Does Your SEM Really Tell the Truth?—How Would You Know? Part 4: Charging and its Mitigation

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ABSTRACT

This is the fourth part of a series of tutorial papers discussing various causes of measurement uncertainty in scanned particle beam instruments, and some of the solutions researched and developed at NIST and other research institutions. Scanned particle beam instruments, especially the scanning electron microscope (SEM), have gone through tremendous evolution to become indispensable tools for many and diverse scientific and industrial applications. These improvements have significantly enhanced their performance and made them far easier to operate. But, the ease of operation has also fostered operator complacency. In addition, the user-friendliness has reduced the apparent need for extensive operator training. Unfortunately, this has led to the idea that the SEM is just another expensive "digital camera" or another peripheral device connected to a computer and that all of the problems in obtaining good quality images and data have been solved. Hence, one using these instruments may be lulled into thinking that all of the potential pitfalls have been fully eliminated and believing that, everything one sees on the micrograph is always correct. But, as described in this and the earlier papers, this may not be the case. Care must always be taken when reliable quantitative data are being sought. The first paper in this series discussed some of the issues related to signal generation in the SEM, including instrument calibration, electron beam-sample interactions and the need for physics-based modeling to understand the actual image formation mechanisms to properly interpret SEM images. The second paper has discussed another major issue confronting the microscopist: specimen contamination and methods to eliminate it. The third paper discussed mechanical vibration and stage drift and some useful solutions to mitigate the problems caused by them, and here, in this the fourth contribution, the issues related to specimen "charging" and its mitigation are discussed relative to dimensional metrology.

Keywords: calibration, charging, measurements, metrology, modelling, scanning electron microscope, SEM, standards, reference materials

1.0 INTRODUCTION

Scanning electron microscopes are used extensively in research and advanced manufacturing for materials characterization, metrology and process control. Earlier papers [1 – 3], discussed some of the potential issues and pitfalls to avoid when quantitative measurements are made with an SEM. The first paper in the series discussed signal generation, instrument calibration, electron beam interactions, and the need for modeling to understand the mechanisms of the actual image generation [1]. Modeling has been discussed at greater length in other papers [4-5].

The second paper in the series, addressed another major issue confronting the microscopist, which is specimen contamination and methods of contamination reduction and its elimination [2]. In a third paper, the additional components of measurement uncertainty induced by mechanical vibration and stage drift and some possible solutions to these issues were discussed [3]. In this, the fourth contribution, some of the issues related to specimen "charging" and methods for its mitigation are discussed. All four of these tutorial papers are unified in the discussion of how these particular problems effect

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[2] Certain commercial equipment is identified in this report to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment identified is necessarily the best available for the purpose.

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dimensional measurements made with the SEM. Over the years, several workers at NIST and other institutions have done a great deal of research into these issues in order to improve the fundamental metrology with particle beam instruments and some of this work, including some historical perspectives, is reviewed and discussed here.

2.0 DISCUSSION

2.1 Specimen Charging. The term "charging" in a particle beam instrument relates to the build-up of either positive or negative potential at or near the surface of a sample while it is being irradiated by a particle beam. Charging results in a significant number of undesirable consequences, and in a few cases, it can be used to the advantage of the researcher (see: Section 4.0). Surface charging causes instability of the secondary electron image intensity which results in variations in the secondary electron yield and detector efficiency. Changes in the surface potential also alters the primary beam landing energy resulting in changes in magnification, beam drift, image distortions and potential errors, even in x-ray microanalysis. All of the issues induced by charging are detrimental to measurement data quality.

Charging has been studied [6 - 9], but is not all that well understood. As discussed more extensively below, one can divide the possible cases of sample charging into four broad categories: **non-charging** - this is the case of metals, i.e., conductive samples where the primary beam electrons can readily travel to ground potential; **un-noticeable charging -** charge build-up is sufficiently minor that the operator does not readily observe obvious charging-related problems during the imaging and measurements of partially conductive, grounded samples. This is the most troublesome case since it often not recognized until after the micrograph has been taken; **evidently charging -** partially conductive samples that still allow limited imaging and measurements; and **grossly charging -** non-conductive and or non-grounded samples that preclude any meaningful imaging or measurements.

Maxwell's equations dictate that charge must be conserved, this is an accounting relationship. When viewing an ideal conductive sample at high accelerating voltage, the sum of the backscatteded electrons leaving the sample (backscattered electron coefficient - η), the secondary electrons leaving the sample (secondary electron coefficient - δ) as signal, may be less than unity but, must be balanced by those electrons flowing to ground, this can be measured as the specimen current. In an ideal case, the specimen current measured (I_{sc}) is a function of the beam energy (E). If incident beam current is represented as (I_{beam}) then:

$$-I_{beam} + (\eta+\delta)I_{beam} + I_{sc} = 0$$

and for a conductive sample, that is typically, the case. Therefore, when (η+δ) is unity the measured I_{sc} is zero.

