A White Paper from XEI

"Application of Plasma Cleaning Technology in Microscopy"

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Introduction

The cleanliness of specimen surfaces and the high vacuum electron microscope environments in which these surfaces are studied or processed have never been more critical than they are today with examination and fabrication nearing the atomic level. Routine manufacturing at the scale required for nanotechnology demands pristine and controlled surfaces in order to create the desired structures. Modern electron and ion microscopes are equipped with sophisticated vacuum systems and can provide these conditions, but maintaining cleanliness over time may be more difficult. One of the ways that scientists have been able to achieve these remarkably unadulterated surfaces has been to subject their samples and microscopes to cleaning by various plasma technologies.

Often contamination is derived from hydrocarbon molecules which even in minute quantities can interact with the electron or ion beam, creating unwanted artifacts in images or data.

While contamination is often considered in terms of examination of non-biological samples, it is useful to point out that exposure of biological and polymeric samples to ion and electron beams can create extensive carbon contamination and the tools used for this work are often in dire need of methods to clean them as well.

Carbon Contamination

The problem of hydrocarbon contamination inside the electron microscope is well documented and has been an issue from the earliest days of electron microscopy. This artifact is the often result of the electron (or ion) beam striking unwanted contaminant molecules and promoting the growth of carbonaceous materials on the surface of the sample. Because fewer low energy, secondary electrons reach the detector from the contaminated surface, the contamination region often appears as a darkened area in the secondary electron image. Typically, in a beam scanning instrument, this contamination layer is in the shape of the rastered pattern on the sample—a rectangle. The effect is more pronounced at high magnifications and lower accelerating voltages—just the conditions under which smaller surface features are often analyzed.

Further, if the contaminated area is measured in an atomic force microscope (AFM), the build-up of this material has been shown to be quite pronounced[i]. Cleaning the specimen before placing it in the scanning electron microscope (SEM) helps, but there is always a small amount of hydrocarbon in the system. Also, the source of these residual hydrocarbons is manifold as they can be left behind from the manufacturing of the tool, part of the vacuum or lubrication system, or derived from the sample or sample handling. Hence, there is the requirement for periodic chamber cleaning.

Previous approaches

While some early decontamination efforts showed moderate success using prolonged purging with dry nitrogen, this was a slow and inefficient process that required overnight, or even several-day periods of flowing gases.

Better results in mitigation of the hydrocarbons were seen by sequestering or trapping them with cold surfaces provided by liquid nitrogen cold traps. As these surfaces eventually become saturated with the immobilized hydrocarbons, this solution is only temporary and the system eventually must be warmed up and cleaned to completely remove the unwanted materials.

Historically, plasma cleaners have been adapted from plasma ashers where the goal was to remove all the organic materials. Commonly used in the semiconductor industry to remove resistive materials composed of polymeric hydrocarbons, these large systems gave rise to smaller, laboratory units which were used to clean samples and parts often destined for examination in vacuum environments such as electron microscopes and other surface analysis tools (right).

Primary Plasma Cleaning Systems

Traditional plasma cleaning systems may be categorized as primary plasma systems. These create a gas plasma via an energy source which ionizes and dissociates a source gas, resulting in physically and chemically active components. Samples intended for cleaning are placed directly into the gas discharge, on or near the electrode plates of the system and receive full exposure to all the aggressive species of the plasma (i.e. ions, free radicals, and by-products).

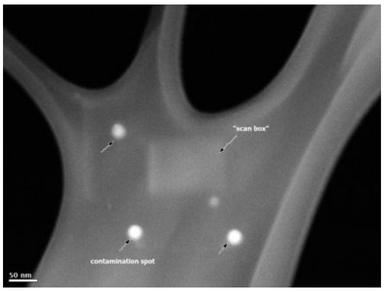
The plasma may be generated with a variety of methods including radio frequency (13.56 MHz) or microwave (2.54 GHz) energy. Lab systems are often of a barrel or cylinder design which can be evacuated with the samples placed inside. When energized, the sample is attacked by the plasma species from all directions (isotropic etch). Parallel plate designs allow for anisotropic etching if the desired result is to remove material solely from a particular direction.

