ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY

An Introduction to ESEM®

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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
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)
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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.
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, 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:

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>SEM</td>
<td>All Scanning Electron Microscopes.</td>
</tr>
<tr>
<td>CSEM</td>
<td>Conventional High Vacuum SEM’s</td>
</tr>
<tr>
<td>ESEM</td>
<td>The Environmental SEM</td>
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<tr>
<td>LV-CSEM</td>
<td>Low Vacuum adaptations of CSEM’s.</td>
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</tbody>
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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.
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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, LaB₆ (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.
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.
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 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. 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.
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.

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.

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 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.
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.

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.
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 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.
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 Kα 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.

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.
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.
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 “arching”.

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.

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.

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.
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 travel.
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 ($m \approx 0$) 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:

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

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:

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

\[
r_{1/2} = 0.0039 d + 1.326 d(p/d)^{1.38}
\]

For typical ESEM conditions \( d = 0.002 \text{ m}, p = 7.5 \text{ Torr} \) 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.
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:

$$n = 9.655 \times 10^{24} \frac{p}{T}$$

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

**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 water vapor.
Upper right - Magnetic tape at 50,000 X in high vacuum. Lower left -
Toner particles at 30,000 X in 5.4 Torr of water vapor. Lower right - Toner particles
at 30,000X in high vacuum.

**3.1.4 Imaging Current**
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:

$$\frac{I(0)}{I_{Total}} = e^{-kpd/V}$$

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

<table>
<thead>
<tr>
<th>Pressure</th>
<th>Imaging Current - Room Temperature, Water Vapor, 20 kV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Torr</td>
<td>Pascals</td>
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<tr>
<td>40</td>
<td>5320</td>
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<td>266</td>
</tr>
<tr>
<td>1</td>
<td>133</td>
</tr>
<tr>
<td>0.5</td>
<td>66.5</td>
</tr>
</tbody>
</table>

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.

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**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.**
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
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
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.
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.

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.

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.
**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.
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 isolated after initial evacuation, a mechanical pump must still be present to evacuate the system. The ESEM 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.
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.

<table>
<thead>
<tr>
<th>Pressure \ Toorr</th>
<th>Pascals</th>
<th>% of Primary Beam Current</th>
<th>ESEM BGPL = 2 mm</th>
<th>LV-CSEM BGPL = 20 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>5320</td>
<td>5%</td>
<td></td>
<td></td>
</tr>
<tr>
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<td></td>
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**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.
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.

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.
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.

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 light microscopy for collateral observations. Finally, they prevent 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 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 - Fern spore (140 million year old fossil)
Right - Foraminifer

Figure 5-4, Left - Hole in photoresist during integrated circuit fabrication process
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 - Orchid petal after thirty minute exposure to saturated water vapor environment
Right - Poinsettia leaf, fully hydrated

Figure 5-7, Left - Poinsettia pollen
Right - Passion Flower pollen

Figure 5-8, Left - Stomata on an Aloe Vera leaf
Right - Root hairs of a beet seedling
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
5.3 CONTAMINATING SAMPLES

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

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 sandstone
Right - Metal particles in uncured resin
CONTAMINATING SAMPLES (CONTINUED)

5.4 DELICATE SAMPLES

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

Figure 5-17, Left - Bread mold
Right - Moth wing scales
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

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


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

The 1995 ESEM bibliography contains listings on papers published.

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151 Neil Baumgarten, SEM for imaging specimens in their natural state, American Laboratory June 1990
Key Words: ESEM, natural state
Abstract: The development of an Environmental Scanning Electron Microscope (ESEM) culminates a long pursued effort to observe under high magnification materials and processes in their natural state. This article sketches this development and cites some current applications.

Key Words: 
Abstract: Scanning electron microscopes offer several unique advantages and they have evolved into complex integrated instruments that often incorporate several important accessories. Their principle advantage stems from the method of constructing an image from a highly focused electron beam that scans across the surface of a specimen. The beam generates backscattered electrons and excites secondary electrons and x-rays in a highly localized "spot." These signals can be detected, and the results of the analysis are displayed as a specific intensity on screen at a position that represents the position of the electron spot. As with television image, after a given period information about the entire field of view is displayed on the screen, resulting in a complete image. If the specimen is thin, the same type of information can be gathered from the transmitted electrons, and a scanning transmission image is thus constructed.
The scanning electron microscope is highly versatile and widely used. The quality of the image is related to its resolution and contrast, which, in turn depends on the diameter of the focused beam as well as its energy and current. Because electron lenses have inherently high aberrations, the usable aperture angles are much smaller than in a light microscope and, therefore, the electron beam remains focused over a relatively large distance, giving these instruments a very large depth of focus.
Scanning electron microscopes are versatile in their ability to detect an analyze a lot of information. As a result modern versions of these instrument are equipped with a number of detectors. Developments are sometimes related to placing the detectors in a geometrically attractive position close to the specimen. Positioning often minimizes aberration and increases resolution. It is not possible to design an ideal microscope because of the many compromises necessary however, certain developments have led to greatly improved microscopes and they are discussed below.

Key Words: 
Abstract: Scientific and even non-technical literature is filled with thousands of scanning electron microscope (SEM) micrographs taken of metals, ceramics, minerals, electronic devices, insects, micro-organisms, and many other types of specimens. This widespread use of the conventional SEM is the result of its ease of operation, high resolution, and large depth of field. However, not all samples are suitable for the conditions imposed by this instrument. For example, insulating specimens have always been something of a challenge because of surface charges generated by the impinging electron beam must be continually drained away to prevent distortion of the image. In addition, samples that outgas, evaporate, melt or decompose under the conditions of operation are generally shunned. To overcome these limitations a new type of SEM or ESEM was developed. This paper illustrates a selection of materials applications and special-purpose experiments developed to utilize the unique capabilities of the ESEM.

Key Words: Environmental Scanning Electron Microscope (ESEM), detector (gaseous ), ionization (gaseous), scintillation (gaseous), insulators (specimen), wet specimens, live specimens
Abstract: The Environmental Scanning Electron Microscope (ESEM) allows the examination of specimens in a gaseous environment. It is based on an integration of efficient differential pumping with a new design of electron optics and detection systems. Backscattered, cathodoluminescence and X-ray detectors can be designed to fit and to perform optimally in the ESEM. The secondary electron signal can be detected with the gaseous detector device, which is a new multipurpose detector. Insulating, uncoated, wet and generally both treated or untreated specimens can be studied.

158 Faith Taylor, Thomas A. Hardt, New Approaches to Ceramic Research Using the Environmental Scanning Electron Microscope., ElectroScan Corporation, Wilmington, MA 01887

Key Words: ceramic

Abstract: During the past three decades the scanning electron microscope (SEM) has been used to study ceramic materials at high magnifications; largely to observe microstructure before and after processes, and to perform elemental micro-analysis of material dispersion. Ceramics technology has recently become more diverse with the advent of exciting applications such as ceramic engines and superconductors. As the ceramics industry has expanded in these new directions, the need for more sophisticated imaging tools has also grown.


Key Words: cryo scanning

Abstract: The Environmental Scanning Electron Microscope (ESEM) (Danilatos, 1983) is widely used for imaging of electrical insulators at high gas pressures (1-20 Torr) because surface charging artifacts can be eliminated and secondary electron contrasts (Peters, 1989) can be used for high resolution microscopy. This condition allows the imaging of water in its liquid (Peters, 1990) as well as frozen state.


Key Words: silicone

Abstract: Scanning electron microscopy and environmental scanning electron Microscopy (ESEM) were used to characterize silicone release coatings on a variety of paper substrates exhibiting different release values. The study showed that the applied coating flows to fill in depressions on rough substrates, leaving thin, easily damaged layers on the high areas after cure. These defects can change the mechanism of the peel.


Key Words:

Abstract: Scientists have long explored ways to modify the SEM in order to allow the direct examination of moist biological specimens without the need to resort to tedious and often damaging dehyration and fixation techniques.


Key Words: sulfate-reducing bacteria, copper

Abstract: Environmental scanning electron microscopy (ESEM) and energy dispersive x-ray analysis (EDS) were used to characterize the topography and chemical composition of biofilm/corrosion layers produced by sulfate-reducing bacteria (SRB) on copper surfaces. The thickness, tenacity, and chemical composition of the sulfide layers, as well as the severity of localized corrosion, varied among the alloys and mixed cultures.
Microorganisms were distributed throughout the copper/nickel/iron-rich surface layers—not on top of these layers as some traditional scanning electron micrographs have indicated.


Key Words: lipid structures
Abstract: Lipid structures present specific preparation and imaging problems for electron microscopy because they are very liable in non-aqueous environments and very beam sensitive. We used Langumir films made from DPPC or films made from lung surfactant lipid extracts as test models for SEM imaging.


Key Words: wet reservoir rocks
Abstract: The barrier to imaging wet or oily specimens in their 'native' states in a scanning electron microscope (SEM) at high resolution and large depth of focus has been broken by the development of a new microscope known as the environmental scanning electron microscope (ESEM). With the new features built into the ESEM, the need for preparing samples with various specimen-destroying preparation techniques has been eliminated. For example, wet reservoir rocks can be imaged and analyzed in their 'native' state, without drying, freezing, or coating with a conductive layer, by saturating the ESEM specimen chamber with water vapor (PH20 = -0.46 psi [3.2 kpal at 25°C [77°F]] during examination. The ESEM also allows dynamic experiments to be performed in a variety of gases at pressures up to 0.6 psi [4kPa) and temperatures up to 1000°C.

This paper was prepared for presentation at the 66th Annual Technical Conference and Exhibition of the Society of Petroleum Engineers held in Dallas, TX, October 6-9, 1991.


Key Words: microbiologically influenced corrosion
Abstract: In aquatic environments, microorganisms attach to metals and colonize the surface to form biofilms producing an environment at the biofilm/metal interface that is radically different from that of the bulk medium in terms of pH, dissolved oxygen, and organic and inorganic species and leading to electrochemical reactions that control corrosion rates. The term microbiologically influenced corrosion resulting from the presence and activities of microorganisms within biofilms at metal surfaces. Microorganisms can accelerate rates of partial reactions in corrosion processes and shift the mechanism for corrosion. Microbiologically influenced corrosion has received increased attention by corrosion scientists and engineers in recent years with the development of surface analytical and electrochemical techniques that can quantify the impact of microbes on electrochemical phenomena and provide details of corrosion mechanisms. Microbiologically influenced corrosion has been documented for metals exposed to sea water, fresh water, demineralized water, process chemicals, food stuffs, soils, aircraft fuels, human plasma, and sewage.


Key Words: sub-micron IC’s, topographic contrast
Abstract: State-of-the-art SEM metrological approaches are discussed to elucidate inherent deficiencies that prevent an accurate assessment of image fidelity in the production or inspection of sub-micron IC’s,
especially on the resist level. The new technique of Environmental SEM is demonstrated to allow topographic contrast generation, unaffected by surface charging, for SAL-601.


Key Words:
Abstract:


Key Words:
Abstract: Researchers in Manchester University’s department of pharmacy are using the United Kingdom’s only example of a new research tool which has many applications in the study of materials used in the pharmaceutical studies.


Key Words:
Abstract: In vitro methodology has been developed to investigate the effects of therapeutic ultrasound on polymer erosion. Enhancement in the rate of polymer erosion was demonstrated using therapeutically acceptable levels of ultrasound on a model class of degradable polymers - polyhydroxides. It was found that ultrasound enhances polymer degradation as demonstrated by the enhanced decrease in polymer molecular weight during the induction period of erosion. Additionally, morphological changes on the surface of ultrasound exposed devices were assessed by environmental scanning electron Microscopy and suggested that cavitation may cause the mechanical disintegration of the polymer surface.


Key Words: Microbiologically influenced corrosion, Environmental Scanning Electron Microscope (ESEM), scanning electron microscopy
Abstract: A newly developed environmental scanning electron microscope (ESEM) coupled with an energy dispersive X-ray spectrometer (EDS) was used to characterize the topography and chemical composition of wet biofilms and corrosion products on metal surfaces in addition to spatial relationships between microorganisms, substratum and corrosion layers. Case studies are presented to demonstrate the applicability and advantages of ESEM/EDS technology in the investigation of microbiologically influenced corrosion (MIC) as compared to traditional methods.


Key Words: biofilm, scanning electron microscope, Environmental Scanning Electron Microscope (ESEM)
Abstract: Descriptions of biofilms and their elemental compositions based on scanning electron micrographs and energy dispersive X-ray analysis cannot be related to the original condition of the biofilm on the surface. Solvent replacement of water removes extra cellular polymeric material and reduces the concentration of
elements bound within the biofilm. In the wet state, bacteria and micro algae are enmeshed in a gelatinous film that is either removed or dried to a thin inconspicuous residue during sample preparation for scanning electron microscopy. The Environmental Scanning Electron Microscope (ESEM), provides a fast, accurate image of biofilms, their spatial relationship to the substratum and elemental composition.


Key Words: reservoir rocks

Abstract: New tools are being developed that allow us to readily explore the structure of porous reservoir rocks and the interaction of fluids with these rocks. The Environmental Scanning Electron Microscope (ESEM), or sometimes called wet SEM, is just such a tool that makes pore level studies easier. The major advantages of ESEM is that the sample does not need to be coated with gold or carbon. This reduces damage to the pore structure and allows liquids to be present in the samples.


Key Words: gallstones

Abstract: Gallstones contain precipitated cholesterol, calcium salts, and proteins. Calcium (Ca) bilirubinate, palmitate, phosphate, and carbonate occurring in gallstones have variable morphologies but characteristic windowless energy dispersive x-ray (EDX) spectra. Previous studies of gallstone microstructure and composition using scanning electron microscopy (SEM) with EDX have been limited to dehydrated samples. In this state, Ca bilirubinates appear as either glassy masses, which predominate in black pigment stones, or as clusters, which are found mostly in cholesterol gallstones. The three polymorphs of Ca carbonate, calcite, vaterite, and aragonite, have been identified in gallstones by x-ray diffraction; however, the morphologies of these crystals vary in the literature. The purpose of this experiment was to study fresh gallstones by environmental SEM (ESEM) to determine if dehydration affects gallstone Ca salt morphology.


