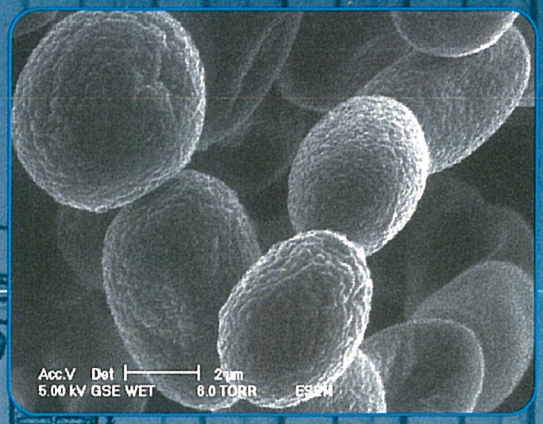
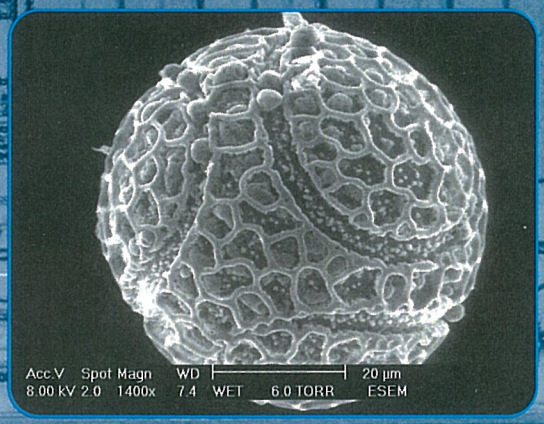
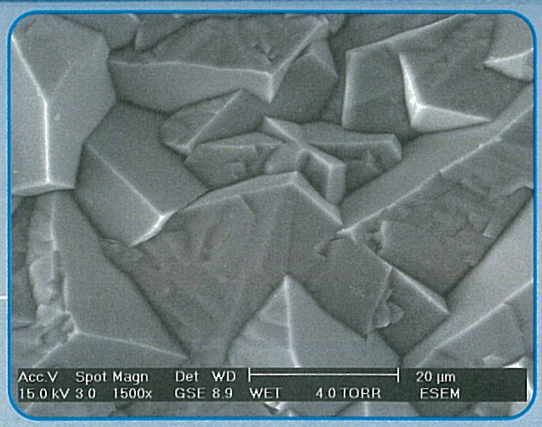
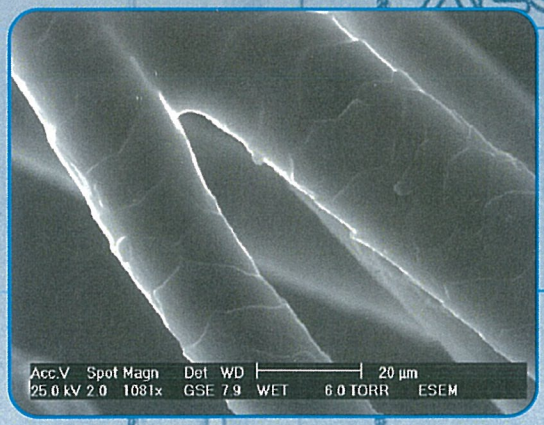
Environmental Scanning Electron Microscopy









An introduction to ESEM®

XL30 ESEM

ESEM®

The Highest Standard in Scanning Electron Microscopy

Philips
Electron
Optics

PHILIPS

ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY

An Introduction to ESEM®

Philips Electron Optics
Eindhoven, The Netherlands

2nd printing

Robert Johnson Associates
2111 Sheffield Drive
El Dorado Hills, Ca 95762

Contents

Preface

Typically, inquirers into Environmental Scanning Electron Microscopy (ESEM®) come from one of two groups. The first group are experts in their own fields but not in scanning electron microscopy (SEM). They simply have something very small that they would like to see. They may have been told that they cannot look at it in an SEM. They need to understand how the ESEM is similar to and different from other SEM's, before they can decide whether it will solve their problem. With this same understanding they are forearmed, if needed, to champion the ESEM against the prevailing wisdom of conventional SEM. The second group are experts in SEM. They need to reconcile the extraordinary performance claims of the ESEM with the fundamental principles of their science, before they will reexamine their beliefs about its capabilities and limitations. We will try here to address the needs of both groups.

Frequently, when addressing a group of microscopists, experienced and neophyte alike, we see among them quite visible expressions of what we have come to call the "Aha! Experience" — "Aha! I didn't know you could do that," or "Aha! That means I could ..." our slogan, "Seeing Things You've Never Seen Before®," comes directly from one customer's "Aha! Experience" during a demonstration. We have written this brief introduction to ESEM hoping to promote a broader understanding and appreciation of the ESEM's remarkable capabilities. If reading it brings you one "Aha!", then your time and ours has been well spent.

This is not the work of one person but of many. Special credit and thanks are due to Ralph Knowles, Philips/ElectroScan Vice President for Research and Development, and Tom Hardt and Trisha Rice, of the Applications Laboratory, for their technical advice and review; Deidre MacDonald for her unerring sense of aesthetics; and perhaps most importantly to Ed Griffith and Al Pick for their guidance (sometimes relentless) toward the goal that this work must serve, first and foremost, the needs of the reader. Of course these contributors cannot be held to account for the way I have used their good advice. All responsibility for remaining errors and omissions is ultimately mine.

ROBERT JOHNSON

As to science itself, it can only grow.
—GALILEO, *Dialogue* (1632)

Would to God your horizon may broaden every day! The people who bind themselves to systems are those who are unable to encompass the whole truth and try to catch it by the tail; a system is like the tail of the truth, but truth is like a lizard; it leaves its tail in your fingers and runs away knowing full well that it will grow a new one in a twinkling.
—IVAN TURGENEV TO LEO TOLSTOY (1856)

1

INTRODUCTION

1.1 WHAT IS AN ESEM?

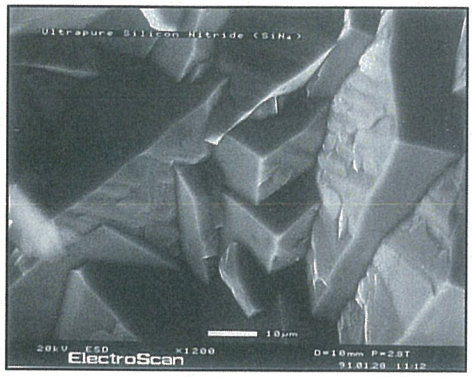
Scanning Electron Microscopes (SEM) began to appear commercially in the mid nineteen sixties. Because of their performance advantages over other types of microscopes, they quickly became an indispensable tool in a broad range of scientific and engineering applications. Although SEM manufacturers continued to refine the technology and made steady improvements in performance and usability, the SEM remained fundamentally unchanged for nearly twenty years. Throughout that time, the SEM's primary limitations, as a general imaging and analytical technique, were the restrictions it imposed on samples by requiring a high vacuum sample environment. Samples had to be clean, dry and electrically conductive. The vast body of technique developed for SEM sample preparation is a tribute to the ingenuity and tenacity of microscopists in the face of these high vacuum constraints.

The mid eighties saw the development of the Environmental SEM or ESEM® (usually pronounced "ee-sem"). Perhaps it would have been better named the Variable Environment SEM since its primary advantage lies in permitting the microscopist to vary the sample environment through a range of pressures, temperatures and gas compositions. The Environmental SEM retains all of the performance advantages of a conventional SEM, but removes the high vacuum constraint on the sample environment. Wet, oily, dirty, non-conductive samples may be examined in their natural state without modification or preparation. The ESEM offers high resolution secondary electron imaging in a gaseous environment of practically any composition, at pressures as high as 50 Torr, and temperatures as high as 1500°C.

The ESEM has opened to SEM investigation a whole host of applications that were previously impossible. Equally important, it has eliminated most of the sample preparation required for those applications that were already possible.

1.2 WHAT CAN IT DO?

The examples on the next page offer a glimpse of the dramatic new capabilities of the ESEM. The reader with a background in electron microscopy will quickly realize that none of these micrographs could have been taken with a conventional SEM.



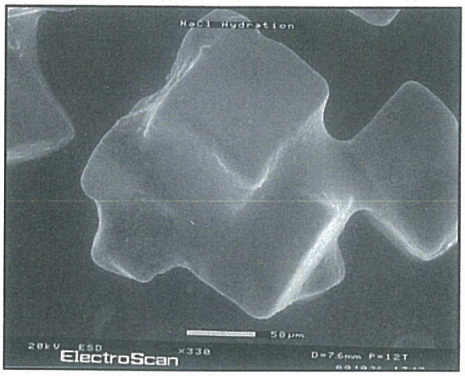
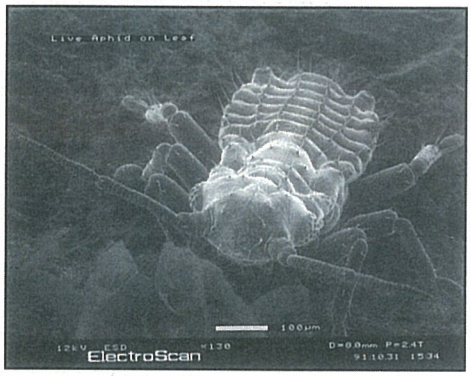
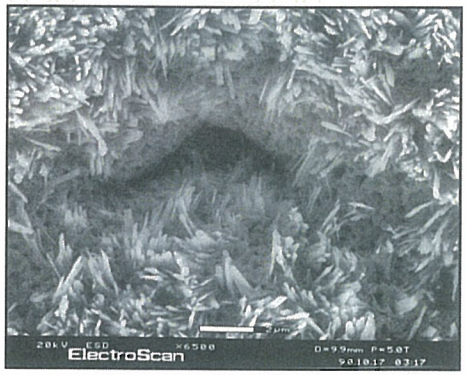
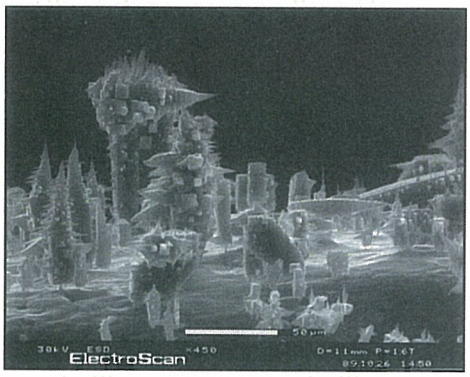


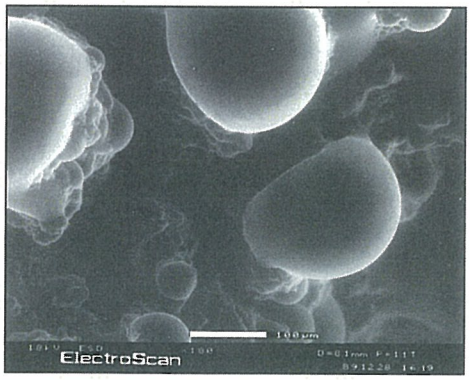
Figure -1-1. *Left - Uncoated ultrapure silicon nitride, an insulator. Note the lack of charging artifacts. Right - Crystals of table salt dissolving in water condensed from a water vapor environment.*





Left - Live green aphid on a rose leaf Right - Iron oxidizing in the chamber at 800°C.





Left - Potassium chloride crystallized from gas in the chamber at 600° C Right - Droplets of oil and water on an oil field core sample

We will return to a more thorough survey of ESEM applications later. For now, it may help to categorize the areas of application where the ESEM has significant advantages.

Nonconductive Gas ionization in the sample chamber eliminates the charging artifacts typically seen with nonconductive samples.

Contaminating The ESEM can image wet, dirty, oily, outgassing samples. The contaminants do not damage the instrument or degrade image quality

Hot The patented Environmental Secondary Detector is insensitive to heat. It can acquire electron images from samples as hot as 1500°C.

Light Emitting The detector is also insensitive to light. It can image incandescent, fluorescent and cathodoluminescent samples without interference. With an accessory light microscope the ESEM can provide simultaneous optical and electron images. Its viewport and chamber illuminator may be used during secondary electron image acquisition.

Delicate Delicate structures often do not survive the sample preparation required for conventional SEM's. The ESEM eliminates the need for conductive coatings, and most other sample preparation.

Hydrated Wet samples need not be dried before viewing in the ESEM. This is especially important for specimens that must remain hydrated in order to retain their structure. The ESEM can provide a saturated water vapor environment, keeping samples fully hydrated indefinitely.

Masked Coatings applied during sample preparation may mask valuable information. For example a gold coating may enhance surface detail but mask internal structure. The process of applying the gold may itself create artifact in the sample. The ESEM does not require samples to be coated.

X-ray The ESEM can acquire X-ray data from insulating samples at high accelerating voltages. This eliminates the potential for X-ray interference from conductive coatings and the need to analyze complicated L and M X-ray lines at low voltages.

Dynamic Much of specimen preparation for the conventional SEM is directed at "fixing" the sample, ensuring that it will not change during image acquisition. Eliminating the need for sample preparation, particularly the need for conductive coatings, opens a whole new realm of investigation in dynamic processes. Tension, compression, deformation, crack propagation, adhesion, heating, cooling, freezing, melting, hydration, dehydration, and sublimation, are but a few examples that come to mind. The ESEM can observe and record these processes directly, as they happen.

Interactive — A Lab Within a Lab The sample environment of the conventional SEM is, by definition, empty, a vacuum. The ESEM may be best understood as a microscopic experimental chamber — a lab within a lab — in which the sample environment can be a component of the experimental system. Interactions between the sample and its environment constitute yet another new universe of potential applications. Consider hydration studies in which samples are wetted and dried by water from the environment, crystal growth from the gaseous environment, corrosion, and etching.

As this listing demonstrates, it is difficult to neatly categorize all of the unique capabilities and applications of the ESEM. The simplest statement might be "All the things a conventional SEM cannot do." But neither is this truly adequate since it presumes a knowledge of the capabilities and limitations of conventional SEM's.

1.3 THIS PRIMER

In this brief primer we will try to provide a basic understanding of the physical principles and design considerations behind the ESEM. You need not have any previous background or experience in SEM. If you do have knowledge in this field you may find some sections too elementary — feel free to skip them. We hope that you will finish with a solid understanding of the ESEM's capabilities and limitations, and the differences between it and a conventional SEM.

This latter point has recently become somewhat confused. The ESEM is in fact unique. Patents protect the essential aspects of its technology. In response to the ESEM, a new class of microscopes, generally known as Low Vacuum SEM's, has appeared. These are essentially conventional SEM's that have been modified to permit limited low vacuum operation. They have neither the range nor the flexibility of the ESEM but do extend, somewhat, the utility of the conventional SEM. We will devote considerable attention to understanding their capabilities and limitations as well.

1.3.1 Terminology

From this point on we will use the following terminology:

SEM	All Scanning Electron Microscopes.
CSEM	Conventional High Vacuum SEM's
ESEM	The Environmental SEM
LV-CSEM	Low Vacuum adaptations of CSEM's.

The next chapter reviews the principles that apply to all SEM's and the limitations of conventional SEM's. Chapter 3 explores the unique principles and design of the ESEM. Chapter 4 examines low vacuum SEM's. The final chapter presents a selection of ESEM application examples.

2

SEM BASICS

SEM's enjoy a tremendous advantage over other microscopies in several fundamental measures of performance. Most notable are resolution — the ability to "see" very small features; depth-of-field — the extent to which features of different "heights" on the sample surface remain in focus; and microanalysis — the ability to analyze sample composition. In this chapter we will examine how an SEM forms an image and the principles that determine resolution, depth-of-field, and microanalytical capability. We will also look at the different signals available in the SEM, particularly as they relate to image resolution. We will conclude with a look at the limitations of conventional SEM's.

Conventional SEM's are a mature, well-understood technology. There are many excellent texts available that describe them in great detail and the reader is directed to them for additional information. Here we will limit our discussion to the rudimentary principles prerequisite to an appreciation of the ESEM. This chapter is intended primarily for readers with little or no knowledge of SEM's and may be skipped by others without penalty.

2.1 DESCRIPTION

All SEM's consist of an electron column, that creates a beam of electrons; a sample chamber, where the electron beam interacts with the sample; detectors, that monitor a variety of signals resulting from the beam-sample interaction; and a viewing system, that constructs an image from the signal.

An electron gun at the top of the column generates the electron beam. In the gun, an electrostatic field directs electrons, emitted from a very small region on the surface of an electrode, through a small spot called the crossover. The gun then accelerates the electrons down the column toward the sample with energies typically ranging from a few hundred to tens of thousands of electron volts. There are several types of electron guns — tungsten, LaB_6 (lanthanum hexaboride) and field emission. They use different electrode materials and physical principles but all share the common purpose of generating a directed electron beam having stable and sufficient current and the smallest possible size.

The electrons emerge from the gun as a divergent beam. A series of magnetic lenses and apertures in the column reconverges and focuses the beam into a demagnified image of the crossover. Near the bottom of the column a set of scan coils deflects the beam in a scanning pattern over the sample surface. The final lens focuses the beam into the smallest possible spot on the sample surface.

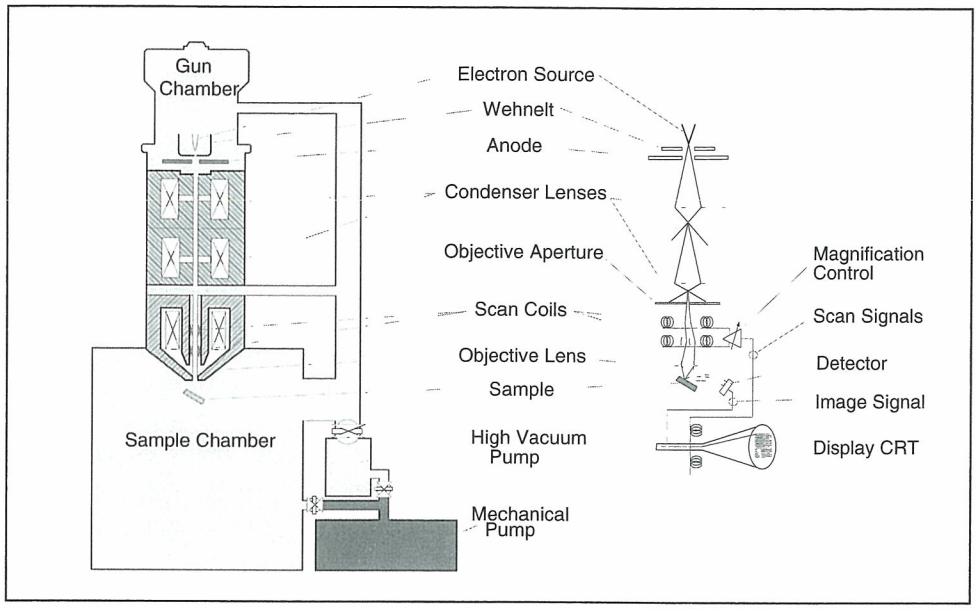


Figure 2-1. *A schematic representation of an SEM. The electron column accelerates and focuses a beam of electrons onto the sample surface. Interactions between the sample and the beam electrons cause a variety off signal emissions. The signals are detected and reconstructed into a virtual image displayed on a CRT.*

The beam exits from the column into the sample chamber. The chamber incorporates a stage for manipulating the sample, a door or airlock for inserting and removing the sample, and access ports for mounting various signal detectors and other accessories. As the beam electrons penetrate the sample, they give up energy, which is emitted from the sample in a variety of ways. Each emission mode is potentially a signal from which to create an image.

2.2 IMAGING PRINCIPLE

Unlike the light in an optical microscope, the electrons in an SEM never form a real image of the sample. Instead, the SEM constructs a virtual image from the signals emitted by the sample. It does this by scanning its electron beam line by line through a rectangular (raster) pattern on the sample surface. The scan pattern defines the area represented in the image. At any instant in time the beam illuminates only a single point in the pattern. As the beam moves from point to point, the signals it generates vary in strength, reflecting differences in the sample. The output signal is thus a serial data stream. Modern instruments include digital imaging capabilities that convert the analog data from the detector to a series of numeric values. These values are then manipulated as desired.

Originally all SEM's used a simple imaging device based upon a cathode ray tube or CRT. A CRT consists of a vacuum tube covered at one end, the viewing surface, with a light emitting phosphor. At the other end are an electron gun and a set of deflection coils. Similar to the SEM, the CRT gun forms a beam of electrons and accelerates it toward the phosphor. The deflection coils scan the beam in a raster pattern over the display surface. The phosphor converts the energy of the incident electrons into visible light. The intensity of the light depends on the current in the CRT electron beam. By synchronizing the CRT scan with the SEM scan and modulating the CRT beam current with the image signal, the system maps the signal point for point onto the viewing surface of the CRT, thus creating the image.

