Image and Diffraction Pattern Rotations in the TEM

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Introduction

When electrons pass through the electromagnetic lenses in a transmission electron microscope (TEM), they follow a spiral path that results in image rotation. In many TEMs, the image or diffraction pattern that appears at the final imaging plane has therefore suffered a significant rotation compared to the actual specimen. The extent of the rotation is equal to the sum of the contributions from each lens. In some recent instruments an extra lens is built into the column to compensate for these rotations. In the case of a scanning TEM (STEM), where the image is created by scanning a focused beam on the specimen, the orientation of the image to the specimen is fixed but can be controlled electronically by the computer processor.

The importance of the lens rotations was first realized from materials science studies of second phases in metallic alloys, where it was necessary to establish the morphology (for example, needle- or plate-like) and orientation of precipitates relative to a matrix phase or to establish the orientation of phase boundaries and dislocation arrays. Later work on the character of dislocation loops led to an appreciation of the importance of a 180° inversion that could occur between the final image and a corresponding diffraction pattern. Some other applications of TEM where image or diffraction pattern rotations have proved important are: (a) making stereo pairs by tilting the specimen to observe the three-dimensional distribution of image features, where the orientation of the tilt axis as applied to the image is needed, (b) tilting a specimen in diffraction mode so as to bring it into a specific crystallographic orientation, and (c) orienting the specimen relative to an EDX detector to avoid having the X-ray path blocked by other features in the microstructure, for example, thick ledges, which can be particularly important for focused ion-beam (FIB) specimens. An excellent account of most aspects of analytical TEM has been given in the book by Williams and Carter [1], and diffraction is particularly well covered by Champness [2].

The traditional way of presenting the lens-induced rotation between the image and diffraction pattern has been in the form of tables or graphs, for example, showing the rotation necessary to correct for the difference between the image at varying magnifications and a diffraction pattern. However, this becomes messy when diffraction work is carried out at various camera lengths, which is often the case using convergent-beam diffraction and when imaging is carried out over a range of magnifications.

The purpose of this note is to draw attention to a simple technique for displaying all that is needed to correct for image or diffraction pattern rotations under normal operating conditions of the TEM using a circular graphical plot. The concept for this type of display is attributed to Dr. E.E. Laufer, previously with the Materials Technology Laboratories. Neither of us has been able to locate the reference, so we believe it may have been published as a Philips Technical Report, which now seems to be unavailable.

Calibration Diagram

Figure 1 shows the results of a full rotation calibration for different imaging modes and varying diffraction pattern camera lengths relative to the position of the rod-type specimen holder in a Philips EM 400T microscope. Note that although this calibration is typical of this model, accurate rotation values will vary from one machine to another. The location of the specimen holder in real space is indicated by the black line at 45° with an arrow head, the tip of which can be considered to be located at the optical axis of the microscope column. The specimen rod axis corresponds to the principal tilt axis (T1) and

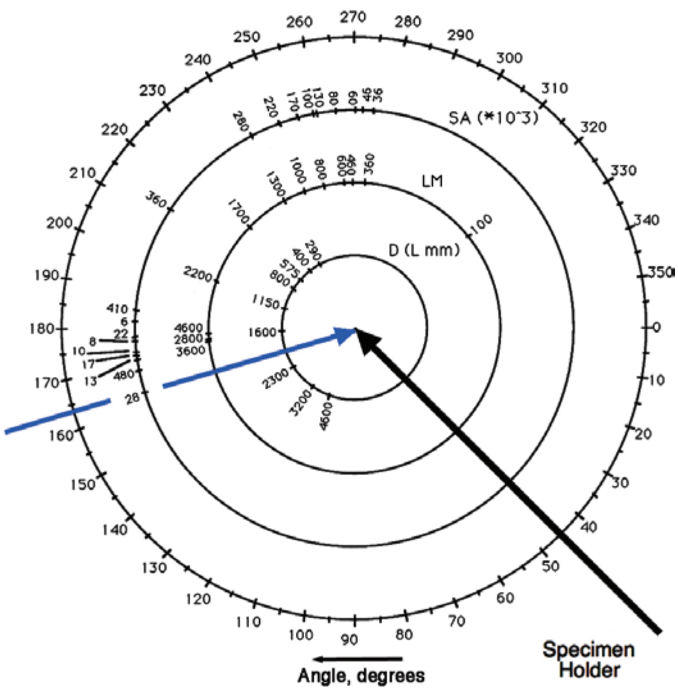

Figure 1: Image and diffraction pattern rotation relative to the side-entry, rod-type specimen holder for a Philips EM400T electron microscope, shown in the form of a circular plot of the type proposed by E.E. Laufer. The blue line indicates the orientation of the specimen holder at 28,000×.

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one of the orthogonal directions for moving the specimen with the translation controls. The virtual orientation of the holder in the images or diffraction patterns is then obtained by rotating this line to the positions ("ticks") indicated on the circles. The blue arrow shown in Figure 1 illustrates the lens-induced rotation of the image at a magnification of 28,000×, which will be used in the examples given below.

