# Ultra High Resolution SEM on Insulators and Contaminating Samples

Trisha Rice* and Ralph Knowles**
*FEI, *Hillsboro, OR, USA and **Eindhoven, The Netherlands*
*trice@feico.com*

Historically, SEM developed as a high vacuum technique requiring sample chamber vacuum of $10^{-5}$ Torr or better. Energetic electrons scatter from any molecules they encounter, so their creation and transport from source to sample, through the focusing lenses of the electron column, requires high vacuum. This imposes a number of limitations on the kinds of samples that can be examined. Samples must tolerate the vacuum environment and the vacuum system must tolerate the sample. Generally, samples have to be solid, clean, dry, and not contain volatile components. Furthermore, since the vacuum insulates the sample from everything except the stub that supports it, non-conductive samples require a conductive pathway between the scanned region and ground to prevent the accumulation of charge deposited by the beam electrons. Non-conducting samples are usually coated with a conductive material such as gold or carbon. In time, low vacuum SEMs were developed to relieve many of these restrictions and to expand the applicability of SEM analysis to new classes of specimens. The first widely used low vacuum SEM was the Environmental SEM (ESEM) introduced by ElectroScan (now part of FEI) nearly two decades ago. Although the word "ESEM" originally designated a specific instrument, it has now come to refer to a collection of technologies that enables high performance low vacuum SEM.

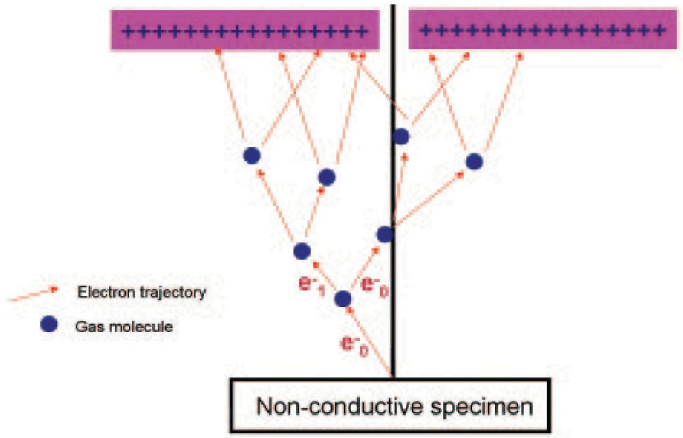


*Fig. 1: In gaseous secondary electron amplification, secondary electrons emerging from the sample surface are accelerated toward the detector by an imposed field. They collide with and ionize gas molecules creating additional electrons and positive ions. The process creates a cascade of electrons that results in proportional amplification of the original secondary electron signal.*

An essential breakthrough in the development of ESEM was the integration of two closely spaced pressure limiting apertures (PLA) in the final lens of the electron column. The regions below, between, and above the PLA are pumped separately to provide a graduated vacuum from as low as 50 Torr, in the sample chamber, to $10^{-5}$ Torr, or better, in the column and emission chamber. Depending on the particular configuration, additional pumping stages may be added to further improve the vacuum in the emission chamber. Locating the apertures close together at the bottom of the column reduces the distance the beam travels through a gaseous environment, the beam gas path length (BGPL), thereby minimizing the effects of scattering from gas molecules.

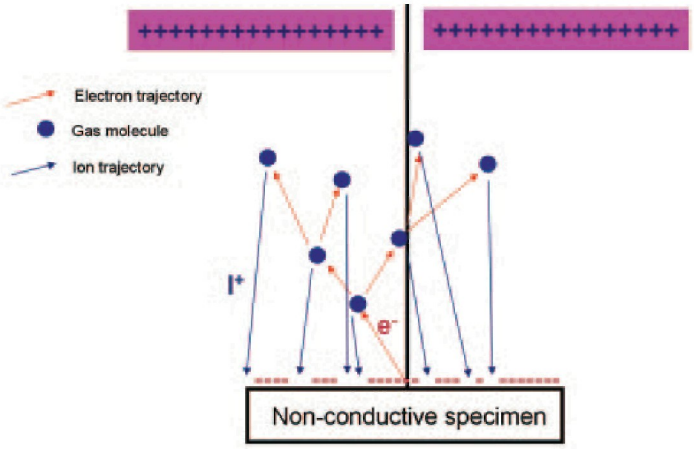


*Fig. 2: Gaseous SE detection provides charge control: as the positive ions created by the cascade migrate to the sample surface where they neutralize charge deposited by the electron beam.*

