Standard Test Method for Determining the Orientation of a Metal Crystal[1]

This standard is issued under the fixed designation E 82; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the back-reflection Laue procedure for determining the orientation of a metal crystal. The back-reflection Laue method for determining crystal orientation **(1, 2)**[2] may be applied to macrograins **(3)** (0.5-mm diameter or larger) within polycrystalline aggregates, as well as to single crystals of any size. The method is described with reference to cubic crystals; it can be applied equally well to hexagonal, tetragonal, or orthorhombic crystals.

1.2 Most natural crystals have well developed external faces, and the orientation of such crystals can usually be determined from inspection. The orientation of a crystal having poorly developed faces, or no faces at all (for example, a metal crystal prepared in the laboratory) must be determined by more elaborate methods. The most convenient and accurate of these involves the use of X-ray diffraction. The "orientation of a metal crystal" is known when the positions in space of the crystallographic axes of the unit cell have been located with reference to the surface geometry of the crystal specimen. This relation between unit cell position and surface geometry is most conveniently expressed by stereographic or gnomonic projection.

1.3 The values stated in inch-pound units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens[3]

2.2 *Adjunct:*

Hyberbolic chart for solving backreflection Laue patterns (1 film positive)[4]

3. Summary of Test Method

3.1 The arrangement of the apparatus is similar to that of the transmission Laue method for crystal structure determination except that the photographic film is located *between* the X-ray source and the specimen. The beam of white Xradiation passes through a pinhole system and through a hole in the photographic film, strikes the crystal, and is diffracted back onto the film. Dark spots, which represent X-ray beams "reflected" by the atomic planes within the specimen, appear on the developed film. The atomic planes these spots represent are identified by crystallographic procedures and the orientation of the metal crystal is determined.

4. Significance and Use

4.1 Metals and other materials are not always isotropic in their physical properties. For example, Young's modulus will vary in different crystallographic directions. Therefore, it is desirable or necessary to determine the orientation of a single crystal undergoing tests in order to ascertain the relation of any property to different directions in the material.

4.2 This test method can be used commercially as a quality control test in production situations where a desired orientation, within prescribed limits, is required.

4.3 With the use of an adjustable fixed holder that can later be mounted on a saw, lathe, or other machine, a single crystal material can be moved to a preferred orientation, and subsequently sectioned, ground, or processed otherwise.

4.4 If grains of a polycrystalline material are large enough, this test method can be used to determine their orientations and differences in orientation.

5. Apparatus

5.1 *X-Ray Tube*—In order that exposure times be reduced to a minimum, the X-ray tube shall have a target that gives a high yield of white X-radiation. The tube voltage shall be near 50 kVp.

5.2 *Back-Reflection Laue X-Ray Camera*— The X-ray camera shall have (*1*) a pinhole system about 6 cm in length with

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[2] The boldface numbers in parentheses refer to the list of references at the end of this method.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

[4] Plate I is available from ASTM Headquarters. Order Adjunct: ADJE0082.

openings of ¼ to 1 mm, (*2*) a flat, light-tight film holder (the hole in the center of the film should be as small as possible, preferably about ⅛ in. (3.2 mm) in diameter), (*3*) a specimen holder, and (*4*) means for setting the crystal-to-film distance at 3.00 cm. These parts may be assembled in various ways depending upon the type of specimen being studied and upon the accuracy desired. The main requirement for accurate results is that the pinhole system shall be precisely perpendicular to the film holder and thus to the film. An aluminum sheet may be placed between the specimen and the film, preferably in close contact with the film, in order to filter much of the secondary X-radiation emitted by the crystal.

NOTE 1—Fig. 1 illustrates a back-reflection Laue camera constructed for use with metallic sheet specimens having grains with a diameter of 0.5 mm or larger. The specimen-to-film distance is fixed at 3 cm and the specimen surface is maintained perpendicular to the incident beam and parallel to the film.

NOTE 2—Fig. 2 illustrates a universal camera with a goniometer head, as adapted for back-reflection Laue studies. With this camera the interpretation of an unsymmetrical pattern may be verified rapidly by rotating the specimen to an angle for which a prominent pole is perpendicular to the film, so that a pattern of recognized symmetry is obtained.

