Basic Principles:

Electron Beam vs. Optical Imaging of Particles

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The only realistic way to understand a population of particles is to measure a sufficiently large distribution of individuals relative to pertinent parameters such as size, shape, and composition. In nearly every case, the most reliable technique for obtaining such a distribution involves the direct imaging of particles such that their dimensional attributes can be accurately established. With today's technology, such characterization can be conveniently performed with a computer-interfaced microscope automatically scanning a substrate on which the particles have been deposited – typically a filter medium.

When microscopic analysis is mentioned, particularly in the context of a routine industrial-type process, a natural reaction is to assume that the familiar light-optical microscope is the obvious candidate. Even many who are aware of the exceptional capabilities of electron-beam instruments (as exemplified by the Scanning Electron Microscope, or SEM) may assume that such instruments are simply too 'high end' for routine applications – the conventional perception of the SEM is that it is an expensive and complex device that cannot be justified unless very high magnifications (i.e., very small particles) are involved. This is a perception that quality-conscious companies around the world are proving false as they increasingly choose electron-beam imaging for critical particle analysis in the automotive, pharmaceutical, defense, mining, steel, forensic and other industries and applications.

A modern electron beam particle analyzer of the type that ASPEX manufactures is a sturdy tool that is at home on the factory floor (or even more hostile environments). Surprisingly to many, the cost of a suitably equipped automated optical unit can approach that of our electron-beam solutions. And when total capability is factored in, the scales tip decidedly in favor of electron beam analysis – even (especially) for coarse particles.

When accurate assessment of particle populations is important, the technique chosen should have the capability to produce trustworthy results. It is the intent of this discussion to simply set out the key technical reasons why the automated electron-beam analyzer should be considered for any program involving the critical analysis of inorganic particulate material in the millimeter to sub-micron range.

