X-ray Analysis in the Low Vacuum SEM (Part 3 of 3)

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Experimental Measurement of Beam Scatter

In this third and final installment on x-ray analysis in the environmental and low vacuum SEM, I will present experimental methods for measuring beam scatter. In my previous two articles I discussed how operating conditions determine beam scatter. It was shown that the type of gas used, the gas pressure in the chamber, the working distance or beam gas path length, and the accelerating voltage all have an effect on how much the electron beam scatters. I also discussed how the beam scatter influences x-ray results by producing x-rays beyond the area of the primary beam. Furthermore, I showed how software models could be used to determine the amount of beam scatter based on different combinations of the four variables (pressure, gas, working distance, and kV).

First, a quick review. In a SEM not under high vacuum, the electron beam travelling from the final aperture in the electron column to the sample surface encounters gas molecules and interacts with these molecules. The result is a beam that starts to look less like a straight-line and more like a cone (Figure 1). The width of the cone (amount of beam scatter) depends on the type of gas in the chamber, the gas pressure, the distance the beam travels in the gas, and the accelerating voltage. The scatter does not take place evenly, *i.e.*, the intensity of the electron beam is not the same at all points along the base of the cone. Under most conditions encountered in an environmental or low vacuum SEM, a majority of electrons are not scattered and remain in the original beam. To get an accurate view of the beam spread one needs to know not only the diameter at the base of the "electron cone", but the intensity profile of the electron beam across this distance (Figure 2). This histogram displays the beam intensity vs. position on a log vertical scale to fit all the data onto the plot. The log vertical scale in Figure 2 makes it look like the beam scatter is a Gaussian distribution, but for the typical range of analysis conditions most of the beam remains unscattered. As indicated by the intensity scale on the right side of the plot, approximately 90% of the beam is unscattered in this example. Also in this example, the beam intensity drops off very quickly a short distance away from the original beam line. The green center portion of the histogram indicates where 80% of the beam scatter takes place. It can be assumed that the 10% of scatter at the edges, shown by the red regions of the histogram, do not produce a statistically significant x-ray signal from the sample.

What has not been discussed so far is how beam scatter can be measured experimentally. What may seem simple at first glance turns out to be quite a difficult problem. Direct and indirect techniques for measuring beam scatter, and their advantages and drawbacks will be discussed. Direct measurement involves taking measurements of the electron beam with some type of electron detecting mechanism. A simple example would be using a piece of film. The cone of electrons formed by the scatter of the electron beam would expose a spot of some diameter on the film. The spot on the exposed film could be measured. Such a technique would provide a reasonably simple method of measuring the diameter of the scattered beam. The biggest drawback to this method involves getting accurate intensity values for the electron beam by correlating beam strength to brightness of the image. The dynamic range of film limits the degree of accuracy one can achieve by this method. The ideal direct measurement would be an electron detector that can be placed under the beam and give a reading of beam diameter and intensity with high spatial resolution. Unfortunately such a detector does not exist. Luckily, there are some simpler techniques available to measure beam scatter indirectly, although these have their limitations.

A more complex variation of the direct measurement method was tried at the National Institute for Standards and Technology (NIST). A monolayer of organic material was deposited on a polished silicon substrate. This sample was then exposed to the electron beam in an environmental SEM. The sample was removed and immersed in a second organic liquid where fluorine ions attached themselves to the beam-damaged area. Subsequent analysis of the sample by secondary ion mass spectroscopy (SIMS) produced a chemical profile of fluorine across the beam-damaged areas. The intensity of the fluorine signal was then correlated to the diameter and intensity of the scattered electron beam. While beam scatter and intensity were measured, several problems were encountered with this technique. High background noise in the form of a low level fluorine signal made it difficult to get a sharp measurement of beam diameter. Also, the organic material reached a saturation point with respect to beam damage and therefore could not provide an accurate measure of electron intensity in the area of the original beam spot. This dynamic range limitation is a typical problem with most direct measurement techniques. The spatial resolution of this technique was limited to about +/- 6 microns. Such resolution prohibits measuring beam scatter at low chamber pressures and short working distances where the amount of beam scatter may be less than the resolution of the technique. Furthermore, this is a complex technique that is not practical outside a specialized research lab.

