

Vocabulary of Surface Crystallography

ELIZABETH A. WOOD

Bell Telephone Laboratories, Incorporated, Murray Hill, New Jersey

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The recent increase of activity in the field of slow-electron diffraction has resulted in a need for agreement on conventions of terminology in the description of a diperiodic *surface structure* relative to a triperiodic *substrate*. Vector description of the surface structure *net* in terms of the substrate surface net is suggested. Fractional Miller indices result when the surface structure *mesh* is larger than the substrate surface mesh. The surface structure may be either that of a deposit or that of the *selvedge* of the substrate.

With the relaxation of one of the diffraction conditions in the diperiodic structure the reciprocal lattice becomes a set of parallel rods intersecting the Ewald sphere continuously with change of wavelength of the incident radiation or change of orientation of the crystal.

The paper discusses the applicability of the seventeen two-dimensional space groups and the eighty diperiodic groups in three dimensions to these diperiodic structures. A comparison is made between the different effects of surface symmetry on etch pits and epitaxial deposits on the cleavage face of muscovite.

INTRODUCTION

THE recent increase of activity in the field of slow-electron diffraction¹ has resulted in a need for agreement on conventions of terminology which was expressed to the writer by some of the workers in this field. With the hope of selecting a set of conventions convenient to use, readily grasped by the reader and acceptable to three-dimensional crystallographers, a memorandum with the above title was circulated among those known to be working on surface structures and made available at the 1962 meeting of the American Crystallographic Association at Villanova University. The conventions suggested there have been revised as a result of comments by readers of the memorandum and are presented here.

NATURE OF THE SURFACE

The surface in question may be entirely clean, consisting only of those atoms that constitute the substrate material or it may have other atoms deposited on it. In either case it is probable that in the outer few atom layers of the substrate the arrangement will differ from that in the bulk material.

The surface structure may be related to that of a parallel planar section of the substrate material by small displacements of atoms or the surface structure may require "reconstructive displacements." [See discussion by M. J. Buerger, *Phase Transformations in Solids* (John Wiley & Sons, Inc., New York, 1951), p. 183.] It is proposed that this region of the specimen be called the *selvedge* by analogy with the selvedge of a

piece of cloth which is a narrow band so woven as to prevent raveling of the edge. (See Fig. 1).

The true *substrate* or *bulk material*, then, has a periodicity normal to the surface ("in the *z* direction") and the *substrate surface* is marked by the disappearance of this periodicity. Since "disappearance" of periodicity depends on the sensitivity of the tool used to test the periodicity, the measured depth of the selvedge, like the results of all physical measurements, depends on the sensitivity of the measuring device.

With the deposit of additional atoms of different type, the structure of the selvedge may change, but in general the selvedge does not disappear. If the deposit is very thin it may not show any periodicity in the *z* direction. If it is thick, it may show such periodicity except in its outermost layers where the deposit material may have a selvedge.

In the following discussion the relations between a substrate structure and a contiguous, related structure, called the *surface structure* are discussed. The "surface structure" is in general *diperiodic*,² which does not necessarily mean that all its atoms lie in a plane, but rather that it is periodic only in two dimensions. Such a surface structure could be the structure of a deposit or it could be the structure of the selvedge of the pure material. For this reason it is desirable to use the general term "surface structure" rather than the term "deposit structure."

The terms "superstructure" and "superlattice" for the structure and the lattice of the superposed material should be avoided because they are already used in the literature to describe an order whose repeat unit is an integral multiple of some smaller repeat unit which is not the true repeat unit of the ordered form but is that of the disordered form. The use of these terms for surface structure would be especially confusing in cases where the repeat distance of the deposit was the same as or smaller than that of the substrate. Furthermore, the surface structure might, in some cases, exhibit what has been known as a superstructure, in which case it would

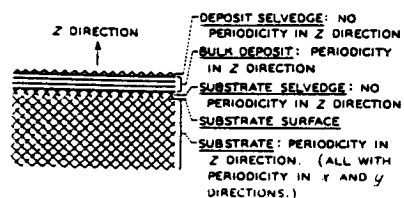


FIG. 1. Diagram of epitaxial film on substrate, illustrating suggested nomenclature.

