Mines Branch Information Circular IC 201

DESCRIPTION AND USE OF AN ION BOMBARDMENT CAMERA AND ANCILLARY EQUIPMENT

by

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ABSTRACT

This paper describes an ion bombardment camera and ancillary equipment consisting of a circular protractor, an orienting sphere, and an optical projector capable of directly producing stereographic and gnomonic projections from ion bombardment ejection patterns.

A unique feature of the equipment described is that it leads to a great simplification of several crystallographic techniques used widely in theoretical metallurgy, material sciences, physics, ceramics and crystal chemistry. It is proposed that the techniques described are also suitable for the teaching of elementary crystallography.

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Direction des mines

Circulaire d'information IC 201

DESCRIPTION ET USAGE D'UN APPAREIL À BOMBARDMENT IONIQUE ET DE SES ACCESSOIRES

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RÉSUMÉ

La présente étude contient la description d'un appareil à bombardement ionique et de ses accessoires, comprenant un rapporteur à limbe complet, une sphère qui permet de déterminer l'orientation des cristaux, et un projecteur optique capable de donner directement des projections stéréographiques et gnomoniques en partant des diagrammes d'éjection résultant du bombardement des cristaux.

L'appareil décrit ci-dessous possède une caractéristique unique qui permet de simplifier grandement plusieurs techniques cristallographiques utilisées en métallurgie théorique, sciences des matériaux, physique, céramique, et cristallographie des composés chimiques. Les techniques décrites pourraient être employées pour l'enseignement de la cristallographie élémentaire.

* Préposé au recherches scientifiques et ** Agent technique, Division de la métallurgie physique, Direction des mines, ministère de l'Énergie, des Mines et des Ressources, Ottawa, Canada.

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A. INTRODUCTION

In addition to its applications as a useful metallurgical tool in etching and surface studies, ion bombardment makes possible several simple experiments which rapidly provide a familiarity with cubic crystal structures. They are based on the phenomenon of the ejection of atoms from metallic single crystals during ion bombardment with energetic inert-gas ions. The fundamental advantage of the method for instructional purposes, over X-ray and electron diffraction and optical methods, is that a limited number of crystallographic directions, rather than a large number of reflections from atom planes, are experimentally recorded. The polar recording of the crystallographic directions, as obtained with the equipment described, greatly simplifies interpretation for students being introduced to crystallography. Furthermore, results are rapidly recorded in 0.5 to 5 minutes without the use of photographic processes.

B. THE PHENOMENON OF ION BOMBARDMENT

The phenomenon responsible for directional ejection from bombarded surfaces is generally believed to be due to penetration of high-speed particles into the specimen and subsequent transfer of momentum back to the surface atoms by impact between atoms along close-packed rows. The only restriction is that the energy imparted outwardly to the surface atom must be above a threshold value characteristic of the crystal. The directions of these close-packed rows are clearly recorded on collectors (Figure 1) by a visible pattern of spots composed of atoms ejected from the crystal during bombardment.

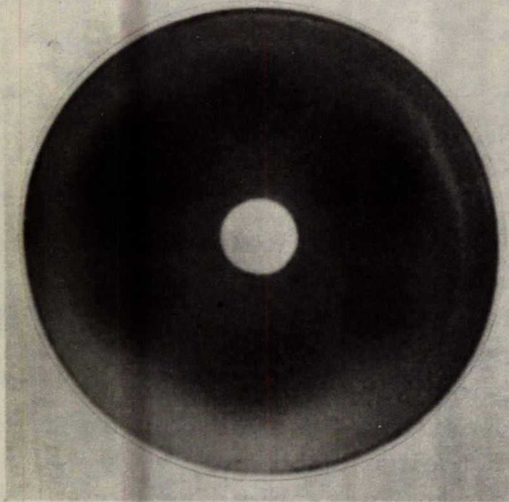


Figure 1(a)

A pattern composed of ejected atoms on a hemispherical collector, obtained from a face-centered cubic (f.c.c) metal single-crystal bombarded vertically with 8 KeV argon ions. It consists of three heavy [110] spots and three lighter spots, presumably from surface twins.

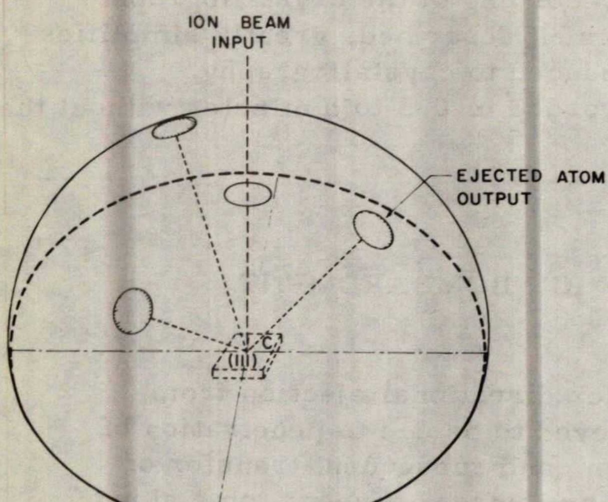


Figure 1(b)

Schematic representation of relationship between specimen, collector, and ion beam. The three spots, symmetrically placed around the normal to the crystal surface, indicate that the crystal is cut parallel to the (111) planes.

Wehner's original suggestion that ejection along the closest-packed directions, viz., [110] in face-centered and [111] in body-centered cubic metals, was due to momentum transfer alone is generally accepted. It has been postulated that in other close-packed ejection directions a mechanism of momentum transfer plus an atomic replacement sequence occurs. These two types of mechanisms are illustrated in Figure 2 for ejection along [110] and [100] directions in f.c.c. crystals. For propagation of momentum along the close-packed [110] directions in f.c.c. crystals, no replacement of atoms occurs, because impacts take place before the atoms have been displaced half their inter-atomic spacing in that direction and they then return to their original positions, as is indicated by the folded arrows. Momentum transfer along [100] directions within the crystal is accompanied by a series of replacements because a displaced atom, having passed the center point before impact, cannot return to its original site and moves into the vacated site of the atom it has just displaced. It should be noted that an atom displaced along [100], at the midpoint of its displacement, is surrounded by a symmetrical ring of four atoms, only two of which, A and B, can be shown in the diagram. This symmetrical ring helps to focus the displaced atom along the [100]direction. Similarly, symmetrical conditions are present in the case of [111]propagation in f.c.c. crystals, where the displaced atoms must pass through two three-atom rings to reach the next atom site. The postulated mechanisms are summarized in Table 1.

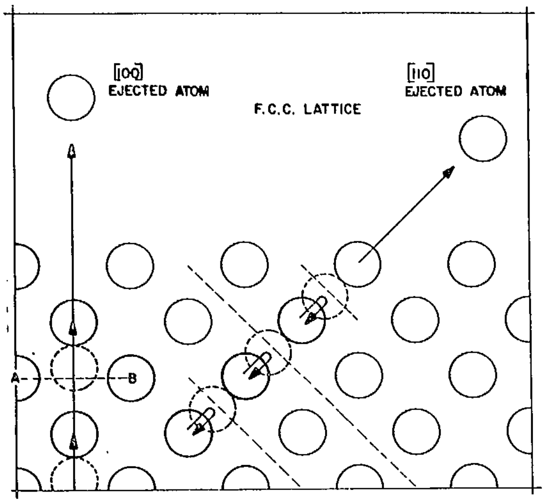


Figure 2.