6. Test Specimen

6.1 The test specimen may be of any convenient size or shape. Normally, the orientation will be determined with reference to a prepared surface and a line on this surface. Surfaces on metal crystals may be prepared by methods ordinarily used in preparing metallographic specimens (Note 3). After final polishing, the specimen shall be etched deeply enough to remove all polishing distortion. This surface shall be examined microscopically to make sure that the etch has removed all scratches or distorted metal. Strain-free surfaces of aluminum, iron, copper, brass, tungsten, nickel, etc., are easily prepared. Great care is needed in preparing surfaces on cystals of metals such as tin and zinc (or their solid solutions), which twin readily on being plastically deformed.

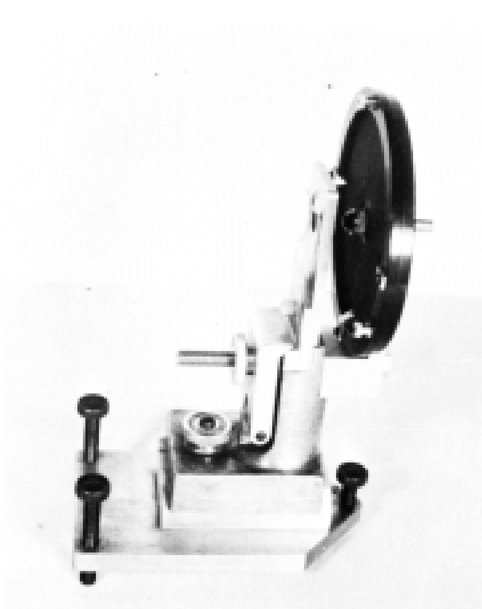

FIG. 1 Back-Reflection Laue Camera for Metallic Sheet Specimens

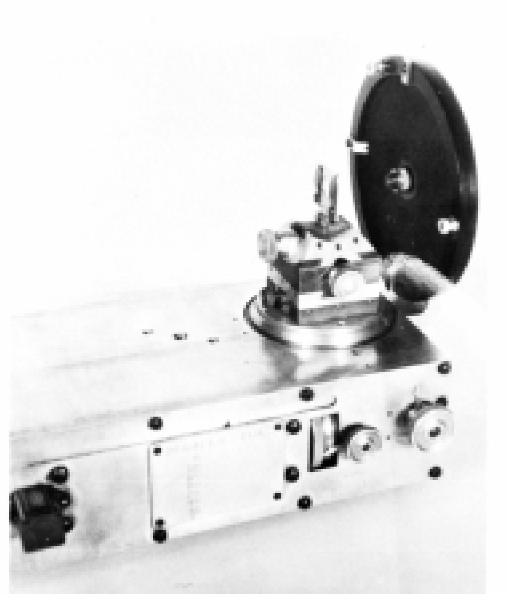

FIG. 2 Universal Camera With Goniometer Head for Back-Reflection Laue Studies

NOTE 3—Reference may be made to Methods E 3, for procedures for polishing specimens.

PROCEDURE

7. Orientation of Specimen and Film

7.1 It is necessary that the orientation relationships between the specimen and film be fixed at the outset (a sketch of this relationship should be made) and be preserved throughout the determinations. For example, this relationship is fixed if (*1*) the exposed specimen surface is parallel to the plane of the film, (*2*) a vertical line inscribed on the specimen surface is parallel to a vertical line on the film, (*3*) the "top" of the film corresponds with the "top" of the specimen, and (*4*) the exposed surface of the film facing the specimen is definitely marked.

8. Back-Reflection Laue Pattern

8.1 The back-reflection Laue pattern, properly prepared, will contain a hundred or more diffraction spots. These spots represent "reflections" of the X-ray beam from all important lattice planes of the crystal that are in position for diffraction. With the crystal-to-film distance of 3 cm and a photographic film 5 in. (127 mm) in diameter or 4 by 5 in. (102 by 127 mm), this will include all important lattice planes that make an angle of less than about 35° with the film; the reflections from all other planes in the crystal will not be intercepted by the film. The diffraction spots form a pattern consisting of many hyperbolic curves; these curves represent crystallographic zones **(1, 2)**. Some of these hyperbolic curves are more prominent (more thickly populated with spots) than others, as they represent crystallographic zones having a higher population of low-indices planes.

