The Effect of Gas Type on Beam Scatter

X-ray Analysis in the Low Vacuum SEM (Part 2 of 3)

Don Chernoff, Small World, Inc.

Last month I addressed the phenomena of beam scatter in the environmental and low vacuum SEM. In that article I covered how beam scatter is affected by chamber pressure, working distance, and accelerating voltage. To briefly summarize, beam scatter becomes worse at higher chamber pressure, longer working distance, and lower accelerating voltages. As the beam scatters, electrons strike the sample some distance away from the primary beam and as a result, generate X-rays from unwanted areas of the sample. It is advantageous for the analyst to keep beam scatter to a minimum to reduce the generation of these X-ray signals. Under conditions of high chamber pressure, long working distance, and low accelerating voltage, it is possible for electrons from the beam to strike the sample on the order of a millimeter or more from the primary beam. This results in an "electron skirt" surrounding the primary beam. For this discussion we can loosely define high chamber pressure to be above about 1 Torr, long working distance to be more than 10 mm, and low accelerating voltage to be below 10 kV. It is important to keep in mind that it is the combination of these variables that leads to beam scatter and that a large reduction or increase in beam scatter is possible, depending on the values selected. I used the term "working distance" as a kind of shorthand to describe the distance the beam travels in the gaseous atmosphere. Once the beam leaves the final aperture until it strikes the sample, it is no longer at high vacuum and is subject to scattering by the gas molecules in the chamber. This distance can be several millimeters more than the working distance, defined as the distance from the pole piece to the sample. A more accurate term for the distance the beam travels in the gaseous atmosphere is "beam gas path length".

There is a fourth variable that affects beam scatter that we have not discussed yet, the gas type. In a typical SEM the working distance (or beam gas path length) for performing X-ray analysis is a fixed value determined by the manufacturer. Accelerating voltage is typically chosen to excite specific X-ray lines and may not be easily changed. Chamber pressure is typically the most easily varied parameter, but for certain samples the chamber pressure must be kept at a minimum value to reduce charging effects or to keep a sample in its natural state. The last variable that the user can adjust to affect the beam scatter is the choice of what gas to introduce into the chamber. Most low vacuum SEMs use air as the operating gas. In the true environmental SEM, air can be used but water vapor is commonly used to look at samples in their hydrated state. For the purposes of this discussion we will discuss gasses used to view non-hydrated samples.

Figure 1 shows a graph of beam scatter vs. chamber pressure for six common gasses. The gasses, listed in increasing order of scatter are helium, water vapor, air, nitrogen, argon, and carbon dioxide. The graph data are from models generated by the software program "Electron Flight Simulator', version E. The graph shows chamber pressures between vacuum and 1 Torr, since this is the typical working range of a variable pressure SEM. The graph shows the percent of the primary beam that is unscattered on the y-axis and chamber pressure on the x-axis. Ideally a gas would cause no scatter at any chamber pressure, and produce a horizontal line at the top of the graph. The amount of scatter is proportional to the atomic number of the gas. Helium exhibits the least amount of scatter so the diameter of the "electron skirt" will be the smallest. Carbon dioxide exhibits the most scatter. Nitrogen exhibits essentially the same amount of beam scatter as air, so for the purposes of discussing beam scatter they can be considered the same.

It is clear from the graph that helium is the gas of choice to reduce scatter, as it exhibits essentially no beam scatter up to 1 Torr chamber pressure. But there are other effects to be considered. A main reason for introducing gas into the SEM chamber is to eliminate the effects of surface charging. The primary mechanism of surface charge neutralization arises when gas molecules in the chamber are ionized by the beam and then combine with the static charge on the sample. Backscattered and accelerated secondary electrons also contribute to this gas ionization process. To eliminate sample charging at the lowest possible chamber pressure, it is desirable to use a gas that ionizes readily. The energy required to ionize helium is approximately double that for nitrogen, implying that the chamber pressure for helium needs to be higher than for nitrogen to get the same charge neutralization effect. This leads to the question of whether the reduction in beam scatter produced by helium is negated by having to use a higher chamber pressure.

Stowe and Robinson addressed the question of using helium by conducting experimental measurements of beam scatter in air and helium. They measured the generation of X-rays from a sample located a precise distance away from the primary beam. The X-ray intensity away from the primary beam was measured under a wide range of pressures for air and helium chamber environments. Air was shown to scatter the beam significantly for all pressures and accelerating voltages tested, while helium showed almost no beam scatter under most conditions. They also performed a test where the pressure of each gas was set to just eliminate surface charging effects. Measurements of beam scatter under these conditions showed that air exhibited more than twice the beam scatter of helium at a distance of 100 microns from the primary beam. The conclusion reached was that even though helium is more difficult to ionize than air, and therefore requires a higher chamber pressure to eliminate surface charging, at the higher pressure it still reduced beam scatter a significant amount vs. air. As far as X-ray analysis is concerned, the reduction in ionization efficiency of helium is more than made up for by the large reduction in scattering of the primary beam.

The choice of gas also has an effect on image quality. Image resolution is less sensitive to the electron skirt than x-ray resolution. Image resolution can remain high even with a large electron skirt, because most of the usable image signal comes from the primary beam and not the skirt. Of course the image quality will still depend to some degree on what gas is used and what type of detector collects the image signal. The relationship between gas and detector type is beyond the scope of this discussion, but needs to be considered when selecting a gas to use in a particular instrument.

At this point you might be considering using Hydrogen as the chamber gas. Hydrogen is lower in atomic number and would be expected to scatter the beam somewhat less than helium. However, due to the obvious safety considerations, hydrogen is not recommended. Most of the literature on low vacuum SEMs involves work performed with helium, air, nitrogen, or water vapor as the chamber gas. Other gasses and gas mixtures can be used to provide specific environments for different samples.

