TECHNICAL DATA SHEET
COATING TEM GRIDS

SPI Supplies
206 Garfield Avenue,
West Chester, PA 19380, USA

Filmed Grids for TEM

Some tips from our grid coating experts!

The control of the thickness of the coating is extremely important, and it is probably the single most difficult aspect of the making of coated grids. A "must" in terms of film thickness uniformity is for the casting liquid surface to be absolutely flat. One should not be picking up film that has formed too close to the "edge" where the liquid could be concave or convex. If the non-uniformities are sporadic, then there could be a contamination problem with the liquid.

The procedures will vary with the exact resin used.

For Formvar™ coated grids, 0.25 to 0.3 % Formvar resin in dry ethylene dichloride. What is not generally appreciated is that there are a number of different grades of the resin called Formvar, but so far as we know, only one of them is optimum for the filmed grid application. If one was using other grades, they might require different concentrations or as many have learned, most of the other grades do not really work at all!

For thicker films, we would suggest increasing the concentration to the range of 0.4 to 0.5%. Thicker films are usually made when one wants to make a holey film with bigger holes. We find that a concentration above about 0.7% results in a film that is literally too thick for any EM application.

If one wants thinner films, then reduce the concentration. But if you are already making relatively decent films, then you are at the borderline in minimum thickness anyhow but the concentration could be reduced a bit more to about 0.2%. However, below 0.2%, the film becomes just too susceptible to cracking, so that becomes the lower limit for reducing the thickness and therefore also, the concentration.

We do realize that this is more easily said than done so we provide some additional guidance. Let us assume we are going to be coating 200 mesh grids. Try to aim for the film, when floating on the water surface, to be silver to barely silver/gold, in other words, nearly colorless. The support film made from that kind of floating film should be quite robust in both mechanically, strung over a grid hole, including a slot grid, and also reasonably stable in the electron beam. In fact the film should be sufficiently robust that a carbon coating should not be necessary, and this is a real advantage when one is looking at nanoparticles, for example which could be confused with grain from a carbon film.

But anyone who has made filmed grids will attest, the real test, at the "end of the day", is how the Formvar film behaves in the vacuum of the TEM and under exposure to the electron beam. These recommended concentrations, and other aspects of the technique are all optimized in order to produce optimum performance, e.g. long lasting films with virtually no drift once in the column and being exposed. But one really does need a TEM right there, next to where the films are being cast, so that they can be instantly inspected and corrective action taken, right there on the spot, e.g. by way of a concentration change. If this is not done, the QC step ends up being when you gear up to do your experiment and then you might very well find out the grids are not usable. Variables such as temperature, humidity and perhaps other factors seem to come into play, so that the optimum concentration one day might not quite be the optimum concentration on some other day. There is even one school of thought that believes that light slowly degrades the solid Formvar which typically might be quite old, since it is used so sparingly.

The method described above, the one that we believe is used by most makers of filmed grids, is actually only one of two methods. The other approach involves dipping a glass slide into the Formvar (or other polymer) solution, with drawing the slide and capturing a thin film of the polymer on the slide. This has also been done with freshly cleaved mica.

While this approach might sound a bit easier, it too has its difficult "step", that one being the stripping of the Formvar (or other polymer) film from the slide. In order to facilitate the removal, or stripping of the films, we would suggest the following procedures:

a. Start with a normal 1" x 3" (25x75 mm) glass microscope slide.

b. Dip the slide into a beaker of saturated Alconox® solution, which has been previously filtered with Whatman #1 filter paper.

c. Wipe the slide dry with a Kimwipe® and then, when dry, with several fresh Kimwipes, dry polish the slide by vigorous rubbing. Some experienced film makers do their rubbing with Lens Tissues. When finished, do a final cleaning with a duster. Sometimes veteran coaters claim they have had problems with a particular brand of slides, but from what we can determine, all brands seem to produce equivalent results, but what can vary is if the particular slides being used are old, and are exhibiting signs of "glass corrosion". If one experiences problems getting the films to release, we would suggest getting a fresh box, making sure that it is not from the same batch, but one that is much more recently manufactured.

   Alternative method:
   Some veteran experts take the slide in its "as received" condition and dip it into absolute ethanol instead of the Alconox solution. The slide is then immediately dried with either SPI Lens Tissue or Ross Lens Tissue. One should immediately proceed with the next step.

d. Apply the Formvar (or other polymer) film, from an ethylene dichloride solution. The Formvar concentration is a bit higher than mentioned above, and will also depend on the speed with which the slide is withdrawn from the solution. In this case, the faster

the slide is withdrawn, and the faster the liquid is allowed to run off, the thicker will be the resulting film. This is the part of this technique that causes the most problems because of the difficulty some persons have in reproducing the conditions of polymer film formation exactly e.g., getting films of uniform thickness, slide to slide.

e. The polymer film, now on the glass slide should be allowed to dry completely.

f. Holding the slide with the polished slide down, scrape the edges of the slide closest to the polished surface with a freshly cleaned razor blade. An acetone/alcohol cleaning combination is the recommended approach.

g. Score the polished surface, that is, the one with the polymer film, with the razor blade, and then blow with an SPI duster any debris that might be remaining.

h. Now using a deep petri dish, one at least several inches deep, and filled nearly to the surface with distilled water, hold the slide at a 45° angle to the water surface, and then slowly lower it into the water itself. When the water reaches the score marks (parallel to the short dimension of the slide), very slowly continue to lower the slide and surface tension effects should then do the rest, with the polymer film smoothly floating off of the slide.

i. At that point, grids can be picked up, one at a time, and dunked into the water and then brought up underneath the floating film and with lifted out of the water and dried.

This second procedure should also result in excellent Formvar or other filmed grids. What makes this part of the process so interesting is that a technique one person finds easy to practice, another finds difficult and *vice versa*.

We would caution the initiated, however, that despite the ease at which it sounds one can make filmed grids of high quality, just remember that this is indeed an art and one does not learn an art in a few minutes. Be prepared to spend some number of hours perfecting your own art in the making of filmed grids for TEM! We have people in our own laboratory who have been making support films for as much as 50 years, and even they have to adjust the dilution, etc. to get films that are consistently beam stable.

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Additional Suggestions on the Removal of Formvar from a glass microscope slide

This is a continuation of our efforts to make everyone in the world an expert grid coater! We find that this is a dying art and we don't want it to become a lost art..... Naturally we hope you will rely on SPI Supplies for your grid coating/filming supplies!

But if you have tried our first recommendation, and still find difficulty getting the Formvar® (also called Vinylec®) film to release, you might want to try the following procedure, taken from the Microscopy List Server, a posting made by Gib Ahlstrand, University of Minnesota:

The procedure is as follows:

1. Rinse the glass slide, both sides, including the edges, except for the end that you are using to hold the slide, with 95% ethyl alcohol, followed by air drying. *Use must be immediate!*

2. Quickly dunk into Formvar solution (0.25 to 0.5% weight/volume in ethylene dichloride) and then retract, drain and allow to air dry.

3. Score around the edges to break the film which is then floated off onto a clean water surface. For scoring of the edges, use the corner of a razor blade, near the edge. Also score the actual corner edge of the slide with the blade held perpendicular to the edge. Also score across the slide near the "top" edge of the Formvar film, near the end you are holding on to to hold the slide, for clean release of the end of the film.

Special precautionary statement: Formvar solutions older than three months when cast as a film on a slide tend to stick to the glass slide surface. We recommend putting the mix date on the bottle of fresh solution, and after three months discard and make new solution, otherwise poor release effects tend to appear.

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