X-Ray Analysis in a Vacuum?

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Performing X-ray analysis on a sample in an SEM used to be relatively straightforward. Straightforward and not always very exciting. Things have gotten a lot more interesting. In just the past few years, the amazing development of variable pressure SEMs has revolutionized the way we can look at samples. At the same time these new machines have changed the way we can image samples, they have also affected how we perform X-ray analysis on those samples.

I apologize in advance for the following gross simplification. For quite a lot of samples, the point and click method of X-ray analysis was and is sufficient to get meaningful data from a sample. Put the beam on a feature, collect a spectrum, and move onto the next sample. Of course, you always have to be aware of the interaction volume the beam creates in the sample. Depending on their energy, electrons in the beam will interact with atoms in the sample and scatter a predictable distance. Since it is these scattering events that create X-rays, the X-rays given off by the sample can be from an area beyond the area being analyzed. But if you are working with homogenous samples, or if your area of interest is several microns in size, you can get by pretty well with the point and click method.

The previous gross simplification is true for samples run in an SEM at relatively high vacuums of 10^{-5} torr or better. At these vacuums, the mean free path for electrons traveling through the vacuum can be measured in feet. Translation: The electrons in the beam will travel from the final lens to the sample in a nice straight line and be focused into a nice tight spot, with no interruptions.

The breakthrough of the variable pressure SEM is the ability to image samples at vacuums much lower than those in a conventional SEM by introducing gasses such as air, nitrogen, helium, water vapor, etc. into the sample chamber. Samples that cannot stand up to high vacuum because of their delicate composition, or because of their water content can now be viewed in their natural state. Samples that normally require conductive coatings can be viewed uncoated, while gas molecules in the chamber harmlessly carry the charge away. Chamber pressures up to several Torr are possible, an increase in pressure of more than 10,000 times over typical high vacuum.

As with most technology, the advent of the variable pressure SEM involves some "engineering tradeoffs". The benefits derived from these instruments take

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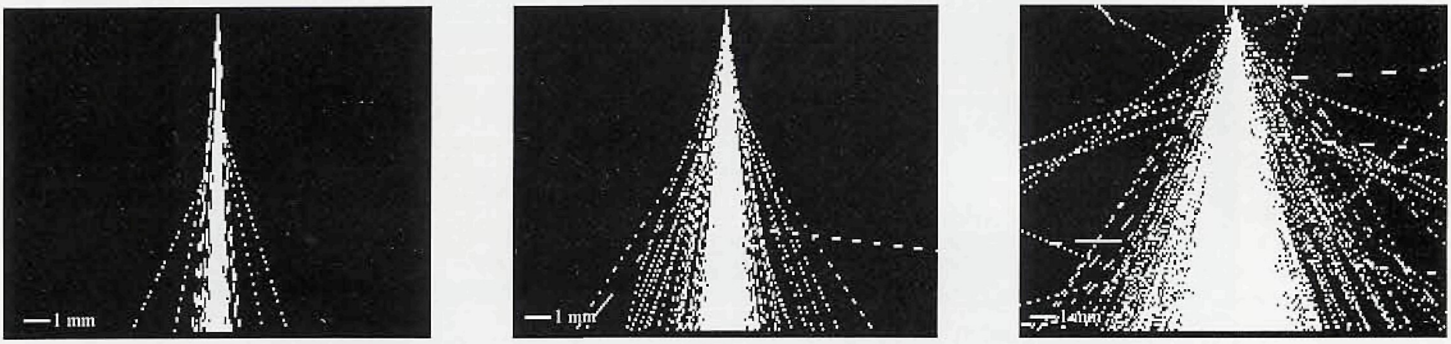
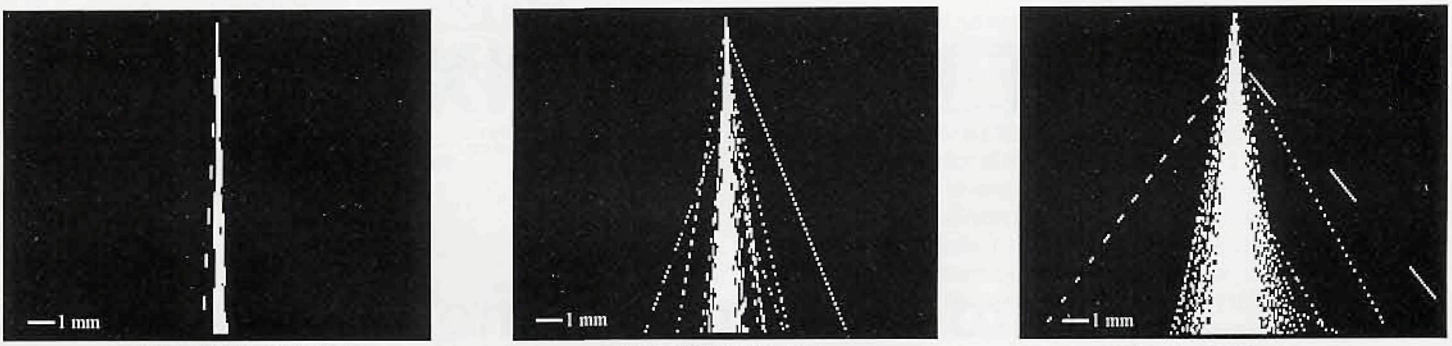


