Using Thickness Monitor to Measure Contaminant Removal by Evactron Cleaning as a Function of Operating Parameters

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Introduction

Since its introduction in 1999, The Evactron® De-Contaminator has demonstrated its effectiveness in removing hydrocarbon contaminants from Electron Microscopes [1]. This is done by using Radio Frequency (RF) generated plasma to produce oxygen radicals which ash contaminants. The Oxygen Radical Source (ORS) is attached to the Electron Microscope chamber, and a controlled leak of oxygen containing gas such as room air is passed through the plasma in order to produce oxygen radicals. It has been difficult to quantify the effectiveness of the decontamination process. Previous methods have monitored contaminated chambers before and after the Evactron process to demonstrate qualitatively that cleaning has occurred [2]. What is needed is a way to introduce contamination in a repeatable fashion into the chamber, and then monitor its removal quantitatively by the Evactron process.

Quartz crystal thickness monitors (QCTMs) are a standard tool for vacuum deposition measurements. They can also be adapted to measure contamination removal by plasma cleaning, as described in a companion paper [3]. Here, they are used to record a thickness loss rate of an oil layer previously deposited on their surface; this loss rate is a measure of the cleaning effectiveness of the Evactron D-C. This method provides a simple and inexpensive way to measure decontamination by the Evactron process, and it allows a determination of the how the effectiveness of the process changes as the operating parameters are allowed to vary.

Results

First Set of Experiments

In the first set of experiments, pump oil was loaded onto the thickness monitor, and the Evactron process was ignited with room air. Refer to the companion paper [3] for details on the experimental methods used; the experimental conditions match that those described in the section “Typical Experiment”. The amount of air leaked into the chamber and the plasma was varied from 0.2 to 0.7 Torr, and the RF power was varied between 10, 14 and 17 W, so that loss rate on the thickness monitor could be measured as a function of different chamber pressures and RF power settings. The results for the QCTM in the center of the chamber (QCTM1 in Figure 2 of ref. [3]) are shown in **Figure 1**. As the pressure increases, the rate of thickness loss decreases. This decrease is due to the higher rate of three-body reactions in the plasma and chamber. These reactions destroy the oxygen radicals which decontaminate the chamber.

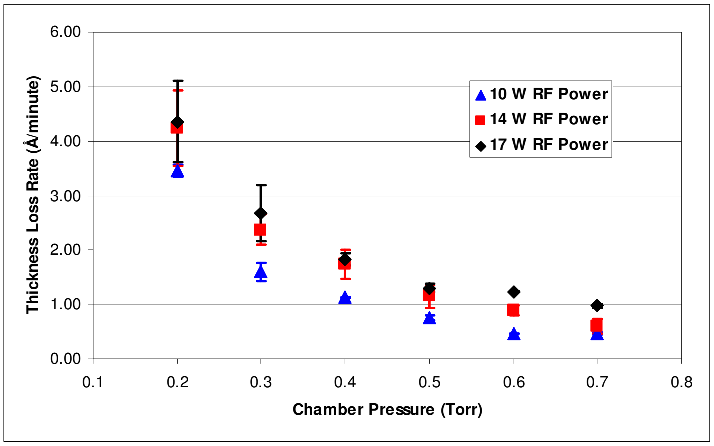

Figure 1: Variation in thickness loss rates as a function of chamber pressure. Data was taken from the QCTM in the center of the chamber. Blue triangles (▲) show thickness loss rates when the Evactron D-C is at 10 W RF power. Red squares (■) show thickness loss rates at 14 W RF power, and black diamonds (♦) show thickness loss rates at 17 W RF power.

It was expected that at very low pressures the efficiency will drop again, due to the lack of oxygen radicals being produced, but this turning point was not reached by our experiments. This may be due to the fact that either the three body recombination rate at low pressures is so small that relatively large numbers of oxygen radicals make it into the chamber. Alternatively, other processes related to the plasma, such as ions or highly energized species, can reach further into the chamber and affect the QCTM thickness loss rate.

As expected for the power variation, an increase in power generally leads to an increase in decontamination efficiency. The increase in decontamination efficiency between 14 and 17 W RF power is not as great as the corresponding increase between 10 and 14 W. Although more oxygen radicals are produced when the RF power is increased, the number of N_2^+ ions in the plasma also increases. These ions destroy oxygen radicals, and decrease the total number of oxygen radicals produced.

