The Application of Monte Carlo Modeling to X-Ray Microanalysis

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X-ray microanalysis (EDS) on a SEM or TEM is a valuable tool for research and industry. No other technique can provide so much information so easily about the chemistry of micro-volumes of a material. Many samples require nothing more than positioning of the electron beam onto the area of interest, collecting a spectra, and 30 seconds later you have your answer. Or do you?

Any technique as widespread and straightforward as energy dispersive x-ray spectroscopy is bound to be misused on occasion. It has been my experience that many users either overestimate the capabilities of EDS to answer difficult materials questions or do not fully appreciate the limitations of the technique and how to work around them. These problems tend to show up as the need to analyze increasingly complex samples increases. The following brief description of the physics of electron beam/specimen interaction may be useful in explaining why problems can exist.

When energetic electrons from the electron beam impact a material, they will penetrate some distance into the material. They will also spread out laterally in the sample due to interactions with the atoms in the material. These interactions are called "scattering events", since the direction of the electron changes after each interaction. The amount of beam penetration and lateral spread is dependent on the energy of the incoming electrons (the accelerating voltage of the SEM or TEM), and the composition of the material. The scattering events are important because it is the scatter of an electron which generates a characteristic x-ray. The more scattering events, the more x-rays are generated. Without sufficient x-ray generation EDS analysis would not be possible.

The net result is that x-rays come from a volume of material much larger than the area where the electron beam first enters the sample. It is a common misconception among analysts that if they put an electron beam on a visible feature, they will be analyzing x-rays exclusively from that feature. Quite often nothing could be farther from the truth. The difference in size between the beam diameter and the resulting electron volume in the material is typically many orders of magnitude. Keep in mind that a typical beam diameter in a SEM is on the order of 100 A. The diameter of the resulting interaction volume can range from a few hundred Angstroms to several microns, depending on the accelerating voltage and the sample composition. Those analysts who think they can always get reliable chemistry data from sub-micron particles or phases may be in for a rude surprise.

Another common mistake arises when analyzing thin films. Many products today are made up of very precise layers of materials; semiconductors and hard disks being two good examples. Failure analysis of defects in these products can be made complicated by that pesky nature of the electron beam to penetrate the sample, as we have discussed. Thus an unsuspecting analyst collects x-ray data from what is thought to be the top layer of the sample when, in fact, they have just analyzed x-rays from multiple layers and are reporting erroneous results.

All hope, however, is not lost. While these spatial limitations are real, they are often tolerable if you can see how they affect your analysis. They can also be minimized by careful control of the analysis conditions. Monte Carlo software models can show you where the beam penetration and x-ray generation are taking place in your sample and help you determine optimum analysis conditions to get better analytical results.

Monte Carlo modeling describes a class of computerized techniques used to generate a picture of the beam specimen interaction for a given sample under specific conditions. Several Monte Carlo algorithms exist, but the most popular and most widely used are written by Dr. David Joy of the University of Tennessee. Monte Carlo modeling techniques were first used during the Manhattan project to model the interaction of electrons with solids. The technique has direct applications to the SEM and TEM user.

The beauty of a Monte Carlo simulation that it literally provides a picture of where the x-rays are coming from in a sample. This allows you to see how large an area you are really analyzing and allows you to see the effect of varying accelerating voltage and sample tilt to achieve more precise analytical results. As mentioned, the amount of beam penetration and scatter depends on the accelerating voltage and sample chemistry. Basically, higher density and higher atomic number materials can stop an electron beam better than low density, low Z materials. Thus for a given accelerating voltage, the amount of beam penetration will be greater in lighter material. Of typical concern to an analyst is how the size of the interaction volume changes in a given material if the accelerating voltage changes. By modeling a sample at various accelerating voltages, the optimum voltage can be determined before the sample is even put into the microscope!

The following simple example illustrates how knowing the interaction volume can help you choose better analysis conditions. The four models shown are for a 1 micron layer of aluminum on a silicon substrate. Figure 1 shows the interaction volume with a 15 kV beam. It is obvious that the beam easily penetrates the aluminum layer and generates considerable x-ray signal from the silicon substrate. Figure 2 shows that by dropping the accelerating voltage to 10 kV you eliminate most but not all of the beam penetration. A still lower kV would guarantee that the interaction volume stays within the aluminum but the lower kV may make it impossible to excite x-rays from other elements which may be present in the aluminum film. Without the option to go to a lower kV, Figure 3 shows that if the sample is tilted 60 degrees, essentially all the interaction volume stays within the aluminum film. This is because tilting the sample effectively thickes the layer directly beneath the beam. Thus the accelerating voltage stays at 10 kV and a more accurate analysis of the aluminum film is possible. The interaction volume shape also changes and approaches the surface as the sample is tilted. This phenomenon can be exploited at high tilt angles to generate a greater proportion of the x-ray signal near the surface and so make the analysis much more surface sensitive. This is shown in Figure 4 where the sample has been tilted 80 degrees.

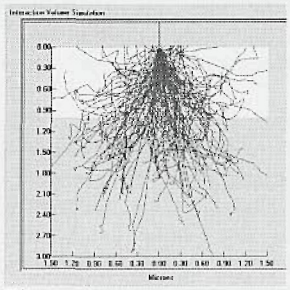

Figure 1

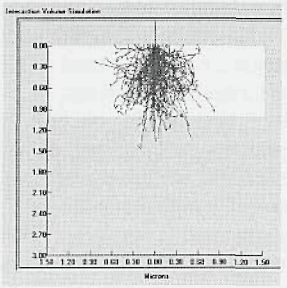

Figure 2

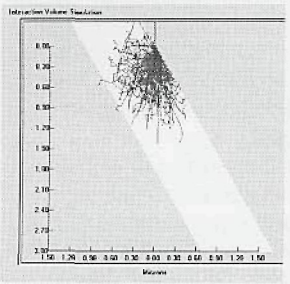

Figure 3

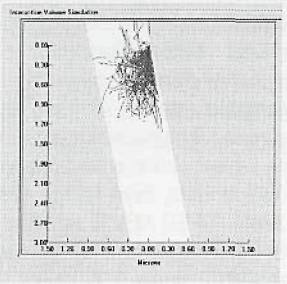

Figure 4

The above models were generated by a Monte Carlo software program developed by my company. The program, entitled "Electron Flight Simulator", is available for IBM compatible PCs (running Widows 3.1) and the Apple Macintosh. The software allows the user to select an accelerating voltage from 1 to 400 kV and sample tilt up to 89 degrees. Any sample chemistry can be modeled simply by typing in the chemical formula. For further information or to request a demo disk, you can call or fax Small World at (415)345-8013. ■

Summary of American Type Culture Continuing Education, Workshops and Conferences for the Balance of 1994:

September 14/16: Growth & Preservation of Animal Viruses.
September 21-23: Microscopy/Photomicrography.
September 26/28: Conventional & Molecular Cytogenetic Techniques.
October 3/6: Insect Cell Culture & Recombinant Protein Expression with the Baculovirus Expression Vector System.
October 19/21: Freezing & Freeze-Drying of Microorganisms.
October 31/November 4: Recombinant DNA: Techniques & Applications.
November 8/11: Polymerase Chain Reaction (PCR) Applications/Cycle DNA Sequencing.
November 16/18: VCell Culture & Hybridomas: Quality Control & Cryopreservation Techniques.

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