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Characterization of a Plasma Cleaner for Electron Microscopy

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Plasma cleaning of electron microscope samples has become important in the preparation of specimens for transmission electron microscopy and scanning electron microscopy. Plasma cleaning a sample prior to electron microscopy can prevent the cross-linking or polymerization of hydrocarbon contaminants on the sample by the electron beam [1]. Such contamination can interfere with the imaging as well as spectroscopic characterization of specimens. Plasma cleaning a sample and holder prior to insertion in the microscope can also prevent hydrocarbon contamination from being introduced into the microscope from the sample or holder.

We can separate hydrocarbon removal into two regimes: bulk cleaning of thick hydrocarbon films, and surface cleaning of the last few monolayers of material. Bulk removal is relatively straightforward to measure with techniques such as electron energy loss spectroscopy, Auger electron spectroscopy, X-ray photoelectron spectroscopy, quartz crystal microbalance, and ellipsometry. Surface cleaning is more difficult to characterize. The contaminant often diffuses across the surface of a sample and is polymerized under the electron beam. In such cases a spectroscopic technique is sampling the contaminant that diffuses into the area of the beam in the time the beam was on as well as the material in that area previously.

We have characterized the Solarus^{TM} Model 950 plasma cleaner from Gatan, Inc. This system uses either Hydrogen/Oxygen or Argon/Oxygen gases controlled by mass flow controllers. The capacitively coupled RF power system automatically matches its impedance to that of the load, insuring consistent power delivered to the plasma. Bulk cleaning rates were measured as a function of position within the specimen chamber, chamber pressure, and hydrogen/oxygen mixture. PMMA resist was spun onto a quartz substrate and baked at 130 C for 10 minutes [2]. PMMA resist is a well characterized photo- and electron-beam-resist material which can be formed into very uniform and stable films. The mass (or thickness) of this film was then measured before and after plasma cleaning. Surface cleaning was measured by exposing the surface to a focused probe in a TEM with a fixed current and spot size. The sample was then tilted and imaged in the TEM and the height of the resulting contamination cone was determined. A sample was considered successfully cleaned if no contamination was visible after exposing an area with a focused probe for 10 minutes.

Cleaning rate increased with sample height and decreased with distance from the center of the specimen chamber (Fig. 1). This is expected since the RF antenna is above the specimen chamber and on-axis. The specimen chamber is cylindrical, and the RF antenna is on-axis and above the specimen chamber. These results indicate that extended objects and multiple samples can be cleaned effectively. Cleaning efficiency as a function of both total chamber pressure and H_2/O_2 ratio was measured. A clear maximum is seen in both curves allowing simple optimization of these parameters. The mass removed during cleaning of PMMA for 1 minute H_2/O_2 plasma was equivalent to a thickness of ~ 5 nm of Carbon.

The results of surface cleaning is exemplified in figure 2. Figure 2a shows a holey carbon film, contaminated with mechanical pump oil and acetone, where a focused probe was held for 1 minute. The dark spot is carbon contamination, and measured ~2.6 μm tall. Figure 2b shows the same area after plasma cleaning for 30 s with H_2/O_2. A focused probe was held on the sample for 10 minutes prior to capturing this micrograph, and no contamination was evident.

References

[1] N. J. Zaluzec, U.S. Patent Number 5,510,624 (1996)
[2] 950 PMMA A11, MicroChem Corp., 1254 Chestnut St., Newton MA 02464