¹ Chronological list of recent references. All references are listed at the end of this article.

TABLE I. The five nets.

Shape of unit mesh	Lattice symbol	Conventional rule of choice of axes	Nature of axes and angles*	Name of corresponding system
General parallelogram (rhomboid)	p	$a < b$	$a \neq b$ $\gamma \neq 90^\circ$	Oblique
Rectangle	p c	The shortest two mutually perpendicular vectors $a < b$	$a \neq b$ $\gamma = 90^\circ$	Rectangular
Square	p	The shortest two mutually perpendicular vectors	$a = b$ $\gamma = 90^\circ$	Square
120° angle rhombus	p	The shortest two vectors at 120° to each other	$a = b$ $\gamma = 120^\circ$	Hexagonal

* The symbol \neq implies nonequality by reason of symmetry; accidental equality may, of course, occur.

be quite appropriate to speak of the superstructure of the surface structure.

FIVE NETS

In triperiodic structures the equivalent points form a three-dimensional *lattice* in which the space units are *unit cells*. In diperiodic structures the equivalent points form a two-dimensional *net* in which the area units are *unit meshes*. The fourteen Bravais space lattices are replaced, in the diperiodic case, by five nets, described in Table I and diagrammed in Fig. 2.

As in triperiodic crystallography, it may sometimes be desirable to use a centered mesh, even though not rectangular, or even a "multiply primitive" mesh, if by so doing the relationships which the author is describing are made more evident. If a centered mesh is used, the letter c must precede the description of the mesh (v.i.). The letter p denotes the primitive mesh, with equivalent points at its corners, but none within the mesh.

In the choice of axes of an *oblique* or *rectangular* mesh, the triperiodic convention $a < b$ should, in general, be retained.

EXISTING CONVENTIONS

The symbols listed below are adapted from those given in Vol. 1 of the *International Tables for X-Ray Crystallography*³ (ITXC). The conventions given there should be followed where possible, as in the choice of most of the symbols in the following list.

Symbols

- a, b : Lengths of unit-mesh edges. Subscript s may be used to distinguish surface structure from underlying structure where necessary.
- \mathbf{a}, \mathbf{b} : Unit-mesh vectors.
- γ : Interaxial angle $x\Lambda y$, $a\Lambda b$.
- x, y :- Directions of crystallographic axes of coordinates.
- x, y : Coordinates of any point within the unit mesh, expressed in terms of a and b units.

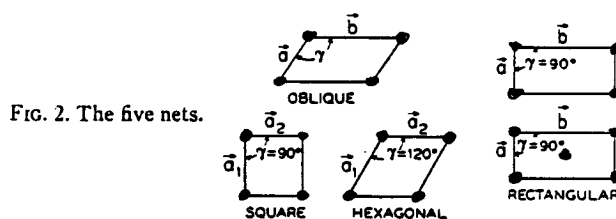


FIG. 2. The five nets.

