Protons, Ions, Electrons and the Future of the SEM

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Abstract. The Scanning Electron Microscope (SEM) is the most widely used high performance microscope in all fields of science but it is now reaching the theoretical limits of its performance. While advances in sources, optics, and detectors, can result in some improvement in performance the ultimate resolution is limited by fundamental physical constraints. One potential alternative is a scanning microscopes utilizing light ions such as H+ and He+. Such an instrument shares all of the benefits of the conventional SEM but is free from the constraints encountered when using electrons and could significantly extend the scope and success of scanning microscopy.

1. Introduction
The scanning electron microscope (SEM) was the first practical imaging device that did not rely on visible light. It was conceived in Germany and the USA in the 1930s and 1940s, finally becoming a practical instrument through the work Prof. Oatley’s group in Cambridge [1] in 1951. Now, nearly 60 years later, present day SEMs are still recognisably direct descendants of that original design and they have become the most widely used high performance tools for imaging and microanalysis with more than 50,000+ instruments in operation world wide producing literally millions of images per day for every area of science and technology. However, even with the benefits of six decades of continuous development, the SEM is now struggling to maintain the level of performance required for current applications in nanoscience, and the worlds of biology, and soft materials. It is therefore necessary to ask if the SEM still has a future and if the current level of SEM performance can be sufficiently enhanced so as to satisfy ever more demanding specifications, or whether alternative technologies must now be considered as replacements.

2. How good is the SEM performance now?
It is regrettable that quantitative data on the imaging performance of SEMs now in operation is difficult to obtain and is little better than anecdotal in quality. This is because of the unwillingness of both manufacturers and users to accept the consequences and potential disappointments of employing techniques for determining resolution that are relevant, reliable, and consistent with those used in other areas of microscopy such as for the TEM, or advanced optical systems. The situation is compounded by the fact that the performance of an SEM depends strongly on the nature of the sample. As a consequence this has led to the development of test specimens such as gold particles on carbon -
whose only merit has been to offer unrealistically high contrast as compared to ‘real’ samples. As a result the data represented as ‘measuring’ SEM performance may be optimistic and provide little guidance as to either the real capabilities, or the operational deficiencies, of these instruments. The resolution of current production, top grade, SEMs is from visual observation certainly of the order of a nanometer (figure 1), and established methods such as the super-position diffractograms or Optical Transfer Function (OTF) analysis of the instrument [2] confirm that such machines can indeed retrieve some image information at the sub-nanometer level (figure 2). In fact several high performance SEMs have been shown to be capable of displaying bright field lattice fringes from carbon (0.32nm spacing) at 30keV [3] when operated in STEM mode (figure 3). Fringe imaging is not, of course, a measure of the probe size but does confirm that the high voltage supply, lens controller, and mechanical stability of the column, are of a quality comparable with those in a TEM. At lower beam energies (3keV or less) chromatic aberration and the reduction in source brightness become dominant factors causing a rapid fall in contrast and signal to noise although information retrieval down to 2-5nm is still achieved. While these levels of performance are an advance on those of a decade or more ago they remain marginal for the routine sub-nanometer scale imaging of most specimens, and are hardly competitive with scanned probe (AFM) tools or, far less, the TEM or STEM so significant enhancements in instrumentation must be sought to close this gap.

3. Can SEM performance be improved?
The imaging performance of an SEM is determined by the probe size at the sample - which is in turn set by the electron optics parameters - and by signal to noise - set by the brightness of the electron source and by the detectors that collect the signal. One important step that has already taken to improve SEM performance has been the replacement of tungsten hairpin thermionic sources with either cold field, or Schottky, emitters either of which offer a significant enhancement in source brightness giving higher currents into the final probe. This, in turn, enhances signal to noise and raises the highest spatial frequency at which image information is transferred to the observer. High brightness sources are also essential for the low voltage (i.e. sub-3keV) operation which is such an important part of contemporary SEM usage.
The detector in the SEM is the first step in the signal chain and is the place where the signal to noise is poorest, so detector efficiency has a major influence on the quality of SEM imaging. In addition a detector should be selective – collecting a maximum of the signal of interest and a minimum of unwanted contributions. Unfortunately it is probably true to say that SEM detectors are never designed, rather they are copied from one instrument to the next and if they generate an image then they are regarded as fully acceptable. Because of this neglect it is probable that there are still opportunities to improve image signal to noise, contrast, and information content by studying, designing and building with the same care given to other components in the instrument. In summary, although much could and should be done to enhance detector performance, there are no novel candidate sources whose brightness could exceed that of either cold field emission guns or Schottky emitters so further improvements in performance must be achieved from the electron-optics.