9. Hyperbolic and Polar Coordinate Charts

9.1 The hyperbolic chart, Fig. 3 (Plate I),[4] and the polar chart, Fig. 4, are used in the solution of back-reflection Laue patterns. Use the hyperbolic chart (reproduced as a positive on photographic film or plate) on the back-reflection Laue pattern in much the same way that a gnomonic (or stereographic) net is used on gnomonic (or stereographic) projections. Locate both horizontal and vertical curves 2° apart *in terms of angles within the crystal*. The horizontal curves are meridians, thus corresponding to crystallographic zones; the vertical curves are parallels. The series of meridian curves shown on the chart represents all possible curvatures that a crystallographic zone of a back-reflection Laue pattern may have; the zone is a straight line only when it passes through the origin.

9.2 The vertical curves are parallels and are used to measure angles along meridian curves. Thus, the angle between two crystal planes that produce two spots on the film may be read directly from the chart. To measure this angle, superimpose the chart on the film with centers coinciding and rotate the plate (or film) until a hyperbolic meridian coincides with the zonal curve connecting the two spots in question; then read the angle between the two planes directly from the set of parallels. Read the angle of inclination of the zone axis to the film directly from the scale of meridian angles.

9.3 A second, though not often needed, operation that may be performed with the aid of the hyperbolic and polar charts is the measurement of the angle between two zone axes (which are represented on the pattern as two intersecting zonal curves). If the point of intersection is located not more than about 10° from the origin, the following procedure is used: Place the chart over the film with centers coinciding so that a meridian coincides with one of the zonal curves. Then rotate the chart about the origin until another meridian coincides with the second zonal curve. The angle or rotation of the chart, measured by means of the polar net, gives the angle between the zone axes producing the two zonal curves. A procedure which may be used for *any* two zonal curves involves a rotation of a few spots of the back-reflection Laue pattern as follows: Superimpose the hyperbolic chart and the film so that the straight-line parallel (the vertical line through the center of the chart) contains the point of intersection of the two zonal curves in question. Then rotate this point of intersection to the origin, and move a (any) point on each of the two zonal curves the same number of degrees (along parallels, of course) in the same direction. Since both zonal curves now pass through the origin they appear as straight lines, and the angle between these radial lines is then the angle between the two zone axes in question (±½ °, if the rotation has been carefully carried out).

