Use of High-Symmetry Zone Axes in Electron Diffraction in Determining Crystal Point and Space Groups

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(Received 21 September 1982; accepted 13 January 1983)

Abstract

A sequence of steps is given for making use of the information available on convergent-beam electron diffraction patterns from high-symmetry zone axes for crystal point- and space-group determination.

1. Introduction

Over a number of years of research, involving the solution of crystal space groups by convergent-beam electron diffraction, we have come to the view that the most effective way of proceeding is by the location and exploitation of zone axes of high symmetry when they exist. There are two main ideas behind this conclusion and a number of subsidiary ones. The main reasons are those of efficiency and reliability. Efficiency is not simply a matter of convenience, it is often the deciding factor whether the job can be completed or not. High-symmetry axes can be rapidly and systematically reached with a little experience (see § 2). Once located, seventeen out of the thirty two possible point groups can be identified from a single high-quality exposure. The remaining fifteen point groups may be decided by a further one or two exposures at the same zone axis. If the diffraction is degrading (as is often the case) by radiation damage or contamination then it is vital to obtain the necessary information as quickly as possible. Even in the absence of such deterioration it is advisable to work from a single zone axis when the crystal of interest is of small dimensions and embedded in a complex microstructure of other crystals. This ensures that the information is obtained from the chosen crystal and minimizes the possibility that the probe has drifted to an adjoining area.

Convergent-beam electron diffraction (CBED) is extremely sensitive to all sorts of subtle effects which are often overlooked in conventional electron diffraction. Weak strains from internal defects such as dislocations or surface contaminants can seriously affect the symmetry of the diffraction patterns. Point defects can affect the large-angle scattering. For this reason it is no great achievement to produce a pattern of low symmetry! On the other hand, having produced a pattern which convincingly reveals a high symmetry it is unlikely to be the result of artefacts. By concentrating on high-symmetry zone axes the chance of spurious results, ever present, is greatly reduced.

Amongst the subsidiary points, there is always a chance that the high-symmetry zone axis occurs for a specimen on which the beam is incident at approximately normal incidence. If, on the other hand, one relies on the contribution of information from different zone axes of a crystal one has to be aware of the more likely influence of specimen inclination.

In this paper we attempt to give a detailed summary of how to obtain and use high-symmetry zone axes for crystal-structure determination. This summary will be given as a sequence of steps to be taken.

2. Point-group determination

2.1. Systematic method for locating high-symmetry axes to give crystal point groups

(a) Set the height on a eucentric goniometer and sweep through the whole range of eucentric tilt (±60° may be possible). Tilt/rotate holders are generally better than double-tilt holders for this work. There are several reasons for this preference.

(i) The holders are thinner and so permit a larger tilt angle without cut-off.

(ii) Less height adjustment is generally required when using the rotation axis than when using the second tilt axis.

(iii) Specimens may be semi-permanently fixed in removable cups greatly reducing the chance of damage (this attractive feature may depend on the design of the holder).

For fine-grain polycrystalline material or small particles it is easiest to perform this tilting operation in the shadow-image mode (second condenser lens out of focus) so that the grain of interest and its diffraction pattern are simultaneously visible. This allows the operator to re-position the region of interest in the electron beam if it shifts during tilting.

(b) Examine the convergent-beam diffraction pattern at low camera length on the viewing screen and look for mirror lines, first in the diffuse scattering (Kikuchi-line array) and then in the diffraction discs.
Table 1. Table indicating the steps to be taken in exploiting the information in high-symmetry zone-axis patterns for crystal point-group determination

The table has been prepared on the assumption that the axis of highest symmetry can be located eventually.