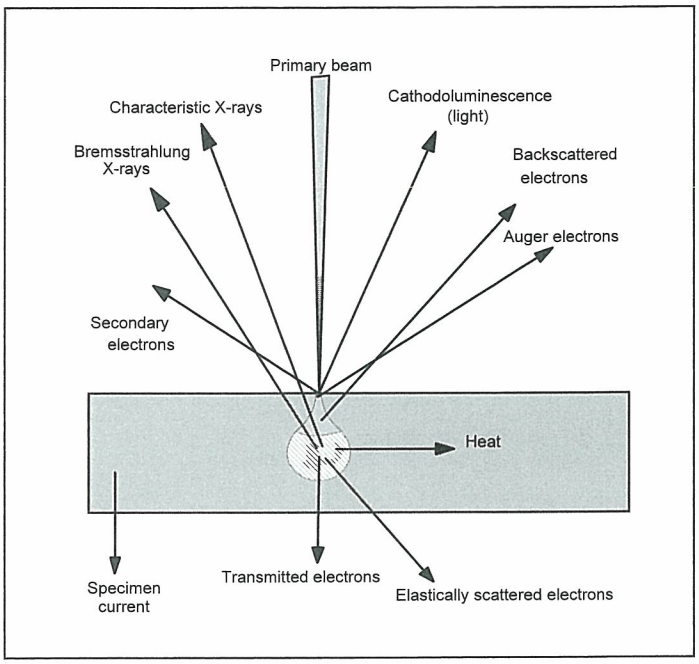


Figure 2-2. *The interactions of beam electrons and sample atoms generate a variety of signals. The most commonly used signals are secondary electrons, backscattered electrons, and characteristic X-rays.*

2.3 ELECTRON OPTICS

2.3.1 Lenses

Magnetic lenses in the electron column bend electron paths just as glass lenses bend light rays. A diverging cone of electrons emerges from each point in the gun crossover, passes through the lens field, and reconverges at a corresponding point in the image plane of the lens. Electrons from all points in the crossover thus pass through the lens to form an image of the crossover at the image plane of the lens. Since the purpose of the column is to project the smallest possible image of the crossover onto the sample surface, its lenses operate in a demagnifying mode. In this mode the image plane is always closer to the lens than the source is. As the cone of electrons converging to a point in the image passes beyond the image plane it begins to diverge again into another cone. In a demagnifying configuration, the divergence angle of the cone beyond the image plane is greater than the divergence angle of the original cone from the corresponding point in the crossover.

Lenses exhibit certain kinds of aberrations. Two of the most important are spherical aberration and chromatic aberration. Spherical aberrations result when paths away from the optical axis are bent more than paths close the axis. Chromatic aberrations result when paths of slower electrons are bent more strongly than paths of faster electrons. Because of these aberrations, all electron paths originating from a given point in the crossover do not converge perfectly on the same point in the image.

2.3.2 Apertures

Apertures are simply small holes, centered on the optical axis, through which the beam must pass. Located at an image plane, an aperture limits the size of the image. Located at a lens plane, an aperture defines the base of the cone of electrons passed from each point in the image, and, thus, the number of electrons transmitted. Here it operates more or less equally on all points in the image of the crossover, and limits total current in the beam. Equally important, an

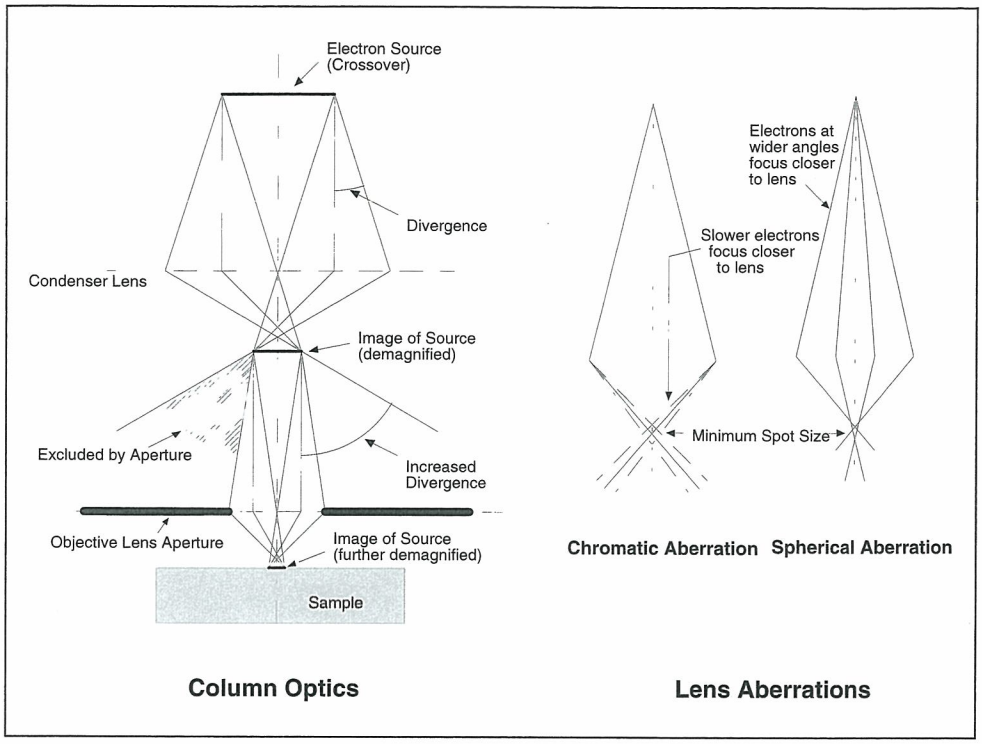


Figure 2-3. *The final aperture limits beam current and reduces the effects of lens aberrations. For any set of lens conditions, there is an aperture size that optimizes the trade off between beam current and spot size. Larger beam currents require larger apertures.*

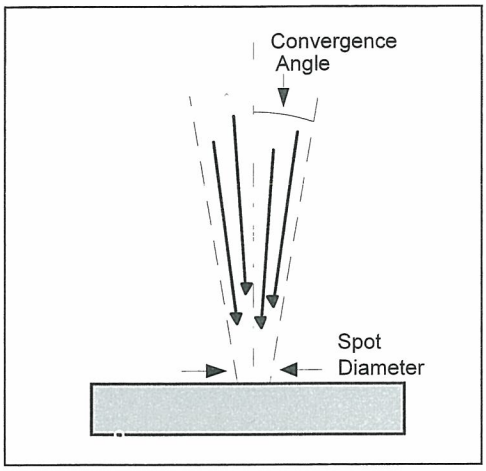
aperture in the lens plane excludes the electrons that are farthest off axis, reducing the adverse effects of lens aberrations. For any beam current there is an optimal aperture size that minimizes the detrimental effects of lens aberrations on spot size. As the beam passes from lens to lens in the column, apertures eliminate the more widely diverging electrons, sacrificing beam current for smaller spot size.

2.3.3 Beam Current

There is a fundamental relationship between beam current and spot size. An increase in one generally increases the other. Larger apertures and weaker lenses yield higher beam currents and larger spot sizes. Smaller apertures and stronger lenses yield smaller beam currents with smaller spot sizes. Some applications, for instance X-ray analysis, need higher current. High resolution imaging, on the other hand, requires the smallest possible spot size.

Beam current requirements ultimately impose a lower limit on spot size. The information in an SEM image consists of variations in signal intensity over time. At lower beam currents, random variations in the signal become increasingly significant. This noise may originate in the detection and amplification chain or, at very low currents, in statistical fluctuations of the beam current itself. As beam current and spot size decrease below some critical level, increasing noise overwhelms improving resolution.

For the purposes of this discussion, we must also distinguish between beam current and imaging current. We will call beam current the current that passes through the last aperture of the electron column. Imaging current is the current remaining in the spot at the sample surface. Imaging current is less than beam current when gas molecules in the sample environment scatter electrons out of the beam. In the high vacuum environment of a conventional SEM beam current and imaging current are essentially the same.



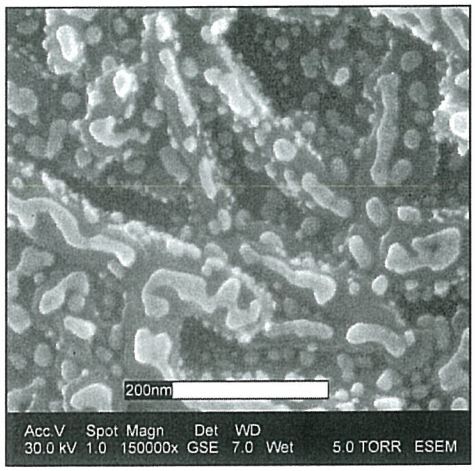


Figure 2-4. Resolution is fundamentally limited by the diameter of the spot formed by the electron beam on the sample surface. The convergence angle determines depth of field.

2.4 RESOLUTION

Resolution is a measure of the smallest feature a microscope can "see". It defines the limit beyond which the microscope cannot distinguish two very small adjacent points from a single point. Resolution is specified in linear units, typically Angstroms or nanometers. Just to keep things interesting, better resolution is called higher resolution, even though it is specified by a lower number. For example 10Å is higher (better) resolution than 20Å.

2.4.1 Spot Size

The size of the spot formed by the beam on the sample surface sets a fundamental limit on resolution. An SEM cannot resolve features smaller than the spot size. In general, low beam current, short working distance and high accelerating voltage yield the smallest spot. Other factors such as type of signal, beam penetration, and sample composition also affect resolution.

2.4.2 Volume of Interaction

Image signals are not generated only at the sample surface. The beam electrons penetrate some distance into the sample and can interact one or more times anywhere along their paths. The region within the sample from which signals originate and subsequently escape to be detected is called the volume of interaction. Signal type, sample composition, and accelerating voltage all impact resolution through their effects on the size and shape of this volume. Figure 2-5 is a schematic representation of the types of signals generated and their specific volumes of interaction. In most cases the volume of interaction is significantly larger than the spot size and thus becomes the actual limit on resolution.

Accelerating Voltage

Accelerating voltage determines the amount of energy carried by the primary (beam) electrons. It affects the size and shape of the volume of interaction in several ways. Higher energy electrons can penetrate more deeply into the sample. Likewise, they can generate higher energy signals that can escape from deeper within the sample. Primary electron energy is also a factor in determining the probability that any particular type of interaction will occur. In all of these instances higher energy tends to reduce image resolution by enlarging the volume of interaction. Higher accelerating voltage can also improve resolution by reducing lens aberrations in the electron column, resulting in smaller spot sizes. Which influence predominates depends upon the specific sample, operating conditions, and signal type.

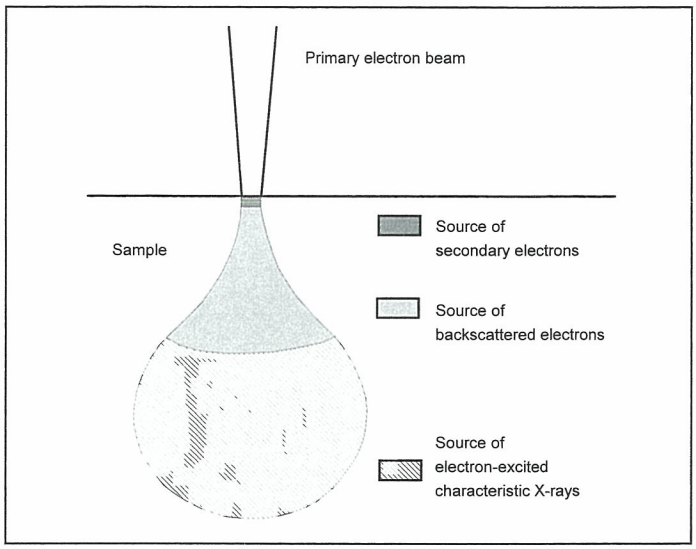


Figure 2-5. *Each type of signal originates within a specific volume of interaction. The size of the volume limits the spatial resolution of the signal. Secondary electrons have the smallest volume, followed by backscattered electrons, and X-rays.*

Sample Composition

Sample composition affects both the depth and shape of the volume of interaction. Denser samples reduce beam penetration and reduce the distance a signal can travel before it is reabsorbed. The resulting volume of interaction tends to be shallower and more hemispherically shaped.

2.4.3 Signal Type

To this point we have discussed a general volume of interaction from which all signals originate. We can divide that volume into specific regions associated with each signal type.

Secondary Electrons

Secondary electrons (SE) are sample atom electrons that have been ejected by interactions with the primary electrons of the beam. They generally have very low energy (by convention less than fifty electron volts). Because of their low energy they can escape only from a very shallow region at the sample surface. As a result they offer the best imaging resolution. Contrast in a secondary electron image comes primarily from sample topography. More of the volume of interaction is close to the sample surface, and therefore more secondary electrons can escape, for a point at the top of a peak than for a point at the bottom of a valley. Peaks are bright. Valleys are dark. This makes the interpretation of secondary images very intuitive. They look just like the corresponding visual image would look.

Backscattered Electrons

Backscattered electrons (BSE) are primarily beam electrons that have been scattered back out of the sample by elastic collisions with the nuclei of sample atoms. They have high energy, ranging (by convention) from fifty electron volts up to the accelerating voltage of the beam. Their higher energy results in a larger specific volume of interaction and degrades the resolution of backscattered electron images. Contrast in backscattered images comes primarily from point to point differences in the average atomic number of the sample. High atomic number nuclei backscatter more electrons and create bright areas in the image. Backscattered images are not as easy to interpret but, properly interpreted, can provide important information about sample composition.

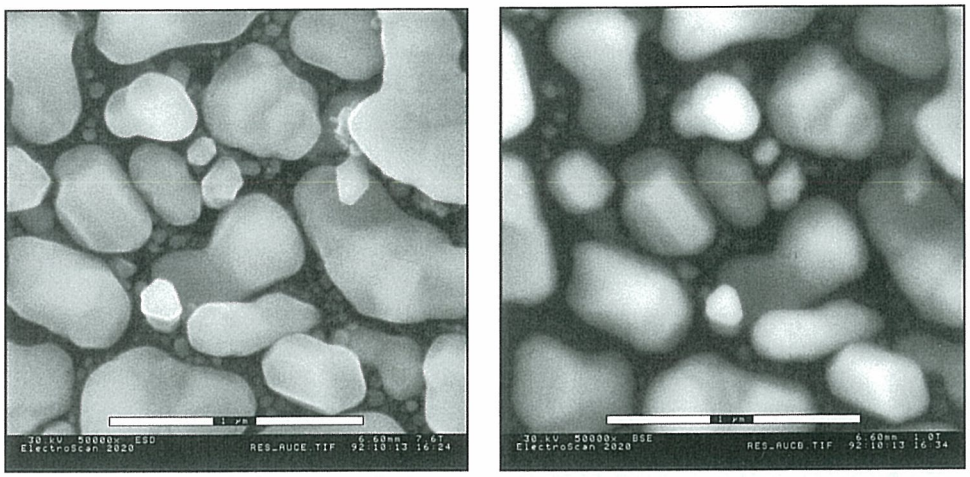


Figure 2-6. *Secondary electron (left) and backscattered electron (right) images of gold on carbon. Gold is a heavy element, providing great atomic number contrast with the carbon background. This type of sample tends to minimize the differences in SE and BSE resolution.*

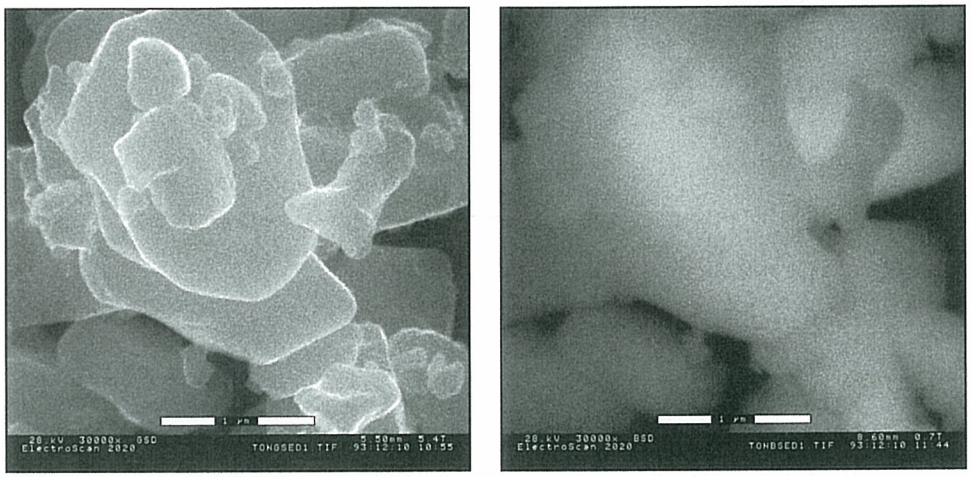


Figure 2-7. *Secondary electron (left) and backscattered electron (right) images of toner particles. A light element matrix, such as this, emphasizes the resolution differences.*

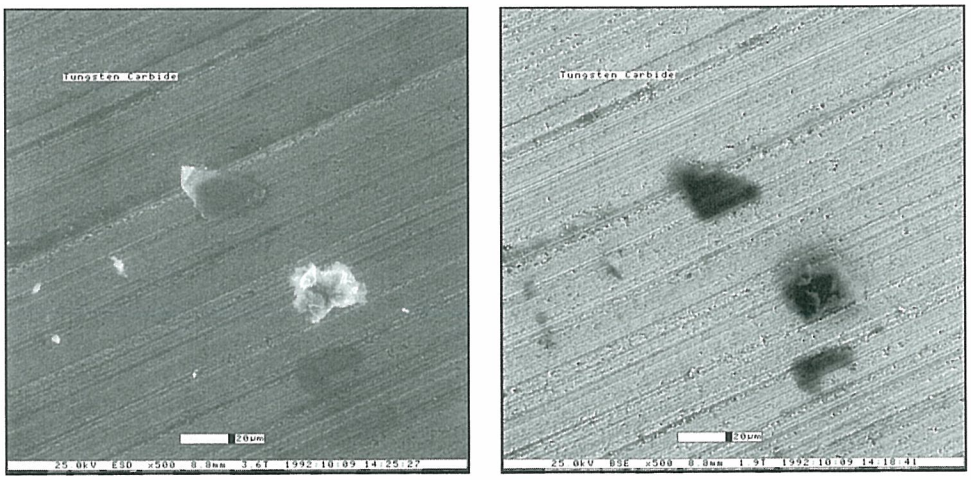
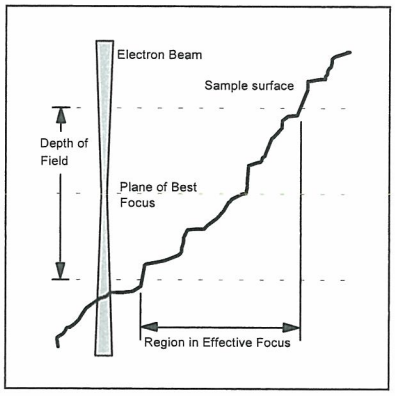


Figure 2-8. *This sample shows light element particles on a tungsten carbide substrate. The SE image (left) shows mostly topographic contrast. Note the surface detail of the particles. Contrast in the BSE image (right) is due primarily to differences in atomic number.*



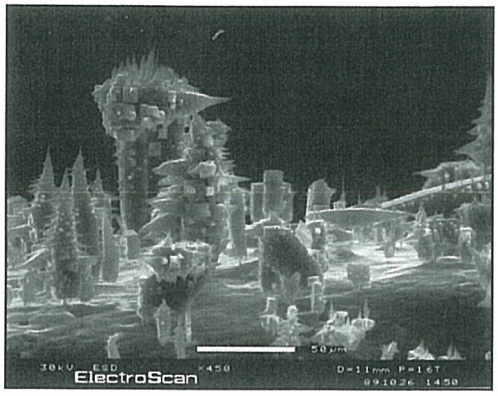


Figure 2-9. *The SEM largely decouples depth of field from magnification. Often the dramatic impact of an SEM micrograph is due more to depth of field than resolution.*

2.5 DEPTH OF FIELD

Compared to light microscopes, SEM's offer a great improvement in depth of field. Depth of field characterizes the extent to which image resolution degrades with distance above or below the plane of best focus. With greater depth of field a microscope can better image three dimensional samples. Although the SEM is best known for its excellent resolution, some of the most dramatic images actually result from its tremendous depth of field.

In a light microscope, the divergence angle of the cone of light collected by the objective lens from each point in the sample determines depth of field. For higher magnifications, this angle is greater and the depth of field shallower. Thus there is a direct trade-off between magnification and depth of field.

The SEM largely decouples magnification from depth of field. The size of the beam scan, relative to the display scan, determines magnification. The convergence angle of the primary beam determines the change in spot size with distance above or below the plane of best focus. Although the convergence angle and spot size are a function of working distance (the distance from the final lens to the sample surface), in all cases the angles are much smaller, and depth of field much greater, than for optical microscopies.

2.6 MICROANALYSIS

Characteristic X-rays

X-rays result when an energetic electron, usually from the beam, scatters an inner shell electron from a sample atom. When a higher energy, outer shell electron of the same atom, fills the vacancy, it releases energy as an X-ray photon. Because the energy differences between shells are well defined and specific to each element, the energy of the X-ray is characteristic of the emitting atom.

An X-ray spectrometer collects the characteristic X-rays. The spectrometer counts and sorts the X-rays, usually on the basis of energy (Energy Dispersive Spectrometry — EDS). The resulting spectrum plots number of X-rays, on the vertical axis, versus energy, on the horizontal axis. Peaks on the spectrum correspond to elements present in the sample. The energy level of the peak indicates which element. The number of counts in the peak indicates something about the element's concentration.

