MICROSCOPY 101

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Repairing Chipped Bell Jars

Chipped bell jars may be "patched" using a stiff epoxy *(e.g.,* Torr Seal). Clean all grease and oil off the bell jar (try using Tilex Soap Scum Remover), then fill the chipped hole with the epoxy, and set the bell jar on a flat, smooth surface covered with waxed paper (so that the surface comes out flat and smooth) while the epoxy cures.

Torr Seal is an epoxy compound especially formulated for use in vacuum systems that was introduced by Varian Associates a number of years ago. It is stated to be good at pressures below 10^{-9} Torr, and is bakeable at temperatures up to 120° C. It adheres to most clean materials (glass, metals, ceramics) and holds up well over long term service. Some companies that handle EM supplies also handle it.

Other similar products are also sold by other companies. For example, Duniway Stockroom Corp. sells a product called "Epoxy Patch" that is stated to be equivalent to Torr seal.

Wilbur C. Bigelow, University of Michigan

As a manufacturer of vacuum evaporators, we have been plagued by the chipping of bell jars for many years. We have used (and sold) Torr Seal and several other epoxies over the years and they are satisfactory for small chips. However, for large chips we have found that epoxy-putty GAPOX10™ works extremely well. It is inexpensive and cures in one hour for sanding and smoothing. We can pull a vacuum of 10^{-7} Torr and have yet to encounter a problem.

Mike Bouchard, Ladd Research

Editor's Note: Allow me to second the above repair method. I've used it to repair a seriously chipped Denton 502A bell jar (it would work on any). After overnight curing (room temperature), the unit pulled as good a vacuum as quickly as it did before the chip. Note that the epoxy must fill the void left by the chip! - Phil Oshel

Using Micro-Fourier Transform Infrared Spectroscopy (FTIR) to Identify Particles:

FTIR is able to identify particulates without extraction, depending on the matrix in which the contaminant is found. I have found it useful, however, to at least "rinse" the contaminant in a solvent (in pharmaceutical applications the solvent is usually water) first. Otherwise, there may be trouble with minor extraneous absorptions which hamper the positive identification of the contaminant, especially with automated search routines.

I have found that a low power (10-60x) stereomicroscope with both reflected and dark field lighting is imperative in the pre-analyis prep of such samples. A clear glass spot plate (or microscope slide with wells) can be used for extraction/cleanup of particles. They can be viewed under the stereomicroscope while the extraction occurs. A good supply of micro-probing tools (available from most SEM supply houses) is essential.

If the particles are teased around in the solvent, and pulled away from the solvent as it evaporates under the heat of the microscope, the particle can usually be cleaned up enough to obtain a good spectra.

Particle size is another consideration. Typically, a useful transmission spectrum can be obtained from particles greater than about 20 µm. Here, the apertures of the FTIR microscope should be adjusted such that they fully encompass the thinnest section of the sample. Reflectance spectra may require larger sizes. Typically, in both of these analysis modes the flatter the sample, the better the spectrum. If the particle or fiber is pliable, I often find it useful to flatten it in a KBr pellet mold. While viewing under the stereomicroscope with reflected light, I then carefully transfer the sample to a KBr (or reflectance) disk for subsequent Micro-FTIR. Fibers generally work quite well in transmission mode. In fact. I once built a library of spectra for the types of fibers found in the cleanroom garments used in the compounding area tor a pharmaceutical product. I subsequently used that library to identifyl/troubleshoot particulate problems for the customer.

Bob Citron, Chiron Vision Corp., Claremont, CA

Staining for Elastin and Collagen:

Elastin and collagen are both quite distinctive in appearance in the TEM, and so they can't be contused with one another. However neither collagen nor elastin stains well with the usual U acetate/Pb citrate combination. Here are our methods (cheap!):

For elastin -

Make up 0.2% orcein in acid alcohol (0.1 g orcein, 5O mL 70% ethanol, 0.3 mL conc. HCl) and filter. Keeps well. Stain grids on drops of this stain *(e.g.,* on a wax sheet, covered) for 30 minutes at room temperature, then rinse with 50% ethanol and stain with uranyl acetate and lead citrate as usual.

We use this to demonstrate the elastin in artery walls, and both newly-forming elastin and "old" elastin stains well. I don't have a reference for this method, I'm afraid.

For collagen -

Make up 0.01% tannin (tannic acid) in water, preferably fresh, and stain grids on drops of this for 3 minutes at 60°C. Rinse with water and then stain with uranyl acetate and lead citrate as usual.

Collagen shows up well, but so do some other extracellular components including elastin sometimes. The reference is: Dingemans, *et al.* (1990) Ultrastructural Pathology 14: 519-527.

Note: We always post-stain with uranyl acetate and lead citrate because we want to see other features in the sections, and it is possible orcein and tannin won't work on their own. So if you want to stain only collagen and elastin, and nothing else, you might have to resort to immunogold methods.

Stephen Edgar, School of Medicine,
University of Auckland, New Zealand