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More Than One Ever Wanted To Know About X-ray Detectors Part IV: Windows for Elements Heavy and Light

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I attend a local entrepreneur's luncheon once a month. Since the small town I live is the home of Word Perfect Corporation, and Novell, Inc. is just down the road, you can imagine that mamy members are doing exciting things with software. When I tell them that MOXTEK makes windows for X-ray detectors they really light up, until I tell them that I mean *real* windows.

X-ray detectors are used in electron microscopy to add chemical element analysis to the imaging capability of the microscope. A typical energy dispersive spectrometer uses a silicon crystal about the size of a shirt button to detect and measure the energy of incoming X-rays. This crystal is cooled to 77 degrees Kelvin, or to the temperature of liquid nitrogen. This cooling lowers the noise and stabilizes the detector. It also makes the detector vulnerable to vapors condensing on its surface. X-ray windows solve this problem by isolating the detector from the environment.

Anything that you put between your sample and the detector is likely to disturb, degrade, and generally make a mess of your precious signal. X-ray windows are no exception. The various window options do this in different ways.

There are several options for X-ray windows used in electron microscopes. These are beryllium, boron nitride, boron hydride, diamond, and polymer. The oldest of these is beryllium. Beryllium is most useful for detecting elements heavier than magnesium. It is possible to use the thinnest beryllium foils (5 µm) for sodium and fluorine. For the light elements (boron, carbon, nitrogen, oxygen, fluorine and sodium) special windows have been developed. These go by the generic name of ultrathin windows. Of the several technologies used to make ultrathin windows, they all have a few things in common. The X-ray transparent membranes are very thin, about 50 $\mu g/cm^2$. This is only a couple of thousand atoms thick. The membrane materials are all remarkably strong and they are made from materials containing only light elements. The membranes are supported on grids to provide strength over large areas.

Boron nitride windows were the first light element windows. They are made with a chemical vapor deposition (CVD) process with the grid a monolithic part of the structure. Diamond windows (which are no longer commercially available) and boron windows (which are available) are also made with CVD, but on a silicon grid. Polymer windows are made of ultrathin superpolymer membranes stretched across a silicon, boron nitride, or tungsten grid. All of these windows are usually coated with a 200 to 800 Å thick layer of aluminum to cut down light transmitted through them and to make them electrically conductive. In addition, a coating of aluminum or aluminum plus aluminum nitride is necessary to stop gas diffusion through the polymer windows.

The transmission of ultrathin windows depends greatly on the window material and the X-ray energy. Windows containing boron, for example, will transmit boron X-rays very well, since the $K\alpha$ emission energy is slightly lower than the boron absorption edge. The boron will strongly absorb carbon X-rays, however. Likewise, diamond windows are very good for carbon and boron X-rays, but poor for nitrogen. Multielement windows such as boron nitride, boron hydride, and polymers have better transmission at absorption edges than pure element windows.

Reliability of ultrathin windows is surprisingly good, considering how thin they are - the transmissive area is about 50 $\mu g/cm^2$. Ultrathin means ultrafragile. You cannot touch an ultrathin window with your finger, bump it with a stage, or clean it with a cotton swab. However, ultrathin windows can be quite reliable. Reliability data is proprietary, so I can only report on MOXTEK results. One spectrometer manufacturer who uses MOXTEK windows has over 1000 ultrathin window systems in the field and an accumulated mean time before failure of 484 weeks as of April 1995 and going up. That is over nine years. When fitting a new thin window system on an old microscope, it would be good to discuss window reliability with both the microscope and EDS manufacturer.

In certain applications, however, reliability can be poor. The biggest problem is particle impact on the window during microscope venting. This causes "bullet holes" in ultrathin windows and depends on the gas dynamics inside the microscope. Many electron microscopes do not have this problem, but some models have a high propensity for window damage. Environmental SEMs, which can expose the window to reactive gases and hot water vapor, are particularly hard on ultrathin windows. The aluminum on polymer windows tend to become etched, which allows water permeation. To solve this problem, MOXTEK has developed a boron membrane window that is immune to attack from corrosive gasses.

Cleaning X-ray windows is a tricky proposition. Even beryllium windows are used near the limit of their strength in order to give good X-ray transmission. If it is clear that the window is contaminated (vacuum oil dripping off the mount is a good sign of this) call the manufacturer for advice on how to clean it. If you do not mind voiding the warranty, an effective, and not-too-dangerous, method of cleaning is to gently run a stream of alcohol across the surface. Do not squirt the window membrane directly. Contact the EDS manufacturer for more details! ■

Front Page Image
Mouse Fibroblasts - Unenhanced & Enhanced

Mouse fibroblasts were labeled with two fluorescent reagents: FITC and acridine orange. The FITC binds to the actin protein in the cell cytoplasm and produces a green fluorescence; the acridine orange reagent binds to DNA and produces a yellow-orange fluorescence. The stained cells were viewed using a Zeiss Axioskop microscope equipped for fluorescence and high-resolution video using a ZVS 47DE color-CCD camera which features real-time, built-in digital edge sharpening. Digital edge sharpening expands the fine details of soft-focus or low contrast images. The enhanced images more closely resemble the subjects as viewed in the microscope oculars.
(Photos courtesy of Carl Zeiss, Inc. Microscope Division, Thornwood, NY 10594)

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