Unfortunately, most of the more interesting samples are not ideal. In most cases, there are differences in the current flow to earth between the I_{beam} and the I_{sc} . Those difference relate to the conductivity, the total signal leaving the sample and how much charge remains:

$$I_{sc} = 1 - (\eta+\delta).$$

In a non-conductor, I_{sc} = O, so charge can accumulate. If η+δ < 1, negative charging will occur, and if η+δ > 1, positive charging will result. In those cases where charge accumulates, the goal is to achieve a ***Dynamic Charge Balance*** so that: η+δ = 1, so the number of electrons injected into the sample by the primary electron beam are balanced by those leaving the sample as signal [8]. Approaches to achieving that balance are discussed in Section 3.1 and Joy and Joy, 1996 [8].

The consequences of charge build-up in an SEM have been known and researched since the early days of television. This is because the early television and the SEM are both closely related technologically in that they were both scanned electron beam systems. Especially notable was the work at RCA Laboratories [10 - 12]. Aspects of that research were directly applicable to the early SEM instruments such as the ones developed by Zworykin, Hillier and Snyder [13] and those at Cambridge University [14 – 15] that ultimately led to the first commercial SEM instruments.

In some ways, charging is very capricious in that one can easily make a sample charge-up grossly (as discussed below), or subtle charging can go on essentially unnoticed and potentially result in significant measurement errors. This capricious

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Figure 1. *Examples of negative charging of a diamond chip. (Left) Micrograph demonstrating minimal charging with low landing energy[3] at 1.0 keV (HFW[4] = 36 μm). (Right) Micrograph showing evidence of strong charging when the landing energy is increased to 10 keV, (HFV = 13 μm).*

nature is largely due to the dynamic nature of charging, to the versatility of the scanning electron microscope, and to the variety of geometries and instrument conditions available in the various particle beam instruments. Often a sample that is charging in one instrument may show no obvious sign of charging in another. There are many reasons why this is the case (as discussed below). In the past, most of the research work has revolved around finding ways to avoid charging. This is quite understandable since this is a rather complicated problem to solve because of the large number of possible instrument and sample variables. It is clear that it is up to the operator to recognize a charging situation and determine the proper conditions necessary to mitigate it and acquire the best images and measurement data.

2.2 Types of Charging. Of the four general cases described above, strictly speaking, it comes down to two types of specimen conditions that can be readily identified. These are non-charging and charging in the particle beam instrument.

2.2.1 Non-Charging. A highly conductive sample, such as bulk gold, channels all of the electrons that it absorbs to ground and no charging (i.e., change in electrical potential) would come about either during image acquisition or after. Clearly, that

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[3]Low landing energy is used here since that term has replaced the term low accelerating voltage because in some of the newer instruments the electron source can emit electrons at high accelerating voltage, but they are decelerated to a lower landing energy in the column and/or at the sample stage. This technique allows the electron optical column to operate more optimally (See: Reference 1). In SEM literature, landing energy is usually given in kilo-electron volts (keV). For example, 15 kV accelerating voltage with no deceleration results in (approximately) a15 keV energy primary electron beam.

[4]Although, horizontal field width and field of view are often used interchangeably (See: Reference 1), HFW has been adopted in this publication since field of view implies a two - dimensional array which is only valid when the beam scan is normal to the sample (zero degrees of tilt).

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is the most ideal situation. Most of the more interesting sample materials are not so cooperative. Even some, seemingly completely conductive metal samples, such as aluminum can have an oxide layer on the surface, can develop a charge depending upon the instrument conditions applied.

2.2.2 Charging. When a material cannot effectively conduct the beam energy imparted to it by the primary electron beam to ground it is often said to be "charging." This build-up of (or a change in) the electrical potential in or around the sample itself can result in detrimental effects to the imaging and any measurements made with the instrument on that sample. Samples may develop a static charge that - depending on the conductivity of the sample and its environment – can be retained for long periods of time, in vacuum. Generally, it is advantageous to allow the static charge to completely drain from the sample, because the changes induced by the primary electron beam of the instrument can be interpreted better and are more repeatably. The accumulated charge in the sample material represents a potential energy, and when it is drained, the sample achieves a more neutralized, more stable, less energetic state. Electrical connections, including surface conduction due to humidity, all play a role in discharging the sample. Two major categories of charging can occur:

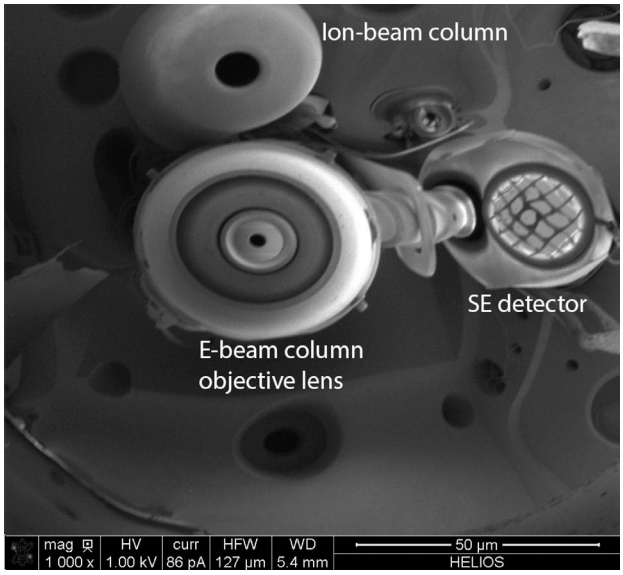

Figure 2. *Example of extreme negative charging causing the primary electron beam to image the inside of the specimen chamber. The instrument "reports" it is scanning a horizontal field width (HFW) of 127 µm however, the stated magnification and HFV recorded on the micrograph are clearly wrong; the HFV is actually approximately 20 cm.*

2.2.2.1 Negative Charging - ($\eta+\delta < 1$). Negative charge build-up occurs when a number of electrons impinging on the sample are trapped within the material and a negative electrical potential builds up. This can be only few volts or as much as the primary electron beam, i.e., several thousands of volts. The most common manifestation of this situation is that the image appears to "glow" (brighter) or cause geometry distortion in the image as electron production is artificially enhanced or the beam is unintentionally deflected (Figure 1). In other cases, marginally adhered particles can be seen to "blast-off" from the specimen stub – never to be seen again (until they land upon a critical component within the column). Fibers, insect antennae and other protruding structures will also be seen waving at the operator as the beam scans across them.

Since most samples are not totally conductive, charging is a common situation; a good deal of scientific literature has been devoted to this topic [16 – 18], as well as, the various references cited below. Negative charging is the most evident and troublesome type of charging and under the most extreme circumstances can disrupt and deflect the electron beam, and cause intolerable distortions. One of the first references to this, for the SEM, was Clarke and Stuart [19]. They formulated an explanation for the "formation of the distorted image of the electron collector of the scanning electron microscope when the instrument is used to observe uncoated insulating materials." This was provided as a cautionary note because they correctly felt it could lead to image misinterpretation when uncoated insulating materials were being observed.

Figure 2 shows an extreme case of charging resulting in a "mirror microscopy-like" image similar to the one described by Clarke and Stuart [19]. In this case, the sample has developed and is retaining a potential at or above that of the primary electron beam. The primary electron beam does not impinge on the sample, as it is scanned over the sample, but it is deflected throughout the specimen chamber generating signal from the internal components of the SEM specimen chamber, such as the final lens, and electron detectors [20, 21]. Even as strange as this mode of instrument operation is, it can also hold a diagnostic function since it can image particles and other contaminants on apertures and the final lens pole piece. Shaffner and Hearle, van Veld and Shaffner, and Shaffner and van Veld [22 - 24], reviewed the phenomenon of charging and also described the mirror mode described above and shown in Figure 2. The extreme negative charging at the sample, causes the primary electron beam to actually become diverted and image the inside of the specimen chamber. Images become grossly distorted and the primary electron beam is deflected as it approaches the sample throughout the chamber when such charging is present. Tilting the sample can direct the beam to various locations of interest. There does not ap-

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pear to be any negative consequences to these actions, but it is startling to the operator the first time it occurs. This is an amusing application of charging, but this is not the main area where the typical charging problem exists. Typically the majority of problems exist between sample ground and just a few electron volts where subtle, un-recognized charging occurs.

Often, charging is obvious, but sometimes it is quite subtle. Negative charging presents an insidious problem for dimensional measurements because there is the potential for it to deflect the beam such that it actually lands nanometers away from its intended location. The amount of deflection can be negligible or it can be significant depending upon the instrument conditions and the 3-dimensional structure being measured. As shown in Figure 3, when the beam approaches a charging structure, its trajectory can be altered and the landing point where the signal is being generated and the point where the instrument scanning system believes the landing point can be different, hence leading to erroneous data and measurements. The delta (Δ) of this measurement is exaggerated for effect, and the amount of deflection is variable and depends on the electrical potential, the structure of charging sample, and on the landing energy[3] of the electron beam. This effect was postulated by Postek [21] for photomask metrology and was later demonstrated by Davidson and Sullivan [22] who calculated the electric fields on dielectric materials and showed, with modeling and experimentation that measurements in the SEM could be compromised by several nanometers if charging of only a few volts was occurring on the sample. Further work in this area needs to be done in order to fully understand the uncertainty that such charging poses to the accuracy of any measurement. However, it is very important to be aware of the potential uncertainty this introduces into the measurement process and to work to eliminate charging in all possible cases.