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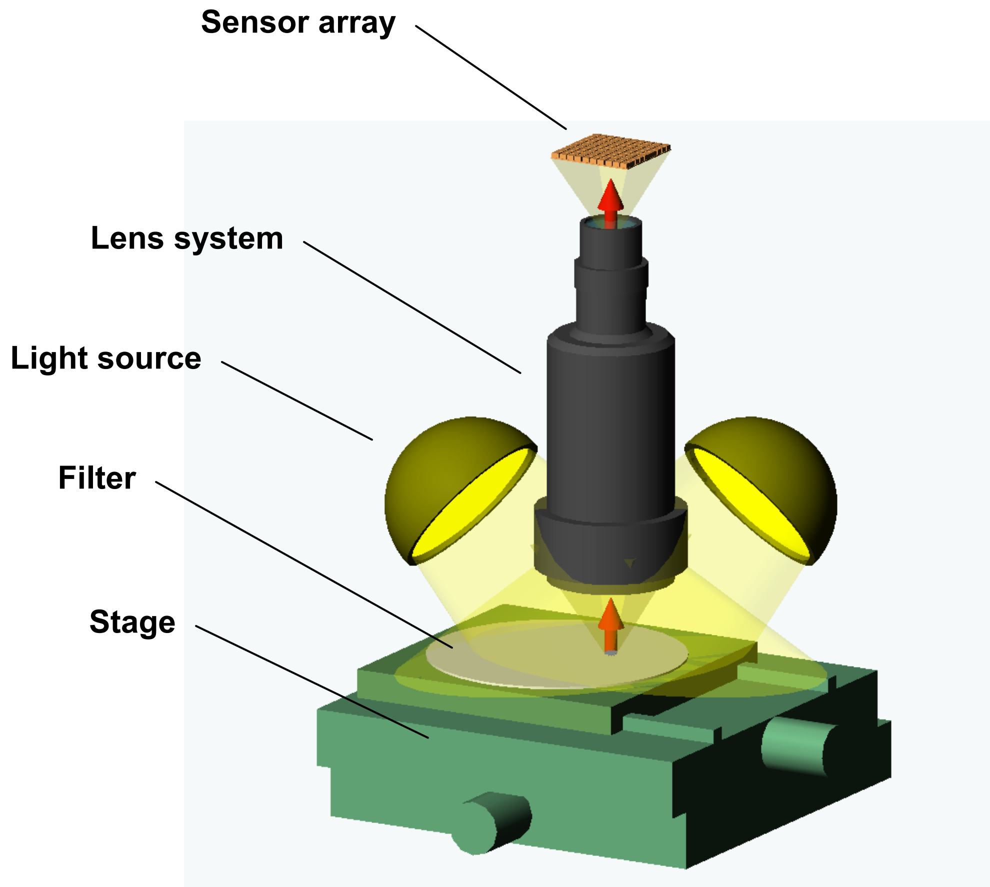
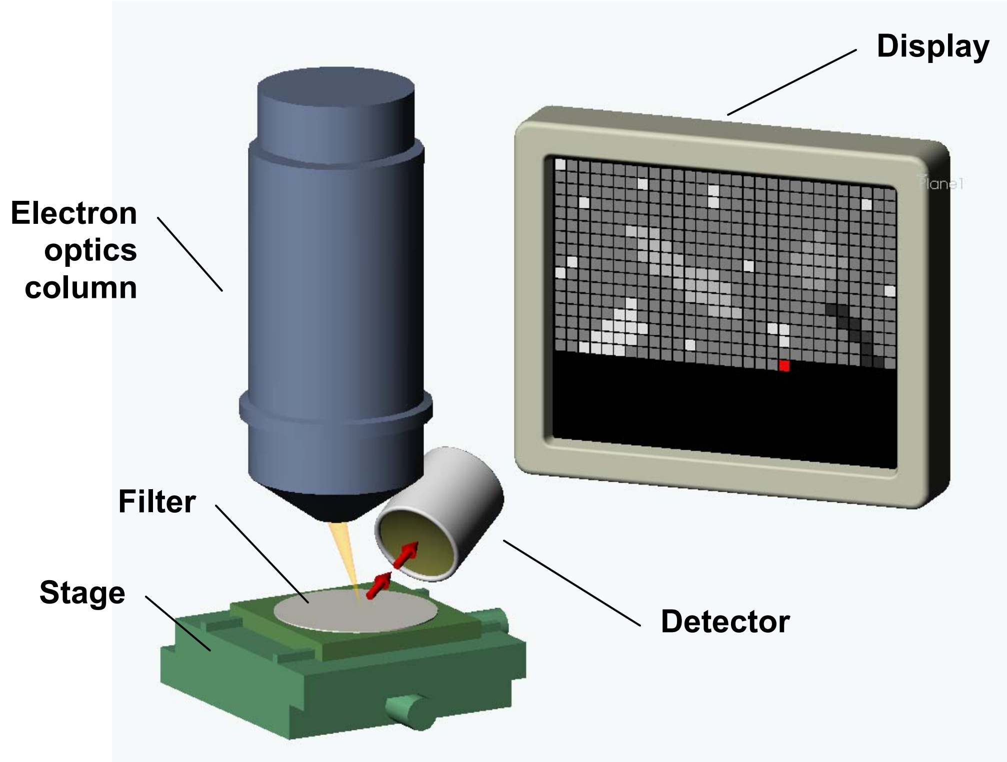
Principle of the Light-Optical Microscope



The figure schematically illustrates the general principle of an optical microscope configured for automated particle analysis. The filter sample is mounted on a motorized stage and illuminated by a diffuse source of light. The image of a tiny portion (field) of the specimen is projected via the lens system onto the sensor array such that each 'pixel' (picture element) is recorded simultaneously as a 'color' value (RGB intensity value). The spatial area of the sample viewed in each field is dictated by the strength of the lens system and the size of the sensor array.

The sensor array is read into a computer memory (not shown) where the image is processed by software to recognize particle boundaries and extract size and shape parameters. When analysis of one field of particles is completed, the motorized stage shifts another field into view.