A face-centered cubic structure sectioned to reveal [110]and [100] directions. Propagation of momentum required for ejection along [110] involves momentum transfer only. However, for [100]ejection, propagation of momentum is accompanied by a transfer of atoms along the row by one atomic spacing.

TABLE 1

Postulated Mechanisms Within the Crystal Responsible for Ejection Along the Three Most Closely-Packed Directions in fcc and bcc Crystals

FACE-CENTERED CUBIC			BODY-CENTERED CUBIC		
Direction	Spacing	Mechanism	Direction	Spacing	Mechanism
[110]	$\frac{a}{\sqrt{2}}$ (.707a)	Momentum transfer only.	[111]	$\frac{\sqrt{3}}{2}$ a (.866a)	Momentum transfer only.
[100]	a	Momentum transfer plus replacement sequence.	[100]	a	"
[111]#	$\sqrt{3}$ a (1.732a)	"	[111]	$\sqrt{2}$ a (1.414a)	Momentum transfer and replacement sequence.

#This direction is rarely recorded.

Patterns from f.c.c. metals will, after light bombardment, usually contain only the family of [110]ejection spots. In some instances, heavier bombardment brings out [100] spots and an additional family of spots close to [611]. These latter are the weaker spots recorded in Figure 1(a) and cannot be accounted for by the mechanisms mentioned previously, because [611]rows are far from being close-packed and direct collision between atoms along these rows would not be possible. It is interesting to note that [611]directions lie only 6° from [411] directions and that the latter are parallel to [110]directions in f.c.c. twins. The parent-twin relationship for f.c.c. is shown in Figure 3. Surface twinning may be the cause of these spots, although alternative theories have been proposed.

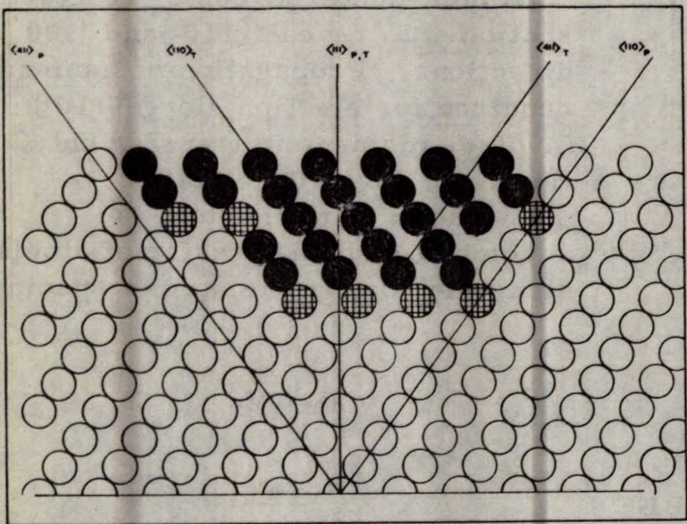


Figure 3.

A face-centered cubic crystal sectioned on a (110) plane and showing the relationship between a twin and its parent. Open circles represent atoms in parent, full circles atoms in the twin, and shaded circles atoms belong to both twin and parent. Note parallelism between [110] of twin and [411]of parent, and vice versa, and that [111]is common to both parent and twin.

Patterns from b.c.c. metals usually contain [111]and [100] spots, which correspond to the two most closely packed directions in these metals. [111] ejection spots are sometimes severely weakened, or even replaced, by satellite spots which, as in the case of f.c.c. metals, may be due to surface twinning. The [100]spots are, therefore, the most reliable in b.c.c. patterns. In general, b.c.c. patterns are inferior to f.c.c. patterns. In the experience of the authors, tungsten single crystals give the best b.c.c. patterns.

C. ION BOMBARDMENT EQUIPMENT

I. The Camera

I(a) Description

A diagram of the ion bombardment camera, which should be connected to a clean vacuum system, is shown in Figure 4. It contains an ion source of the glow-discharge type with a large output. The glow-discharge region and the bombardment chamber are separated by the thin, easily replaceable, 0.25-mm aluminum cathode (A) in contact with the sealing O-ring (B), and pierced with a central hole of about 0.5-mm diam. The hole at the upper end of the cylindrical aluminum anode (C) is slightly tapered to accommodate a short length of polyethylene tubing used as a vacuum seal between the drawndown glass capillary(s) and the anode. The supply gas, controlled by a pressure regulator (not shown) and passing through a needle valve (U) and the capillary, enters the glow-discharge region from a 1-mm axial hole at the lower end of the anode, the sharp edge of which helps to concentrate the glow discharge opposite the hole in the cathode for maximum output; the outer edge of the anode is slightly rounded for the same reason. To prevent attack on the plastic near the glow discharge, the glass sleeve (D) surrounds the anode and is supported by the O-ring (E). It is necessary that this sleeve contact the plastic at its upper end and the cathode at its lower end.

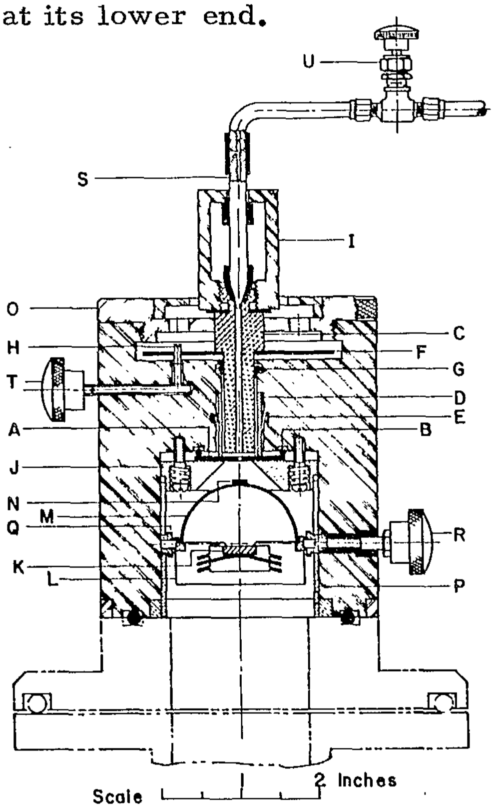


Figure 4.

Cross-section of ion bombardment camera. (A) aluminum cathode, (B) cathode sealing O-ring, (C) aluminum anode, (D) glass sleeve, (E) supporting O-ring, (F) copper cooling disk, (G) anode sealing O-ring, (H) anchor pin, (I) plastic fitting, (J) spring-loaded copper disk, (K) single-crystal specimen, (L) combined specimen holder and collector ring, (M) hemispherical collector, (N) brass retaining band, (P) supporting framework, (Q) insulating bushings, (R) tilting knob, (S) glass capillary, (T) insulated high voltage terminal post, (U) needle valve.

The anode-cathode distance is adjusted, without rotation of the anode, by turning a threaded cover (O) which supports it. The anode carries the copper cooling disk (F) which eliminates the possibility of the anode O-ring (G) becoming overheated. Air cooling of the copper disk is achieved by a series of holes in the cover. A potential of several kilovolts is supplied, at the terminal(T), to the anode through its cooling disk by contact with the anchor pin (H), used to prevent the anode assembly from rotating. Electrical contact with the anode is avoided by making the upper gas input fitting (I) of plastic. For operating safety the power supply should be of the low-current-capacity RF type.

