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screw elements the symbol of that element is given and the '*G*' or '*S*' symbol is omitted.

The following paragraphs give information on the real-space interpretation of GS band formation, and their specific extinction rules, considered useful in structural interpretation.

Real-space interpretation of extinction conditions. Dynamic extinctions (GS bands) are essentially a property of symmetry in reciprocal space. However, since diagrams from *IT* I and A are used there is a need to give an equivalent real-space description. These bands are associated with the half-unit-cell-translational glide planes and screw axes represented in these diagrams. Inconsistencies between 'conventional' and 'physical' real-space descriptions, however, become more apparent in dynamical electron diffraction, which is dependent upon three-dimensional scattering physics, than in X-ray diffraction. Also, the distinction between general (symmorphic) and specific (non-symmorphic) extinctions is more basic (in the former case). This is clarified by the following points:

(i) Bravais lattice centring restricts the conditions for observation of GS bands. For example, in space group *Abm*2 (No. 39), '*A*' centring prevents observation of the GS bands associated with the '*b*' glide at the [001] zone-axis orientation; this observation, and hence verification of the *b* glide, must be made at the lower-symmetry zone axes [0*vw*] (see Table 2.5.3.5). In the exceptional cases of space groups $I2_12_12_1$ and $I2_13$ (Nos. 24 and 199), conditions for the observation of the relevant GS bands are completely prevented by body centring; here the screw axes of the symmorphic groups *I*222 and *I*23 are parallel to the screw axes of their non-symmorphic derivatives. However, electron crystallographic methods also include direct structure imaging by HREM, and it is important to note here that while the indistinguishability encountered in data sets acquired in Fourier space applies to both X-ray diffraction and CBED (notwithstanding possible differences in HOLZ symmetries), this limitation does not apply to the HREM images (produced by dynamic scattering) yielding an approximate structure image for the (zone-axis) *projection*. This technique then becomes a powerful tool in space-group research by supplying phase information in a different form.

(ii) A different complication, relating to nomenclature, occurs in the space groups $P\bar{4}3n$, *Pn*3*n* and *Pm*3*n* (Nos. 218, 222 and 223) where '*c*' glides parallel to a diagonal plane of the unit cell occur as primary non-symmorphic elements (responsible for reciprocal-space extinctions) but are not used in the Hermann–Maugin symbol; instead the derivative '*n*' glide planes are used as characters, resulting in an *apparent* lack of correspondence between the conventionally given real-space symbols and the reciprocal-space extinctions.

(*Note*: In *IT* I non-symmorphic reflection rules which duplicate rules given by lattice centring, or those which are a consequence of more general rules, are given in parentheses; in *IT* A this clarification by parenthesizing, helpful for electron-diffraction analysis, has been removed.)

(iii) Finally, diamond glides (symbol '*d*') require special consideration since they are associated with translations $\frac{1}{4}, \frac{1}{4}, \frac{1}{4}$, and so would appear not to qualify for GS bands; however, this translation is a result of the conventional cell being defined in real rather than reciprocal space where the extinction symmetry is formed. Hence '*d*' glides occur only in *F*-centred lattices (most obviously Nos. 43, 70, 203, 277 and 228). These have correspondingly an *I*-centred reciprocal lattice for which the zero-layer two-dimensional unit cell has an edge of $a^{*\prime\prime} = 2a^*$. Consequently, the first-order row reflection along the diamond glide retains the reciprocal-space anti-symmetry on the basis of this physical unit cell (halved in real space), and leads to the labelling of odd-order reflections as $4n + 2$ (instead of $2n + 1$ when the cell is not halved). Additionally, although seven space groups are *I*-centred in real space with the conventional unit cell (Nos. 109, 110, 122, 141, 142, 220 and 230), these space groups are *F*-centred with the transformation $a'' = [110]$, $b'' = [1\bar{1}0]$, and correspondingly *I*-centred in the reciprocal-space cell as before, but the directions [100], [010] and reflection rows *h*00, 0*k*0 become replaced by directions [110] (or $[1\bar{1}0]$) and rows *hh*0, $h\bar{h}0$, in the entries of Table 2.5.3.5.