- z :- Direction normal to x - and y -directions.
- z : Coordinate of any point in this direction, expressed in terms of angstrom units, positive above (away from substrate) and negative below some arbitrarily chosen plane.
- u, v : Coordinates of any net point, expressed in terms of a and b as units.
- $[uv]$: Indices of a direction in the direct (real-space) net.
- $\langle uv \rangle$: Indices of a "form" of directions (related by symmetry).
- (hk) : Indices of a set of parallel rows.
- hk : Indices of the reflection from a set of parallel rows; coordinates of a rod in the reciprocal lattice as measured on any plane normal to the rods.
- $\{hk\}$: Indices of a form of sets of rows or of the reflections from such a form.
- a^*, b^* : Lengths of reciprocal-lattice unit-mesh edges.
- $\mathbf{a}^*, \mathbf{b}^*$: Unit-mesh vectors in reciprocal space.
- γ^* : Interaxial angle in reciprocal space.
- $d_{(hk)}$: Interrow spacing of rows hk .
- A, A^* : Unit-mesh area in real and reciprocal space, respectively.
- $[uv]^*$: Indices of that direction in reciprocal space defined by a line from the origin through the points $h_u k_v$, or from the origin through the "rods" $h_u k_v$ and normal to these rods. (Examples: $[u0]^*$ through the reciprocal lattice points 10, 20, 30, etc.; $[uu]^*$ through the reciprocal lattice points 11, 22, 33, etc.; $[2u \cdot u]^*$ through the reciprocal lattice points 21, 42, 63, etc.)
- $\langle uv \rangle^*$: Indices of a "form" of directions in reciprocal space (related by symmetry).
- p, c : Primitive and centered nets (two-dimensional lattices).
- 1, 2, 3, 4, 6: Rotation axes.
- $\bar{1}, \bar{2}, \bar{3}, \bar{4}, \bar{6}$: Inversion axes.
- 2_1 : Screw axis.
- m : Mirror reflection plane.
- a, b, n : Glide planes.
- n, m : Any integers.

Further conventions indicated in the ITXC, Vol. 1, are: (1) the conventional orientation of the unit mesh with the y axis horizontal and pointing to the right, the x axis down the page either straight or slanting to the

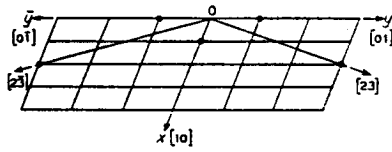


FIG. 3. Indices for directions $[uv]$.

left as in Fig. 2; (2) Miller indices (hk) of a set of parallel rows are the reciprocals of the row intercepts on the x and y axes; a direction is indicated by $[uv]$, where u and v are the coordinates of a net point (in terms of mesh units) which, with the origin, defines the direction (see Fig. 3) (ITXC p. 10).

These conventions follow the triperiodic conventions, with the omission of the final symbol. In the hexagonal triperiodic case the indices $(hkil)$ are commonly used. The omission of the final symbol in this case leaves three indices and might result in confusion due to the fact that most authors will be using the three-index notation to refer to the triperiodic substrate, depending on the two-index notation to denote diperiodicity of the surface structure. Therefore the i index should not be used. It may always be derived from h and k in the hexagonal system by the relationship $i = -(h+k)$.

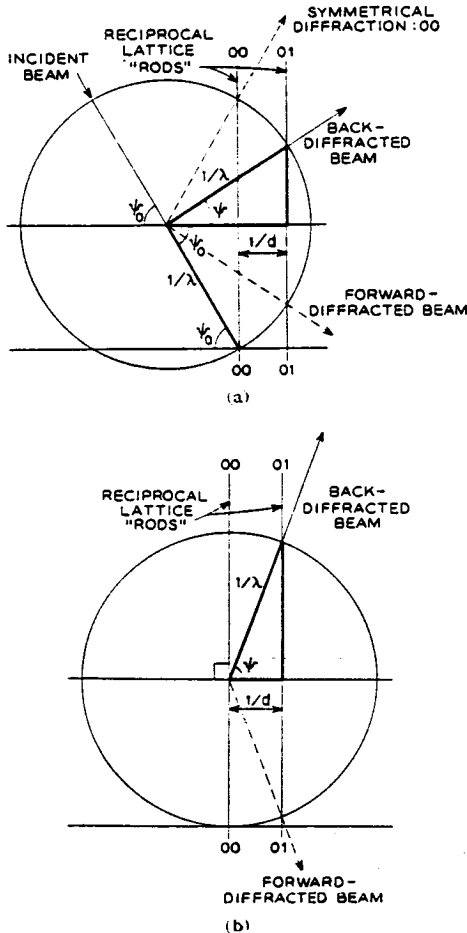


FIG. 4. Sections of the reciprocal lattice of a diperiodic structure with the Ewald sphere. (a) General case, $m\lambda = d(\cos\psi - \cos\psi_0)$; (b) normal incidence, $m\lambda = d \cos\psi$.

