

Analytically Speaking

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Characterization of Deposits, Coatings & Electroforms-Vector Calculations

Editor's Note: In last month's column, the Miller indices of a diffraction pattern were considered. We now turn to the necessary vector calculations.

To index the diffraction pattern, it is plane of a family that caused a spot. To discover which planes of the {111} and {220} families caused Spots No. 2 and 3, the equation for the angle between two vectors is used. The cosine of the angle ϕ between the vectors h_1k_1l_1 and h_2k_2l_2 is given by

$$\cos\phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{(h_1 + k_1 + l_1)^{1/2} (h_2 + k_2 + l_2)^{1/2}}$$
(1)

Any plane of the {111} can be assumed to have caused Spot No. 2. The (111) plane is selected. It may be recalled from previous discussions that for cubic lattices, the direction perpendicular to a given set of planes has the same three indices as the plane. Accordingly, the direction from Spot No. 1 to Spot No. 2 is the [111] direction because the (111) planes were assumed to have caused Spot No. 2. The direction or vector from Spot No. 1 to Spot No. 3 is perpendicular to the [111] direction, so the angle ϕ is 90°. Cos ϕ , therefore, is zero. The direction from Spot No. 1 to Spot No. 3 has to be of the <220> family of directions because it was previously determined that Spot No. 3 was caused by planes of the {220} family. Substituting $h_1 = 1$, $k_1 = 1$ and $l_1 = 1$ for the [111] direction into Eq. (1) results in

$$1xh_2 + 1xk_2 + 1xl_2 = 0 (2)$$

If the substitution $h_2 = 2$, $k_2 = -2$ and l_2

= 0 is made, this equation is satisfied. So Spot No. 3 has the indices [220], which is a direction belonging to the <220> family.

To index the other spots, vector addition is used. As already shown, the magnitude and direction from Spot No. 1 to Spot No. 2 is the [111] vector. The vector from Spot No. 1 to Spot No. 4 is the negative of that from Spot No. 1 to Spot No. 2. Then Spot No. 4 has the indices [111]. The vector from Spot No. 1 to Spot No. 5 is the negative of the vector from Spot No. 1 to Spot No. 3 and has the indices [220]. The vector between Spot No. 2 and Spot No. 6 is the same as that from Spot No. 1 to Spot No. 3. So, adding [220] to [111] is done by adding the components of the vectors. Consequently, the vector $h_{1}k_{2}l_{2}$, which is the sum of the [111] and [220] vectors, is $h_3 = 1 + 2 = 3$, $k_3 = 1 - 2 = -1$ and $l_3 = 1 + 0 = 1$, yielding [311] for Spot No. 6. By similar addition, the indices for Spot No. 8 are obtained. Adding [220] to [111] yields [131] for Spot No. 7. The vector from Spot No. 8 to Spot No. 1 is twice the vector from Spot No. 4 to Spot No. 1. Spot No. 8, therefore, has the indices [222]. It should be noted that spots equidistant from Spot No. 1 have the same values of $(h^2 + k^2 + l^2)$. as required by the equation for the indexing procedure (from last month's column):



Fig. 1—Electron diffraction pattern of a polycrystalline silver vapor deposit.



Fig. 2—Transmission electron micrograph of a nickel electrodeposit.

$$h^2 + k^2 + l^2 = (Ra_0K)^2$$
 (3)

This condition can be used as a check to determine whether the indexing is correct.

The plane parallel to the surface of the specimen in the area selected by the aperture is the one of greatest practical interest. As previously discussed, this plane has the same Miller indices as the direction or vector perpendicular to it. This vector is perpendicular to all directions shown in Fig. 1. It is, therefore, the



Fig. 3—Electron diffraction pattern of a single grain in a nickel electrodeposit.

cross product of any two non-parallel vectors in the diffraction pattern and is, for example, the cross product of [111] and [220]. The cross product $[h_3k_3l_3]$ of two vectors $[h_1k_1l_1]$ and $[h_2k_2l_2]$ is given by $h_3 = k_1l_2 - l_1k_2$, $k_3 = l_1h_2 - h_1l_2$ and $l_3 = h_1k_2 - k_1h_2$. The cross product of [220] and [111] is, therefore, [224]. A <224> direction is parallel to a <112> direction. Accordingly, the plane perpendicular to the incident electron beam belonged to the {112} family. This plane produced the pattern of Fig. 1.

To determine grain size unambiguously, the field-limiting aperture is placed around an area such as that marked A in Fig. 2. This aperture, located below the specimen in the TEM, determines the area from which the electron beams form the diffraction pattern. If the diffraction pattern is one of a single grain, such as shown in Fig. 2, the area within the aperture is indeed a single grain. By placing the aperture around a specific area in the image of the specimen, it can be determined whether it is a single grain. The size of the grains is, therefore, the area that vielded the diffraction pattern like Fig. 2. If twins are present in a grain, extra spots may appear in the diffraction pattern.

From the indexed diffraction pattern, it is possible to determine the directions of various elements of the structure of the specimen. For example, the direction of the twins seen in the area marked **B** would be the same as that of the vector in the diffraction pattern from that area parallel to it. The twins in Area **B** were found to be parallel to a <220> vector in the pattern. The direction of the twins was then <110>. In some

microscopes, the image rotates, depending on the magnification. Calibration of this rotation between the diffraction pattern and the images as a function of magnification is usually provided with each TEM. This rotation must be taken into account when determining the direction of structural elements from vectors in the diffraction pattern.

A diffraction pattern of an area containing a number of grains is shown in Fig. 3. The pattern consists of rings. According to Eq. (3) all planes having the same value of the sum of the squares of the indices, have the same value of **R**. Conse-

quently, the electron beams diffracted by planes in the variously oriented grains, having the same sum of the squares of the Miller indices, form a ring. In some rings, as seen in Fig. 3, the spots resulting from the various diffracted electron beams are recognizable. The constant \mathbf{K} in Eq. (3) can be determined from a diffraction pattern, such as shown in Fig. 3. This figure is a diffraction pattern of a polycrystalline silver deposit. Because silver has face-centered cubic crystal structure, the ring having the smallest radius was caused by planes having the smallest sum of the squares of the Miller indices that are either all odd or all even. These indices are those of the {111} family. By substituting 3 for $(h^2 + k^2 + l^2)$ [because {111} is the smallest set of indices that are all odd or all even] and the radius of the smallest ring and the value of a_0 of silver in Eq. (3), the value of **K** can be calculated. P&SF

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