<table>
<thead>
<tr>
<th>Action</th>
<th>Result</th>
<th>Action</th>
<th>Result</th>
<th>Action</th>
<th>Result</th>
<th>Conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sweep through range of eucentric tilt</td>
<td>Neither WP nor BF mirror</td>
<td>Rotate through 90° and repeat eucentric tilt</td>
<td>BF mirror without WP mirror</td>
<td>Rotate about BF mirror line</td>
<td>Additional symmetry</td>
<td>Point groups 1, 1, 3, 3. Look for evidence of a threefold axis. Do ±G dark-field experiments to test for inversion†</td>
</tr>
<tr>
<td>Neither WP nor BF mirror</td>
<td>Rotate about BF mirror line</td>
<td>Additional symmetry</td>
<td>No additional symmetry</td>
<td>WP and BF mirrors</td>
<td>No additional symmetry</td>
<td>Point groups 2, 4, 4, 6. Distinguish 4, 4 by WP and BF symmetries</td>
</tr>
<tr>
<td>BF mirror without WP mirror</td>
<td>Rotate about WP mirror line</td>
<td>Additional symmetry</td>
<td>No additional symmetry</td>
<td>WP and BF mirrors</td>
<td>No additional symmetry</td>
<td>m, 2/m, 4/m, 6/m, or 6. Distinguish by symmetry of unique axis</td>
</tr>
<tr>
<td>WP and BF mirrors</td>
<td>Rotate about WP mirror line</td>
<td>Addition symmetry</td>
<td>No additional symmetry</td>
<td>See Table 2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

†See Notes on Tables 1 and 2

Notes on Tables 1 and 2

(1) Table 1 has been prepared assuming the use of a tilt/rotate holder. Slight modifications are required in the case of a double-tilt holder.

(2) Results may have to be combined from different crystals with overlapping ranges of orientation in the case of low-symmetry crystals or holders with more limited ranges of tilt.

(3) In order to be certain of the presence of a mirror line in a pattern it may be necessary to take an exposure and examine the negative.

(4) Accurate alignment is required to examine symmetry properties. The best way to achieve this is by using the translation controls on the second condenser aperture for the final stage of alignment (≥1°).

(5) It is assumed that three-dimensional diffraction data is available. This may be in the form of fine lines crossing the zero-layer reflections (thick crystals) or else as individual reflections in one or more higher-

themselves. If no mirror lines are located, rotate the specimen through 90°, recenter the eucentric tilt axis and sweep through the range of eucentric tilt once again (or use the second tilt axis).

If no mirror lines are then located the crystal is almost certainly triclinic (see Table 1).

(c) Once a mirror line is located, use a combination of rotation (or second tilt) and tilt to move along the mirror line (rotate about the diffraction vector normal to the mirror) and look for zone axes with additional symmetry properties. Refer to Tables 1 and 2. In the event that a single whole pattern or bright-field mirror is located check to see if a high-symmetry axis exists perpendicular to the mirror, i.e. a four- or sixfold axis. If it does, one can tilt to obtain CBED results from this axis. Note that it might be helpful to have available a small computer and graph plotter so that the goniometer coordinates can be converted into points on a stereographic projection by appropriate computer routines.
Table 2. Table indicating use of axes of highest symmetry in determining crystal point groups

Entries in brackets have higher symmetry axes which occur elsewhere in the tables and would not therefore arise in an analysis based solely on the axes of highest symmetry.

<table>
<thead>
<tr>
<th>WP symmetry</th>
<th>BF symmetry</th>
<th>Diffraction group(s)</th>
<th>Comment</th>
<th>Point group(s)</th>
<th>Distinguishing test or other comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>m</td>
<td>2mm</td>
<td>ml</td>
<td>Isolated</td>
<td>mm2</td>
<td>Axis 45° away has DG 4mm which identifies the point group</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>(43m)</td>
<td>4mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Two 45° apart</td>
<td>mm2</td>
<td>4mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Two 30° apart</td>
<td>mm2</td>
<td>6mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>One 45° away from a zone axis</td>
<td>mm2</td>
<td>4mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>One 30° away from a zone axis</td>
<td>mm2</td>
<td>6mm</td>
</tr>
<tr>
<td>2</td>
<td>2mm</td>
<td>2m</td>
<td>2mm or 2mm1</td>
<td>mm2</td>
<td>HOLZ ring diameter distinguishes m3. For mm2 and mm3 the DG’s 2mm and 2mm1 have to be distinguished by ( \pm G ) DF or by internal DF symmetry*</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2mm1</td>
<td>mm2</td>
<td>4mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2mm1</td>
<td>mm2</td>
<td>6mm</td>
</tr>
<tr>
<td>3</td>
<td>3m</td>
<td>3m</td>
<td>Isolated</td>
<td>mm2</td>
<td>HOLZ ring diameter</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2mm</td>
<td>mm2</td>
<td>4mm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2mm1</td>
<td>mm2</td>
<td>6mm</td>
</tr>
<tr>
<td>3</td>
<td>6mm</td>
<td>3mm</td>
<td>3m or 6mm1</td>
<td>mm2</td>
<td>HOLZ ring diameter</td>
</tr>
<tr>
<td>4</td>
<td>4mm</td>
<td>4mm</td>
<td>4mm or 4mm1</td>
<td>mm2</td>
<td>HOLZ ring diameter</td>
</tr>
<tr>
<td>6</td>
<td>6mm</td>
<td>6mm</td>
<td>6mm or 6mm1</td>
<td>mm2</td>
<td>HOLZ ring diameter</td>
</tr>
</tbody>
</table>

