More Than One Ever Wanted To Know About X-ray Detectors

Part V: Wavelength - The "Other" Spectroscopy

Mark W. Lund, Ph.D., MOXTEK, Inc.

The use of x-ray spectrometry in electron microscopy has been a powerful market driver not only for electron microscopes but also for x-ray spectrometers. More x-ray spectrometers are sold with electron microscopes than in any other configuration. A general name for the combination is AEM, or analytical electron microscope, though in modern times AEM can include other instrumentation such as electron energy loss spectroscopy and visible light spectroscopy. In previous articles I have discussed energy dispersive spectrometers (EDS). These use semiconductor crystals to detect the x-rays and measure the energy deposited in the crystal. A second type of x-ray spectrometer measures the wavelength of the x-rays, and so is called "wavelength dispersive spectrometry" (WDS).

Wavelength spectrometers use crystals to diffract x-rays similar to the diffraction of visible light by gratings. The regularly spaced array of atoms (or molecules) in the crystal diffract x-rays. Unlike diffraction gratings, however, the crystal reflection only reflects one wavelength for each angle of incidence. This is due to the difference between the two-dimensional diffraction of a grating and the three dimensional diffraction of a crystal lattice. Diffraction from a three dimensional structure is called Bragg diffraction. In Bragg diffraction the angle of reflection is equal to the angle of incidence just as if the crystal were a mirror. Only one wavelength and its shorter wavelength harmonics can be reflected for a given lattice spacing and angle of incidence. This means that most crystal spectrometers must either be scanned or remain at one fixed wavelength. The exception to this statement is a small class of spectrometers that use a geometry that allows the x-rays to simultaneously intercept the crystal at a range of angles, a different angle for each segment of the crystal. These have very small collection solid angles and require a position sensitive detector to record the spectrum.

A crystal can only diffract x-rays that are shorter in wavelength than twice the distance between the atoms (or molecules). This means that there is not one crystal that will work for the whole x-ray range. Short wavelength x-rays can use crystals of typically 1 to 5 angstrom spacings. X-rays from the lighter elements need crystals with wider spacings, which are typically organic crystals with large molecules. These crystals exist at spacings shorter than about 13 angstroms and cannot be used for elements lighter than fluorine.

For the lightest elements artificial crystals are used, either Langmuir-Blodget films or, more recently, sputtered multilayers. Langmuir-Blodget films are made from soaps that contain heavy metals. The soap molecules can be floated on water with their hydrophilic ends in the water and their hydrophobic ends up, giving a uniform layer one molecular layer thick. When a substrate is repeatedly dipped into the water, the soap layer is picked up onto the surface one layer at a time, making an artificial crystal with the molecules aligned within each layer. The layer stack can then be used to diffract x-rays. Sputtered multilayers are made by physical vapor deposition, with alternating layers of heavy and light elements. These multilayers can be made at custom spacings from about 15 angstroms to 200 angstroms or more. They have higher reflectivities than the soap films and are more stable.

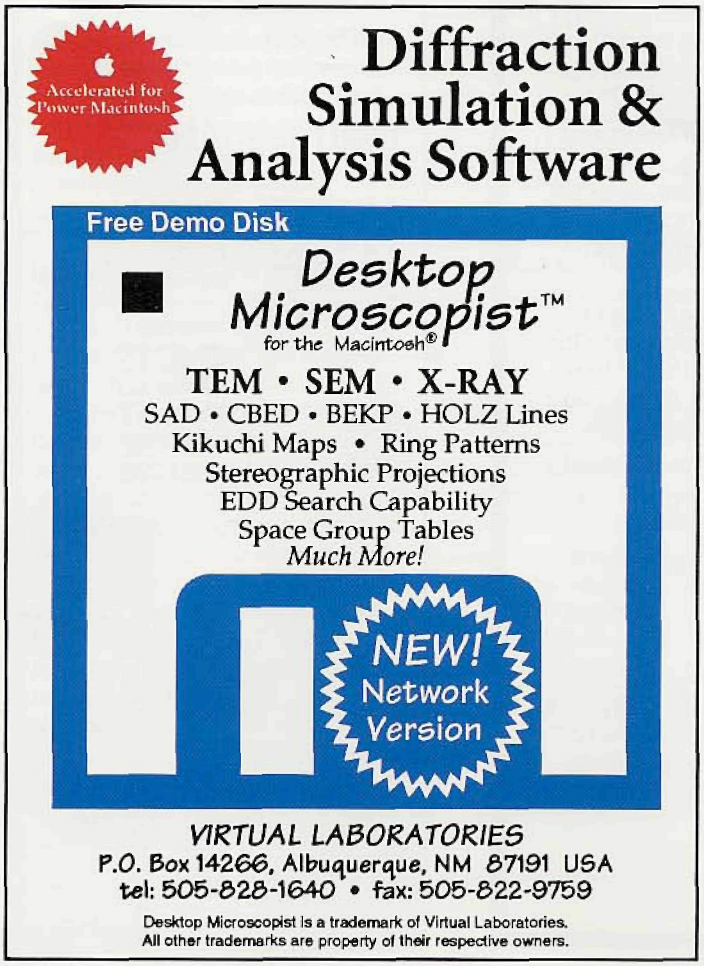
The first x-ray spectrometer-electron microscope combinations were special purpose WDS instruments. The microscope was specifically modified to have a spectrometer bigger than the column on which it was built. The combinations were useful as prototypes of analytical microscopes and were used to develop AEM techniques, but it was not until EDS was developed that analytical electron microscopes became popular.