The pressure necessary to maintain the glow discharge is some tens of microns, while the pressure in the bombardment chamber must be sufficiently low so that an ejected atom has a high probability of reaching the collector without collision with gas molecules, viz., a micron or less. At one micron, the mean free path for argon is about 4.5 cm. The low pressure in the specimen chamber is obtained by rapid pumping of the system so that the necessary pressure drop along the hole in the cathode is obtained.

It is not possible to obtain directional ejection from metallic surfaces when they are covered with an oxide. Hence, before bombarding easily oxidized metals, such as aluminum, it is necessary to be sure that air has been removed from all parts of the camera. Since it is not possible to rapidly pump down the glow-discharge region through the small cathode hole, the cathode is temporarily held away from the sealing O-ring (B) by pressing down on the plastic fitting (I) at the upper end of the anode for a short period.

The bombardment chamber contains the single crystal specimen (K), which may be as large as 18 x 18 x 6 mm; the combined specimen and collector holder (L); the hemispherical collector (M), cut from a ping-pong ball; its retaining band (N); and the supporting framework (P), which also serves to ground the cathode. All but the latter are insulated from ground by bushings (Q), so that a microammeter may be connected between the exposed shaft of (R) and ground in order to monitor specimen current (which should be at least 50 microamperes for rapid results). The knob (R) is used to tilt the assembly for inspection of the pattern at any time during bombardment. The collector is protected from the ion beam, during inspection periods, by the brass band N.

The use of hemispherical collectors, with the specimen at the center of curvature, ensures that all ejecting directions are recorded. Since the ejected atoms arrive in directions normal to the collector, an accurate estimation of the center of gravity of each spot can be made. These are then carefully marked with pinholes. Spots from some metals tend to fade and in these cases the pinholes also ensure a permanent record. Hemispheres are accurately cut from ping-pong balls with a sharp blade or jeweller's saw by using the jig shown in Figure 5(a). The center of the hole for the input beam marks the direction of the normal to the bombarded surface and must be accurately punched at the top of the hemisphere in the jig shown in Figure 5(b).

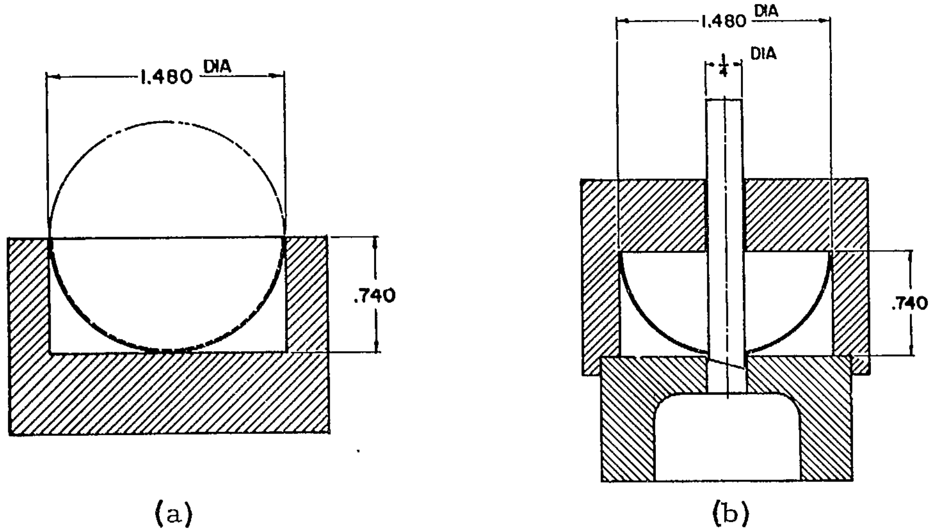


Figure 5. (a) Jig used for cutting hemispheres from ping-pong balls.

(b) Jig used for punching hole marking normal to surface and through which the ion beam passes.

I(b) Practical Operating Ranges

Voltage	-	7-10 kV
Current	-	25-75 microamperes
Pressure	-	0.25-0.75 micron of argon
Time	-	0.25 to 5 minutes

It is important not to bombard the sample for too long, because the spots become broader as background increases, thus decreasing the accuracy of angle determinations. Frequent inspection of the pattern during bombardment is required. Bombarding time depends on the crystal used and its surface condition. For instance, a (111) silver sample which has been chemically etched so as to remove all its cold-worked layer would require only 15 seconds bombardment at 8 kV, 50 μa and a pressure of 0.5 micron. If insufficiently etched, the same sample would require 5-10 minutes bombardment.

Single crystals of a number of metals are available commercially. However, single-crystal ingots from which single crystals of desired orientation must be cut may be grown in the laboratory by slowly withdrawing from a furnace a molten charge contained in a long boat. Lead, having a low melting point and not requiring a vacuum, is probably the easiest to prepare and gives a good pattern. All cold-worked material must be removed from cut surfaces by careful etching and electrolytic or chemical polishing.

D. CRYSTALLOGRAPHIC PROCEDURES

I. Use of the Circular Protractor for Measuring Angles Between Crystal Directions

The angles between directions recorded on the collector may be easily determined by the use of a circular protractor (Figure 6) with an opening in the centre to fit the ping-pong ball and a small anchor pin at 0° to fit into one of the pinholes marking an ejection direction. By the use of Table 2, which contains only pertinent angular relationships, it is possible to readily identify the directions recorded.

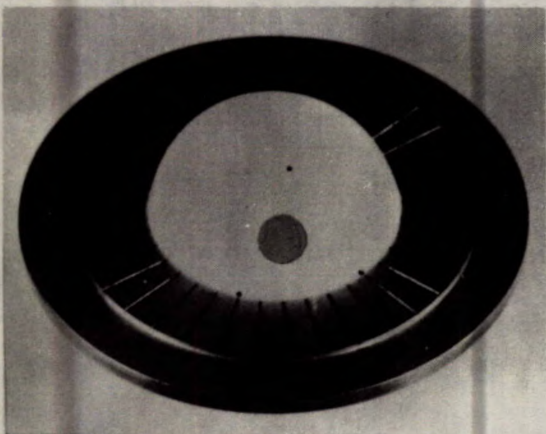


Figure 6.

Circular protractor with white hemispherical collector positioned for measurement of angle between two pinholes which mark positions of centers of ejection deposits on inner surface of collector. By reference to Table 2, indices of ejecting directions may be determined.

TABLE 2

Directions of Interest in Patterns from the Cubic System and Approximate Angles Between Pairs

Face-centered cubic pattern				Body-centered cubic pattern			
Measured Angles ± 1°			Direction Pairs	Measured Angles ± 1°			Direction Pairs
36°	144°		[110] - [111]	36°	144°		[110] - [111]
		or	[110] - [116]	45°	135°		[100] - [110]
45°	135°		[100] - [110]	55°	125°		[100] - [111]
60°	120°		[110] - [110]	60°	120°		[110] - [110]
78°	102°		[116] - [116]	70°	110°		[111] - [111]
77°	103°		[110] - [116]	90°			[100] - [100]
90°			[110] - [110]			or	[110] - [111]
		or	[110] - [111]				

In the cubic systems, directions are also normals, or poles, of planes of the same indices, and thus it becomes possible to determine the angle between certain planes.