Principle of the Scanning Electron Microscope (SEM)



The principle of the Scanning Electron Microscope (SEM) is schematically illustrated in the figure. The electron optics 'column' produces a highly focused beam of electrons that impinges on a point of the filter specimen. The interaction of the beam with the specimen induces a signal that is sensed by the detector. The sensed intensity of the response varies as the beam is sequentially scanned over consecutive points of the specimen and this variation of intensity is recorded in the computer memory as a gray-scale image. The spatial area of the image collected is dictated by the spacing between adjacent beam points on the specimen.

Electronic displacement of the beam allows multiple particle fields to be analyzed without moving the stage; stage motion is reserved for large field displacements.

Since a focused electron beam can be produced only within a relatively high vacuum, the beam, specimen, and detector are housed in a vacuum enclosure (not shown).

Useful magnification

The useful magnification* that can be achieved by an optical microscope is constrained by the diffraction of light in the lens system and is limited to an ultimate value of approximately 1000X. However, in practice, optical magnifications of greater than 100X are rarely employed for particle analysis tasks.

The useful magnification that can be achieved by a SEM is defined by the size of the focused spot projected on the specimen, and this too is ultimately limited by the diffraction of the focused electron beam.

A great deal could be said about the theoretical diffraction-limited resolution of both the SEM and the optical microscope. On the basis of wavelength alone, an overly-simplistic analysis would predict the SEM to be capable of more than four orders of magnitude greater ultimate magnification. However, after other practical considerations are factored in, a general rule-of-thumb is that SEMs used in industrial applications** are routinely capable of producing useful magnifications about 100 times greater than that of their light-optical counterparts. As a consequence, whereas an optical microscope has difficulty imaging particles down to the 20 micron size, a SEM begins to ‘run out of steam’ for practical imaging of particles below about 200 nanometers.

In practice, the ability of a SEM to optically resolve small features is rarely, if ever, the limiting factor in its practical utility for automated particle analysis. Rather, it is the susceptibility of the SEM’s mechanical systems to environmental factors such as vibration and thermal drift which typically defines the limits of practical application. Fortunately, such issues are not limiting factors for the particle sizes of concern to the vast majority of applications – typically micron size and above.

Surprisingly to the many who falsely assume that the virtues of an electron microscope solely involve high magnification, it will be seen that in many practical applications the magnification advantage, though clearly an important consideration, ends up being less significant than others.

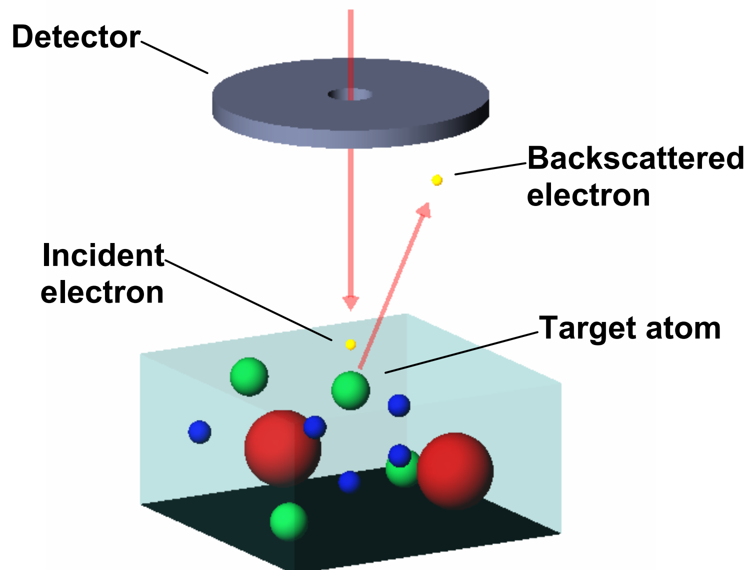
*The concept of ‘magnification’ is not useful in any absolute sense for defining the imaging performance of computer-collected images, since it implies the projection of the ‘magnified’ image on a display screen of particular size, but since most people have a practical sense of the visual magnification achieved by lens systems, the term is here used as a basis of comparison.