Extinction rules for symmetry elements appearing in Table 2.5.3.5. Reflection indices permitting observation of *G* and *S* bands follow [here 'zero-layer' and 'out-of-zone' (*i.e.* HOLZ or alternative zone) serve to emphasize that these are zone-axis observations].

(i) *Vertical glide planes* lead to '*G*' bands in reflections as listed ('*a*', '*b*', '*c*' and '*n*' glides):

*h*0*l*, *hk*0, 0*kl* out-of-zone reflections (for glide planes having normals [010], [001] and [100]) having $h + l$, $h + k$, $k + l = 2n + 1$, respectively, in the case of '*n*' glides, or *h*, *k*, *l* odd in the case of '*a*', '*b*' or '*c*' glides, respectively;

*h*00, 0*k*0, 00*l* zero-layer reflections with *h*, *k* or *l* odd.

Correspondingly for '*d*' glides:

(*a*) In *F*-centred cells:

*h*0*l*, *hk*0, 0*kl* out-of-zone reflections (for glide planes having normals [010], [100] and [001], having $h + l$, $k + l$, or $h + k = 4n + 2$, respectively, with *h*, *k* and *l* even; and (space group No. 43 only) zero-layer reflections *h*00, 0*k*0 with *h*, *k* even and $= 4n + 2$.

(*b*) In *I*-centred cells:

hhl (cyclic on *h*, *k*, *l* for cubic groups) out-of-zone reflections having $2h + l = 4n + 2$, with *l* even; and zero-layer reflections *hh*0, $h\bar{h}0$ (cyclic on *h*, *k*, *l* for cubic groups) having *h* odd.

(ii) *Horizontal screw axes*, namely 2_1 or the 2_1 component of screw axes 4_1, 4_3, 6_1, 6_3, 6_5, lead to '*S*' bands in reflection rows parallel to the screw axis, *i.e.* either *h*00, 0*k*0 or 00*l*, with conventional indexing, for *h*, *k* or *l* odd.

(iii) *Horizontal glide planes* lead to zero-layer absences rather than GS bands. When these prevent observation of a specific GS band (by removing the two-dimensional conditions), the symbol '–' indicates a situation where, in general, there will simply be absences for the odd-order reflections. However, Ishizuka & Taftø (1982) were the first to observe finite-intensity narrow bands under these conditions, and it is now appreciated that with a sufficient crystal thickness and a certain minimum for the *z*-axis repeat distance, GS bands can be recorded by violating the condition for horizontal-mirror-plane (m') extinction while satisfying the condition for *G* or *S*, achieved by appropriate tilts away from the exact zone-axis orientation [see Section 2.5.3.3(iv)].

2.5.3.5. *Space-group analyses of single crystals; experimental procedure and published examples*

2.5.3.5.1. *Stages of procedure*

(i) *Zone-axis patterns*. The first need is to record a principal zone-axis pattern. From this, the rotational order *X* of the vertical axis and associated mirror (including glide-line) components are readily observed (see all examples).

This pattern may include part of the higher-order Laue zone; in particular the closest or first-order Laue zone (FOLZ) should be included in order to establish the presence or absence of horizontal glide planes, as illustrated in Fig. 2.5.3.3. The projection approximation frequently applies to the zone-axis pattern, particularly when this is obtained from thin crystals (although this cannot apply by definition to the FOLZ). This is indicated by the absence of fine-line detail in the central beam particularly; identification of the projected symmetry is then straightforward.

(ii) *Laue circle patterns*. Next, it is usual to seek patterns in which discs around the Laue circle include the line m_R (Fig.

2.5.3.2). The internal disc symmetries observed together with those from the zone-axis pattern will determine a diffraction group, classifying the zero-layer symmetry. [Fig. 6(*c*) of Goodman & Whitfield (1980) gives an example of Laue-circle symmetries.]