The many operating conditions of the microscope could, in principle, be arranged on just one circle, but the resulting overlap and close proximity of some of the data would make it difficult to use. On the example shown, three circles were chosen. The inner circle represents rotations of the diffraction patterns, and the other two show results for the low-magnification and high-magnification (selected area, "SA") ranges respectively. The outer circle shows the rotation angles, clockwise from 0°, which was arbitrarily defined by the horizontal in the image plane. The orientation of the specimen holder in the column then falls at +45°. The corrections for the lens-induced rotations can be made by positioning images and DPs directly on the calibration diagram itself at the appropriate magnifications or camera lengths.

Calibration Procedure

The measurements needed to produce this type of plot are simple and have been well documented in the literature [1, 2]. With a side-entry holder, some benchmark rotations of the image relative to the specimen can be established by taking a double exposure before and after translating the holder rod along its axis at a number of convenient (that is, not too high) magnifications. The exposures should show movement of a well-defined feature in the microstructure of any suitable specimen, for example, Figure 2. Take care to avoid backlash effects by translating the holder smoothly along one direction only for the recorded images. This also establishes the *sense* of the rotation, thus revealing possible image inversions. The relative rotations over a large range of magnifications in image mode can be measured using a sample with a straight edge such as a crystal of α-MoO_3, which can also be used to establish the rotation of diffraction patterns at various camera lengths relative to the images. It is worth noting that the long edge of the pseudo-orthorhombic crystal is parallel to the [001] direction [1, 2], not the [100] direction as given in some early textbooks. For recording the diffraction patterns, selected-

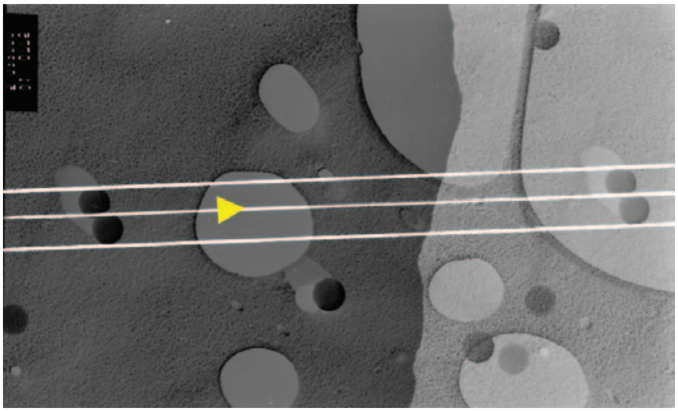

Figure 2: Double-exposure images of a carbon film containing latex balls and gold islands to reveal the specimen rod axis in an EM4OOT microscope, showing the direction of translation of the rod at a specific magnification.

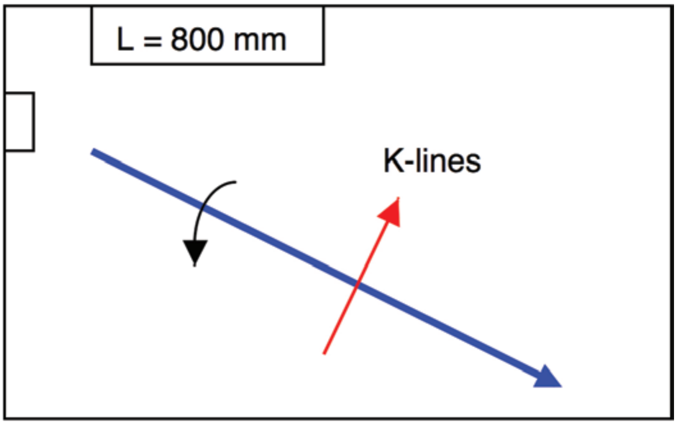

Figure 3: Showing (schematically) the direction of movement of intersecting Kikuchi lines at a camera length of 800 mm, as a result of a clockwise tilt about the principal rod axis (compare to Figure 1).

area diffraction (SAD) is preferable, with the condenser lens strongly over-focused to give sharp spots for accurate measurements. Cross-sectional specimens of semiconductors containing planar epitaxial layers are also useful for checking DP versus image rotations. One such specimen is marketed as the "Mag-i-cal" standard [3], which can also be used for magnification calibration.

The sense of the rotation is equally important for diffraction patterns and this can be easily checked by observing the direction of motion of the point of intersection of two Kikuchi (K) lines in a convergent beam pattern on tilting the sample about the rod axis (Figure 3) and repeating the procedure for the second, perpendicular tilt axis. Again, backlash effects are likely and must be avoided. A very useful technique for confirmation purposes is to note the orientation of a specimen edge in a defocused diffraction pattern [4].

Use of the Calibration Diagram

I have used Laufer's rotation plots in materials science work on a great variety of materials, in both a Philips EM400T and an FEI CM20FEG, and found them to be an invaluable aid to microanalysis. The following diagrams are derived from Figure 1 to illustrate how such a diagram can be used. Note that these are schematic, so that the angles used may not be exact compared to those in Figure 1. The *correction* needed to rotate the diffraction pattern relative to the image is the *inverse* of the actual rotation (see Figure 4). It is therefore easy to carry out experiments requiring the correlation of image features, such as precipitates or defects, with diffraction patterns using any combination of magnifications and camera lengths.