** The present discussion is applicable to tungsten-source SEMs operating in the 10-30 kilovolt beam energy range typical for routine microanalysis applications.

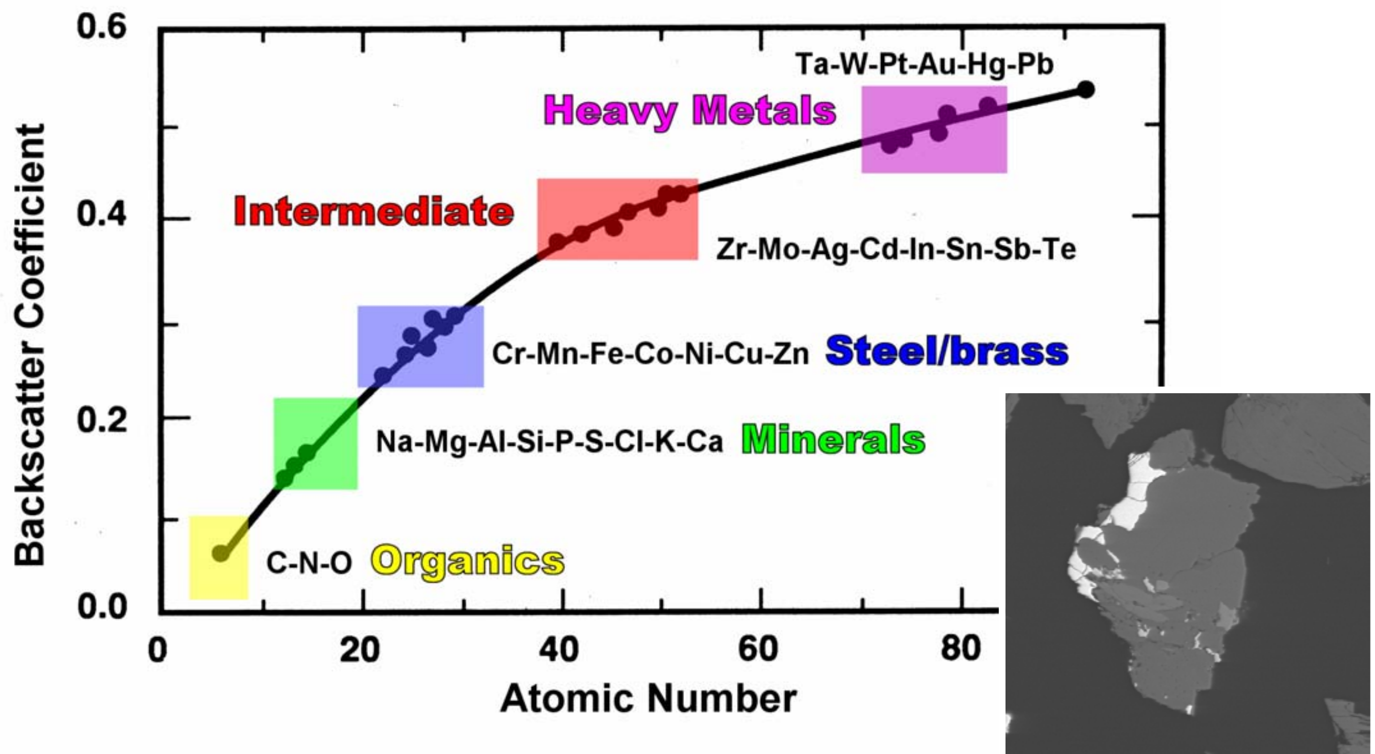
Contrast Mechanisms

The information in an image comes from the various contrast mechanisms by which a subject feature interacts with the illumination source to produce variations in the reflected signal. The rich and subtle contrast mechanisms involved with the interplay of light and matter endow our visual world with great beauty, but can also make interpretation of visual images 'beastly' difficult. Color, for example, is a powerful visual cue, but one whose origins involve so many complexities as to often obscure, rather than enhance the interpretation of images.