Advantages:

- Provides a direct method of measuring beam scatter and intensity

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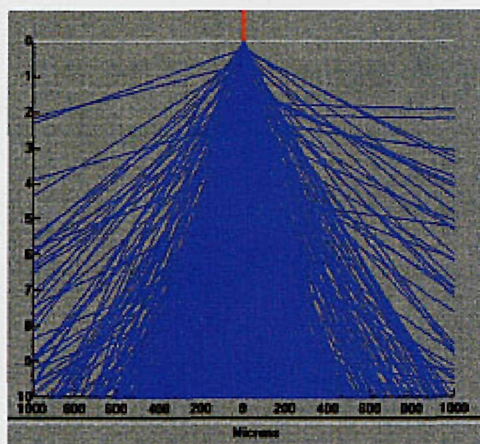

Figure 1: Beam scatter plot showing electron scatter more than 1000 microns on either side of the beam. Vertical scale shows working distance in mm from the final aperture to the sample surface.

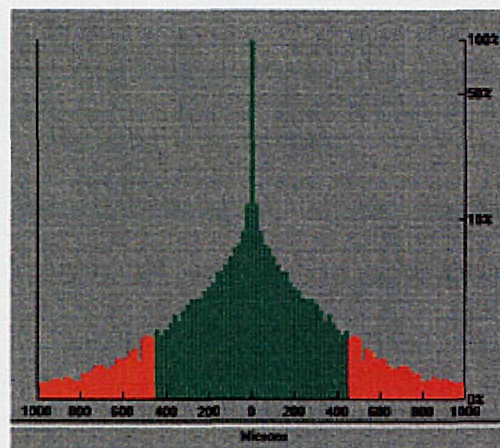

Figure 2: Histogram of electron intensity vs. distance from the beam. Vertical log scale shows percent of original beam intensity. Approximately 80% of beam scatter takes place in green center portion.

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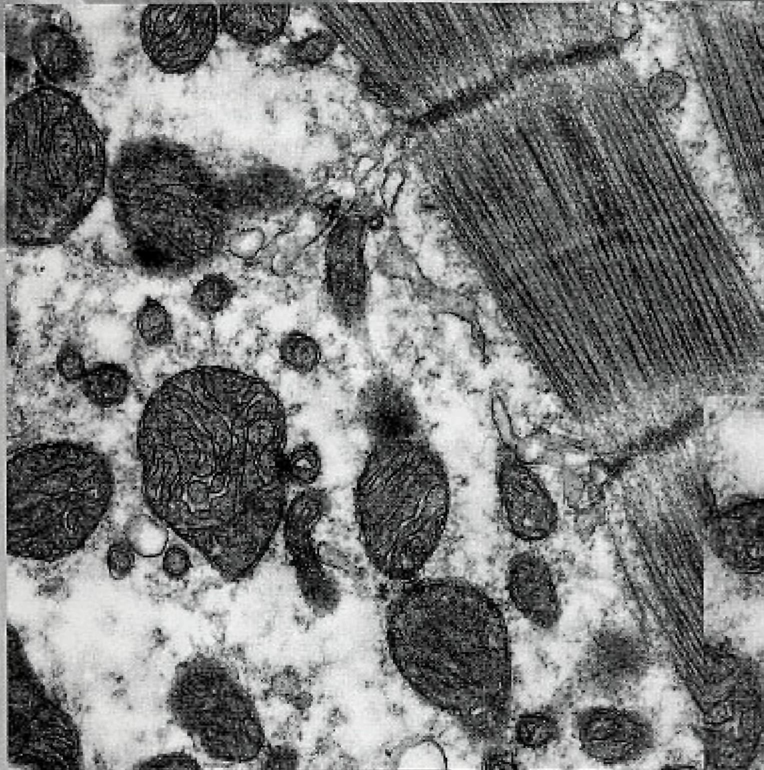

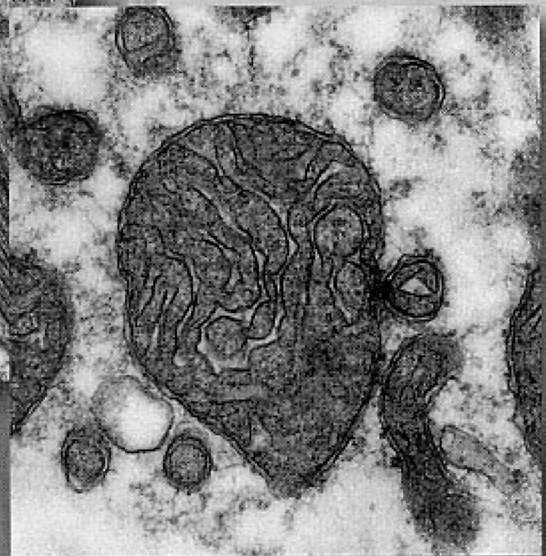

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Drawbacks:

- Difficult, time consuming, requires specialized equipment
- Poor resolution +/- 6 microns
- Dynamic range limits accurate measurement of beam intensity