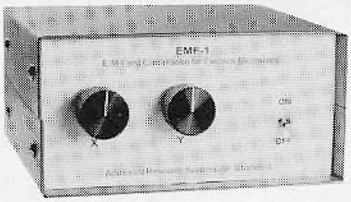
0.1 Torr Chamber Pressure | 1 Torr Chamber Pressure | 10 Torr Chamber Pressure

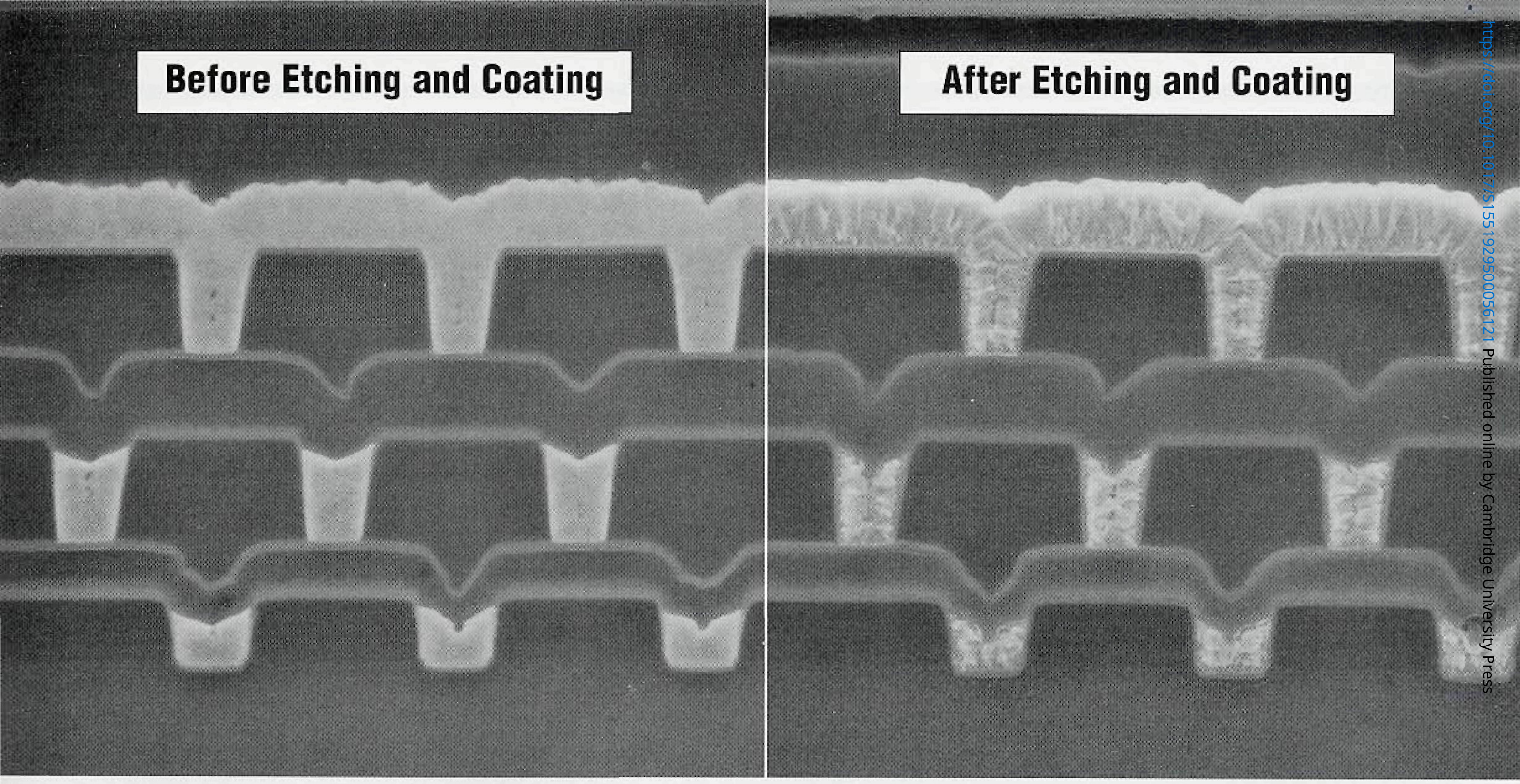
Figure 1. Electron beam spread in air



0.1 Torr Chamber Pressure | 1 Torr Chamber Pressure | 10 Torr Chamber Pressure

Figure 2. Electron beam spread in helium





Before Etching and Coating

After Etching and Coating

Before and after images showing the polished cross-sectional view of a typical semiconductor device. The sample was etched for 5 minutes at 6 kV and coated with Au/Pd. In the etched image, the detailed grain structure of the tungsten plugs are plainly visible.

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place at elevated chamber pressure. As the pressure goes up, the number of gas molecules in the chamber goes up as well. The probability of electrons in the beam hitting a gas molecule, and being scattered, goes up with the pressure. This effect is similar to the beam spread in a solid sample, although to a much lower degree. If the beam is scattered on its way to the sample, some electrons will hit the sample away from the area of interest. This effect is what makes X-ray analysis in a variable pressure SEM so interesting.

Think of the electron beam under high vacuum conditions as looking like a straight, thin line. Now, under low vacuum conditions, imagine the beam looking more like a cone, spreading out as it travels to the sample. The higher the chamber pressure, i.e., the more gas molecules in the chamber, the more scattering and the larger the base of the cone. As the electron beam spreads out, more of the x-ray signal will be generated away from the original area of interest. If the beam spread (base of the cone) is larger than the area being analyzed, the resulting X-ray spectrum will contain information from an unwanted region of the sample.

