

More Than One Ever Wanted To Know About X-Ray Detectors Part 3: Who Put Promethium In My Soup?

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The combination of electron microscope and x-ray spectrometer is a very powerful tool. Not only can one see a sample in great detail, but one can determine, and even map, the chemical elements. In Part 1, I discussed some of the basics of energy dispersive x-ray spectroscopy (EDS, EDX). In Part 2, I went into detail about the construction of the detector, and discussed counting statistics. In this installment I will discuss the interaction between the x-ray and the detecting crystal, things that can go wrong in the process, and the phenomena which can make false peaks in a spectrum.

An energy dispersive detector measures the energy of an x-ray by absorbing it, then measuring the energy absorbed. In Si(Li) detectors the x-ray is absorbed in a silicon crystal. The x-ray's energy is split several ways. Most of the energy goes to produce phonons. Phonons are just crystal lattice vibrations, which can be thought of as sound or as heat. The remainder of the energy goes to produce free charge-electrons which are removed from their home atoms with enough energy to move freely through the lattice. These leave vacancies at home, which also can be seen to move freely through the crystal, hopping from one atom to the next. These are what is termed as "holes," and act just like positively charged electrons. Under the influence of an external electric field these electrons and holes will move in opposite directions, causing current to flow in the detector electrodes until they hit the electrode, where they are absorbed back into the lattice. The amount of charge that flows in the external circuit during this time is the signal, and is proportional to the energy of the absorbed x-ray.

One thing that can go wrong with this process is that the absorbed x-ray might excite a silicon atom in the detector to emit a silicon x-ray. If this x-ray is then re-absorbed in the crystal there is no problem, but if the new silicon x-ray escapes from the crystal without being absorbed it leaves an energy deficit equal to the energy of the silicon k line. These events will cause a separate peak to form 1.74 keV below the real peak. This is called an "escape peak." Only x-rays that have higher energy than 1.84 keV (the silicon absorption edge) can produce escape peaks, and because escapes are rare they are usually only seen when a strong peak is present. Their height is typically less than one percent of their parent peak.

Another thing that can go wrong is overlap. EDX detectors can only

detect one x-ray at a time. This includes not only signal x-rays, but background x-rays and stray electrons from the microscope beam. If two x-rays come in at exactly the same time they will look like a single x-ray to the detector. The energy the detector will report to the computer will be the sum of the two energies. This causes artifacts known as "sum peaks."

If two x-rays come in together, but not exactly at the same time, the second x-ray generates a new pulse of charge before the previous one has been fully collected. This give the same effect as the scale at the doctor's office—when you step on it the nurse always puts her foot on it (which is why you always weigh more at the doctor's). If uncorrected this will give a high energy tail to the peak. This type of coincidence can be detected by the electronics, which will reject both x-rays. The circuit that does this is called the "pile-up rejector."

Another artifact that can be seen with Si(Li) detectors is a small silicon peak. This probably comes from fluorescence of silicon in parts of the detector that are outside the active region—the flange or the thin dead layer at the surface.

Interferences are lines from two elements that the spectrometer cannot resolve. This can take two forms. An example of the first is calcium versus potassium. These are neighboring elements which the spectrometer can easily resolve if they are in similar concentrations. In biological analysis, however, there are often high concentrations of potassium and low amounts of calcium. The huge potassium peak has a tail that overlaps the calcium, making it difficult to detect.

The second form of interference is due to L or M lines of heavy elements interfering with K lines of light elements. An example of this is the interference of the vanadium $L\alpha$ and $L\beta$ and the oxygen $K\alpha$.

Artifact lines from these phenomena show up in the spectrum, and so a search for possible elements will often show unexpected elements. Usually these matches come from the heavy elements. Since they each have many lines there is a good chance one will match your artifact. It can be quite a surprise to find promethium or francium in your sample. If it is a surprise, check for artifacts. A good way to do this is to look for other lines for the same element. For example, if the promethium $L\alpha$ line at 5.432 is found, look for the $L\beta$ line at 5.960 keV, or the M lines. Usually you will find that the surprises are artifacts, and that your sample is actually quite boring! ■


First get your facts; then you can distort them at your leisure.
...Mark Twain


Front Page Image

SEM Image of a Negative Electron Affinity (NEA) Diamond Surface

Scanning electron micrograph (SEM) of a natural type IIa (100) diamond surface. The crystal had been especially treated with atomic hydrogen to create a negative electron affinity (NEA) diamond surface. In the NEA condition, the conduction band lies below the vacuum energy level and the surface energy barrier for electron escape is removed. As a result, the secondary electron yield is greatly enhanced and the image contrast originates from further beneath the surface than is normally observed in a secondary electron image. The contrast is due to a dense array of dislocations clustered in orthogonal rows in $\langle 011 \rangle$ directions that likely act as free electron traps. Consequently, secondary electron emission is reduced in the near vicinity of the dislocations producing the dark spots that compose the image. The micrograph is 0.75 mm square.

This micrograph was supplied by David P. Malta of Research Triangle Institute, 3040 Cornwallis Rd., Research Triangle Park, NC 27709. Co-workers on Diamond NEA technology development at PTI are John B. Posthill, Trevor P. Humphreys, Raymond E. Thomas, Michael J. Mamtini, G. Gill Fountain, Ron A. Rudder, George C. Hudson and Robert J. Markunas. The project is funded by the Ballistic Missile Defense Organization through the Office of Naval Research.

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Don Grimes, Editor

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