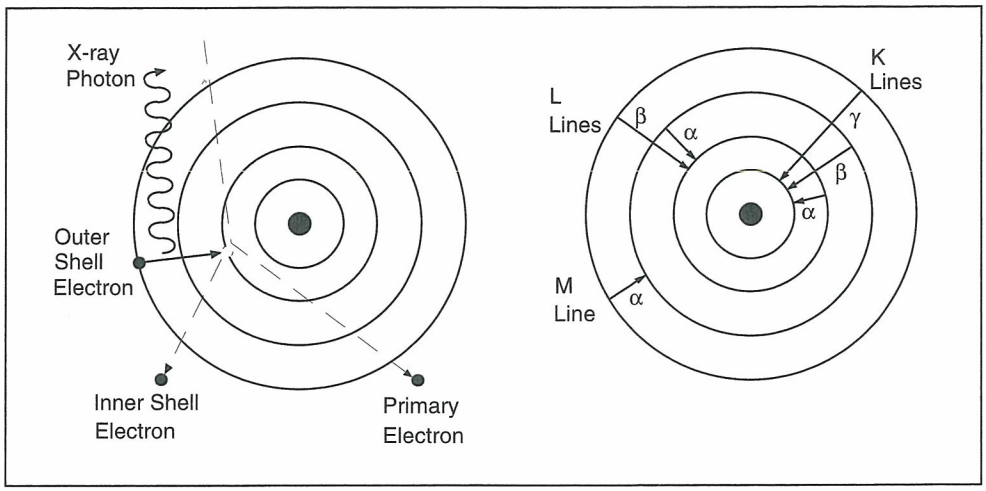


Figure 2-10. *Characteristic X-rays are generated when an inner shell vacancy is filled by a higher energy outer shell electron. The energy of the X-ray equals the difference between the electron energies and is characteristic of the emitting element.*

X-ray Lines

Most elements have multiple energy shells and may emit X-rays of several different energies. The various emission "lines" are named for the shell of the initial vacancy — K, L, M, etc. A Greek letter subscript indicates the shell of the electron that fills the vacancy. Thus a K_α line results from a vacancy in the K shell filled by an electron from the next higher shell, L in this case. The nomenclature and the peak structures can become very complex, particularly for high atomic number elements with a multitude of shell and sub-shell energy levels.

Some general rules apply to the various spectral lines. 1) For a given element, lower line series have higher energy — gold K lines have higher energy than gold L lines. 2) Within a line series, higher atomic number elements emit higher energy X-rays — oxygen K lines are higher energy than carbon K lines. 3) Lower line series have simpler structure than higher line series. K lines are simple and distinct. L and M lines, become progressively more complex and overlapping.

X-ray Maps

For a number of reasons, the X-ray signal provides a much poorer image than electron signals. One reason is the distance X-rays can travel through the sample, generating a large volume of interaction and poor spatial resolution (see Figure 2-5). Another reason is the inherent X-ray background signal (Bremsstrahlung) that, combined with intrinsically low characteristic X-ray signal levels, yields a poor signal to noise ratio.

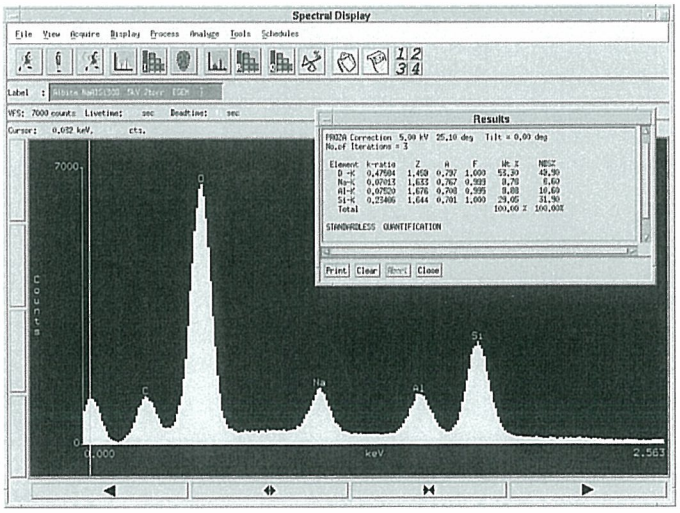


Figure 2-11. *An energy dispersive X-ray spectrometer displays peaks at energies characteristic of elements present in the sample.*

Figure 2-12. X-ray maps plot the location and intensity of characteristic X-ray emissions over the field of view. These images show (clockwise from upper left) a secondary electron image, an oxygen map, a magnesium map, and an aluminum map.

X-ray images are generally referred to as maps, rather than images. Setting the spectrometer to register a "dot" on the imaging device when it detects an X-ray of the appropriate energy creates a "dot map", showing the spatial distribution of the corresponding element. Given sufficiently long collection times, the digital imaging capabilities of current generation EDS systems can generate gray level maps that show relative X-ray intensity at each point (Figure 2-12). Even a gray level map does not approach the quality of an electron image.

X-ray Analysis

Because of its poor spatial resolution, the X-ray signal is more often used for analysis than imaging. A qualitative analysis seeks to determine the presence or absence of elements in the sample based on the presence or absence of their characteristic peaks in the spectrum. A quantitative analysis tries to derive the relative abundance of the elements in the sample from a comparison of their corresponding peak intensities, to each other, or to standards. The many interactions that may occur between characteristic X-rays and sample atoms make quantitative analysis very complex.

2.7 WHY AN ESEM ? — SEM LIMITATIONS

Although conventional SEM's have superior resolution, depth of field, and microanalytic capabilities, they also have a number of limitations. Almost all of these limitations derive from the high vacuum a CSEM must maintain in its sample chamber.

2.7.1 SEM Vacuum Constraints

That CSEM's developed as high vacuum systems is probably due more to the historical context of their development than to strict technical requirements. The column required a high vacuum in order to generate and focus the electron beam. The sample chamber required a high vacuum to permit the use of available secondary electron detectors. The simplest design, then, was to allow the chamber and the column to share a common high vacuum environment. At the time, the penalties paid for this approach must have seemed small compared to the performance benefits.

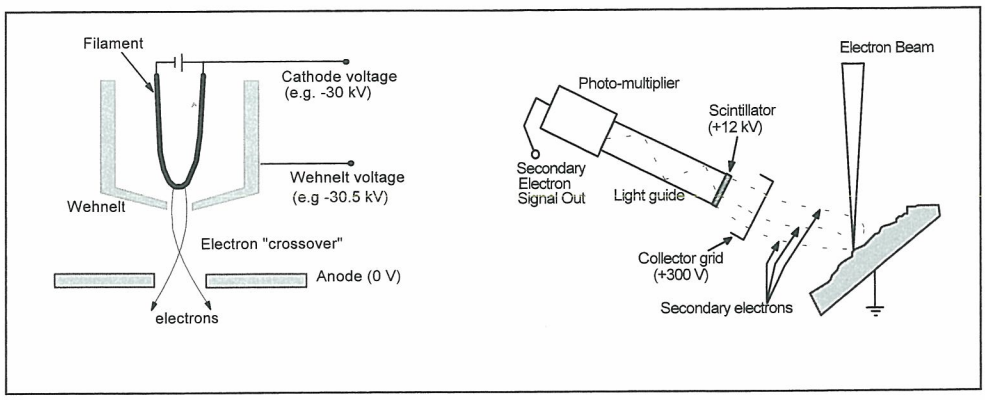


Figure 2-13. The high voltages used in the electron gun and Everhart-Thornley secondary electron detector require a high vacuum environment.

Gun Chamber and Column

All electron guns, regardless of type, are very sensitive to vacuum levels. Gas in the gun chamber can interfere with electron emission and degrade or destroy the electron source, be it tungsten, LaB_6 or field emission. Moreover, the gun uses very high voltages to accelerate the electrons down the column. The fields generated by these voltages are strong enough to ionize any gas present, providing a path for electrical discharge or "arcing".

Gas in the column can also interfere with the formation and transmission of the beam. Since the focal lengths of the magnetic lenses are relatively long, the beam electrons must travel a considerable distance (typically tens of centimeters) from the gun to the sample. Gas molecules along the beam path can scatter the electrons and degrade column performance.

ET Detector

The secondary electron detector used in most conventional SEM's is known as an Everhart-Thornley (ET) detector, named for its inventors. Like other secondary electron detectors, it uses a positive bias of a few hundred volts to attract the low energy secondary electrons and increase its collection efficiency. The detector field has little effect on higher energy backscattered electrons. Having entered the detector through the collector grid, secondary electrons are immediately accelerated by a higher voltage field (ten to twelve thousand volts) toward a scintillator. The scintillator converts the electron signal to light, which then passes through a light pipe to a photomultiplier tube. The photomultiplier tube amplifies the light signal and converts it back to an electronic signal. An electronic amplifier further amplifies and conditions the signal before passing it along to the imaging system. Because of its exposed high voltage elements, an ET detector can only function in a high vacuum environment. In a gas environment, it too will arc, often damaging or destroying itself in the process.

2.7.2 Sample Constraints

What constraints does the high vacuum requirement impose on samples? In the simplest terms, CSEM's require that samples be vacuum tolerant, vacuum friendly and electrically conductive.

Vacuum Tolerant

Vacuum tolerant means that the sample is not changed by the high vacuum environment of the sample chamber. A piece of metal is, generally, vacuum tolerant. A volatile coating on that same piece of metal is not. A delicate biological structure, perhaps supported by internal hydrostatic forces, is not. Much of specimen preparation for the CSEM involves the substitution of non-volatile materials for volatile sample components. Accomplishing this without altering the sample is difficult at best. CSEM sample preparation and analysis

has been called "the art of creative artifact." The science lies in correctly interpreting the observed artifact.

Vacuum Friendly

Vacuum friendly is really the opposite perspective on vacuum tolerant. Vacuum friendly describes the impact of the sample on the instrument. Will the sample degrade the vacuum enough to damage the detector or electron gun? Will it leave deposits on the apertures of the electron column, degrading imaging performance? Will it leave sufficient contamination on the walls of the sample chamber to interfere with subsequent observations?

Electrically Conductive

The connection between electrical conductivity and sample chamber vacuum requirements is less obvious. The electron beam deposits considerable charge in the sample. In conductive materials, the charge flows through the sample stage to ground. In insulating materials, the charge accumulates, causing local variations in secondary electron emissions and, in extreme cases, deflecting the electron beam itself. All of these effects are classified as charging artifacts.

Techniques for eliminating charging artifacts on nonconductive samples fall generally into two categories: conductive coatings, and low voltage charge balancing. Applying a thin conductive coating to the sample provides a path to ground and dissipates the local fields caused by accumulating charge. A heavy element coating, such as gold, may also improve signal strength and apparent resolution. As with any sample preparation, coating raises the issue of preparation artifacts. Does the coating process itself significantly alter the sample? Moreover, an image of a gold coated sample is an image of the coating not the sample. Are they the same?

Coatings may interfere in other ways. For example, a gold coating renders invisible the gold particles sometimes used as labels. In microanalysis, sample X-rays may be absorbed by the coating or obscured by coating X-rays. Gold absorbs X-rays very efficiently and emits interfering X-rays at several energies. Even carbon, a light element coating, can cause unacceptable interference.

Low voltage charge balancing works by balancing the charge deposited in the sample by the electron beam against the charge emitted from the sample as various signals. The balance is a function of accelerating voltage, sample composition, and local topography. Charge balancing generally requires accelerating voltages between a few hundred and two thousand volts, exacting a penalty in spot size and, potentially, in resolution. Furthermore, since the balance is specific to local composition and topography, it may be difficult to achieve simultaneously over the entire field of view. Finally, low voltages complicate X-ray analysis by requiring the use of more complex L and M lines.

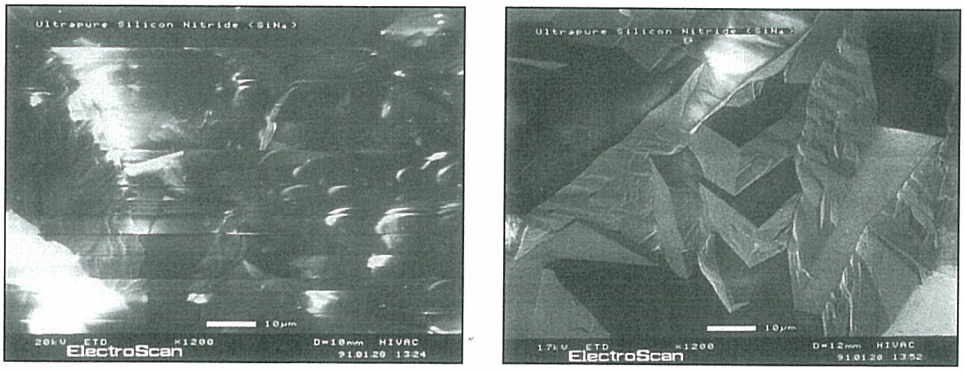


Figure 2-14. *Left - In high vacuum, at 20 kV, charging artifacts are apparent on this insulating sample. Right - Even low accelerating voltage (1.7 kV) may not eliminate charging uniformly over the entire field of view.*

2.8 SUMMARY

SEM's offer superior performance compared to light microscopes, particularly in resolution, depth of field, and microanalysis. An SEM can form an image from a variety of signals. Of the most commonly used signals, secondary electrons offer the best resolution and carry information about surface topography. X-rays carry the best information about sample composition but have poor spatial resolution. Backscattered electrons occupy the middle ground offering a medium resolution image carrying significant but non-specific compositional information.

Though SEM's offer superior performance, they are limited by their high vacuum requirements to samples that are vacuum tolerant, vacuum friendly and electrically conductive. Certainly the number of applications that do not meet these criteria must far exceed the number that do. In some cases, sample preparations can extend the conventional SEM's application. Even when successful, these techniques are expensive and time consuming. More importantly, they unavoidably call into question the integrity of the information derived from the modified sample. What does it look like in its natural state?

3

THE ESEM

The previous chapter ended with a question, "What does it look like in its natural state?" It was exactly this question that led to the development of the Environmental Scanning Electron Microscope. Researchers in Australia wanted to look at wool in its natural state — wet, oily and dirty — definitely vacuum intolerant, very vacuum unfriendly and highly non-conductive. They realized that the solution lay in eliminating the high vacuum requirement in the sample chamber. To do this they had to cross two technical hurdles. First they had to separate the vacuum environment of the electron column from the environment of the sample chamber. Second, they needed a secondary electron detector that could function in this non-vacuum sample environment. Their solutions to these problems are the keys to the development of the Environmental SEM.

3.1 VACUUM SYSTEM

All SEM's require high vacuum conditions in the electron gun, where high voltages are used to generate and accelerate the electron beam. High vacuum is also desirable throughout the column, where gas molecules can scatter electrons and degrade the beam. In the ESEM, multiple Pressure Limiting Apertures (PLA's) separate the sample chamber from the column. The column remains at high vacuum while the chamber may sustain pressures as high as 50 Torr.

The balance of gas flows into and out of the ESEM sample chamber determines its pressure. Gas flows out of the sample chamber to the column through the pressure limiting apertures, at a rate determined by each aperture's size and the pressure differential across it. Gas flows into the chamber from a selected source through an automatic metering valve controlled by the operator. Changing the inflow rate changes the vacuum level in the chamber. The environmental gas admitted to the chamber may be inert or may comprise one of the reactants in the experimental system. The choice of gases is limited primarily by practical considerations such as toxicity, flammability, and chemical reactivity with components of the chamber and vacuum system.

Prior to the ESEM some work was done using a single PLA to separate the sample chamber from the column, but conflicting optical and vacuum requirements — an aperture large enough to pass the beam but small enough to limit gas flow — permitted only limited benefits.

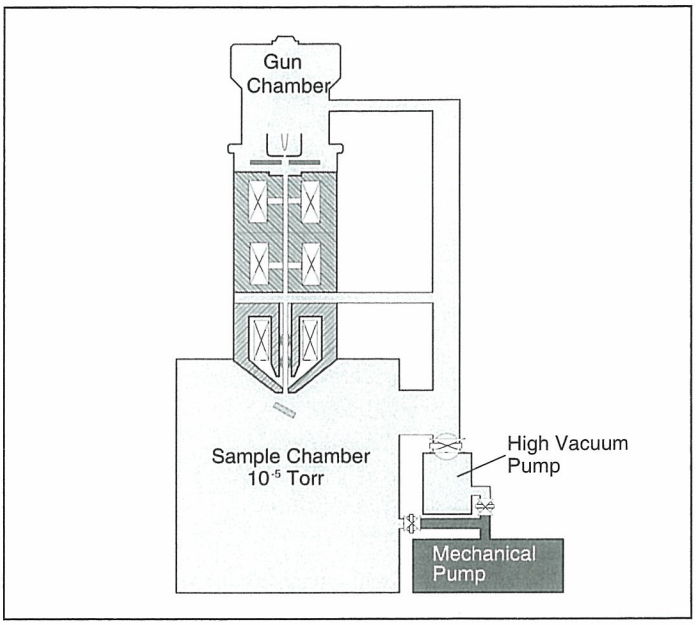


Figure 3-1. *In a CSEM the column and specimen chamber share the same vacuum.*

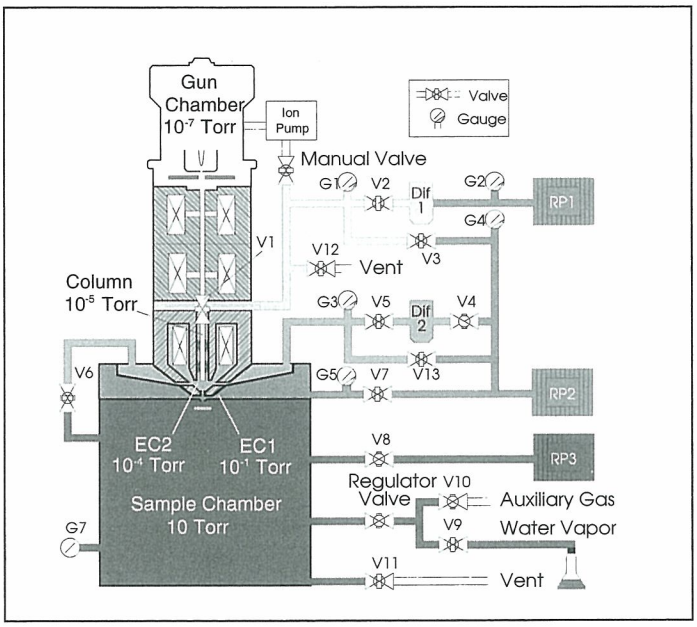


Figure 3-2. *In the ESEM the vacuum system is divided into as many as five stages of increasing vacuum, separated by pressure limiting apertures. In this schematic the stages are the sample chamber, first environmental chamber (EC1), second environmental chamber (EC2), column, and gun chamber.*

3.1.1 Multiple Pressure Limiting Apertures

The essential breakthrough in the design of the ESEM vacuum system was the integration of two closely spaced pressure limiting apertures into the final lens of the electron column. The regions below, between, and above the PLA's are separately pumped 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 gun. Depending on the particular configuration, additional pumping stages may be added to further improve vacuum in the gun. By using multiple apertures, the designers were able to decrease the pressure differential across each aperture and use larger aperture diameters, while still achieving a large total pressure difference between the column and the sample chamber. By locating the apertures close together at the bottom of the column they reduced the distance the beam has to

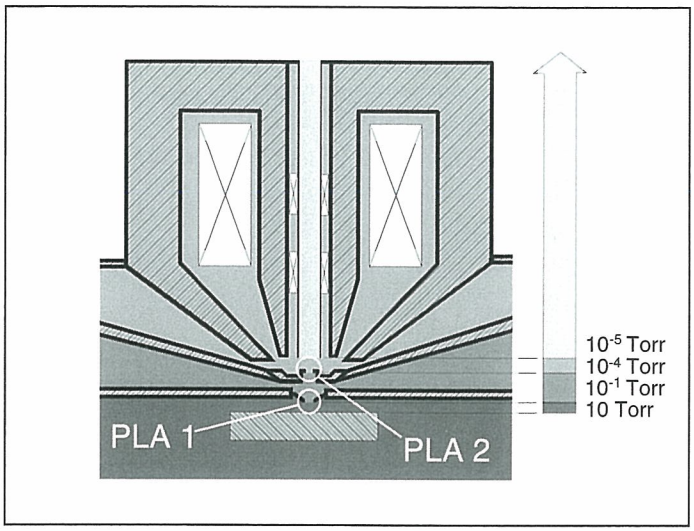


Figure 3-3. *Two Pressure Limiting Apertures (PLA's) integrated into the final lens assembly permit vacuums as low as 50 Torr in the sample chamber while maintaining high vacuum conditions in the gun. Keeping the apertures close together at the bottom of the column minimizes the effects of electron scattering.*

travel through the higher pressure stages. This type of vacuum system is protected by multiple patents and is available only in the ESEM.

3.1.2 Beam-Gas Interactions

If resolution in an SEM depends on its ability to focus the beam electrons into the smallest possible spot on the sample surface, how can the ESEM maintain its performance in a gaseous environment? Does the gas not scatter the primary electrons and degrade resolution? Yes, and no. Yes, the gas scatters electrons. No, it does not necessarily impact resolution. In order to understand the effects of the gas on the beam we must look more closely at electron scattering.

Although scattering may occur anywhere along the beam path from gun to sample, apertures in the column prevent most electrons scattered there from ever reaching the sample. Most scattering that could affect resolution occurs between the final pressure limiting aperture at the bottom of the column and the sample surface, hence the need to reduce this distance to a minimum.

It is of the utmost importance to understand that scattering is a discrete process, not a continuous one. Each individual electron is deflected only when it passes within a certain critical distance of a gas molecule. Otherwise, it continues on its original trajectory. Thus each electron has a finite, integer number of collisions before it reaches the sample surface. There is a statistical distribution that describes this kind of process, called a Poisson distribution. According to this distribution, the fraction of electrons that falls into each number-of-collisions category depends only on the average number of collisions for all electrons. Most importantly, even when the average number of collisions per electron is large, some small fraction of electrons still falls into the zero-collisions category.