Interrow Spacing Formulas for the Five Nets

Oblique:

$$\frac{1}{d^2_{(hk)}} = \frac{h^2}{a^2 \sin^2\gamma} + \frac{k^2}{b^2 \sin^2\gamma} + \frac{2hk \cos\gamma}{ab \sin^2\gamma}$$

Rectangular, p and c :

$$1/d^2_{(hk)} = (h/a)^2 + (k/b)^2$$

Hexagonal:

$$1/d^2_{(hk)} = \frac{4}{3}[(h^2 + hk + k^2)/a^2]$$

Square:

$$1/d^2_{(hk)} = (h^2 + k^2)/a^2$$

THE RECIPROCAL LATTICE

The reciprocal lattice for the triperiodic case is discussed in the *International Tables* (ITXC, Vol. 1, p. 11). Together with the sphere of reflection (the Ewald sphere) it forms a powerful tool for working in the field of x-ray and electron diffraction. In the triperiodic case the diffracted beam is generated when a reciprocal lattice point coincides with the Ewald sphere. Any change in orientation of the crystal (movement of the point) or change in the wavelength of the beam (change in the radius of the sphere) destroys the condition for diffraction. In the diperiodic case, the removal of periodicity in the third dimension relaxes one of the diffraction conditions and diffraction occurs continuously with continuous change of wavelength or rotation of the diperiodic structure. Therefore the reciprocal lattice is most usefully considered as an array of lines or one-dimensional "rods" of infinite extent normal to the plane of diperiodicity.

Figure 4 shows a section through such a reciprocal lattice with the Ewald sphere passing through an arbitrarily chosen origin. Referring to Fig. 4(a), where ψ_0 = angle of incidence, ψ = angle of diffraction, d = periodicity distance and λ = wavelength of electron beam, m = any integer (here, 1),

$$\cos\psi = [(1/d) + (1/\lambda) \cos\psi_0] \lambda$$

$$\cos\psi = (\lambda/d) + \cos\psi_0$$

$$\lambda = d(\cos\psi - \cos\psi_0)$$

(Laue equation for the diperiodic case).

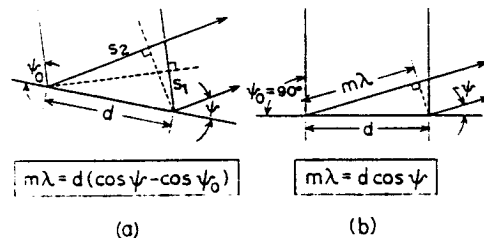


FIG. 5. Diagram of diffraction from surface rows: (a) general case; (b) normal incidence.

In Fig. 4(b) the special case is shown in which the incident beam is normal to the plane of periodicity of the surface structure and therefore parallel to the reciprocal lattice rods. In this case,

$$\cos\psi_0 = 0$$

$$\lambda = d \cos\psi.$$

Note that ψ varies inversely with λ .

The same derivations in real space are shown in Fig. 5. Note that the angle of incidence ψ_0 is not equal to the angle of diffraction ψ except for the 00 beam (Fig. 6), for which the path difference for beams scattered from equivalent scatterers is zero.

RELATION BETWEEN THE SURFACE MESH AND THE SUBSTRATE MESH

Since the structure of the substrate is the initially known structure, it is taken as the basis of reference. Its net parallel to the surface is the reference net. For example, if the surface of a cubic substrate crystal is the (111) surface, the substrate net is hexagonal (see Fig. 2) and the surface net is referred to that.

TABLE II. Definition of surface mesh in terms of substrate mesh.

