Quantification of Contamination Using Quartz Crystal Thickness Monitors

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Introduction

Hydrocarbon (HC) contamination is a persistent problem for users of electron microscopes (EMs), often leading to image distortion and interference with nanoprobing [1-4]. Quantifying the amount of contamination present by easy, low cost methods has been a challenging problem.

The Evactron® De-Contaminator (D-C) has been available for contamination removal in EMs since 1999 [5]. The Evactron D-C uses low power radio frequency (RF) generated plasma in order to produce oxygen radicals which clean the EM. The source of the oxygen radicals is an oxygen containing gas (usually air) which is introduced into the plasma through a controlled leak. The oxygen radicals chemically react with the HCs to form volatile oxidation products such as H_2O, CO and CO_2. These volatile compounds can be pumped out of the EM chamber.

The efficiency of the Evactron process can depend on several parameters; the pressure measured in the plasma, the power of the RF generator, the location in the chamber of the cleaning, and whether there are any obstacles between the plasma and the area to be cleaned. As of yet, no studies have been done to determine what effect these parameters have on the efficiency of the Evactron process.

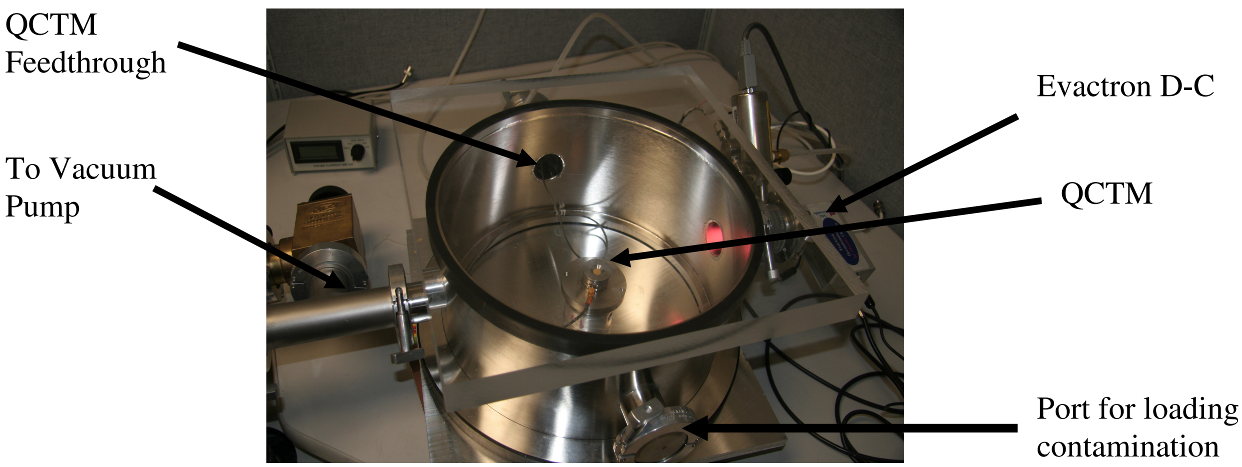
It is very easy to qualitatively demonstrate the efficiency of the Evactron process; a light HC deposit can be removed from a mirrored surface within minutes. However, a quantitative method of determining the efficiency of the Evactron process is needed. Here at XEI Scientific, we have decided that using a quartz thin film thickness monitor (QCTM) is the best method for monitoring the efficiency of the Evactron process. Quartz crystal thin film thickness monitors are a standard technique for measuring the vacuum deposition of thin films and are available from many manufacturers [6].

Experimental

Set-up and Equipment

The experimental set-up is shown in the picture below (**Figure 1**). The size of the vacuum chamber used for the experiments is 30 cm diameter and 15 cm in height. It has four KF40 ports equidistant from each other. One of these ports is reserved for the vacuum pump; either an oil pump or a scroll pump was used in this system. The port opposite the vacuum line is reserved for the Evactron D-C Oxygen Radical Source (ORS). The third port has the electronic feedthroughs for the QCTMs, and the last port is used for introducing contamination into the chamber.