The average number of collisions (m) provides a basis for defining three different scattering regimes. For the Minimal Scattering Regime, the average ranges from 0 to 0.05. At the upper limit (m = 0.05) 95% of the electrons in the beam have no collisions. Conventional SEM's operate in the lower portion of this range ($m \rightarrow 0$) where scattering effects on the beam are insignificant.

At the other end of the spectrum is the Complete Scattering Regime. Here the average number of scattering events per electron is greater than 3 and 95% or more of the electrons are scattered at least once. In this range the beam is generally broadened and not useful for SEM imaging.

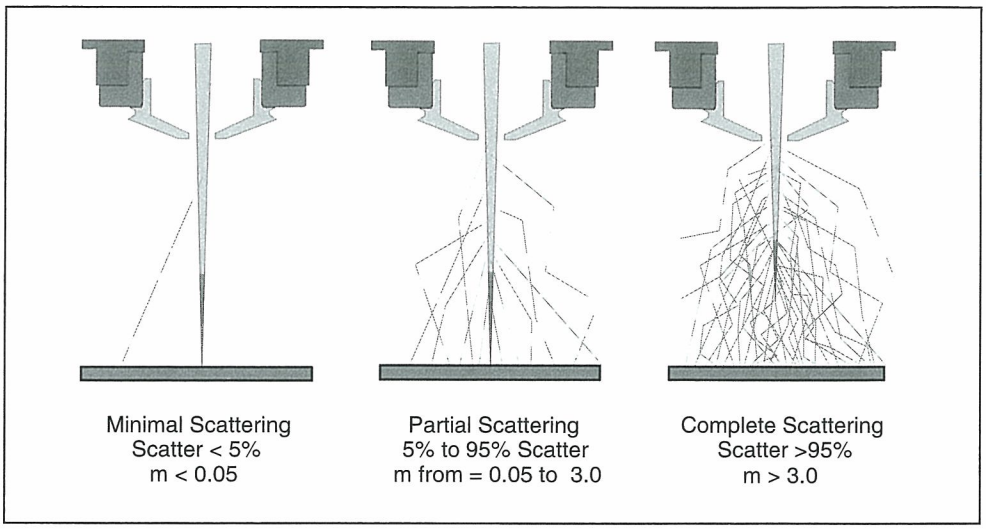


Figure 3-4. *It is useful to define three scattering regimes based on the average number of scattering events per electron, m. Conventional SEM's operate in the Minimal Scattering Regime. ESEM's and LV-CSEM's operate in the Partial Scattering Regime. The Complete Scattering Regime is not used for SEM imaging.*

Between Minimal Scattering and Complete Scattering is the Partial Scattering Regime. Here the average number of scattering events ranges from 0.05 to 3. Over this range, 95% to 5% (respectively) of electrons pass without scattering. This fact carries profound implications for imaging in the ESEM.

Aside: A Little Statistics (Very Little)

In a Poisson Process, events occur randomly over a continuum of time or space. The scattering of electrons by gas molecules is such a process. In a Poisson Distribution, the probability that any number of events will occur within a specified interval is a function only of the mean of the distribution. For electron scattering, the parameter of interest is the number of collisions each electron has with gas molecules along its path. The interval is the distance the electron travels through the gas. The mean of the distribution is the average number of collisions per electron, for all electrons.

The Poisson Distribution is described mathematically as:

$$P(x) = m^x e^{-m}/x!$$

where: $P(x)$ is the probability an electron will scatter x times
m is the average number of scattering events per electron
e is the base of natural logarithms, 2.71828...

For $x=0$, the probability that an electron will not scatter at all, the equation reduces to:

$$P(0) = e^{-m}$$

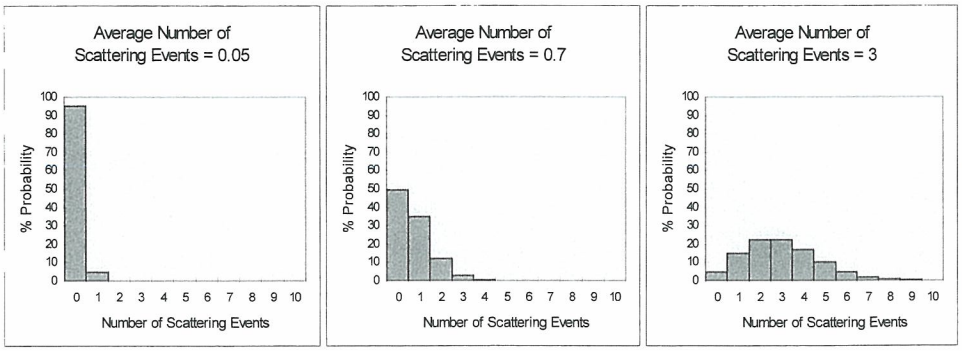


Figure 3-5. *A Poisson distribution is determined entirely by its mean. At the lower limit of the Partial Scattering Regime, 95% of electrons do not scatter. At the upper limit, only 5% do not scatter.*

3.1.3 Imaging Resolution

It seems reasonable to expect the electron beam to broaden gradually, but maintain its Gaussian profile, with increasing gas pressure. This, in fact, does not happen. Instead, the spot loses current without broadening, until it eventually disappears below the background.

Think of the beam as divided into two components, scattered and unscattered. The unscattered component remains well focused in the original spot on the sample surface. The scattered component, called the beam "skirt", falls in some broader distribution. The overall intensity profile of the beam is the sum of the two component profiles. The intensity of the skirt relative to the intensity of the spot determines the degree to which the skirt interferes with imaging.

Limited experimental data suggest the following relationship for the skirt half radius ($r_{1/2}$), the radius encompassing half of the scattered electrons:

$$r_{1/2} = 0.0039\, d + 1.326\, d(pd)^{1.38}$$

For typical ESEM conditions (d = 0.002 m, p = 7.5 Torr) the skirt half radius is about 16 micrometers. This is tremendously larger than the spot half radius of a few nanometers. Even at the upper limit of the partial scattering regime, the 5% of electrons not scattered are concentrated in an area many orders of magnitude smaller than the area of the skirt. As a result, the skirt electrons contribute only a very low level background signal that is easily discarded. As long as current sufficient to form an image remains in the spot, image resolution is unaffected.

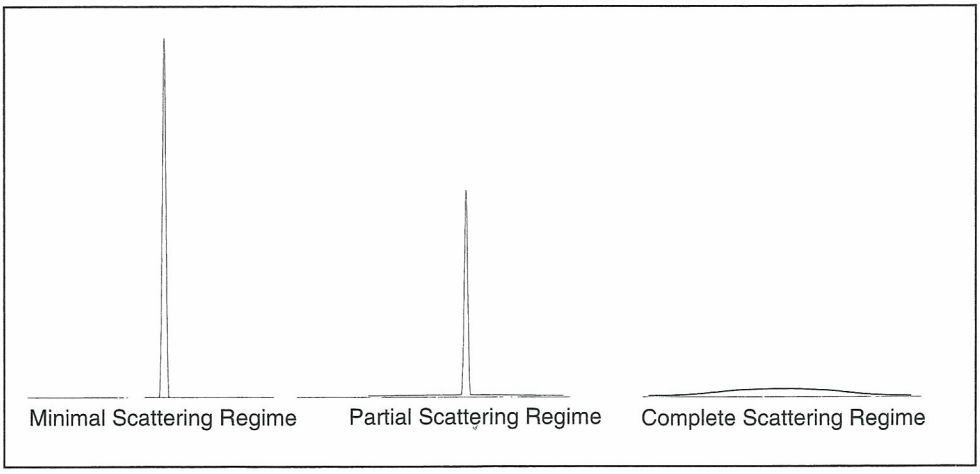


Figure 3-6. *The shape of the beam intensity profile depends on the scattering regime. As scattering increases the beam loses current to a very broadly dispersed "skirt". The unscattered component loses intensity but does not broaden, remaining within the original spot on the sample surface.*

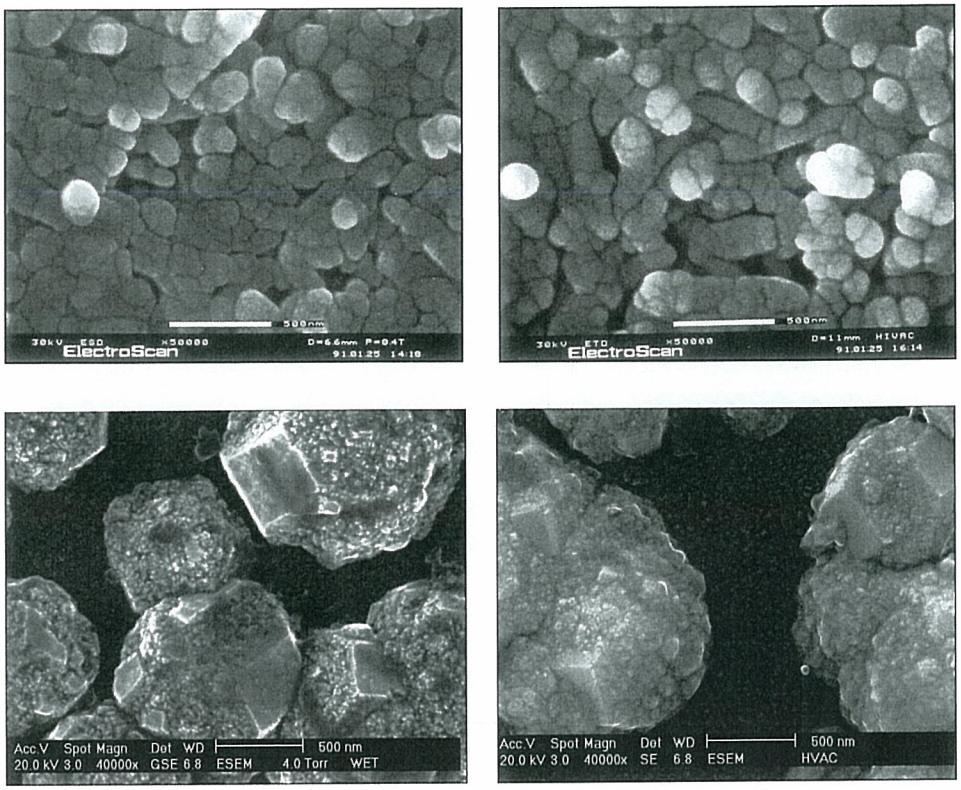


Figure 3-7. *As these micrographs demonstrate, there is no inherent loss of resolution in a gaseous environment. Upper Left - Magnetic tape at 50,000 X in 8.4 Torr of watervapor. Upper right - Magnetic tape at 50,000 X in high vacuum. Lower GSE image of diamond film in WET mode. Compare with SE image of same specimen in High Vac mode.*

3.1.4 Imaging Current

Imaging in a gaseous environment is thus limited by the useful current remaining in the unscattered beam spot, not by beam spreading. How does the statistical parameter m, the average number of scattering events per electron, relate to the operational parameters the microscopist can control, such as pressure and working distance? What is the ESEM's useful operational range?

Intuitively, m should depend on the number of gas molecules per unit volume (n), the effective size of the molecules (s) and the distance the electron travels through the gas (d in meters, also called Beam Gas Path Length or BGPL).

For known temperature (T in °K) and pressure (p in Torr), the ideal gas law gives n as:

$$n = 9.655 \text{ X } 1024\ p/T$$

In the energy range of beam electrons, the angular deflection and energy loss for each scattering event are relatively small. Therefore, for m less than three, the path length through the gas, d, very nearly equals the straight line distance from the final PLA to the sample surface. Working distance is usually defined as the distance from the bottom of the final lens to the sample surface. In the ESEM the final PLA extends below the bottom of the final lens so the path length is less than the working distance by some fixed amount.

The effective size, s, of a molecule is called its scattering cross section. A detailed discussion of the determination of scattering cross sections is beyond the scope of this work. Both theoretical derivations and experimental

measurements are available. It is sufficient here to note that scattering cross section is specific to each type of gas molecule, and that it varies inversely with the energy of the beam electron (V) — higher energy electrons are less likely to scatter.

It can be shown that, in the Partial Scattering Regime, the average number of collisions per electron is given by:

$$m = s\ n\ d$$

Combining this with the previous equations for n and $P(0)$, and collecting the constants with scattering cross section into a single constant (k) specific to gas type, we can derive an equation for the fraction of electrons not scattered:

$$I_{(0)} / I_{Total} = e^{-kpd/TV}$$

The table below shows this fraction, as a percentage, for water vapor at various pressures under typical ESEM operating conditions.

Imaging Current - Room Temperature, Water Vapor, 20 kV		
Pressure Torr	Pascals	% of Primary Beam Current ESEM - BGPL = 2 mm
40	5320	5%
20	2660	23%
10	1330	48%
7	931	60%
5	665	69%
2	266	86%
1	133	93%
0.5	66.5	96%

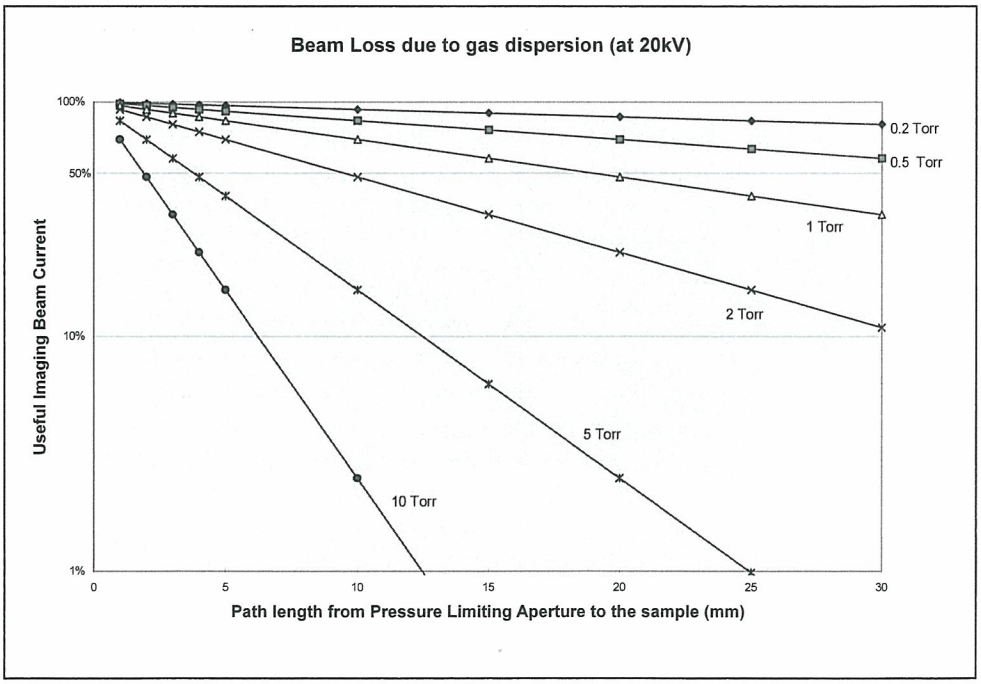


Figure 3-8. *Plots the percentage of useful imaging current (unscattered electrons remaining in the original spot) for various combinations of gas path length and sample chamber pressure. In this example the gas is water vapor, and the accelerating voltage is 20 kV.*

3.2 ENVIRONMENTAL SECONDARY DETECTORS

Secondary electrons provide the highest resolution images. Unfortunately, the Everhart-Thornley (ET) detector used in the CSEM cannot function in the gaseous environment of the ESEM. In its place the ESEM uses a proprietary Environmental Secondary Detector (ESD). The most recent generation of the ESD, the Gaseous Secondary Electron Detector (GSED), provides better discrimination against parasitic electron signals. Both ESD and GSED are patented and available only in the ESEM

3.2.1 ESD

In its simplest form the ESD is a conical electrode, about a centimeter in diameter, positioned apex down, concentric with the beam, at the bottom of the pole piece. The beam passes through the detector, exiting from the integrated final pressure limiting aperture. The detector's location directly above the sample eliminates the need to tilt the sample for improved detector efficiency

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 environmental 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.

The ionization characteristics of the gas in the sample chamber affect the imaging process directly. The more easily the gas ionizes, the higher the amplification gain will be. Varying the detector bias modulates the gain and permits the use of a variety of different gases. The most commonly used environmental gas is water vapor. It ionizes easily to provide excellent imaging performance. It is convenient and non-toxic. Last but not least, it is an abundant component of our own environment and, thus, frequently of interest as part of the experimental system under observation.

Because the ESD does not use a photomultiplier tube it is insensitive to light. Light from the sample, for example, incandescence from heated samples, cathodoluminescence, or fluorescence, does not interfere with imaging. Likewise, the detector permits the use of the chamber viewport or an integrated optical microscope, with illumination, during image acquisition.

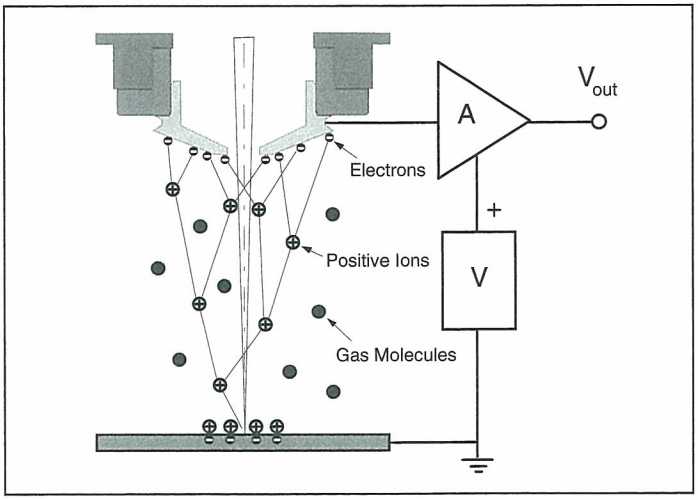


Figure 3-9. The Environmental Secondary Detector uses gas ionization to amplify the secondary electron signal. In nonconductive samples, positive ions are attracted to the sample surface as charge accumulates from the beam. There they effectively suppress charging artifacts.

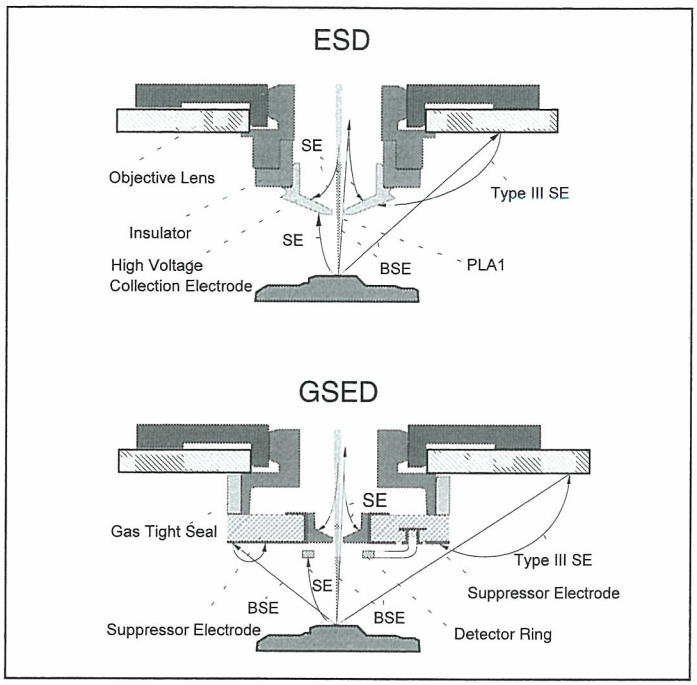


Figure 3-10. *The Gaseous Secondary Electron Detector (GSED) discriminates against backscattered electrons and parasitic Type III secondary electrons to improve image quality and apparent resolution.*

3.2.2 GSED

The Gaseous Secondary Electron Detector (GSED) is a refinement of the original ESD. It improves image quality by discriminating against spurious signals from backscattered electrons and type III secondary electrons.

BSE's

All secondary electron detectors, conventional or environmental, are also sensitive to backscattered electrons (BSE). Backscattered electrons have an angular emission distribution with a maximum normal to the sample surface. Conventional detectors are typically located to the side of and some distance away from the sample. This reduces the number of BSE's that reach them. Because the ESD is directly above and very close to the sample, it collects more backscattered electrons than conventional secondary detectors. The backscattered contribution degrades the contrast and resolution of the secondary electron signal.

Type I, II, III SE's

Secondary electrons (SE) are classified into three types based on their origin. Type I secondary electrons result from interactions between beam electrons and sample atoms, and escape only from a very shallow region where the beam enters the sample. These are the secondaries that carry high resolution image information. Type II secondary electrons result from interactions between sample atoms and backscattered electrons as the backscattered electrons exit through the sample surface. Since BSE's may travel a considerable distance through the sample before escaping, type II SE's have relatively poor resolution. Type III secondary electrons occur when a backscattered electron collides with the walls of the sample chamber or some other component of the microscope. They generally follow the intensity and resolution of the backscattered signal.

