Improving the Resolution of Sputter-coated Films

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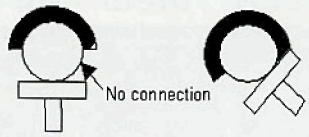
After an inquiry from the Microscopy Listserver, I went back to my 1980 copy of Scanning Electron Microscopy, volume I. Several authors had investigated the structure of thin metal films by depositing the films onto carbon-film-covered TEM grids and imaging the films at high magnification. There were several proposals for new devices that have since become standards for high-resolution coaters[1, 2], but the Listserver inquiry was for a fine conducting film suitable for high-resolution SEM from an existing sputter coater[3]. There were several factors studied that influenced the fine structure of the films. The first was the materials sputtered: for a given set of conditions of voltage, current and time, platinum gave the finest film, 60% gold-40% palladium (Au/Pd) the next finest and pure gold the least fine. Other materials such as tungsten[4], tantalum, nickel, chromium and molybdenum were also tried and gave very fine films with almost undetectable structure, but they will all oxidize, so they are not good for long-term storage of specimens after sputtering. I tried a nickel target in my coater, coated a carbon-covered grid and examined the resultant film in the TEM. The film was thin and the structure was almost undetectable, but I never used it on SEM specimens, so I don't know how well it coated the specimens for practical use.

The second factor that influenced the sputtered structure was the temperature of the substrate and sample as they were being sputtered[3]. Lowering the temperature seems to reduce the mobility of the metal atoms after they hit the sample surface, preventing them from agglomerating into large clumps and keeping the film small-grained and thin. Some sputter coaters have sample cooling capabilities and I have heard of someone making a sample holder block, insulated with teflon and grounded with a wire, which was chilled in a freezer or liquid nitrogen before being put in the coater. You must let it warm up completely after coating and before venting the sputter coater, to prevent moisture condensation, but it does make the coating finer-grained if all the other parameters remain constant. A third factor was accelerating voltage. Some sputter coaters allow you to adjust the voltage and I found that I could lower the voltage on my unit to 700 V and 25 mA current for three minutes to sputter Au/Pd and yield a fine enough film for my use. If I used pure gold I could lower the current to 20 mA. The argon pressure was maintained at 150 to 200 millitorr.

While doing experiments on the imaging of charge in a ceramic sample that sintered together a conductive (SiC) phase with a non-conductive phase (Al_2O_3), I tried to do a very short (ten second) coating, under my usual conditions of 700 V and 25 mA, to retain a bit of charging to differentiate the phases. To my surprise this rendered the surface completely conductive. I found it worthwhile, particularly with smooth samples, to try very short coatings and re-coat again if the first was not quite enough. This is very dependent on the surface area of the sample, but the film has much less structure when it just starts to nucleate than after prolonged bombardment has heated the substrate and re-crystallized the thin film. I have found that with difficult samples, such as fluffy bugs, it is better to do several short coats, turning the sample between each coat, and to use some conductive paint to connect the area of interest to the sample stub, so the coating does not have to conduct too far. Tilting the sample on its side for a second coat is useful to get the coating under edges or to connect round particles to the substrate. See following illustration.

Since I do not have a field-emission SEM, I cannot evaluate the structure of the finest coatings, so these measures are enough to provide featureless coating for my use. Imaging a gold-sputtered film on polished graphite is actually a good way to check a high resolution SEM for astigmatism and sharpness. ■



Coating a second time with the sample at a 45 degree tilt may help complete the path to ground

1. Peters, K. R. 1980. Penning Sputtering of Thin Metal Films for High Resolution Electron microscopy. SEM/1980/I, SEM Inc., AMF O'Hare, IL. 143-154.
2. Franks, J., Clay, C. S., Pease, G. W. 1980. Ion Beam Thin Film Deposition. SEM/1980/I, SEM Inc., AMF O'Hare, IL. 155-162.
3. Echlin, P., Broers, A. N., Gee, W. 1980 Improved Resolution of Sputter-coated Metal Films. SEM/1980/I, SEM Inc., AMF O'Hare, IL. 163-170.
4. Slayter, H. S. 1980. High Resolution Metal Coating of Biopolymers. SEM/1980/I, SEM Inc., AMF O'Hare, IL. 171-182.

