Methods for Preparing SEM Specimens for Micrography with Black Backgrounds

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The basic trick is to produce a very smooth, featureless background, preferably using low Z (atomic number) or low density materials. The following three methods exploit in 1: round glass coverslips, in 2: double stick tape and/or colloidal graphite paint for backgrounds, and in 3: a combination low Z plus high Z conductive coating strategy is described which when used in combination with 2, also produces the desired background effect.

1. I've done the following for pollen grains, rust spores, and soil particles (dried down onto glass coverslip support surface from water or ethanol, they just stick naturally), should work with larger samples, eg., insects, seeds, provided that any paint you use to mount the sample to the glass coverslip does not spread out beyond the edges of the sample so you don't see it in the background. Mount a cleaned (EtOH rinse, air dried) 1 cm diameter round glass coverslip onto a standard aluminum stub using double stick tape (or paint) and edge the coverslip with colloidal graphite paint to provide a conductive path to the aluminum and for extra adhesion. Mount large samples to the coverslip using a tiny dot of colloidal graphite paint, or using the pin mount method (first apply dab of paint to one end of pin, use wet end to pick up sample, seed or insect, etc, let dry, then apply dab of paint to other end of pin and mount to center of coverslip - keep it balanced - and dry completey). Heavy metal coat on a rotary stage as usual and view in the SEM. In spite of the fact that the glass (silicon & oxygen) coverslip is dense and coated with gold-palladium, the pollen grains, for example, are brighter than the background and when setting up the SEM exposure it is often quite easy to drop the featureless background into the black by appropriate settings of your SEM's gain and blacklevel (or brightness) controls. If you can't drop the background brightness totally into the black without also getting areas of your sample too dark, it may be possible, with a bit of dodging of the subject in the darkroom when making a print from the negative, to burn in the background to black. The key here is that the background is featureless so that there are no edges of paint globs or other debris whose deges will light up in secondary electron imaging.

2. Similar to the above, is to use a smooth surfaced double stick tape to mount your samples to avoid using paint (for very small samples) or as a smooth, low Z background for the pin mount technique. Some double stick tape surfaces develop tiny cracks or hole patterns in them as a result of being in the vacuum, beam, or whatever and may not provide a feature-free background. Spreading carbon paint with a brush to get a low Z background may work if the paint is quite thinned out by adding solvent but sometimes it leaves too many brushstrokes or paint chunks visible and that detail may show up in the background as bright edges.

3. This method gives a low-Z background with a high-Z coating on your sample: Mount samples (directly or pin-mounted) on double stick tape or with a spot of carbon paint onto a stub that has been previously painted with carbon paint (and dried) to hide the aluminum, so you have a low-Z background. Set up your vacuum evaporator rotary stage to zero degrees tilt if you can. We have a Ladd rotary tilt stage whose "whirling disk" can be re-mounted to zero degrees tilt, that is, horizontal. Set up the evaporator for an overhead carbon rod evaporation on one set of electrodes; set up another set of electrodes for a very low angle metal (Au-Pd) coating of less than 3 degrees elevation; straight on. After pump-down, set the stage rotating and do the two coatings, in either order. Very little metal coating will hit the stub surface because of the low angle of coating. A lot of metal will hit the target, tho' it may be a bit thin on top of the target, hence the overhead carbon coating to get some extra conductivity on top surfaces without adding high Z coating to the background. This method has worked very well for imaging seeds from 0.5 to 4.0 mm diameter in backscattered imaging (I wanted even "overhead" illumination that BSE gives) with black backgrounds. Should also work OK for secondary electron imaging (gives typical "side" illumination). ■