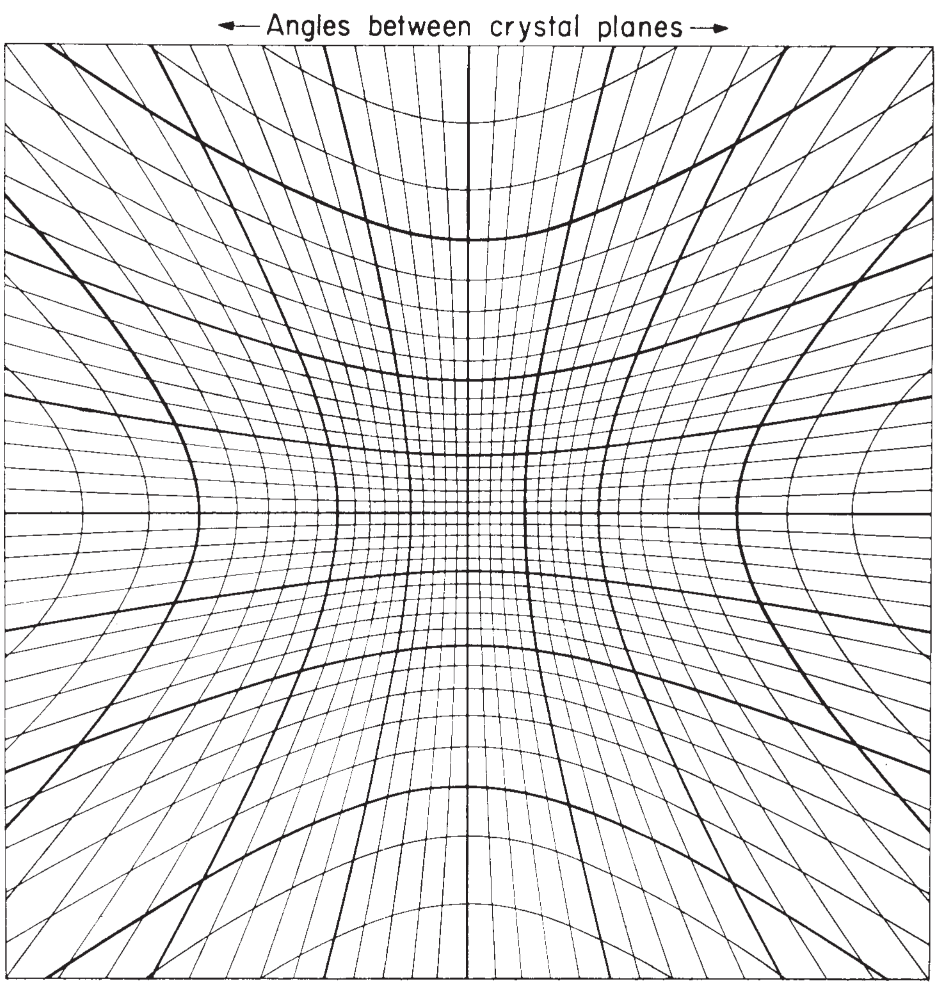

FIG. 3 Hyperbolic Chart for Solution of Laue-Back-Reflection Patterns

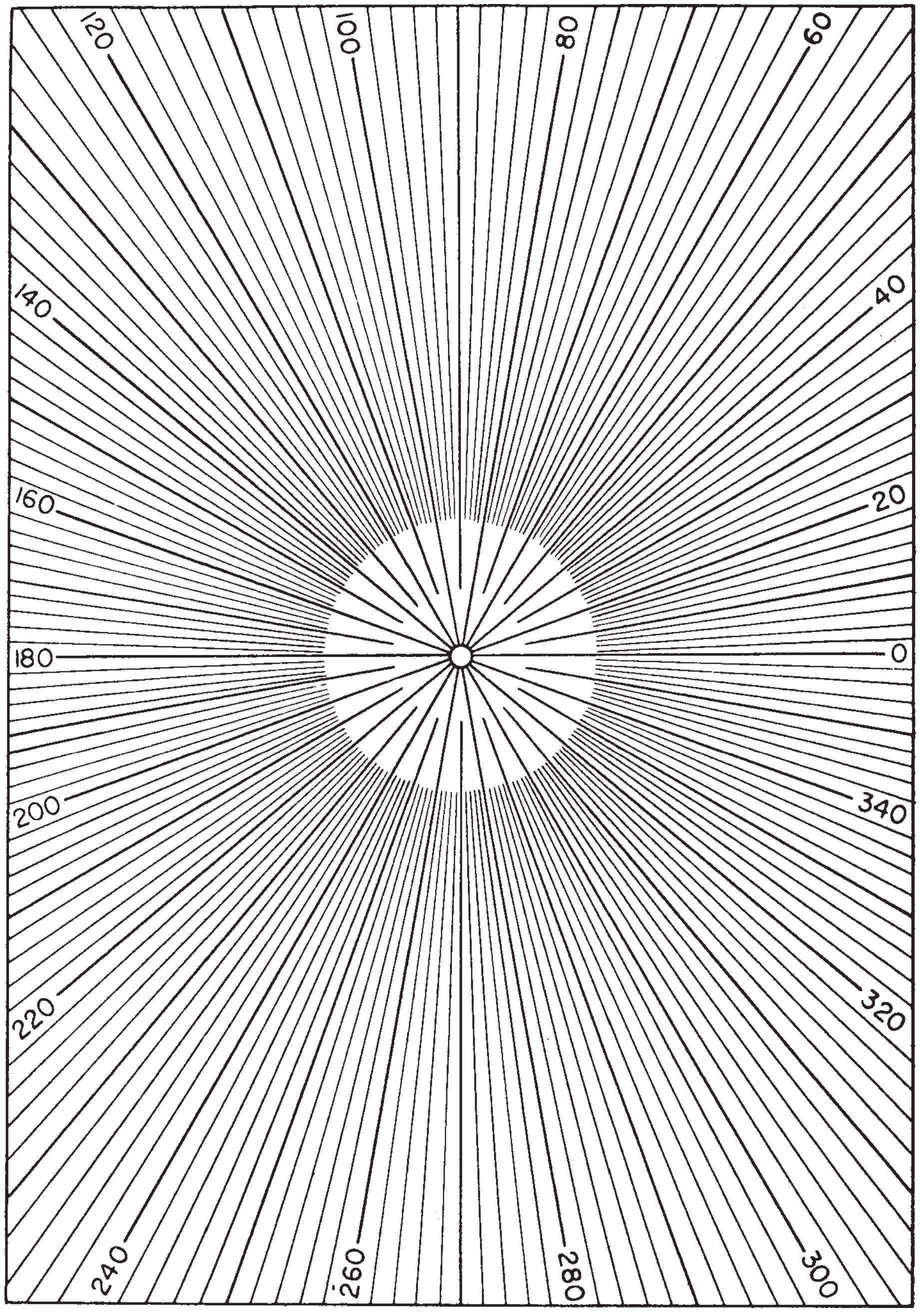

FIG. 4 Polar Chart for Solution of Laue Back-Reflection Patterns

This operation is the same as the operation required to measure the angle between any two intersecting great circles on a stereographic projection.

10. Interpretation of Unsymmetrical Back-Reflection Laue Patterns

10.1 The most rigorous method for solving an unsymmetrical pattern is by preparing a stereographic projection with its plane parallel to the plane of the film. Read the film from the side opposite that of incident radiation, so that the projection corresponds to viewing the crystal from the position of the X-ray tube. Inscribe a reference line on the film through the central spot and parallel to a prominent direction in the specimen, and measure all azimuth angles with respect to this line. Methods for plotting the projections are described by Barrett **(4)**. Methods for identifying prominent spots and zones are summarized in 10.2 to 10.6, inclusive.

10.2 After some experience has been gained, it will be found that back-reflection Laue patterns may be solved by inspection alone. The following remarks should be of assistance in the development of a systematic approach: At least one standard stereographic projection **(5)** of the lattice being studied shall be prepared. This projection shall include ⟨100⟩, ⟨110⟩, and ⟨111⟩ zones, and if the crystal is face-centered cubic, the projection shall include all the poles of the forms {100}, {110}, {111}, and {113}; if body-centered cubic, the projection shall include {100}, {110}, {111}, and {112}. This standard projection shall be studied until one has become familiar with the relative positions of poles and their angular separations, the symmetry characteristics of each pole, the important zonal curves passing through each pole, etc.

10.3 In Figs. 5-8 are reproduced standard stereographic projections of a cubic crystal with the {100}, {111}, {110}, and {112} poles at the center. These projections illustrate orientations having four-fold, three-fold, and two-fold axes of symmetry and a plane of symmetry, respectively. Note that the standard cubic, or {100}, projection is made of 24 identical triangular areas.