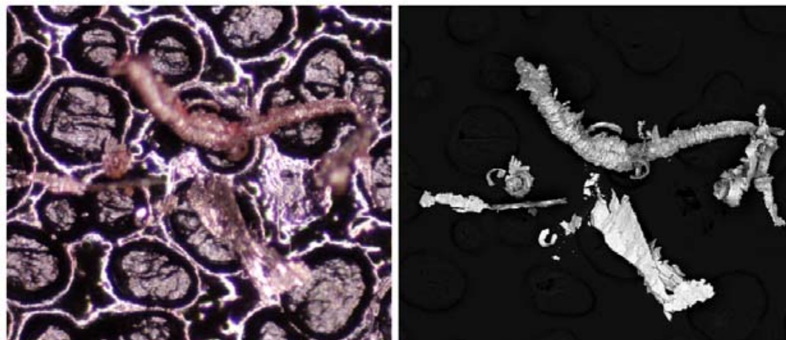
By way of contrast (pun intended), electron-beam interaction mechanisms tend to provide simpler and more useful means of feature discrimination. The signal that is most commonly used for producing SEM pictures (micrographs) is the **secondary electron** response, which is very sensitive to surface detail, and with its realistic shadowing, produces a pleasing 'three dimensional' appearance. However, the less familiar phenomenon of **backscattered electrons** provides a contrast mechanism that is particularly useful in automated particle applications.



The backscatter contrast mechanism involves the elastic scattering of incident electrons off of the atoms of the specimen. Since even the lightest atoms are orders of magnitude more massive than electrons, the effect is like a ping-pong ball bouncing off a bowling ball – the electron recoils with nearly all of its initial energy. An annular detector element arrayed symmetrically around the incident beam intercepts these backscattered electrons. Though the illustration is not even remotely close to true scale, it does correctly suggest that the probability of a backscattered event will be proportional to the average size of the atoms in the specimen material.



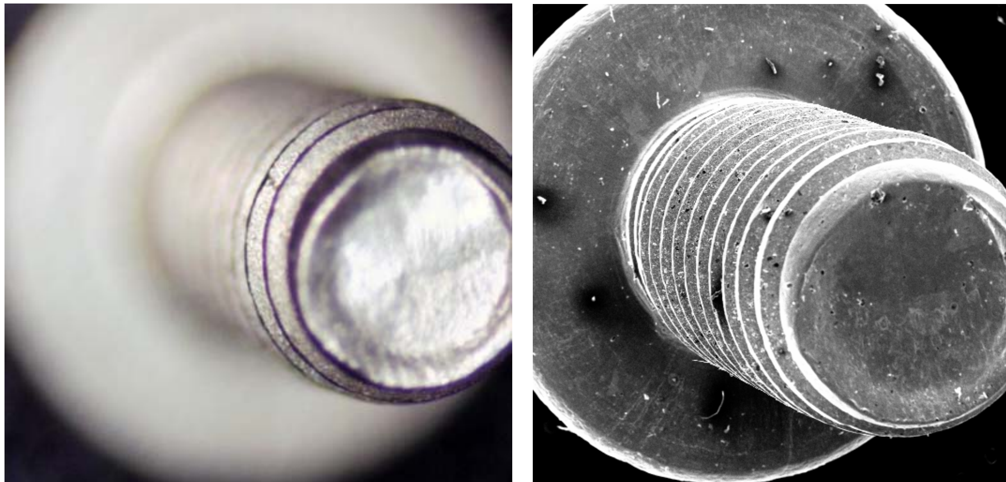
The backscattered electron signal gives rise to highly predictable atomic number contrast, as indicated in the above graph. Because the probability of backscatter recoil is proportional to the average atom size at the point where the beam strikes the sample, an abundance of large massive atoms produces a stronger signal than lighter ones. As shown in the graph, this trend is highly regular and can be used to differentiate different categories of materials by their brightness alone. The inset shows the image of a particle of ore mineral imbedded in a plastic mounting medium. Because the plastic is a low atomic number material (principally hydrogen, oxygen, and carbon) it registers as virtually black. The 'gangue' (commercially worthless mineral matrix) registers as a dark gray, whereas the valuable metallic ore components show up as various lighter shades