There are several ways to deal with this problem. The most obvious is to keep the chamber vacuum as low as possible, without damaging the sample or inducing charging effects. Another is to keep the working distance as short as possible. The shorter the working distance, the less chance the beam has to spread before it reaches the sample. The choice of gas plays a significant role in the amount of beam scatter. Helium is the gas of choice to keep beam scatter to a minimum. A helium atmosphere will scatter the electron beam significantly less than an atmosphere of heavier atoms such as air. If all else fails, another option, sample permitting, is to first do the imaging at low vacuum, then switch to high vacuum to perform X-ray analysis.

How do you know if the beam scatter is going to cause you a problem? The ability to determine the amount of beam scatter would prove very useful for selecting the best conditions to analyze a given sample. The same software technique of monte carlo modeling commonly used to view the size of the interaction volume in the sample can be employed to see how much the beam scatters in a variable pressure SEM. Software models can be performed that give a precise view of the beam scatter for a given set of variables. To produce a model, values for accelerating voltage, working distance, chamber pressure, and gas type need to be entered into the software. The user can then try different pressures, gasses, and working distances and see how much the beam scatters. This type of modeling lets you choose the best conditions to keep beam scatter within the limits of the feature you are trying to analyze.

The following examples were created with the software program "Electron Flight Simulator LV".

Figure 1 is a series of models showing beam spread for various chamber pressures with air as the source gas. Figure 2 shows the same models for a Helium atmosphere. Both figures use an accelerating voltage of 20 kV and a working distance of 15 mm.