II. Use of Orienting Spheres for Determining Orientation of a Crystal

Ejection patterns formed on hemispherical collectors are suitable for making orientation determinations by direct contact with an illuminated sphere on which are engraved the ejection directions, viz., principal directions of cubic metals. The center of the hole, cut accurately at the top of the hemispherical collector, marks the direction of the normal to the specimen surface. With care in marking the centers of the deposits with pinholes, it is possible to obtain an accuracy in orientation determination of 2-3°.

II(a) Orientation in Terms of Angle Between the Surface Normal and the Principal Directions Defining the Unit Triangle

For determination of orientation by this method, use the sphere, shown in Figure 7, with calibrating circles centered at the corners of the basic spherical triangle defined by the three lowest index directions [001], [101] and [111]. These directions are indicated on the sphere by □, ○ and △ respectively.

Figure 7.

Hemispherical collector on illuminated orienting sphere engraved with principal directions of cubic metals. Calibrating circles at 10° intervals are drawn from the corners of the basic triangle.

Shift the collector over the surface of the sphere until the best correspondence is obtained between the pattern spots and the engraved directions. The angles between the center of the hole, marking the normal to the surface, and the engraved directions may then be read off from the sphere by using the calibrating circles engraved at 10° intervals. Any two of these angles are sufficient to completely describe the orientation, as may be seen by examination of the basic triangle in Figure 8.

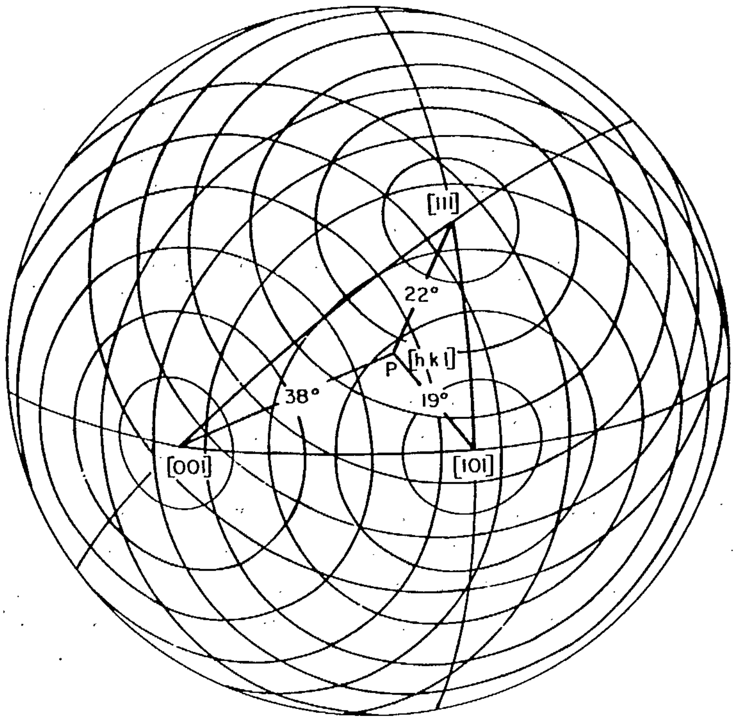


Figure 8.

P [h k 1] indicates the position of the normal to a specimen surface. It lies at approximately 38° to [001], 19° to [101], and 22° to [111].

II(b) Orientation in Terms of Angles Between Surface Normal and the Orthogonal Co-ordinate Axes of a Cube

For this determination, the sphere with the calibrating circles centered on the cubic directions [001], [010] and [100] is used. The advantage of this method over the one previously described is that the cosines of the angles measured are directly proportional to the Miller indices of the surface normal. The collector is shifted over the surface of the sphere to give the best correspondence of its spots with the engraved ejection directions on the sphere, indicated as before by □, ○ and △. It is then possible to read off the angles which the surface normal makes with the three cubic directions [001], [100] and [010], by using the calibrating circles.

Figure 9 illustrates the angular relationship between a direction P [h k 1] and the three cubic directions. In the example given it is found to make angles of 38°, 58°, and 74°.

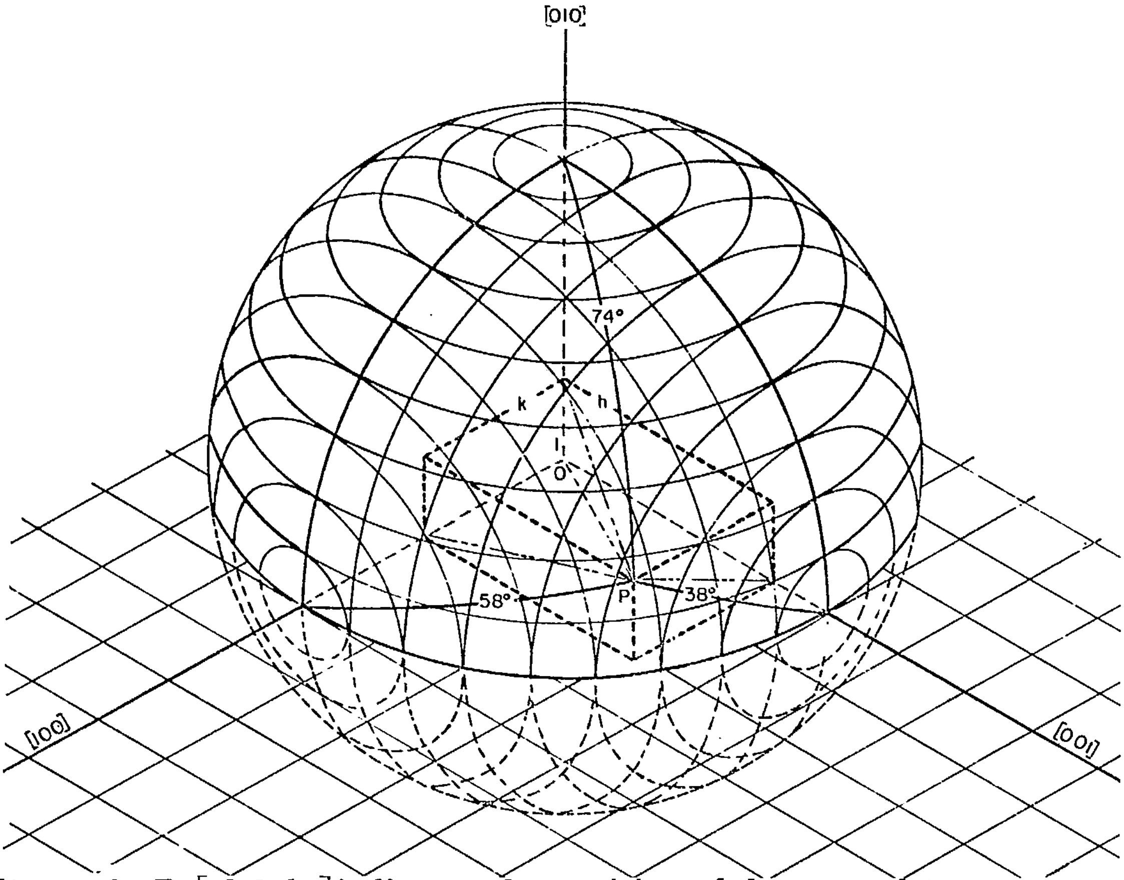


Figure 9. P [h k l] indicates the position of the normal to a specimen surface. It lies at 38° to [001],58° to [100], and 74° to [010] . The components of the vector OP are h, k, and l.

