



MICROSCOPY

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We appreciate the response to this publication feature - and welcome all contributions. Contributions may be sent to Phil Oshel, our Technical Editor at:

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Embedding Polymers That Dissolve in Embedding Resins

Some polymer fibers and small polymer particles tend to dissolve in any of the standard embedding systems. We solved this problem by taking the following approach:

1) Take a flat, clear embedding mold, and fill it half way with either SPI Pon™ 812 or one of the other popular "Epon substitute" resins.

2) After polymerizing into a block, apply sparingly to some of the fibers (or particles), preferably in a dry form. If deposited from a liquid, surface tension forces interfere with being able to coat the underside of the fibers because of greater particle agglomeration.

3) Then metallize with Pt in a sputter coater. Au is OK if you don't have Pt handy, but Pt tends to be less likely to smear when thin sectioning.

4) Then "disturb" the fibers (by shaking, or a slight blast with a duster) to the extent that at least some have been moved with the unmetallized side up and metallize again. We do this three times. The idea is to encapsulate the fibers in a passivation layer to provide protection from contact with the embedding resin.

5) The cavity can then be filled up with resin and when cured, the block sectioned, resulting in undisturbed and unmodified (by the resin) cross-sections for TEM. The resin should be put in, in two steps, the first being a very thin layer to cover the fibers and prevent them being disturbed by the second layer. The embedding cavity is then filled by the second layer.

Metallizing could be done using osmium instead of platinum since in theory at least, an osmium coating should be better than platinum because of osmium's amorphous nature and zero grain size. The benefits of osmium plasma coating can be found on the SPI Supplies website at www.2spi.com. ■

Disclaimer: SPI Supplies manufactures and distributes some of the products mentioned so we would have a vested interest in their greater use. We have also been performing these kinds of laboratory analytical services since 1970.

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How to Keep Embedding Resin Bottle Caps and Threads Clean

Epoxy resins used for embedding in transmission electron microscopy are viscous liquids that are quite messy if not handled properly. For example, the stock component bottles frequently trap a small amount of liquid (anhydride or resin) between the glass bottle cap and threads that often makes removal of the caps very difficult. Even careful pouring and wiping of the threads after each use may not completely eliminate this problem. I find that wrapping the threads of newly opened bottles with Teflon plumber's tape keeps the lid from sticking. Teflon is inert and will not react with the resin components. The ideal solution would be for some EM supplier to provide bottles with Teflon or polypropylene coated threads and caps. Perhaps the entire container could be made of polypropylene, a relatively inexpensive plastic that will not permit epoxies to adhere.

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A Quick Test To detect Depolarization Of Scanned Probe Microscope Piezo Elementse

I did a little experiment to measure the change in capacitance with depolarization of the piezo elements of a SPM. I measured on average of 30% reduction in capacitance after depoling.

I took five piezo elements from my junk drawer, measured the capacitance before, and after, being depoled. I believe the piezo material is PZT5A or something very similar. I depoled the piezos by heating them with a hot air gun. All measurements were made with the piezos at room temperature.

Piezo Element	Poled Capacitance	Depoled Capacitance	% Change In capacitance
#1	14.7 nF	10.3 nF	-29.9%
#2	14.6 nF	9.8 nF	-32.9%
#3	14.1 nF	10.4 nF	-26.2%
#4	14.8 nF	9.2 nF	-37.6%
#5	13.8 nF	10.0 nF	-27.5%
Average:			-30.8%

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Preventing Particle Agglomeration When Preparing Powders For Examination In The TEM

We have looked at a variety of powders (including ceramic powders), and have found that much of the agglomeration that is seen is probably occurring while the drop of the dispersion is drying on the grid. Long times in the ultrasonic will likely not help prevent this. We often found that if the particles are not sintered together then a few minutes of sonication will do.

Other points to keep in mind are:

Dilute your dispersion as much as possible (there should be a barely noticeable change in the turbidity of the liquid). This will minimize agglomeration during drying. Generally, I do not wait for the liquid to completely dry on the grid. Instead, after 10 to 20 seconds, I remove the excess liquid using the corner of a filter paper or tissue.

If this does not work, try spraying the dispersion onto the grids. Kits for doing this are available from various EM suppliers and they will also help minimize agglomeration during drying.

Another way is to mount your carbon coated grids onto a rotating plate (I have improvised with a Dremel tool), so that when you place a drop on the grid it will spread out and minimize agglomeration.

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