BSE Discrimination

The GSED occupies the same physical location as the ESD (see figure 3-10). It is fabricated as a printed circuit board. A seal on the back side joins a housing screwed into the pole piece and vacuum manifold of the ESEM. The final PLA is

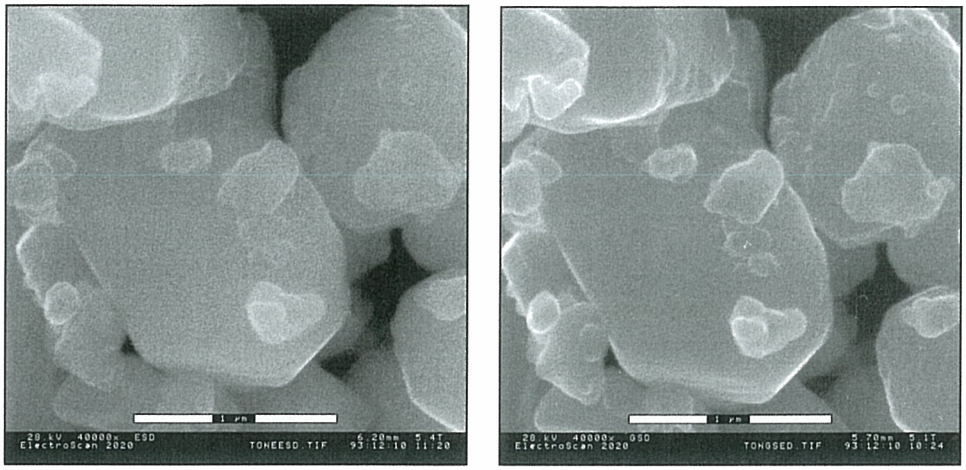


Figure 3-11. *Left - ESD image of toner particles. Right - GSED image of the same sample. Note the enhanced edge effects, indicative of the increased secondary electron contribution to the image signal.*

integrated into the printed circuit board. The suppressor electrode covers the lower surface of the assembly and is in physical and electrical contact with the PLA. The detector ring is suspended below and parallel to the suppressor electrode. The electrode and the detector ring are biased to shape the detector field. Low energy SE's will follow paths influenced by the shape of this field. Since the ring is closer to the sample than the suppressor, it creates a stronger field and attracts a larger share of SE's than its apparent relative area represents.

Type III SE Discrimination

BSE's impacting the suppressor electrode have the potential to create Type III electrons. Since the suppressor is positively biased, type III's created there are unable to escape to the detector ring. Because of its size and position, the suppressor electrode also prevents most type III's generated elsewhere in the sample chamber from reaching the detector ring.

The original ESD did not isolate the pressure limiting aperture from the detector electrode. BSE's passing through the PLA can create type III's within the pole piece and first environmental chamber. The back side of the ESD detector electrode collects these type III's. The GSED separates the PLA from the detector ring. The PLA and suppressor electrode shield the detector ring from Type III's generated within the pole piece.

The GSED's ability to discriminate against type III secondary electrons and backscattered electrons significantly enhances the quality and apparent resolution of images from the ESEM. Figure 3-11 compares images taken with the ESD and GSED detectors.

3.2.3 Charge Suppression

One of the greatest benefits of the ESEM is the absence of charging artifacts. Charging artifacts occur in conventional SEM's when charge deposited by the beam accumulates in insulating samples. The fields induced by charging cause local variations in secondary electron emissions, and deflections of the primary beam. Both interfere with imaging. In the ESEM, positive ions, generated by the signal amplification process, are attracted to the sample surface as charge accumulates. There, they suppress the local fields and effectively eliminate charging artifacts (See Figure 3-9).

Charge suppression in the ESEM permits the imaging of nonconductive samples in their natural, uncoated state. The mechanism operates at all accelerating voltages, freeing the microscopist to manipulate beam energy for purposes other than charge balance. It permits simultaneous imaging and X-ray

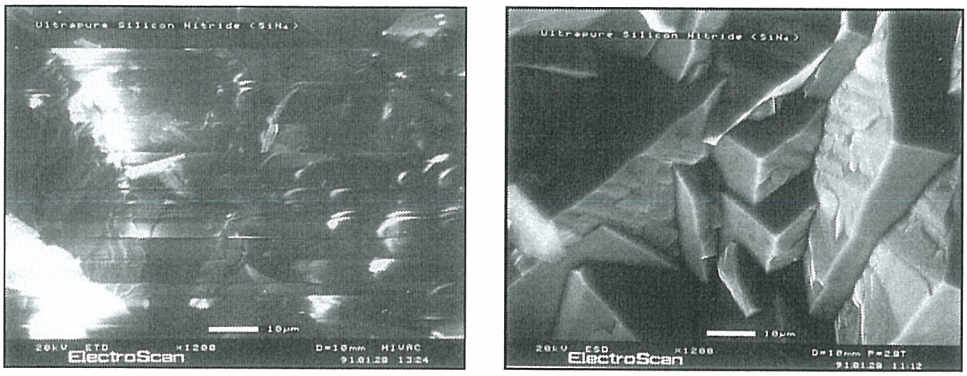


Figure 3-12. Gas ionization in the ESEM suppresses charging in insulating samples. Left - Silicon nitride, an insulator, in high vacuum. Right - The same sample in a 2.8 Torr water vapor environment.

analysis using less complex, higher energy K-lines. It is self-adjusting, suppressing charge as and where it occurs across the image field.

3.3 X-RAY ANALYSIS IN THE ESEM

The lack of charging artifacts in the ESEM has direct benefits for X-ray analysis. It eliminates the interference of sample coatings and it permits analysis at higher accelerating voltages on non-conductive samples. However there are additional variables to be considered in optimizing the ESEM for X-ray analysis.

3.3.1 Lack of Interferences

Any coating applied to a sample contributes to the characteristic X-ray spectrum. X-rays from the coating can interfere with the detection and counting of X-rays from sample elements having lines of the same energy. For instance, gold, a commonly applied conductive coating, has M lines at the same energy as the K-lines of sulfur. Coatings also absorb X-rays generated in the sample. Gold, being a heavy element, is also particularly good at absorbing X-rays. The absence of conductive coatings on ESEM samples eliminates the potential for absorption and interference.

3.3.2 Sufficient Excitation Energy

X-ray analysis is easier and more accurate using simple well-separated peaks. These are generally the lowest order peaks for a given element. K-lines are simpler than L-lines, which are simpler than M-lines. K-lines are also higher energy than L-lines, which are, in turn, higher energy than M-lines. In order to excite characteristic X-rays efficiently, the beam energy must be two to three times the energy of the line of interest. All of these factors conspire to make X-ray analysis at higher beam energies very desirable. Low beam energies are sometimes used in conventional SEM's to reduce charging. Unfortunately, X-ray analysis then becomes difficult or impossible. Once again the absence of charging artifacts in the ESEM frees the analyst to select operating conditions best suited to the task at hand.

3.3.3 Skirt X-rays

The X-ray signal has intrinsically low resolution and is not suitable for imaging in the conventional sense. In the ESEM, skirt electrons can further degrade X-ray spatial resolution.

When forming a secondary or backscattered electron image, the background contribution of the skirt electrons is easily discarded while still retaining sufficient signal to form a high resolution image. This kind of signal processing is a threshold discrimination and works well when there is a large difference

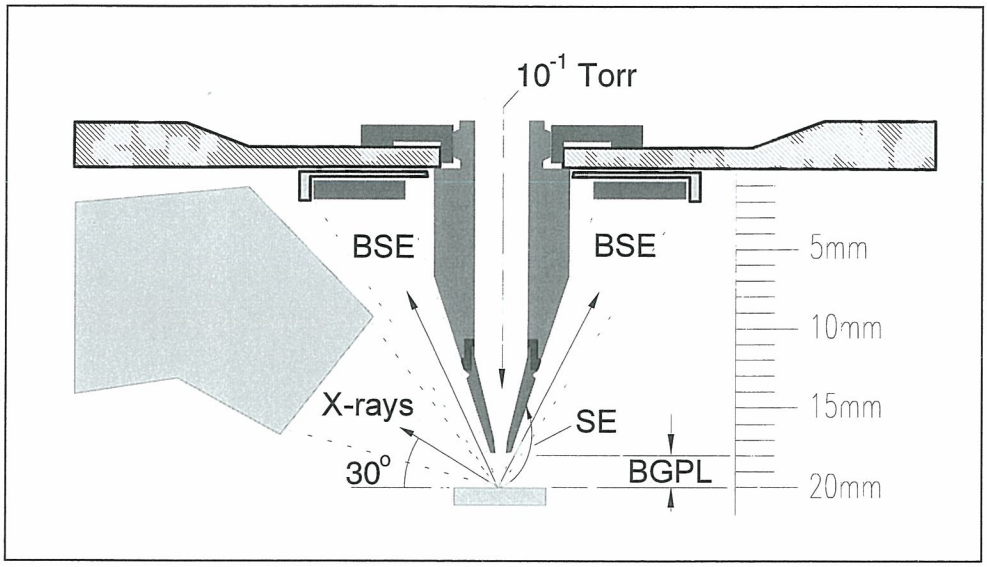


Figure 3-13. A specially designed ESD keeps the gas path length short while allowing sufficient room for a solid state backscattered electron detector and an X-ray detector (thirty degree take-off angle).A short gas path length minimizes X-rays generated by skirt electrons.

between signal intensity and background intensity. This is not the case for X-rays. The inherently weak X-ray signal is further reduced by the exclusion of all X-rays not having the specific energy characteristic of the element of interest. This weak signal is superimposed on a relatively large background signal (Bremsstrahlung). The poor counting statistics and low signal to noise ratio make threshold discrimination ineffective. Every X-ray counts. Under some conditions, obviously inappropriate for X-ray analysis, skirt electrons generate X-rays at points hundreds of microns from the center of the beam. The analyst must always remain aware of the potential for spurious X-rays generated by skirt electrons.

Minimizing Skirt X-rays

The skirt is formed by electrons scattered out of the beam by gas molecules. The displacement of a scattered electron from its original destination on the sample surface is a function of the scattering angle, and the remaining distance to the sample from the scattering site. Each successive scattering event increases the potential range of displacement. Thus the size of the electron skirt depends on the beam gas path length and the sample chamber pressure. Minimizing the path length reduces the likely displacement from any one scattering event and reduces the number of times an electron is likely to scatter. Minimizing the pressure also reduces the scattering probability.

In the ESEM, the secondary electron detector is directly above the sample at the bottom of the pole piece. A long working distance version of the ESD lowers the sample away from the pole piece while still maintaining a short gas path. In this configuration, the X-ray detector can be positioned close enough to collect X-rays at an efficient thirty degree take-off angle, while skirt size is kept to a minimum by a gas path length of only 2 mm.

3.3.4 Environmental Gas X-rays

There is one other source of X-ray background to be considered. Beam electrons also excite X-rays from the environmental gas. These will appear as a constant low level signal characteristic of the gas composition. Again, minimizing pressure and gas path length reduces this signal. The gas in the chamber may also be selected to avoid specific interferences with elements of interest.

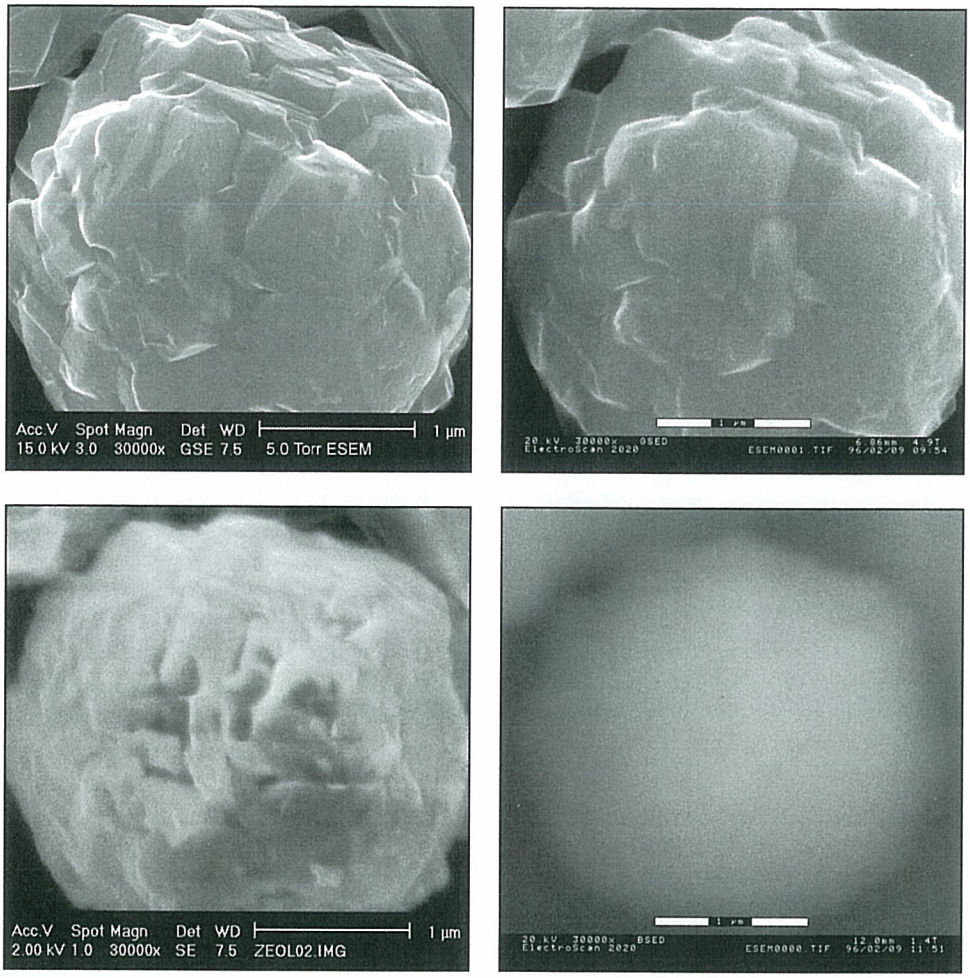


Figure 3-14. *This series of micrographs demonstrates the imaging capability of the ESEM. The sample is a zeolite. It is nonconductive and uncoated. Upper left is a high resolution secondary image taken at 15 kV in an ESEM with a field emission gun at 5 Torr. Upper right is a secondary image from an ESEM with a LaB6 electron gun at 20 kV, 4.9 Torr. The lower left image was made with a field emission gun in high vacuum using low accelerating voltage (2 kV) to reduce charging. On the lower right is a backscattered electron image taken at 20 kV and 1.4 Torr (LV-CSEM conditions) with a LaB6 gun.*

3.4 SUMMARY

There are two key technologies that differentiate the ESEM from all other SEM's. The first is its multiple aperture, graduated vacuum system. This system maintains a high vacuum in most of the electron column while permitting relatively high pressures in the sample chamber. The gas in the sample chamber does scatter some electrons from the beam. However, within a scattering regime known as Partial Scattering, corresponding to a certain range of operating conditions (pressure, gas path length, temperature, accelerating voltage, and gas type), beam scattering does not degrade image resolution.

The second key technology is the Environmental or Gaseous Secondary Electron Detector, using gas ionization to detect and amplify the secondary electron signal. Gas ionization also suppresses charging artifacts on insulating samples. The detectors are insensitive to light and heat.

The ESEM facilitates X-ray analysis by eliminating potential interferences from coatings. It also permits the analysis of uncoated insulating samples at higher accelerating voltages, where X-ray peak structures are less complex. The ESEM does introduce additional considerations to X-ray analysis, among them, the influence of chamber pressure and beam gas path length on X-ray spatial resolution, and the contribution of X-rays from the environmental gas.

4

LOW VACUUM - CONVENTIONAL SEM's (LV-CSEM's)

What is an ESEM and what is not? When the ESEM was introduced this was not a difficult distinction to make. It was the only SEM specifically designed with elevated sample chamber pressure as its primary operating condition. Since the advent of the ESEM, several SEM's have appeared which permit operating pressures intermediate between a conventional SEM and an ESEM. Are they ESEM's or CSEM's?

The historical context of the ESEM's development provides a practical definition. The inventors specifically wanted to look at liquid and hydrated samples. This dictates an operating pressure of at least 4.6 Torr, the minimum vapor pressure required to maintain liquid water at 0°C. Though somewhat arbitrary, this definition is quite useful, since it derives from one of the most valuable capabilities of the ESEM.

The key enabling technologies of the ESEM are its multiple pressure limiting apertures, and its environmental secondary electron detectors. These technologies are both protected by patent and available only in the ESEM. They are directly responsible for the ESEM's ability to offer high resolution imaging at pressures above 4.6 Torr and, therefore, may constitute the most specific basis for a definition of the ESEM.

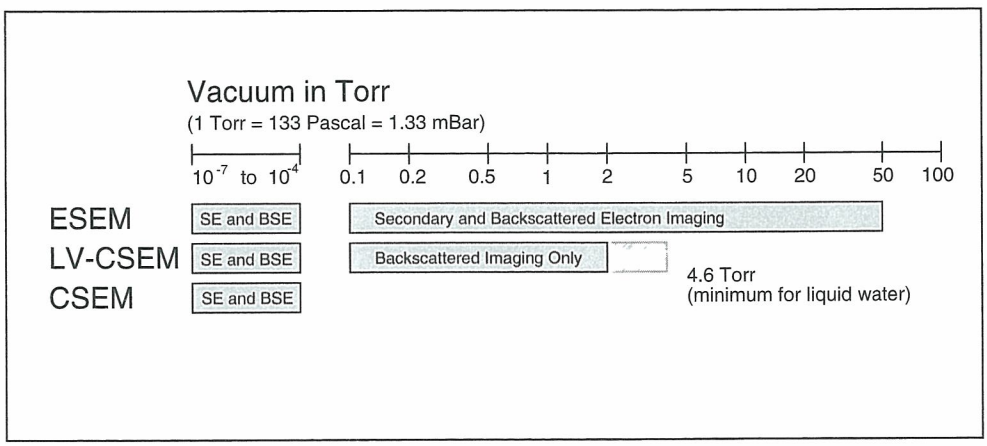


Figure 4-1. *The ESEM operates at pressures as high as 50 Torr and offers both SE and BSE imaging. LV-CSEM's are limited to 2-4 Torr and can offer only BSE images in low vacuum mode.*

Other low vacuum SEM's have no significant technological distinctions from conventional SEM's. We will refer to them here as low vacuum conventional SEM's (LV-CSEM's) They are readily distinguished from the ESEM, in terms of capability, having, without exception, maximum operating pressures in the 2 to 4 Torr range and no secondary electron imaging capability in low vacuum mode. This chapter looks at the design compromises made in LV-CSEM's, and at their capabilities and limitations.

4.1 VACUUM SYSTEMS

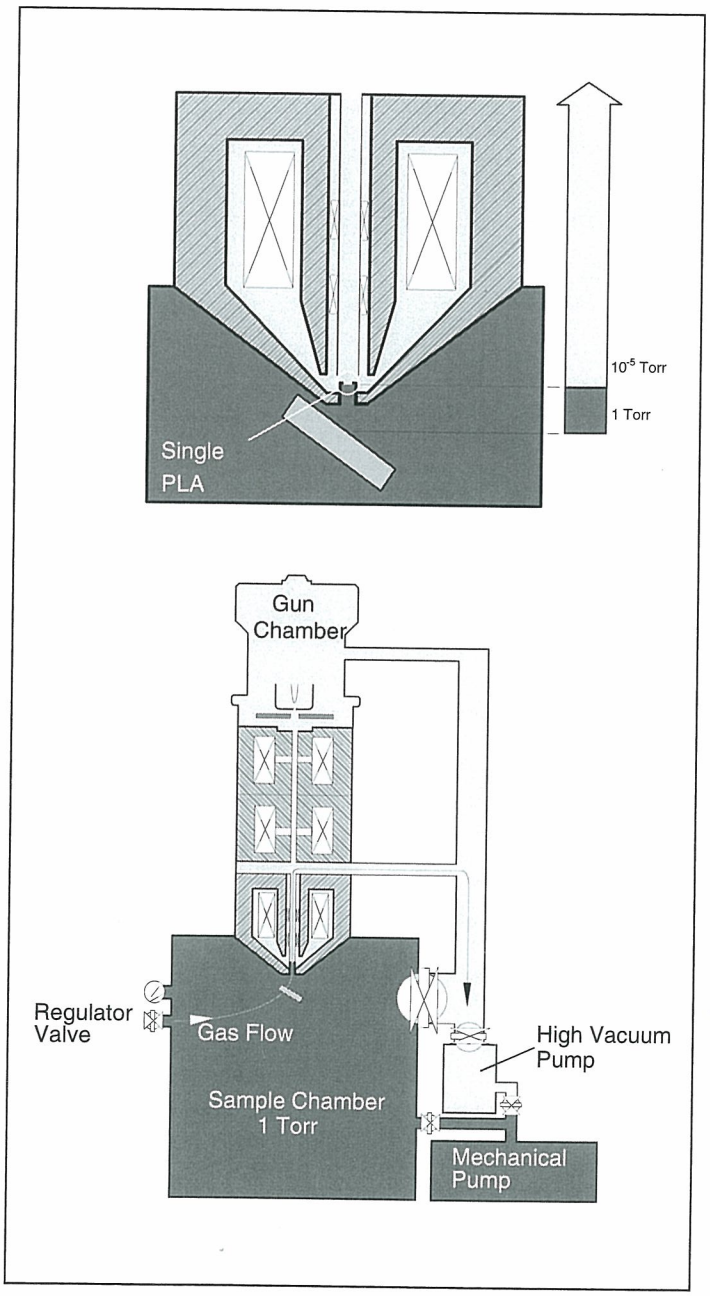


Figure 4-2. *An LV-CSEM has only a single Pressure Limiting Aperture (PLA). The size of the aperture determines the pressure differential that can be maintained between the column and the sample chamber. It also limits the current available in the electron beam*

In operation, the mechanical pump is isolated from the sample chamber. The balance of gas flow in from the regulator valve, with gas flow out through the PLA, sets the pressure in the chamber. Likewise, the balance of gas flow in through the PLA with gas flow out to the pumping system, determines the pressure in the column.