The three components of the direction [h k l] may be determined by taking cosines of the angles 38°, 58° and 74° whose values are 0.798, 0.530 and 0.276. These values are proportional to the Miller indices, which are always expressed as whole numbers. Hence the Miller indices of P [h k l] are [798 530 276]. However, for practical purposes a simpler relationship of the indices to each other should be sought. From inspection, 798: 530: 276 is approximately equal to 3:2:1. Hence, the Miller indices of direction P [h k l] are close to [3 2 1].

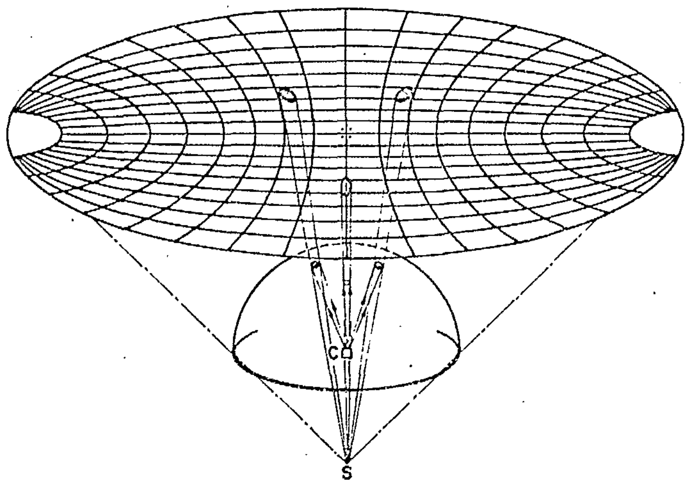
Having determined the indices of the direction in the above manner, it is a simple matter to determine δ , the angle this direction makes with any other direction [$h_1k_1l_1$], by applying the formula

$$\cos\ \delta = \frac{hh_1 + kk_1 + ll_1}{\sqrt{(h^2 + k^2 + l^2)\ (h_1^2 + k_1^2 + l_1^2)}} .$$

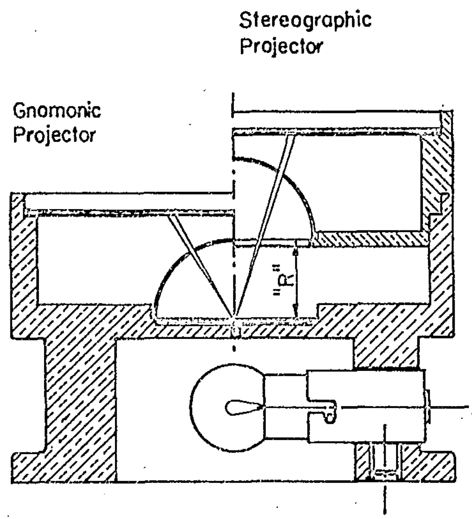
The direction [3 2 1] is found to make angles of 22°, 38° and 19° with the directions [111], [001] and [101] respectively. These correspond with the angles shown in Figure 8. Hence, [h k l] in Figure 8 is actually a [3 2 1] direction.

III. The Use of Stereographic and Gnomonic Projections

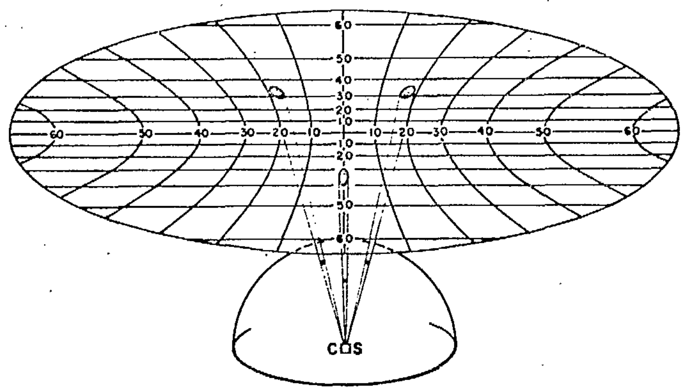
Stereographic and gnomonic projections are means of representing the angular relationships of directions in a three-dimensional crystal on a two-dimensional plane. Either type of projection can be made optically from the hemispherical collectors on which the ejection directions of an ion-bombarded crystal have been directly recorded. The stereographic projection is by far the most common and all possible directions can be recorded. In the gnomonic projection the angular range is limited. In Figure 10, (a) and (b) illustrate the geometry of the two types of projections and (c) illustrates a practical projector.



(a)



(c)



(b)

Figure 10.

(a) Schematic representation of the production of a stereographic projection directly from the collector by placing light source S at bottom of diameter. (b) Schematic representation of the production of a gnomonic projection by raising light source to equatorial plane. (c) Dual-purpose projector for producing stereographic and gnomonic projections directly from a hemispherical collector.

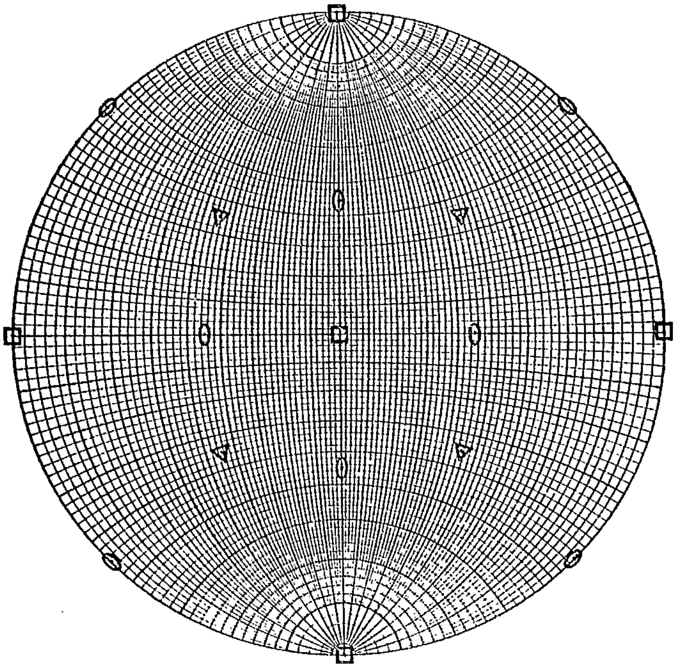
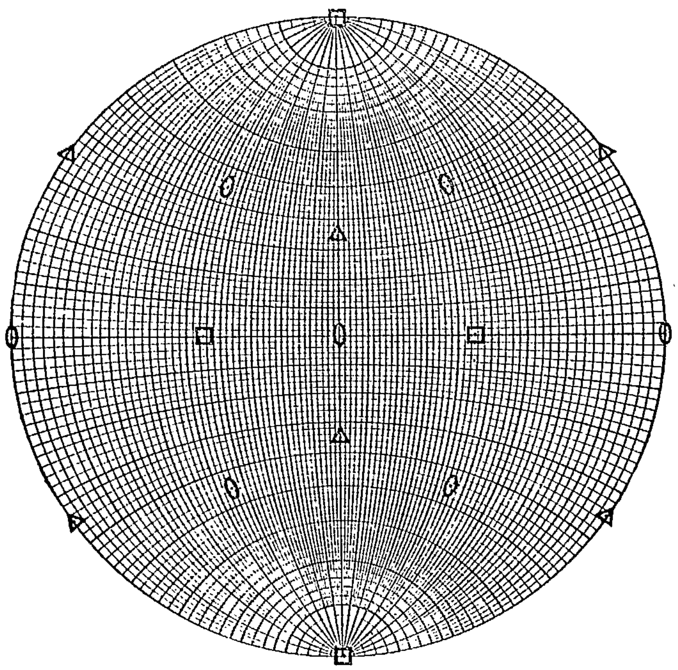