Key Words: ivory

Abstract: Two ivory fragments from the Metropolitan Museum of Art’s Ancient Near Eastern Art Department were examined by scanning electron microscopy (SEM): MMA #36.70.12 and MMA #36.70.37J. Each fragment was examined in two types of SEM: a new type - Environmental Scanning Electron Microscope (ESEM ElectroScan Corporation) - which permits microscopy in a near ambient environment (specimens need not be subjected to high vacuum or surface coating); and conventional SEM (Amray 1600T equivalent).


Key Words: Sn-Pb
Abstract: Sn-Pb alloys are the most often used solder materials in the microelectronics industry for the interconnection of components to substrates. The expanded use of surface mount technology has increased the importance of the mechanical properties of solders. This is because, in addition to providing electrical contacts, the solder functions as structural members by mechanically supporting surface mounted devices on circuit boards.


Key Words: solder

Abstract: Environmental Scanning Electron Microscope (ESEM) was used to monitor eutectic tin/lead solder paste reflow, demonstrating the capability to visualize solder paste reflow which is an important component attach process for printed circuit boards. The effect of three different atmospheric conditions (air, nitrogen and a reducing atmosphere) during reflow were studied. A unique furnace/controller system was used to simulate a typical industrial reflow process. A reducing atmosphere produced the best reflow while lowering the melting point of the solder versus nitrogen or air.


Key Words: chlorite acid sensitivity in sandstone reservoirs

Abstract: The effect of HCl on authigenic chlorite in three different sandstone’s has been examined using an Environmental Scanning Electron Microscope (ESEM), together with conventional analytical techniques. The ESEM enabled chlorites to be directly observed in situ at high magnifications during HCl treatment and was particularly effective in allowing the same chlorite areas to be closely compared before and after acid treatment. Chlorites were reacted with 1M to 10M HCl at temperatures up to 80°C and for periods up to 5 months. After all treatments, chlorites show extensive leaching of iron, magnesium and aluminum, and their crystalline structure is destroyed. However, despite these major compositional and structural changes, chlorites show little or no visible evidence of acid attack, with precise morphological detail of individual plates preserved in all samples following acid treatments. Chlorite dissolution, sensu stricto, does not occur as a result of acidization of the host sandstone’s.

Acid-treated chlorites are likely to exist in a structurally weakened state that may make them susceptible to physical disintegration during fluid flow. Accordingly, fines migration may be a significant engineering problem associated with the acidization of chlorite-bearing sandstone’s.


Key Words: sandstone reservoirs, illite-smectites

Abstract: The water sensitivity of authigenic smectite- and illite-rich illite-smectites in sandstone reservoirs has been investigated using an Environmental Scanning Electron Microscope (ESEM). The ESEM enabled the illite-smectites to be directly observed in-situ at high magnification during freshwater immersion, and was also particularly effective in allowing the same selected illite-smectite areas to be closely compared before and after freshwater treatments. The tendency of authigenic smectite-rich illite-smectite to swell on contact with fresh water varies greatly. Smectite-rich illite-smectite may cosmically swell to many times its original volume to form a gel which greatly reduces porosity and permeability, or may undergo only a subtle morphological change which has little or no adverse effect on reservoir quality. Authigenic illite-rich illite-smectite in sandstone’s does not swell when immersed in fresh water. Even after prolonged soaking in fresh water, illite-rich illite-smectite particles retain their original morphology. Accordingly, illite-rich illite-smectite in sandstone’s is unlikely to cause formation damage if exposed to fresh water based fluids.

185 H. M. Wallace, P.J.R. Uwins and C. A. McConchiel, Investigation of pollen-stigma interactions in Macadamia and Grevillea using ESEM, Department of Entomology, University of Queensland, St.
All members of the plant family Proteaceae are protandrous with the pollen being released prior to the onset of stigma receptivity. In many genera, including Macadamia and Grevillea, the released pollen is presented at flower opening on a specialized swollen region of the style. The pollen surrounds the receptive stigmatic surface but self pollination is minimized by asynchronous maturation of the pistil and cells that interact with the pollen. In addition to this temporal and physical separation of the male and female components of flower development, a partial self incompatibility mechanism has been reported in some genera (Sedgley et al 1990). After self-pollination in Macadamia, growth of pollen tubes is inhibited in the upper pistil and pollen contents may be prematurely discharged through a sub-terminal pore in the distorted pollen tube tip (Sedgley, 1983). This self incompatibility mechanism is thought to limit initial nut set in Macadamia.

There have been several foodborne disease outbreaks associated with the consumption of sprouted seeds (Andrews et al, 1982; O'Mahony et al, 1990; Portney et al, 1976). In 1988 for example, in Sweden, a large outbreak of salmonellosis was associated with the consumption of mungbean sprouts. The imported mungbean seeds, were found to be contaminated with Salmonella species (O'Mahoney et al, 1990). Later in the same year, another outbreak of salmonellosis in the United Kingdom was also linked to the consumption of mungbean sprouts. As the origin of the Salmonella in both cases was found to be contaminated seed, a detailed study was initiated to identify points of attachment and distribution of the bacteria on seeds with different coat types which might reveal why these bacteria are so resistant to conventional seed treatments (Andrews et al, 1982; Fordham et al 1975).

First systematic study of vacuum and pressure characteristics of JEOL JSM-2 SEM. The use of a single PLA coinciding with objective aperture does not allow short clearance, because of the image distortion and bright halo from the aperture. A double aperture solved the problem. This allowed pressures up to 68.5 mbar with 20keV with a modified scintillating BSE detector. The small diameter hole in the detector was used for detection at the short working distance as opposed to the “hemispherical or wide angle” geometry used Robinson. Wet wool fibers under very stable environmental conditions could be examined at room temperature.

Key Words: atmospheric SEM, ASEM, open SEM, high pressure, BSE, acronym ASEM

Abstract: A new detection configuration for the SEM has been devised, which allows the imaging of the surface of a specimen in the open room, i.e., at atmospheric pressure. Such a device gives rise to a new microscope: the Atmospheric Scanning Electron Microscope (ASEM). In this configuration, a backscattered electron detector is placed between the pressure limiting aperture and the electron column. The electron beam passes through the final aperture, reaches the sample in the open room and the backscattered electrons passing through the same final aperture reach the detector. The principle has been tested and the result reported. The size of the aperture used was 22µm. The acronym ASEM has been introduced for the first time.


Key words: atmospheric SEM, open SEM, high pressure, BSE, ESEM, acronym ESEM, acronym ASEM

Abstract: Further advances on the development of ASEM and ESEM are reported. The acronym ESEM has been introduced for the first time. These two terms correspond to two different configurations, both of which allow the examination of specimens at any pressure up to one atmosphere. In ASEM the detector is placed above the PLA, whereas in ESEM the detector is placed below or is contiguous or integral with PLA. First time two-stage differential pumping is introduced in the SEM, via two apertures and an additional rotary pump. The PLA diameter for ASEM was increased to 30µm. Scintillating BSE detectors are used. The concept of a portable electron optics column to examine specimens at room conditions is introduced. Specimens were no more limited by size, portability or nature: the idea is to take the microscope to the specimen and examine its natural surface.


key words: wool, applications

Abstract: An overview of ESEM with particular emphasis on its application to studies of wool fibers.


key words: applications, plant material, botanical structures, live specimens

Abstract: An environmental scanning electron microscope (ESEM) has been developed which allows the examinations of live and wet plant specimens. The results are compared with those obtained using similar material that has been hydrated or prepared by conventional techniques. The plant materials can survive the hypobaric pressure and beam irradiation, especially if the latter is controlled.


key words: high pressure, differential pumping, pressure characteristics, PLA, leak rate, aperture conductance, objective aperture, room temperature, wool, BSE, multiple-backscattered electrons (MBSE), design & construction of ESEM & ASEM, field of view, ASEM

Abstract: This is the first in a series of reports on the design and construction of an atmospheric or environmental SEM. The introduction of better vacuum pumping between the objective and pressure limiting aperture (PLA) has allowed the use of relatively large pressure limiting apertures, i.e., up to 57µm for operation at atmospheric pressure or up to 400µm for operation at saturation water vapor pressure and at one atmosphere, at room temperature are presented. The first part of experimentation and analysis on the vacuum characteristics of the new system together with different detection configuration is also presented. An integrated detector/PLA
A system is proposed. By a multiple-backscattered electron (MBSE) imaging mode it is shown that the surface of a specimen can be imaged although the BSE detector is placed below an (opaque) specimen.

**Abstract:**

Fresh, fixed and critical point dried specimens of rat trachea, stomach and skin were examined with the ESEM. Results showed that in tissues with fine surface detail such as trachea and stomach there was some loss of contrast and resolution in the image when they were viewed in the fresh state. In a “harder” tissue such as skin, quite clear images could be obtained. A comparison of fresh and critical point dried skin showed some distortion in the form of increased desquamation, and shrinkage in the critical point dried tissue. At present, the advantages of this technique seem to lie in the fact that some features of biological tissues which may be masked by processing could be revealed. Living tissue can be examined. It is time saving as tissues do not need to be fixed, dehydrated and critical point dried.

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**Key words:** biological ESEM, tissues, fresh materials, live specimens, applications

**Abstract:**

Advances in ASEM and ESEM are presented. The PLA is tilted to avoid the effects of a gas jet forming above PLA. A wedge shaped BSE detector is used. The multiple-backscattered electrons contribute to the image. Low (7) keV at TV scan rate is used to image salt crystal formation. Wool and live plant specimen is imaged at one atmosphere with 15keV. The PLA size of ASEM has been dramatically increased to 140µm for operation at one atmospheric pressure.

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**Key words:** low keV, TV scan rate, gas dynamics, PLA tilt, wedge-shape BSE detector, multiple-backscattered electrons (MBSE), ASEM, live plant in atmosphere, live specimens

**Abstract:**

Advances in ASEM and ESEM are presented. The PLA is tilted to avoid the effects of a gas jet forming above PLA. A wedge shaped BSE detector is used. The multiple-backscattered electrons contribute to the image. Low (7) keV at TV scan rate is used to image salt crystal formation. Wool and live plant specimen is imaged at one atmosphere with 15keV. The PLA size of ASEM has been dramatically increased to 140µm for operation at one atmospheric pressure.

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**Key words:** low keV, TV scan rate, gas dynamics, PLA tilt, wedge-shape BSE detector, multiple-backscattered electrons (MBSE), ASEM, live plant in atmosphere, live specimens, rat tissue, live ants, field distortion, bright halo, critical review, Leptospermum flavescens, BSE detector (thin), BSE detector (wedge), wool, radiation effects, applications, review, outline

**Abstract:**

A critical review of ESEM and its applications to date is presented. Wool fibers subjected to various treatments, wet (fresh) rat tissues, crystallization and rewetting o salts, and some radiation effects have been examined. A wedge shaped BSE detector together with a tilted aperture allowed the use of relatively low (7 keV) at TV scan rate.

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**Key words:** review (brief), review, outline

**Abstract:**

Brief review of ESEM is presented.

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**Key words:** gaseous detector device

**Abstract:**

First disclosure of gaseous detector device (GDD). This is a novel method in electron microscopy whereby the gaseous ionization produced by the signal-gas interactions is used for imaging.
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<td>211</td>
<td>Danilatos, G.D. (1983b) A gaseous detector device for an environmental SEM. Micron and Microscopica Acta 14:307-319.</td>
<td>This is the first paper announcing the gaseous detector device, by which the ionization produced in the gas by the signal-gas interactions is used for imaging. Ionizing radiation’s such as BSE and SE electrons produce positive ions and free electrons in the gas. These charge carriers can be collected by electrodes placed in various positions in the specimen chamber. The contrast varies with gas pressure, electrode positioning and electrode or specimen bias level and polarity. The variation of ionization current measured with a Faraday cup and with a ring electrode was measured. An inversion of contrast and corresponds to a “cross over” point of the ionization current collected by the Faraday cup as we raise the pressure. Various contrast phenomena are recorded.</td>
<td>gaseous detector device, ionization, SE detection with GDD, BSE detection with GDD, GDD-low bias, GDD</td>
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<td>212</td>
<td>Danilatos, G.D., and Postale, R. (1983) Design and construction of an atmospheric or environmental SEM-2. Micron 14:41-52.</td>
<td>This is a continuation of reports on the design and construction of an ESEM and ASEM. It presents a thorough experimental investigation of the gas dynamics in the system. Experiments specifically aimed to establish how the vacuum in the electron optics system was affected by the relative positioning of the objective and pressure limiting aperture, as well as the pumping speeds employed, specimen chamber pressure, geometry and size of apertures, and by other means. Further, the influence of the jet deflectors, to control the effects of this jet on the microscope system were studied quantitatively using specifically designed apparatus. In addition, the study of the pressure gradients below the pressure limiting aperture revealed that specimens can be placed as close as one radius from the aperture and still experience an almost saturated vapor pressure environment. The results of the present study are currently being used in the design of an optimum detection configuration. A preliminary result has allowed the use of 140 µm pressure limiting aperture to observe specimens at atmospheric pressures as well as the use of accelerating voltages (e.g. 7 kV) at TV scanning rates to record video cassette dynamic phenomena, including wetting or recrystallizing salt solutions, etc.</td>
<td>gas dynamics, jet, jet deflectors, jet length, high pressure, differential pumping, pressure characteristics, PLA, leak rate, objective aperture, room temperature, BSE, design &amp; construction of ESEM &amp; ASEM, field of view, ASEM</td>
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<td>214</td>
<td>Danilatos, G.D., and Brancik, J.V. (1984) A microinjector system in the environmental SEM. Eighth Australian Conference on Electron Microscopy, Brisbane, Australian Academy of Science, Abstracts:34.</td>
<td>Some applications of ESEM required the invention of a device for the injection of micro droplets of liquid onto the specimen in situ. The practical problem of transferring liquid from ambient pressure into the hypobaric environment has been solved by the following system: A small cavity behind a capillary needle in the specimen chamber is filled or emptied with a liquid by means of two tubes leading outside the microscope. The internal diameter of the needle (20 µm) was chosen so as to conduct sufficient liquid when externally applied under pressure, whilst at other times allowing a negligible air leak. The needle can be moved in three dimensions with controls independent from the microscope stage. To accommodate the moving needle and specimen within 1 mm from the pressure limiting aperture a BSE detector integrated with PLA was constructed. This arrangement has been used to obtain video recordings of various systems.</td>
<td>microinjector, liquid flow, applications</td>
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<td>215</td>
<td>Danilatos, G.D., Denby, E.F., and Algie, J.E.</td>
<td>1984</td>
<td>The effect of relative humidity on the shape of Bacillus apiarius spores. Current Microbiology 10:313-316.</td>
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<td>216</td>
<td>Danilatos, G.D.</td>
<td>1985</td>
<td>Design and construction of an atmospheric or environmental SEM (part 3). Scanning 7:26-42.</td>
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<td>217</td>
<td>Danilatos, G.D., and Brooks, J.B.</td>
<td>1985</td>
<td>Environmental SEM in wool research present state of the art. Proc. 7th Int. Wool Textile Research Conference, Tokyo, I:263-272.</td>
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<td>219</td>
<td>Danilatos, G.D.</td>
<td>1986b</td>
<td>Color micrographs for backscattered electron signals in the SEM. Scanning 8:9-18.</td>
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frame. In addition, the mixing of signals from the gaseous detector device together with their use for color imaging is also examined.