## Gaseous secondary detection

Another essential breakthrough in the design of the first ESEMs was the development of gaseous secondary electron detectors. Everhard-Thornley detectors, the standard for early high vacuum systems, arc in a gaseous environment and self destruct. Newly developed gaseous detectors consist essentially of an electrode located just above the sample surface in the vicinity of the beam. A positive potential of a few hundred volts, applied to the detector, attracts secondary electrons emitted by the sample. As the electrons accelerate in the detector field they collide with gas molecules. The resulting ionizations create additional electrons, called gaseous secondary electrons, and positive ions. This process of acceleration and ionization repeats many times resulting in a proportional cascade amplification of the original secondary electron signal. The detector collects this signal and passes it directly to an electronic amplifier.

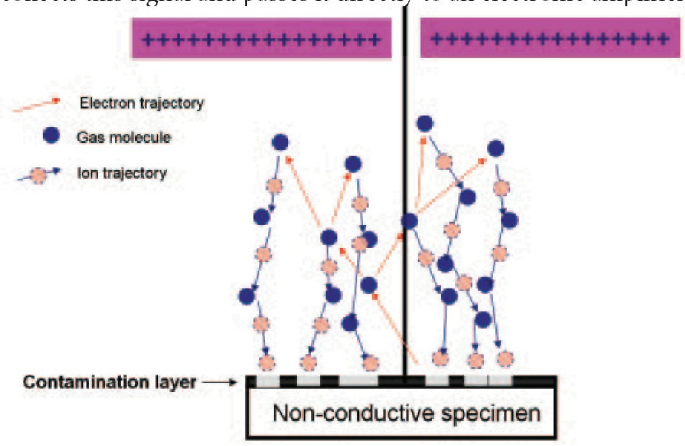


*Fig. 3: Gaseous SE detection also provides contamination control: At the sample surface positive ions not only eliminate charge but also mediate the chemical conversion of polymerized hydrocarbons to volatile compounds that can be pumped away by the vacuum system.*

## Reduced charging without coating

An ancillary benefit of gaseous secondary electron detection is the elimination of sample charging. Charging occurs in high vacuum SEMs when charge deposited by the beam electrons accumulates in insulating samples. The fields induced by this induced specimen charging cause local variations in secondary electron emissions, and deflection of the primary beam. Both interfere with imaging. In gaseous secondary electron detection, positive ions, generated by the signal amplification process, are attracted to the sample surface as

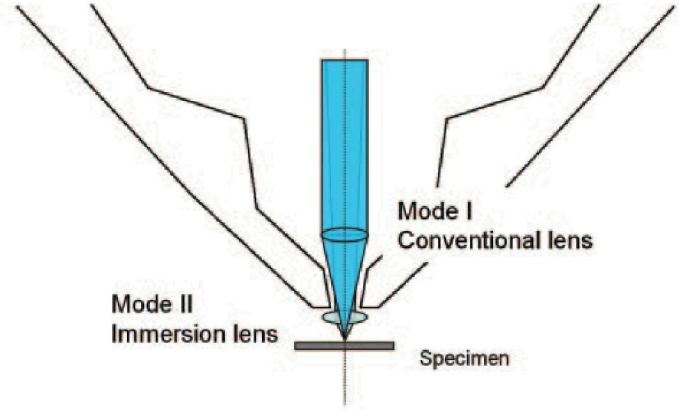


*Fig. 4: With an immersion lens the sample is immersed in the lens field allowing tighter focusing of the electron beam.*

