

The Effect of Collimators on Evactron Cleaning of EDS Windows

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Introduction

Electron microscopes need cleaning to remove hydrocarbon (HC) contamination. The Evactron De-Contaminator (D-C) removes HC contamination by using oxygen radicals generated by plasma produced by a radio frequency (RF) discharge in the Evactron D-C's Oxygen Radical Source (ORS). Previous studies have shown that Evactron cleaning of UTW windows increases carbon sensitivity [1] and that there may be sight corrosion of the Aluminum film coating of these windows [2]. In many systems, there is an aluminum alloy collimator with a 15 mm base that fits over the window. Above the window is a cone with an entrance port up to 4 mm in diameter for the X-rays to pass through in order to be detected. The oxygen radicals are mainly carried by flowing gas in a viscous flow regime to the surfaces to be cleaned. The window is situated behind an orifice in a non-flow region; the surface HC should be oxidized less by the Evactron cleaning process simply because the oxygen atoms will have a more difficult time reaching the window. The goal of this work is to quantify the effect of the Evactron process on surfaces guarded by the collimator.

XEI Scientific, Inc. has developed a quantitative technique to determine the efficiency of the Evactron process under various conditions. This technique uses a thickness monitor to measure the loss rate of an oil layer which had previously been allowed to accumulate on the monitor. More detail on this technique is presented in another paper [3]. In order to measure the effect of the collimator on the oil thickness loss rate due to the Evactron process, the collimator is placed over a thickness meter prior to pump down. The rate of thickness loss with the collimator covering the monitor is compared to the loss rate when the monitor is completely exposed to the oxygen radicals produced by the Evactron process.

Experimental

Effectiveness of the Evactron® cleaning process was determined by using two McVac quartz crystal thin film thickness meters (QCTMs) placed in a mechanically pumped vacuum chamber. The QCTMs are 8 mm in diameter. The outputs from the QCTMs are read using a McVac MDM-160 thickness monitor. A thin film is introduced onto the QCTMs as described in another paper[3]. The QCTMs were then moved to the center of the chamber approximately 14 cm from the Evactron® ORS. The collimator was placed over one of the TMs; the other TM was left uncovered as a reference. A piece of Teflon tape was placed around the base of the collimator so that the only opening between the TM and the rest of the system was the opening at the top of the collimator.

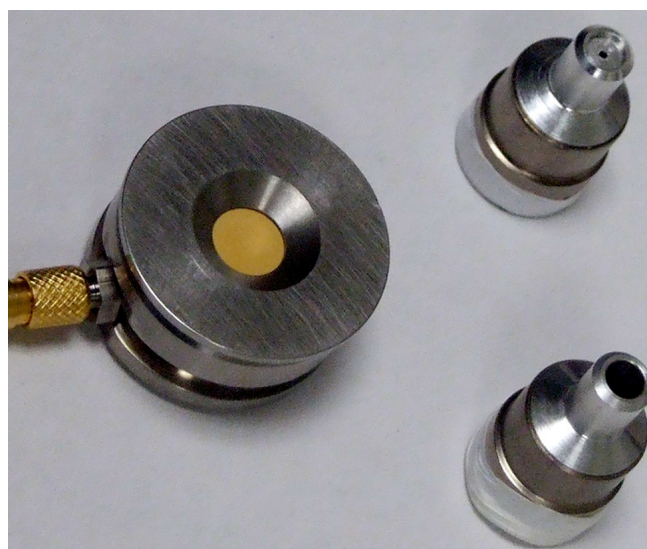
The collimator was provided by Oxford Instruments. It is in the shape of a tapered tube, with a large opening (1.5 cm) on the bottom and a small opening (0.4 cm) on the top. It is 2.5 cm in height. An insert was fabricated to reduce the diameter of the top opening. When the insert is in place, the diameter of the collimator is reduced from 4 mm to 1 mm.

In all cases, room air was used as the oxygen gas source. The air was leaked into the ORS by means of a metering valve, and the pressure in the ORS was monitored with a Pirani gauge. A LabView™ program was used to monitor the change in pump oil thickness on each of the TMs simultaneously.

The quartz crystal thin film thickness meter and the collimators are shown in [Figure 1](#).

Figure 1:

Quartz crystal thickness meter (left) and Oxford Instruments collimator with (top right) and without insert (bottom right). Teflon tape has been placed around the base of the collimators.