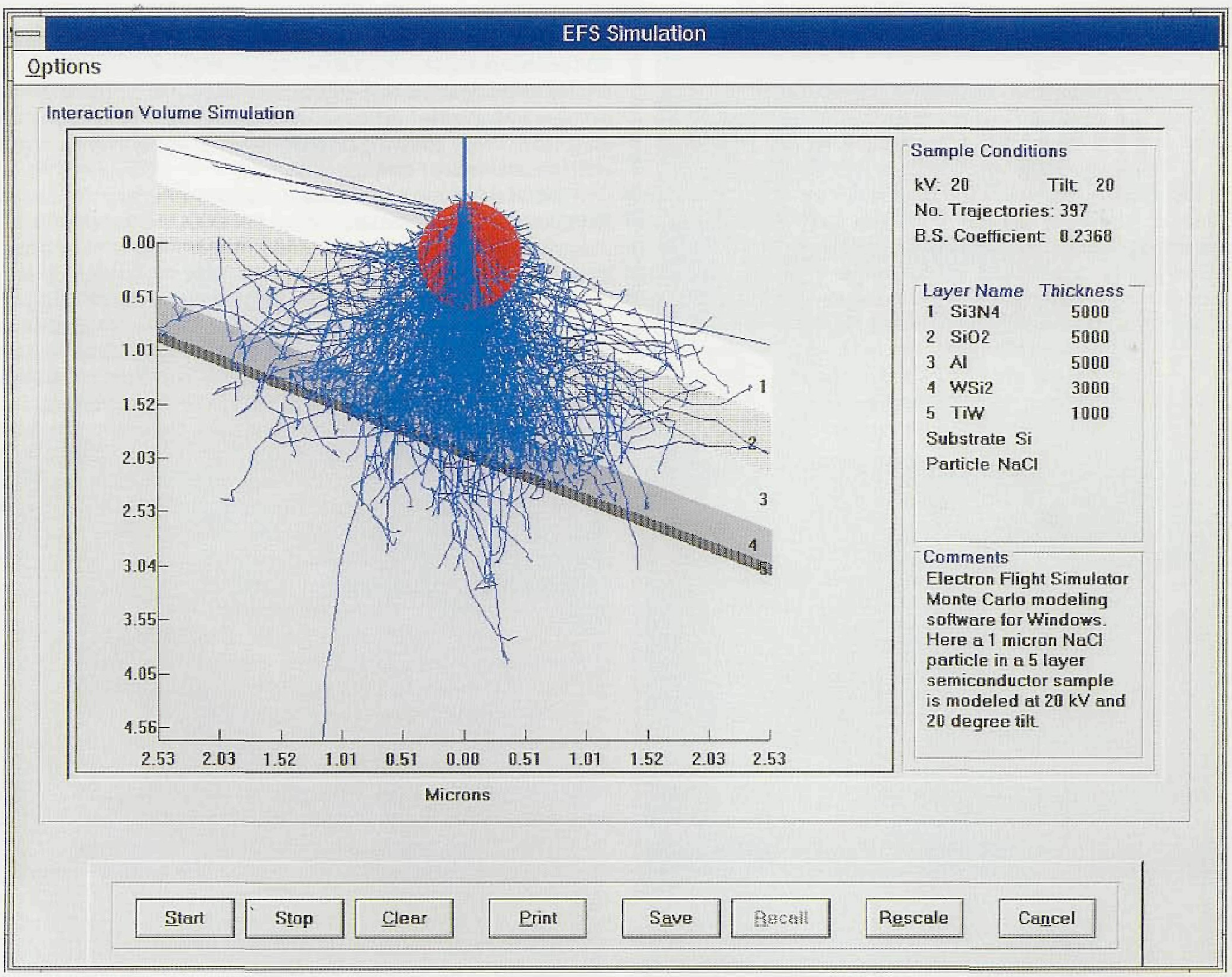
Today there are many applications of WDS, and several companies make WDS spectrometers for use with both TEMs and SEMs. The advantages of WDS are higher resolution and lower background. Typical resolution of a WDS spectrometer is 20 eV (at Mn $K\alpha$), whereas the best EDS spectrometers have 130 eV. At Na $K\alpha$ the WDS can give as low as 3 eV FWHM versus 80 eV for EDS. These advantages give WDS the edge in measurements where minimum detectable limits are important. They also make possible measurements such as potassium in a high background of calcium, which is important in some areas of medicine and biology. The disadvantage of WDS is low throughput. WDS spectrometers collect a relatively small solid angle and must be slowly scanned to obtain a spectrum. A recent commercial development is the integration of both WDS and EDS in one unit with the hardware and software to collect, analyze and display their spectra simultaneously.