Conclusion

Practically any gas can be used in the environmental or low vacuum SEM. The main considerations for choosing a gas are reducing the surface charge on the sample, providing a specific environment for studying the sample, and minimizing scatter of the primary beam. Helium produces the least amount of beam scatter of all gasses tested.

Beam scatter results will affect image quality and X-ray spatial resolution, but not equally. Under most conditions, the image quality is not affected by the electron skirt produced when the beam is scattered by the chamber gas. However, the electron skirt can generate X-rays from regions well beyond the area of interest. Use of a low atomic number gas is one variable the analyst can use to minimize the size of the electron skirt and thereby improve the spatial resolution of X-ray analysis. Gas type should also be selected with respect to the other variables that contribute to beam scatter: chamber pressure, working distance, and accelerating voltage. ■

https://doi.org/10.1017/S1551929500068619 Published online by Cambridge University Press

References

Small World: Electron Flight Simulator, version E (1997)
http://www.small-world. net
Dchernoff@aol.com
(703) 849-1492

Joy, David: Environmental SEM Monte Carlo Simulation (1997)

Philips Electron Optics and Robert Johnson Associates: Environmental Scanning Electron Microscopy, An Introduction to ESEM (1996)

Stowe, Sally, and Robinson, Viv: The Use of Helium Gas to Reduce Beam Scattering in High Vapour Pressure Scanning Electron Microscopy Applications, Scanning Vol.20, January 1998

Wight, Scott: Beam Size in the Environmental SEM: A Comparison of Model and Experimental Data, Surface and Microanalysis Science Division, National Institute of Standards and Technology, Gaithersburg, MD 20899

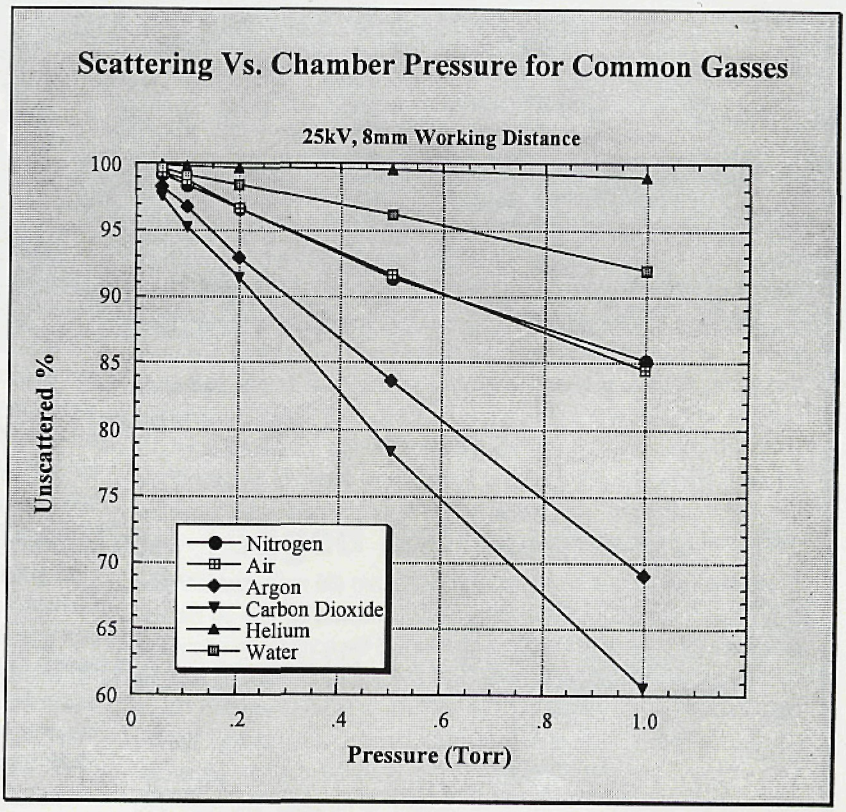

FIGURE 1. Beam Scatter vs. Chamber Pressure for 6 Different Gasses.

SAMx

Are you searching for an Automation system?
Would you like EDS/WDS Imaging?
Would you like to have EDS/WDS Analysis?

Then we are the company you're looking for.

We are the leading developer of EPMA/SEM automation, imaging, and combined EDS/WDS analysis under Windows95/NT. We are the pioneers in Thin Film analysis with STRATA released in 1991. We are fully Internet compatible, all software upgrades can be downloaded directly from our web site.

We listen to our customers needs and we deliver. Here are just a few:

- **XMAS Automation:** We can upgrade your EPMA / SEM equipped with WDS.
- **PCIMAX with IDFIX:** Get your SEM equipped with imaging and combined EDS.
- **TN2WIN and KV2WIN:** Convert your old TRACOR/KEVEX data files to PC.
- **STRATAGem:** Our 2nd generation of Thin Film STRATA is ready. Get your copy today.

For more detailed information on our products, we invite you to visit our web site at: **www.samx.com**

Please be sure to stop by our Booth #236 at Microscopy & Microanalysis '98 in Atlanta for your personalized demonstration. We look forward to seeing you there.

SAMx Support for Applications in Microanalysis and x-ray

4, rue Galilée / 78280 Guyancourt - France / Tel: 011 33 1 30 57 90 25 / Fax: 011 33 1 30 48 95 65 / email: jfthiot@samx.com
Bldg. M2048, Swarr Run Road, Lancaster PA 17601 / Tel.: (717)299-0599 / Fax: (717)299-2022 / email: jbarney@samx.com

Circle Reader Inquiry #10