Another effect of the elevated chamber pressure is the gas molecules do not just interfere with the incoming electron beam, but they can also interact with the X-rays leaving the sample on their way to the detector. The gas molecules will act to absorb some small portion of the emitted X-rays, thereby changing the measured intensities. This could lead to errors in quantitative analysis. The effect will obviously be more pronounced for low energy X-rays, as they are more readily absorbed by the gas molecules. Software correction methods will no doubt evolve to deal with this new wrinkle in quantitative analysis.

My conclusion: Enjoy the new found capabilities the variable pressure SEM brings to your sample analysis needs. Just don't be surprised when your X-ray spectrum gives you peaks you don't expect. Take time to consider the effects of the chamber atmosphere and pressure on the accuracy of your X-ray data, and set up your analysis accordingly. ■

Acknowledgments:
Dr. David Joy, University of Tennessee VPSEM monte carlo model.
Electron Flight Simulator 3.1LV, Small World (703) 849-1492, dchernoff@aol.com

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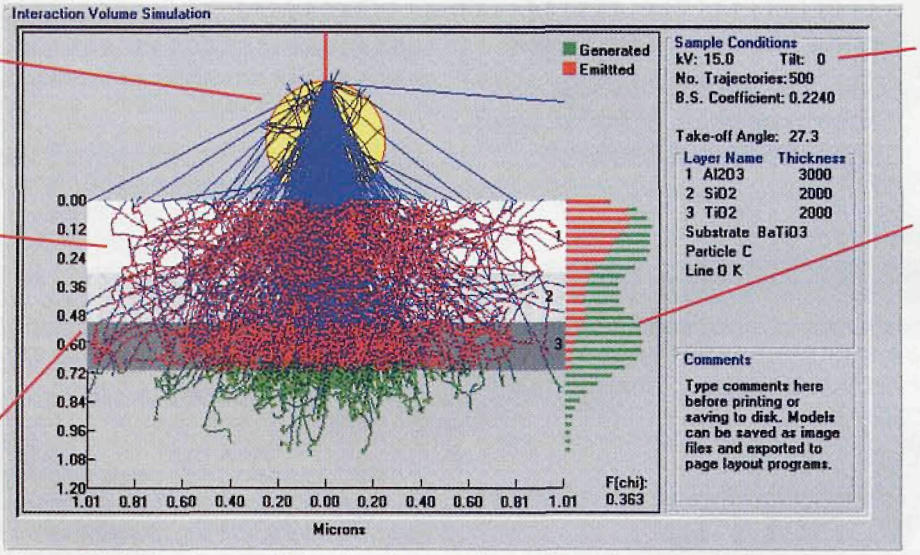
Display the PhiRhoZ curves to show generated (green) and emitted (red) x-ray intensity vs. depth.

Generate simulated spectra , based on your detector crystal, window, and geometry.



Sample Conditions
kV: 15.0 Tilt: 0
No. Trajectories: 500
B.S. Coefficient: 0.2240

Take-off Angle: 27.3

Layer Name	Thickness
1 Al2O3	3000
2 SiO2	2000
3 TiO2	2000

Substrate BaTiO3
Particle C
Line O K

Comments

Type comments here before printing or saving to disk. Models can be saved as image files and exported to page layout programs.

Electron Flight Simulator model showing electron and x-ray interaction volume in a complex sample

How Will it Help Me?

Electron Flight Simulator was designed with one goal in mind: to help you do a better analysis. Critical information about beam penetration, interaction volume size, and x-ray production is at your fingertips.

Knowing how the beam penetrates and spreads in a sample will give you increased confidence that your data is accurate. It helps explain your results to non-experts, and train new analysts. Save time, effort, and money by modeling a sample before ever putting it in the microscope. The software shows the best conditions to use.

How Do I Use It?

Vary the accelerating voltage and sample tilt until the interaction volume model is the correct size and shape for the feature you want to analyze. If you want to analyze thin films or small particles, the model enables you to determine the best accelerating voltage to avoid picking up unwanted x-ray signals from the substrate. Even measure film and particle thicknesses.

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