This final pair of images powerfully demonstrates the advantage of the backscattered electron contrast mechanism for the automatic characterization of particles. Both images are of the same metallic particles extracted from an automatic transmission and mounted on a carbonaceous adhesive substrate. The image on the left was obtained with a 50X optical microscope. The image on the right was obtained with the Personal SEM using backscattered electron contrast. It goes without saying that establishing the true number and dimensions of the particles from the right image (SEM) is going to be a vastly simpler and consequently more accurate enterprise than analyzing the optical image on the left.

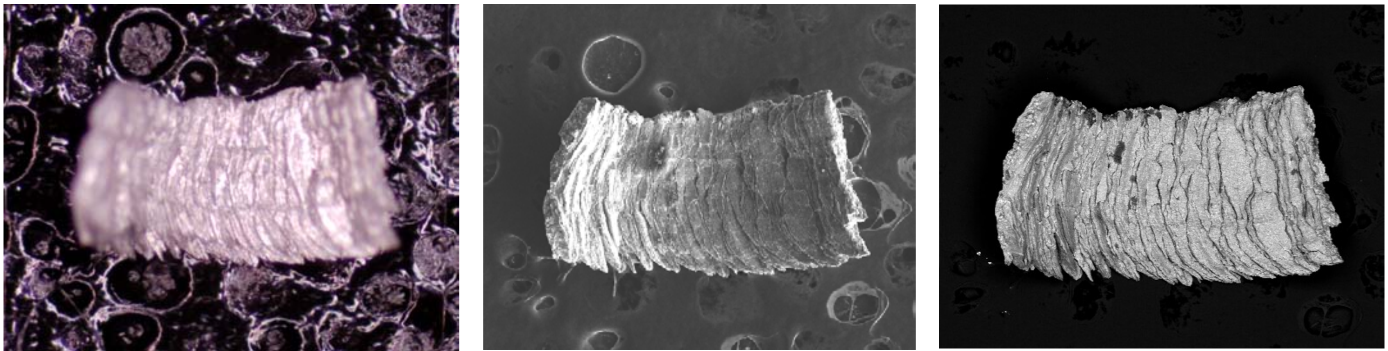
Depth of focus

Any photographer knows that a camera lens needs to be refocused when moving between near and far objects in order to render images crisply. When both distant and close objects are in the field of view, one or the other (or both) will be out of focus. Strictly speaking, a subject is in perfect focus at only one precise distance, but in practical terms, the image remains suitably sharp over a range of distances, which is termed the ***depth of focus*** (or equivalently, depth of field). In general terms, the depth of focus becomes large when the distance to the object is much larger than the diameter of the limiting aperture of the lens. Because optical microscopes need to employ rather large lenses at close quarters in order achieve high magnification, they have an intrinsically small depth of focus. By comparison, because the limiting aperture of a SEM must be made very tiny, it has a correspondingly huge depth of focus – easily thousands of times greater than an optical microscope in ordinary situations!



The pictures tell the story. The object is a small screw, about ¼ inch in length. When viewed with an optical microscope as on the left, only a very narrow slice of the depth is in satisfactory focus. By comparison, the PSEM image on the right remains in sharp focus over the entire length of the screw (and beyond)! (By the way, the PSEM image in this case was rendered with the secondary electron detector, which does not alter the depth of focus, but does provide realistic depth cues that help our brain interpret the image as a three-dimensional object.)

In numerical terms, the depth of focus for an optical microscope equipped with a 100X objective is typically under a micron, whereas for a typical PSEM configuration, it can be over ten centimeters at the same magnification! At higher magnifications, the depth of field of the SEM does become much smaller, but is always orders of magnitude larger than for an optical microscope.



