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Simple Method to Determine the Rotation Between a TEM Image and Diffraction Pattern

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Accurate knowledge of the diffraction pattern orientation with respect to the corresponding TEM image is essential for transferring crystallographic information between the two. This is important for identifying crystallite orientation, for indexing dislocations, and for determining polarity from convergent beam electron diffraction (CBED) patterns. Correct identification of sample polarity has recently become more critical due to the growing technological importance of noncentrosymmetric materials. In wide bandgap nitrides and ferroelectric oxides, polarity causes internal electric fields and affects material growth, dopant incorporation, and morphology as well as electrical and optical properties. Accurate determination of polarity is, therefore, crucial for understanding the growth and for developing applications of these materials.

The traditional method to determine the rotation between the diffraction pattern and the image uses double exposures of a standard sample (typically α-MoO_3) and its diffraction pattern and also requires knowledge of the lens system to account for 180° inversions as lens are turned on or off.[1] In most modern microscopes these inversions are electronically eliminated and frequently the angle between the diffraction pattern and image is also corrected for. However, due to the symmetry of zone axis diffraction patterns, even of noncentrosymmetric materials, this correction can be off by 180°. The standard procedure to determine the absolute orientation of the diffraction pattern requires a sample with a platelet (or nanowire) shape and under focusing the diffraction pattern by decreasing the current through the diffraction lens so that an image of the sample is visible in each diffraction spot.[2,3] A critical aspect of this is that the pattern be under focused; if the diffraction lens is over focused the sample image in the spots is inverted and the measured orientation will be 180° off.

We present here a novel method to determine the correct orientation of the diffraction pattern relative to the sample image. The technique utilizes the diffraction pattern produced in the image plane when the beam is focused in the object plane and the sample is shifted above the object plane. It does not rely on the sample shape and can be used on any sample with a large enough crystalline area to obtain a diffraction pattern. The method is illustrated in Fig. 1 with images of a GaN lamella.

A CBED pattern, taken with the sample oriented along the [1-100] zone axis, is shown in Fig. 1 a). When the sample was tilted slightly along a <0001> direction the CBED pattern becomes asymmetric as shown in Fig. 1 b). The images in Fig. 1 a) and b) were both taken in diffraction mode with the electron beam condensed in the image plane. Switching to image mode and raising the sample above the object plane produced the image in Fig. 1 c). When the sample is positioned above the object plane, the orientation of the diffraction pattern corresponds with the orientation of the sample image. This pattern can then be compared with the diffraction pattern in the back focal plane of the objective lens, Fig. 1 b), to determine the rotation between the image and back focal planes. Comparing Fig. 1 b) and c) it is evident that there is a 180° rotation between the diffraction pattern and the image plane in the

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microscope used. The importance of raising the sample, rather than lowering it, is illustrated by Fig 1 d) which was taken with the sample positioned below the object plane, resulting in a 180 ^{o} inversion of the diffraction pattern [4].

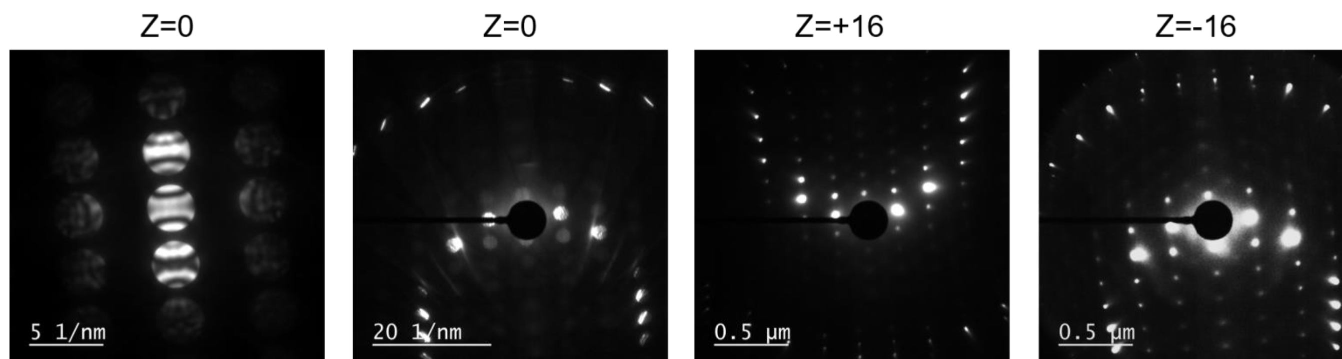

Figure 1. Images of a GaN lamella. a) CBED pattern taken along the [1-100] zone axis, with a 30 μm condenser lens aperture (CLA) and a 20 cm camera length. b) An asymmetric CBED pattern taken after the lamella was tilted slightly off the [1-100] zone axis along a <0001> direction, with a 30 μm CLA and an 8 cm camera length. c) The corresponding pattern taken under the same conditions as b), but in image mode at 5 kX with the sample shifted 16 μm above the object plane. The 180 ^{o} rotation evident between the diffraction mode and image mode patterns is the instrument induced rotation for the microscope used. d) Pattern taken under the same conditions as c) but with the sample shifted 16 um below the object plane.

References:

[1] J.W. Edington, “1 The Operation and Calibration of the Electron Microscope (Philips Technical Library)”, (The MacMillan Press, London, U.K., 1974) pp. 23-24.
[2] M. De Graef, “Introduction to Conventional Transmission Electron Microscopy”, Cambridge Solid State Science Series (Cambridge University Press, Cambridge, U.K., 2003); pp. 273–275.
[3] D.B. Williams and C.B. Carter, “Transmission Electron Microscopy Part 1: Basics”, (Springer Science1 Business Media, New York, New York, 2009); pp. 167–168.
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