Second Set of Experiments

A second set of experiments compared the cleaning efficiency between air and various other gas mixtures. The first gas mixture tested was a mixture of dry O_2 and N_2 (30% O_2, balance N_2). For this experiment, a single QCTM was used and was placed in the middle of the chamber. It was found that the dry O_2/N_2 mixture does not produce as good a cleaning efficiency as room air, as seen in **Figure 2**. This observation suggests that water, which is a constituent of room air and is dissociated by plasma into hydroxyl radicals (OH) may play a role in the remote plasma cleaning process.

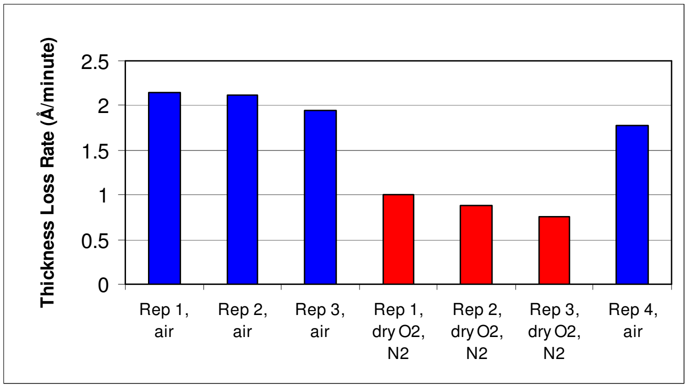

Figure 2: **Comparison in thickness loss rate recorded using room air and a mixture dry O_2 and N_2. A single QCTM placed in the middle of the chamber was used for all of these experiments, which were run with the chamber pressure at 0.4 Torr and the RF power at 14 W. Three experiments measuring the thickness loss rate for air were performed, followed by three experiments with the dry O_2/N_2 mixture and then a final experiment with air.**

This experiment was repeated with a mixture of dry O_2 and Argon (40% O_2, balance Ar). ***No thickness loss at all was observed for the oxygen/argon mixture.***

This experiment was also repeated using dry, industrial grade O_2. A comparison of the initial activity of Evactron cleaning was made between O_2 and air. In these experiments, a fresh layer of pump oil was deposited before each cleaning run.

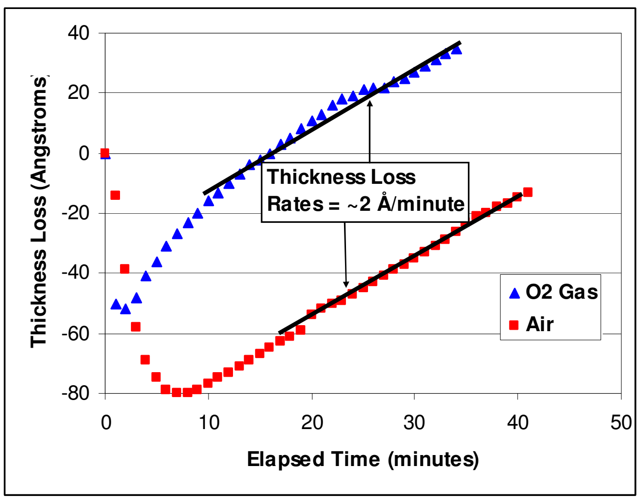

Figure 3: **A comparison of the initial activity of Evactron cleaning between using either industrial oxygen, denoted as blue triangles (▲), or room air, denoted as red squares (■), as the radical source. A fresh layer of pump oil was deposited before each run. These cleaning runs were done at 14 W RF power and a chamber pressure of 0.5 Torr.**

As can be seen in **Figure 3**, both O_2 and air finish their respective runs with a thickness loss rate of ~2 Å/minute. However, there are differences in the initial ***induction period***, during which time the oxygen radicals are incorporated into the oil layer, causing an increase in the thickness of the oil layer, and then only later are volatile compounds produced from the oil layer, causing it to decrease. The induction period is much shorter for the cleaning run with the industrial O_2 used versus the room air. This observation suggests that products (such as NO), made from O_2/N_2 plasma, incorporate themselves into the oil layer and are more difficult to remove by subsequent oxygen radical attack.

Third set of experiments

A third set of experiments changed the distance between the ORS and the thickness meter. Companion data to the data shown in **Figure 1** was collected from the QCTM on the side of the chamber (QCTM2 in Figure 2 of ref. [3]) and is shown in **Figure 4**. The cleaning rates are much less on the side of the chamber than in the middle. The oxygen radicals travel through the chamber from the ORS to the pump port; there is a longer path for the radicals to take to the side of the chamber versus the center, and this longer path increases the likelihood that they will be consumed in three body reactions. Also, the difference in cleaning efficiency is greater between 14 and 17 W RF power than it is between 10 and 14 W RF power. This difference suggests that more RF power causes oxygen radicals to be created either with more velocity or with a greater angular spread as they leave the ORS port.