These three images of the same metal chip (approximately 1 mm in length) illustrate the superiority of electron-beam analysis for particles. The image on the left is again via an optical microscope. The one in the center was acquired by a PSEM using the secondary electron detector, and the one on the right with its backscattered electron detector. We note how the modest curvature of this chip causes it to go out of focus at the edges in the optical image whereas the two PSEM images are crisply rendered everywhere. Although the secondary electron image in the center is the most visually 'interesting' due to its rich surface detail and shadowing, the backscattered image at the right, with its 'flat' contrast, best lends itself to rapid and accurate sizing. (For automated analysis, 'boring' images are best!)

These images illustrate the surprising fact that even for rather large particles, where the limited magnification of the optical microscope is not an issue, the huge depth of field and powerful contrast mechanisms available via electron-beam imaging make it the clear choice for accurate analysis.

But what about small particles? If particles are small and uniform in height, or perhaps even flat, shouldn't an optical microscope be able to render them more consistently? In principle, "yes", but in practice, particles will be presented on substrates that cannot be relied upon to be perfectly flat. For analysis of particles on filters, for example, a typical experience is that optical microscopy is not reliable for particles below about 50 microns in size.

By the way, the depth of field limitations of an optical microscope as illustrated here may not be as apparent when a sample is viewed through the microscope's eyepieces. This is because the human eye adds an additional ability to 'fine focus' the image and thus the apparent depth of focus is increased. However, this useful feature of the human visual system isn't available for static images.

Dynamic Control

There seems to be a natural assumption that electron microscopes are intrinsically more complicated, fragile, and costly than their light-optical cousins. Such truth as there is to this perception can be attributed to the fact that basic optical microscopes are fundamentally simple mechanical devices with centuries of evolution behind them, whereas the SEM is a relatively recent electronic development which has not, until recently, significantly evolved from its research-laboratory roots.

There is no question that a basic optical inspection microscope is fundamentally a smaller, simpler, less expensive unit than even the most basic SEM. However, try to automate such a unit with anywhere near the versatility enjoyed by the PSEM, and the perspective changes radically. The simple fact is that an optical microscope is a fundamentally mechanical device that requires the addition of complex and sophisticated actuators in order to provide computer control of such basic attributes as magnification, illumination, and focus. Similarly, the optical microscope evolved as an adjunct to the human eye, and when it is to be used for automated imaging, it must be equipped with an electronic camera and readout. A SEM's optical system by contrast, is intrinsically electronic, and a significant part of the cost and complexity of old-style SEMs involved the interface to the human operator, providing mechanical controls to manipulate and a display to view. In the simplest terms, a SEM is an imaging device which seems made for computer control whereas an optical microscope is usefully interfaced to a computer only with considerable effort.

This basic fact has obvious ramifications relative to cost and reliability. The cost of a suitably high-grade automated optical microscope system comes as a shock to many. And the kind of actuator required to adjust the focus of an optical microscope, for example, involves mechanical components subject to wear and failure. By contrast, adjustment of the magnetic focusing lens of a SEM involves only a change in drive current – a simple matter for modern electronics and there is nothing to 'wear out'. But the biggest virtue of the electron-beam imaging system is the speed and dynamic range over which it can be adjusted. Even sophisticated optical 'zoom' lenses struggle to approach a factor of ten in magnification change, whereas any SEM can 'zoom' continuously through at least four orders of magnification change – and do so virtually instantly, without any kind of mechanical adjustment. As a consequence, a truly optimized electron-beam particle imaging system, such as the units engineered by ASPEX, can dynamically adjust to imaging requirements in ways that no optical system can approach.