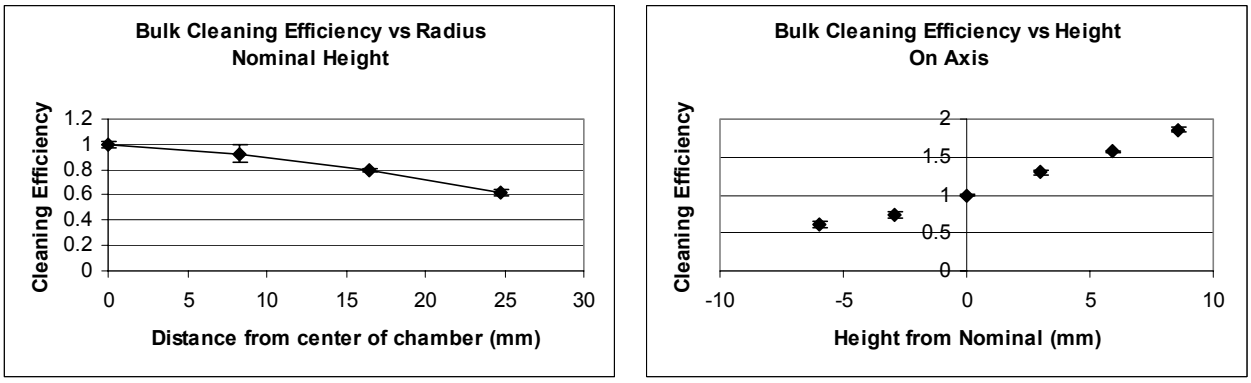

Fig. 1. a) Cleaning efficiency as a function of distance from the specimen chamber axis, normalized to 1 for the nominal height on the axis of the chamber. b) Cleaning efficiency as a function of height.

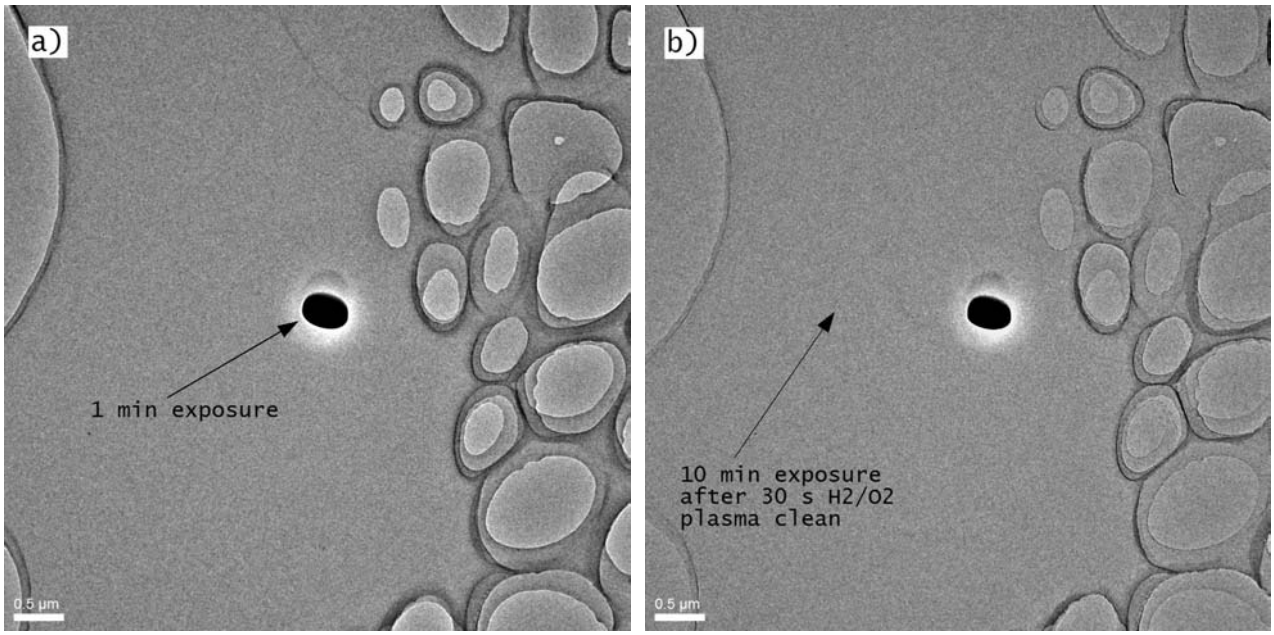

Fig. 2. a) Micrograph of holey carbon film contaminated with pump oil and exposed by focused electron beam for 1 minute. b) Same area after cleaning in Gatan Solarus Model 950 with 30 seconds H_2/O_2 plasma then 10 minute exposure with focused probe. No contamination is evident from the 10 min exposure, verifying a clean surface.

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