Illustration	Real space	Reciprocal space
Fig. 7	$a_s = 2a$ $b_s = 2b$	$a_s^* = a^*/2$ $b_s^* = b^*/2$
Fig. 8	$a_s = 3a$ $b_s = b$	$a_s^* = a^*/3$ $b_s^* = b^*$
Fig. 9	$a_s = 4a + 2b$ $b_s = -2a + 2b$	$a_s^* = a^*/6 + b^*/6$ $b_s^* = -a^*/6 + b^*/3$

The vectors defining the mesh of the surface structure are expressed in terms of the underlying mesh as shown in Table II, illustrated by Figs. 7-9.

Shorthand terms for the mesh defined by vectors parallel to those of the substrate mesh are "2x2" (or simply "2") for Fig. 7 and "3x1" for Fig. 8. If a centered mesh is chosen, this shorthand term should be preceded by *c*. A *c* (2x2) surface structure as well as (1x2) and (1x3) structures are illustrated in photographs of ball models in a review paper¹ in *Science* by MacRae (1963). The shorthand term is sometimes put in parentheses to separate it from neighboring symbols or text.

FIG. 6. The "00 reflection."

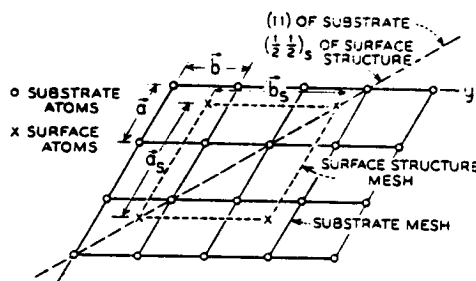
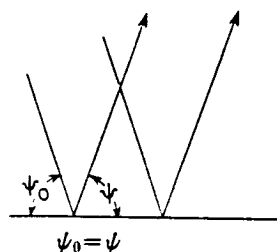


FIG. 7. A 2x2 surface structure.

For full identification, the shorthand term should be preceded by the name of the substrate and the orientation of the substrate surface and followed by the name of the deposit, if a deposit is present. Examples: Si(100)4, a clean (100) silicon surface with a diperiodic selvedge structure in which the mesh edges are parallel to those in the (100) plane of the bulk crystal, but four times as long in both *x* and *y* directions; Si(100)12-P, a (100) silicon surface with phosphorus on it resulting in a diperiodic surface structure in which the mesh edges are parallel to those in the (100) plane of the bulk silicon crystal but twelve times as long in both *x* and *y* directions; Ni(011)3x1-0, an (011) surface of nickel with oxygen on it, resulting in a diperiodic surface structure in which the mesh edges are parallel to those in the (011) plane of the bulk nickel crystal, with *a*, the same length as *a* in the (011) plane of the nickel crystal and *b*, three times the repeat distance in the (011) plane normal to *a*.

In some cases the mesh of the surface structure may have two or more orientations in various parts of the

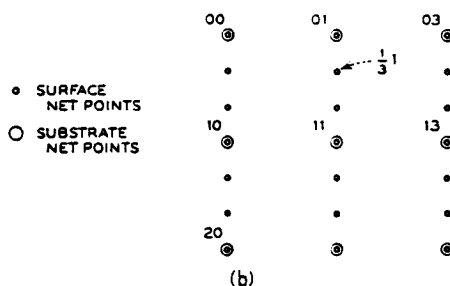
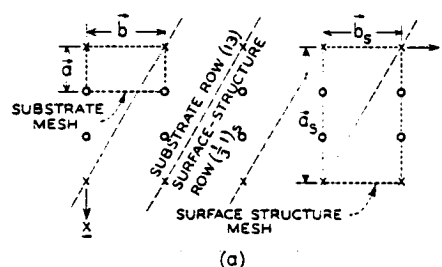


FIG. 8. A 3x1 surface structure. (a) real-space; and (b) reciprocal-space diagrams.

