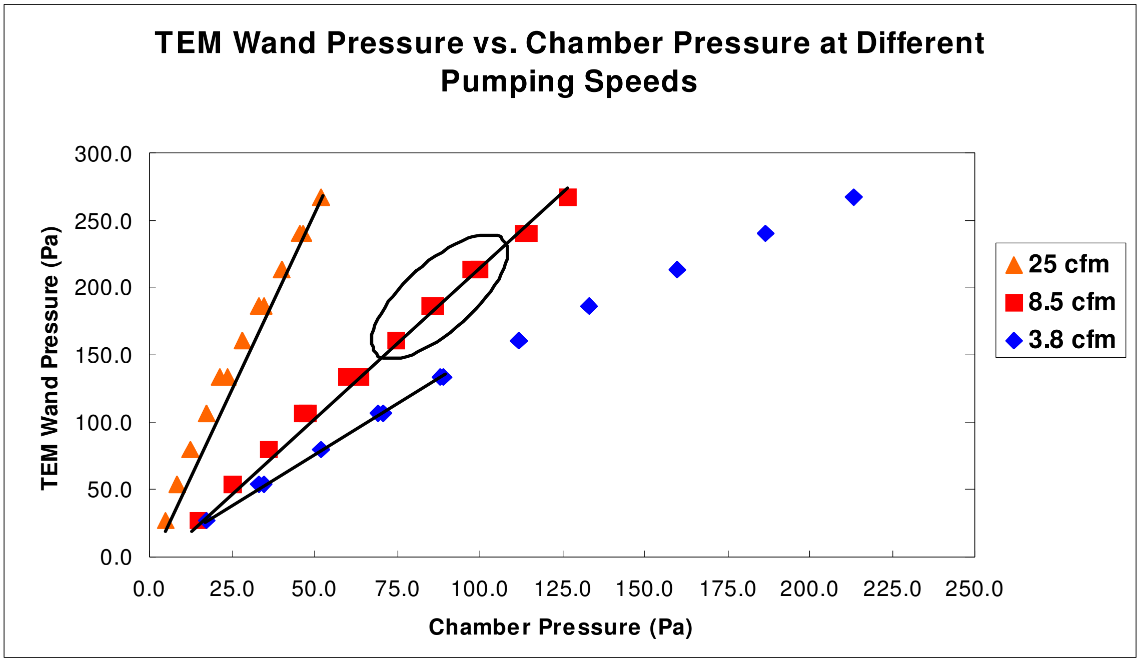
A new method for using the Evactron D-C to clean TEMs is reported here. The RF electrode used to create the oxygen radicals is now mounted on the end of a TEM sample rod. The impedance matching network and gas delivery hardware are placed on the other end of the sample rod, which is hollow to allow oxygen containing gas to reach the electrode.



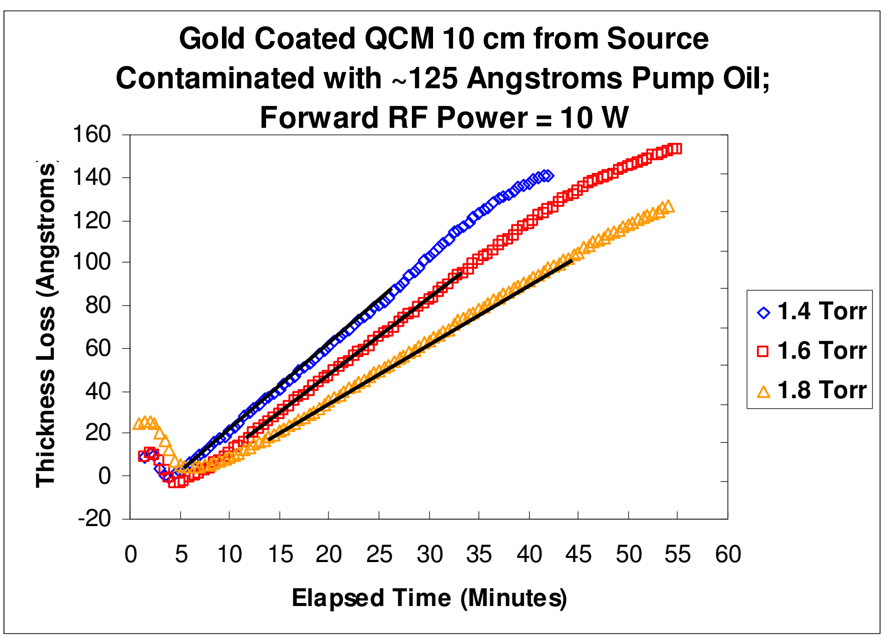
The picture above is of the Oxygen Radical Source (ORS) on a TEM Holder. The RF electrode is on the right hand side near where a TEM sample would normally be. The manual metering valve, shut-off valve, pressure sensor, and impedance matching network are on the left hand side of the picture and are outside the TEM chamber during operation of the ORS.