Three factors - chromatic aberration, spherical aberration, and diffraction limiting - determine the probe diameter and profile (figure 4). The dominant contributions are diffraction limiting and chromatic aberration and because these move in opposite senses as a function of the beam convergence $\alpha$ then there is an optimum value of $\alpha$ which gives the highest current into the smallest probe. Typically this optimum $\alpha$ is a few milliradians in size, a value which severely limits the incident beam current (which is proportional to $\alpha^2$) although providing a useful depth of field in the image (proportional to $1/\alpha$). The route taken by high performance STEMs and TEMs to overcome the constraints of these limitations is to ‘correct’ the spherical (Cs) and chromatic (Cc) aberrations associated with the probe forming, or objective, lens. Reducing Cs and Cc magnitudes from millimeters to micrometers shifts the optimum beam convergence from a few mrads. to 30 or 40mrads, making it possible to generate sub-angstrom probes containing 20-30x more current than for the equivalent uncorrected system. The same procedure can be applied to the SEM [4], and prototype aberration corrected SEMs (ACSEMs) have been successfully demonstrated by several companies (for example FEI Inc. and JEOL Inc.) although only one such instrument – from Holon – seems to have made its way into production.

In fact there are several reasons why a full fledged ACSEM is unlikely to become the SEM of the future. First, the increase in beam convergence $\alpha$ made possible by aberration correction reduces the imaging depth of field at high magnification settings to a nanometer or less – which makes the ACSEM of little use for real world samples. Second, optimization of aberration correctors is achieved by a complex, computer controlled, iterative, procedure. For TEMs and STEMs this is guided by the analysis of the Ronchigrams generated from ultra-thin transparent metal foils, but for an SEM imaging
a bulk sample there is no equivalent of a Ronchigram and so the correctors must be adjusted by observing the real space image. This is slow, unreliable, hard to optimize, and easily confused by effects such as sample charging and contamination.

Two limited forms of aberration correction for SEMs are, despite these issues, presently in use. One, the retarding field mode – is in fact the most widely used form of aberration correction for any electron beam system. In this technique, the incident beam of energy $E_B$ is abruptly decelerated to some lower final energy $E_F$ over a distance $L$ in the region between the probe forming lens and the sample surface. In this case the effective aberration coefficients of the combined optical system are then \[ Cs = -Cc = L \frac{E_F}{E_B} \]

and so are independent of the actual lens parameters. For example, a 5keV beam decelerated to 500eV over a distance of 1mm creates effective aberration coefficients in the micrometer range. Significant enhancements in resolution are achievable with minimal effort and consequently this approach has become the standard for the semiconductor industry’s “Critical Dimension-SEMs” which perform nanometer scale metrology of devices. However the retardation mode is difficult to engineer if high landing energies are required, and does not work well for samples which are small in size, or have prominent topography. Alternatively, the electron beam can be ‘mono-chromated’ to reduce its energy spread ($\Delta E$) and hence the associated chromatic aberration error ($\sim \alpha \Delta E/E$) to enhance performance. The “Uni Colore” system from FEI Inc. employs a dispersive lens structure built into the Schottky emitter source which is paired with a slit aperture to eliminate the lowest energy electrons from the beam and so reduce the energy spread from about ~0.7eV to below 0.3eV - a value comparable with a cold field emission source. The improvement in resolution achieved at low energies is significant and requires no elaborate alignment or optimization but this enhancement comes at the expense of total beam current and is of less value at high energies (10keV and above).

4. What happens next?
Within the energy range 0.5-30keV over which the SEM is most commonly used, and even allowing for the more widespread adoption of the kinds of improvements discussed above, there is little scope for substantial gains in SEM performance. Firstly, electrons have a wavelength which at low energies is a significant fraction of a nanometer in size making diffraction limiting an impassable barrier. Increasing the beam energy minimizes this problem but also leads to a rapid rise in beam penetration. Thus better resolution is achieved - but only at the expense of losing surface specific detail in favour of deep sub-surface information carried by backscattered electrons. While the enhanced brightness of the electron source at higher energies puts more current into the focused probe this gain is offset by the reduction in signal intensity which results from the reduction in secondary electron yield that occurs. There is, thus, neither any instrumental development nor optimal of operational conditions which can sufficiently enhance the performance to maintain the competitive position of the SEM. Comparable alternative technologies must therefore be devised and exploited instead.