key words: gaseous detection device , GDD , multi-electrode GDD

Abstract: Two wires are used for the gaseous detection device (GDD). Directionality contrast is produced. By adding the outputs from GDD, atomic number contrast is produced equivalent to that obtained with a pair of scintillating BSE detectors. By subtracting the outputs, topography contrast is produced. By inverting the electrode bias, the contrast is inverted.


key words: terminology , acronyms ESEM and ASEM , solid state detectors

Abstract: Brief review of ESEM. Proposal to unify the terms of ASEM and ESEM to one only, namely, ESEM. Design of integrated solid state detectors for the general ESEM.


key words: radiation effects , wool

Abstract: Various beam irradiation effects on wool fibers are reported. The type and amount of beam effects depend on (a) the electron beam: accelerating voltage, current intensity, scanning mode (raster or other, line density, magnification), (b) the environment: nature, pressure and temperature of gas and (c) the specimen: composition, structure, texture and orientation.


key words: specifications of ESEM , commercial ESEM , integration , universal ESEM

Abstract: A summary of ESEM specifications is presented with a view to designing a commercial ESEM. The concept of integrating various fundamental components is introduced: For a commercial instrument we should integrate (a) objective lens and scanning coils with (b) differential pumping and with (c) detection systems, all in a new design. The concept of a universal ESEM is introduced, whereby ESEM can perform both as a conventional SEM and as an ESEM.


key words: cathodoluminescence , gaseous scintillation , gaseous detection device , GDD , generalized GDD

Abstract: A novel detection means for the environmental SEM (ESEM) is described. Certain gases, apart from being the environmental conditioning medium, can also act as a scintillator detector. All signals, such as secondary and backscattered electrons which can cause a particular gas to luminescence, can be detected. It is further concluded that the gas can act as a generalized detector device for all signal-gas reactions provided some suitable parameter can be monitored. New possibilities in the detection of specimen cathodoluminescence created by the ESEM are demonstrated.


key words: microinjector , liquid flow , applications
Abstract: The microinjector previously developed and reported has been used in various applications. It is possible to form a droplet standing at the tip of the needle of the microinjector and move around the object under examination to make contact with the liquid. The subsequent wetting, absorption or reaction of the liquid can be viewed at TV scanning rates and a video recording can be made for further analysis. With this system it has been possible to observe the wetting and removal of different components from the surface of greasy (raw) wool fibers. Apart from the specific wool applications, other industrial and scientific investigations can benefit. Video recordings of various liquids and liquid transport is shown. The flow and absorption of water by paper tissue at various “instants” is captured. The fast changes of configuration of liquids can be captured in real time for subsequent study. The ESEM equipped with this system has created new possibilities for surface physics and chemistry.

key words: high pressure, differential pumping, pressure characteristics, PLA, leak rate, aperture conductance, BSE, gas dynamics, jet, definition of ESEM, terminology, gas flow, gas equations, beam-gas interactions, beam-specimen interactions, specimen-signal interactions, signal-gas interactions, gas-specimen interactions, beam-signal interactions, scattering cross-sections, beam profiles, electron skirt, skirt, electron distribution, beam diameter, interaction volume in gas, gaseous reactions, ionization, scintillation, gas scintillation, ion concentration, electrostatic Pinch effect, secondary electrons, cathodoluminescence, x-rays, Auger electrons, gaseous detection device, resolving power, signal-to-noise-ratio, SNR, radiation effects, charging, contamination, damage, ESEM operation, application, oligo-scattering, plural scattering, single scattering, multiple scattering, molecular flow, continuum flow (viscous effusion), mean free path, beam spread, reduced variables, Gaussian distribution, Faraday cup, spot width, beam transfer, salts, contrast, resolution, live specimens, wool, probe, electron probe, distribution, skirt radius, cross-section, elastic cross-section, scattering

Abstract: A comprehensive survey on the fundamentals of ESEM is presented. A formal definition of ESEM is proposed. The state of gas in ESEM is given, namely, the basic equations frequently needed, an analysis of the gas flow, calculation of conductance and experimental assessment of gas flow. The general interactions in ESEM are outlined by way of pointing to all possible combinations of dual component systems. Above all, the main thrust of this survey concentrates around the fundamental question of electron beam scattering and distribution in gas. Until this time, there have been conflicting reports on whether the useful beam spot spreads or not. Applying complex mathematical formulas that describe the electron scattering and distribution in a plural scattering regime, a definitive answer was found for the first time: Fraction of electrons is removed from the original beam in vacuum and is redistributed in a very broad “skirt” surrounding the remaining intact fraction at the center. This result is further confirmed by careful experimentation.

This finding is extremely significant, because it means that the resolving power of ESEM can be maintained in the presence of gas. The preservation of a core electron beam with the same distribution as in vacuum occurs over a finite beam travel distance at a given gas pressure. This regime is characterized by the condition that the average number of collisions per electron is less than three, and it has been termed "oligo-scattering" regime. In the course of this study, calculations of scattering cross-sections for atomic and molecular gases and profiles of skirts for a "point" beam and for a Gaussian distribution beam have been found. Furthermore, the beam-gas interaction volume, ionization of gases, ion concentration, and electrostatic effects are analyzed. All detection modes, namely, BSE, SE, CL, x-ray and Auger electrons are discussed in detail. The multipurpose gaseous detection device is reviewed. Analytical equations of signal-to-noise-ratio and a thorough examination of contrast and resolution is undertaken. The beam radiation effects, namely, charging, contamination and damage are discussed. Some basic considerations for the operation and application of ESEM are outlined. This survey is an up-date of the state of the art in ESEM at present.

key words: resolution, profile, electron distribution, skirt, scattering
Abstract: This is a summary on the electron beam profiles and resolution based on a previous extended survey.

key words: contrast, resolution, signal-to-noise-ratio
Abstract: This is a summary on contrast and resolution based on a previous survey.

key words: chemistry in ESEM, surface chemistry
Abstract: A brief review of ESEM with emphasis on chemistry in the system.

key words: resolution micrograph, integration ESD/BSE, ElectroScan ESEM. review, outline
Abstract: A concise review of ESEM with early micrographs from the ElectroScan ESEM. Efficient scintillator design in conjunction with a sharp tip ESD. The fundamental aspects of ESEM are outlined in the four page extended abstract.

Key Words:
Abstract: Environmental Scanning Electron Microscope (ESEM) eliminates sample preparation and allows materials to be examined in their natural states.

key words: integration, PLA-detector, GDD, high pressure, design & construction of ESEM & ASEM, ASEM, GDD above PLA
Abstract: A new detection configuration using the gaseous device in the environmental scanning electron microscope (ESEM) is demonstrated. First, the pressure-limiting aperture (PLA) is used simultaneously as a biased electrode to collect the ionization current produced in the gaseous environment of the microscope. Second, a wire electrode is placed above the pressure-limiting aperture, and it is shown that enough signal from the specimen escapes through the aperture to produced satisfactory images. These detection configurations allow the use of high specimen chamber pressures, namely, well above 200 mbar. Above this pressure level, life is fully sustainable. This report presents one example of unify the detection of signals both below and above the PLA by use of the gaseous detector device. This unification further confirms the need for unifying also the terms ASEM and ESEM into one, namely, ESEM, as proposed earlier.

Abstract: Review abstract.

key words: GDD, theory of GDD, imaging parameters; induction, displacement current, electron temperature, ion temperature, electron mobility, ion mobility, mobility, diffusion, electron diffusion, recombination, electron attachment, ionization, ionization energy, terminology, terms, discharge, amplification, gain, Townsend coefficient, Townsend factors, drift velocity, plane electrodes, parallel plates, Paschen law, primary processes, secondary processes, time response, frequency response, scintillation GDD, spectroscopy, statistics, energy resolution, Geiger-Muller counters, proportional counters, detection volume, amplification volume, electrode geometry, multi-electrode GDD, cylindrical geometry, avalanche, fast electrons, slow electrons, probe, object, gas, walls, electrons, ions, rays, slow, fast, light, specimen current, absorbed current

Abstract: A comprehensive theoretical survey and analysis of the gaseous detector device is presented. It is established that the true and correct nature of signal generated on various electrodes is induction. As long as there are moving charges in the inter-electrode space, a signal current flows in the external circuit. The theory of induced signals in general and in the ESEM, in particular, is given. It is shown that the conventional notion of "specimen absorbed current" is misleading and can lead to erroneous results in ESEM. An image can be made even if no "absorbed" current by the specimen is present. The electrical conductivity of a specimen is responsible for the after-effects of charging and image distortion in the vacuum SEM. The magnitudes of imaging parameters in ESEM are calculated, and a realistic picture is conveyed. This survey includes a detailed collection and analysis of various physical parameters such as electron and ion temperature, electron and ion mobilities, electron diffusion, recombination, electron attachment and effective ionization energy. To describe the new complex physical phenomena in electron microscopy, a new terminology is necessitated and proposed. The discharge characteristics with regard to electrode geometry, nature of gas, and electrode bias are explained. The amplification or gain characteristics of the GDD are thoroughly analyzed for various electrode geometry's. The limits and advantages are determined. Apart from the ionization GDD, the scintillation GDD is shown to have some unique advantages in performance. Considering the spectroscopy, statistics and energy resolution it is proposed that nuclear methods and instruments can be transferred and properly adapted to electron microscopy in general and, in particular, to environmental scanning transmission electron microscopy and to ESEM. Practical tips and construction details are gathered for efficient designs of GDD.

236 Klaus-Ruediger Peters, Surface Imaging of the Natural Air Interface of Hydrated Lung Tissue, Molecular Imaging Laboratory, Dept. of Radiology Biomolecular Structure Analysis Center, University of Connecticut Health Center, Farmington, CT

237 Klaus-Ruediger Peters, Introduction to the Technique of Environmental Scanning Electron Microscopy, Molecular Imaging Laboratory, Dept. of Radiology Biomolecular Structure Analysis Center, University of Connecticut Health Center, Farmington, CT


key words: induction, specimen current, absorbed current

Abstract: Further demonstration and review of the mechanism of detection by indication is presented. Salt crystals resting on a glass plate were imaged both by a flat electrode above the specimen and a flat electrode below the glass plate. The images were equivalent (the same) after inverting one of them. This proves that the conventional concept of "absorbed" specimen current, is both redundant and non-existent. Instead, detection by induction is the ever present mechanism for all specimens, conducting and insulating.


key words: review, ElectroScan, outline
Abstract: An up-date of ESEM is presented. Applications and imaging by use of the ElectroScan ESEM are included.


key words: flow field, flow properties, temperature, speed, density, pressure, PLA, gas jet, pressure gradients, Monte Carlo, gas dynamics

Abstract: The flow properties, namely, gas density, temperature and speed are presented for the case of a flat pressure limiting aperture (PLA). It is important to know the variation of these properties in the immediate neighborhood of the PLA, through which the electron beam passes and near which the specimen must be placed. The direct simulation Monte Carlo method was used. The gas jet forming through an aperture was actually imaged with gaseous detection device.