Indirect measurement of the beam spread and intensity involves measuring some secondary effect produced by the electron beam. Since most SEMs have integral EDS x-ray systems, x-ray is the most likely candidate, although other signals such as Auger or EELS could be investigated. Several researchers have used x-ray intensity as an indirect method of measuring beam diameter and intensity. Research on beam scatter conducted at NIST and the Australian National University used one variation of this technique. In its simplest form, a polished sample is prepared usually consisting of a piece of metal with a relatively high atomic number embedded in a low Z matrix. The edge between the metal and the matrix is moved a precise distance away from the electron beam and an x-ray spectrum is collected. This process is repeated over multiple distances until a database of spectra is collected. By plotting the intensity of x-rays produced from the metal vs. distance away from the primary beam, a picture of the beam scatter and intensity can be built. A big advantage of this technique is that the data gathering can be done in most SEM labs with relatively simple sample preparation.

Correctly interpreting the data collected with this technique is more of a challenge. Since beam scatter takes place 360 degrees surrounding the electron beam, the ideal specimen for collecting x-ray data would be a sample consisting of a series of annular rings of different materials spaced very closely together. Unfortunately such a sample is difficult or impossible to make. Because the typical sample used for this type of data collection is a piece of metal that corresponds to a small fraction of a circle, the x-ray intensity data collected must be interpolated to represent an x-ray signal coming from 360 degrees surrounding the beam. Since each distance from the primary beam represents the radius of a different circle, the x-ray intensity collected at each distance must be treated differently.

The spatial resolution of the x-rays being used and the accuracy of the stage also limit the resolution of this technique. A typical value for spatial resolution of the x-ray signal in a high atomic number material is on the order of 1 or 2 microns due to electrons penetrating vertically and spreading laterally in the sample. Typical SEM stages will have a mechanical accuracy on the order of several microns making it statistically improbable to move the sample to a position with a precision of better than a few microns. Therefore it would be expected that the spatial resolution of this technique would be on the order of +/- 10 microns under typical conditions. Of course there are high precision stages available that can be positioned repeatedly with an accuracy of one micron or better, but they are very expensive and not available to most SEM labs. It also should be mentioned that some older and poorly maintained SEMs might have stage positioning errors of tens of microns. A major advantage of this technique is that x-ray intensity values can be accurately measured under the primary beam and essentially all the way out until the signal intensity drops off to background. Therefore the dynamic range problems associated with trying to measure beam intensity vs. brightness, as discussed above, are avoided.

Advantages:

- Relatively simple data collection
- Basic determination of beam scatter can be done in most labs
- Wide dynamic range makes it possible to get accurate intensity values for primary beam

Drawbacks:

- Impossible to make ideal sample
- Data interpretation is difficult
- Spatial resolution of x-rays is a limiting factor in overall resolution of technique
- Accuracy of stage is a limiting factor in resolution of technique

The techniques described here were performed by researchers with an interest in getting precise measurements of beam scatter under various conditions. It goes without saying (but I will say it anyway) that most analysts do not have the time or inclination to duplicate this research. Having said that, I would recommend the x-ray technique described above be used as a simple demonstration of beam scattering in most SEM labs. Any analyst trying to do environmental or low vacuum x-ray analysis in the SEM needs to be aware of the problem of beam scatter causing x-ray signals to be generated large distances away from the primary beam. Even those analysts who are aware of this problem may not realize the extent to which the beam can be scattered under certain operating conditions. It is an eye opening experience to place a sample several tens or hundreds of microns away from the beam and still pick up x-ray signals from that sample. For anyone skeptical as to the extent of this problem on collecting meaningful x-ray data, I suggest you try it in your lab. Not only will it show you just how severe this problem can be; it will teach you to take greater care when analyzing samples at elevated pressures.

Conclusion

The advantages and drawbacks of some experimental techniques for measuring beam scatter and intensity in the environmental and low vacuum SEM have been discussed. It is very difficult to accurately measure the amount of beam scatter that takes place in the environmental and low vacuum SEM, although it is relatively simple to see the beam scatter effect by collecting x-rays from a sample placed a known distance from the primary electron beam. This discussion only mentions a few techniques that have been tried. Other techniques to measure beam scatter are possible. Signals such as those collected by Auger and EELS could be used to measure beam scatter with higher spatial resolution and more accurate electron energies. Ideally there would be a detector that could be placed under the beam to provide direct measurements of beam scatter distance and intensity. Until such a detector is developed, the most practical method of determining the intensity and diameter of beam scatter is to use software to model beam scatter based on user-defined values for gas type, chamber pressure, working distance, and accelerating voltage. Software models, when correlated to high precision experimental techniques, provide a simple and inexpensive method of determining beam scatter for a wide range of conditions. ■

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