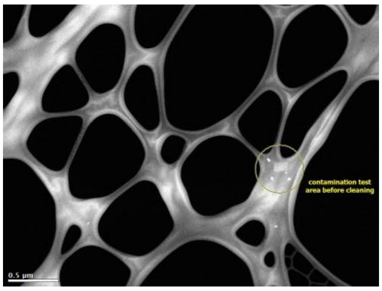
Numerous references exist whereby researchers have proven these tools can be used to clean either specimens or sample holders destined for examination in electron microscopes and that these cleaned items improve the imaging and analytical results of the tools[ii]. However, cleaning samples and parts destined to go into the microscope mitigates only part of the problem: it leaves the issue of the cleaning of the chamber itself unaddressed.

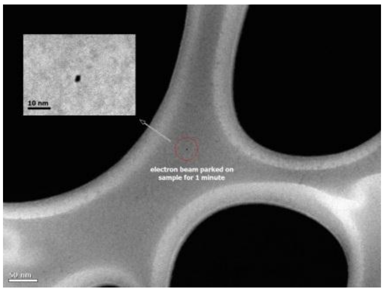
Conventional plasma cleaners

The original work by Zaluzec[iii] resulted in a patent and commercial products that use direct plasma to clean samples and parts destined for use in electron microscopes. This has allowed scientists to remove substantial contamination artifacts from their images. Follow up work, such as that of Isabel and Fischione[iv], continued to demonstrate the value of plasma cleaning to remove problematic hydrocarbons and even to clean contaminated samples if they have already suffered beam-induced carbonaceous polymerization on their surfaces. Most of these direct cleaners use argon or argon/oxygen mixtures. In these systems, the samples are directly exposed to the energetic ions, and even at low power (10–20 W) there is the possibility of surface ablation and damage of sensitive samples such as lacy carbon films.

Recent confirmation that downstream plasma cleaning is safe for these sensitive samples can be seen in the accompanying three STEM HAADF micrographs of lacy carbon support films (provided by Vincent Hou of Nanolab Technologies). From the left, the first image is a low magnification examination of a sample before cleaning. The second image is a higher magnification of this sample clearly showing contamination artifacts caused by examination in the STEM mode (both from spot and scanning modes). After cleaning for 15 minutes in the Evactron® SoftClean™ Chamber, the third image shows that even after one minute of stationary electron beam exposure, no contamination is observed and the clean sample is preserved.



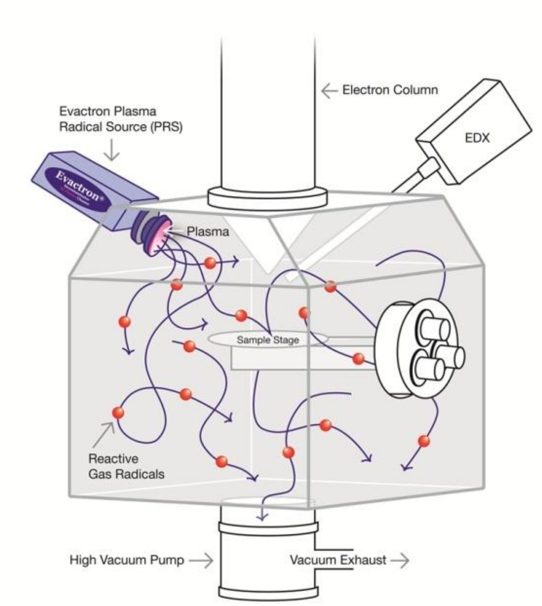




Plasma cleaners are now common in the electron microscope (EM) suite with users in many disciplines cleaning both samples and sample holders prior to microscopy. Direct plasma cleaners are available from several manufacturers. These vary both in sophistication and price. However, these instruments do not address the internal surfaces of the microscope. Although the amounts of mobile hydrocarbon contaminants contained by those internal surfaces might be miniscule, the scales at which observations are now being pursued are such that the issue requires an effective resolution if progress is to be maintained. Any proposed solution which rests upon a requirement for the disassembly and manual cleaning of components is unlikely to be practical and – even if pursued – would entail a level of manual intervention that would potentially place a disproportionate economic burden on any assessment of research costs and benefits.

Downstream Plasma Cleaning Systems

In 1999, XEI Scientific patented and introduced a radio frequency (RF) plasma product that used the technique of secondary or downstream plasma cleaning to address the problem of cleaning internal surfaces of vacuum chambers in electron microscopes. A schematic of this downstream plasma process is shown here.