Abstract: Short review of flow field properties.


key words: flow field, flow properties, pressure PLA, gas jet, Monte Carlo, gas dynamics, sharp PLA

Abstract: Further results from a study on gas dynamics in ESEM are presented. The flow field around a conical-sharp pressure limiting aperture is analyzed by the direct simulation Monte Carlo method. The variation of flow field properties with different specimen clearances is presented. One conclusion is that the specimen surface pressure is practically unaffected by the gas flow when the specimen is placed further away than one PLA diameter. The calculation of gas density gradients along the axis of the system is needed for the determination of mass density and electron scattering.


key words: scintillation, GDD, gaseous scintillation, secondary electrons, SE, control specimen

Abstract: An alternative way to detect secondary electrons in a gaseous environment is by use of the gaseous scintillation that accompanies an electron avalanche. Generally, apart from ionization we also have electron excitation as the electrons collide with gas molecules in the multiplication process. The gaseous scintillation can be detected with a suitable light pipe/PMT (photomultiplier) system. Results showing definite SE image are presented. It was found that the SE images could also be obtained at TV scanning rates, which shows that the GDD frequency response is very broad. This is consistent with short electron transit times previously calculated.


key words: universal ESEM, flow field, flow properties, pressure PLA, gas jet, pressure gradients, Monte Carlo, gas dynamics, sharp PLA, beam transfer, transition region, electron diffusion, mass thickness, noise propagation, solid scintillating BSED, charge neutralization, low vacuum SEM, low voltage SEM, signal-to-noise ratio, SNR, detector efficiency, E-T detector, SE detector, surface charge accumulation, critique, review, critical issues, grids, YAG, YAP, cathodoluminescence, CL, x-rays, multi-electrode GDD

Abstract: This is both a review and a survey into some critical issues of the environmental scanning electron microscope (ESEM). Some new concepts and designs are also presented. An attempt to unify various detection modes is made. In ESEM, the gas flow around the main pressure limiting aperture establishes a density gradient
through which the electron beam passes. Electron beam losses occur in this transition region and in the uniform gas layer above the specimen surface. In the oligo-scattering regime, the electron distribution consists of a widely scattered fraction of electrons surrounding an intact focused probe. The secondary electrons are multiplied by means of gaseous ionization and detected both by the ionization current and the accompanying gaseous scintillation. The distribution of secondary electrons is governed by the applied external electric and magnetic fields and by electron diffusion in the gas. The backscattered electrons are detected both by means of the gaseous detection device and by solid scintillating detectors. Uncoated solid detectors offer the lowest signal to noise ratio especially under low beam accelerating voltages. The lowest pressure of operation with uncoated detectors has been expanded by the deliberate introduction of a gaseous discharge near the detector. The gaseous scintillation also offers the possibility of low noise detection and signal discrimination. The “absorbed” specimen current mode is re-examined in the conditions of ESEM and it is found that the current flowing through the specimen is not the contrast forming mechanism: it is all the electric carriers in motion that induce signals on the surrounding electrodes. The electric conductivity of the specimen may affect the contrast only indirectly, i.e., as a secondary, not a primary process. The ESEM can operate under any environment including high and low pressure, low or rough vacuum and high vacuum; it also operates at both high and low beam accelerating voltage, so that it may be considered as the universal instrument for virtually any application previously accessible or not to the conventional SEM.

key words: review, universal ESEM, operational range, parameters, ablation, spectrometry, mass spectrometer, x-rays, skirt, transition, Mach disk, SE, BSE, Cl

Abstract: This is a review of ESEM whereby the main principles and instrument design considerations are unified in order to define the range of various operational parameter as we vary pressure. It is shown that the environmental scanning electron microscope is the natural extension of the scanning electron microscope. The former incorporates all of the conventional functions of the latter and, in addition, it opens many new ways of looking at virtually any specimen, wet or dry, insulating or conducting. The environmental scanning electron microscope is characterized by the possibility of maintaining a gaseous pressure in the specimen chamber. All operational parameters can be varied within a range which is a function of pressure. It can be used with all types of gun and basic modes of detection and, hence, it can be applied to both morphological and to microanalytical studies. It has opened many novel ways of looking at specimens and phenomena not previous accessible with scanning electron microscopy. A model for specimen charge distribution and dissipation is proposed. The interface of a mass spectrometer by sampling the gas flowing through the PLA is suggested; this would give rise to high resolution ablation mass spectrometry. An outline of present approaches to the problem of electron skirt in microanalysis is presented.

key words: field emission, wafers, electronic devices, line width measurement, inspection, testing, low keV, Nikon Corporation, Critical Measurement, applications

Abstract: This is a review with particular emphasis on the application of ESEM technology to the examination of electronic devices. Because of its universal capabilities, ESEM is ideally suited for inspection and testing, in general. In particular, microelectronic devices can now be studied faster, better and more reliably, or even in ways not previously feasible. For wafer pattern measurements, the high resolutions required need not be compromised by the use of a very low keV incident beam. It is much better to use rather a compromise voltage of, say, 3 keV to avoid specimen damage and yet to allow a high resolution. At the same time, specimen charging is avoided by maintaining a gaseous pressure around 100-200 Pa. A dedicated instrument with a field emission gun has been developed for linewidth measurements (Critical Dimension Measuring SEM, Nikon Corporation). Apart from inspection and testing, ESEM has potential for a wide range of applications in microprocess industry and research, because the natural surface of a specimen can be placed directly under the beam. There seems only the need to control the amount and nature of gas for each particular application. In fact, the very presence of a
controlled gaseous environment has opened up many new possibilities for beam-specimen and gas-specimen interactions. Electron beam-induced chemical reactions and applications to direct writing and associated processes can be given a new impetus. Resist materials and processing techniques can also be studied in a new way. Both etching and carbonaceous depositions have been observed and it is envisaged that these processes can be used in a controlled way during imaging to monitor those processes and to put them to practical use. Electron lithography and micro fabrication are yet to see the benefits of ESEM. Hot and cold stages can be incorporated in the microscope for studies of materials in situ at high, intermediate and low temperatures. Already, several studies on soldering techniques with this technology have appeared in the literature, and transition phases of materials from very low to very high temperatures can be readily monitored. The range of ESEM uses can only be underestimated at this early stage of development.


Abstract: A universal system of detectors can now be incorporated in ESEM so that the complete pressure range from high pressure to high vacuum can be used. The GDD is integrated with solid scintillating materials together with an optimized gas dynamics system. An array of electrodes ( grids and apertures) serve in the detection, separation and control of various signals. They are combined with highly efficient scintillating materials and/or light pipes. This system should be incorporated at the lower part of an electron optics column. Thus, all main modes of detection can be represented. Secondary (SE) and backscattered (BSE) electron signals, cathodoluminescence (CL) and x-ray microanalysis can be practiced at any pressure. In addition, a mass spectrometer can be interfaced for analysis of the gas flowing through PLA1. By directing the electron beam at the feature of interest, ESEM produces an ablation mass spectrometer with very high resolution and sensitivity, in an equivalent but much better system than that used with a laser beam.


Abstract: An outline is presented of the first commercial environmental scanning electron microscope (ESEM) made by ElectroScan Corporation. A concise description of this instrument and its operation, from a user’s perspective, is given. More specifically, the description includes the electron optics, pressure stages and control, detection modes, resolution and ancillary equipment.


Abstract: Two comprehensive and updated lists of publications on environmental scanning electron microscopy are compiled. One list contains mainly those papers dealing with the development and instrumentation, while the other deals with the applications of the technique. A brief introductory summary of the field is presented.

C.E. Jordan and A.R Marder , A Model For Galvanneal Morphology Development The Physical Metallurgy of Zinc Coated Steel.

Abstract: Cross-sectional and planar views of galvanneal coatings were studied to characterize morphology development. Cross-sectional analysis of coatings annealed under different time-temperature conditions showed the formation of three distinct morphological coating types. The morphology types were classified as Type-0, Type-1, and Type-2 and represent an under alloyed, a marginally alloyed, and an over alloyed coating structure, respectively. The structures were analyzed to quantify the chemistry associated with each morphology. Planar observation of the coatings during annealing was performed in-situ in an environmental...
SEM. Burst-like structures were found to form during annealing, and the role of bath aluminum content on their formation was studied. From these results a phenomenological model for galvanneal morphology development is proposed.


Key Words: Analytical Techniques, Environmental Scanning Electron Microscope (ESEM), Fiber Morphology, Fiber Structure, Microscopy, Nondestructive Testing, Surface Examination

Abstract: Use of the environmental scanning electron microscope (ESEM) for surface examination and observation of textile materials under varying conditions is explored. The ESEM allows imaging of specimens without coating and drying, as well as the observation of dynamic phenomena. Examples of textile structures viewed with the ESEM are shown, including time series images of fabric absorption and structure change under wetting and heating. The advantages of ESEM over regular scanning electron microscopy (SEM) are also discussed.


Abstract: An image analysis technique, called image intensity matching, is developed and is shown to be suitable for studying the complex sub-pixel deformation on ESEM images. The technique is general but is applied here to dimensional changes that occur as cement past shrinks. The technique is based on a minimum mean square error criterion. The relationship between the present technique and the maximum cross correlation method is analyzed mathematically, and the latter is shown to be a special case in the minimum mean square error criterion. Noise is removed by a normalization process. The optimal window size is analyzed. The deformation parameters (for the problem in two dimensions) of rigid body translation, expansion or shrinkage, shear, and rotation in a deformed image can be determined. The resolution is calibrated using a simulated image shift and is shown to be about 0.2 pixel.

253 P. Forsberg and P. Lepoutre, ESEM Examination of Paper In High Moisture Environment: Surface Structural Changes and Electron Beam Damage. Scanning Microscopy 8 (1)

Key words: Wood Fibers, paper, fiber-rising, electron microscopy, surface properties, contact angle, supercalendered paper (SC), and light-weight coated paper (LWC).

Abstract: Supercalendered and coated papers (SC and LWC) were examined using and environmental scanning electron microscope (ESEM). Moderate structural surface changes were observed as water condensed on the surface in a high moisture environment. The changes were fully or partially reversible depending on the sample origin. A wide range of contact angles could be observed when condensing water on uncoated wood fibers. While there was no visible indication or irradiation damage on the commercial paper samples examined nor on mechanical pulp fibers, attempts to look at chemical pulp fibers during wetting to examine fiber swelling were unsuccessful because of very rapid irradiation damage.

254 P. Forsberg and P. Lepoutre, ESEM Examination of the roughening of paper in high moisture environment. Presented at the 1993 PTS Symposium in Munich, Germany

Key Words: Fibers, Paper, Fiber-rising, Electron microscopy, Surface properties, LWC, SC, Gloss

Abstract: Supercalendered filled (SC) and light weight coated (LWC) papers were examined using an environmental scanning electron microscope (ESEM). Large structural surface chances were observed during condensation of water on the surface in a high moisture environment. The changes were fully or partially reversible upon drying depending on the sample origin. In the case of LWC papers, underlying fibers protrude and appear extensively swollen. Careful examination of the base stock confirmed that all Kraft fibers were collapsed into ribbons but, during wetting, there was no evidence of ribbon-to-tube shape change of individual fibers. Swelling, which could be very substantial since the WRV indicates a potential swelling of more than 100% took place from flat ribbon to swollen ribbon. Upon drying the swollen ribbons shrunk back to their original
dimensions. Once supercalendered, the LWC base stock did show evidence of some collapsed mechanical fiber segments returning, irreversibly, to their uncollapsed state. In the case of SC papers, significant surface roughening during wetting was clearly seen but there was no indication of ribbon-to-tube shape changes either. Roughness and gloss changes measured after wetting on a laboratory printing press agreed well with ESEM observations.


Key Words: Environmental Scanning electron microscopy, individual fibers, mechanical properties, digital image correlation, recycling, tensile, fiber failure

Abstract: Relationships between virgin fiber types, fiber production techniques and mechanical properties are well understood and documented (e.g.1). For recycled fibers, however, these same relationships are confounded by unquantified degrees of further mechanical and chemical damage (2). To gain a more comprehensive understanding of the impact of recycling on secondary fibers, the potentially deleterious effect of recycling upon fiber mechanical properties must be quantified. In this study individual fibers, both recycled and virgin were tested in tension within and environmental scanning electron microscope. Fiber failure characteristics of both recycled and virgin fibers are reported. The influence of both natural and processing induced gross defects were seen to be highly influential in controlling mechanical behavior. The importance of defects and the implications for modeling the behavior of fibers is explained.

256 G.D. Danilatos, Introduction to the ESEM, Instrument Microscopy Research and Vol. 25, #5&6

Key Words: Environmental SEM, Scanning electron microscope, Gaseous detection, Differential pumping

Abstract: An outline is presented of the first commercial environmental scanning electron microscope (ESEM). A concise description of this instrument and its operation, from a users perspective, is given. More specifically, the description includes the electron optics, pressure stages and control, detection modes, resolution, and ancillary equipment.


Key Words: ESEM, Hydrophobic, Hydrophilic, Water Condensation, Water droplets

Abstract: With the ability to perform dynamic experiments in the environmental electron microscope (ESEM), the evaluation of microporous polymer membranes via a scanning electron microscope has advanced beyond morphological and elemental analysis. By adjusting sample temperature and environmental chamber pressure, the process of condensing water onto the porous membrane surface can be achieved. In doing so, assessments about the uniformity of wetting in hydrophilic membranes can be obtained based on how the liquid water spreads. Variations in the shape of condensed water droplets formed on non-water wetting structures will reflect the degree of hydrophobicity. This technique has proven useful in the characterization of hydrophobic spots on chemically modified hydrophilic structures and the dynamic examination of irregular wetting patterns in naturally hydrophilic membranes.


Key Words: Conservation, Water, Relative Humidity, Cracking

Abstract: The preservation of nineteenth-century albumen prints is of great concern to collection managers and to conservators of photographic materials. In the field of art conservation preservation techniques incorporating aqueous treatments are often used to enhance the long-and short-term stability of historical artifacts.
or art objects. In a study of the interaction of water with albumen photographs, experiments were carried out in the ESEM to follow the real time effects of water on the prints. The experiments were designed to observe the effects of a range of relative humidities and liquid water on samples of expendable historic albumen prints, utilizing the advantages of imaging in the presence of water vapor. All albumen photographs exhibit a fine network of cracks in the albumen protein layer. Average crack width is approximately 10 am, as observed in the ESEM, a 4.25-fold increase in the width of a single crack (at 50% RH), viewed normal to the surface, resulted from a single controlled excursion to high relative humidity and swelling and shrinkage in thickness, and a 5% and 9% swelling and shrinkage along the width of a fragment of the albumen/image layer when the sample was immersed in water and dried. The visual information gained through the use of the ESEM helped to focus a materials investigation and served as a foundation for a study which shows that aqueous treatment causes increased cracking of both unsupported albumen and the albumen/image layer in prints.


