EDUCATIONAL ARTICLE: WHEN TO USE SELECTED-AREA DIFFRACTION AND WHEN TO USE CONVERGENT-BEAM DIFFRACTION

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ABSTRACT
Selected area diffraction and convergent-beam diffraction are the two most generally used diffraction methods in the transmission electron microscope. This tutorial note discusses the strengths and weaknesses of each method. Convergent-beam electron diffraction is the most useful method and should be generally used. Selected area diffraction should be used only in certain specific cases which are listed.

Keywords: Tutorial, TEM, Diffraction, Convergent-beam, Selected-area.

RESUMEN
En el microscopio electrónico de transmisión hay dos técnicas principales para obtener diagramas de difracción: difracción de área selecta y difracción de haz convergente. En este trabajo didáctico se presentan las ventajas y desventajas de ambos métodos. La difracción de haz convergente es la técnica preferida en la gran mayoría de los casos y debe utilizarse casi siempre. Se presenta una lista de los pocos casos específicos en que esta recomendado el uso de difracción de área selecta.

Palabras Clave: tutorial, MET, Difracción, Haz–convergente, Área–selecta.

INTRODUCTION

There are two basic techniques for obtaining diffraction patterns in the transmission electron microscope. They are selected-area diffraction and convergent-beam diffraction [1]. This tutorial note discusses when it is appropriate – and best – to use each of these techniques. There are other diffraction techniques in the TEM but they are of more specialist and specific application and are not considered in this paper. These special techniques include precession [1], large-angle convergent-beam diffraction (LACBED) [2] and versions of microdiffraction and nanodiffraction [3] that use a parallel beam for illumination.

These techniques are discussed in several books [1-7]. The most accessible general introduction to all of these methods is the book by Williams and Carter [1].

This note assumes that the reader is familiar with both selected-area and convergent-beam diffraction. If this is not the case the reader is directed to the books mentioned above. We will limit ourselves here to a brief reminder of how each technique is implemented.

In all forms of diffraction in the transmission electron microscope, the aim is to obtain a diffraction pattern from a specific area of the sample. It is important that the area of sample that gives rise to the pattern can be clearly defined and identified on an image of the sample. Different techniques achieve this in different ways.

Selected-area diffraction

In selected-area diffraction, a large area of the sample is illuminated with the electron beam, but not all of the illuminated area contributes to the pattern. The diffracting area is limited by an aperture following the sample. This aperture is in a plane conjugate to the sample, and only that area within the aperture contributes to the diffraction pattern.

The practical sequence of operations to obtain the pattern is:

1. Set up starting conditions: The sample in focus, preferably at the eucentric height.
2. Spread the illumination to be approximately parallel.
3. Insert the area-selecting aperture.
4. Go to diffraction mode.
5. Adjust the diffraction focus as needed.

Convergent-beam diffraction.

In convergent-beam diffraction, the area of sample that contributes to the pattern is limited by ensuring that the incident electron beam strikes only the chosen area of the sample.

The practical sequence of operations to obtain the pattern is:

1. Set up starting conditions: The sample in focus, preferably at the eucentric height.
2. Choose an appropriate spot size.
3. Use the condenser lens control to focus the illumination onto the sample in the form of the smallest possible spot or "probe".
4. Go to diffraction mode.
5. Adjust the diffraction focus as needed.

Initial comparison of the two methods.

The first and most striking difference between the two methods is in the character of the illumination. In the SAD case, the illumination of the sample is with a parallel (or approximately parallel) beam. In contrast, in the case of CBED, the beam is focused onto the sample. The electrons are incident on the sample in all the directions within a cone defined by the condenser aperture.

The result of this is that, if there is no sample present, a SAD pattern consists of a single bright spot. In the case of CBED in the absence of a specimen, the pattern obtained, is a single disc uniformly illuminated, and having a diameter determined by the size of the condenser aperture. In each case we refer to this beam as the direct beam or the bright-field beam. When a sample is introduced, two kinds of interaction between the sample and the beam occur: elastic scattering and inelastic scattering. If the sample is crystalline, as we shall suppose for the moment, the elastic scattering takes the form of Bragg reflection and electrons are diffracted into well defined directions at fixed angles with respect to the incident electrons. Thus in SAD, the diffraction pattern consists of an array of sharp spots, each spot displaced from the direct beam by a vector determined by the crystal structure. In the case of CBED, each point in the bright-field disc acts as a source. An array of spots (similar to the array in the SAD case) is associated with each point in the disc. Thus the diffraction pattern consists of an array of discs. The discs are all of the same size as the direct-beam disc.

Inelastic and diffuse scattering.

Electrons which are scattered in the sample by inelastic or diffuse scattering processes are not constrained – as is the case for Bragg reflection – to fall in positions related specifically to the direction of the incident beam. They can be scattered into all directions as the name "diffuse" implies.

Advantages and disadvantages.

1. CBED patterns can be (and generally are) obtained from areas of the sample which are much smaller than is possible for SAD. It is not true that the area contributing to a SAD pattern is determined solely by the size of the area-selecting aperture. There is also a fundamental limit related to the spherical aberration coefficient of the objective lens [1–8]. Although this limit will be a function both of the properties of the objective lens and of the camera length (or more precisely, the range of diffraction angles included in the pattern), it is often reasonably assumed that an

area of about 1 μm in diameter is the smallest that can be studied with SAD. In CBED, the diffraction pattern can be obtained conveniently from an area down to 40 nm or less in a microscope with a thermionic source and down to less than a nm with a field-emission gun.