Preparation and Use of Needles and Micropipets for Handling Very Small Particles

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small drops of nonane carefully guided with a needle over the particles, any oil in them can be extracted right on the crystal for IR analysis (see Figure 17). The reason that this extraction works is because the whole drop deposited by the micropipet, as well as the particle, is in the field-of-view at 10 or 20X. One can watch the drop going to dryness and one is able to observe a small amount of oily residue after the nonane evaporates. One can immediately mark the position of the residue with a tungsten needle and run a blank next to the residue by placing a drop of pure solvent to check for a deposit. Large drops, such as those taken directly from the 15 mL ground glass bottles, would not work on the small KBr crystal or any other surface if the sample is 100 times smaller than the drop. Also, it would be difficult to keep the large drop together as it goes to dryness.

Frequently, a tiny piece of polyester filter may be used to remove a micro drop of oil from a hard-to-reach place. To extract the oil from this filter for further analysis, a small drop of nonane is deposited on the surface of the KBr and immediately the filter, held on the tip of a needle, is dipped in the solvent. Most of the oil will remain with the solvent and, as the solvent evaporates, one will see an oily drop appear on the surface of the crystal. The position of the drop is marked with the needle because small drops are hard to relocate once the field-of-view is changed.

These are just some of the ways that tungsten needles and micropipets have been used. There are many more. ■

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Anna is this years recipient of the Chamot Medal awarded by the State Microscopical Society of Illinois. This annual award, recognizing an individual who has made outstanding contributions to the field of microscopy, will be presented at the INTER/MICRO-2000 conference this June. And if you would like to observe her photomicrography talents, take a look at the "Polypropylene with Phthalocyanine Blue Pigment" photomicrograph on the Nikon 2000 calendar.

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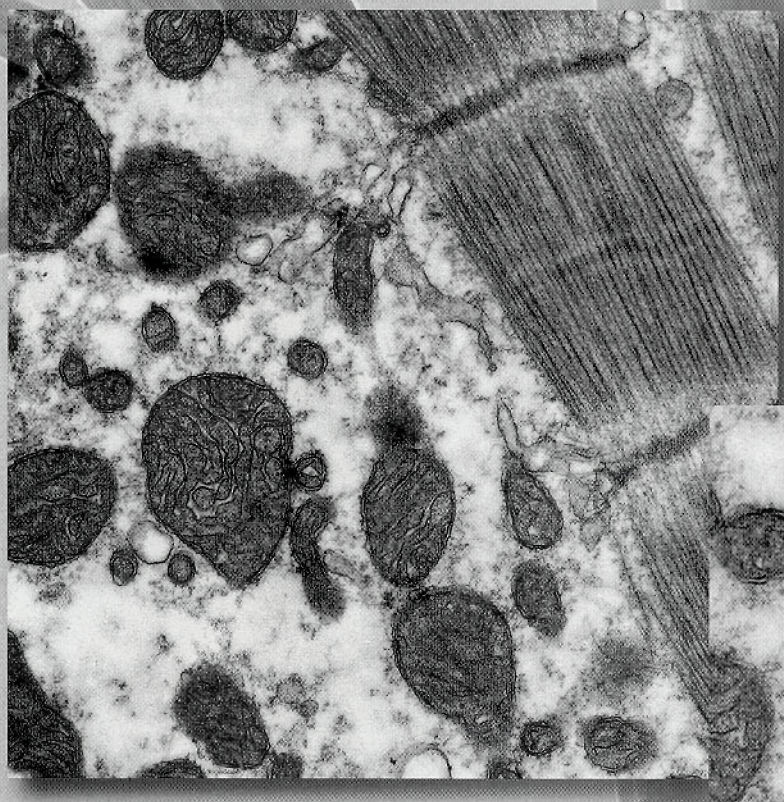

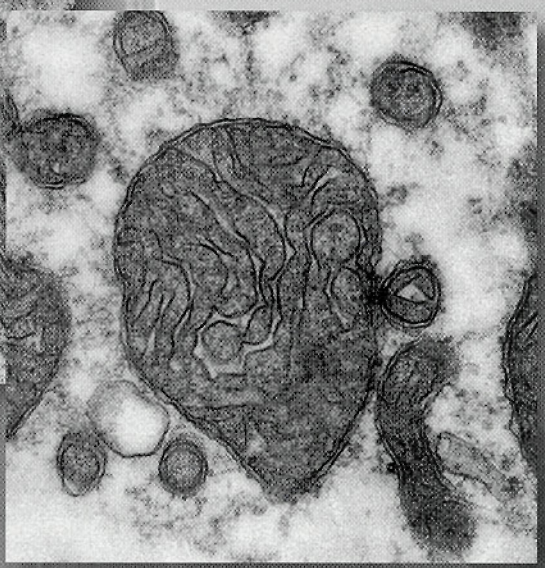

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