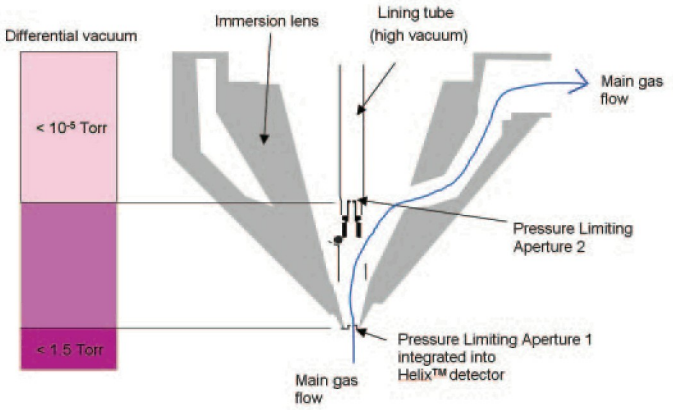


*Fig. 5: Through-the-lens pumping extends the high vacuum region of the column closer to the final lens and provides more efficient removal of gas from the region.*

charge accumulates. There, they suppress the local fields and effectively eliminate charging artifacts. The elimination of charging also eliminates the need to apply conductive coatings that can obscure image detail and interfere with compositional analysis.

## Reduced contamination

Another advantage of low vacuum operation is the inherent suppression of sample contamination. Contamination usually appears as a dark rectangle covering an area that has been scanned by the electron beam. It results from the accumulation of hydrocarbons

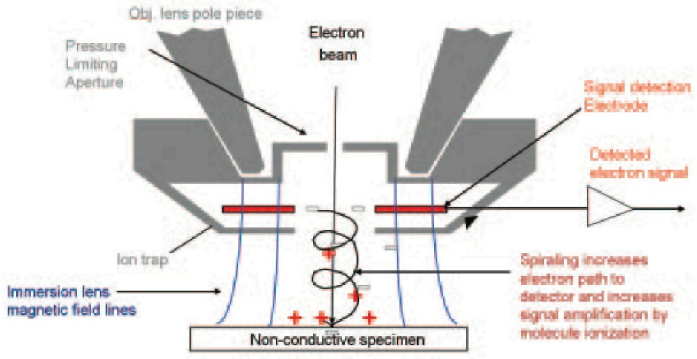


*Fig. 6: The Helix detector takes advantage of the immersion field to force electrons along a long spiral path. The longer path results in more ionizations and higher gain while maintaining a short working distance.*

polymerized by the beam. Good vacuum design can eliminate the vacuum system as a source of contamination, however, it cannot eliminate contamination introduced by the sample itself. Many of the new nanomaterials are particularly troublesome in this regard since they are often produced using wet chemistry processes. A mechanism to reduce or eliminate contamination in a low vacuum SEM is essential and the same ions that neutralize charge also inhibit the accumulation of surface contamination. As they migrate to the surface, these ions experience multiple collisions with neutral gas molecules and they arrive with energies too low to sputter. At the surface they appear to mediate the chemical conversion of hydrocarbons into volatile compounds that are then pumped away by the vacuum system.

## Nova NanoSEM

Although low vacuum SEM has been widely accepted for its ease of use and flexibility, it has not been regarded as an ultra high resolution technique. Other technologies, such as the development of low voltage SEM, have provided alternatives for charge control in a high vacuum environment but they do not address the issue of contamination. In fact, sensitivity to contamination effects increases at low voltage because of the decrease in beam penetration. The new FEI Nova NanoSEM has been designed from the ground up to provide ultra high resolution in a gaseous environment that addresses both charging and contamination.