*See Notes on Tables 1 and 2

order Laue zones (HOLZ's). In case of doubt whether three-dimensional diffraction is playing a significant part in the pattern symmetries it is helpful to have the facility of continuous variation of the microscope accelerating voltage. If significant pattern changes occur for 2 keV changes of the accelerating voltage it is fair to assume that three-dimensional diffraction effects are present.

(6) To study the intensities or intensity distributions of individual reflections it is necessary to use a second condenser aperture which is small enough to exclude overlap between the various orders of reflection (in HOLZ’s as well as in the zero layer).

(7) To make an accurate determination of the repeat distance along the zone axis it is helpful to have a large second condenser aperture so that considerable overlap occurs between the intensity distributions of adjoining reflections. This procedure ensures the generation of continuous rings of intensity in the HOLZ’s to which circles may be fitted. The diameter of the circles may need correction for lens distortions (Steed, 1981) and where a Laue zone consists of a multiplet of continuous lines it is recommended that one takes the innermost (smallest diameter) of these (Steed, 1980). A low-camera-length setting and large angular view are essential for this step. The spacing \( H \) of reciprocal-lattice planes along the direction of incidence is given by the equation

\[
G = \sqrt{2KH},
\]

where \( K \) is the electron wave number and \( G \) is the radius of the HOLZ. Note that, strictly speaking, HOLZ radii can only be used as a guide to point groups and the conclusion reached should be tested by tilting to the appropriate axes and examining the symmetry.
Table 3. Use of a single principal zone-axis pattern to determine the crystal point group

<table>
<thead>
<tr>
<th>WP</th>
<th>BF</th>
<th>Diffraction groups</th>
<th>Possible point groups and zone axes</th>
<th>Distinguishing tests</th>
<th>Number of point groups determined</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>m</td>
<td>mR</td>
<td>16 possible point groups</td>
<td>If HOLZ diffraction reveals a monoclinic cell then its point group is 2[w0w]</td>
<td>1</td>
</tr>
<tr>
<td>m</td>
<td>2</td>
<td>1R</td>
<td>m[010] or 3m(1120)</td>
<td>Use HOLZ to distinguish monoclinic from trigonal 2mm</td>
<td>2</td>
</tr>
<tr>
<td>2mm</td>
<td>m1R</td>
<td>m[010], 4m(010) or</td>
<td>mm2(100), 4mm(010) or 2mm</td>
<td>If HOLZ diffraction reveals an orthorhombic cell then mm2(100) and if a cubic cell then 3m(110).</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>2mm</td>
<td>2mm</td>
<td>(110), 6mm</td>
<td>If HOLZ reflections indicate tetragonal (100) then 4mm and if hexagonal (1100) then 6mm</td>
<td>5</td>
</tr>
<tr>
<td>2mm</td>
<td>2mm</td>
<td>mR</td>
<td></td>
<td>Use HOLZ to see if orthorhombic, tetragonal, hexagonal, or cubic. Use zero-layer diffraction pattern to distinguish between two cubic point groups. If tetragonal (110) then 422</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>If HOLZ reveals that crystal is tetragonal then 4/mmmm(100) or (110), if cubic then m3(100) or m3m(110). The two cubic point groups may be distinguished by the zero-layer pattern. If hexagonal (1120) then 6/mmm</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>4R</td>
<td>42[001] or 43m(001)</td>
<td>Distinguish by HOLZ diffraction</td>
<td>2</td>
</tr>
<tr>
<td>2mm</td>
<td>4mm</td>
<td>4Rmm</td>
<td></td>
<td>Cubic cases can be distinguished from trigonal by HOLZ</td>
<td>–</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>3 or 6R</td>
<td>3 or 3[001], 23 or m3(111)</td>
<td>Distinguish by HOLZ diffraction</td>
<td>2</td>
</tr>
<tr>
<td>3m</td>
<td>6</td>
<td>3lR</td>
<td>32[0001], 432(111)</td>
<td>–</td>
<td>1</td>
</tr>
<tr>
<td>3m</td>
<td>6mm</td>
<td>3lR</td>
<td></td>
<td>–</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>4mm</td>
<td>4Rmm</td>
<td>42[001] or 432(001)</td>
<td>Distinguish by HOLZ diffraction</td>
<td>2</td>
</tr>
<tr>
<td>4mm</td>
<td>4mm</td>
<td>4Rmm</td>
<td></td>
<td>If HOLZ reveals crystal is cubic then m3m</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>6mm</td>
<td>6Rmm</td>
<td>622[0001]</td>
<td>–</td>
<td>1</td>
</tr>
</tbody>
</table>

