Analytically Speaking

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Characterization of Deposits, Coatings & Electroforms—The Use of Optical Microscopy

Optical Microscopy

Optical microscopes were used exclusively for the examination of the structure of deposit, coatings and electroforms until 1949. Because of the poor depth of focus of optical microscopes, one can only view the structure of polished and etched (metallographically prepared) cross sections.

"Depth of focus" is how clearly objects appear that are at various distances from the lens. Poor depth of focus can be described as when an object close to the lens appears in sharp focus, and an object in the background appears "fuzzy." The reason the as-plated surface of an electrodeposit cannot be examined with optical electron microscopy is that if protrusions appear in sharp focus, the depressions look fuzzy—or vice versa.

The Most Important Properties Of Microscopes

There are three very important properties of microscopes: Depth of focus, resolution and contrast. *Resolution* is how closely two points or lines in the structure may be separated and still be distinguishable. The wavelength of visible light and the numerical aperture of the lens (its light-gathering ability) limit the resolution of optical microscopes. Therefore, points or lines separated less than about a micrometer cannot be distinguished.

Contrast is determined by the shades of dark and light in the image. Two points that have the same shade obviously cannot be distinguished from each other. The classic example of poor contrast is a picture of a white rabbit in the snow. In optical microscopy, contrast is primarily determined by whether structural features are inclined or parallel to the surface.

What Can Be Controlled

The three aspects of microscopes that can be controlled are the magnification, the illumination and the focus. The magnification of optical microscopes is determined by the product of the magnifying power of two lenses and the effective distance between them. The two lenses are the objective lens and the eyepiece. For metallographs that are optical microscopes with attachments for photographing the structure, the distance between the eyepiece and the camera also determine the magnification.

Optical microscopes, as the name applies, use visible light for observing the structure. Most also have means for polarizing the light or tinting it. Polarized and tinted light can aid in the identification of non-metallic inclusions. Focusing primarily is accomplished by changing the distance between the specimen surface and the objective lens. For objective lenses of high magnifying power, this distance is so short in air that the lens touches the specimen surface. By immersing the lens in a drop of oil, the numerical aperture is changed so that the distance-to-focus between the specimen surface and the lens is lengthened.

Contrast is determined by whether structural features are parallel to the surface or inclined. Microscopes are generally operated in the so-called "bright field mode." In this mode, the light reflected by structural features perpendicular to the incoming light beam is reflected back into the objective lens and the features therefore appear bright. Features inclined to the incoming light beam reflect it outside the lens, and therefore appear darker.

To attain contrast, some portions of structure must be inclined to the incoming beam. The main purpose of etching (attacking the specimen, usually with an acid) is to attain contrast. A polished surface would not exhibit contrast because it wholly reflects the light into the objective lens and therefore appears uniformly bright. By etching, some structural features become inclined to the beam and, as a result, appear darker. In this way, the grain structure can be distinguished. Through the use of certain etchants, grains are attacked differently, depending on which crystal plane is parallel to their surfaces. Different grains exhibit various shades of light and dark.

The as-plated surfaces of deposits that are not fully bright generally show structural features that are inclined and therefore do not require etching. As was previously discussed, the whole surface would not be in focus. Some inclined features may look like grain boundaries. They are often the crevices surrounding nodules, however. Even if the depth of focus permits being able to observe the structure, the grain size should not be determined from observations of the as-plated surfaces of deposits. The cross sections of deposits need to be polished and then etched. Polishing a specimen, especially a relatively soft metal, can result in severe plastic deformation. This plastically deformed layer must be removed by repeated etching and slight repolishing or it will obscure the true structure. **P&SF**

Next month: *Scanning Electron Microscopy*