10.4 Crystallographic zones are of great importance in the solution of back-reflection Laue patterns. For the face-centered cubic lattice, the important zones, arranged in order of importance, are ⟨110⟩ and ⟨100⟩; for the body-centered cubic lattice these are ⟨111⟩, ⟨100⟩, and ⟨110⟩. For any lattice, the most important zone is always that one whose axis is the line of closest atomic approach. For body-centered-cubic, this zone axis is ⟨111⟩, and every unsymmetrical pattern will contain at least one of these; likewise, every face-centered-cubic pattern will contain at least one of these; likewise, every face-centered-cubic pattern will contain at least one (usually two) ⟨110⟩ zones.

10.5 The most important spots are those originating from planes having widest spacing in the lattice. For face-centered cubic crystals, these important spots are {111}, {100}, and {110}, for body-centered cubic, they are {110}, {100}, and {112}. The above-listed planes and zones are all that need be considered in the solution of patterns, because every unsymmetrical back-reflection Laue pattern will contain at least two of the listed diffraction spots, and in order to obtain a complete solution of a pattern it is necessary only to identify two important diffraction spots, or one important spot and a zonal curve. An important spot on a back-reflection Laue pattern may be recognized easily because (*1*) it is (comparatively) isolated from its neighbors, (*2*) it is a point of intersection of a large number of zonal curves, and (*3*) it is of rather high intensity. A spot is identified by the angles between the important zonal curves that intersect at the spot in question, or by its position in relation to that of some other important spot. The indexing of the separate points is also much simplified by using a tabulated summary of the possible angular separations for the crystal form investigated. Such a summary made by Stahlein and Schlechtinig **(6)** for the body-centered cubic lattice is reproduced in Fig. 9. Data for the face-centered cubic lattice are given in Table 1 **(7)**. Peavler and Lenusky **(8)** calculated the angles of intersection of planes up to {554} in the cubic system and listed them in tabular form.