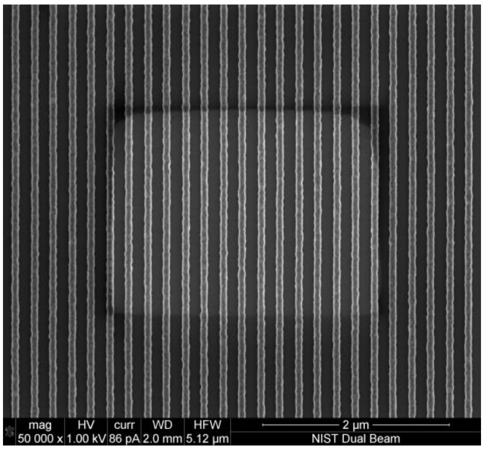


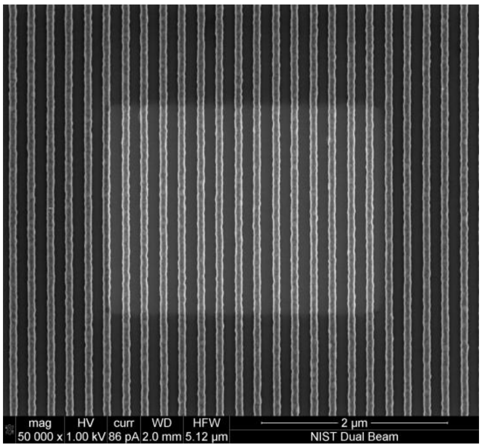
How it works

This type of system produces the active plasma in a remote chamber (called a Plasma Radical Source or PRS) and transfers the active species to the cleaning chamber via gas flow, relying primarily on the chemical activity of the reactive radicals produced by the plasma.

Experiments with different gases to create the plasma have shown room air to be an excellent source of oxygen to create reactive radicals and efficiently crack hydrocarbon molecules. It has the benefits of being available, free, and safe. Additionally, through the choice of other noncorrosive gases for producing radicals, different chemical etch processes may be selected while benign regimes for sensitive components and optimized chemistries may be obtained for the fast removal of unwanted contaminants.

While the energetic ions are generally contained in the external PRS, reactive gas radicals are allowed to drift through the vacuum chamber and come into contact with the sample and internal surfaces. Photons in the plasma are in the Vacuum UV (VUV) wavelengths, and VUV energy is very effective in breaking most organic bonds, that is, CH, CC, C=C, CO, and CN. Thus, high molecular weight contaminants are broken into smaller components. A second cleaning action is carried out by the various oxygen species created in the plasma (O2+, O2, O3, O, O+, O, ionized ozone, metastably-excited oxygen, and free electrons), which combine with organic contaminants to form H2O, CO, CO2, and low molecular weight hydrocarbons. Exhibiting relatively high vapor pressure, these compounds are easily pumped out of the microscope by the vacuum system. An illustration of the effects of plasma cleaning on a NIST reference sample is shown here, before (left) and after (right) treatment.

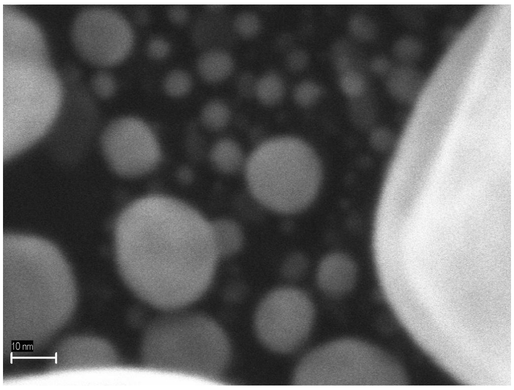


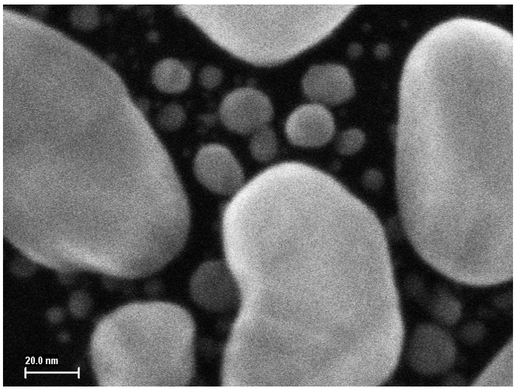


This downstream plasma cleaning technique has proven to be extremely useful in electron and ion column instrumentation as the technique can remove unwanted hydrocarbon contamination from the insides of complex instrumentation without disassembly. This example is the Evactron® PRSv equipped with a KF40 flange and impedance matching electronics configured for SEM or dual beam FIB use.

