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

The First in a Series

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Spiderman carefully slid the sample of mud into the microscope. As he increased the magnification he caught sight of a tiny spherical crystal. *Zooming in on it, he said "Let's see what it's made of". He reached over and* flipped on the x-ray detector. As the spectrum formed on the screen a gasp went through the small group of researchers: "Kryptonite. . .the intruder is from another comic strip!"

The primary function of a microscope is to get a closer look at a sample. Many times a closer look is enough. Other times you need as much information as possible to solve a problem. In the above example, a reading of the chemical elements contained in the sample gave important clues about the sample's origin. X-ray analysis in electron microscopy combines elemental analysis with high resolution imaging. Of all the analytical techniques available to microscopists, it is the most highly developed and easiest to use.

An electron microscope uses an electron beam to form an image of an object. As the image is formed, the electron beam interacts strongly with the sample, creating (along with heat, light, and sound) x-rays. The electron beam gives an image of the sample. The x-rays contain information about the composition of the sample. Adding an x-ray spectrometer to an electron microscope can form a very powerful instrument.

When the electron beam strikes the sample it can generate x-rays in several ways. As the electrons strike the sample many are slowed, stopped, or deflected. The resulting change of momentum produces a broadband x-ray spectrum called continuous radiation or bremsstrahlung. As the incoming electrons disturb the inner shell electrons in the sample, the atoms produce characteristic radiation in the form of x-ray spectral lines, just as the outer shell electrons produce light spectra and as vibrating molecules produce infrared spectra. Most of the x-rays produced in an electron microscope come from these two mechanisms.

The x-ray spectrum can be measured in two different ways. The most common directly measures the energy of each x-ray and is termed energy dispersive spectrometry (EDS). The other method measures the wavelength of the x-rays and is termed wavelength dispersive spectrometry (WDS). In the future I plan to discuss WDS in detail, but for now I will stick to EDS, since it is the most common.

Energy dispersive spectroscopy is like measuring the speed of a baseball by putting a thermometer in the catcher's mitt. An energy dispersive spectrometer measures the energy of each x-ray by letting the x-ray dissipate all its energy in a semiconductor crystal. Most of the energy is converted to phonons, but a predictable fraction of the energy goes to create free charges in the form of electron hole pairs. As these charges move through an electric field applied to the crystal a transient current flows, stopping when the charges either reach the electrodes or are trapped by crystal defects. The integrated current (or charge) from this event is proportional to the energy of the x-ray.

Making this measurement is literally a heroic effort. A magnesium Kα x-ray will produce about 300 electron hole pairs. A boron Kα X-ray will produce only 50 electron hole pairs. Current state of the art is to have a mean error in this measurement of 3 electrons. Literally hundreds of thousands of hours have been spent developing more efficient crystals, lower noise electronics, and optimized pulse processing to reach this level. If you have a Si(Li) detector system, you are in the presence of one of the lowest noise preamplifiers ever built.

Any semiconductor could be used as the detection crystal. The practical ones (in 1994) are silicon, germanium, mercuric iodide, and cadmium telluride. Of these only silicon and germanium are used in electron microscope spectrometers. Because of their relatively small band gaps, silicon and germanium must be cooled to liquid nitrogen temperatures to eliminate thermally generated dark current.

Silicon EDS detectors are used most of the time. It has so far proven impossible to purify silicon to the point that it has high resistivity at 77°K. This is primarily due to the presence of low levels of boron, which produces holes that carry leakage current. If you could match up a small atom willing to donate an electron with each boron atom you could "compensate" the crystal to have no net carriers at low temperatures.

This is what is done in a lithium drifted silicon (or Si(Li)) detector. Lithium is the only monovalent atom small enough to diffuse into silicon at low temperatures. If the diffusion is done in an electric field the lithium atoms (which are ionized at the drifting temperature) will drift into the crystal to form an opposing field. When the field in the crystal is canceled by the drifted lithium ions, the lithium stops drifting and the crystal is exactly compensated. After the drifting is completed the crystal must be kept cold to prevent the lithium from drifting out of it.

Germanium can be purified to intrinsic levels and does not need to be lithium drifted. Since germanium has a smaller band gap than silicon, an x-ray will generate more charges in a germanium crystal than a silicon crystal. This gives the germanium slightly better resolution. There are many technical problems with the production of germanium x-ray detectors. They have only recently been introduced into the marketplace.

EDS can detect all elements heavier than lithium. As the elements increase in atomic number the energies of their x-rays increase. The very light elements (beryllium through fluoride) require special detector ultrathin windows for detection. Elements heavier than sodium can be detected with standard beryllium window detectors.

P.S.: Kryptonite can be detected only under very special conditions. ■

This article is intended as an overview of the field of x-ray spectroscopy and is the first of a series. Future articles will go deeper into this powerful technique. Coments, suggestions, etc. on the series are invited: Mark Lund, MOXTEK, INC. Tel.: (801)225-0930, Fax: (801)221-1121.

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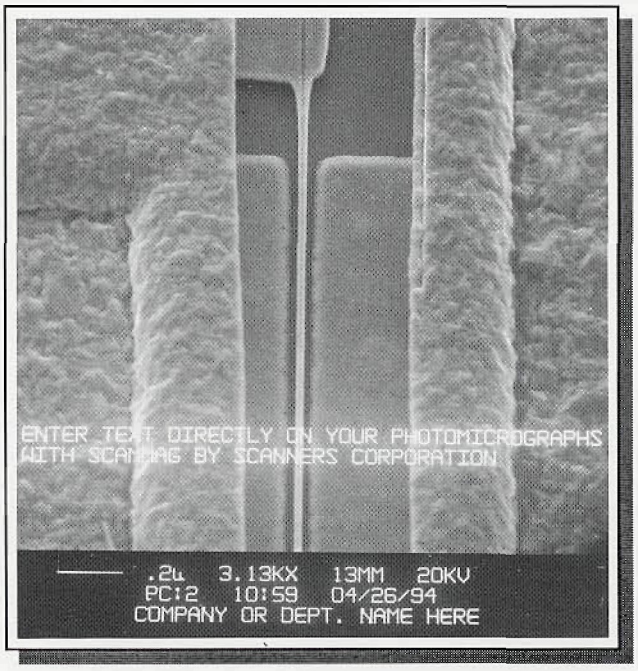

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