2.2.2.2 Positive Charging - (η+δ > 1). Positive potential can build up when more electrons are emitted from the sample than the primary electron beam provides. The positively charged regions rather than glowing brighter, get darker, because the secondary electron (SE) emission is reduced, many of the SEs are attracted back to the sample surface. Positive charging turns the scanned area dark and it is often confused with the build-up of contamination (which was discussed in Reference 2). Positive charging is far less detrimental than negative charging, and it is usually restricted only to a few volts of electrical potential. The main result is a loss of some valuable signal electrons as they are re-absorbed by the positively charging surface [27, 28]. Figure 4 shows an interesting effect of the deposition of positive charging on a thin oxide film sample. The initial "writing" of the dark lines was carried out by the automatic exposure (contrast, brightness) setting circuitry that was scanning only over the partial field, resulting in the widely spaced dark scan lines. The acquisition of the final overall image was then taken with that exposure setting.

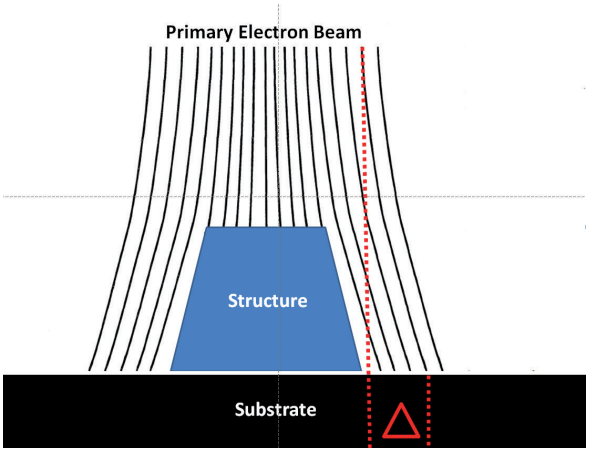

Figure 3. *Artistic representation of potential beam deflection due to charging. Charging of structures can result in the potential of beam deflection of several nanometers (re-drawn from Davidson and Sullivan [26]). The magnitude of the deflection (Δ) is a function of a number of factors as discussed in the text.*

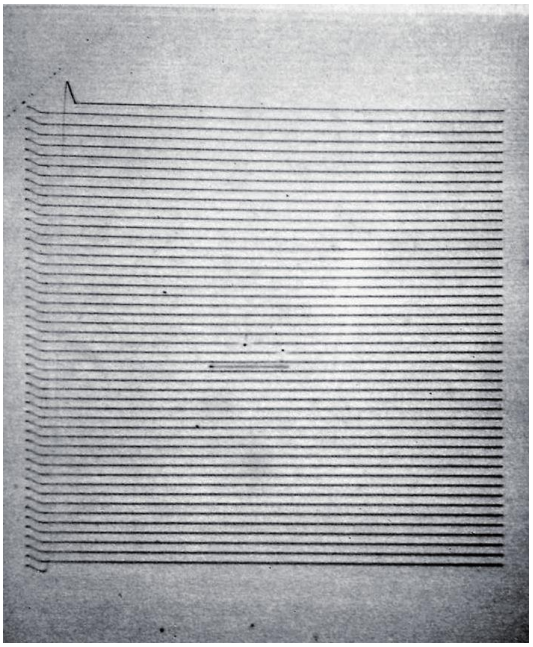

Figure 4. *Positive charging on a thin oxide film sample showing dark lines where the primary electron beam was scanned over the sample during the automatic exposure setting routine. In the case shown, the partial field scanning for the automatic exposure (contrast and brightness) adjustment resulted in positive charging on the portion of the sample exposed by the beam. After the initial adjustment, the final overall image was taken. Note the scan initiation, over-scanning and retrace can be clearly seen. The micrograph was taken at 1.0 keV (HFW = 2 250 μm).*

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If that sample was allowed to remain in the instrument for a period of time, or removed and put back into the instrument, the dark lines will have disappeared since the charge dissipated due to the venting of the chamber.