Cleaning cycle

Cleaning is done at higher pressures than those that typically exist when the microscope is in operation. However, the process is quite fast and often can be accomplished immediately after a vent or sample exchange cycle. Also, once a system is initially cleaned, maintenance can usually be accomplished with a weekly cleaning of 10 minutes or less. This obviously depends on the type and cleanliness of samples being inserted into the scope. The photos below show an example of improvement in the image quality of a gold-on-carbon SEM resolution sample. The image on the left was taken before cleaning with the Evactron® process, and the image on the right is the result after plasma cleaning.





A further benefit of this technique is that its benign nature has proven safe for common, but sensitive materials found inside of electron microscopes (such as X-ray detector windows), and has been used for years with no adverse effects observed by a very large number of researchers.

Today's scientists are not only imaging and measuring at a nanoscale level; they are also manipulating matter and creating features of nanometer dimensions. This capability requires absolutely clean surfaces before manipulation. Recent work by Mancevski has shown that downstream plasma cleaning was essential for successful vapor phase cutting of CNTs using a nano-manipulator system[v].

Plasma radical sources are now available in a number of configurations for most makes and models of electron and ion microscopes. Standard PRS units commonly allow for air or oxygen and oxygen/argon mixtures. These units have KF 40 flanges adapted for most SEMs and Dual Beam FIB/SEMs. Further, because they are portable, these systems may be moved about cleaning a number of different electron microscopes in the laboratory. A high vacuum Conflat flange version is available for surface analysis tools or other high vacuum chambers and provides for the use of hydrogen gas. Versions of the PRS specific for TEMs where a patented hollow cathode is inserted through the sample insertion port are in final design and testing with most major TEM suppliers. This unique design delivers the cleaning capability directly to the hard to reach area where beam and sample interact in the TEM.

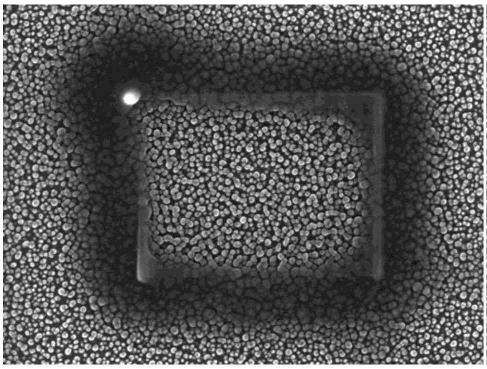
XEI's Evactron® TEM Wand™ is shown here with the plasma source mounted at the distal end of a TEM sample exchange mechanism.

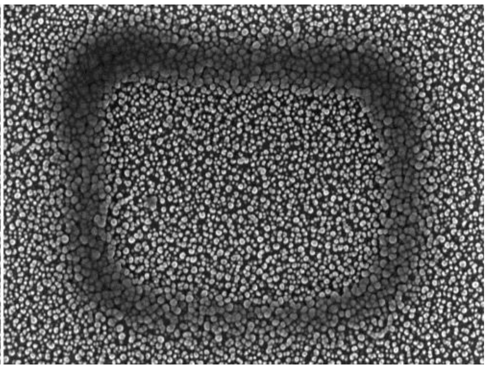
Downstream Plasma Cleaning in Metrology

Workers at National Institute of Standards and Technology in Washington, DC (NIST) are strong believers in removing all contamination from both samples and chambers. The work of the NIST nanoscale metrology group needs highly accurate scanning electron and helium ion microscopy down to sub 1nm resolution[vi], and this demands contamination-free operation. Repeatable results require a high degree of cleanliness so that over the few minutes of measurement, the sample does not change noticeably. This also includes the need for consistent secondary electron emission (yield). Cleaning regimes utilising a liquid nitrogen trap, clean nitrogen gas bleeding, cryo, special pump oil, oil free methods, and others had all been deployed at NIST without reaching the standards required by the Institute.

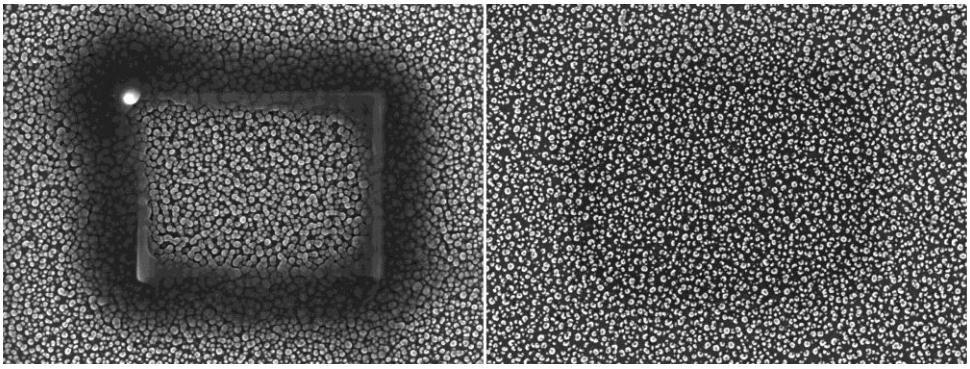
However, the downstream plasma cleaning method that is deployed at NIST has led to their adoption of the technology – using the XEI Evactron® decontamination system – since it has been assessed as the only current mechanism that will allow the NIST contamination specifications to be met. NIST does not endorse a specific product or brand, but is a leading advocate of the use of plasma-based SEM cleaning. Implementation and regular use of these methods has made it possible to eliminate the effects of electron beam induced contamination.