Key Words:  Apherulite, Polymer etching, Deinking, Paper recycling

Abstract:  This article describes two cases in which the advantages of the ESEM have been exploited in unanticipated ways. First, we have found that etching occurs as the electron beam scans the surface of uncoated polymers in the ESEM. The surface topography caused by etching, as seen in the ESEM images, reflects the morphology of crystalline structures in the polymers. This technique has been valuable in the study of such textures in polymers. The second applications is related to our use of the ESEM in support of research on the deinking of paper. In this effort we have learned that unconventional contrast mechanism can be used during ESEM imaging to distinguish between inked and non-inked areas of newsprint. Under usual operating conditions, ESEM imaging does not distinguish between inked and non-inked areas. However, at relatively low sample chamber pressures the non-inked areas appear brighter than inked areas in ESEM images.


Key Word:  Environmental Scanning Electron Microscopy, Algae, Fungi

Abstract:  Microorganisms, including bacteria, fungi, protozoa, and micro algae, are composed, predominantly of water which prohibits direct observation in a traditional scanning electron microscope (SEM). Preparation for SEM requires that microorganisms be fixed, frozen or dehydrated, and coated with a conductive film before observation in a high vacuum environment. Sample preparation may mechanically disturb delicate samples, compromise morphological information, and introduce other artifacts. The environmental scanning electron microscope (ESEM) provides a technology for imaging hydrated or dehydrated biological samples with minimal manipulation and without the need for conductive coatings.

Sporulating cultures of three fungi, a Sperrgillus sp., Cunninghamella sp., and Mucor sp., were imaged in the ESEM to assess usefulness of the instrument in the direct observation of delicate, uncoated, biological specimens. Asexual sporophores showed no evidence of conical displacement or disruption of sporangia.

Uncoated algae cells of Euglena gracilis and Spirogyra sp. were examined using the backscatter electron detector (BSE) and the environmental secondary electron detector (ESD) of the ESEM. BSE images had more clearly defined intracellular structures, whereas ESD gave a clearer view of the surface. E. gracilis cells fixed with potassium permanganate, Spirogyra sp. stained with Lugol’s solution, and Saprolegnia sp. fixed with osmium tetroxide were compared using BSE and ESD to demonstrate that cellular details could be enhanced by the introduction of heavy metals. The effect of cellular water on signal quality was evaluated by comparing hydrated to critical point dried specimens.

260  L.M. Egerton-Warburton, B.J. Griffin, and J. Kuo  Microanalytical studies of Metal Localization in Biological Tissues by Environmental SEM. Microscopy Research and Technique 25:406-411

Key Words:  Bulk frozen hydrated samples, Eucalyptus, Aluminum, Manganese
Abstract: The presence and distribution of Al and Mn in floral and seed tissues of eucalyptus from Al-contaminated soils was analyzed using energy-dispersive X-ray microanalysis (EDS) in an environmental scanning electron microscope (ESEM). EDS by ESEM determined the distribution of elements between tissue types was suitable for intact samples or those with lower available moisture or intact specimens. The analytical technique was not appropriate for highly vascular samples. Other factors influencing the detection of elements within the tissues. EDS-detectable levels were significantly correlated with tissue concentrations determined by atomic absorption spectrophotometry for Mn but not for Al.

261 P.J.R. Uwins, M. Murray, and R.J. Gould Effects of Four Different Processing Techniques on the Microstructure of Potatoes: Comparison with Fresh Samples in the ESEM Microscopy Research and Technique 25:413-418

Key Words: Potato microstructure, Freeze-substitution, Chemical fixation, Fresh unprocessed potato samples

Abstract: Four common scanning electron microscope (SEM) processing techniques involving freeze-substitution and chemical fixation were compared with fresh unprocessed samples imaged in an environmental scanning electron microscope (ESEM) using small pieces of potato tubers as test specimens. Potato tubers were chosen for this investigation because of their high moisture content and, consequently, the common need for extensive processing for conventional, high vacuum SEM imaging. ESEM results showed that the fresh unprocessed specimens were essentially unaltered, showing clear potato cell structure, morphology, and cell content. However, processed networks of material stretching across the surface of cells. These structures may represent fibrillar material or may be artifact caused during processing. Chemical fixation almost entirely destroyed the microstructure of these potato samples.

262 L.C. Gilbert and R.E. Doherty, Using ESEM and SEM to compare the Performance of Dentin conditioners Microscopy Research and Technique 25:419-423

Key Words: Environmental Scanning Electron Microscope, Smear Layer, Dentin Bonding, Tenure, Scotch Bond 2, Syntax, Universal Bond 3, Teeth, Dentistry, Adhesion

Abstract: A comparison of four dentinal conditioners was performed utilizing a traditional scanning electron microscope (SEM) and the new technology, the ElectroScan environmental scanning electron Microscope/ESEM Both ESEM and SEM analysis verified current theorized mechanisms of adhesion to dentin surfaces look like. Increases in information from the "surfaces" of uncoated specimens and the reduction in specimen preparation time were associated with ESEM analysis.


Key Word: Biocorrosion, Sulfate-reducing bacteria, Biofilm, Desulfovibrio, Electron microscopy

Abstract: The biofilm attributed to Desulfovibrio vulgaris growing in the presence of ferrous metals was examined with an environmental scanning electron microscope. This novel microscope produced images of iron sulfide colloids and other iron containing structures that had not been reported previously. A plaque composed of iron sulfide enveloped the surface of the corroding metal while crystals containing magnesium, iron, sulfur, and phosphorus were present in the culture where corrosion was in progress. A structure resembling the tubercule found in aerobic corrosion was observed on stainless steel undergoing biocorrosion and the elements present in this structure included sulfur, iron, chloride, calcium, potassium, and chromium.

264 L.F. Keyser and Ming-Taun Leu, Morphology of Nitric Acid and Water Ice Films Microscopy Research and Technique 25:434-438

Key Words: ESEM, Surface reaction, Polar stratospheric clouds

Abstract: Ice films have been used to simulate stratospheric cloud surfaces in order to obtain laboratory data on solubility’s and heterogeneous reaction rtes. to obtain intrinsic uptake and surface reaction probabilities
which can be applied to atmospheric models, it is necessary to carefully characterize these films. In the present study, environmental scanning electron microscopy (ESEM) is used to study thin films of both water ice and nitric acid ice near the composition of the trihydrate. The ices are formed by vapor deposition onto aluminum or borosilicate-glass substrates cooled to about 200º K. Micrographs are recorded during the deposition process and during subsequent annealing at higher temperatures. The results show that the ice films are composed of loosely consolidated granules, which range from about 1 to 20 am in size at temperatures between 197º and 235º K. Cubic water ice is sometimes observed at 200º K and converts to the hexagonal form at slightly higher temperatures. The loose packing of the granules confirms the high porosity’s of slightly higher temperatures. The loose packing of the granules confirms the high porosity’s of these films obtained from separate bulk porosity measurements. Average surface areas calculated from the observed granule sizes range from about 0.2 to 1m 2g-1 and agree with surface areas obtained by gas-adsorption (BET) analysis of annealed ice films. For unannealed films, the BET areas are about an order or magnitude higher than the ESEM results and imply that the unannealed ices contain microporosity which is lost during the annealing process. The present results have important implications for the extraction of intrinsic reaction probabilities from laboratory rate data.


Key Words: Microscopy, Groundwater, Pollution, Radioactive Waste, Transport, Remediation

Abstract: Environmental colloids are toxic or radioactive particles suspended in ground or surface water. These hazardous particles can facilitate and accelerate the transport of toxicants and enhance the threat to humans by exposure to pathogenic substances. The chemical and physical properties of hazardous colloids have not been well characterized nor are there standard colloid measurement of their size distribution, zeta potential, chemical composition, adsorption capacity, and morphology. The environmental scanning electron microscope (ESEM) by ElectroScan, Inc., analyzes particle sizes, composition, and morphology. It is also used in this study to identify the attachment of colloids onto packing or rock surfaces in our development of a colloid remediation process. The ESEM has confirmed the composition of groundwater colloids in our studies to generally the same material at the surrounding rock. The morphology studies have generally shown that colloids are simply small pieces of the rock surface that has exfoliated into the surrounding water. However, in general, the source and chemical composition of ground water colloids is site dependent. We have found that an ESEM works best as a valuable analysis tool within a suite of colloid characterization instruments.


Key Words: Cotton, Milkweed, Kapok, Polypropylene, Biocomponent fiber, Biconstituent fiber, Adsorption, Absorption, Capillary action

Abstract: Oil sorption capacities of various natural and man-made fibrous sorbents were compared in a simulated seawater bath containing oil. Natural sorbents such as milkweed, kapok, cotton, and wool showed higher sorption capacities than man-made sorbents such as polyester, polypropylene, viscose rayon, nylon 6, nylon 66, and acetate. Sorption capacities of the natural sorbent were over 30g oil/g fiber. No definite advantages were observed using man-made bicomponent and biconstituent fibers over regular man-made fibers with respect to their sorption capacity.

Analyses of sorption mechanisms using an environmental scanning electron microscope revealed that an oil deposit disappeared from the fiber surface after a certain time interval in milkweed, kapok, and cotton. This suggested the sorption of oil in these fibers occurred through capillary action, probably due to their hollow lumens. Contrarily, adsorption, a surface phenomenon, would be the most prominent mechanism for oil sorption of wool fibers due to large amounts of surface wax, irregular scaly surfaces, and crimp. Effects of both adsorption and absorption were shown in the oil sorption of man-made fibers, depending upon the type and shape of the sorbent. Dumbbell like oil deposits were seen on the fiber surface in certain oleophilic man-made fibers, because of a partial wetting of oil on the fiber surface. For some hydrophilic man-made fibers such as polyvinylalcohol and
copolymer of isobutylene-maleic anhydride, the physical configuration of the fiber was a decisive factor in determining oil sorption capacity of the sorbents.

267 Chao Lung Hwang, Ming Liang Wang, and Shuke Miao Proposed Healing and Consolidation Mechanisms of Rock Salt Revealed by ESEM Microscopy Research and Technique 25:456-464

Key Words: Crushed rock salt, ESEM, Deformation, Healing mechanism, Consolidation mechanism

Abstract: The grain boundary heading behavior of crushed rock salt was mainly studied by employing the environmental scanning electron microscope (ESEM) to study the consolidation mechanism of rock salt backfill. Dedicated miniature round rock salt specimens were prepared for observation of the water trapping effect by using a cold stage in the ESEM to reach saturation conditions. Comparable high pressure pellets were prepared for measuring the crystal growth. Consolidation tests using materials made at different pressures and containing different moisture levels were conducted in order to construct the proposed mechanism. Direct observation of specimens in the ESEM resulted in viewing water trapped on the surface and the formation of a water meniscus between two particles. The concentration of brine at the grain boundary was observed as contributing to the amount of recrystallization process may be redrawn. The amount of water therefore has a great effect on the consolidation of rock salt and is possibly due to the sliding rotation, or crushing of the contact zone of the granular material. From such a study, tentative healing and consolidation mechanisms can be deduced.


Key Words: ESEM, Liquid hydrocarbons, hydrocarbon reservoirs, Clay minerals, Chlorite, Illite/smectite, Calcite, Fluid sensitivity

Abstract: The environmental scanning electron microscope (ESEM) has been used to image liquid hydrocarbons in sandstone’s and oil shales. Additionally, the fluid sensitivity of selected clay minerals in hydrocarbon reservoirs was assessed via three case studies: HCl acid sensitivity of authigenic chlorite in sandstone reservoirs, freshwater sensitivity of authigenic illite/smectite in sandstone reservoirs, and bleach sensitivity of a volcanic reservoir containing abundant secondary chlorite/corrensite. The results showed the suitability of using ESEM for imaging liquid hydrocarbon films in hydrocarbon reservoirs and the importance of simulating in situ fluid-rock interactions for hydrocarbon production programs. In each case, results of the ESEM studies greatly enhanced prediction of reservoir/borehole reactions and, in some cases, contradicted conventional wisdom regarding the outcome of potential engineering solutions.


Key Words: Coatings, Copper thick films, Crystallization, Hydroxyapatite, Propellants

Abstract: In this article we describe a number of studies involving the direct observation of microstructural evolution. In general these investigations were carried out to establish the mechanistic paths involved. The materials studied range from fibers being evaluated for use in high temperature ceramic composites to energetic materials used as propellants. In particular we discuss the room temperature imaging of materials difficult to image by conventional means and the use of the chamber atmosphere to influence microstructural evolution. Imaging of hydroxyapatite formed by chemical means is briefly described as an example of a difficult microstructure. Microstructural evolution during calcium aluminate cement hydration relies on the chamber atmosphere to control moisture loss from the hydrating specimens. In some instances microstructural evolution with heating occurred independently of the chamber atmosphere. Grain growth in PZT films formed by sol-gel processes depends strongly on temperature but does not appear to depend on the chamber atmosphere. This is also the case for the combustion of nitroamine propellants in that their combustion’s does not depend on access to an indirect role in determining microstructure. However, the mechanistic path driving microstructural evolution in copper-based inks used as conductive paths on electronic substrates is atmosphere dependent. These inks are formulated from copper powder, glass, and an organic binder, and the interaction of the binder with an oxidizing atmosphere allows it to be butted out before significant interaction occurs between the copper powder and the
glass. Finally, the microstructural variations during the oxidation of structural composites at high temperature were used to allow assessments of their likely failure mechanisms.

