

Environmental Contamination Sources and Control in High Resolution Scanning Electron Microscopy

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Traditionally, contamination control in SEMs has focused on pump oils, finger prints, dirty specimens, and good vacuum practice in manufacturing. The use of dry pumps at all stages of the vacuum system of new FE SEMS, and the use of better vacuum practices on the part on users and manufacturers have made environmental hydrocarbons, the hydrocarbon background contamination of our world, a significant source of the remaining hydrocarbons in electron microscope vacuum systems. These environment sources of hydrocarbons (HC) cause a loss of resolution and contrast in imaging at the highest levels of magnification.

The Semiconductor industry and associated nano-sciences have brought on a demand for instruments that can image structures <5nm in size at < 2KV. Instrument manufacturers have responded with Field Emission (FE) instruments that can give better than 200KX magnification with ease and at high contrast with low KV beams. Control of contamination has taken more importance as semiconductor manufacturers move to ever smaller dimensions. It is already common to be looking with low KV (<2KV) at features <10nm in size that are close to the resolution limits of the instruments. In such cases, the smallest amount of HC in the chamber can cause a loss of resolution and contrast. The electron beam reacts with any stray HC in the beam path or on the surface to create HC ions that then condense and form a hydrocarbon gunk on the area being scanned. Despite dry pumps and LN traps, these artifacts and contamination haze continue to be formed. What is the source and what can be done?

It is important to understand that SEMs are not designed from the ground up as ultra high vacuum instruments. Unlike XPS and Auger spectrometer systems that require ultra high vacuum to obtain high resolution energy information from the emitted electrons, electron microscopes only look at electron flux. Backscatter detectors do have some energy discrimination, but the energy filtering is crude and does not need ultra high vacuum. High vacuum SEMs are not bothered by water vapor and can do imaging with pressures in the low 10⁻⁵ Torr range in the specimen chamber. But good SEM imaging is disrupted by HC. Roughing pump oil in the chamber with a partial pressure of 10⁻⁷ Torr will cause black squares to form on specimen images.

Exposure of samples and surfaces in a vacuum chamber to room air is an under recognized source of environment HC contamination in microscopy. Long known and understood in the surface analysis, HC collects on all surfaces exposed to room air. Room air is full of traces of amounts of organic vapors from plant, animal, and synthetic sources. XPS and Auger spectroscopy reveal a carbon background on everything exposed to it. The effective ways the rid surfaces of these carbon compounds are to drive them off or destroy them with heat, sputtering, or chemical etch. Solvent cleaning may leave residues. A freshly prepared specimen will show little if any contamination, but the same sample left exposed to room air overnight or longer, even in a protective container, will show surface carbon build up.

Environmental HC can also enter into an EM chamber whenever the chamber is opened for specimen exchange or instrument servicing. Servicing the instrument for stage repairs, detector exchange, pole piece replacement, etc. can bring in HC from the room air. During servicing, cleaning parts with solvents is the traditional method, the results are uncertain until the system is pumped down and a image taken.

Environmental contamination will also accumulate on specimens used for system performance tests. The common gold on Carbon specimens are unusually contaminated by exposure to environmental HC in side their carrier cases. These specimens are best when plasma cleaned before use. Plasma cleaning of specimens before they are placed in the EM chamber is a commonly applied method to stop contamination from reaching the EM chamber. Desk top plasma cleaners for cleaning specimens and TEM stages are available form several manufacturers. These cleaners use exposure to Argon, Oxygen, or mixed gas plasmas to sputter etch the HC layer off the surface of the specimen and stage. Used for too long they cause sputter damage to the specimen surfaces.

The XEI Scientific Evactron Anti-Contaminator offers an alternate cleaning method that removes environmental HC and HC from any other source from the inside of electron microscopes and dual beam FIBs. Using a remote RF plasma to make O radicals from air, Evactron cleaning uses the pumping differential to flow the Oxygen radicals though the specimen chamber and oxidize off the HC from all surfaces. The process is quick to remove the typical low levels of HC present, and it possible to complete cleaning in less than 5 minutes on many specimens and chambers.

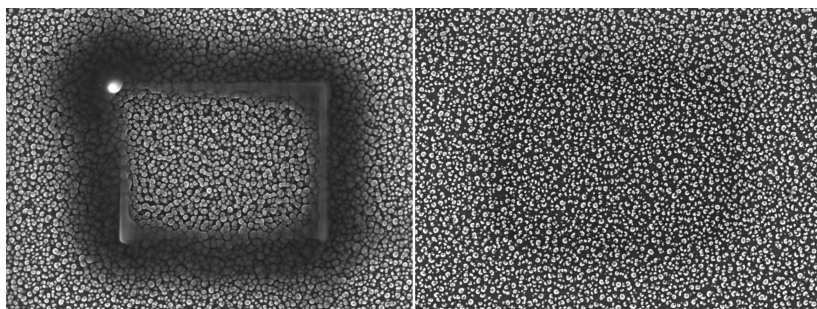


Figure 1. Two locations of a silicon "grass" sample irradiated for 10 minutes before (left) and after (right) the use of Evactron anti-contamination device. 50KX. [1]

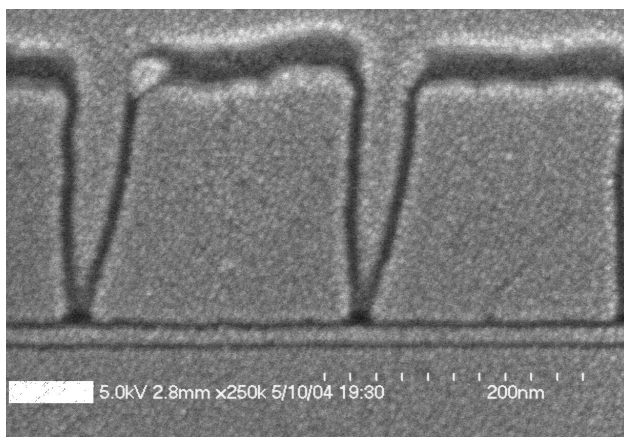


Figure 2. Example of resolution improvement after Evactron cleaning. Small sputter Pt islands are visible on a semiconductor cross section at 250 KX at 5 KV on a 6 year old Hitachi S4700.

References:

- [1] Andr s E. Vlad r, Michael T. Postek and Ronald Vane* Active Monitoring and Control of Electron Beam Induced Contamination Proc. SPIE Vol. 4344 (2001), 835.