Coating Effects On BSE Imaging

Woody White, McDermott Technology, Inc.
woody.white@att.net

Poorly conducting specimens can be examined without coating by using a variable pressure SEM. However, many labs may only have a high vacuum SEM, or for other reasons, choose a high vacuum mode. In order to examine insulating specimens in a high vacuum SEM (using operating conditions conducive to BSE and X-ray analysis) specimens must be coated with a conductive thin film.

The perspective of this article is from a materials point of view, but the principles remain the same for biological examinations requiring similar information.

Back Scattered Electron (BSE) image contrast is primarily a function of the average atomic number of an imaged area. This is particularly true for polished specimens where there is no topography to contribute to contrast. The BSE coefficient is *the ratio of back scattered electrons to incident (beam) electrons.* Higher atomic number elements have a higher BSE coefficient (*i.e.*, are more "reflective"). A higher rate of detected BSEs will normally produce a brighter image response.

Higher atomic number (Z) compositions are brighter.

Lower atomic number materials are darker.

BSE imaging is invaluable when searching a specimen for variations in composition. An uncoated, polished, conductive specimen is ideal for most SEM procedures, including BSE imaging. With such a specimen, variations in compositions can be seen using BSE imaging that cannot be detected by most Energy Dispersive Spectrometers (EDS).

The real world, however, is seldom that nice! Conductive coatings must be used which will influence the examination. The obvious goal is to use a coating that will minimize this influence. The coating of choice will depend on the examination goals. Considerations must be given to a number of variables including the secondary electron (SE) resolution requirements, BSE sensitivity, X-ray analysis requirements (for both interference lines and loss of detestability limits), etc. There are always exceptions, but in general, the thinner the layer and the lower the atomic number of the film, the better.

Electron penetration (and subsequent escape as a BSE) depends on the composition of the specimen (and the coating), beam incident angle and the beam potential. "Monte Carlo" computer programs are available to model the analysis volume generated. Under many operating conditions, subsurface composition variations can be observed using BSE imaging. The ability to subsurface image permits the specimen to be coated for conductivity and maintain composition contrast. There is unfortunately a trade-off. *Any coating will reduce apparent sensitivity to composition* changes. A coating material and method must be selected to provide conductance while minimizing sensitivity loss.

Beryllium may make the ideal film for many BSE exams, but is not practical because of its toxic properties. Carbon is the best practical material for most BSE and X-ray examinations. Since carbon is difficult to sputter, it is typically applied by evaporative coating.

For enhanced secondary electron imaging resolution, specimens are often sputter coated with gold, palladium, Au/Pd alloys or similar alloys. The thickness of these sputtered noble metal films must be minimized to maximize BSE sensitivity.

To quantify the loss of BSE sensitivity with increasing gold film thickness, the following experiment was performed.

The specimen was a polished tool steel mount, imaged under nearly identical conditions for each test. There is only a small amount of unintended topographic relief resulting from hardness differences in the specimen (polishing artifact). This material has

DESCRIPTION OF IMAGES IN THE FOLLOWING FIGURES

- Matrix is lowest atomic number phase (darkest).
- Large phase is next higher Z. Small phase is highest (brightest)
- The left-to-right straight line through the image is the line of analysis.
- The stepped, wider line is a plot of the image brightness along the line of analysis

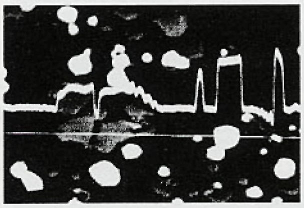


Figure 1: Uncoated surface, relative response is 100%.

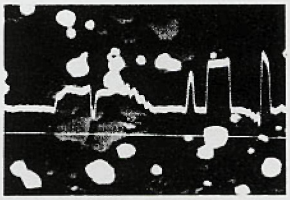


Figure 2: Carbon coated surface. Response time: 89%. Applied using yarn evaporator. Thickness not determined.

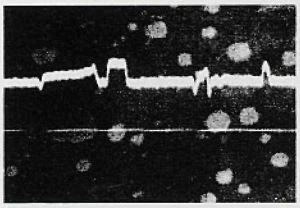


Figure 3: Gold coated (~4 nm). Response:30%

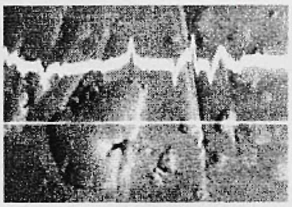
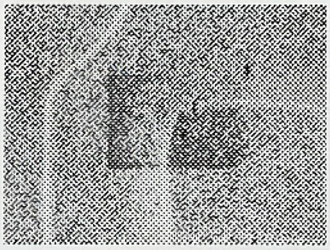
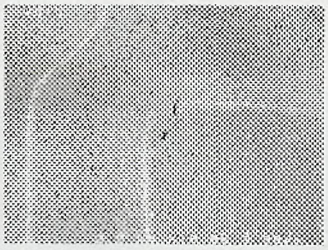


Figure 4: Gold sputter coated (~12 nm). Response: Almost none. Most is topographic, not composition





precipitates of several atomic numbers (Z), best seen in the "uncoated" condition (Figure 1). Composition contrast from the specimen is shown to reduce as the coating "Z" is raised and/or the coating thickness increased.

During the experiment, the absolute brightness was adjusted to yield usable images. As the gold coating thickened and became a more significant portion of the BSE analysis volume, the "apparent" average Z of the specimen increased. The brightness (dark level/DC level) adjustment was set for approximately the same specimen matrix gray level for each example. The contrast (BSE detector gain) and beam current were constants.

The figures show that thicker, higher "Z" coatings increasingly mask the underlying material.

Similar problems also occur with x-ray analysis (EDS, WDS). Not only can characteristic x-ray lines from coatings mask other elements, but detestability limits for elements in the specimen will be reduced as the coating increases. This is especially significant when analyzing for lighter elements.

Graphic results of the experiment are shown in Figures 1 - 4. Figure 1 is the uncoated specimen that is referenced as a 100% BSE response. The carbon coated surface is shown in Figure 2 and exhibits a response that is 89% of the uncoated surface. This thickness, applied using a "yarn" type evaporator is estimated to be in the realm of 100 nm. Figure 3 shows the reduced response from an indicated 4 nanometers of gold. It is only 30% as intense as the uncoated surface. In Figure 4, additional 8 nanometers of gold were applied for a total thickness of 12. At this thickness and beam potential, almost all composition contrast is lost. Nearly all BSEs are originating from within the coating rather than the specimen. The accuracy of the gold thickness is not established. A quartz crystal (oscillator) supplied as an accessory to the sputter coater was used to obtain the reported coating values.

The relative effects are valid but, unfortunately, the beam voltage used for this experiment is not available. The voltage was likely on the lower end (5-10 kV). At higher voltages (20-30 kV) the coating effect would be reduced as the analysis depth is increased and the coating signal becomes a smaller component of the total BSE signal. ■