**Field Emission** – Field emission sources provide the highest brightness, and permit the highest resolution. Only Shottky field

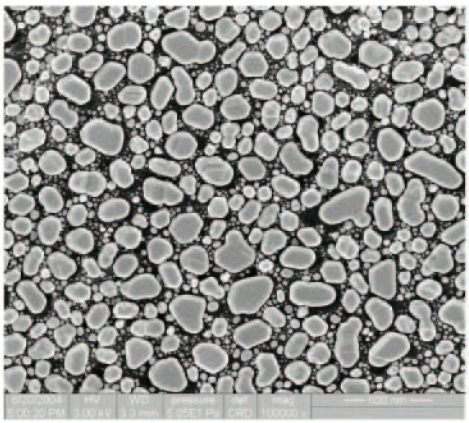

*Fig. 7: This image of a gold-on-carbon resolution standard was acquired in low vacuum mode. The Nova NanoSEM provides 1.8 nm resolution in low vacuum at 3 kV.*

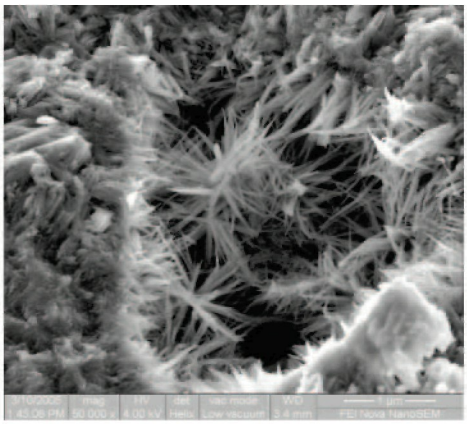

*Fig. 8: This image of cement (stored wet) demonstrates the Nova NanoSEM's insensitivity to evaporation of water or outgassing of other volatile components from the sample. Courtesy of Dr. Möser, Bauhaus Universität Weimar.*

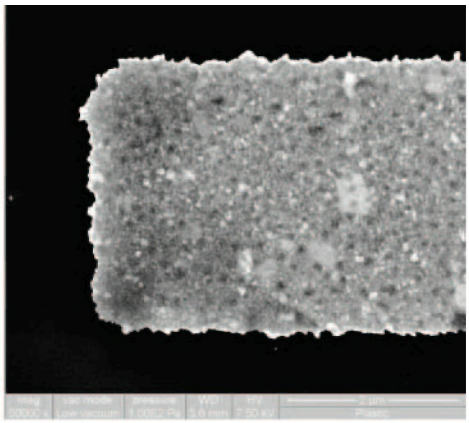

*Fig. 9: The subsurface information in this image of an insulating plastic from an electronics application cannot be seen in low voltage images or when the sample is coated.*

*Fig. 10: Diamond films are notorious for accumulating contamination. The gaseous amplification process removes contamination, even that which originates from the sample itself.*

*Fig. 11: In e-beam deposition, organometallic gases injected near the sample surface are decomposed by the beam depositing the metal fraction in a pattern that is determined by the beam scan pattern.*

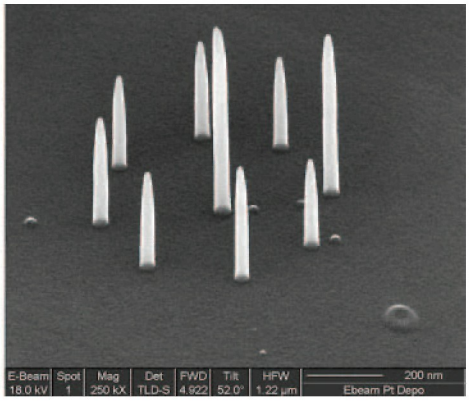


*Fig. 12: These platinum pillars were created by e-beam deposition.*

emission provides both the brightness required for UHR imaging and the high beam current (up to 22 nA) and the stability essential for high precision analysis.

**Immersion Lens** – The monopole immersion lens puts the sample within the lens field for improved resolution. It provides full resolution on tilted samples and permits the imaging of large samples at high resolution.

**Working Distance** – Short working distances permit optimal resolution.

**Vacuum** – Through-the-lens pumping extends the high vacuum of the column closer to the final lens field and increases pumping efficiency for reduced scattering and improved optical performance.

**In Lens Detection** – In-lens detectors for SE and BSE supplement conventional in-chamber detectors. In-lens detection works with the immersion lens field to improve collection efficiency and increase signal to noise ratios.