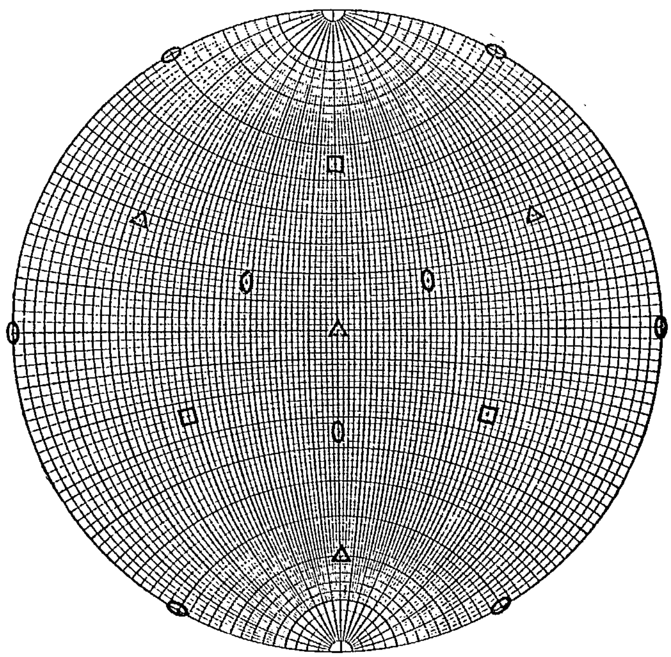
Figure 11.

Stereographic nets with [001], [011] and [111] poles indicated by □ , ○ and △ respectively.

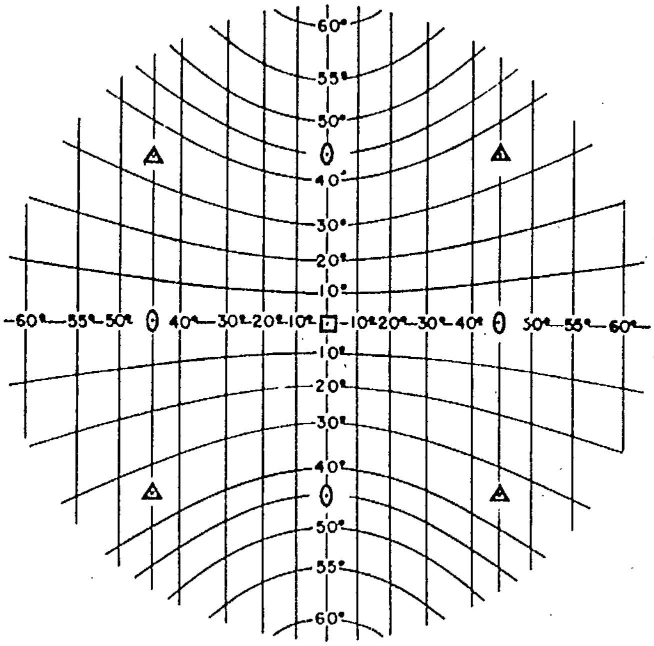
(a) Standard [001] stereographic projection for cubic crystals.



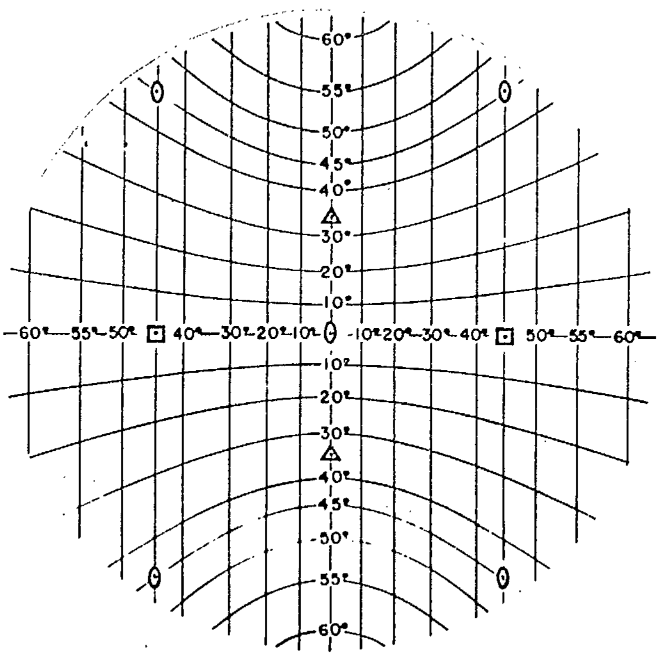
(b) Standard [011] stereographic projection for cubic crystals.



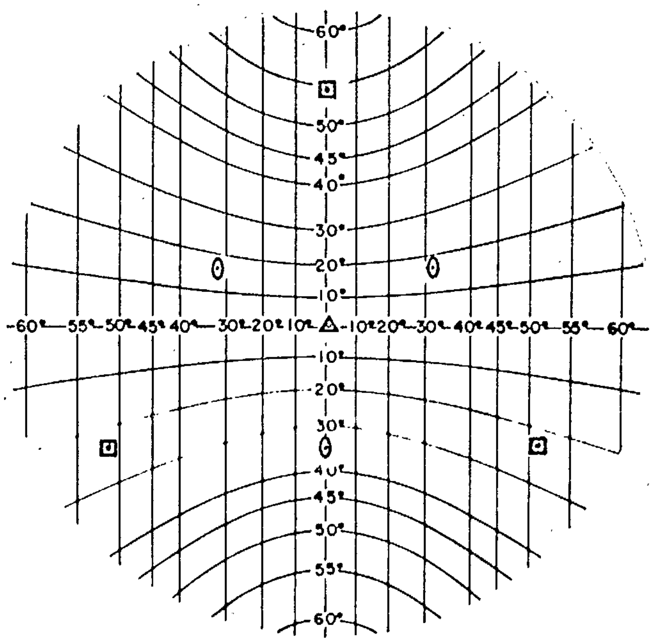
(c) Standard [111] stereographic projections for cubic crystals.



(a) Standard [001] gnomonic projection for cubic crystals.



(b) Standard [011] gnomonic projection for cubic crystals.