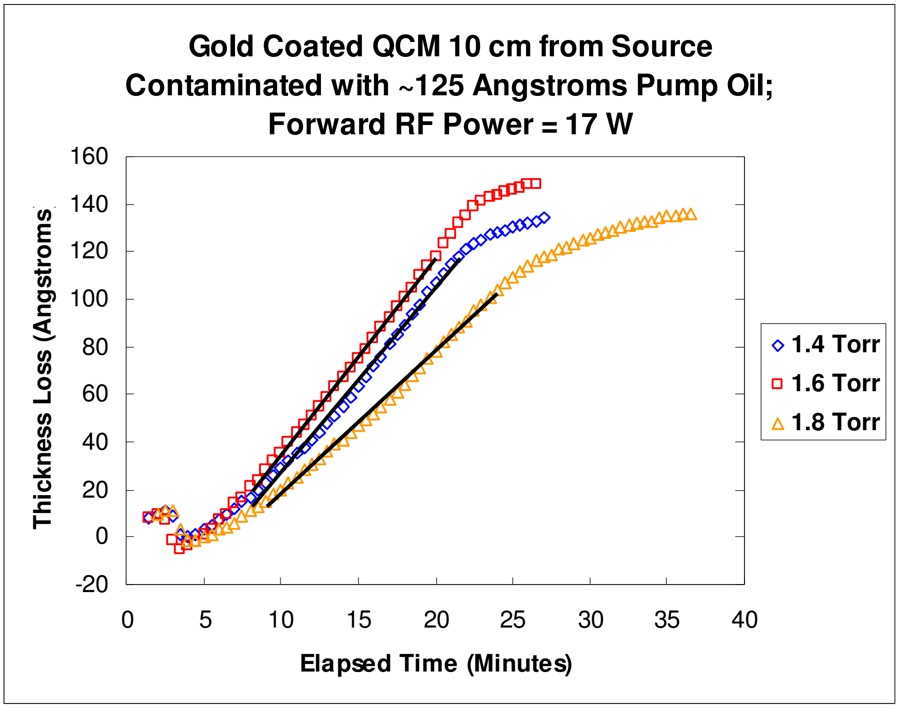


The picture above shows the remote plasma cleaner system using the TEM Holder. A shroud surrounding the valve assembly allows the user to manipulate the ORS on a TEM Holder more easily and provides a more aesthetic package.



The Graph above shows the pressure difference between the pressure sensor on the ORS on a TEM Holder and the pressure at the electrode inside a vacuum chamber being pumped down at various pumping speeds. **The lines** are linear fits to the data. Based on these fits and the different pumping speeds, the **conductance through the ORS on a TEM Holder is estimated to be 3.4±0.4 L s^{-1}**. **The oval** shows the pressure region in which the cleaning rate and oxygen concentration experiments were done.
(133 Pa = 1 Torr)