(8) The symmetry observed is derived from the symmetry of the whole specimen studied. The presence of undetected faults in the studied region can have a marked effect on this symmetry and the pattern symmetry would then no longer bear a simple relation to the crystal point group (Johnson, 1972; Tatlock & Steeds, 1973). One reliable method of detecting the presence of such faults is to examine the bright-field symmetry of a systematic diffraction row. If the pattern lacks a mirror line at its centre the crystal contains faults (the effect of non-systematics may be eliminated by rotating about the systematic axis).

(9) Strictly speaking the effect of inclined specimens interferes with the method of analysis proposed here. Steep specimen-wedge angles or high specimen inclinations could produce complicating effects. In practice such complications are only infrequently encountered (Goodman, 1974) and even then only for inclination greater than about 20°.

(10) For examining the bright-field symmetry, it is necessary to use the largest possible second condenser aperture which does not cause overlap of the zero-layer reflections. For close reflections it may be hard to see any detail unless very thick crystals are used (the scale of the patterns becomes finer as the thickness increases). Alternatively, bend-contour (Tanaka, Saito, Veno & Harada, 1980) or double-rocked (Eades, 1980) patterns may be used in appropriate specimens.

(11) The broken line in Table 1 implies that this step is unlikely to occur.

(12)* The star in Table 2 refers to the presence of an internal diad axis in any DF reflection when the beam is incident normal to a mirror plane in the point group. Reference to Table 2 in Buxton, Eades, Steeds & Rackham (1976) or else the work of Tanaka (1981) is recommended for this step.

(13)† The diffraction group relating the ±G dark-field symmetries always contains at least the element 2R for any zone axis in each of the eleven centrosymmetric groups (see Tables 2 and 4 in Buxton et al., 1976). For example, ±G reflections in point group 1 are not related except by the identity operator (DG1) whereas the equivalent DG is 2R for point group 1.

(14) WP stands for whole pattern, BF and DF refer to bright (direct beam) and dark field respectively. Point symmetries shown by the pattern as a whole (WP), i.e. the zero layer and/or the higher-order Laue zones (HOLZ's) are always shared by the direct beam (BF). The direct beam may also exhibit additional symmetry properties not found in the pattern as a whole.

2.2. Deduction of point group from a single principal-axis pattern

(a) The whole-pattern and bright-field symmetries listed in Table 3 provide sufficient information to
decide four point groups unambiguously (4, 6, 6m2, 622). Strictly, we need only the zero-layer pattern to determine both the BF and WP symmetries, including three-dimensional effects. However, low-camera-length micrographs containing HOLZ reflections can be a valuable cross-check on the WP symmetry when the visibility of HOLZ contrast in the zero-layer reflections is relatively weak. In addition, the HOLZ ring diameters are frequently a useful guide to the Bravais lattice and crystal class (see Table 3). Then another 17 point groups can be determined and are added to the list in Table 4. The eleven undetermined groups are 1, 1, 2/m, mmm, 4, 4/m, 3, 3, 3m, 6, 6/m.