10.6 It is often desirable to know the orientation of directions not represented on the back-reflection photograph. A simple vector method for doing this which requires only slide rule calculations using data for two or at most three spots on the film has been developed by B. F. Decker **(9)**.

NOTE 4—*Examples of Solution of Back-Reflection Laue Patterns*—In Fig. 10 and Fig. 11 are reproduced unsymmetrical back-reflection Laue patterns of tungsten crystals (body-centered cubic); Fig. 12 is a tracing of the obviously important zones and spots of Fig. 10. A measurement of angles between the spots of Fig. 12 gives the following:

ab = 45°
ac = 35½ °
bc = 30°

The fact that angle *ab* measures 45° means that one of these spots may be {100} and the other {110}. Now, in as much as {110}:{112} = 30° and {100}:{112} = 35¼ °, the following must be true: *a* = {100}, *b* = {110}, and *c* = {112}. This solution may be checked in various ways. For example, note that four important zonal curves pass through spot *a*, and

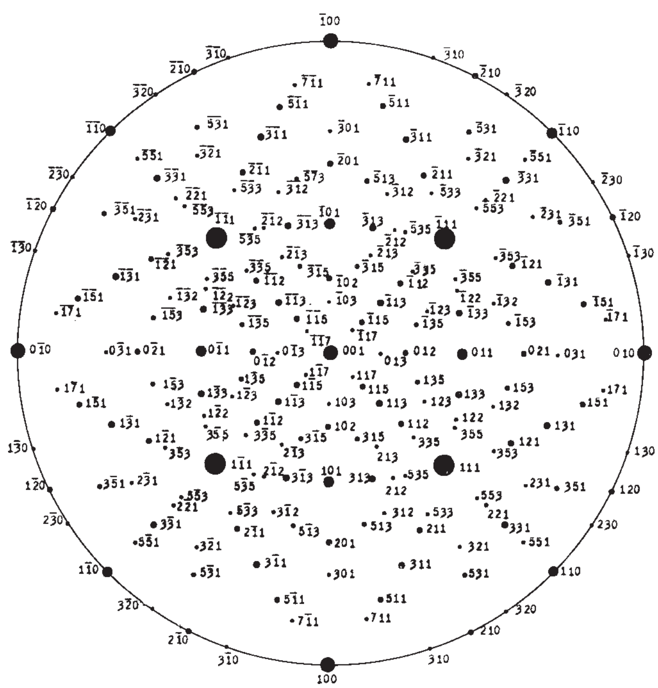

FIG. 5 Standard {001} Projection for a Cubic Crystal

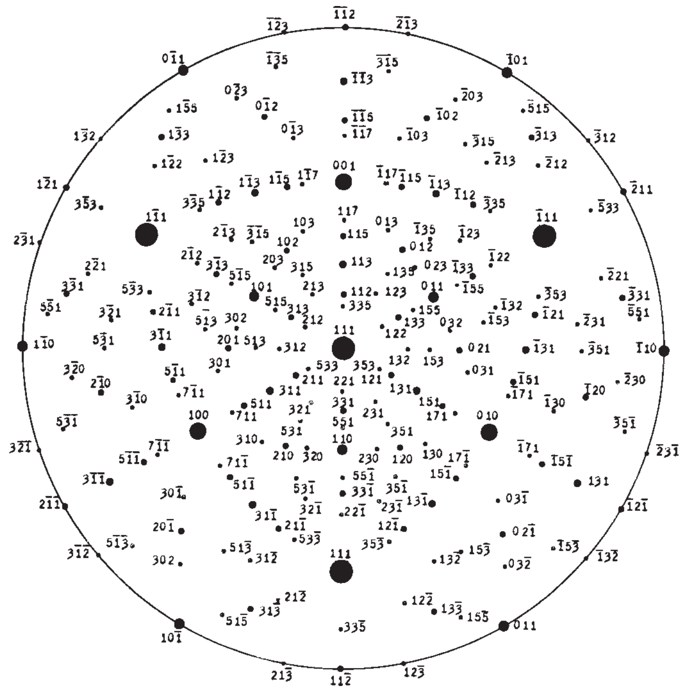

FIG. 6 Standard {111} Projection for a Cubic Crystal

