Insights on Diffraction

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This article presents ideas on some topics related to electron diffraction in the TEM. These are in regard to topics that I have come to think of as standard parts of what it means to do microscopy. However, they represent insights that not all users share (or even agree with, maybe).[1]

Kikuchi lines

Kikuchi lines are of great use in orienting a sample. Unfortunately, in modern microscopes, Kikuchi lines are not seen in selected-area diffraction (SAD). This is because immersion lenses send parallel electrons, from different parts of the sample (like the Kikuchi lines from a flat specimen), to different places in the diffraction pattern. Thus Kikuchi lines are blurred and generally not useful whenever, as in SAD patterns, a large area of the sample contributes to the diffraction pattern. This has been known for a long time now,[2] but I find that many people are still unaware of it. In convergent-beam (CBED) patterns, this problem does not arise and the Kikuchi lines are clear and strong. Because a CBED pattern is obtained from only a small region of the specimen, the immersion lens effect is avoided, moreover the blurring of the lines due to sample buckling is also avoided. The presence of high-contrast, sharp Kikuchi lines is one of the strongest arguments for using CBED as the normal form of diffraction and using SAD only for special applications (unfortunately many people still do the reverse).

Kikuchi lines are sharper and have more contrast in CBED than in SAD for the reasons given above. They are also brighter. There is a different reason for this. Consider two diffraction patterns, one obtained by CBED and one by SAD, with the same total number of electrons in each case. In the two patterns the number of diffusely scattered electrons will be the same and they are distributed over the same area, therefore the intensity in the diffuse background will be the same. However, in SAD the elastically-scattered electrons in the direct beam (and in the Bragg peaks) are all at the same place in the diffraction pattern, giving a sharp intense peak, whereas in the CBED pattern the same number of elastically-scattered electrons is distributed across discs, which are therefore of lower intensity. As a result, the ratio (intensity in the Bragg peaks) / (intensity in the diffuse background) is much higher for SAD than for CBED. Thus CBED is much better when looking at Kikuchi lines.

Setting the Deviation Parameter

An important part of experimental electron diffraction is the setting of the deviation parameter. Two different circumstances arise: Setting the pattern exactly to a zone axis and setting a chosen deviation under two-beam conditions. To set up at a zone axis it is normal to use the mechanical tilts to get fairly close to the zone axis, but then to use the beam tilts for the fine alignment. In the case of two-beam conditions, it is generally necessary to do all the tilting mechanically. Zone-axis orientations are usually sought to obtain information from the diffraction pattern itself, for example, to get the symmetry or the lattice parameters. A high-quality image of the sample is not needed at the zone-axis orientation. Because an image is not required, the beam can be misaligned (by tilting the beam to get the diffraction pattern centered

on the zone axis): the diffraction pattern is not affected by the misalignment. On the other hand, in the case of two-beam conditions, the information sought comes from the image as much as (or in many cases more than) from the diffraction pattern. In the two-beam case, it is necessary to keep the microscope accurately aligned, in order to get a good image. Thus, it is essential to do all the tilting mechanically. Fortunately, since tilt about only one axis is required, this is not as difficult as it would be in the case of tilting to a zone axis.

Early textbooks are written as if the sample is flat and the deviation parameter has the same value across the whole field of view. The immersion lens problem, mentioned above, means that, even if the sample is perfectly flat, the deviation parameter will change across the image of a sample. This is made evident by the fact that bend contours are always visible in images in modern microscopes. The standard method of setting the deviation parameter (using the Kikuchi lines) is of limited use under these circumstances. The position of the Kikuchi lines is only relevant to one place in the image. It may make more sense to set the deviation parameter by looking at the image and watching the bend contour as the sample is tilted.

Selected-area diffraction and convergent-beam diffraction

Convergent beam diffraction should be considered the standard form of diffraction in the TEM. It should be the method used routinely. Does this mean that there is no reason ever to use SAD? Despite my views on the superiority of CBED, there are a couple of things (only a couple) that SAD does better than CBED. The first of these is the formation of ring patterns from polycrystalline samples or from amorphous materials. In CBED,

electrons elastically scattered by a particular diffraction vector appear in the pattern as a disc. When the scattering is into well-separated beams, this is just fine. But when the diffraction is distributed continuously, as in the case of a ring pattern, the effect of the convergent beam is to blur the pattern. The same argument can be used to show that SAD reveals more clearly the distribution of the diffuse scatter between the main reflections, when it is not uniform.[3]

The second strength of SAD patterns is that they reveal the presence of weak reflections with much greater sensitivity than CBED patterns. This is because the ratio of the intensity of an elastic peak to the inelastic background is much greater in SAD (see above). Thus weak peaks, for example from superlattices, can easily be overlooked in CBED, but will be quite clear in SAD.[4]
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References

1. D. B. Williams and C. B. Carter, *Transmission Electron Microscopy*, Plenum (New York 1996)

J. C. H. Spence and J. M. Zuo, *Electron Microdiffraction*, Plenum (New York 1992)

J. M. Cowley (Ed.) *Electron Diffraction Techniques* (2 volumes) Oxford (Oxford 1992)

2. K. K. Christenson and J. A. Eades, *Skew Thoughts on Parallelism* Ultramicroscopy 26 (1988) 113-132

3. J.M. Cowley, *Diffraction Physics*, North Holland (Amsterdam 1981) Chapter 12, 249-271

J. Gjonnes, *Disorder and defect scattering, thermal diffuse scattering, and amorphous material*, in J. M. Cowley (Ed.) *Electron Diffraction Techniques* Oxford (1992) Volume 2, 223-259

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