2. It is hard or impossible to obtain useful information from the intensities of the spots in a SAD pattern. For example, the intensities of the spots in SAD should not be used as an indication of sample symmetry. This is because the diffraction pattern comes from a substantial area of sample. Within that area, there will almost always be variation in the sample thickness, in the sample orientation and in the crystal perfection. Thus the pattern obtained is a complex average over diffraction patterns from many different conditions. Although the positions of the spots have a clear meaning, the intensities of the spots are generally of little value. In contrast, since the area that generates the diffraction pattern is so small, in CBED, the pattern corresponds to a particular place, a particular thickness and (if the operator chooses carefully) a region free from defects. Therefore, the intensities in CBED patterns are meaningful and can be used to obtain information about the sample (see item 7, below).
3. If the sample is polycrystalline, SAD will produce a ring pattern with sharp rings. CBED will produce a pattern of rings in which each ring is an annulus having a width equal to the diameter of the direct beam. Thus for this application SAD is to be preferred, since the rings in a convergent-beam pattern from a polycrystalline region will be ill defined and often overlap. Similarly, diffraction patterns from amorphous samples should be taken by SAD. CBED patterns from amorphous samples will be unnecessarily blurred.
4. Sometimes it is desirable to obtain diffraction patterns from a region of the sample which contains more than one grain. For example, if we wish to determine the relative orientation between two grains (to answer the question: are these twins, perhaps), it may be easiest to take a diffraction pattern including both grains. Such patterns can become quite complex, with many reflections close together. In such a case a SAD pattern with its sharp spots may be preferred.
5. In most samples, the diffuse scattering between the Bragg peaks is structureless (simply dropping in intensity as you move away from the elastic peaks). However, in some samples with particular kinds of disorder, there is structure in the diffuse scatter which can be informative about what is going on in the sample [1-7,9]. In CBED patterns, any such structure in the diffuse scatter will be blurred. This structure in the diffuse scatter is – like the Bragg peaks – tied to the position of the direct beam. Therefore, in CBED, where there are many incident beam directions, the structure is spread out.
6. In CBED patterns the diffuse scatter between the reflections is brighter relative to the Bragg discs than is the case for SAD patterns. This can be seen as follows: In a SAD pattern, there will be a certain ratio between the intensity of a Bragg peak and the intensity of the surrounding diffuse background. A CBED pattern can be thought of as a superposition of SAD patterns, one for each position in the disc. Each of these superimposed patterns has its own diffuse background and they all get added together. The intensity in the Bragg discs does not get added together, however, since the peaks fall in different places not on top of each other. The result of this is that weak reflections are more readily seen in SAD than in CBED. Reflections that are very weak – the kind of reflections that arise from superlattice ordering, for example – may be completely invisible in CBED patterns.
7. The variation in intensity within the discs of a CBED pattern carries a lot of information and there is no

equivalent for SAD patterns. The intensity within the discs can be used to determine:

- Crystal symmetry [1-7,10,11]
- Changes in lattice parameter and strain [1-7,12]
- The identification of a phase [1-7,13,14]
- The thickness of a crystalline sample [1-7,15]

All of these are now well established techniques and can be considered standard. There are also more advanced applications of CBED with no analogue for SAD diffraction [1].

8. Kikuchi lines appear in the diffuse scatter in electron diffraction patterns. The Kikuchi lines are very valuable in determining the orientation of the sample and in tilting to desired orientations [1-7]. Kikuchi lines are far more clearly visible in CBED patterns than in SAD patterns [16]. In fact, Kikuchi patterns are hardly visible at all in SAD patterns. This is not the result of scattering physics. It is the result of the design of transmission electron microscopes for the last three decades. In order to achieve high resolution performance – and, indeed, to get the strong condenser focusing needed for both CBED and energy-dispersive x-ray spectroscopy (EDS) – virtually all TEMs made during this period have been made with immersion lenses. When the objective lens is an immersion lens, the sample sits within the magnetic field of the lens. The result of this is that the electrons follow helical paths, which – in turn – has the result that Kikuchi lines from different parts of the sample fall in different places in the diffraction pattern [16]. The larger the area of the sample contributing to the diffraction pattern, the more the Kikuchi lines will be blurred, even if the sample is perfectly flat. Hence, CBED is greatly to be preferred for tilting and aligning the sample – for "navigating in reciprocal space".

SUMMARY

Convergent-bean diffraction overall is easier than selected-area diffraction and provides, in general, much more useful information about the sample. It should therefore be used as the standard method of diffraction in the TEM. Only rarely do circumstances arise when it is preferable to use SAD.

If you want to:

- check for the presence of weak reflections,
- make a ring pattern from a polycrystalline region,
- get diffraction from a region with more than one grain,
- obtain diffraction from amorphous samples,
- look for structure in the diffuse scattering,

then use selected-area diffraction.

In all other cases, use convergent-beam diffraction.

Use convergent-beam diffraction as your main method, your normal method, of doing diffraction in the transmission electron microscope.

Since it is better to use convergent-beam diffraction than selected area diffraction in the great majority of cases – and since it is also easier – one can legitimately ask how it is that most microscopists still use SAD as their primary diffraction mode. The answer surely has to be historical accident. Until the mid-1970s, the microscopes in use could not produce convergent-beam diffraction patterns. The condenser lens was too far from the sample and it was not possible to produce a large enough convergence angle in the illumination. That changed with the advent of immersion lenses and, at that point, everyone should have changed from SAD to CBED. However, the world was full of people who knew how to use SAD and almost no one who knew how to use CBED. Those who taught microscopists continued to teach SAD, it was what they knew. It is time for a change.

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