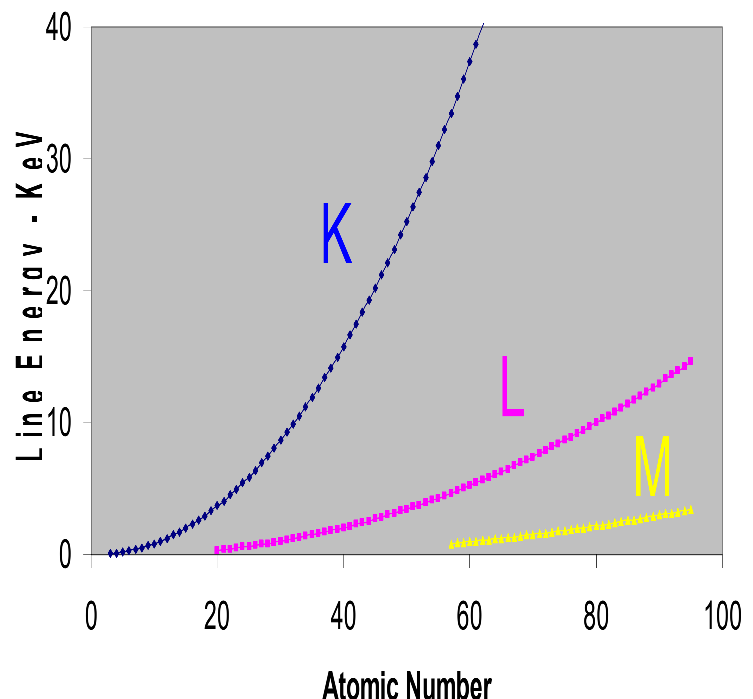
Compositional Analysis

It's quite easy to compare the compositional analysis capabilities of the electron-beam-based particle analyzer with its light-optical counterpart – the latter has none! To be sure, light microscope systems can be programmed to make use of color cues, but this is an inexact and unreliable way to characterize composition. Though there are optical spectroscopies that provide chemical information, they have not yet proven robust enough for routine automated particle identification applications. But electron beam instruments have for the past fifty years routinely analyzed the x-ray emissions that are produced by the beam's interaction with the specimen to obtain accurate compositional information.

The material processes that produce visible light involve *molecular* interactions. By contrast, x-ray production derives from *atomic* effects. Simply speaking, an energetic electron penetrates deeply into an atom's core structure and thus is sensitive only to the species of atom. Visible light, which is thousands of times less energetic, interacts at the much-more weakly bound outer levels where atoms interact with each other to form molecules. Since molecular chemistry can be incredibly complicated, spectroscopies based on its light emissions are also – though there is a wealth of information carried in the visible light signature, it defies simple 'rules'. For example, there are undoubtedly thousands, if not millions, of ways by which light of a specific wave-length can be produced, and though a particular molecular structure can be relied upon to produce that wavelength, there are just too many alternatives to permit a simple identification of a compound on that basis alone.

X-ray production, however, since it occurs deep in the atom where it is not affected by factors such as temperature, pressure, and chemical bonding, is characterized by simple and extremely consistent emission patterns.

Moseley's Law states that the energy of an element's characteristic x-ray emissions will vary as the square of the element's atomic number (see graph). This relationship, first discovered nearly 100 years ago, is so predictable that it was the means by which the proper order of the periodic table of the elements was finally established!



Since every element above lithium produces characteristic x-rays when excited by energetic electrons, the detection of such emissions thus provides a simple and unambiguous way of identifying which elements are present within a sample – and with care, the relative abundance of each. Consequently, during automated particle analysis by an electron beam, the final step involved in classifying a particle is often to collect its x-ray spectrum. From analysis of this spectrum the particle then can be placed in an appropriate material category, such as a particular alloy of stainless steel, for example. Such analysis, which in a modern unit can be conducted within seconds or less, thus provides a definitive identification of the elemental composition of the particulate material.

X-ray analysis is not 'perfect', in the sense that it cannot discriminate between two compounds with identical elemental constituents that are chemically combined in different ways (i.e., different molecular structure). Thus, x-ray analysis is not a useful means of differentiating most organic compounds, which are composed principally of carbon, oxygen, and hydrogen linked up in an enormous variety of configurations. However, for identification of most inorganic materials (as well as those organic compounds characterized by significant constituents of other elements, such as sulfur, chlorine, fluorine, etc.) this is the most powerful technique available. For rapid and accurate identification of metallic or mineral particulates, the capability to perform rapid and precise x-ray analysis on particles with spatial dimensions down into the micron range establishes electron-beam analysis as the premier technique for particle classification.