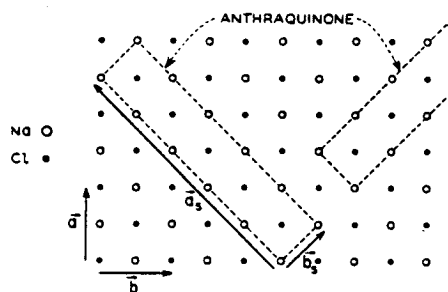


FIG. 9. Diagram of the orientation of anthraquinone on the (100) face of sodium chloride.

specimen because these orientations involve symmetrically equivalent directions of the substrate structure. An example (suggested by Dame Kathleen Lonsdale) is the occurrence of anthraquinone on the (100) face of sodium chloride in the orientations shown in Fig. 9. Here $a_s = 2\frac{1}{2}a \pm 2\frac{1}{2}b$ and $b_s = \frac{1}{2}a + \frac{1}{2}b$, where a_s represents the a lattice vector of the pseudo-orthorhombic lattice of monoclinic anthraquinone and b_s represents the monoclinic b lattice vector. A shorthand designation for this situation might be $\text{NaCl}(100)5 \times 1 - 45^\circ - A$, but would have to be defined in vector terms at the beginning of any discussion.

For Fig. 10, Lander prefers the designation "2\3" to indicate that both of the surface mesh dimensions bear this relation to the substrate mesh edges. Any such shorthand designation should be explained, if used, and the unambiguous vector definition exemplified in Table II should also be given.

Since the rows of the surface structure derive their Miller indices from the substrate net, fractional Miller indices commonly occur, as illustrated in Figs. 7 and 8. In Fig. 10, the rows which would have Miller indices (01) on the surface net would have Miller indices $(\frac{1}{2} \frac{1}{2})$ on the substrate net. Row (10) on the surface net would be $(\frac{1}{2} \frac{1}{2})$ on the substrate net. Row (11) on the surface net would be $(0\frac{1}{2})$ on the substrate net.

Since fractional indices are inconvenient in structure computations, it will generally be so that in the later stages of analysis the author will wish to change to a more convenient mesh, as indicated below.

STRUCTURE DESCRIPTION

The description of the size and orientation of the diperiodic net does not tell us where the atoms are any more than does the description of the triperiodic crystal lattice. The symbols and units for describing atom positions within the unit mesh are given in Table I. The z coordinate (normal to the surface) cannot be expressed in fractions of a cell edge as it is in the triperiodic case since the surface structure lacks periodicity in this direction. It should therefore be expressed in angstrom units above (away from the substrate) or below some arbitrary

surface designated by the author of the paper describing the structure.

The choice of an origin (upper left corner of the mesh in Fig. 2) is arbitrary. In Fig. 10 two choices are shown, one with the origin at an atom in the surface structure, the other with the origin superposed over that of the substrate net. Although the latter choice would facilitate description in terms of the substrate net, the former choice, or some other, might facilitate description in terms of published diperiodic-group positions which are discussed below. Indeed an author might therefore shift from one to the other in different parts of a paper, but should, of course, clearly describe the relationship between the two choices.

DIPERIODIC GROUPS

The 230 space groups published in the *International Tables*³ have proven an essential aid to the determination of triperiodic structures, although the simple structures that were first determined were determined without reference to them.⁴ Simple diperiodic structures have been determined without the aid of diperiodic groups, but the need for consideration of diperiodic groups will probably be felt as more complicated surface structures are considered.

The seventeen strictly two-dimensional plane groups have been published in the *International Tables*.³ They do not include any symmetry elements which would involve admitting the existence of the third dimension such as, for example, a twofold axis lying in the diperiodic plane.

There is another set of plane groups, the 80 diperiodic groups in three dimensions, mentioned on page 56 of the *International Tables*, which do include such elements, while still excluding any element which requires periodicity normal to the plane, such as a screw axis normal to the plane. These were recognized many years ago⁵ and have now been arranged by the writer⁶ in the form used in the *International Tables*. It is anticipated that they will be included in a future volume of the *Tables*.