(b) Additional use of internal dark-field symmetry. A further six point groups may be determined at a single principal axis in this way. These are 2/m[010], mmm(100), 4[001], 4/m[001], 6[0001], 6/m[0001].

(c) Additional comparison of ±G dark-field intensity distributions (or use of another zone axis in addition to the unique axis) 3m[0001] (1120). 

(d) ±G dark field essential 1, 1, 3, 3. 

(e) The conclusions arrived at should be treated with caution because of the possible existence of low-symmetry crystals having cell dimensions close to those of crystals of higher symmetry (pseudo-symmetry).

(f) The process of constructing the Bravais lattice from HOLZ reflections described below (§ 3.1) implies that both the crystal class and the zone axis can be determined from a single micrograph. In Table 3, the zone axis is combined with the diffraction group to distinguish four extra point groups (4mm(100), 6mm(1100), 422(110) and 6/mmm(1120)).

(g) For some space groups, containing suitably oriented glide planes, the presence of a mirror normal to the zone axis can be inferred directly from the absence of reflections in the mesh which occur in the HOLZ rings. Then the dark-field tests mentioned in (b) are eliminated for three of the six point groups (2/m, mmm and 4/m).

3. Space-group determination

3.1. Bravais lattice

By projecting the lattice of reflections in each of the HOLZ's onto the zero-layer mesh it is possible to deduce the Bravais lattice in reciprocal space and hence the real-space Bravais lattice. Note that it is important to infer the existence of absent zero-layer reflections from the HOLZ's or else incorrect conclusions can be reached (e.g. spinel, Steeds & Evans, 1980; garnet, Raghavan, Steeds & Petkovic-Luton, 1982). Small second condenser apertures and large-angle diffraction patterns are required for this exercise.

3.2. Dynamic absences

When a reflection, forbidden by kinematical diffraction theory, appears on a principal line of a zone-axis pattern it generally reveals a central line of absent intensity in a well-oriented pattern. The existence of such a line of absence indicates that the electron beam is incident on the crystal either parallel to a glide plane or perpendicular to a screw axis as shown in Fig. 1 of Gjonnes & Moodie (1965; see also Portier & Gratias, 1981). The screw axis which produced the absence is a 21 axis or one of higher symmetry which includes the 21 operation, i.e. 41, 43, 61, 63, 65 but not 31, 32, 42, 62, 64. The line of absence, sometimes called a Gjonnes-Moodie line, has a number of characteristics which may be used to distinguish it from accidental lines of absence fortuitously occurring in similar positions. The characteristics are as follows:

(i) Alternate reflections along a systematic line in a given layer must all show the characteristic line of absence.
(ii) The lines of absence become narrower as the specimen thickness increases.
(iii) The absences occur for all thicknesses and all microscope operating voltages.
(iv) By satisfying the Bragg condition for any particular order with a line of absence a second line of absence will be observed orthogonal to the first (the 'black cross'). This condition is strictly only obeyed in zero-layer diffraction where three-dimensional diffraction effects are relatively weak. Exceptions are found in the cases of 100 spinel or diamond structures where 002 reflections are observed as a result of multiple diffraction effects involving the next layer of the reciprocal lattice (Steeds & Evans, 1980). In this case the second crossing line of absence is not observed when the Bragg condition is satisfied (just as predicted by Gjonnes & Moodie, 1965). However, since three-dimensional diffraction generally subtracts intensity from a zero-layer pattern the rules worked out by Gjonnes & Moodie for three-dimensional absences are not usually applicable. However, the information about screw axes and glide planes can be deduced by an alternative and equally simple method which takes account of three-dimensional diffraction (see § 3.3). The fact that the characteristic effects listed

Table 4. The 21 point groups determined from a single zone-axis pattern

<table>
<thead>
<tr>
<th>Monoclinic</th>
<th>Orthorhombic</th>
<th>Trigonal</th>
<th>Tetragonal</th>
<th>Hexagonal</th>
<th>Cubic</th>
</tr>
</thead>
<tbody>
<tr>
<td>m</td>
<td>mm2</td>
<td>3m</td>
<td>4, 4mm</td>
<td>6, 6mm</td>
<td>m3</td>
</tr>
<tr>
<td>2</td>
<td>222</td>
<td>32</td>
<td>42m, 4/mmm</td>
<td>6/m2, 6/mmm</td>
<td>43m</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>422</td>
<td>622</td>
<td></td>
</tr>
</tbody>
</table>

The 21 point groups determined from a single zone-axis pattern.