4.1.1 Single Pressure Limiting Aperture

The ESEM's patents restrict LV-CSEM's to the use of a single pressure limiting aperture. In effect this forces the final aperture to serve a dual function as both a pressure limiting aperture and a beam limiting optical aperture. When it is small enough to sustain a useful pressure difference between the column and the sample chamber, it is also small enough to limit the current available in the beam. In some designs the final physical aperture may directly serve both functions. In other designs, where a projection aperture higher in the column limits the beam, the final PLA limits the effective size of the projection aperture. In either case, the final physical aperture is subject to conflicting optical and vacuum requirements. These result, ultimately, in performance compromises.

Aperture Size

In an LV-CSEM, a mechanical pump initially evacuates the sample chamber. When the chamber reaches a predetermined pressure, an isolation valve closes between it and the mechanical pump. During operation, a diffusion or turbomolecular pump maintains high vacuum in the column. Gas flows continuously out of the specimen chamber, into the column, through the PLA. A regulator valve meters gas into the sample chamber at a rate controlled by the operator. The chamber and column settle to equilibrium pressures, determined by the various gas flows, and manipulated by the regulator valve.

In the column, the vacuum level is determined by the balance between gas outflow, to the high vacuum pumping system, and gas inflow, through the PLA. Since the vacuum level required in the column and the high vacuum pumping capacity are both fixed for any system, they also fix the maximum inflow permitted through the PLA. Gas flow through an aperture is proportional to the pressure difference and the area of the opening, for a given gas, temperature, and type of flow. At the maximum permitted PLA flow rate, the size of the aperture therefore determines the pressure differential between the chamber and the column and, consequently, the maximum permitted pressure in the chamber. Smaller apertures permit higher pressures but reduce beam current. Larger apertures permit higher beam current but reduce chamber pressure.

Increasing the performance of the vacuum system is not as simple as increasing the size and speed of the pumps. Since the mechanical pump is

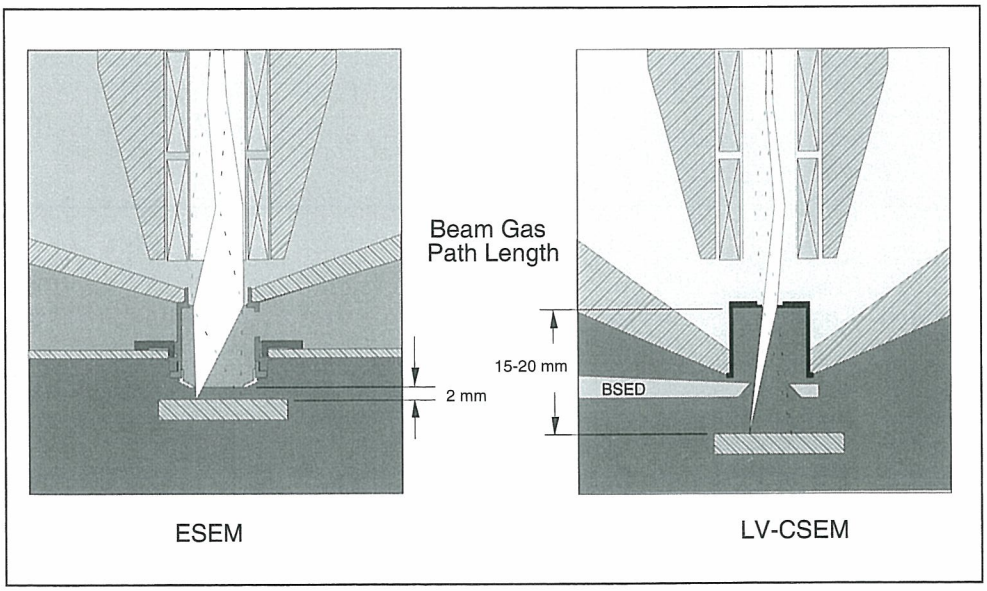


Figure 4-3. *The LV-CSEM must combine both optical and vacuum functions in a single aperture. The compromise requires a smaller aperture and longer beam gas path length than the ESEM.*

isolated after initial evacuation, a faster pump may improve pump down time, but does not affect the maximum operating pressure in the sample chamber. In the high vacuum system of the column, performance is determined by the combination of the pump capacity, and the pipe conductance between the pump and the column. In practice, pipe conductance is the more difficult to improve. Most systems, particularly adaptations of conventional designs, are pipe limited.

In an ESEM, the use of multiple apertures permits smaller pressure differences, and therefore larger diameters, at each aperture, while still maintaining a greater total pressure difference between the column and the sample chamber. These larger apertures do not impose a practical limit on beam current. Moreover, the entire vacuum system of the ESEM is designed for optimal performance in the environmental pressure range.

Aperture Position

The same principles that govern electron scattering in the ESEM, also apply in the LV-CSEM. In order to maintain their imaging capability, they too must operate in the partial scattering regime. In the equations for electron scattering, pressure and beam gas path length are equivalent. That is, an increase in path length has exactly the same effect as an increase in pressure. For a given degree of scattering, shorter path lengths permit higher pressures.

Ideally, then, the final PLA, should be as close as possible to the sample surface. However, an optical aperture in this position (close to the focal plane of the lens), limits the field of view. For the large apertures used in an ESEM (500 to 1000 micrometers), this limit is not overly restrictive. The small PLA's (typically 75 to 200 micrometers), required to attain useful chamber pressures in an LV-CSEM, make this position impractical.

SEM's use scan coils located above the final lens plane to move the beam through the scanning pattern. Most use a technique called double deflection, in which the beam is first diverted to one side, and then back again the opposite direction, to pass through the principal plane of the lens at the optical axis. This point, about which the beam pivots as it scans, is sometimes called the rocking point. An aperture at this point limits beam current equally for all points in the image plane, and can be a minimum size without limiting the field of view. In an LV-CSEM, the need to pass a maximum current through a minimum aperture dictates that the aperture be located here. Unfortunately, this point is typically 5-10 mm above the bottom of the pole piece. Since most LV-CSEM's require an additional 5-10 mm of working distance below the pole piece, the beam gas path length is 10 to 20 mm, an order of magnitude greater than in the ESEM. The result is a fundamental limitation — to remain in the partial scattering regime, LV-CSEM's must operate at pressures an order of magnitude less than an ESEM.

4.1.2 Performance Limitations

The design of an LV-CSEM vacuum system, with a single PLA, requires a variety of compromises between optical and vacuum considerations. How do these technical compromises translate into practical limitations?

Upper Pressure Limit

LV-CSEM's have maximum chamber pressures in the range of 2 to 4 Torr. Probably the most significant sacrifice to lower sample chamber pressure is the loss of the capability to keep wet samples wet. The minimum pressure needed to sustain liquid water is about 4.6 Torr. At lower pressures wet samples desiccate quickly and unavoidably. Low chamber pressures also preclude sample wetting and relative humidity experiments.

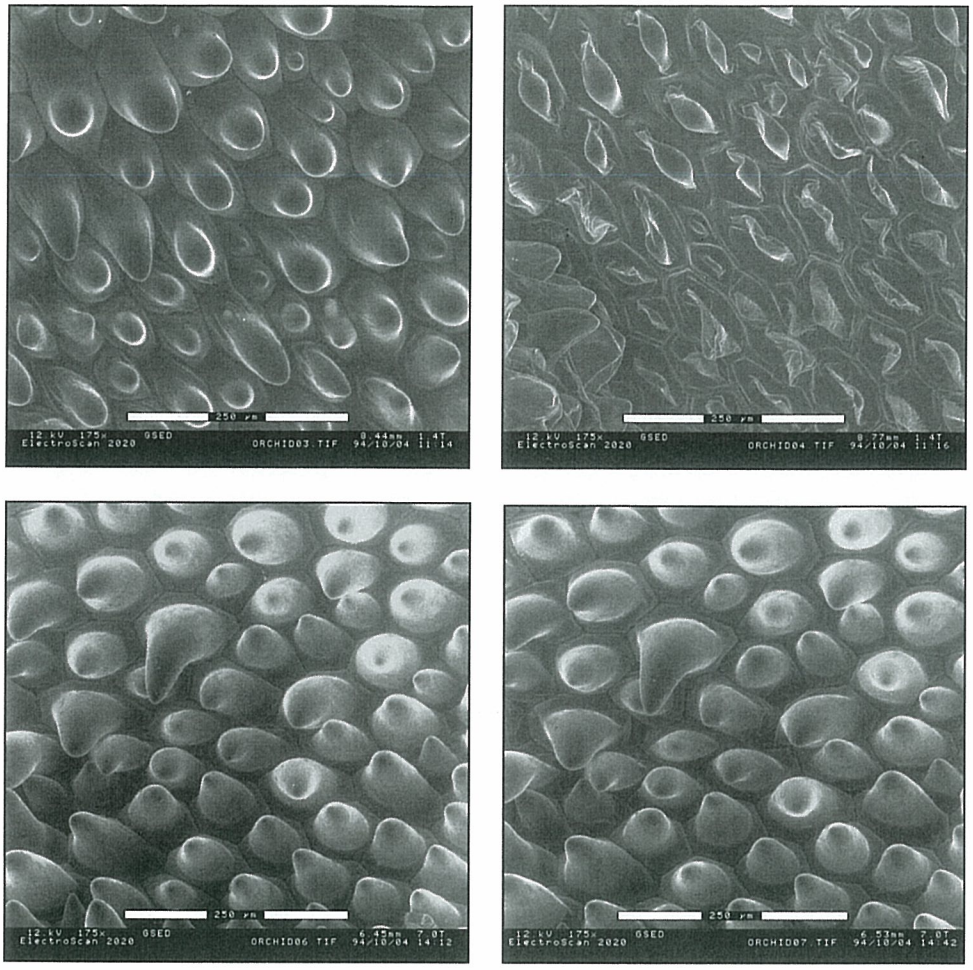


Figure 4-4. *This set of micrographs demonstrates the importance of adequate pressure capability in maintaining hydrated samples. The sample is an orchid petal. The upper pair were taken before and after a two minute exposure to a 1.4 Torr water vapor environment at 6 degrees C. Dehydration is obvious. The lower pair is the same sample before and after a thirty minute exposure to a 7.0 Torr water vapor environment at 6 degrees C. At this temperature and pressure, the water vapor environment becomes saturated and dehydration does not occur.*

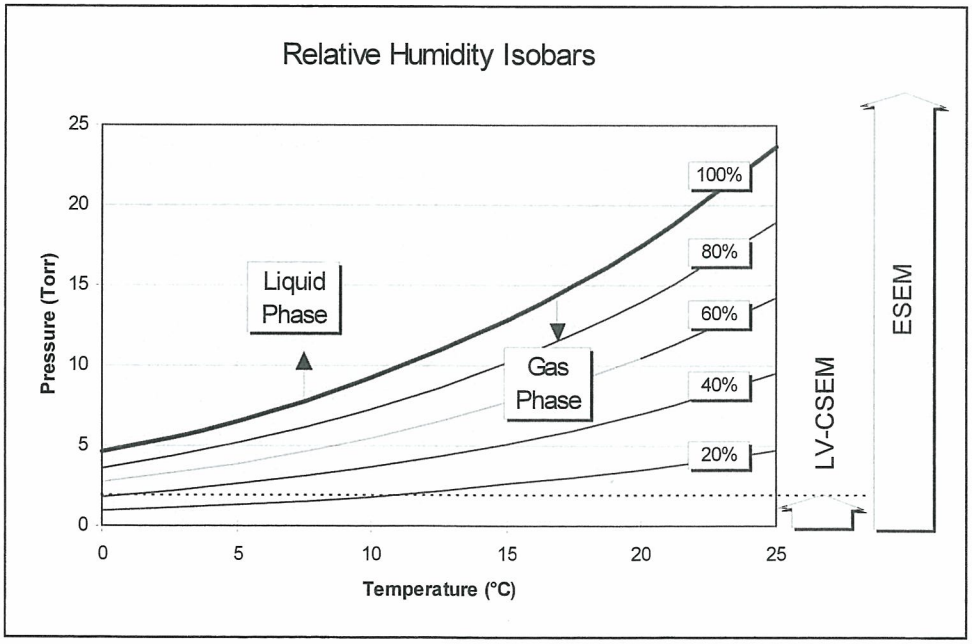


Figure 4-5. *The minimum pressure that can sustain water in the liquid phase is about 4.6 Torr at 0 degrees C. Higher temperatures require higher pressures.*

Imaging Current

Imaging current is the unscattered electron current, remaining in the spot on the sample surface, from which high resolution images are formed. The LV-CSEM pays a double penalty in imaging current. Vacuum requirements prevent the use of larger apertures to increase overall beam current. Long beam gas path lengths multiply losses due to scattering. The table below compares scattering losses in the ESEM with those in an LV-CSEM.

Imaging Current - Room Temperature, Water Vapor, 20 kV			
Pressure Torr	Pascals	% of Primary Beam Current ESEM BGPL = 2 mm	% of Primary Beam Current LV-CSEM BGPL = 20 mm
40	5320	5%	
20	2660	23%	
10	1330	48%	0.1%
7	931	60%	0.6%
5	665	69%	2.5%
2	266	86%	23%
1	133	93%	48%
0.5	66.5	96%	69%

Environmental Gases

In the LV-CSEM, any gas or contamination that passes the single PLA has direct access to the gun chamber. This leads to concerns about the types of gas permitted. Most LV-CSEM's permit only air or dry nitrogen. Every ESEM includes an auxiliary gas manifold and permits the use of practically any gas.

Contamination

Contamination of the beam limiting aperture distorts the shape of the beam, causing astigmatism in the image. In systems using the same aperture to limit both current and pressure, the aperture is directly exposed to contamination from the sample environment. In the ESEM two PLA's protect the projection aperture from contamination.

Contamination of the chamber does not usually interfere with imaging in low vacuum mode. However the same levels of cleanliness that cause no problems in low vacuum mode can lengthen pump down times or completely prevent operation in high vacuum mode. Because of their limitations in low vacuum mode, LV-CSEM's are typically used as conventional high vacuum SEM's most of the time, reserving low vacuum operation for occasional use in special applications. If their high vacuum performance is to be maintained, they must either avoid contaminating samples or be shut down for cleaning after each low vacuum use. Unfortunately, this often leads to a reluctance to use of the low vacuum capabilities at all. In the ESEM there is no performance penalty in low vacuum mode. Most ESEM's, though perfectly capable of high vacuum operation, are used almost exclusively in environmental mode.

Field of View

Because of its location in the lens plane, the PLA in an LV-CSEM does not limit the field of view. Although the PLA does limit the field of view in the ESEM, the use of relatively large apertures provides sufficient field for most applications. When optimized for large field imaging, the ESEM offers full field magnifications of less than 50X, corresponding to field sizes greater than 2 mm. It is worth noting that the ESEM can operate in high vacuum mode as well, with no PLA and an unrestricted field of view.

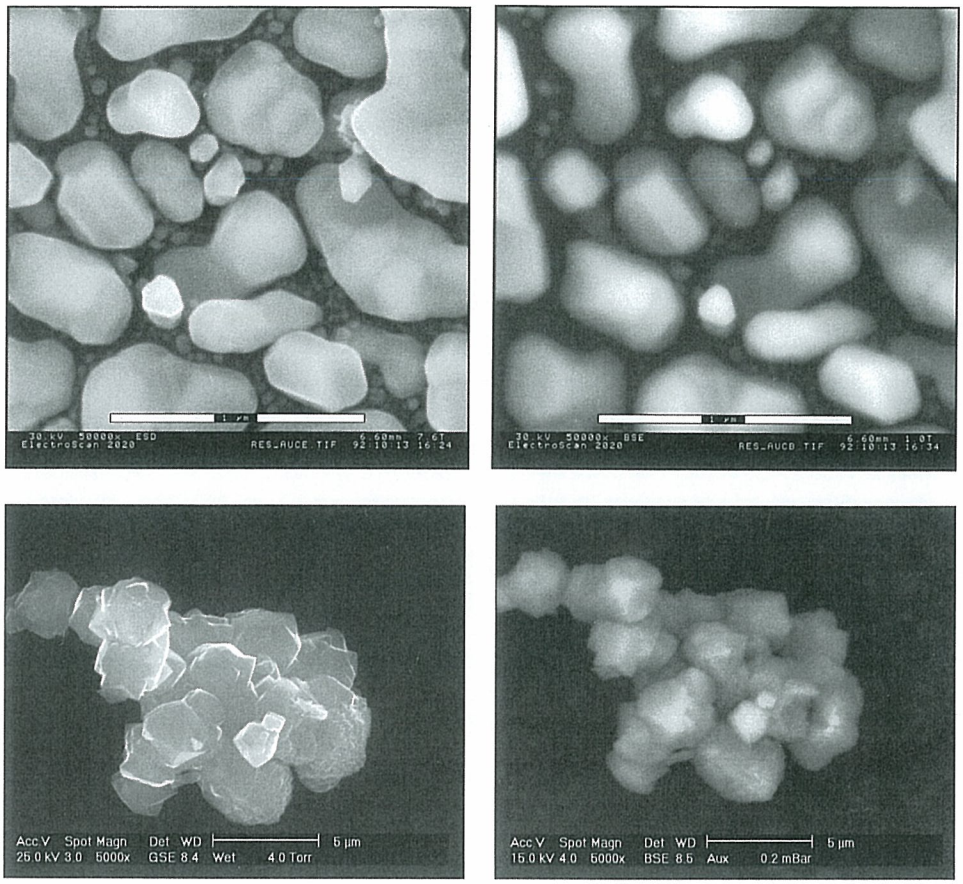


Figure 4-6. *The micrographs on the left are secondary electron images taken in the ESEM at pressures of 7.6 and 5.4 Torr. On the right are backscattered electron images of the same samples at 1.0 and 0.7 Torr. They demonstrate the resolution capabilities of the ESEM and LV CSEM's under typical operating conditions. Note the strong dependency in the BSE images on sample type. Note detail on the zeolite grains, visible only in secondary electron images. Compare backscattered images.*

4.2 Signal Detection—BSE only

One of the most important differences between the ESEM and LV-CSEM's lies in their signal detection systems. The ESEM uses a proprietary Environmental Secondary Detector to detect secondary electrons in the gaseous environment. Because conventional secondary electron detectors cannot function in a low vacuum environment, LV-CSEM's can provide only backscattered electron images in low vacuum mode.

4.2.1 Resolution

The micrographs in Figure 4-6 compare secondary and backscattered images for different samples. Note that the resolution loss is strongly dependent on the sample type. Among manufacturers and users of SEM's, gold on carbon has become a de facto standard for demonstrating image resolution, and with good reason. Gold's high atomic number limits resolution loss due to beam penetration and its complex topography offers plenty of secondary electron contrast. This sample also offers tremendous atomic number contrast between gold and carbon. It is perhaps the best possible sample to minimize resolution loss in a comparison of secondary and backscattered electron images. The toner particle micrographs, a low atomic number sample, show much greater resolution difference.

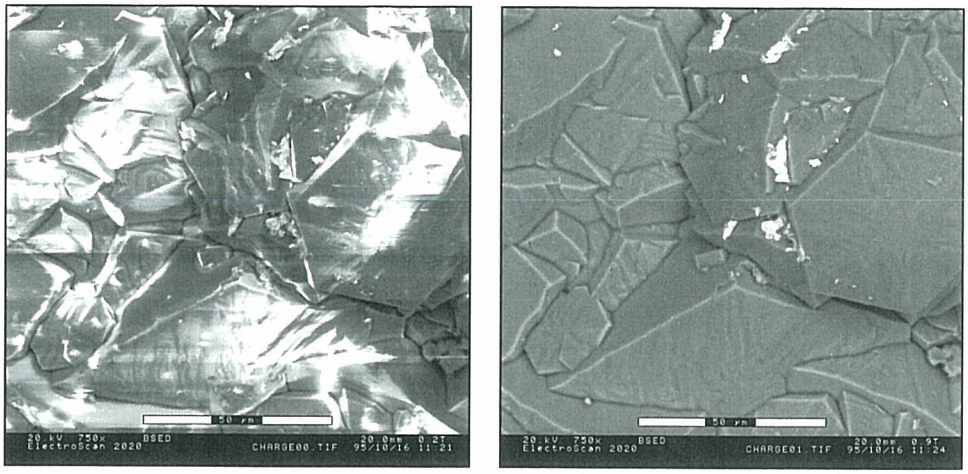


Figure 4-7. *Though BSE's are less sensitive to charging than SE's, interference can still occur. The image on the left was taken at 0.2 Torr, 20 mm gas path, on the right at 0.9 Torr, 20 mm gas path. Imaging may be difficult when low pressure is required, e.g. during X-ray analysis.*

4.2.2 Charge Suppression

Although charging has less effect on higher energy backscattered electrons, gas ionization in the LV-CSEM may still be insufficient to eliminate charging artifacts on some samples. Lacking the ions created by the ESD's gas amplification, the LV-CSEM must rely solely on ions created by the beam. Unfortunately, every ionization caused by a beam electron removes that electron from the imaging current. The result is a direct trade of imaging current for charge suppression. In situations where low chamber pressure is required, as during X-ray analysis, it may be impossible to maintain sufficient current for imaging and sufficient ionization for charge suppression. Operationally, charge suppression in the LV-CSEM may be controlled only by adjusting accelerating voltage or chamber pressure, neither of which is very convenient. In the ESEM, positive ions are created both by beam electrons and by accelerated secondary electrons as part of the cascade amplification of the ESD. Varying the ESD field strength provides convenient control of charge suppression.