2.2.3 Diagnosing Charging. Positive and negative charging can be diagnosed quite easily to determine the proper landing energy and the sample's conductivity, a further discussion can be found in Joy and Joy [9]:

- Set-up the instrument to the proper instrument operating conditions.
- Locate an area of interest and focus on that area at a high magnification or the magnification where one you plan to do the majority of the work (the effect of charging is exacerbated at higher magnifications).
- Irradiate the sample for a few seconds within the area selected.
- Reduce the magnification by a factor of 5 and observe the sample.
- If a bright raster pattern appears (which may slowly disappear upon going to the lower magnification), negative charging is probable. Therefore, try lowering the landing energy a few 100 eV. Then, repeat the procedure at a different location.
- If a dark raster pattern appears, and then (possibly) quickly disappears, positive charging is probable (Figure 3). If that occurs, raise the landing energy a few hundred volts. Then repeat the procedure.
- If the dark square remains, then positive charging is not likely to be the problem. Beam induced contamination is more likely the problem (see: Reference 2).

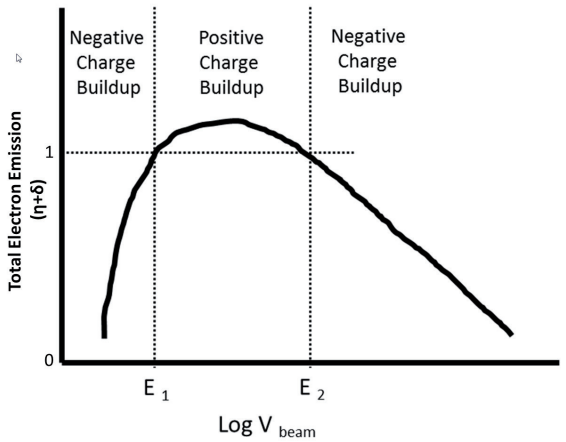

Figure 5. *Total electron emission curve. The E_1 and E_2 points are the landing energies where Dynamic Charge Balance is achieved and no sample charging is expected to occur.*

3.0 SOME METHODS FOR CHARGE MITIGATION

Studies of the phenomenon of sample charging were carried out since early work with the SEM. The SEMs relative similarity to early cathode-ray tube and television research led to many useful and parallel conclusions. The two most common approaches to the mitigation of charging are low accelerating voltage (low landing energy) operation and coating the sample with a thin conductive metal or carbon layer. Other possible solutions are discussed later in Section 3.3.

3.1 Low Accelerating Voltage Observation. Low accelerating voltage (landing energy) operation was possible with most SEMs since the early days, but the imaging was generally poor due to instrument design, poor signal-to-noise ratio and lower resolution [29, 30]. It was not until the latter 1980s when scanning electron microscopes were able to routinely view most samples in a non-destructive, uncoated manner. Many innovative instrument improvements took place which eventually changed instrument operation and the terminology used to low landing energy techniques.[3] The notable improvements that spurred this was the availability of high brightness electron sources such as lanthanum hexaboride and field emission electron sources and later frame storage electronics which evolved into the current digital imaging electronics. Non-destructive, low landing energy operation became common in semiconductor manufacturing where insulating samples (such as oxides and photoresist) are viewed routinely on the production lines. Early research work in cathode ray tubes and television found that generally, at low landing energies, a charge balance could be achieved when an electron beam impinges on an insulating surface. Thornley [31] reported that at low (1-2 keV) landing energies the secondary electron coefficient could be greater than unity, as shown on Figure 5.

For most non-conductive materials, E_1 and E_2 are the points where the total electron emission is equal to 1. Joy and Joy have published data on a number of E_2 points [9]. It is thought that the E_1 and E_2 points are relatively stable for a particular sample and set of instrument conditions being applied (landing energy, beam current, tilt, etc.) and they are the energies at which the sample is in charge balance. At that point, the number of electrons injected into the sample by the primary

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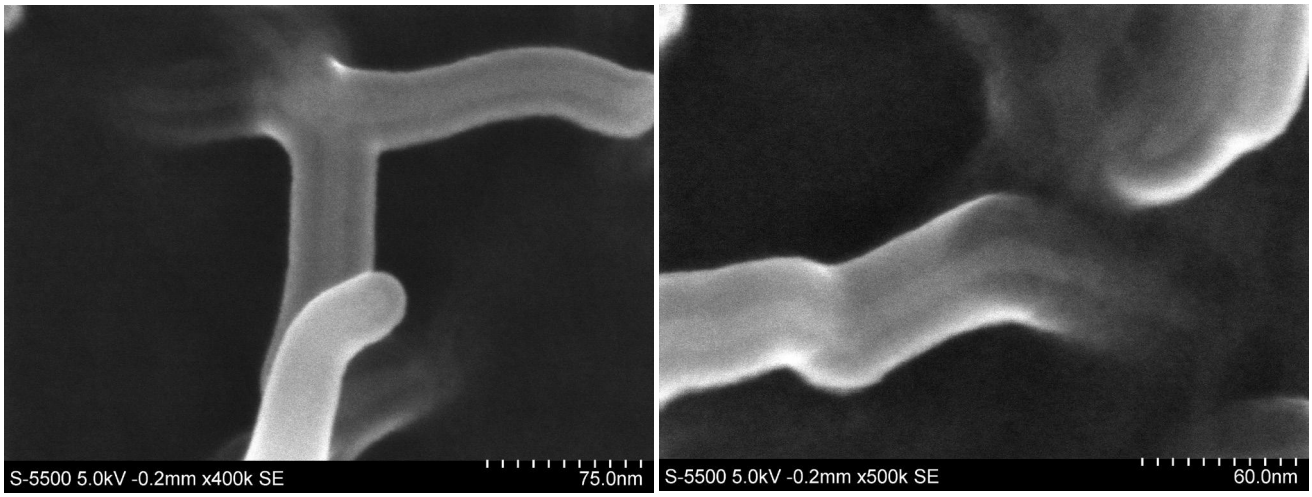