Figure 1: Picture of vacuum chamber used for QCTM studies with Evactron D-C

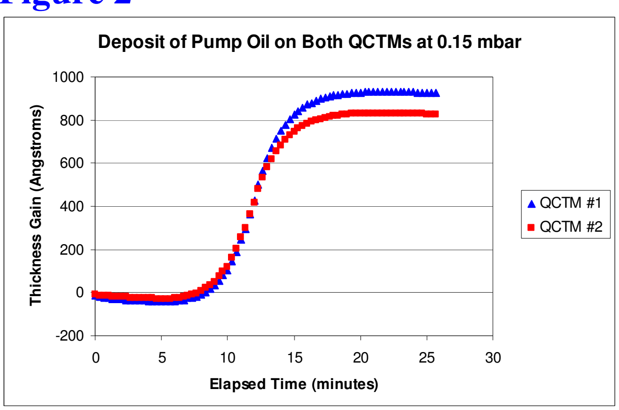


The signals from the QCTMs are monitored by a thickness monitor (McVac - MCM 160). Two QCTMs can be monitored with this device, and their output can be sent via an RS232 cable to a PC, where a LabVIEW™ program can record the output as a function of time. A separate vacuum gauge can be used to monitor the pressure inside the chamber.

Depositing a Contamination Layer onto the QCTM

The following procedure is used to deposit contamination onto the QCTMs. Between 5-7 drops of liquid contamination (typically pump oil supplied by Duniway Stockroom Corp., part # MPO-190-1, but flaxseed oil is also used) are placed inside of an 11 cm long vacuum tube. One end of the tube is attached to the chamber, and a leak valve is attached to the other end of the tube. The QCTMs are placed close to the port with the vacuum tube attached. The chamber is pumped down, and its pressure is adjusted to ~0.15 mbar using the leak valve. The vacuum tube is heated until a layer of oil ~0.05 um is deposited on the QCTMs. A typical trace of thickness gain as a function of time on the QCTMs is shown in **Figure 2**. The tube is then allowed to cool, and the chamber is opened. Excess oil in the vacuum tube, on the walls of the chamber, and on the mounts for the QCTMs is removed using isopropyl alcohol wipes.

Figure 2

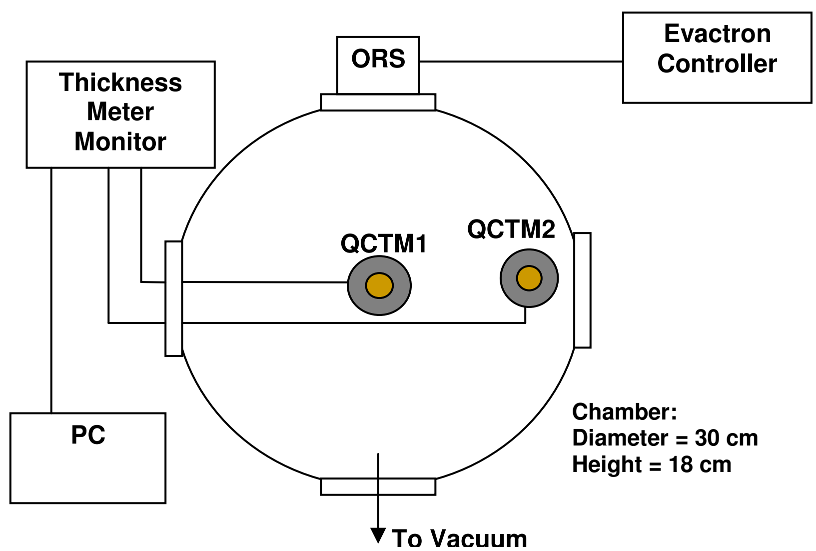


It should be pointed out that this procedure deposits far more contamination in the chamber than would ever be expected in normal EM operating conditions. The goal of this procedure is not to recreate EM contamination. Instead, it is to insure that enough oil is deposited on the QCTMs so that a series of experiments can be performed with them.

Typical Experiment

A typical experiment with the Evactron D-C and two QCTMs is run in the following way. One QCTM is placed in the center of the chamber and the other QCTM is placed on the side of the chamber by the port used for loading contamination.