4.2.3 Sensitivity to Light and Heat

Conventional secondary and backscattered electron detectors use light sensitive components as part of the detection chain. They also include materials that do not tolerate high temperatures. As a result, they are not suitable for a wide range of applications. They cannot be used to observe fluorescent, cathodoluminescent, or incandescent samples. They are limited, either directly, by transferred heat or, indirectly, by sample incandescence, in their ability to observe hot samples. They preclude the use of a sample viewport and chamber illuminator during electron image acquisition.

4.3 X-RAY ANALYSIS

X-ray analysis in a gaseous environment requires additional considerations. Principal among them is minimizing skirt size by using a short beam gas path length and low pressure. In the calculation of beam current loss for the partial scattering regime, pressure and path length are equivalent. They can be freely traded, one for the other, without increasing scattering losses. The same is not true with respect to skirt size. Skirt size is a function of the average scattering angle and the path length. Think of the skirt size as the base of a cone with its apex at the PLA. For a given average scattering angle, fixed by the type of gas and accelerating voltage, the diameter of the base is proportional to the height of

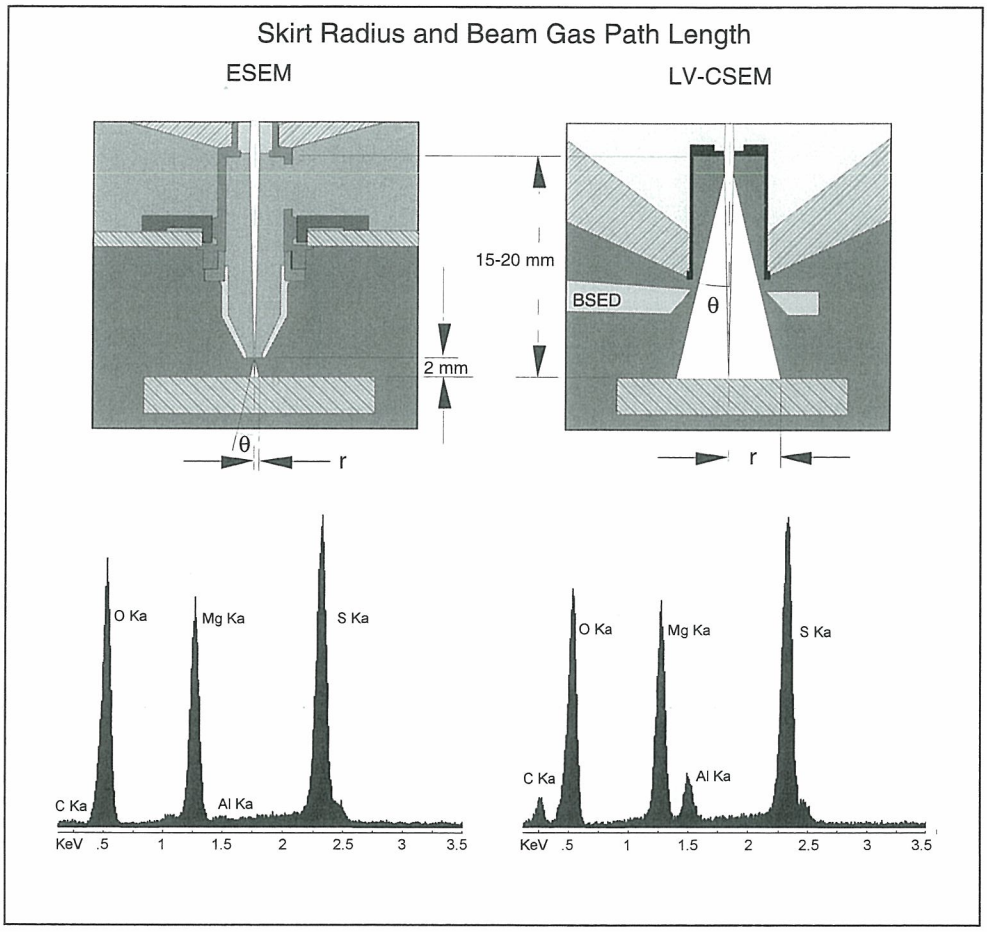


Figure 4-8. *Skirt size is a function of beam gas path length and average scattering angle. The long BGPL required in an LV-CSEM results in skirt sizes many times greater than the ESEM. Large skirts generate X-rays far from the analytical target.The X-ray spectra shown here were acquired from the same sample, a crystal of Epsom salt nearly 900 microns in size. Even on this large sample the LV-CSEM spectrum shows peaks generated by skirt electrons falling on the aluminum stub and carbon paint holding the sample. Al and C peaks are absent in the ESEM spectrum.*

the cone. A path ten times longer yields a skirt ten times broader. The long gas path lengths required in the LV-CSEM multiply the difficulties caused by skirt generated X-rays.

4.4 SUMMARY

Because LV-CSEM's are unable to offer the key technologies of the ESEM — the multiple aperture, graduated vacuum system and the Environmental or Gaseous Secondary Electron Detector — they incorporate a series of compromises. Their operating pressures are limited by conflicting optical and vacuum demands placed on the single pressure limiting aperture. They cannot maintain liquid water in the chamber nor keep a hydrated sample from drying. Their small apertures limit total beam current. The multiplied effects of scattering over an increased gas path length further limit their imaging current. They lack any secondary electron imaging capability in low vacuum mode. They have insufficient gas ionization to suppress charge on some samples. They are sensitive to light and heat. Their broad electron skirts complicate X-ray analysis.

LV-CSEM's perform best as conventional high vacuum SEM's with occasional use in low vacuum mode. In some cases, charge suppression for instance, their distinction from the ESEM is a question of degree. However, in most cases — secondary electron imaging, hydrated samples, environmental gas selection, X-ray analysis, and more — they do not offer equivalent capability in any degree.

5

APPLICATIONS

This chapter contains a selection of images chosen, each, to represent a class of similar applications, and as a collection, to demonstrate the tremendous range of ESEM applications. This is by no means an exhaustive survey. For additional examples please see the ESEM Image Library and the ESEM Bibliography.

5.1 NONCONDUCTIVE SAMPLES - UNCOATED

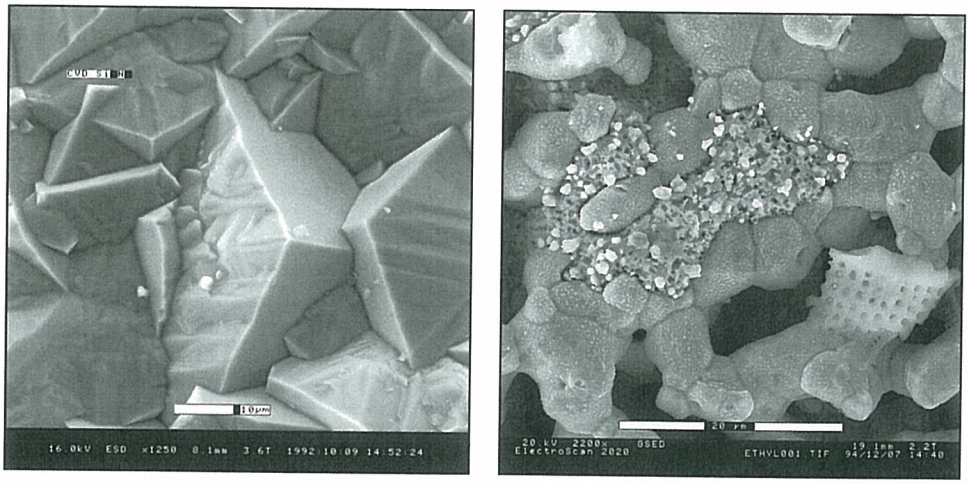


Figure 5-1, Left - Silicon nitride
***Right** - Ceramic*

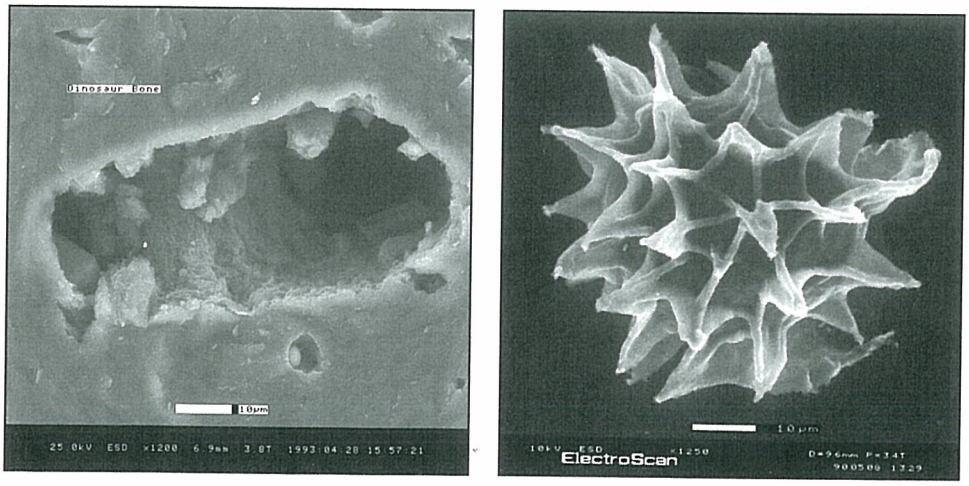
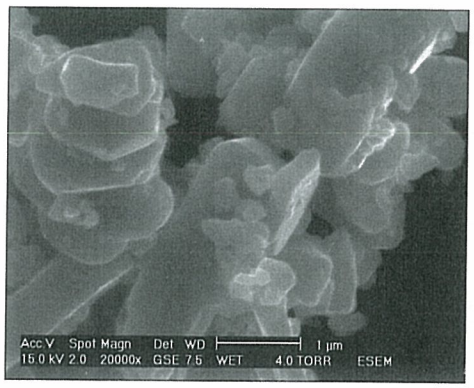


Figure 5-2, Left - Partially fossilized dinosaur bone
***Right** - Aquatic fern megaspore (135 million year old fossil)*

NONCONDUCTIVE SAMPLES - UNCOATED (CONTINUED)

Figure 5-3, Left - *Toner particles from toner cartridge, laser printer*
Right - *Detail of toner particle image 80.000x*



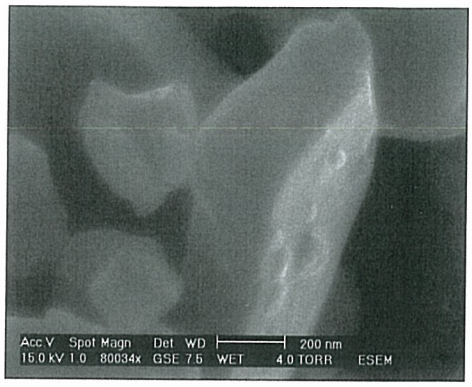
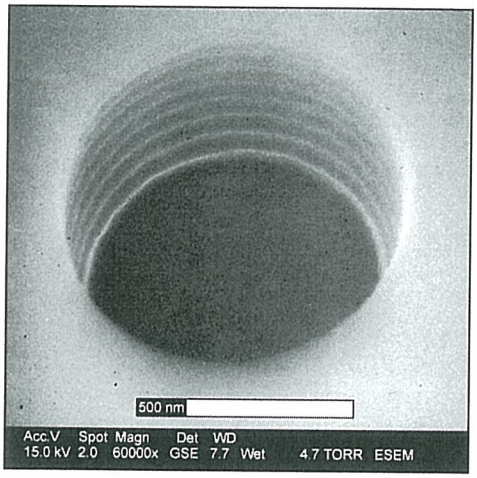


Figure 5-4, Left - *Hole in photoresist during integrated circuit fabrication*
Right - *Pharmaceutical inhaler crystals*



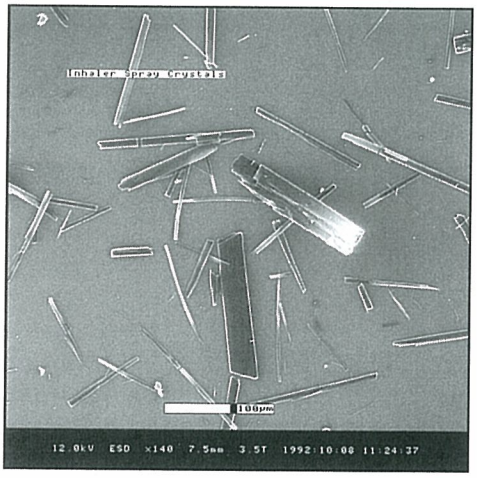
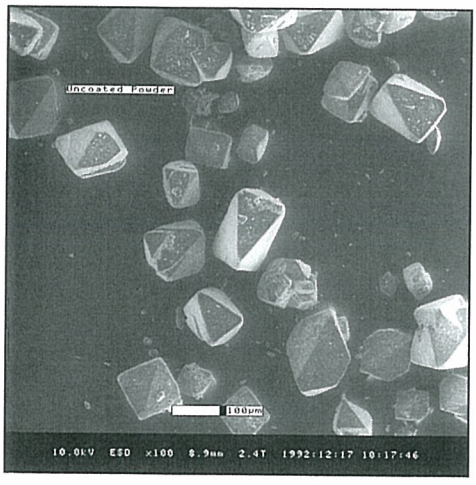


Figure 5-5, Left - *Artificial sweetener crystals*
Right - *Rouge on nylon from a forensic investigation*



5.2 HYDRATED SAMPLES

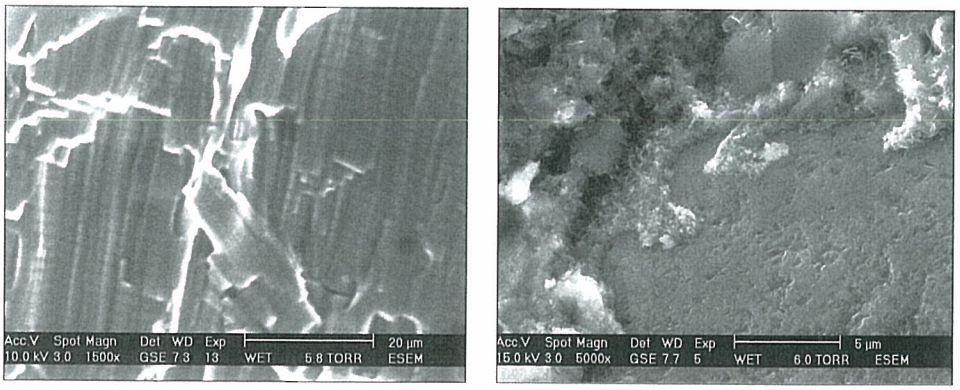


Figure 5-6, Left - *Muscle fabric just underneath the skin of a rat in its original hydrated state*
Right - *Concrete - Cement interface*

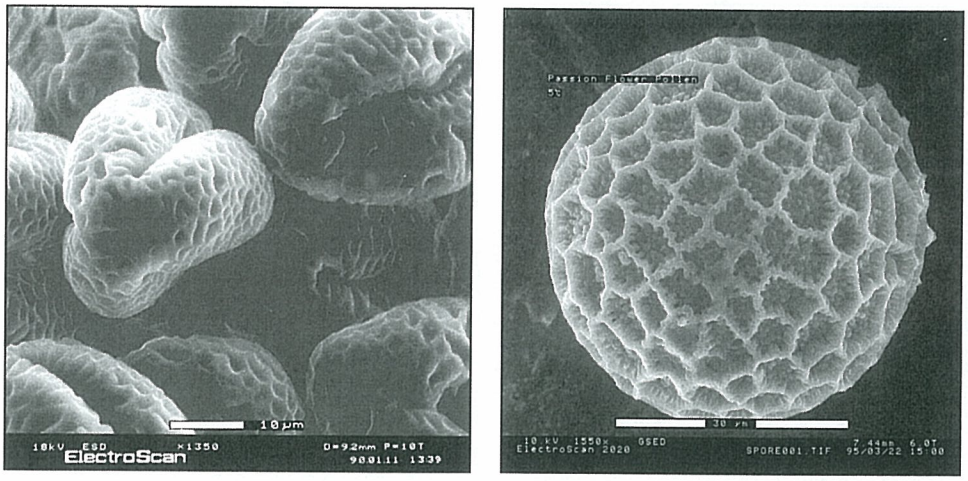


Figure 5-7, Left - *Poinsetta pollen*
Right - *Passion Flower pollen*

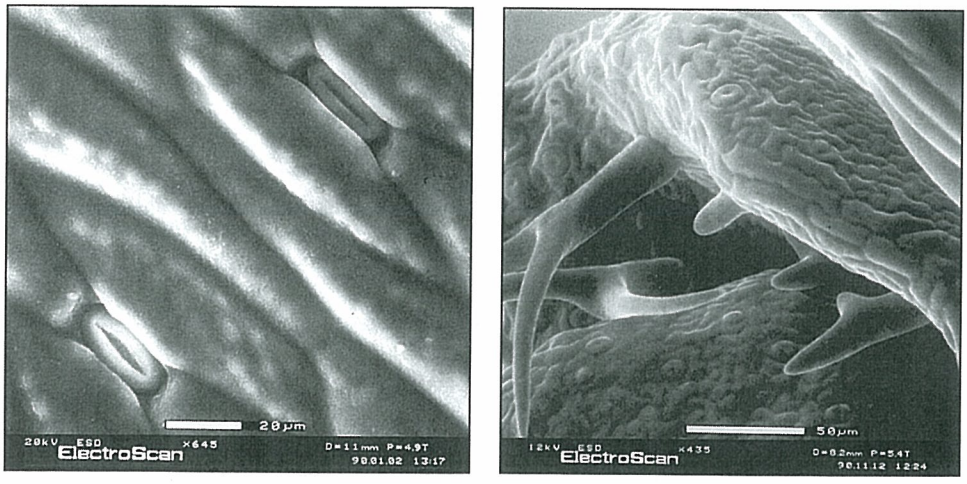
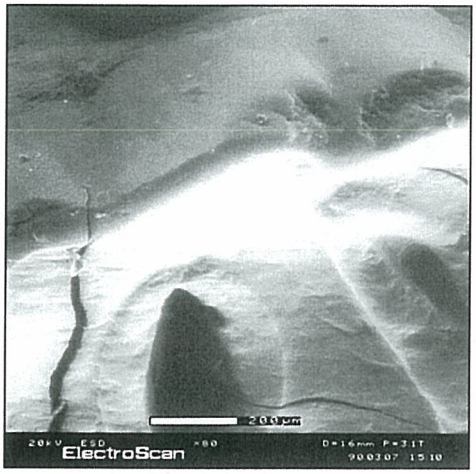


Figure 5-5, Left - *Stomata on an Aloe Vera leaf*
Right - *Root hairs of a beet seeding*

HYDRATED SAMPLES (CONTINUED)



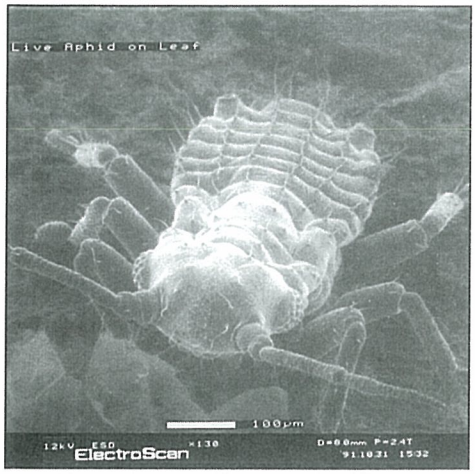
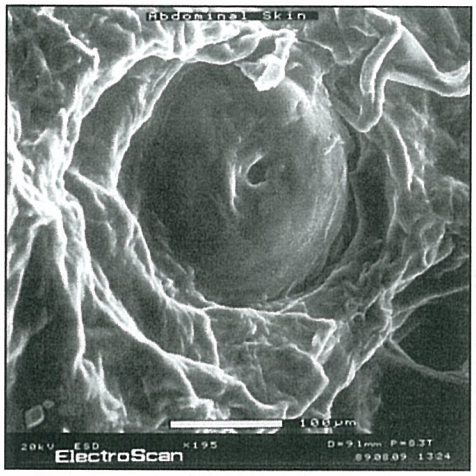


Figure 5-9, Left *- Rat tooth at 80X*
Right *- Living aphid*



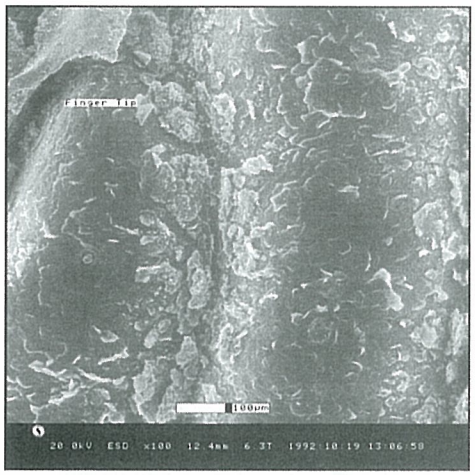
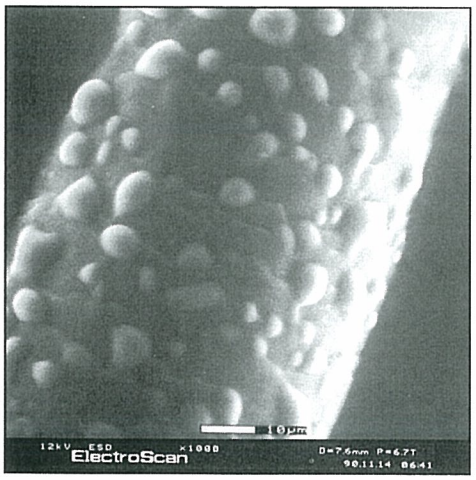