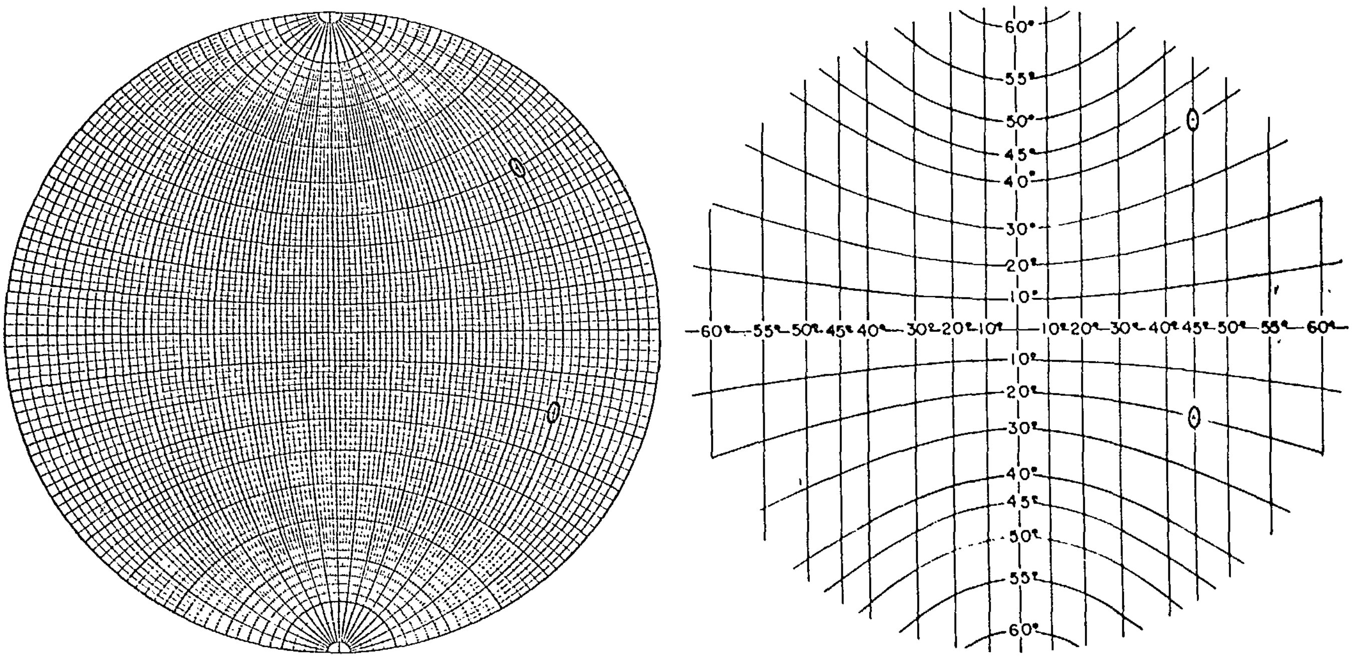
(c) Standard [111] gnomonic projection for cubic crystals.

Figure 12.

Gnomonic nets with [001], [011] and [111] poles indicated by □, ○ and △ respectively.

III. (a) Determination of Angles Between Crystal Directions, Using Stereographic and Gnomonic Nets

Since angles between any two points on the surface of a sphere must be measured along great circles, it follows that any measurements of angles using projections must be made on projections of great circles. In the stereographic projection the meridianal lines, running from pole to pole, are projections of great circles. In the gnomonic projection the straight lines are projections of great circles.



(a) The Stereographic Projection. (b) The Gnomonic Projection.

Figure 13. For determining the angles between any two directions, these must be marked on tracing paper and rotated until they fall on common great circle projections. In both (a) and (b) the two directions shown are 60° apart.

III. (b) Determination of Orientation of Crystals, Using Stereographic and Gnomonic Nets

Having carefully pierced the hemispherical collectors at the center of all the [110] deposits, stereographic and gnomonic projections can now be made with the projector. Stereographic projections are the most useful, since all angles up to 90° to the normal can be recorded on a net whose diameter is twice that of the collector. A gnomonic projection using the same size net can only record angles up to 63° from the surface normal, and a net of infinite size would be required to record angles of 90° from the

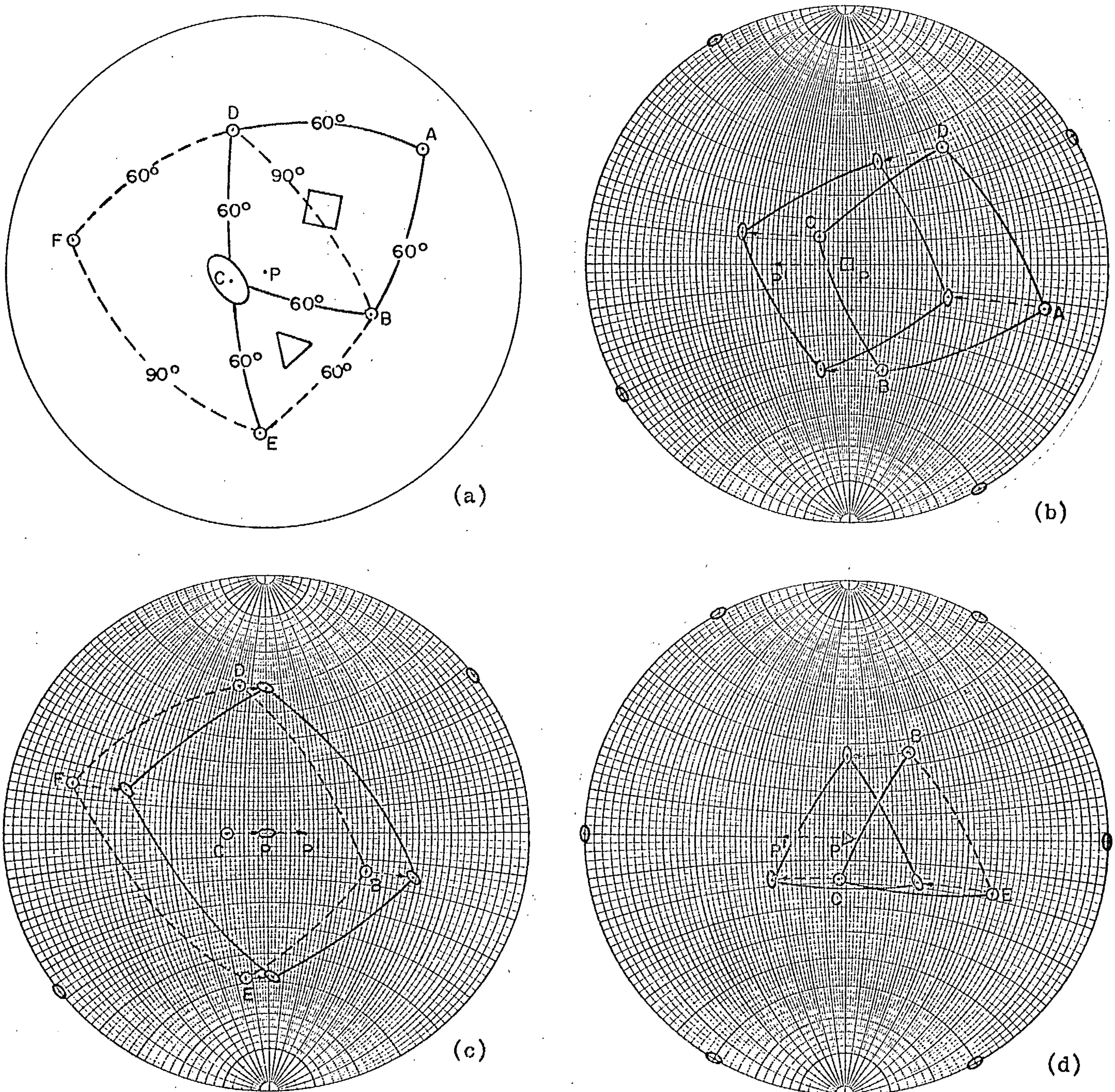


Figure 14. Stereographic projections.