5. Ion imaging – the new scanning microscopy?
A promising option is to consider the use of ion, rather than electron, beams. As first pointed out by Levi-Setti more than thirty years ago [6], ions are much more massive than electrons and so their wavelength $\lambda$ is at least 50 to 100 x smaller than that of an electron of the same energy. Since scanning beam systems are always ultimately limited in performance by diffraction, reducing $\lambda$ by such a large factor makes a substantial improvement in resolution a definite possibility. A further consequence of the large mass of an ion relative to an electron is that, at the same energy, its velocity is much smaller. For example, for He + ions (mass 7297x that of an electron) their velocity $v_{He}$ compared to that of an electron $v_e$ of the same energy is (under non-relativistic conditions)

$$v_{He} = v_e * 0.012$$
This difference in velocities has important implications. When a charged particle enters a material the rate at which its energy is lost depends on the ‘stopping power’ of the material which in turn determines not only the range of the particle but also the profile of the energy deposition along the trajectory and, consequently, the magnitude and depth distribution of the various interactions and signal emissions that occur. Thompson [7] showed that stopping power is a function of particle velocity not energy and if, as shown in figure (5) the stopping power of electrons and of ions is plotted as a function of their velocity then the normalized curves resemble each other closely although their absolute magnitude may differ significantly. For both projectiles the maximum in the stopping power occurs at a velocity of about $6 \times 10^8$ cm/sec which for electrons correspond to an energy of about 200eV but to about 700keV for a He$^+$ ion. Electron microscopes thus operate on the right hand side of the profile where the stopping power falls with increasing energy – and consequently as the energy is increased the range increases rapidly, most of the energy is deposited at the end of the range i.e. deep in the sample, and secondary electron and other emissions fall as the energy rises. It is this chain of events which leads to the problem of ‘optimizing’ the SEM discussed above. For ions the situation is quite different. A proton or a He$^+$ ion microscope operating at 30-50keV is on the left hand side of the profile where stopping power is rising with energy. An increase in beam voltage therefore results in only a relatively modest increase in range, the majority of signal generation occurs at the surface where the energy and hence the stopping power is highest, and the overall SE yield rises. Because of this behaviour for ion beams all parameters of interest - from source brightness and wavelength to beam range and signal production – are simultaneously optimized as the energy is increased.

6. The Scanning Ion Microscope (SIM)
To exploit the potential of its greatly reduced wavelength a scanning ion microscope (SIM), like any SEM, also requires a high performance beam source and quality lenses. As shown by Levi-Setti [7] protons can be generated in a standard Butler-Crewe cold field emission gun by the expedient of replacing the usual high vacuum with a low pressure of hydrogen. Helium ions, in the ZeissSMT ‘Orion’ instrument [8], are generated from a field ionization source developed from that devised by Muller in 1951 for the field ion microscope. In both cases the ion source can be operated at modest
vacuum levels, and exhibits both a brightness which is comparable with a field emission electron gun and a nanometer source size. Ion optical lenses are usually of the simple ‘Einzel’ electrostatic type but offer competitive aberration characteristics. All of the components required for competitive ion microscope performance are therefore already available. Ion induced secondary electron (iSE) emission is - as in an SEM – the imaging mechanism of the most immediate interest. The iSE yield (iSE/ion) is a factor of 5 to 10 time higher than the electron yield (SE/electron) from the same material and rises with beam energy only reaching a maximum for protons and He+ at energies in the range 400-700keV. Consequently the signal to noise of a SIM is superior to that of an SEM for comparable beam currents. For protons and helium ions the effective beam range R (nm) in a material of density ρ (gm/cc) at energy E(keV) is well described \([9]\) by the relation

\[ R_{\text{ion}} = 80.(E^{0.73}/\rho) \] (3)

which compares with the Kanaya-Okayama \([10]\) estimate of

\[ R_{\text{electron}} = 80.(E^{1.66}/\rho) \] (4)