(iii) *Alternative zone axes or higher-order Laue zones*. Finally, alternative zone or higher-order Laue-zone patterns may be sought for additional three-dimensional data: (*a*) to determine the three-dimensional extinction rules, (*b*) to test for centrosymmetry, or (*c*) to test for the existence of mirror planes perpendicular to the principal rotation axis. These procedures are illustrated in the following examples.

2.5.3.5.2. *Examples*

(1) *Determination of centrosymmetry; examples from the hexagonal system*. Fig. 2.5.3.4(*a*) illustrates the allocation of planar point groups from [0001] zone-axis patterns of β-Si_3N_4 (left-hand side) and β-GaS (right-hand side); the patterns exhibit point symmetries of 6 and 6*mm*, respectively, as indicated by the accompanying geometric figures, permitting point groups 6 or $6/m$, and 6*mm* or $6/mmm$, in three dimensions. Alternative zone axes are required to distinguish these possibilities, the actual test used (testing for the element m' or the centre of symmetry) being largely determined in practice by the type of crystal preparation.

Fig. 2.5.3.4(*b*) shows the CBED pattern from the $[11\bar{2}0]$ zone axis of β-Si_3N_4 (Bando, 1981), using a crystal with the corresponding cleavage faces. The breakdown of Friedel's law between reflections 0002 and $000\bar{2}$ rules out the point group $6/m$ (the element m' from the first setting is not present) and establishes 6 as the correct point group.

Also, the GS bands in the 0001 and $000\bar{1}$ reflections are consistent with the space group $P6_3$. [*Note*: screw axes 6_1, 6_3 and 6_5 are not distinguished from these data alone (Tanaka *et al.*, 1983).]

Fig. 2.5.3.4(*c*) shows CBED patterns from the vicinity of the $[1\bar{1}02]$ zone axis of β-GaS, only 11.2° rotated from the [0001] axis and accessible using the same crystal as for the previous [0001] pattern. This shows a positive test for centrosymmetry using a conjugate reflection pair $1\bar{1}01/\bar{1}10\bar{1}$, and establishes the centrosymmetric point group $6/mmm$, with possible space groups Nos. 191, 192, 193 and 194. Rotation of the crystal to test the extinction rule for $hh2\bar{h}l$ reflections with l odd (Goodman & Whitfield, 1980) establishes No. 194 ($P6_3/mmc$) as the space group.

Comment: These examples show two different methods for testing for centrosymmetry. The $\pm H$ test places certain requirements on the specimen, namely that it be reasonably accurately parallel-sided – a condition usually met by easy-cleavage materials like GaS, though not necessarily by the wedge-shaped refractory Si_3N_4 crystals. On the other hand, the 90° setting, required for direct observation of a possible perpendicular mirror plane, is readily available in these fractured samples, but not for the natural cleavage samples.

(2) *Point-group determination in the cubic system, using Table 2.5.3.3*. Fig. 2.5.3.5 shows [001] (cyclic) zone-axis patterns from two cubic materials, which serve to illustrate the ability to distinguish cubic point groups from single zone-axis patterns displaying detailed central-beam structures. The left-hand pattern, from the mineral gahnite (Ishizuka & Taftø, 1982) has 4*mm* symmetry in *both* the whole pattern and the central (bright-field) beam, permitting only the BESR group $4mm1_R$ for the cubic system (column III, Table 2.5.3.3); this same observation establishes the crystallographic point group as $m3m$ (column V of Table 2.5.3.3). The corresponding pattern for the χ-phase precipitate of stainless steel (Steeds & Evans, 1980) has a whole-pattern symmetry of only 2*mm*, lower than the central-beam (bright-field) symmetry of 4*mm* (this lower symmetry is made clearest from the innermost reflections bordering the central beam). This combination leads to the BESR group 4_Rmm_R (column III, Table 2.5.3.3), and identifies the cubic point group as $\bar{4}3m$.