This set of images was generated by Andras Vladar at NIST. They illustrate carbon contamination on a sample in a scanning electron microscope. The left images (i & iii) show the build-up after scanning for ten minutes at 5kV and 10pA. The upper right image (ii) shows the effect of using a cryo trap. Contamination is reduced but is still clearly present. Contrast this with the lower pair of images. The right hand image (iv) shows the result of using downstream plasma cleaning with almost no contamination observed.





(i) Contamination build up (ii) Contamination reduced with use of a cold trap

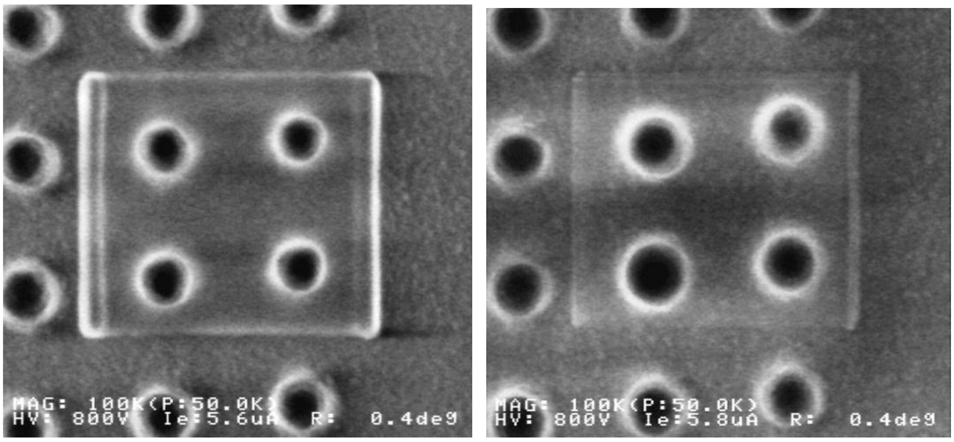


(iii) Contamination build up   (iv) Almost complete prevention of contamination

Other applications

Critical Dimension measurements

Comparison imaging to examine the effects of contamination on critical dimension (CD) measurements has shown that these image artifacts can affect dimensional measurements[vii]. In CD work, modification of dimensions by the SEM imaging process causes a loss of precision in the measurement. Using a very clean Hitachi 6280, the test pattern (left image) began to show filling-in of the holes after a 20-minute scan. After *in-situ* cleaning of the chamber and the specimen, a repeat of the measurement showed no filling of the holes and a much-reduced scan mark (right image).



(CD test pattern showing fill-in of the holes during scanning (left) while after *in situ* cleaning of the chamber and specimen, a repeat of the measurement shows no filling of the holes and a much reduced scan mark.)

Artifact removal

It is also well established that cleaner vacuum systems can assist in removing spurious analytical artifacts. Often, carbon analysis can contain contributions that do not originate from the sample but can be due to contamination. Work by Strein and Allred[viii] showed that the antechamber of an X-ray photoelectron spectroscopy system was introducing a thin layer of carbon onto samples, making carbon analysis unreliable. Downstream plasma treatment in the antechamber removed the contamination.

Analytical microscopy and spectrometry

Horiuchi et al[ix] have also shown that analytical transmission electron microscopy (TEM) results on polymer brush samples could be accomplished with a system cleaned using downstream plasma. Electron energy loss spectrometry (EELS) in imaging mode could be used for high-resolution carbon mapping.