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Key Words:  Non-destructive techniques , SEM , FE-SEM

Abstract:  Process visualization can be a very powerful tool for understanding dynamic processes. Process visualization requires a non-destructive inspection will be described and compared. The applicability of these non-destructive inspection methods to process visualization will be compared and contrasted. Particular attention will be paid to a recent development in this area, the environmental scanning electron microscope (ESEM) which is inherently a non-destructive inspection technique with the advantages of electron microscopy for superior magnification and depth of field capability. The ESEM offers a unique platform for process visualization studies. The majority of process visualization is currently done using optical microscopes with hot stages for observing morphological effects top down (optical microscopes) or from the side (contact angle). Major limitations of these optical methods include lack of magnification, poor depth of field, and clouding of optics.

Process visualization is best carried out utilizing a non-destructive technique, such as the ESEM, since invasive sample preparation techniques such as conductive coatings alter the sample and make interpretation more difficult. Common process variables such as thermal profiling and the effect of ambient conditions have been examined using the ESEM. Other process variables that could be of interest in the future will be discussed. There are limitations in the ability of the ESEM to reproduce actual process conditions, such as pressure and mass flow rate trade-offs. The ESEM can also be combined directly and indirectly with other analytical techniques to determine the composition of the sample and/or by products of a reaction that is being monitored.

This paper will serve as an overview and introduction for several papers which deal in depth with specific process visualization applications which utilize the ESEM. A series of illustrative examples of previous work will be referenced and briefly discussed. The examples will emphasize the importance of non-destructive testing techniques in material science and semiconductor applications. The application window of the ESEM for process visualization will be explored, including trade-offs in process conditions that can be examined. Observation of dynamism processes include examples such as corrosion studies of various materials such as stainless steel and thermal studies of industrially relevant processes such as ceramic processing, soldering, and sealing.

Morphological and compositional process visualization applications will be presented. An example of morphological applications observed is solder reflow and inter-metallic formation as a function of the materials used and the atmosphere during processing. Morphological coupled with compositional applications include monitoring outgassing products from solder paste and Ag/glass die attach material.

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Key Words:  Solder joining , Ambient effects , Solder oxidation , Solder microstructure , Solder morphology , Atmosphere effects , Solder

Abstract:  The ESEM is ideally suited to study soldering processes. We have used it to observe solder reflow and joining in ambient gases. It reproduces effects of atmospheric pressure reflow in a hot stage light microscope, but with much better clarity and depth of field. Compared to a regular SEM, the ESEM offers advantages of atmosphere control and ability to observe the solder samples without carbon or gold coating. These coatings could interfere with the oxidation/reduction reactions which occur at the solder/ambient gas interface. Very thin surface films, especially oxide layers, dramatically influence the flow of liquid solder and the ability of solder to wet or join to another surface. Fluxless processes in particular are ideally suited for study in the ESEM. We have used the ESEM to observe dynamic fluxless soldering and have recorded events on videotape for later stop-action still pictures and slow motion photography. Examples of these processes are shown to illustrate the ESEM capability. Included are solder deformation structure, balling reflow of eutectic solder in hydrogen, balling reflow of eutectic solder in nitrogen, joining of two solder disks in nitrogen, and dynamic melting and freezing of an off-eutectic dendritic alloy. All of these are observed in the absence of flux.
Abstract: Copper samples, hot solder (eutectic) dipped and thermally aged, were cross-sectioned and placed in and environmental scanning electronic microscope (ESEM). While in the ESEM the samples were heated for ~2.5h at 170ºC to stimulate the growth of additional Cu/Sn inter-metallic compound. The intent of the study was to obtain a continuous real-time videotape record of the diffusion process and compare the observations to static SEM images reported to represent long-term, naturally aged inter-metallic growth. The video obtained allows the observation of the diffusion process and relativistic growth phenomena at the Cu, Cu₃Sn,Cu₆Sn₅, and solder interfaces as well as effects on the bulk Cu and solder. Effects contrary to earlier reports were observed; for example, growth rates of Cu₃Sn were found to greatly exceed those of Cu₆Sn₅.

Key Words: Inter-metallic compounds, ESEM, SEM

Abstract: This paper provides a summary of some in situ, high-resolution studies of solder spreading reactions on microelectronic circuit metallizations. Experiments are described that focus on the use of the environmental scanning electron microscope, or ESEM. Those experiments have been complemented by studies using optical hot-stage microscopy and have been supplemented by additional analytical tools such as energy dispersive x-ray microanalysis, Auger and ESCA to evaluate chemical processes. Two general results from dynamic scanning electron microscope observations are that 1) molten solder alloys undergo a segregation process during spreading in which a "precursor" film spreads in advance of the bulk solder and 2) the spreading front, which may be enriched in Sn from Pb-Sn or Bi-Sn solders, or in from Pb-In solders, spreads along high-reactivity features of the metallization surface as a reacting "precursor" film. A third observation from these tests is that, in unconfined geometries, the reactive metallization, if not sufficiently thick, can be dissolved by the solder before wetting is complete, leading to de-wetting of the solder. Both the kinetics and extent of spreading of these films and the relationship of these phenomena to the commonly measured contact angle and wetting forces are currently being examined by a range of complementary techniques. Information gathered in these studies shows that process temperature as well as composition, reactivity, and relative amounts of the solder and metallization species should all be factors of interest to the those responsible for control of soldering processes.

Key Words: Metallurgical reactivity, Inter-metallics, Precursor films, Surface energy

Abstract: The significance of the ElectroScan environmental scanning electron microscope (ESEM) as a processing tool from studying dynamic morphological changes under controlled temperature/atmosphere conditions was evaluated. The ability to observe dynamic processes in situ, which cannot be achieved by other means, is critical to understanding microstructural formation.

Processing of printed copper thick films on ceramics was used as a test case, wherein morphological changes associated with the steps of organic binder removal and sintering of copper particles were observed/examined in real time. Good agreement was seen between microstructures obtained in the ESEM and those achieved in a belt furnace when similar process variables were used. When processed in atmospheres which were proven to induce sintering in a conventional belt furnace, tapes in real time. Determination of critical event temperatures was achieved-that is, binder burnout occurring between 270º and 350ºC, onset of oxidation at 520 ºC, and sintering starting at 770 ºC.

It was thus verified that the microstructural changes during the copper thick film sintering process can be observe in situ using an ESEM.
Bibliography


Key Words: ESEM, In situ, Microelectronics, Stainless steel tubing

Abstract: An ElectroScan ESEM was used for in situ corrosion studies on cold drawn electropolished 316L stainless steel tube surfaces in the as-received and passivated conditions. Corrosion product was removed as it formed and the tube surface was viewed before, during, and after corrosive attack. The corrosion process was followed in situ, and the sample features most susceptible to corrosion (draw lines, inclusions, etc.) were identified. In addition, X-ray photoelectron spectroscopy (XPS) was used to study the changes in surface chemistry after corrosive attack. This information provided clear evidence of relevant corrosion mechanisms and relative corrosion susceptibility.

276  G.D. Danilatos, Bibliography of Environmental Scanning Electron Microscopy Microscopy Research and Technique 25:529-534

Key Words: ESEM, Survey of ESEM, Review of ESEM, Development of ESEM, Uses of ESEM, Applications of ESEM

Abstract: Two updated lists of publications on environmental scanning electron microscopy are complied. One list contains mainly with the applications of the technique. A brief introductory summary of the field is presented.

277  B. Caveny, Cement Hydration Study Using the Environmental Scanning Electron Microscope ICMA Proceedings

Key Words: cement

Abstract: This paper will detail a brief study of hydrating cement using the ESEM. The ESEM is capable of working at much higher pressures than conventional SEM, thus allowing wet samples to be examined in a more natural environment. A class H oil well cement will be studied in several degrees of hydration’s and ESEM photomicrographs will be shown of the various microstructures observed.

278  Robert Pope and Raymond W. Scheetz, Dynamic Events Related to Humidity Changes on Botanical Samples Imaged with the Environmental SEM, Dept. of Biological Sciences, University of Southern Mississippi, Hattisburg, MS 39406-5018

Key Words: humidity, botanical samples

Abstract:


Key Words: microbiologically influenced corrosion, ESEM, SEM

Abstract: A newly developed Environmental Scanning Electron Microscope (ESEM) coupled with an energy dispersive x-ray spectrometer (EDS) was used to characterize the topography and chemical composition of wet biofilms and corrosion products on metal surfaces in addition to spatial relationships between microorganisms, substratum and corrosion layers. Case studies are presented to demonstrate the applicability and advantages ESEM/EDS technology in the investigation of microbiologically influenced corrosion (MIC) as compared to traditional methods.
Bibliography

280 P.A. Wagner, B.J. Little, R.I. Ray, Biofilms: An ESEM Evaluation of Artifacts Introduced During SEM Preparation, Naval Oceanographic and Atmospheric Research Laboratory, Stennis Space Center, MS 39529-5004

Key Words: Biofilm, scanning electron microscope, environmental scanning electron microscope

Abstract: Descriptions of biofilms and their elemental compositions based on scanning electron micrographs and energy dispersive x-ray analysis cannot be related to the original condition of the biofilms on the surface. Solvent replacement of water removes extra cellular polymeric material and reduces the concentration of elements bound within the biofilm. In the wet state, bacteria and micro algae are enmeshed in a gelatinous film that is either removed or dried to a thin inconspicuous residue during sample preparation for scanning electron microscopy. The Environmental Scanning Electron Microscope (ESEM) provides a fast, accurate image of biofilms, their spatial relationship to the substratum and elemental composition.


Key Words: Electron-Sensitive Resist, sub-micron IC’s,

Abstract: State-of-the-art SEM metrological approaches are discussed to elucidate inherent deficiencies that prevent accurate assessment of image fidelity in the production or inspection of sub-micron IC’s, especially on the resist level. The new technique of Environmental SEM is demonstrated to allow topographic contrast generation, unaffected by surface charging, for SAL-601.


Key Words: cement, cryogenics

Abstract:


Key Words: gallstones

Abstract: The purpose of this experiment was to study fresh gallstones by Environmental Scanning Electron Microscope (ESEM) to determine if dehydration affects gallstone Ca salt morphology.


Key Words: ultrasound, polymer erosion

Abstract: In vitro methodology has been developed to investigate the effects of therapeutic ultrasound on polymer erosion. Enhancement in the rate of polymer erosion was demonstrated using therapeutically acceptable levels of ultrasound on a model class of degradable polymers—polyanhydrides. It was found that the ultrasound enhances polymer degradation as demonstrated by the enhanced decrease in polymer molecular weight during the induction period of erosion. Additionally, morphological changes on the surface of ultrasound exposed devices were assessed by Environmental Scanning Electron Microscopy and suggested that cavitation may cause the mechanical disintegration of the polymer surface.

285 Wang Peiming, Li Pingjiang, Chen Zhiyuan, Research on the Morphology of Cement Hydrates by SEM, State Key Laboratory of Concrete Materials Research, Tongji University, Shanghai, 200092, China

June, 1995
Key Words: hydrate, morphology, hydration space, environmental pressure

Abstract: This paper throws light on the morphology change of cement hydration products under the observation of SEM. The results show that the morphologies are changed, some even significantly, with the sample grown within different free space, or under the observation of different vacuum degree to others.

286  Bill Caveny, Gant McPherson, Lance Brothers, Sudhir Mehta, Crystal Phases of Cement Paste Cured in High Temperature CO$_2$ Environment

Key Words: geothermal wells

Abstract: Completion of geothermal wells in hostile environments require laboratory studies to determine which cement blends might work best for a given set of conditions. This paper details some data obtained from blends that were cured in CO$_2$ environments at 316°C. XRDA, ESEM and light microscopy and other methods were used in the analyses.


This paper was prepared for presentation at the 66th Annual Technical Conference and Exhibition of the Society of Petroleum Engineers held in Dallas, TX, October 6-9, 1991.

This paper was selected for presentation by an SPE Program committee following review of information contained in an abstract submitted by the author(s). Contents of this paper, as presented, have not yet been reviewed by the Society of Petroleum Engineers and are subject to correction by the author(s). The material, as presented, does not necessarily reflect any position of the Society of Petroleum Engineers, its officers, or members. Papers presented at the SPE meetings are subject to publication review by the Editorial Committee of the Society of Petroleum Engineers. Permission to copy is restricted to an abstract of 300 words, illustrations may not be copied. The abstract should contain conspicuous acknowledgment of where and by whom the paper is presented.

Write Publications Manager, SPE, PO Box 833836, Richardson, TX 75083-3836, U.S.A.

Key Words: petroleum

Abstract: The barrier to imaging wet or oily specimens in their “native” states in a scanning electron microscope known as the Environmental Scanning Electron Microscope (ESEM). With the new features built into ESEM, the need for preparing samples with various specimen-destroying preparation techniques has been eliminated. For example, wet reservoir rocks can be imaged and analyzed in their “native” state, without drying, freezing or coating with a conductive layer, by saturating the ESEM specimen chamber with water vapor {P$_{H_2O}$=0.46 psi [3.2kPa] at 25°C [77°F]} during examination. The ESEM also allows dynamic experiments to be performed in a variety of gases at pressures up to 0.6 psi [4kPa] and temperatures up to 1000°C.

This paper presents the key features of the ESEM which distinguish it from the conventional SEM, and results of some ESEM feasibility studies important in petroleum technology such as matrix acidization, water flooding, and clay and cement hydration. The results indicate that the ESEM combined with the energy-dispersive x-ray spectroscopy is a powerful new technique capable of providing a new understanding of many exploration and production related studies not previously possible with conventional “high-vacuum” SEM microscopy.


This paper was prepared for presentation at the SPE Int’l Symposium on Formation Damage Control held in Lafayette, Louisiana, 7-10 February 1994.