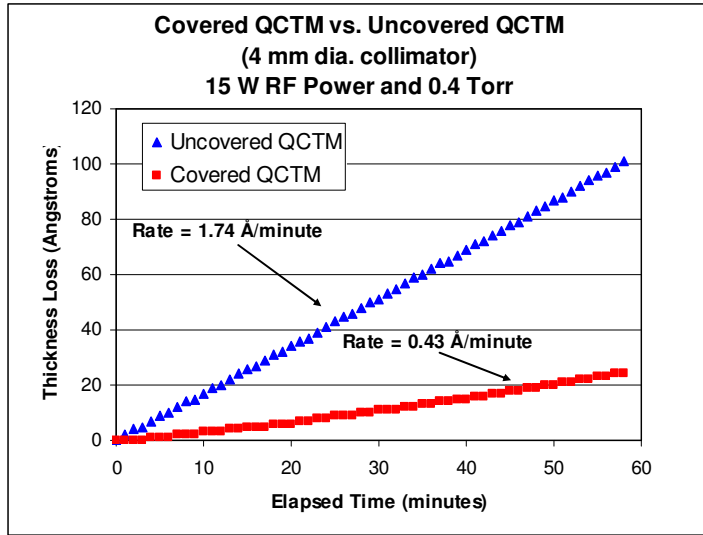


Results

First Set of Experiments

The first set of experiments was done without the 1 mm insert; the diameter of the collimator is 4 mm. The results of this experiment are shown in [Figure 2](#). In this graph, thickness loss (in Angstroms) is shown as a positive number. When the Evactron process is turned off, almost no thickness loss is seen on either QCTM. The rate for the covered QCTM is about 4x less than the uncovered QCTM, which is the difference in area between the size of the quartz crystal QCTM and the size of the collimator opening. Oxygen radicals are able to diffuse through the collimator and remove contamination from the TM with minimal loss on the walls of the collimator. The collimator is made from aluminum, and the native aluminum oxide surface will not remove oxygen radicals very efficiently. The rate of thickness loss is determined by the geometry of the experiment.

Figure 2:



For air, the Knudsen number (Kn) is

$$Kn = \frac{1}{(20.2 \times P \times d)}$$

where P is the chamber pressure in Torr and d is the collimator diameter [4]. The Knudsen number is the ratio of the mean free path over the diameter of the opening. As this number increases, the likelihood of oxygen radicals getting past the collimator restriction to the covered QCTM should decrease. It should be kept in mind that lowering the pressure also increases the number of oxygen radicals reaching the collimator.

The first experiment was repeated at a lower pressure to see what effect a change in the Knudsen number will have on the ratio between the uncovered and covered thickness loss rates. The results are shown in **Table I**.

Table I: Pressure Study with 4mm Collimator at 15 W RF Power

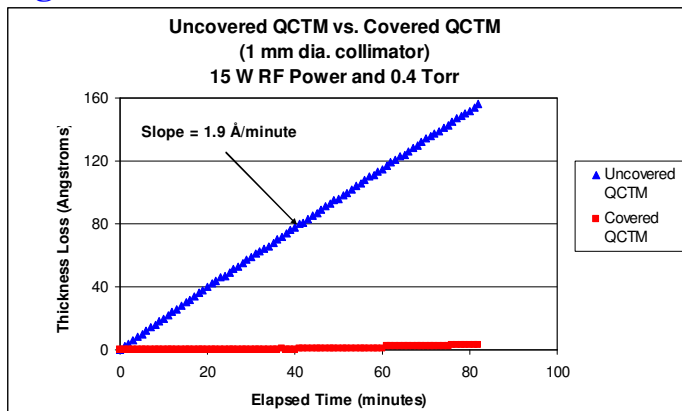
Pressure (Torr)	Knudsen Number	Uncovered Rate (Å/minute)	Covered Rate (Å/minute)	Ratio (Uncovered Rate/Covered Rate)
0.4	0.03	1.74	0.43	4.1
0.4	0.03	1.79	0.46	3.9
0.2	0.06	4.15	0.51	8.1
0.2	0.06	4.12	0.52	7.9

The ratio between the uncovered and covered rates doubles when the chamber pressure is halved, suggesting that the Knudsen number can be used to ascertain the shielding effect of the collimator.

Second Experiment

The second experiment is a repeat of the first one, except that the 1 mm insert is placed in the collimator. The results of this experiment are shown in **Figure 3**. This time, however, there is a radical difference between thickness loss rates. For the uncovered QCTM, the thickness loss rate was measured to be $1.9 \text{ \AA}/\text{minute}$. Using the geometric arguments from the results of the first experiments, one would expect the loss rate of the covered QCTM to be around $0.2 \text{ \AA}/\text{minute}$ and the total thickness loss after 80 minutes to be around 16 Angstroms. However, the rate of thickness loss for the covered QCTM and the 1 mm insert was actually around $0.04 \text{ \AA}/\text{minute}$. There is a change in the Knudsen number of the oxygen radicals entering the top of the collimator. When the collimator opening is reduced from 4 mm to 1 mm, the Knudsen number increases 0.12, further into the transition region between viscous and molecular flow [4]. It may be more difficult for the oxygen radicals to move through the top of the collimator if they are in a flow regime that is more molecular than viscous.