**Gaseous Detection** – The Helix detector combines short working distance with high gain for optimal resolution in low vacuum mode. A large field gaseous detector allows rapid surveys of large areas. Finally, a gaseous analytical detector (GAD) provides a very short beam gas path length to minimize spurious signals from scattered electrons during X-ray analysis.

## Helix Gaseous Secondary Electron Detector

The heart of the Nova NanoSEM's high resolution capability on insulating and contaminating samples is the new Helix gaseous secondary electron detector. Its designers were challenged to resolve the conflicts between gaseous performance and high resolution that were inherent in previous designs. SEMs in general provide highest resolution at shortest working distance. This is particularly true in the case of an immersion lens where the sample must be close enough to the objective lens to be immersed in lens field. It was also desirable to reduce the distance the beam travels through the gas to allow operation at the lowest possible beam energy. Low beam energy reduces beam penetration and optimizes the amount of surface information carried by the signal. These factors call for a gaseous detector that operates at a very low pressure in a very small space. On the other side of the equation are the requirements for gas amplification. The longer the path traveled by the secondary electron through the gas, and the higher the pressure, the greater the amplification.

The Helix detector resolves these conflicts by using the magnetic field of the immersion lens to lengthen the path of the gaseous secondary electrons. A charged particle, like an electron, moving through a magnetic field experiences a force that causes it to follow a spiraling path. The Helix detector is designed to take advantage of this phenomenon to increase the path of the secondary electron to many times the straight line distance between the detector and the sample. Gains as high as 5,000X have been measured for the Helix detector.

The Helix detector allows the Nova NanoSEM to be the first UHR FEG SEM to specify a resolution in gaseous mode of 1.8nm @ 3 kV, without compromising high vacuum resolution of 1 nm @ 15 kV and 1.8 nm @ 1 kV.

## Helix Charge Control

Gaseous amplification creates a large number of positive ions, far more than are needed for charge neutralization. Excess ions can interfere with imaging in a way similar to the electron charging effects that they are intended to eliminate. This usually manifests itself as difficulty in imaging large insulating samples. The problem is magnified in the Helix detector with its very high gain. The solution is simply to introduce another electrode between the detector and the sample that functions as an ion trap.

## E-beam deposition and nanofabrication

The FEI Nova NanoSEM can be configured with extensive e-beam deposition capability. In e-beam deposition an organometallic gas is injected into the vicinity of the beam at the sample surface. Under electron bombardment the gas molecules decompose. The metal deposits on the sample surface and the volatile organics are pumped away by the vacuum system. E-beam deposition can create complex three dimensional structures with nanoscale precision. The gas injector mounts through a port in the sample chamber and delivers its gas through a small retractable needle. Beam gas chemistries are available to deposit platinum, gold and tungsten. Also available are a 4K X 4K digital scan generator and a small step, piezo driven, 6-inch stage.

## Conclusion

The FEI Nova NanoSEM is the first ultra-high resolution, low vacuum SEM designed specifically to image charging or contaminating samples. It incorporates an array of technologies to achieve unprecedented resolution in both low vacuum and high vacuum modes: a field emission source, immersion lens, in-lens detectors and the Helix gaseous secondary electron detector. It also offers a complete solution for e-beam deposition.

**It's the same with microanalysis...**

**you want your supplier to always be there for you.**

At EDAX, we take enormous pride in providing today's best micro-characterization technologies. But this is not the end. In fact, installing your system is just the beginning of your relationship with EDAX.

We are equally proud of our unparalleled global support for all our customers. EDAX provides frequent, in-depth training classes, skilled service engineers on call to assist you and scientists to advise you in your specific applications.

Next time you are in the market for X-ray microanalysis, EBSD or micro-XRF systems, come to EDAX. We will not let go of you after the sale is made.

Discover what good service and support are all about at:

**www.edax.com/thinkEDAX**

EDAX® ...advancing materials characterization TSL™

AMETEK®