Nanoscale applications

Having pristine surfaces is an absolute requirement for nanomanipulation and nanofabrication. The previously reported work by Mancevski has shown that downstream plasma cleaning was essential for successful vapor phase cutting of carbon nanotubes using a nanomanipulator system[x]. Also, electrical measurements made by positioning minute probes on circuits, using nanopositioning systems located inside SEMs and FIBs, require that the probes be free of contamination in order to make good contacts. *In situ* cleaning of these devices is a requirement for accurate measurements and plasma cleaners are considered almost an essential accessory for nearly all of these new tools.

Product Development

The fundamental technology associated with downstream plasma cleaning has been further developed to increase the practical utility and availability of the system. In its earliest forms, plasma cleaning required separate systems for the cleaning of samples on the one hand and electron microscopes on the other. This was a result of the PRS unit typically being mounted only on the electron columns and not available for use on the desktop. As a consequence, market research indicated that many electron microscopists did not have ready access to a plasma cleaning system.

The natural solution was to combine both desktop and column cleaning in the same tool. Describing one such development, XEI created first the Evactron® SoftClean™ Chamber. This is a small, versatile chamber which holds TEM sample holders, SEM samples or small parts destined for examination in the microscope. This chamber allows users of existing systems to mount their Evactron® plasma radical source onto the chamber, delivering the same downstream plasma cleaning technique and achieved pre-cleaning without the

need of another, direct plasma cleaning tool. This way, sensitive samples can be chemically etched safely and without exposure to high energy ion bombardment and potential damage. It can also be used as a safe storage repository for clean samples when it was not in use as a cleaning chamber. However, the user is required to physically cable and un-cable the instruments as the controller can only support one PRS unit at a time.

In a further development, a combined cleaning system was introduced, the XEI Evactron® CombiClean™ decontaminator. Using a similar cleaning chamber, this provides a new system controller which can support multiple PRS units simultaneously. Now the user can simply select from the control panel whether to use the internal PRS cleaning on the combined chamber or to clean the electron microscope. Incorporating all of the features of the original enabled the system to clean parts going into the microscope and act as a storage chamber as well as control the column cleaning PRS unit.

Conclusions

Downstream plasma cleaning has evolved into a very effective method to get the best possible images and analytical data from sophisticated electron and ion column tools. These systems have proved to be a reliable technique to remove problematic hydrocarbon contamination from samples, holders and microscopes themselves. This downstream plasma cleaning technology is allowing researchers in various fields to optimize performance from their microscopes and investigate, image, analyze and manipulate materials. XEI alone can account for over 1,300 installations of their tool on nearly all makes and models of SEM and Dual Beam FIB/SEMs. Today most new high-resolution tools come equipped with some form of downstream plasma cleaning upon delivery from the factory. Further, service personnel often carry a portable version of the system when they make service and preventative visits in the field in order to maximize SEM performance.

References

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[iv] TC Isabell et al., Micros Microanal 5 (1999) 126–35.

[v] Mancevski, V. and Rack, P. D. Vapor Phase Cutting of Carbon Nanotubes Using a Nanomanipulator Platform. Materials Science & Technology 2010 Conference and Exhibition, October 17–21, 2010, Houston, TX.

[vi] A Vladar, C Archie, and B Ming, Meas Sci Technol 22 (2011) 024004, doi: 10.1088/09570233/22/2/024004.

[vii] A Vladar, NIST, personal communication.

[viii] L Strein and D Allred, “Use of commercial RF Plasma Cleaner in eliminating adventitious carbon contamination in an XPE system,” Microscopy and Microanalysis poster, 2008.

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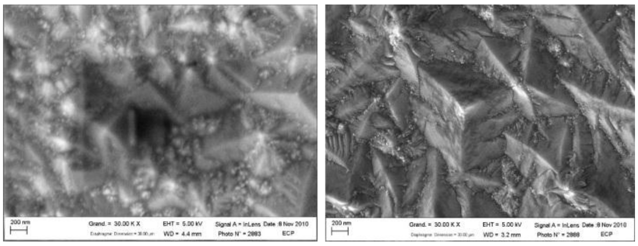
Examples of Evactron® Cleaning

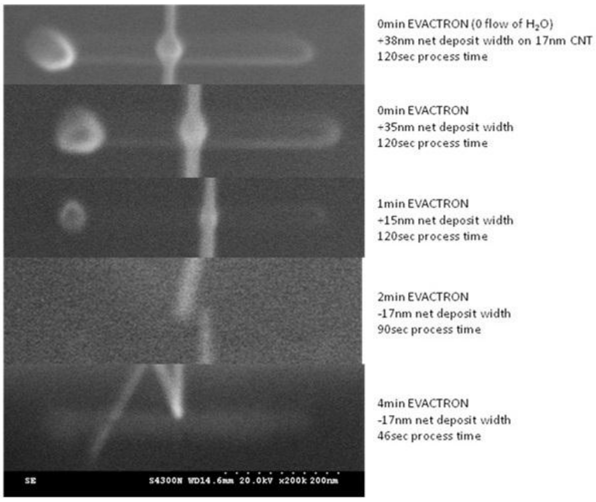


Before After

Images courtesy of (CNRS/ECP-MSSMAT)