Figure 3: Schematic of typical experiment to determine cleaning efficiency of Evactron D-C using QCTMs



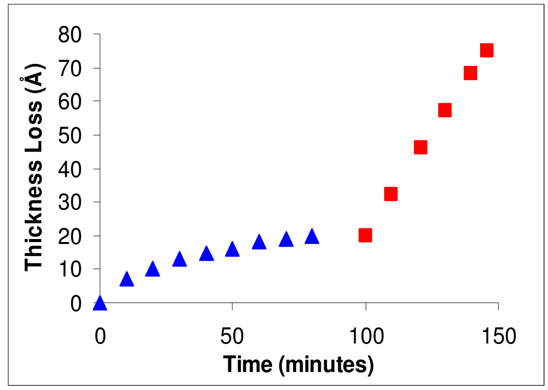
Results

Behavior of the Contamination on the QCTM

When the QCTMs with the oil deposit are placed in the vacuum chamber and allowed to be pumped on, the oil is removed through evaporation. The rate of removal is rapid at first, but then slows down to an almost negligible rate, as seen in **Figure 4**. When the Evactron D-C is turned on, the rate of oil removal increases again.

Figure 4:

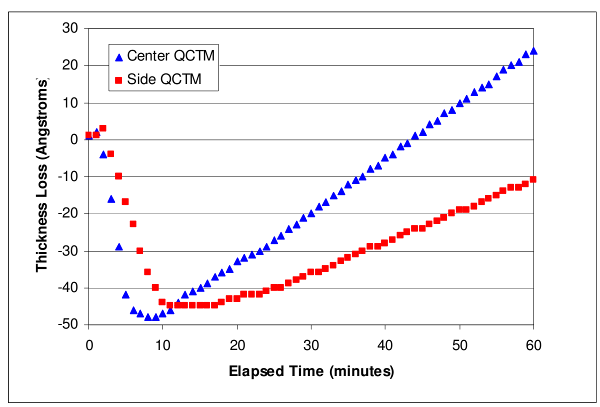
Here is a measurement of oil thickness loss versus time using a single QCTM. Ambient chamber pressure was 0.6 Torr. Blue triangles (▲) show thickness loss before Evactron process started. Red squares (■) show thickness loss after start of Evactron process. Power of RF generator was at 14 W.



If, on the other hand, the Evactron D-C is turned on shortly after a fresh layer of oil has been deposited, there is an initial increase in the thickness, as seen in **Figure 5**. Our interpretation of this behavior is that when the Evactron process has started, the oil in the contamination layer begins to be oxidized by the radicals produced in the plasma. However, there is an ***induction period*** during the initial Evactron D-C activity. During this period the oxygen radicals are incorporated into the oil layers, causing an increase in the thickness of the oil layer. Note also the delay between the minima in the center QCTM signal and the side QCTM signal, due to the longer time needed for the oxygen radicals to reach the side QCTM.

Figure 5:

Initial activity seen during Evactron D-C cleaning immediately after depositing ~0.065 um pump oil onto both QCTMs. The chamber pressure is set to 0.4 Torr and the RF power is set to 14W. Blue triangles (▲) show thickness loss of the center QCTM, while red squares (■) show thickness loss at the side QCTM. Note the *induction period* for both traces, during which the thickness of the contamination layer increases due to incorporation of oxygen radicals into the layer.



Once enough oxygen radicals reach the surface and are incorporated into it, the oil layer can be turned into volatile compounds and removed. This removal, or thickness loss, occurs at a steady rate, as seen after 10-15 minutes elapsed time in **Figure 5**. The thickness loss rate is repeatable under the same Evactron D-C operating conditions.

We have been able to demonstrate that a quartz crystal thickness monitor can be used to measure the rate of decontamination by the Evactron process. The monitor can now provide a measure of Evactron process efficiency as a function of chamber pressure, RF power, and location of surface to be cleaned relative to the Evactron process and the pump. We can also determine the effect on the Evactron process on what gas mixture is used in the plasma and whether there are any obstacles, such as collimators, between the surface to be cleaned and the Evactron D-C.

References

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