Figure 5-10, Left *- Sweat pore, porcine abdominal skin*
Right *- Skin of a human finger tip, forensic sample*



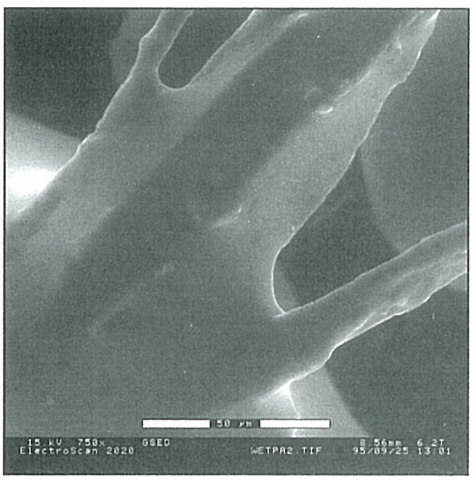


Figure 5-11, Left *- Human hair with water droplets*
Right *- Wet paper*

HYDRATED SAMPLES (CONTINUED)

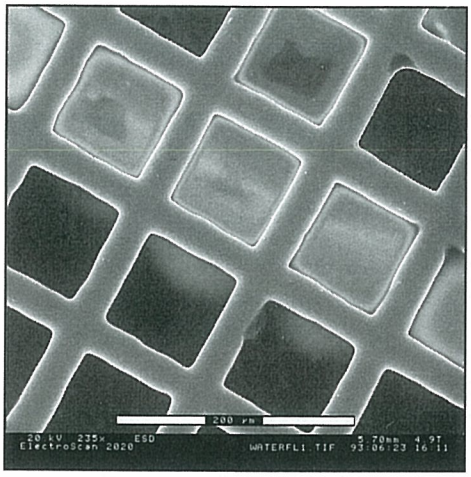
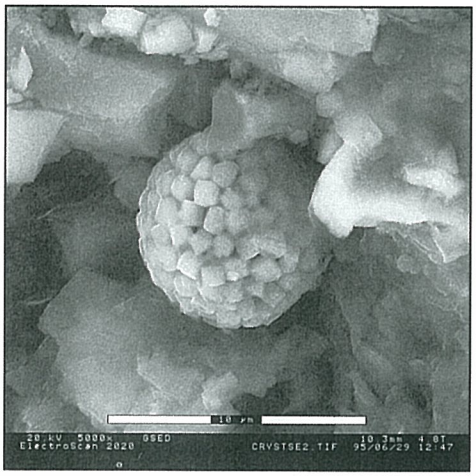


Figure 5-12, Left - *Bacteria and red blood cells on tooth root tissue*
Right - *Water film on a copper grid*

5.3 CONTAMINATING SAMPLES



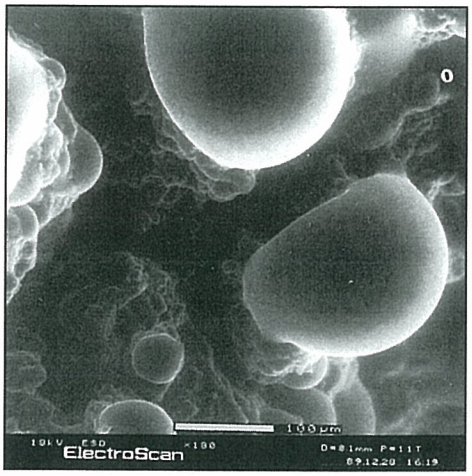
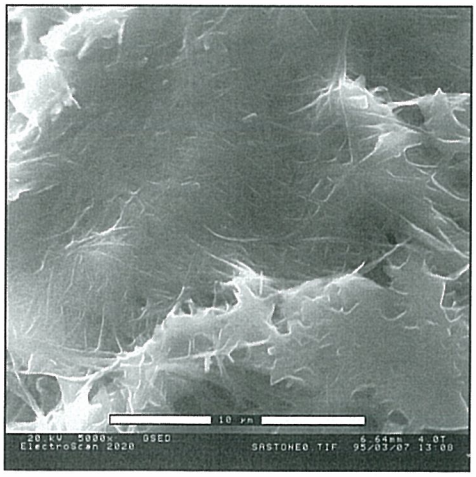


Figure 5-13, Left - *Crystallized structure discovered in oil saturated sandstone*
Right - *Droplets of oil and water on a geological sample*



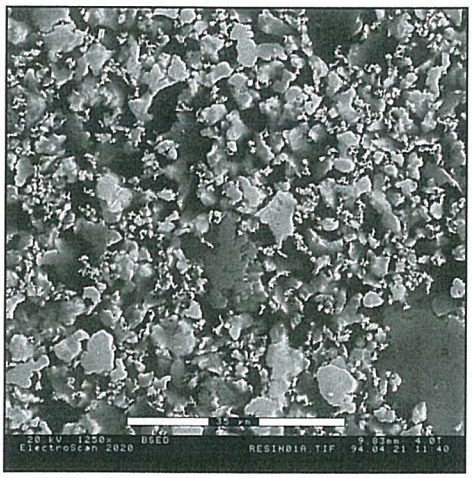
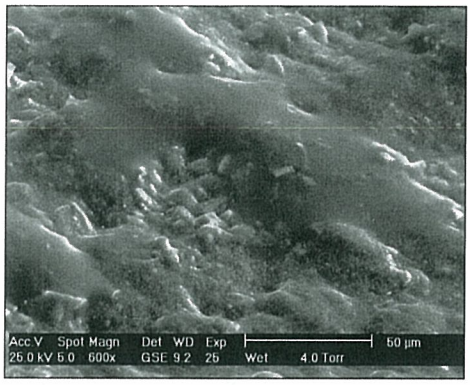


Figure 5-14, Left - *Crystal fibers in water saturated sand stone*
Right - *Metal particles in uncured resin*

CONTAIMINATING SAMPLES (CONTINUED)

Figure 5-15, Left - *Oil contamination on 17th century painting. Note the pigments partly visible through the oil drop.*
Right - *Exhaust particle from diesel engine exhaust pipe*



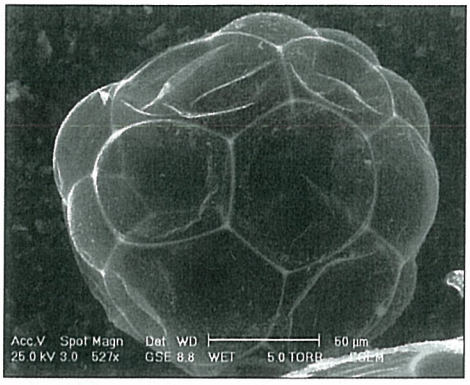


Figure 5-16, Left - *Fungus on a pine needle*
Right - *Fungal hyphae with calcium oxalatecrystals*

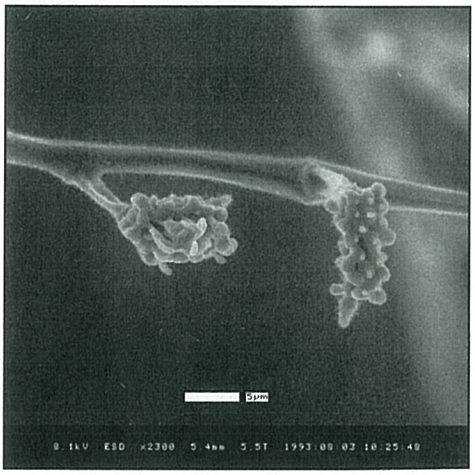
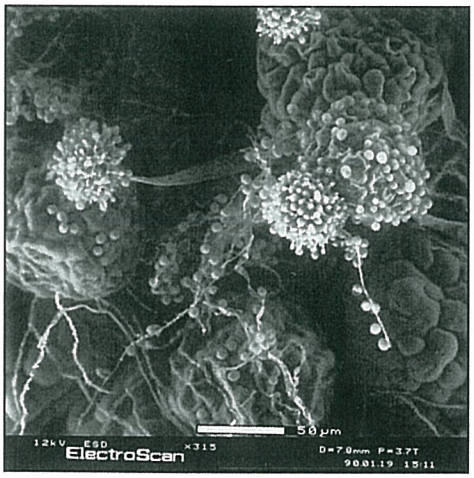
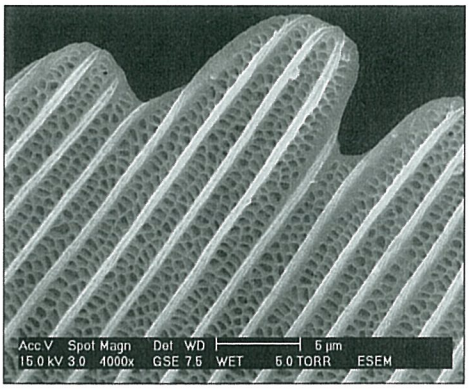


Figure 5-17, Left - *Brend mold*
Right - *Butterfly wing*





5.5 COATING INTERFERENCE

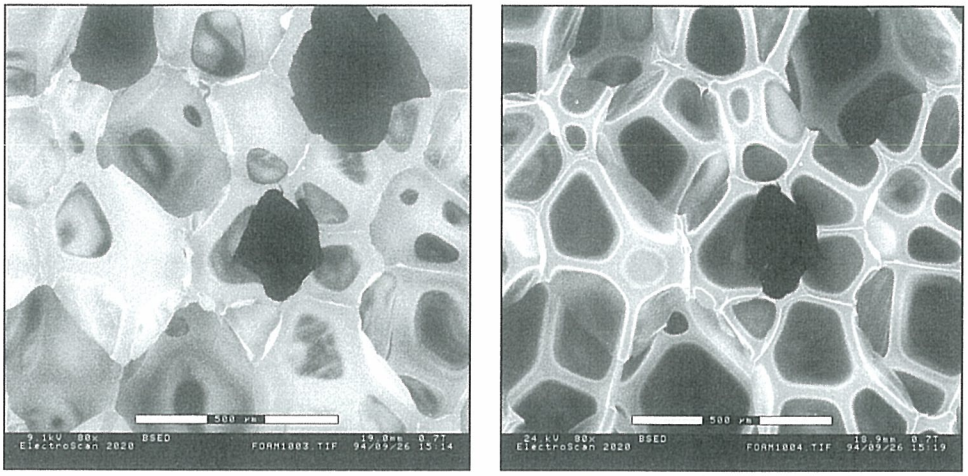


Figure 5-18 *Left - Styrofoam at 9.1 kV* ***Right*** *- The same sample at 24 kV. The difference in the two micrographs is due to the greater penetration of the higher energy beam. If the sample had been coated with gold for conductivity, the internal structure would have been masked.*

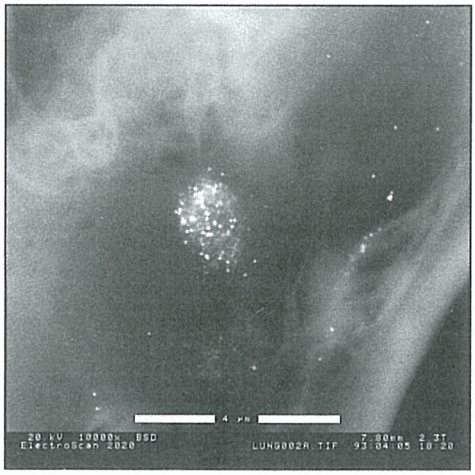


Figure 5-19, Left *- Lung tissue labeled with 20 nanometer gold particles. A gold coating would have obscured the labeling particles*

5.6 PHASE TRANSITIONS

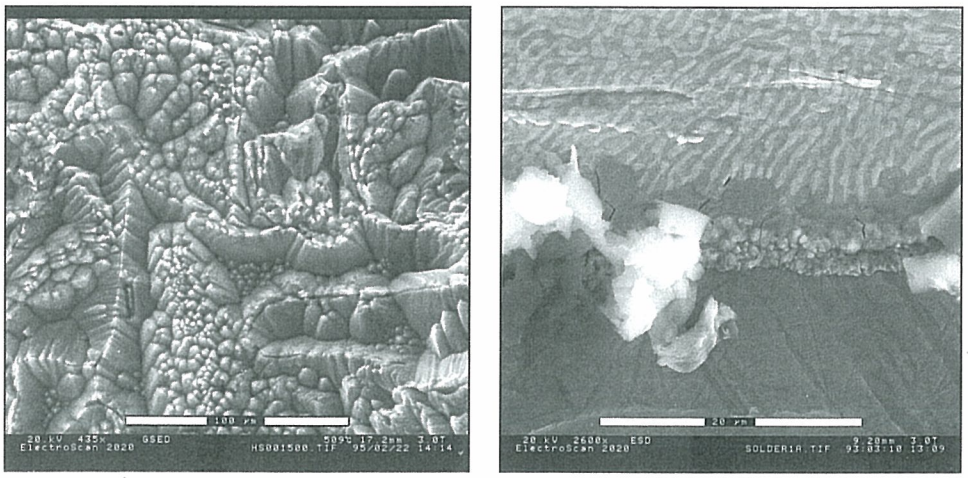
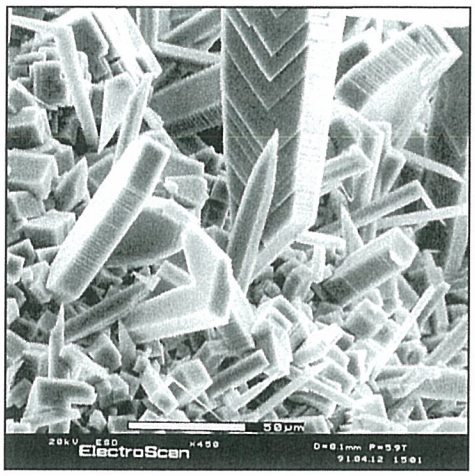


Figure 5-20, Left *- Surface of pure silicon melted and resolidified in the ESEM.* ***Right*** *- Solder on copper melted in the ESEM. With a high temperature hot stage, the ESEM can provide electron images of samples at temperatures as high as 1500°C.*

PHASE TRANSITIONS (CONTINUED)



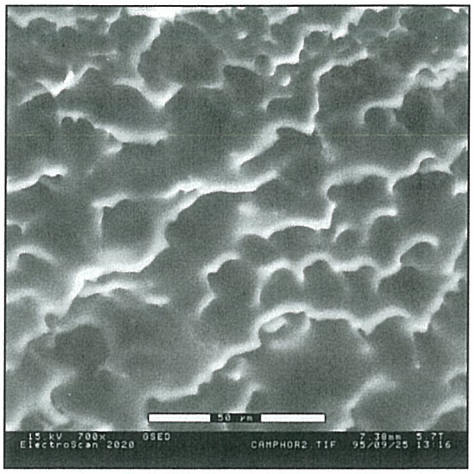


Figure 5-21, Left *- Potassium chloride crystals grown from vapor in the ESEM at about 600°C.*
Right *- Camphor sublimating to vapor*

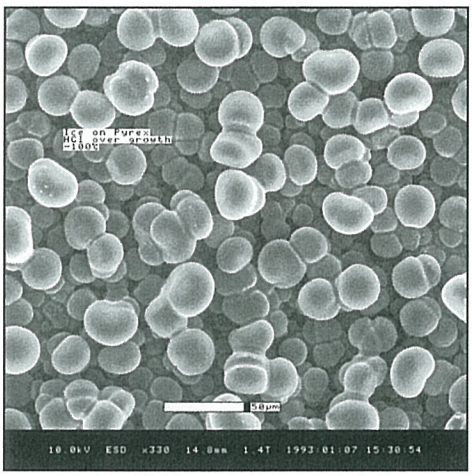
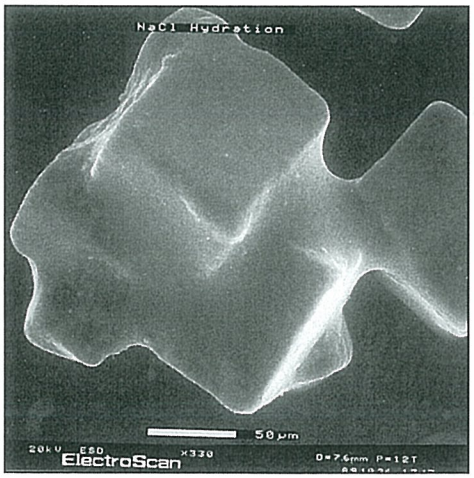


Figure 5-22, Left *- Ice crystallized from vapor in the ESEM*
Right *- Crystal of hydrochloric acid ice formed over a layer of water ice on Pyrex.*

5.7 HYDRATION PROCESSES



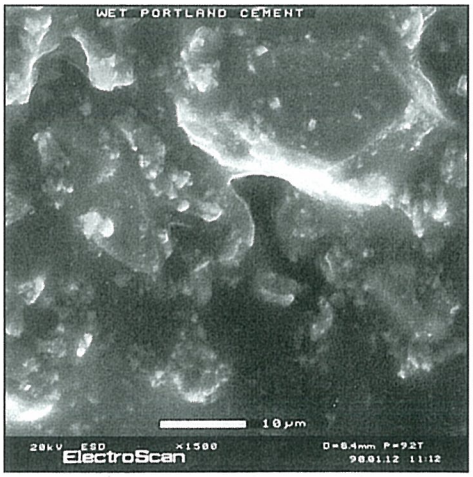


Figure 5-23, Left *- Crystals of table salt begin to dissolve in water condensed from the chamber atmosphere*
Right *- Portland cement wetted by water condensed from the ESEM atmosphere*

5.8 OXIDATION/CORROSION

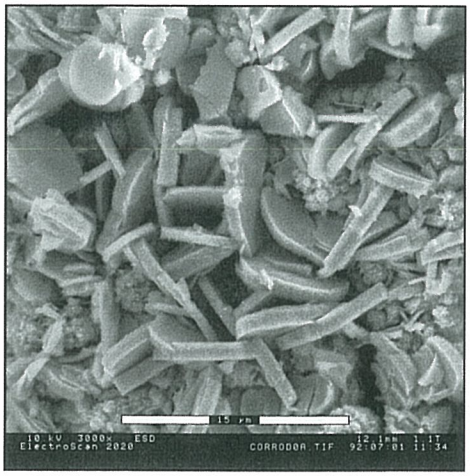
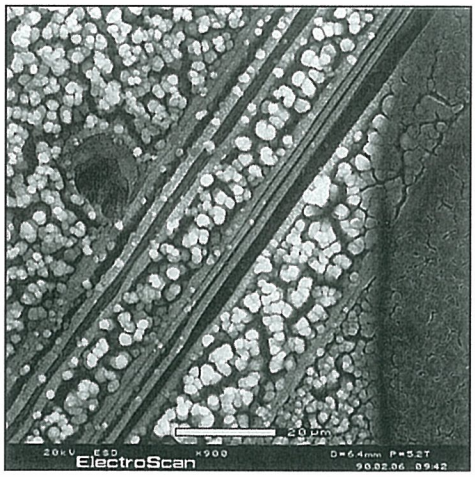


Figure 5-24, Left - *Oxide grows around a small sulfur inclusion in a piece of iron at 860°C in a pure oxygen atmosphere*
Right - *Iron sulfide crystals grown on stainless steel*

5.9 THERMAL/MECHANICAL/CHEMICAL STRESS



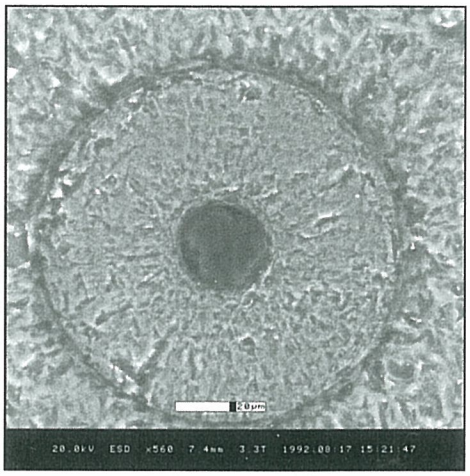


Figure 5-25, Left - *A droplet of liquid toluene has etched away the matrix of a plastic composite*
Right - *In a high temperature oxidizing environment, cracks develop at the fiber/matrix interface of a silicon carbide reinforced composite.*

Figure 5-26, Left - *Separation occurs at the fiber/matrix interface during tensile failure of a polypropylene reinforced cement*
Right - *Cracks develop at high temperature in a carbon-carbon composite*

Further Reading

G. D. Danilatos, "Foundations of Environmental Scanning Electron Microscopy," in Advances in Electronics and Electron Physics 71, 109–250, 1988

J. I. Goldstein, D. E. Newbury, P. Echlin, D. C. Joy, A. D. Romig, C. E. Lyman, C. Fiori, E. Lifshin, *Scanning Electron Microscopy and X-ray Microanalysis, 2nd Edition* (Plenum Press, New York, 1992)

K. F. J. Heinrich, Electron Beam X-ray Microanalysis (Van Nostrand Reinhold, New York, 1981)

J. E. Johnson, E. M. Griffith, G. D. Danilatos, eds. Microscopy Research and Technique 25(5&6), August 1993

Index

Philips Electron Optics
Building AAE, P.O. Box 218
5600 MD Eindhoven, The Netherlands
Tel. +31 40 2766768, Fax. +31 40 2766786
E-mail: marcom@eo.ie.philips.nl
http://www.peo.philips.com

This document is printed on chlorine free
produced paper
Printed in the Netherlands 98-02
Data subject to change without notice
9498 701 21812

FEI and Philips - Global vision, focused solutions