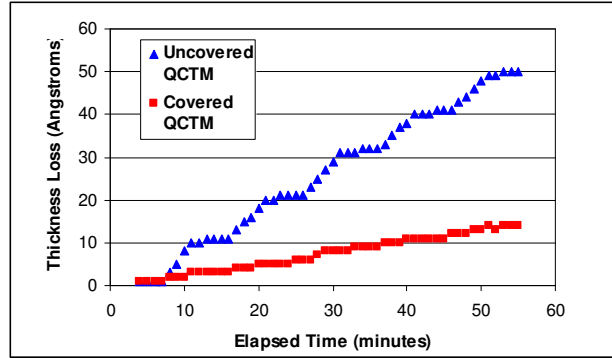
Figure 3



Third Set of Experiments

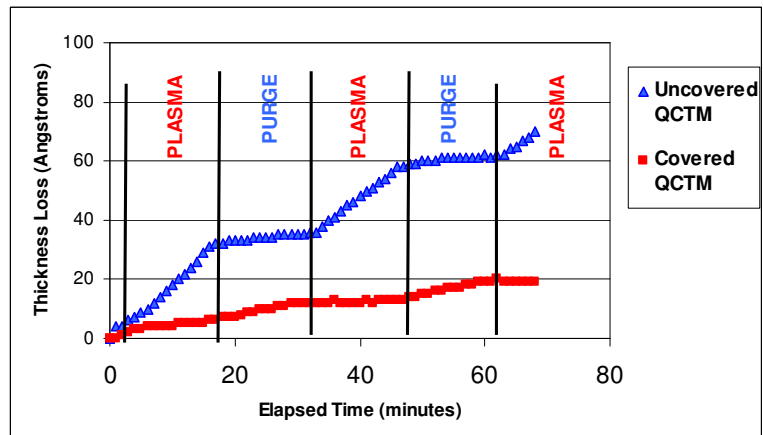
It has been noted that there is a time delay between when the initial Evactron D-C cleaning starts on the uncovered TM and when this cleaning starts on the covered TM. It was thought that if a purge step was introduced into the cleaning process, it would remove oxygen radicals before they could reach the covered TM. The result would be that there would be a greater shielding effect on surfaces protected by a collimator, while still allowing Evactron D-C cleaning on unprotected surfaces. Hoping to exploit this effect, the next experiment cycled the Evactron D-C cleaning process between running the oxygen radical producing plasma and then turning off the plasma in order to purge the chamber. In the example shown in **Figure 4**, the collimator with the 4 mm top opening was used. Room air was used for the purge step. The uncovered TM had a thickness loss rate about 4x greater than the covered TM; this ratio is the same as the first experiment, which was done under the same experimental conditions but without the plasma/purge cycling. The results do not show the hoped for increase in the shielding effect, which would be seen as a greater difference between the loss rates of the uncovered and covered TMs. This experiment was repeated with differences in the purge pressure used and with difference in the plasma and purge timing, but no increase in the shielding effect was seen.

Figure 4: Effect of cycling plasma and purge steps on covered and uncovered QCTMs. The collimator with the 4 mm opening was used. The plasma steps ran at 0.4 Torr and 15 W RF power for 5 minutes. The purge steps ran at 1 Torr for 5 minutes using room air. The plasma and purge steps were cycled 5 times.



An intriguing effect was found in one repetition of the above experiment; the results are shown in **Figure 5**. The decrease in the thickness layer for the covered QCTM occurs during the purge step and not during the plasma step. This result suggests that there may be procedures which would remove contamination from areas which are partially covered (such as an EDS window behind a collimator) without exposing those areas to direct Evactron D-C cleaning. By judiciously using plasma and purge cycling, it may be possible to clean parts of a vacuum chamber that are 1) difficult for the oxygen radicals to reach and 2) contain equipment which is sensitive to oxygen radicals. More work will be done to study this effect.

Figure 5: Effect of cycling plasma and purge steps on covered and uncovered QCTMs. Time periods during which each step was run are marked in the figure. The collimator with the 4 mm opening was used. The plasma and purge steps ran at 0.4 Torr for 15 minutes. The plasma step was run at 15 W RF power.



Conclusions

The results suggest that an increase in the Knudsen number will decrease the ability of the oxygen radicals to get through the collimator. The Knudsen number can be increased by lowering the pressure and making the collimator diameter smaller. It has been seen in our laboratory that lower pressure increases cleaning efficiency, so lowering the working pressure of the Evactron® De-Contaminator should also be beneficial to the entire process. Although the plasma/purge cycling did not have the hoped-for shielding effect, future work may point to a pressure/timing regime which would allow cycling to have the desired effect of keeping oxygen radicals away from sensitive detectors, while still cleaning the entire chamber.

References

- [1] P. Rolland, V. Carlino, and R. Vane, Proc. Microsc. Microanal. 10 (Suppl 2) (2004) 964 CD
- [2] R. Vane, C. Roberts, and V Carlino, Microsc. Microanal. 10 (Suppl 2) (2004) 966CD
- [3] C. G. Morgan, M. M. Gleason, and R. Vane, Microsc. Microanal. (2007) *submitted*.
- [4] A User's Guide to Vacuum Technology, 2nd Ed., John O'Hanlon, John Wiley & Sons, 1989.