Note: The table includes all the point groups that can be determined from a single zone-axis pattern, as well as those that can be deduced from additional data. The entries in the table are as follows:

- **Monoclinic**: This group includes reflections that are parallel to the unique axis of the crystal.
- **Orthorhombic**: These reflections are parallel to the three unique axes.
- **Trigonal**: This group includes reflections that are parallel to any of the three axes.
- **Tetragonal**: These reflections are parallel to the two axes.
- **Hexagonal**: This group includes reflections that are parallel to any of the three axes.
- **Cubic**: This group includes reflections that are parallel to any of the three axes.

The table also includes additional information about the point groups, such as the presence of additional symmetry elements, and the conditions under which they can be determined. This information is important for deducing the crystal structure from the diffraction patterns.
Table 5. The six different cases of dynamic absences along a single systematic line of reflections

Symbols used follow Tables 4.1.6 and 4.1.7 of International Tables for X-ray Crystallography (1969).

--- has been used to indicate a general glide plane parallel to the direction of incidence. It also includes ....... and ----

<table>
<thead>
<tr>
<th>WP</th>
<th>BF</th>
<th>Diffraction group</th>
<th>Orientation of mirrors with respect to line of absences in a zone-axis pattern (orthogonal lines are principal axes)</th>
<th>Minimum number of symmetry elements responsible for absence</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>m</td>
<td>mR</td>
<td><img src="image" alt="Diagram" /></td>
<td>Screw axis perpendicular to beam 2, 4, 6 or 6i.</td>
</tr>
<tr>
<td>m</td>
<td>m</td>
<td>2mm mR</td>
<td>(a) <img src="image" alt="Diagram" /></td>
<td>Screw axis perpendicular to the beam and to a mirror plane; 2/m, 6/m.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>or</td>
<td></td>
</tr>
<tr>
<td></td>
<td>m</td>
<td>m</td>
<td><img src="image" alt="Diagram" /></td>
<td>Glide plane parallel to beam.</td>
</tr>
<tr>
<td>m</td>
<td>2mm</td>
<td>m1R</td>
<td><img src="image" alt="Diagram" /></td>
<td>Screw axis (2, 6) plus parallel glide plane.</td>
</tr>
<tr>
<td>2mm</td>
<td>2mm</td>
<td>2mm or 2mm m1R</td>
<td><img src="image" alt="Diagram" /></td>
<td>As (b) above with an extra mirror plane parallel to the beam and perpendicular to the glide plane.</td>
</tr>
<tr>
<td>2mm</td>
<td>2mm</td>
<td>2mm mR</td>
<td><img src="image" alt="Diagram" /></td>
<td>Screw axis (2, 4, 6, 6i or 6) axis perpendicular to a 2-fold axis or 2, both perpendicular to the beam.</td>
</tr>
</tbody>
</table>

above also apply to the cases of 4, 4, 6, 6, 6i, and 65 screw axes calls for some comment. Odd reflections 00l (l = 2n + 1) along a line with kinematic absences will have crossing Gjønnes-Moodie lines for zone axes which permit multiple-diffraction routes. Although these cases have not been discussed in detail in the previous literature the conclusion follows at once from the projection argument of Gjønnes & Moodie (1965).

3.3. Dynamical absences in the case of three-dimensional diffraction (Steed, Rackham & Shannon, 1978; Shannon, 1979)

The absences may be observed in either the zero layer or the HOLZ’s. The zero layer absences may be subdivided into two cases, those with a single line of absences and those with absences along two perpendicular axes. In addition, absences occur in the HOLZ’s themselves as a result of glide planes parallel to the incident beam.

3.3.1. Single line of absences in zero layer. There are six different cases of this type, illustrated in Table 5.

3.3.2. Two orthogonal lines of absences in zero layer. There are six different cases of this type and they are illustrated in Table 6. One diffraction group, m1R, with WP and BF symmetries m and 2mm, respectively, has been deliberately omitted from Table 6 because no example with orthogonal rows of absences in the zero-layer pattern occurs along any zone axis within any space group. Details of the proof are not given here, but the method relies on first extracting the possible combinations of zone axes and point groups with DG ml from Tables 3 and 4 in Buxton et al. (1976), and then examining the relevant space groups for the presence of suitably oriented glide planes and screw axes. In addition, there must exist dynamical diffraction routes within the zero layer to the forbidden reflections.