WDS spectrometers use an angle scan to generate a spectrum. The crystal moves and rotates and the detector moves along at twice the speed to keep up with the mirror reflection, giving what is called a theta-two-theta scan. This takes a lot of space and precision mechanics. Since collimating optics are not available in the x-ray region, the crystals are efficient only over a small solid angle and collection efficiency is low. Detectors are typically flow proportional counters, which have a high count rate capability and sensitivity to even the softest x-rays.

X-ray spectrometry is a good example for pondering nature's constraints. EDS does not have to be scanned, and so can detect an entire spectrum at once. On the other hand, an EDS spectrometer can only detect one x-ray photon at a time and does not care whether the x-ray is in your range of interest or not. This could cause your maximum count rate to be exceeded by spectra outside your range of interest. WDS spectrometers can withstand very high background or signal count rates, but can measure only one wavelength at a time. Simultaneous WDS spectrometers always give up either collection area, or spectral range in order to give a simultaneous spectrum. These constraints are not easily resolved, and so the best spectrometer depends on the exact application. This leaves room for both EDS and WDS in modern analytical electron microscopes. ■

> *It is a capital mistake to theorize before one has data. Insensibly one begins to twist facts to suit theories, instead of theories to suit facts.* *Sir Arthur Conan Doyle*





Sample Conditions

kV: 20 Tilt: 20
No. Trajectories: 397
B.S. Coefficient 0.2368

Layer Name	Thickness
1 Si3N4	5000
2 SiO2	5000
3 Al	5000
4 WSi2	3000
5 TiW	1000
Substrate Si	
Particle NaCl	

Comments

Electron Flight Simulator Monte Carlo modeling software for Windows. Here a 1 micron NaCl particle in a 5 layer semiconductor sample is modeled at 20 kV and 20 degree tilt.

NEW! Electron Flight Simulator Version 2.0 for the SEM and TEM

The above image was created with the new version 2.0 Electron Flight Simulator, Monte Carlo modeling software for Windows. This model shows a 1 micron sodium chloride particle partially embedded in a five layer semiconductor film structure on a silicon substrate.

The software is used to visualize beam penetration and spread in a sample so you can see where the x-ray signal is being generated. It allows you to test different microscope conditions like accelerating voltage and sample tilt, before running the sample, to predetermine the best way to perform x-ray analysis. Practically any sample you can think of can be modeled. Electron Flight Simulator takes away the guesswork and lets you see where your x-ray data comes from in your sample. Any SEM/TEM lab doing x-ray analysis can use it. It is perfect for training students and technicians, and to explain your x-ray results to other engineers.

Version 2.0 adds many exciting features including: Modeling up to 5 different layers, either stand-alone or on a substrate. Modeling particles and inclusions of any size and chemistry, use your mouse to dray and drop the particle anywhere in the sample. Modeling films at 90 degree tilt to simulate cross sections or grain boundaries, drag and drop the beam into any starting position. Display x-ray generation volume to see where the x-rays are generated for any x-ray line you specify. Overlay the x-ray generation volume onto the electron interaction volume model for a more precise look inside your sample. Display the PhiRhoZ depth of x-ray generation plot to identify depth of maximum x-ray generation - and more.

Electron Flight Simulator is available from Small World, P.O. Box 25284, San Mateo, CA 94402. Phone/Fax: (415)345-8013. EMail: DCHERNOFF@AOL.COM. Demo disks are available upon request.

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