(a) A stereographic projection of an ejection pattern from a f.c.c. single crystal. Angles between directions have been measured and are indicated on the projection. (b), (c) and (d) Standard [001], [011] and [111] stereographic projections for cubic crystals upon which directions from the pattern shown in (a) were shifted along latitude lines until they coincided with directions of the standard projections. Shifts of 32°, 17° and 29° were made on the [001], [011] and [111] nets respectively, indicating that the pole of the crystal surface was at those angles to the three principal directions.

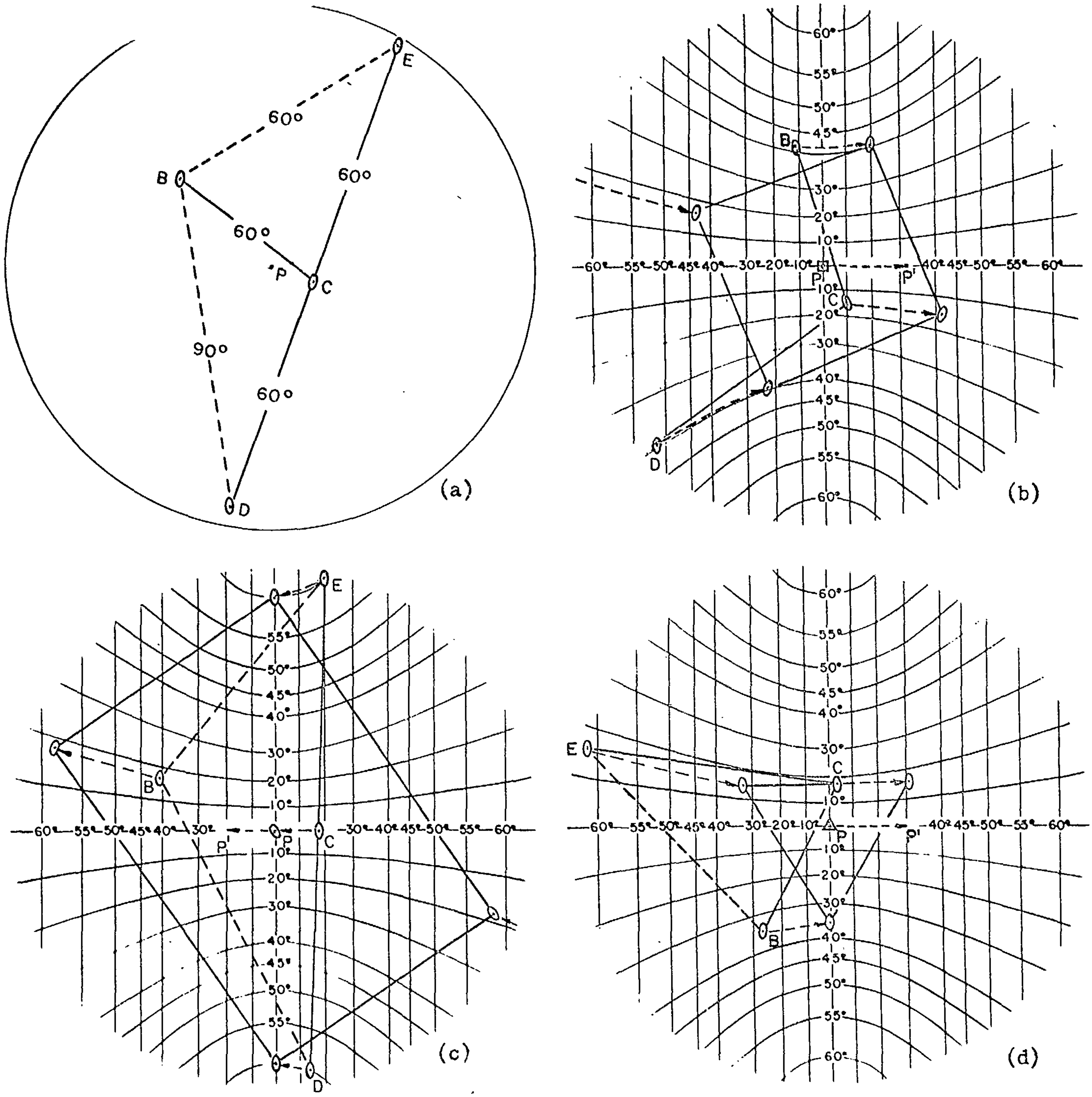


Figure 15. Gnomonic projections.

(a) Gnomonic projection of an ejection pattern from a f.c.c. single crystal whose stereographic projection was shown in the previous figure. Angles between directions have been measured and are indicated on the projection. (b), (c) and (d) Standard [001], [011] and [111] gnomonic projections for cubic crystals upon which directions from the pattern shown in (a) were shifted along latitude lines until they coincided with directions of the standard projections. Shifts of 32°, 17° and 29° were made on the [001], [011] and [111] nets respectively, indicating that the pole of the crystal surface was at those angles to the three principal directions.

normal. The gnomonic net is therefore seldom employed. Angles between [110] directions should be measured and the pattern carefully examined for symmetry. For example, the four [110] type directions A, B, C and D on the stereographic projection (Figure 14(a)) outline a surface on a sphere with four 60° sides, indicating that a 4-fold symmetry axis (viz. [001]) lies at its center of symmetry. This may be confirmed by examination of Figure 11(a). Similarly, B, E, F and D outline a surface on a sphere with two opposite sides of 60° and two of 90°. This indicates that an axis of 2-fold symmetry (viz. [011]) lies at its center, as confirmed by spot c. The directions B, C and E outline an equilateral spherical triangle with 60° sides; hence a 3-fold axis (viz. [111]) lies at its center.

After analyzing the pattern as described, it is most convenient to make a second tracing including only those [110] directions of interest for determining the angle of the surface normal relative to [001], [011] and [111]. A tracing is then superimposed on the stereographic projection of chosen orientation indicated in Figures 14 (b), (c) and (d), and rotated about its center until a position is found, with the help of a stereographic net, such that spots on the tracing and those on the stereogram fall on common latitude lines. The spots may then be made to coincide by shifting them along these latitude lines. The amount of shift, in degrees, should be the same for all spots and represents the amount the crystal should be rotated so that its stereographic projection coincides with any chosen standard projection. As a consequence, the normal to the surface, P, is shifted an equal amount to position P'. Figures 14 (b), (c) and (d) show the superposition on the standard stereographic nets of only those [110] directions of direct interest for each orientation.

The procedures for gnomonic projections, as shown in Figures 15 (a), (b), (c) and (d), are similar to those outlined above. Symmetry is not as obvious, as fewer spots are recorded due to the limited angular range of the gnomonic projection. It is therefore not as easy to use as the stereographic method, and is not recommended for beginners.

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