Figure 6. *Micrographs of cellulose nanofibrils that have been coated with osmium vapor in order to reduce the charging. The 6-7 nanometer visible core is likely the cellulose and the remaining thickness is the osmium vapor coating. Images taken at a landing energy of 5 keV (HFV, left = 250 nm, HFV, right = 316 nm).*

electron beam is equal to the total of those electrons leaving the sample and thus no specimen charging presumably occurs. Usually there is a small range of voltages to which a sample can be exposed, up to and including the E_2 value. E_2 is the most stable value and is usually chosen for uncoated observation since it is found at a higher accelerating voltage, thus enabling a higher resolution operating condition for the instrument. However, the optimal landing energy needed is always dependent on the required sample information. A high voltage primary beam will image layers deeper into the sample, while low voltages will provide more information from the sample surface. So compromises must always be optimized. Additionally, newer particle beam instruments with ultra-low voltage/high resolution capability can work acceptably in the E_1 region without significant compromise to the resolution.

3.2 Specimen Coating. Traditionally, over-coating the non-conducting specimen with a heavy metal, conductive, material (gold, gold/palladium, and osmium) has been the most commonly used method to overcome charging. Coating also increases the secondary electron emission from the sample especially if the sample is composed of low atomic number materials (especially biological). The one thing that must be remembered is that, if a sample is coated, signal is mainly being generated from the flux of electrons originating from the coating acting as a protective shell and not necessarily the sample of interest. In addition, a myriad of coating artifacts, such as cracking, can result. Adding the appropriate amount of coating has always been a complicated decision based upon the needed conductivity and the amount of artifacts one can tolerate. Vacuum evaporation (gold, gold/palladium), sputter coating (gold, gold/palladium) and aqueous or vapor deposition of osmium have all been used. The philosophy and techniques can be found in Postek et al., [32]. For x-ray microanalysis often carbon coating is also helpful in reducing charging and diminishes the effects of stray artifacts in the analysis [33].

A good continuous coating can mitigate charging, but can also introduce coating artifacts such as a change in surface details. Coating also increases the size of the structures being observed relative to the thickness of the coating applied. Therefore, interpretations can be compromised. Figure 6 shows a nanocellulose material that has been coated with a deposition of a few nanometers of osmium vapor. Note that the core (observed through the coating) is about the expected 6-7 nanometers in diameter for the cellulose nanomaterial but, surrounding it is several additional nanometers of coating. Therefore, coating a nanoparticle potentially compromises the measurements especially on nano-sized particles and structures.

3.3 Other Potential Solutions. The simplest approach is often the best approach. Hence today, non-destructive low accelerating voltage operation is the first method usually applied to an unknown sample, then coating may be tried if needed. Seasoned microscopists usually begin by applying low landing energies to an unknown sample, unless they know coating will not compromise the imaging or measurements. However, as discussed below, other methods have also been used with varying degrees of success.

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3.3.1 Charge Neutralization. Prior to the availability of high resolution imaging at low landing energies, Crawford [34, 35] and others reported good success with specimen charge neutralization. In this case, the charge build-up is neutralized, as it builds up, by a beam of very low energy ions. The ions act to stabilize the surface potential, at the "ion zero kinetic-energy point, independent of the nature of the insulating surface." [34] This requires the installation and optimization of a charge neutralization device in proximity to the sample. The unit is positioned above the specimen and below the final lens in the specimen chamber of the SEM. Because of the amount of specimen chamber real estate needed by the device and the prevalence of low landing energy microscopy with high-brightness field-emission instruments, this method is not often practiced, today. In the scanning helium ion microscopes there is an option for an electron flood gun to work to neutralize the positive charging caused by the ions.