for electrons. The ratio of electron to ion beam range is therefore of the order of magnitude ‘E’ (i.e. 30x more at 30keV) confirming that the iSE image will always contain significantly more surface information than the electron signal at all energies. The iSE image is further enhanced by differences in the origin of the SE between electrons and ions. For electron irradiation typical 50% of the total SE signal is generated by exiting backscattered electrons, the resultant ‘SE2’ contribution being of poor resolution as compared to the SE1 contribution generated by the incident beam. In the case of ions while the ratio iSE2/iSE1 is comparable to the value for electrons at low energies this falls steadily with increasing energy both because of the reduction in the ion backscatter yield and because the lower stopping power of the backscattered ions reduces their iSE generation efficiency. Consequently at higher energies the iSE signal tends towards being pure SE1 in character which further improves the contrast of the high resolution image. In addition (figure 6) ion induced SE images display high sensitivity to changes in surface chemistry, high contrast from channelling interactions with the crystallography of a sample, and an impressive depth of field which is the result of the small convergence angle (less than 1 milli-rad) typically chosen.

Figure 6. Helium ion beam secondary electron image of an aluminum registration mark. Note the clarity of surface topography, the visibility of surface contamination, and the large depth of field. Beam energy 35keV, horizontal field of view 4.5 micrometers. Image courtesy Dr. L.Scipioni, ZeissSMT
Figure (7) shows an example of a He+ ion SE image of a gold on carbon specimen recorded at 35keV. The material contrast between the gold and carbon is strong and clearly reveals detail that is not evident in electron induced SE images of the same material. Diffractogram and contrast transfer function analysis of such images demonstrates the information limit to be well below 1nm, and ultimately to be limited by signal to noise rather than by probe size or mechanical instability. The beam range, and secondary electron yields, from both proton and helium beams are quite similar at the same energy and both have the virtue that, because of their low mass and high velocity, their nuclear stopping power component is small and falls with increasing energy so they cause little sputter damage from specimens. Scanning ion microscopes using argon or gallium beams would also offer many valuable benefits. However, because such ions are massive compared to H+ and He+ high beam energies (>1MeV) are necessary to generate useful secondary electrons yields comparable to those produced by lighter ions. Heavy ion beams are also more prone to exhibit multiple ionization states which increase the chromatic spread of the beam and so degrade the probe size, and to producing significant sample damage as the result of their substantial nuclear interactions.

Scanning ion microscopes do also have some operational limitations. Even for H+ and He+ beams the probe current may need to be restricted to the 1-10pA range in order to minimize damage, to nanoscale objects, and to limit the energy broadening of the beam which could occur as the result of the Boersch effect [11]. Sample charging which unlike the electron situation is always positive in polarity, and which is substantially higher in intensity than that encountered with electrons, is also to be expected on all poorly conducting and insulating samples unless some form of charge compensation such as a low energy electron flood gun is applied. Finally, X-ray microanalysis by light ion beams is not generally possible except at beam energies in excess of 1MeV [12] although energy spectroscopy of the Rutherford Back Scattered ion (RBS) signal might provide a method for surface analysis with extremely high depth resolution.
7. Conclusions
The SEM has established itself as a uniquely powerful and versatile tool for imaging and analysis, but
the performance of the instrument is now constrained by limits set by the wavelength of the electron,
and the problem that there is no one beam energy which permits all of the parameters of performance
to be simultaneously optimized. A scanning microscope which employs a beam of light ions combines the convenience and familiarity of the SEM with advantages that come from the shorter wavelength of ions and their higher stopping power. This in turn leads to restricted beam ranges, enhanced surface sensitivity, and higher secondary electron yields. Contrast effects associated with sample crystallography, and with surface chemistry, are an order of magnitude stronger than for an electron beam. Increasing the energy of the ion beam to 100 or 200keV from the current 30 to 50keV levels would increase source brightness by a factor of 10x, reduce the ion wavelength by a factor of 3x, and increase secondary yields by a factor of 3-4x while only increasing beam range by a factor of 3-4x. Such an instrument would have the potential to achieve sub-angstrom resolution in SE mode -without aberration correction of any type - under such conditions, and would position Scanning Microscopy to remain the universal imaging technique of choice.

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