Which set of diperiodic groups is appropriate for surface structures? Certainly the structures are not strictly planar: the atoms of the selvedge in many cases do not all lie in the same plane. But would an atom above some plane (parallel to the surface) be symmetrically related to an atom on the other side of the plane? Strictly

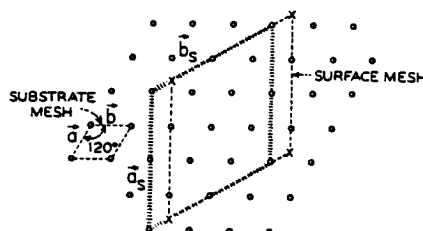


FIG. 10. A surface net not parallel to the substrate net.

speaking the atoms could not be symmetrically equivalent since one is closer to the substrate than the other and therefore in a different force field. From this point of view one would say that only the seventeen strictly two-dimensional space groups would be useful. However, it is frequently so, in triperiodic crystallography, that the symmetry of a crystal structure closely approximates a symmetry that is higher than its true symmetry and that the use of the higher-symmetry space group is of great help in determining the structure. From this point of view one would say that the 80 diperiodic groups in three dimensions are likely to be useful in the solution of diperiodic surface structures. Their application to this field was suggested to the writer by A. L. Patterson.

NOTE CONCERNING SURFACE SYMMETRY

A plane or axis of symmetry of the substrate crystal that is normal to the surface in question is of course a plane or axis of the substrate surface, but planes and axes of the substrate crystal that are not normal to the surface are not. Note that this means that in any substrate surface that does not have an n -fold axis normal to it with n even (as, for example, the (111) surface of any cubic crystal) any direction $[u_1v_1]$ is not symmetrically related to the opposite direction $[\bar{u}_1\bar{v}_1]$.

COMPARISON OF EFFECTS OF SURFACE SYMMETRY IN MUSCOVITE

An example of this in the a direction in muscovite and other micas is shown by the excellent photographs of etch pits recently published by Patel and Ramanathan.⁷ The outlines of the pits on two sides of a thin muscovite flake, traced from their Fig. 9(b) (1962) are shown in Fig. 11(a). Note that the shape of each pit has the mirror plane m which is normal to the cleavage surface (the cleavage is parallel to the paper) but not the twofold axis parallel to b . The two pit-shapes are related to each other by the twofold axis, since the $+a$ direction, $[100]$, on the top surface is thus related to the $-a$ direction, $[\bar{1}00]$, on the bottom surface. The "bulging side" of the rhomb-like pits is directed toward the acute angle of the monoclinic lattice [Fig. 11(b)] as determined by the writer of the present paper from x-ray diffraction patterns from muscovite etched with hydrofluoric acid.

When ammonium iodide is deposited epitaxially on a cleavage surface of muscovite it is oriented as shown in Fig. 11(c). The deposited crystal *does not* have a mirror plane normal to b as the substrate structure and its etch pits do. The epitaxial deposition of ammonium iodide on mica will be further discussed in another paper.

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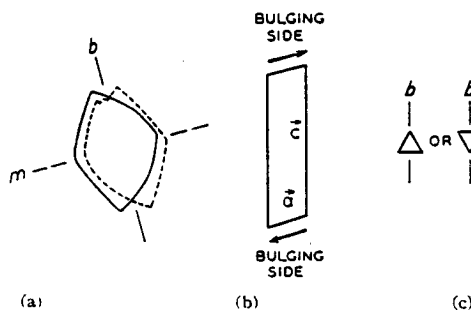


FIG. 11. (a): Diagram of etch pits on opposite cleavage surfaces of muscovite mica [traced from Fig. 9(b), Patel and Ramanathan,⁷ 1962]; (b): Relation between etch-pit shapes and crystallographic directions in muscovite; (c): Relation of epitaxially deposited ammonium iodide crystals to the b direction in the cleavage face of muscovite.

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