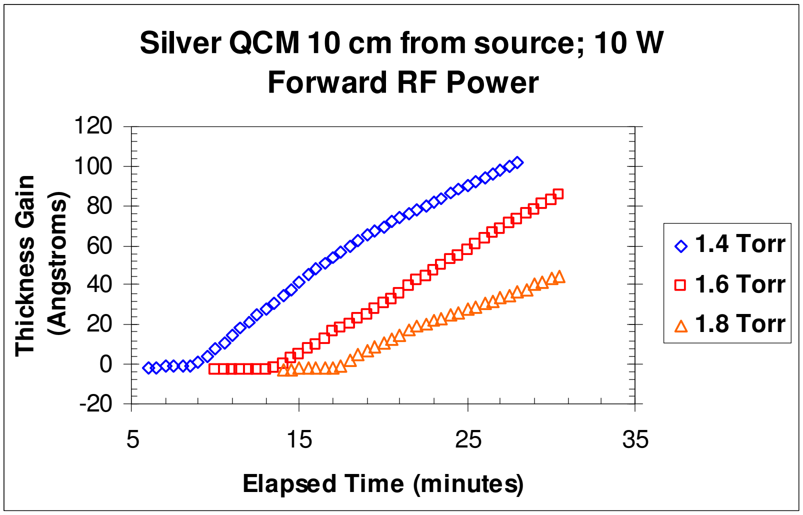


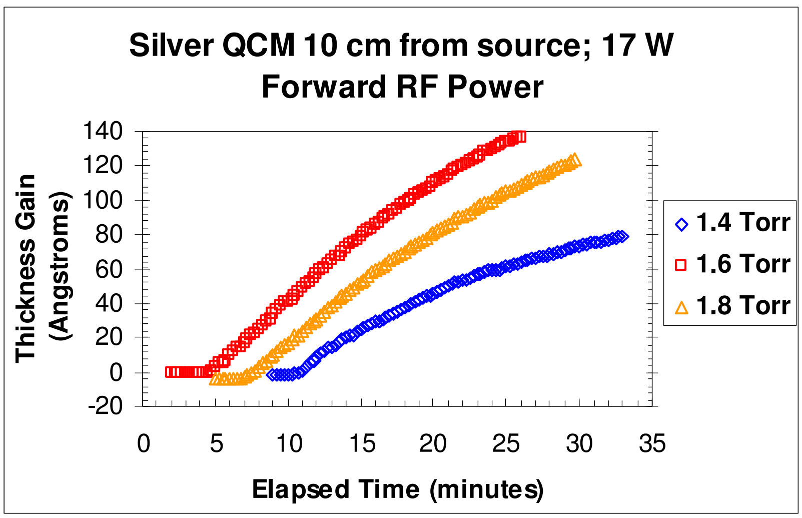
The previous two graphs show removal of hydrocarbon contamination by the **TEM Holder Evactron D-C with room air as the oxygen containing gas**. The cleaning rates were determined using a gold-coated Quartz Crystal Microbalance (QCM) using a method previously described [3]. A small amount of mechanical pump oil (Duniway Stockroom, Part# MPO-190-1) was placed into an 11 cm long vacuum tube with a leak valve attached to one end of the tube and a vacuum chamber on the other. Once the chamber was pumped down, the leak valve was adjusted so that the pressure in the chamber was roughly 0.4 Torr. The tube was then heated, and the deposition of hydrocarbons onto the QCM, typically around 12.5 nm, was monitored using a QCM monitor (McVac - MCM 160). The contaminated QCM was then placed in a separate chamber ~10 cm from the electrode of the ORS on a TEM Holder. Measurements were done at two power and three TEM Holder pressure settings.

Table: Cleaning Rates for Evactron D-C on a TEM Holder (QCM is 10 cm from source)

Press. (Torr)	Rate (Å min^{-1}) (10 W)	Rate (Å min^{-1}) (17 W)
1.4	4.0	7.7
1.6	3.6	8.1
1.8	3.2	6.1

The Table above shows the results. They generally follow the previously observations that increasing RF power and decreasing pressure lead to larger cleaning rates [3]. However, it should be noted that at 17 W the cleaning rate at 1.4 Torr (TEM Holder Pressure) is slightly less than the cleaning rate at 1.6 Torr.





The graphs above show the results of tests with silver-coated QCMs using room air and performed at the three pressure and two power settings used in the gold-coated contaminated QCM tests. The thickness changes on the silver-coated QCMs are due to exposure to oxygen atoms and can be predicted by the Deal-Grove Model [4]. The Model can be used to determine the oxygen atom concentration at the QCM (10 cm from source). The table below shows the results of the tests using the silver-coated QCMs.

Oxygen Radical Concentration for Evactron D-C on a TEM Holder
(QCM is 10 cm from source)

Press. (Torr)	Concentration (10^9 cm^{-3}) (10 W)	Concentration (10^9 cm^{-3}) (17W)
1.4	4.5	5.2
1.6	3.3	5.0
1.8	2.3	4.1

CONCLUSION

A new means of removing hydrocarbon contamination from TEMs by using the Evactron D-C configured in a TEM Sample Holder has been demonstrated. It has been shown that the Evactron D-C on a TEM Sample Holder can produce oxygen atom radicals and remove hydrocarbons.

REFERENCES

[1] A. Vladár, M. Postek and R. Vane, Proc. SPIE 4344 (2001) 835; N. Sullivan, T. Mai, S. Bowdoin and R. Vane, Micros. Microanal. 8 (Suppl 2) (2002) 720CD; P. Rolland, V. Carlino and R. Vane, Micros. Microanal. 10 (Suppl 2) (2004) 964CD; R. Vane and V. Carlino, Micros. Microanal. 11 (Suppl 2) (2005) 900

[2] S. Horiuchi, T. Hanada, M. Ebisawa, Y. Matsuda, M. Kobayashi and A. Takahara, ACS Nano, 2009, 3(5), 1297-1304

[3] M. M. Gleason, C. G. Morgan, and R. Vane, Micros. Microanal. (Suppl 2) (2007) 1734CD; C. G. Morgan, M. M. Gleason and R. Vane, Micros. Microanal. 13 (Suppl 2) (2007) 1736CD

[4] A.K. Srivastava and P. Sakthivel, *J. Vac. Sci. Technol. A* 19 (1) 2001 97; C. G. Morgan and R. Vane, Micros. Microanal. 15 (Suppl 2) (2009) 814CD