With the aid of the dynamical absences in three-dimensional diffraction the crystal space group may
Table 6. The seven cases of dynamic absences along two orthogonal lines

<table>
<thead>
<tr>
<th>WP</th>
<th>BF</th>
<th>DG</th>
<th>Minimum number of symmetry elements responsible for absences</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>2mm</td>
<td>2mm</td>
<td>Orthogonal screw axes, orthogonal to the beam direction.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>21 perpendicular to 21, 41, or 43</td>
</tr>
<tr>
<td>2mm</td>
<td>2mm</td>
<td>2mm or</td>
<td>Two perpendicular glide planes</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2mm1</td>
<td></td>
</tr>
<tr>
<td>m</td>
<td>m</td>
<td>2mm</td>
<td>2 screw axis perpendicular to a glide plane (N.B. although there are 41 screw axes perpendicular to glide planes in cubic F and l-centred space groups dynamic absences do not occur because there are no multiple diffraction routes to the forbidden reflections).</td>
</tr>
<tr>
<td>4</td>
<td>4mm</td>
<td>4mm</td>
<td>Orthogonal screw diads normal to a tetrad axis (4, 41, 42 or 43) which is parallel to the beam direction OR an orthogonal set of three 41 or 43 screw tetrad axes with one axis parallel to the beam</td>
</tr>
<tr>
<td>2mm</td>
<td>4mm</td>
<td>4mm</td>
<td>Orthogonal screw diads normal both to an inversion tetrad axis and to the beam direction</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(a)</td>
<td></td>
</tr>
<tr>
<td>4mm</td>
<td>4mm</td>
<td>4mm</td>
<td>Orthogonal glide planes, parallel to an inversion tetrad axis</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(b)</td>
<td></td>
</tr>
<tr>
<td>4mm</td>
<td>4mm</td>
<td>4mm or</td>
<td>Orthogonal glide planes, parallel both to a tetrad axis (4 or 41) and to the beam direction</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4mm1</td>
<td></td>
</tr>
</tbody>
</table>
Table 7. Table giving the number of possible space groups once the point group and Bravais lattice have been determined

<table>
<thead>
<tr>
<th>No.</th>
<th>Point groups and Bravais lattices which give the space group directly</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>P1, P1, P6, I4, P3, R3, F23, R32, F222, C2, P4</td>
</tr>
<tr>
<td>24</td>
<td>P2, Pm, Cm, R3m, R3m, C222, I222, Fmmm, C2/m, I4, I4/m, P6/m, Fm3, Im3, F432, I432, I422, I3m, P23, I23, P43m, F43m, I43m, Fmm2</td>
</tr>
<tr>
<td>4</td>
<td>P3, Pm3, Cm2, I2m2</td>
</tr>
<tr>
<td>17</td>
<td>P2/m, P222, Cm2, Pm2, P3m, P3m, P4, P4/m, I422, I4/m, P6/m, Fm3, I4/m, P6/m, Fm3, Im3, F432, I432, I422, I3m, P23, I23, P43m, F43m, I43m, Fmm2</td>
</tr>
<tr>
<td>4</td>
<td>P6, P622, P32, Cmmm</td>
</tr>
<tr>
<td>3</td>
<td>P422, P4mm, P42m</td>
</tr>
<tr>
<td>1</td>
<td>Pm2</td>
</tr>
<tr>
<td>2</td>
<td>P4/mmmm</td>
</tr>
</tbody>
</table>

be determined with the aid of Tables 5 and 6. In fact, once the point group and Bravais lattice have been determined there are generally only a few space groups to decide between (see Table 7). In twelve cases this information is sufficient to determine the space group directly.

4. Conclusion

Use of convergent-beam electron diffraction from small perfect crystals has greatly simplified the job of deducing the crystal point and space groups.

One of us (RV) wishes to acknowledge the support of the Science and Engineering Research Council for a research assistantship. We also acknowledge the critical comments of Dr J. A. Eades.

References