3.3.2 Fast, TV-Rate Imaging. Welter and McKee (1972) [36] demonstrated that fast scanning using a high-brightness field-emission electron microscope could alleviate charging problems. They stated that "if a layer of charge is put down on the specimen and reinforced at a scan rate faster than the average discharge rate," charge equilibrium could be reached. They used a fixed TV scan rate of 1155 lines per frame and 15 frames/sec. and provided reasonable imaging even at low landing energies. This work paved the road for the more modern instruments displaying 60 frames/sec. (or greater). TV-rate imaging is now common on most instruments. But, it took successful demonstration of the concept of fast scanning with good signal-to-noise ratio to prove that charging could be mitigated in this manner.

3.3.3 Backscattered Electron Imaging. One of the earliest approaches to charge mitigation in the SEM was to employ backscattered electron collection rather than secondary electron collection. Charging of the sample affects the secondary electron image far more than the higher-energy backscattered electrons. Most laboratory SEMs are equipped with a mechanism whereby the bias of the collection screen at the front of the SE detector can be grounded or negatively (reverse) biased, thus rejecting the SE and only allowing those high-energy BSEs that are in the proper geometrical relationship to the detector to be collected. Alternatively, dedicated backscattered electron detectors can be employed. Tilting the sample toward the detector is, not only, helpful to improve signal collection but alsom signal strength. BSE detection is also used on uncoated samples in the table-top instruments. Alternatively, the low loss technique developed by Wells [37] was shown to provide high-resolution images of the sample surface while mitigating the charging.

3.3.3.1 Low-Loss Electron Imaging. Low loss imaging is a subset of backscattered electron imaging where the electrons are energy filtered in such a manner that only those that have minimally interacted with the sample are collected. These are the low loss electrons. These electrons have been demonstrated to have greater surface sensitivity and reduced apparent charging [38 – 41]. Overall, sample charging is not eliminated and beam deflection by surface charging can still occur - the charging is not dissipated, just ignored. If the charge builds up sufficiently, deflection of the primary electron beam is still possible.

3.3.4 Conductive Spray. Prior to the prevailing use of high-resolution low landing energy microscopy, experiments were undertaken to use a "conductive" spray to eliminate charging. As early as 1957, Wells [15] described experiments with several potential anti-static materials. It is notable that, conductive spray was reported to be successfully used on polymers by Sikorski et al. (1967) [42] to view polymers with no or reduced charging at high landing energies. A "conductive film aerosol" was marketed in 1980, as a commercial product, but was taken off the market several years later. A similar product has been recently revived as ConductCoat [43]. This product appears to have some success in reducing charging on some materials, but an overall comparison if this material to low landing energy operation has not been done, nor have the effects on instrument or specimen contamination been fully studied.

3.3.5 Variable Pressure SEM. It is clear that, charging must be overcome in order to obtain any meaningful data from the SEM. Gross charging can readily distort the image and subtle charging can deflect the beam and lead to measurement error. An alternative that has not been fully explored for metrology is the employment of variable pressure or "environmental" microscopy [44 - 47]. This methodology uses a gaseous environment to neutralize the charge. For various technical reasons, high-pressure microscopy has mostly been employed for specimens of a biological nature, not for many semiconductor samples. Figure 7 shows several images of photomask samples taken at high landing energies usingt variable pressure technoloogy demonstrating no charge accumulation. Photomasks are very prone to charging [48]. It has been reported that high accelerating voltage, injection of air of as little as 20 Pa ~0.15 Torr into the specimen chamber can reduce the charging

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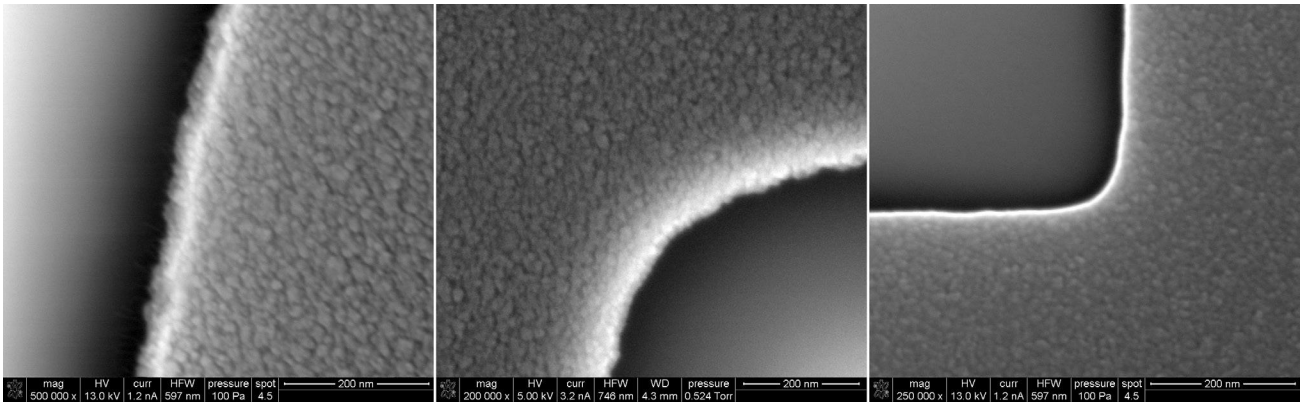