This paper was selected for presentation by an SPE Program committee following review of information contained in an abstract submitted by the author(s). Contents of this paper, as presented, have not yet been reviewed by the Society of Petroleum Engineers and are subject to correction by the author(s). The material, as presented, does not necessarily reflect any position of the Society of Petroleum Engineers, its officers, or members. Papers presented at the SPE meetings are subject to publication review by the Editorial Committee of the Society of Petroleum Engineers. Permission to copy is restricted to an abstract of 300 words, illustrations may not be copied. The abstract should contain conspicuous acknowledgment of where and by whom the paper is presented.

Write Librarian, SPE, PO Box 833836, Richardson, TX 75083-3836, U.S.A.

Key Words: acid, core flood tests,

Abstract: A new laboratory procedure has been developed to study formation damage mechanisms and improve acid stimulation designs using an Environmental Scanning Electron Microscope (ESEM) coupled with core flood tests. An ESEM has the unique capability of observing a sample wet or dry, in its natural, uncoated state. The core plugs used in core flooding experiments can be observed at the same locations, before during and after treatment with workover and completion fluids, without any cleaning, drying or metal coating processes.
The direct effects of the simulation and completion fluids on the formation minerals can be seen along with any changes to the initial porosity. The ESEM is combined with energy-dispersive x-ray spectroscopy (EDS) to allow elemental analysis of precipitates or observation of changes in the elemental composition of the clay minerals.


Key Words: ESEM, Fluid/Rock Interactions, Hydrocarbon Production.

Abstract: At Texaco’s Exploration and Production Technology Department in Bellaire, TX, research is being conducted on core material using the Environmental Scanning Electron Microscope (ESEM). In the past, conventional SEM’s have proven to be a very useful tool for describing reservoir rocks. They are capable of providing details at a sub-micron scale thus revealing rock characteristics which aid in the understanding of the complex diagnostic history and petrographic properties of the rock. The ESEM allows these petrographic studies to go one step further. Because of its specialized design, the ESEM allows viewing of any sample, wet or dry, in its natural state. Also a microinjection port allows water and other fluids to be placed directly on the sample during imaging. Dynamic events such as dissolution and precipitation can be observed in real-time and recorded through the use of a VCR.

* Diagnosis refers to all of the physical, chemical and biological changes that a sediment is subjected to after a grain has been deposited but before it is metamorphosed.

# Petrographic properties are rock properties described in hand specimen and thin section analysis.


Key Words: ESEM, Environmental Scanning Electron Microscopy.

Abstract: The Environmental Scanning Electron Microscope (ESEM) is one of the most exciting new developments in the field of Electron Microscopy. The ESEM differs from conventional Scanning Electron Microscopes (SEM) by being able to examine materials including liquids and oils in their natural state with no prior sample preparation. Accessory equipment, cooling, heating and manipulating devices allow the manipulation of samples thus making it possible for the first time to image dynamic processes such as wetting, drying, absorption, corrosion, melting, crystallization, curing and fracturing at high magnification. (Author abstract) 24 Refs.


Edward M. Griffith III and G.D. Danilatos Foreword

G.D. Danilatos Introduction to the ESEM Instrument

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291b Paul Messier and Timothy Vitale, Cracking in Albumen Photographs : An ESEM Investigation

291c James H. Rask, John E. Flood, John K. Borchardt, and Greg A. York, The ESEM Used to Image Crystalline Structures of Polymers and to Image Ink on Paper

291d Scott A. Wight and Cynthia J. Zeissler, Environmental Scanning Electron Microscope Imaging Examples Related to Particle Analysis


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G.D. Danilatos, Bibliography of Environmental Scanning Electron Microscopy
Junhui Li; Pecht, M. Engel, P. A.; Chen, W. T., Dynamic investigation of thermal and sorptive effects on electronic packages -

Key Words: microscopes; scanning electron

Abstract: Temperature and relative humidity affect electrical, chemical, mechanical, and thermo-mechanical properties of microelectronic packages. Attention has been focused on the environmental effects that results in various physical failures. The scanning electron microscope is, a useful tool for visually characterizing these failures, but its use is restricted because the specimens examined must be coated. The environmental scanning electron microscope functions like a traditional high-quality scanning electron microscope, and also allows the researcher to examine unprepared, uncoated specimens. This makes it possible to view wet or moist specimens in their natural states. In this paper, several experimental efforts using the E-SEM technique are discussed. The first was a study of thermal effects on L-band microwave monolithic integrated circuits. The second investigation concentrated on adhesive properties of multi layer thin film polyimides. The third study focused on thermal and humidity cycling effects on the interfacial bonding characteristics of resin-fiber interfaces near plated-through-holes in printed wiring boards.


Key Words: microscopes; scanning electron

Abstract: Moiré fringe patterns can occur when high-frequency line arrays are observed in the scanning electron microscope. We have applied this phenomenon to local deformation measurement in a glass-fiber-reinforced plastic and in a plated-through-hole. In the GFRP, local strain measurements were made by interpreting the moiré fringe patterns over gage lengths from 10 to 30 mum at a 0-90 ply interface during tensile testing. Load shedding by the transverse ply was evident from the fringe patterns. On a cross section of a plated-through-hole, inhomogenous strains were observed.


Key Words: Backscattered Electron Detector, Environmental Cell, Specimen Charging, Atomic number contrast, Materials Characterization

Abstract: The combination of scanning electron microscope, environmental cell modification and energy-dispersive X-ray detector has been employed, as a system, in materials characterization. The system requires no specimen preparation, produces images which display high atomic number contrast together with good topography contrast, and enables different phases, detected by their atomic number contrast, to be quickly identified by X-ray analysis. The lack of specimen preparation means that analysis can be extremely rapid, with time periods as short as one minute being achieved for receiving, imaging and analyzing the specimen.


Key Words

Abstract: A Portland cement mortar was mixed onboard a recent space shuttle mission to study the effects of a microgravity environment on the production of a hardened mortar. A control sample was subsequently
prepared in the same mixer under gravity conditions on the earth’s surface. The purpose of this study is the
examination of the cement paste microstructure to determine the effects of microgravity on its development.
Optical microscopy of thin sections of mortar and scanning electron microscopy of fracture surfaces of mortar
were chosen for the analysis. Thin-section analysis suggested that the extent of aggregation of the calcium
hydroxide crystals was less under the influence of microgravity. Scanning electron microscopy suggested that the
microgravity allowed for the formation of a microstructure with a much more distinct void structure.

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(2) 213-412 (1994). Elsevier, ISSN 0927-0256

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densities in Cd$_x$Zn$_{1-x}$S.

297b J. Kohanoff Phonon spectra from short non-thermally equilibrated molecular dynamics simulations

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297d J. Hutter, H.P. Lothi and M. Parrinello Electronic structure optimization in plane-wave-based density
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environmental scanning electron micro-scope

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distortion effects on bulk modulus and band structure in Li-alloys

297g A. Fischer and A. Pyzalla-Schieck Calculation of thermal micro residual stresses in materials
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297h D. Faken and H. Johnsson Systematic analysis of local atomic structure combined with 3D computer
graphics

and pressure dependence of band gaps in GaAs and GoAs$_x$P$_{1-x}$

297j M. Sluiter Introducing distant interactions in the cluster variation method

297k Chen Haoran, Yang Quangsan and F.W. Williams A self-consistent finite element approach to the
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297l M.J.W. Greuter and L. Niesen Molecular dynamics simulation of the lattice dynamics of solid Kr

297m V. Vydra, K.M.A. El-Kader and V. Ch6b Influence of variations of temporal pulse shape in excimer
laser processing of semiconductors

297n L.-W. Wang and A. Zunger Large scale electronic structure calculations using the Lanczos method

297o C.S. Wu and L. Dorn Computer simulation of fluid dynamics and heat transfer in full-penetrated
TIG weld pools with surface depression

297p Ph. Lambin, L. Philippe, J.C. Charlier and J.P. Michenaud Electronic band structure of multilayered
carbon tubules
Abstract: The natural state of biological specimens is hydrated at temperatures above 0°C. These specimens cannot be placed directly into a conventional SEM because the water would vaporize and interfere with the electron generation and/or detection systems. To overcome this problem, a number of dehydration techniques have been developed, including air drying, critical point drying (Anderson 1951, 1956) (reviewed by Cohen 1977) and freeze drying from water or non-aqueous solutions (Boyde 1974, de Harven et al 1977). These techniques have enabled many specimens to be examined - whilst retaining a shape somewhat similar to their original structure. However, they do not prevent dimensional distortion during dehydration and quite large volume reductions can still occur (Boyde 1976, Boyde et al 1977). Also, the surface appearance of many samples can be radically altered by the different dehydration techniques employed.

The best method of overcoming these distortions during dehydration is to avoid dehydration. Two separate techniques have been developed to enable hydrated specimens to be examined in an SEM. The first involves lowering the vapor pressure) freezing the specimen in liquid nitrogen or liquid nitrogen cooled freon (Echlin et al 1970, Echlin 1971, Nei et al 1973, Robinson 1975b). Freezing prevents the vapor from interfering with the electron generation and detection systems, provided sufficiently low temperatures are reached and maintained during examination. These techniques enable a large range of specimens to be successfully examined without dehydration, although they have some limitations, including the possibility of ice crystallization damage.

However, the natural state of biological specimens is not frozen and it is desirable to examine many specimens in their natural state and/or in contact with water.

The second major technique of examining hydrated specimens is to examine them in an environmental cell, which isolates the high water vapor pressure from the electron generation and detection equipment. This paper discusses the various methods of performing this isolation and the factors which limit their performance.

Key Words:

Abstract: The use of a wide angle backscattered electron detector in a scanning electron microscope, which has the capability of the specimen chamber pressure being controlled independently of the column pressure, provides a simple technique for examining frozen hydrated specimens. Large specimens have been examined
within 1 min. of being placed on the stub and have been examined for many hours without charging artifacts or distortion due to dehydration.

300  V.N.E. Robinson , Facility of Applied Science, University of New South Wales, PO Box 1, Kensington, N.S.W., 2033, Australia, A wet stage modification to a scanning electron microscope, Journal of Microscopy, Vol. 103, Pt 1, January 1975, pp. 71-77.

Key Words:
Abstract: A modification to the vacuum system of a JSM2 scanning electron microscope has enabled hydrated specimens to be placed inside the chamber of the instrument and to be surrounded by water vapor at a pressure up to approximately 1.3kPa (10 Torr). The surface topography was observed by detecting the backscattered electrons using a wide angle backscattered electron detector placed close to the specimen. The microscope was operated in the normal scanning mode which allowed the examination of the surface topography of the specimens, whilst still retaining the depth of focus which is a feature of the SEM. This modification has enabled resolution of approximately 0.2 µm to be obtained from biological specimens partially immersed in water at temperatures just above 0°C.


Key Words:
Abstract: The Environmental Scanning Electron Microscope (ESEM) and new image analysis techniques are used to document the microstructure of cement pastes. The ESEM is shown to be vital to the imaging of cement hydration products at early age. An image intensity matching technique, which computes deformation between images with a resolution of 0.1%, is used in conjunction with the ESEM. This technique is used to document drying shrinkage, which is defined as the length change associated with loss of water. The effects on drying shrinkage of w/c, curing temperature, age, and drying rate are investigated. The magnitude of shrinkage is shown to vary as a function of scale, from individual particles to an area or average behavior. Shrinkage is shown to be controlled by both microstructure and hydration product properties. W/C is shown to vary the microstructure while curing temperature, age, and drying rate affect the properties of the hydration products. Experiments and modeling show there are restraining effects from calcium hydroxide and hydrous cement that accounts for differences in shrinkage between single particle and average behavior. Modeling is also used to demonstrate that a cement paste shrinks more at older ages when normalized by the shrinkage of the calcium silicate hydrate and porosity. Experimental results form the basis for a more complete understanding of the (micro)structure-property relationships for cement paste.

302  R.E. Cameron and A.M. Donald , Minimizing Sample Evaporation In the Environmental Scanning Electron Microscope, Polymers and Colloids Group, Cavendish laboratory, Madingley Road, Cambridge, CB3 OHE, United Kingdom

Key Words: Environmental Scanning Electron Microscope (ESEM), evaporation, condensation, pumpdown procedure
Abstract: The ElectroScan Environmental Scanning Electron Microscope (ESEM) enables the observation of wet samples to be made by eliminating air but allowing water vapor into the sample chamber. However, evaporation from and condensation on the sample may occur during the pumpdown sequence used to reach this state which means that the sample may not be in its natural state when viewed if due care is not taken. In this paper, the pumping system of the ESEM is described mathematically and expressions derived for the evaporation and condensation. This treatment is then used to calculate the optimum pumpdown sequence. The importance of using the optimized procedure is illustrated with micrographs of fat emulsions.

Abstract: Conventional scanning electron microscopy (SEM) is firmly established as a standard analytical tool. However, there have always been limitations on the samples which may be observed and the experiments which may be performed. Firstly, conducting coatings are needed on insulating samples to avoid build-up of charge and the consequent deterioration in image quality. This had rendered dynamic experiments on insulators difficult, since any change in the sample damages the coating. Secondly, the high vacuum within microscope has meant the samples must not contain any volatile species. Samples which are normally hydrated, for example, must be dried or frozen before observation. The Environmental Scanning Electron Microscope (ESEM) overcomes both these limitations. This article describes the principles behind the operation of the ESEM and discusses the new classes of experiment now possible. Examples of the use of ESEM are taken from a range of scientific disciplines.

Key Words: Environmental Scanning Electron Microscope (ESEM)


Abstract: The Environmental Scanning Electron Microscope (ESEM) eliminates the high vacuum requirement of conventional SEM, allowing the analysis of unprepared, wet samples.