(3) *Analysis of data from FeS_2 illustrating use of Tables 2.5.3.4 and 2.5.3.5*. FeS_2 has a cubic structure for which a complete set of data has been obtained by Tanaka *et al*. (1983); the quality of the data makes it a textbook example (Tanaka & Terauchi, 1985) for demonstrating the interpretation of extinction bands.

Figs. 2.5.3.6(*a*) and (*b*) show the [001] (cyclic) exact zone-axis pattern and the pattern with symmetrical excitation of the 100 reflection, respectively (Tanaka *et al.*, 1983).

(i) Using Table 2.5.3.4, since there are GS bands, the pattern group must be listed in column II(ii); since a horizontal 'b' glide plane is present (odd rows are absent in the b^* direction), the symbol must contain a 'b'' (or 'a'') (*cf.* Fig. 2.5.3.3). The only possible cubic group from Table 2.5.3.4 is No. 205.

(ii) Again, a complete GS cross, with both G and S arms, is present in the 100 reflection (central in Fig. 2.5.3.6*b*), confirmed by mirror symmetries across the G and S lines. From Table 2.5.3.5 only space group No. 205 has the corresponding entry in the column for '[100] cyclic' with *GS* in the cubic system (space groups Nos. 198–230). Additional patterns for the [110] setting, appearing in the original paper (Tanaka *et al.*, 1983), confirm the cubic system, and also give additional extinction characteristics for 001 and $1\bar{1}0$ reflections (Tanaka *et al.*, 1983; Tanaka & Terauchi, 1985).

(4) *Determination of centrosymmetry and space group from extinction characteristics*. Especially in working with thin crystals used in conjunction with high-resolution lattice imaging, it is sometimes most practical to determine the point group (*i.e.* space-group class) from the dynamic extinction data. This is exemplified in the Moodie & Whitfield (1984) studies of orthorhombic materials. Observations on the zero-layer pattern for Ge_3SbSe_3 with a point symmetry of 2*mm*, and with GS extinction bands along odd-order $h00$ reflections, together with missing reflection rows in the $0k0$ direction, permit identification from Table 2.5.3.4. This zone-axis pattern has the characteristics illustrated in Figs. 2.5.3.3 and hence (having both missing rows and GS bands) should be listed in both II(ii) and II(iii). Hence the diffraction group must be either No. 40 or 41. Here, the class *mmm*, and hence centrosymmetry, has been identified through non-symmorphic elements.

This identification leaves seven possible space groups, Nos. 52, 54, 56, 57, 60, 61 and 62, to be distinguished by *hkl* extinctions.

The same groups are identified from Table 2.5.3.5 by seeking the entry *GS* '−' in one of the [001] (cyclic) entries for the orthorhombic systems. With the assumption that no other principal zone axis is readily available from the same sample (which will generally be true), Table 2.5.3.5, in the last three columns, indicates which *minor* zone axes should be sought in order to identify the space group, from the glide-plane extinctions of 'G' bands. For example, space group 62 has no $h0l$ extinctions, but will give $0kl$ extinction bands 'G' according to the rules for an 'n' glide, *i.e.* in reflections for which $k + l = 2n + 1$. Again, if the alternative principal settings *are* available (from the alternative cleavages of the sample) the correct space group can be found from the first three columns of Table 2.5.3.5.

From the above discussions it will be clear that Tables 2.5.3.4 and 2.5.3.5 present information in a complementary way: in Table 2.5.3.4 the specific pattern group is indexed first with the possible space groups following, while in Table 2.5.3.5 the space group is indexed first, and the possible pattern symmetries are then given, in terms of the standard *International Tables* setting.

2.5.3.6. *Use of CBED in study of crystal defects, twins and non-classical crystallography*

(i) Certain crystal defects lend themselves to analysis by CBED and LACBED. In earlier work, use was made of the high sensitivity

references