"We have had very good experiences with the Evactron system. We use it with almost all samples. It has allowed us to use the full possibilities of our SEM, in particular to be able to perform really high magnification routinely (something we could previously do only occasionally and only with very well prepared samples)."

- *Thierry Auger, CNRS/ECP-MSSMAT*

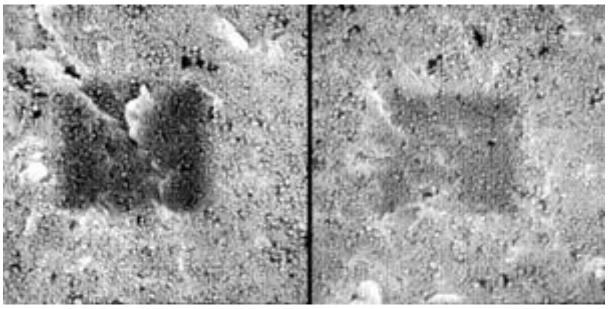


Vapor-Phase Cutting of Carbon Nanotubes Using a Nanomanipulator Platform
MS&T'10, October 18, 2010

Xidex
NANODEVICES AT WORK

Xidex Corporation reported at the 2010 MS&T conference in Houston, TX, that "the Evactron Decontaminator reduces the competitive carbon deposition and enhances the etching rate in vapor-phase cutting of carbon nanotubes using their nanomanipulator platform."

- *Vladimir Mancevski, Xidex*



"Black focusing squares on resolution test specimens are a common problem. Before cleaning the instrument for the first time, a black square covers the scanned area. After cleaning for 5 minutes, the square is greatly reduced."

-*Andras Vladar NIST*

Evactron Successes

The Evactron® De-Contaminator System has been used to solve various contamination problems by users. This is a partial list illustrating its use.

Hitachi S4700: Spansion, Sunnyvale, California, USA
Improved resolution was observed, on a 5 year old instrument so that sputtered PT grains could be imaged at 5kV and < 3 nm resolution.

FEI 235 Dual Beam FIB: FEI Company, Hillsboro Oregon, USA
The FEI Company was having problems meeting contamination specifications on new Dual Beam 235 FIBs during the final manufacturing test. Solvent cleaning and wipe downs of chamber and stage failed to control the problem on some units. The problem was thought to be with residual machining oil on parts.

With less than four hours of Evactron cleaning over two days during a demonstration, the contamination deposition rate was brought down from being 2 times over the FEI specification amount to less that 10% of the specification. FEI has purchased Evactron units for manufacturing to make sure all Dual Beam FIBs are clean on departure.

FEI 235 Dual Beam FIB: AMD, Sunnyvale, California, USA
The FEI FIBs make use of a Gas Injection Systems (GIS) for metal deposition using the ion beam. This technique uses organometallic gases that react with the ion beam to deposit metals on the scanned areas. Organic gases, released as a by-product, contaminate the chamber and the specimen surface.

AMD uses the Dual Beam FIB to study the structure of sub 170 nm copper vias for process control. The use of GIS platinum deposition guns had released organics into the chambers, creating foggy images of these small structures. The installation of an Evactron system allows for faster and clearer imaging of these very small structures.

JEOL 6400: Kimberly Clark, Neenah Wisconsin, USA
A JEOL 6400 was used for imaging wood and paper products. The microscope had also an oily window problem on the EDS Detector. Installing an Evactron system cleaned up the chamber and periodic use prevents build-up of contamination on the wall of the chamber. Additionally, oil stopped building up on the EDS Detector. No damage to the EDS window has been reported.

JEOL 6400 and JEOL 845: IBM Canada, Bromont, Quebec, Canada

This semiconductor research lab had persistent problems with contamination until the Evactron system was installed on these two SEMs.

Hitachi S-4500: IBM San Jose, California, USA

The user images magnetic disks coated with a lubricant. Over time, the lubricant had heavily contaminated the chamber and images of defects had to be obtained solely on the first pass. After cleaning with an Evactron De-Contaminator (D-C), contamination problems disappeared. The user now cleans the chamber with an Evactron D-C every 2 to 4 weeks, as needed. The system also has an EDS detector and after the installation of the Evactron system, the oily window problems have stopped.