The result of tilting the specimen about the rod axis (the principal tilt axis) to create a stereo pair is illustrated in Figure 5a, where the blue line represents the orientation of the tilt axis in the image. The stereo pair images must therefore be oriented as indicated in Figure 5b. It is rarely necessary to distinguish top and bottom surfaces of the specimen in stereo images, but if that is required it is important to arrange the two images so that the one on the right has been tilted in an anticlockwise sense relative to the left-hand image (based on viewing the specimen rod in the direction toward the column center).

A more difficult operation is to orient a crystalline specimen to obtain stereo images of features showing strong diffraction

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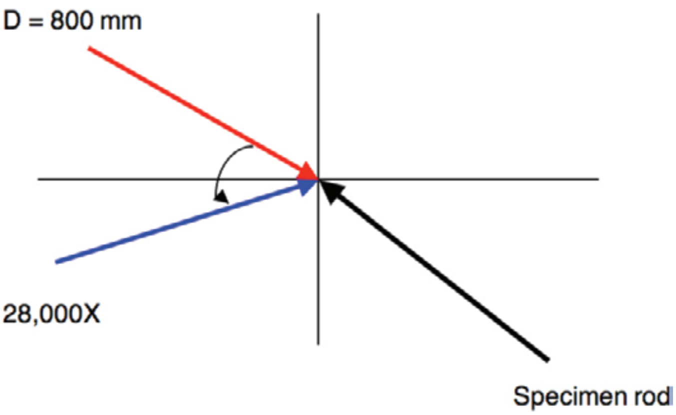

Figure 4: An example illustrating the rotation required to *compensate* for the rotation of a diffraction pattern, L = 800 mm, relative to an image at 28,000×.

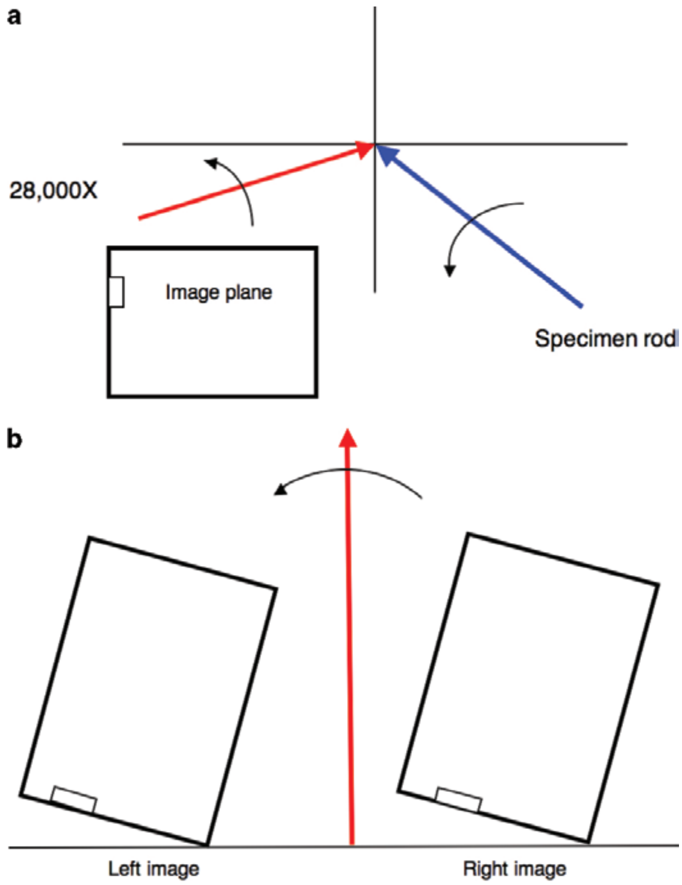

Figure 5: (a) Tilting the specimen about the principal rod axis to create a stereo pair, showing the sense of tilt in images at 28,000× and (b) the reorientation needed for the images in a stereo viewer to correctly retain top and bottom surfaces in the resulting 3-D image.

contrast as in the case of dislocations in crystals. Here, it is necessary to maintain the same operating reflection (**g**-vector) for each image of the stereo pair by tracking along a pair of Kikuchi lines (K-lines) in diffraction mode. The Laufer plot is invaluable for such work, which requires frequent switching between imaging and diffraction mode. It is therefore useful to be able to anticipate the direction of movement of the K-lines while observing the specimen in *imaging* mode, assisting with the difficult task of maintaining a constant area of the specimen in the field of view. The tilt axis needed for viewing the stereo pairs in this particular case is then simply the direction of the operating reflection (that is, perpendicular to the K-line pair). Knowing which way the K-lines will move for a given tilting operation is also useful for orienting the specimen so as to excite a single strong reflection for dislocation Burgers vector analysis in crystals.

The recognition of possible 180° inversions is intrinsic in the Laufer plot and becomes crucially important for identifying the character (vacancy or interstitial) of irradiation-induced dislocation loops [5]. For such work there are two possible outcomes of the imaging experiments and only one of them is correct!

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