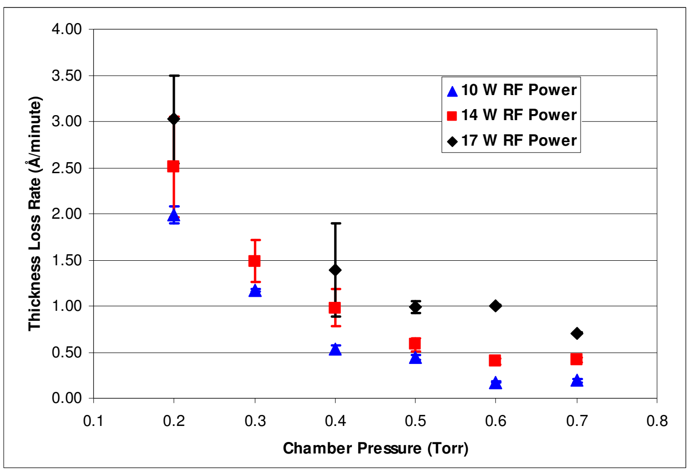

Figure 4: Variation in thickness loss rates as a function of chamber pressure. Data was taken from the QCTM on the side of the chamber. Blue triangles (▲) show thickness loss rates when the Evactron D-C is at 10 W RF power. Red squares (■) show thickness loss rates at 14 W RF power, and black diamonds (♦) show thickness loss rates at 17 W RF power.

Also, a study was done to see what effect the distance between the ORS and the thickness meter has on the cleaning efficiency; the results are shown in **Figure 5**. These experiments were done with flaxseed oil loaded onto the thickness monitor, with the RF power at 14 W, and with the chamber pressure at different levels. A single QCTM was placed on an imaginary line between the ORS port and the vacuum port. The efficiency of the Evactron process increased, and the pressure dependence was greater the closer the thickness meter was to the ORS. These changes can also be explained by the number of oxygen radical destroying three-body reactions occurring. The higher the chamber pressure and the longer the oxygen radicals travel before they react on the thickness monitor, the more likely they will be destroyed.

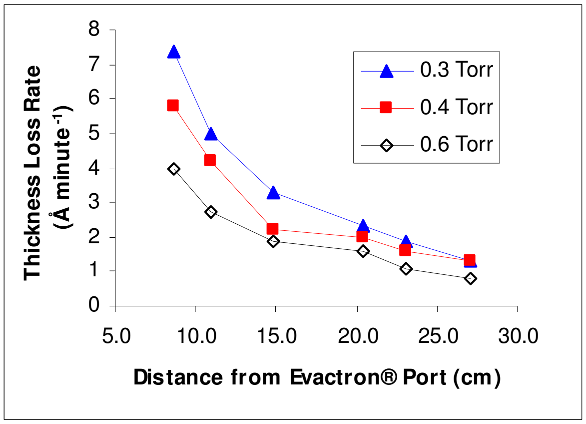

Figure 5: **Variation in thickness loss rates as a function of distance between ORS and vacuum port. A single QCTM was placed on an imaginary line between the ORS and the vacuum port. The RF power was set at 14 W. Blue triangles (▲) show thickness loss rates when the chamber pressure was at 0.3 Torr. Red squares (■) show thickness loss rates when the chamber pressure was at 0.4 Torr, and open black diamonds (◊) show thickness loss rates when the chamber pressure was at 0.6 Torr.**

Conclusions

The cleaning efficiency of the Evactron process was quantified by using quartz crystal thin film thickness meters. Chamber pressure, RF power, gas type used, and distance between the ORS and the thickness meter were the parameters used in this study.

Lower pressure and higher power leads to greater cleaning efficiency. This efficiency is less the further away from the ORS the area to be cleaning is located. The results indicate that three body collisions play an important role in determining the cleaning efficiency of the oxygen radicals, although there might be chemistry due to species created directly in the plasma, such as high energy radicals or ions, at low pressure.

Dry O_2/N_2 mixtures do not provide good cleaning efficiency. Dry O_2/Argon mixtures do not provide any cleaning. From our experiments, industrial grade O_2 provides as good cleaning efficiency as air, but the induction period, the amount of time needed to start removing hydrocarbons from the surface, is much shorter than air. Therefore, industrial grade O_2 gas is superior to air for cleaning chambers.

References

[1] http://www.evactron.com/index.html
[2] R. Vane and V. Carlino, Micros. Microanal. 11 (Suppl 2) (2005) 900
[3] C. G. Morgan, M. M. Gleason, and R. Vane, Micros. Microanal., (2007) *submitted*