Double-Coating Of Three-Dimensional SEM Specimens

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The Problem:

Many of the SEM specimens which we are asked to deal with are highly three-dimensional non-conductive objects, such as silica beads, pollen grains and spores, small inorganic and organic crystals such as zeolites and drug preparations, starch grains, etc. These often cause severe charging problems which result in all of the familiar and tiresome charge-related image artefacts, such as flare, excessive contrast, specimen movement, and variable scan lines. Charging can also make it very difficult to obtain high-resolution images or do satisfactory analysis.

The Cause:

Although mounting the specimens on conductive carbon tabs can help to reduce these effects, more effective measures are often required to control them. In most of these cases the cause of charging is that only line-of-sight surfaces of the specimen are coated, and deeply overhung or undercut surfaces receive little coating. Consequently although the upper hemisphere of a spherical specimen will be covered with a conductive layer, little metal will accumulate on its underside, and it will remain electrically insulated from the carbon tab and the stub. Therefore, despite precious metal coatings thick enough to degrade fine structure, some of these specimens still persist in charging very badly.

The Solution:

A simple tip for dealing with this is to coat both the top and the bottom of all particles. I find the simplest and most reproducible way to achieve this is to dust the particles onto the surface of a temporary support made of a slightly tacky material. Silicone rubber, or the non-slip mats made by Dycem Limited (http.//www.dycem.com), hold the particles sufficiently securely to allow their bottoms to be coated. Once the bottoms have been coated the particles can be picked up on a specimen stub covered with a carbon-adhesive tab, and the stub is then returned to the coater to coat the tops. The improvement in specimen quality which results is dramatic. Charging is well controlled, especially on well-separated single particles, and the specimen can now be examined at high tilt angles if necessary without any risk of scanning uncoated areas. Clumps, aggregates and overlapping particles may still show some charging, but can usually be avoided. The method works well on specimens of all sizes from 250 nm or less to 1 mm or more, but larger specimens may require a more aggressive adhesive, such as a very low-tack pressure-sensitive adhesive. Best of all, the top surface of the specimen now only needs a much lighter coating than would otherwise be necessary, so true ultrastructural work is possible.

Comments:

Some arising from discussion with Dr. Charles Garber:

It is essential that a conductive adhesive used to pick up the specimens following the first coating is conductive. If it is not, then the particles will remain electrically isolated after the top-coating and the point of the exercise will have been lost.

Light pressure is applied to the specimens during transfer from the temporary mount. Although the temporary mount and adhesive are both soft, compliant materials, pressure could result in distortion or fracture of very fragile particles. Make sure the specimens are robust enough to withstand such forces.

It is possible that size-selection may occur during transfer processes. If size distributions are important, the selection properties of the system must obviously be investigated. ■

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