Key Words: Environmental Scanning Electron Microscope (ESEM)


Abstract: It has been suggested that acids in the cold polar ice sheets may exist as aqueous mixtures at grain boundaries. This assumption can correctly predict the d.c. conductivity of polar ice, but this does not prove the existence of acids or liquid veins at grain boundaries, and this remains controversial. In this study we used a scanning electron microscope (SEM) equipped with a cold stage and an energy-dispersive X-ray microanalysis facility, to determine the location of sulfur in ice from the Antarctic Peninsula. As expected, sulfur was undetectable in the bulk of the ice. However, at the junctions where three grains met (tri-junctions), sulfur was found in concentrations greater than 1M in areas of <1 µm². Calculations show that between 40 and 100% of the sulfuric acid present in this ice was found at the tri-junctions, and would have been liquid at ice-sheet temperatures. This finding, if general, has considerable implications for many of the physical properties of polar ice.

Key Words: Antarctic ice, Sulfuric acid

306 Leon F. Keyser, Ming-Taun Leu, Surface Areas And Porosities Of Ices Used To Simulate Stratospheric Clouds. Earth and Space Sciences Division Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA 91109.

Key Words: ice

Abstract: Low temperature ices formed by deposition from the vapor phase are used as laboratory simulations of stratospheric ice clouds. To obtain intrinsic reactivities of these ices, detailed information on their physical structure is required. Surface areas, bulk densities and porosities are determined for H2O ice and HN03-H2O ices formed or annealed at temperatures from 85 to 265 K. Bulk densities and porosities are determined photogrammetrically. Scanning electron microscopy is used to obtain particle sizes and to study the morphology of the ices at several temperatures. Total surface areas are obtained from BET analysis of gas adsorption isotherms. Comparisons of the experimental isotherms with non-porous reference samples are used to determine the particle porosity due to micro and meso pores as well as the external area of the ice particles. Pore-size analysis yields the internal surface area and an estimate of the particle porosity that agrees very well with the porosity obtained from the comparison plots. The sum of the internal and external areas is consistently lower than the BET area; this plus the evidence for porosity obtained from the comparison plots indicates that part of the BET value is due to pore filling and, thus, cannot be considered a true surface area. External and internal surface areas as well as particle porosities are found to decrease sharply with temperature between 85 and 240 K, although bulk
porosities change very little. This suggests that the observed surface loss is due to pore closure, particle growth, and sintering.


Key Words: solder

Abstract: Preliminary results from solderability studies of Pb/Sn solder alloys wetting Au metallization are presented. The focus of these studies is to elucidate the physiochemical aspects of solderability, in particular the relationship between liquid composition and wetting. Spreading front morphology is discussed and a possible mechanism for observed rapid spreading sequences is advanced. A composition study of a spreading sequence exhibiting a precursor film is presented along with a discussion of the role of the film in the overall wetting process.


Key Words: Environmental SEM, hot-stage SEM, solders, microstructure, dispersion strengthening, microstructural refinement

Abstract: Solidification behavior of solders has a critical effect on the resulting microstructures and hence mechanical properties. Therefore, it is essential to understand the effects of soldering processing parameters on microstructure to engineer optimum microstructures. Microstructural changes in the solder that occur during the reflow process were studied in a hot-stage environmental scanning electron microscope (ESEM). An off-eutectic Sn6O-Pb4O solder and a dispersion strengthened solder with the same Sn-Pb ratio were reflowed in an ESEM, and changes in the microstructure were recorded on video tape. The dissolution and nucleation of grains during melting and solidification were observed. It was found that the microstructure of the conventional solder became coarser when it was allowed to solidify from a melt where the proeutectic lead was not completely dissolved. Grain refinement was observed in dispersion strengthened solder where the dispersoids acted as heterogeneous nucleation sites.


Key Words: Environmental Scanning Electron Microscope (ESEM), high pressure scanning electron microscopy, drying of paper, cryogenic scanning electron microscopy

Abstract: Environmental Scanning Electron Microscope (ESEM) is a new groundbreaking technique that images samples in a gaseous environment at pressures up to 2.7kpa (20 Torr). This paper reports an exploration of the capabilities of the ESEM for examining the wetting and drying of paper, and the drying of a coating formula. Breaking of fibers was seen during wetting and drying of 80# paper. But images must be interpreted with great care because electron beam radiation can damage wet cellulose fibers. In a coating formula dried in the ESEM, alignment of kaolin platelets was examined. This paper also assesses the potential for combining ESEM and cryogenic SEM for advantages of both.


Key Words: Pulp, Kraft, Thermo-mechanical pulp, Lignin, Electron Microscopy, ESEM Degradation, Irradiation
Abstract: Experiments have been made to try to explain the striking difference in irradiation in the scanning electron microscope between kraft and TMP fibers. TMP fibers remain intact while kraft fibers degrade quickly at high humidity, rendering them featureless. The water extractives and the benzone-toluene extractives from TMP, abietic acid and finally lignin were added to kraft handsheets in order to determine which wood component protects the carbohydrates in TMP. Only lignin gave protection against irradiation damage. The exact mechanisms of degradation, presumably caused by free radical reactions, of protection by lignin and of the accelerating effect of water are not known. It appears that lignin protects cellulose when it is intermixed with it (secondary wall) as well as when it forms a sheath around it (middle lamella).


Key Words: fiber-rising, scanning electron microscopy

Abstract: Encapsulation and controlled release of flavours and fragrances has revolutionized the food and fragrance industries. Microencapsulation is a process in which small amounts of liquids, solids or gases are coated with materials which provide a barrier to undesirable environmental and/or chemical interactions (e.g., heat, moisture, oxidation) until release is desired. Conventional Microencapsulation techniques include spray-drying, liquid phase methods employing coacervation and in-situ polymerization. Typical advantages to encapsulating foods and fragrances are outlined.


Key Words: flavours and fragrances

Abstract: A computer controlled loading fixture has been designed to allow in-situ observation of fracture processes during bending deformation of metal/ceramic microlaminates in an Electroscan Environmental Scanning Electron Microscope (ESEM). The stage has the capability of accommodating either 3 or 4 point bending experiments. A unique design feature of the stage is that the specimen surface remains at a fixed distance from the secondary electron detector and, hence, in focus during bending: the sample rests on the fulcrum which remains in a fixed position while the restraints that grip the ends of the sample descend on a ball slide. The system is controlled by an MacIntosh computer - Instruments NB-MIO-16L-9 data acquisition card. National Instruments LabVIEW82 software is used to control the stage displacement and to record the load cell and transducer outputs. The operation of this instrumentation in the ESEM is illustrated by the study of fracture processes in ceramic and ceramic/metal microlaminate films deposited on ductile metallic substrates.
Po-Fu Huang, Barbara J. Turpin, David J. Phipho, David B. Kittleson, Peter H. McMurry, Cloud Processing of Diesel Chain Agglomerates, for submission to Journal of Aerosol Science, Particle Technology Laboratory, University of Minnesota, Minneapolis, MN 55455 Publication Number 875, August 1993.

Key Words: cloud processing

Abstract: Diesel engines emit chain-agglomerate particles that can serve as cloud condensation nuclei. This research uses an Environmental Scanning Electron Microscope (ESEM) to study the effect of cloud processing on morphologies of individual diesel chain-agglomerates. Particles produced using a Caterpillar 3304 diesel engine from fuels with sulfur contents of 0.84%, 0.32% and 0.034% by weight were collected on silicon wafer substrates. They were subjected to 1-3 water condensation - evaporation cycles in the ESEM. This process was recorded on video tape, and digitized images of individual particles were used to find the particle's fractal dimension before and after each condensation - evaporation cycle.

Significant collapse occurred in particles generated from both the mid-range sulfur fuel (0.32% S) and the low sulfur fuel (0.034% S). The average fractal dimension of the particle images increased from 1.56 to 1.76 and from 1.40 to 1.54 for particles from low and mid range sulfur fuel respectively. We observed no significant morphological change in particles from high sulfur fuels. The experiments reflect lower limits for the degree of collapse that diesel chain-agglomerate particles undergo during atmospheric cloud processing.

Mehta, S., Jones, R., Chatterji, J., and McPherson, G. Effects of amorphous and crystalline silica on phase chemistry, microstructure and strength of set cement at elevated temperatures., ARCO Exploration and Production Technology, Plano, Texas 75075, Halliburton Energy Services, Duncan, Oklahoma 73533

Key Words: Strength retrogression of cement pastes cured at elevated temperature is a well-known phenomenon. To offset this decrease in compressive strengths, 35 to 70% crystalline silica is added to cement slurries that are cured at or above 110°C (230°F). Crystalline silica addition minimizes the conversion of C-S-H gel to a-C2SH, which is thought to be the primary cause for strength retrogression at elevated temperatures. However, it has been also observed that if crystalline silica is replaced by amorphous silica such as silica fume, the beneficial properties of silica additions may not be realized. The reason for this anomalous behavior of anomalous silica is not well understood.

In this paper results of environmental scanning electron microscopy (ESEM), cryogenic scanning electron microscopy (Cryo-SEM) and x-ray diffraction (XRD) studies of high temperature, cured class H oil well cement pastes are presented in an attempt to delineate the effects of crystalline silica and amorphous silica additions on microstructure, phase chemistries and compressive strengths of pastes cured for one, three and seven days at 110°C (250°F). The results show that in one day cured samples there is a distinct difference in the hydrated phases present as well as in the nature of the matrix cohesiveness. These differences are reflected in the much higher compressive strength of the crystalline silica sample compared to the amorphous silica or neat samples. For 3 and 7 days cured cements, however, the chemical differences tend to diminish, but the compressive strengths of amorphous silica samples remain at or near that achieved after one day curing; whereas, for the crystalline silica samples the strength increases by a factor of two after 7 days. These observations indicate that, for controlling strength retrogression at high temperatures, amorphous silica additions to cements should be avoided since they can create major matrix defects and irregular gel chemistry with Ca/Si ratio that does appear to be suitable for conversion to a more stable tobermorite matrix.

Roger B. Bolon, Craig Robertson, X-RAY & MICROSTRUCTURAL E-SEM ANALYSIS OF REACTIONS AND NONCONDUCTING MATERIALS IN GASEOUS ENVIRONMENTS., GE Corporate Research & Development, Schenectady, NY 12301

Key Words: Biologists have long been interested in the ability to look at samples in their natural wet state without tedious sample altering preparations. This need led to the development of a new commercial instrument and technique, capable of looking at samples in saturated water vapor as well as in a variety of other gases, called
environmental scanning electron microscopy (E-SEM). The key features of the instrument are an extensive
differential pumping system between the chamber and column and a gas amplification secondary electron detector
(GED). Together these developments provide new capabilities for looking at materials and performing dynamic
experiments not possible by traditional SEM techniques. This paper summarizes the key features which make the
E-SEM different and presents a collection of experimental results from an ongoing feasibility study exploring new
applications for materials characterization.

D.A. Lange, Sujata, K., and H.M. Jennings, CHARACTERIZATION OF CEMENT-WATER
SYSTEMS, Northwestern University, Evanston, IL

Key Words:

Abstract: Mechanical properties of cement and concrete are controlled largely by the microstructure which
develops during hydration. The major hydration product, calcium silicate hydrate (C-S-H), forms physical bonds
between cement particles providing structural integrity to the bulk material.

Recent advances in electron microscopy, like the Environmental Scanning Electron Microscope (ESEM) from ElectroScan
Corporation, have made possible direct observation of physical changes in hydrating cement. In the ESEM, the specimen
chamber remains at pressures of 1-20 torr during imaging. Various gases and water vapor may be introduced into the chamber.
A microinjector mechanism permits water to be added during imaging of the specimen. Dissolution and precipitation
processes are observed uninterrupted and recorded on videotape.

New insights into cement hydration are emerging as a result of these new techniques. We have been able to watch the
microstructure change when individual cement particles dissolve and precipitation products form as water is added to dry
cement. Adjoining particles join to form bridges in the early stages of hydration, much as in the sintering process. C-S-H is
first seen to form on the inside surfaces of pores and voids. The composition of the material is obtained from dot maps by an
Energy Dispersive X-Ray analyzer coupled with an Image Analysis system.

Other ESEM projects in progress include studies of effects of drying of cured cement, effects of different cement mixing
techniques, and analysis of cement grout microstructures.

Hoyberg, K.; Knaggs, H., Environmental scanning electron microscopy of microcomedones -

Key Words: Electron Microscopy ; Environmental

Abstract: Environmental Scanning Electron Microscopy (ESEM) allows the direct observation of wet, dry,
and nonconductive specimens without sample preparation. Microcomedones extracted from cyanoacrylate biopsy
technique were examined via ESEM. ESEM provides a viable technique to monitor the surface of a substrate
before and after treatment to determine the efficacy of products. This technique will also be used to study the
effects of other known anti-acne agents as well as acne products. 3 Refs.

Key Words: 

Abstract: Supercalendered filled (SC) and light weight coated (LWC) papers were examined using an environmental scanning electron microscope (ESEM). Large structural surface changes were observed during condensation of water on the surface in a high moisture environment. In the case of LWC papers, underlying fiber protrude and appear extensively swollen. SC papers show significant roughening during wetting but there was no indication of ribbon-to-tube shape change of individual fibers.


Key Words: electron microscopy, environmental

Abstract: Environmental Scanning Electron Microscope (ESEM) has been used to study the every early pre-induction, and induction physical processes that occur in the hydration of tricalcium silicate. An in-situ experimental technique is described which allows direct, real time observation of the sub-micrometer morphological changes that take place during this reaction. The results of this investigation are correlated with kinetic data obtained by differential scanning calrimetry (DSC). In this way, microstructural evolution has been identified with the stages of very early hydration. Upon first contact with water, a gelatinous coating was seen to form at grain surfaces and a crystalline secondary product was observed at the end of an extensive dormant period. These findings are viewed in the light of previous "wet" and "dry" microscopy studies, and are discussed within the framework of ordinary Portland cement as a possible explanation of induction. Comment is made as to the suitability of environmental SEM for analysis of such materials.

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