The results were presented at a 1999 Microscopy & Microanalysis (M&M) conference in the paper “The Removal of Contamination Deposits From Defects in Thin Film Magnetic Disks By Oxidative Cleaning Inside The SEM”, by Sharon Myers and Ronald Vane.

Hitachi S-4500: AMD, Sunnyvale, California, USA

An Evactron system was installed on a new SEM to prevent contamination. It has been operated by an automatic system for 2 minutes cleaning, every day. No contamination problems have ever been noted on this SEM.

Hitachi S-4700: NIST Gaithersburg, Maryland, USA

NIST borrowed and later purchased an Evactron D-C for controlling contamination in an Hitachi S-4700 SEM. Their research on contamination control resulted in the paper "Active Monitoring and Control of Electron Beam Induced Contamination" Proc. SPIE Vol. 4344 (2001), 835, by András E. Vladár, Michael T. Postek and Ronald Vane. This study found that the Evactron device was "effective in cleaning the vacuum of the specimen chamber of laboratory and production metrology SEMs."

Hitachi S-4700: Oak Ridge National Laboratory, High Temperature Materials Lab, Oak Ridge, Tennessee, USA

Roughing pump oil back-streaming through the sample exchange chamber was causing dirty specimens and chamber contamination that could not be controlled with a liquid nitrogen (LN) cold finger. Contamination is controlled by use of both the plasma cleaning and the nitrogen purge features of an automatic Evactron system.

Hitachi S-4700: University of Illinois, Champaign-Urbana, Illinois, USA

A three year old Hitachi S-4700 SEM had constant contamination problems since being installed. The user had added a turbomolecular pump and heated micromaze foreline trap, but they made little difference. LN traps, top and bottom, had been kept in service constantly, but failed to stop contamination problems.

The user purchased an Evactron system with the automation package. The automation package operates the Hitachi SEM evacuation system with the proper delays to allow for the cooling of the heated aperture before venting the chamber to Evactron operating pressure. The automatic package then operates the plasma clean cycle for 2 minutes. The system is then ready to image specimens again, 40 minutes after the Evactron cleaning cycle.

Multiple cleaning cycles were done during installation for testing and demonstration purposes over two days. After installation was completed, an old gold on carbon specimen was imaged. This specimen had shown chronic contamination problems in the past with black squares forming quickly. After Evactron cleaning of the chamber, the specimen showed no black square formation even with dry LN traps during this first test. Cleanliness is maintained by operating the Evactron system on a weekly maintenance cycle.

LEO 1550 and JEOL 6400: University of California, Berkeley, California, USA Microfabrication Laboratory

Student-used SEMs were heavily contaminated by student use on dirty specimens. The SEM maintenance technician does not have time to watch over every user. LEO 1550 SEM has a valveless turbopump pumping system and no load lock. The SEM technician did not want to teach users how to clean with Evactron system and did not want to spend time doing cleaning himself.

The Evactron De-Contaminator (D-C) was set up to operate during every pump down cycle for 100 seconds. By adjusting the gas leak and the vacuum set points for plasma operation, the cleaning takes place while the SEM chamber pumps down through the 0.9 Torr to 0.4 Torr pressure zone. Operating the Evactron D-C adds less than one minute to pump down time and was not noticed by most users. Pump down to high vacuum was speeded up because UV light generated by the plasma is effective at desorbing water vapor from walls during the cleaning cycle in the roughing mode. Cross contamination between specimens has disappeared. Higher resolutions are being observed. UCB Physics Department ordered a third Evactron D-C for the new FEI Sirion SEM in 2003 for use at Lawrence Berkeley Labs.

LEO 1525: NIST, Boulder, Colorado, USA

NIST purchased an Evactron De-Contaminator to assist in backscatter electron diffraction measurements. The slightest layer of carbon interferes with these measurements. Evactron cleaning of the specimen and chamber before measurement ensures the cleanest possible measurements. By adjusting the leak rate, pressure can be maintained in the Evactron cleaning zone for up to five minutes, if needed.

LEO 1550: Intel, Sacramento, California, USA

The microscope is staying clean and pump-down speeds are increased. UV light from the Evactron system desorbs water from chamber walls during pump-down with Evactron cleaning. This system has no sample airlock so the Evactron De-Contaminator is used to remove both hydrocarbons and water vapor from the chamber.