Figure 7. *SEM Micrographs of several chromium photomask samples using the variable pressure SEM. (Left) 13 keV landing energy (HFW =597 nm); (Center) 5keV landing energy (HFW = 746 nm); (Right) 13 keV landing energy (HFW =597 nm).*

potential of an insulator at the surface by as much as an order of magnitude [49]. For accurate metrology, this methodology affords a path that minimizes, if not eliminates, the need for charge modeling. Modeling of charging is exceptionally difficult since each sample, instrument and operating mode can respond to charging in different ways. This methodology shows great potential if optimal balance can be achieved in a reproducible manner. This methodology, although potentially desirable for charge neutralization, has not been seriously employed in photomask or wafer metrology [50]. This is largely because there is not an instrument available for full-scale production samples with high throughput. VPSEM was proven to be useful for photomask metrology [51] but no in-line instrument was developed to use the technology, either. Variable pressure microscopy offers advantages of possible application of higher accelerating voltages and different contrast mechanisms [51].

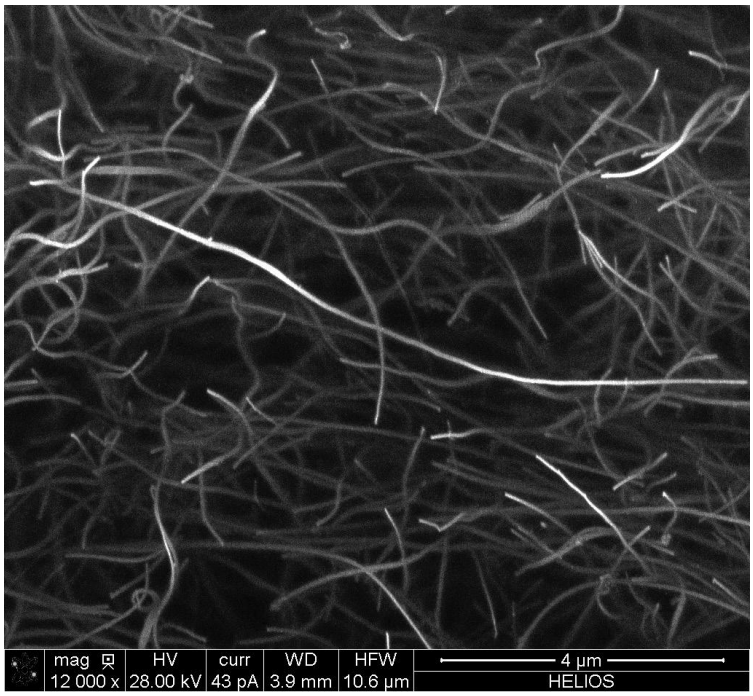

Figure 8. *Micrograph of tangled multiwall CNT structures in an epoxy matrix taken at 28keV (HFW = 10.6 µm) those that are sharper reside close to the surface and others are several nanometers below the surface (See: Reference 55).*

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4.0 ADVANTAGES AFFORDED BY SAMPLE "CHARGING"

On the other side of the coin, charging can be used and advantageously controlled. Charge contrast forms the basis of several imaging modes such as voltage contrast (VC) and electron beam induced conductivity (EBIC). Both of these methods are used extensively in semiconductor electronics testing and quality control [27, 52, and 53].

4.1 Charge Contrast. More recently, some conductive materials buried on non-conducting matrices have been shown to be successfully imaged using charge induced contrast (Figure 8). By properly choosing the instrument operating conditions, sub-surface imaging of materials, such as carbon nanotubes (CNT) in polymers (epoxy) can be imaged even embedded as deeply as several hundred nanometers [54, 55].

5.0 CONCLUSION

Charging is an inevitable consequence of particle beam microscopy of non-conductive samples. It is clear that charging must be overcome in order to obtain meaningful and repeatable data from the SEM. Coating of the sample to make it conductive is only one solution, which could lead to artifacts. Gross charging readily distorts the image and subtle charging can deflect the beam and hence can lead to measurement error. Charging can be overcome with judicious application of the methods discussed in this presentation. For general imaging, charging can be useful and it may create interesting micrographs, but for measurements it can lead to a great deal of error if the operator is not careful.

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