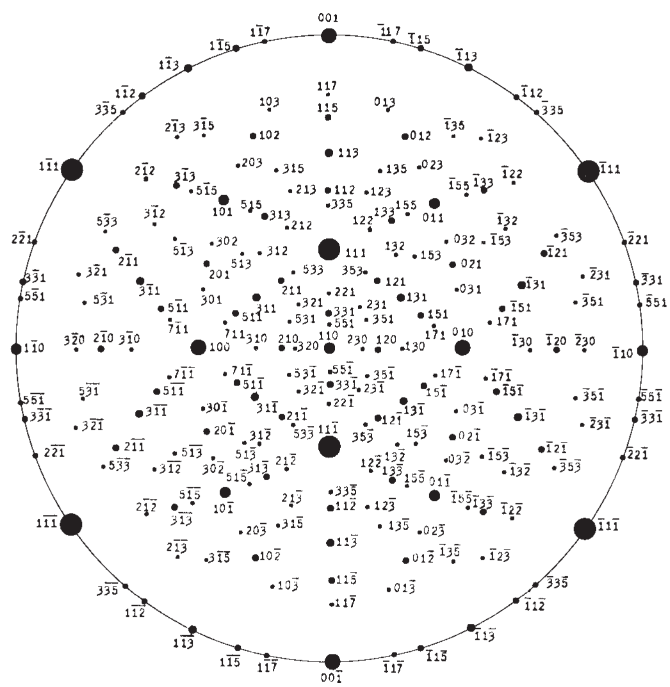

FIG. 7 Standard {110} Projection for a Cubic Crystal

that these zones are at angles of 45° with each other. This is possible only when the symmetry about the point is four-fold; hence spot *a* must be {100}.

The tracing of important zones and spots in Fig. 11 is shown in Fig. 13. The pattern symmetry about spot *a* is obviously four-fold; angle *ab* measures 35^{+} °. In as much as {100}:{112} = 35¼ °, *a* = {100} and *b* = {112}.

11. Precision and Bias

11.1 *Precision*—With reasonable care, the orientation of a crystal can be determined to an uncertainty of about + or − ½ °. With considerable care, including checks on the accuracy and uniformity of the two charts, the uncertainty may be improved to one or two tenths of a degree. It is recommended

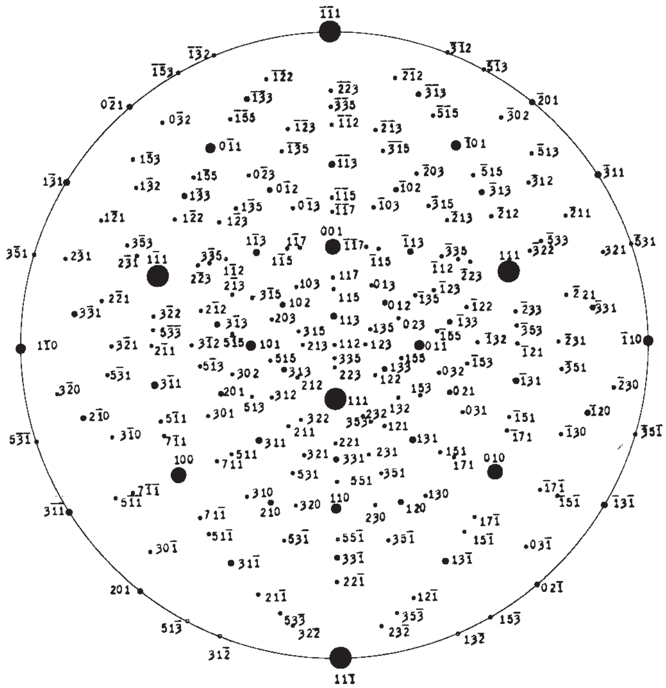

FIG. 8 Standard {112} Projection for a Cubic Crystal

that the orientation be over-determined (many spots measured) because of the number and proximity of interplanar angles.

11.2 *Bias*—There is no known bias in this test method.

12. Keywords

12.1 back reflection Laue X-ray; hyperbolic chart; metal crystal; orientation; polar chart; standard projection; X-ray diffraction

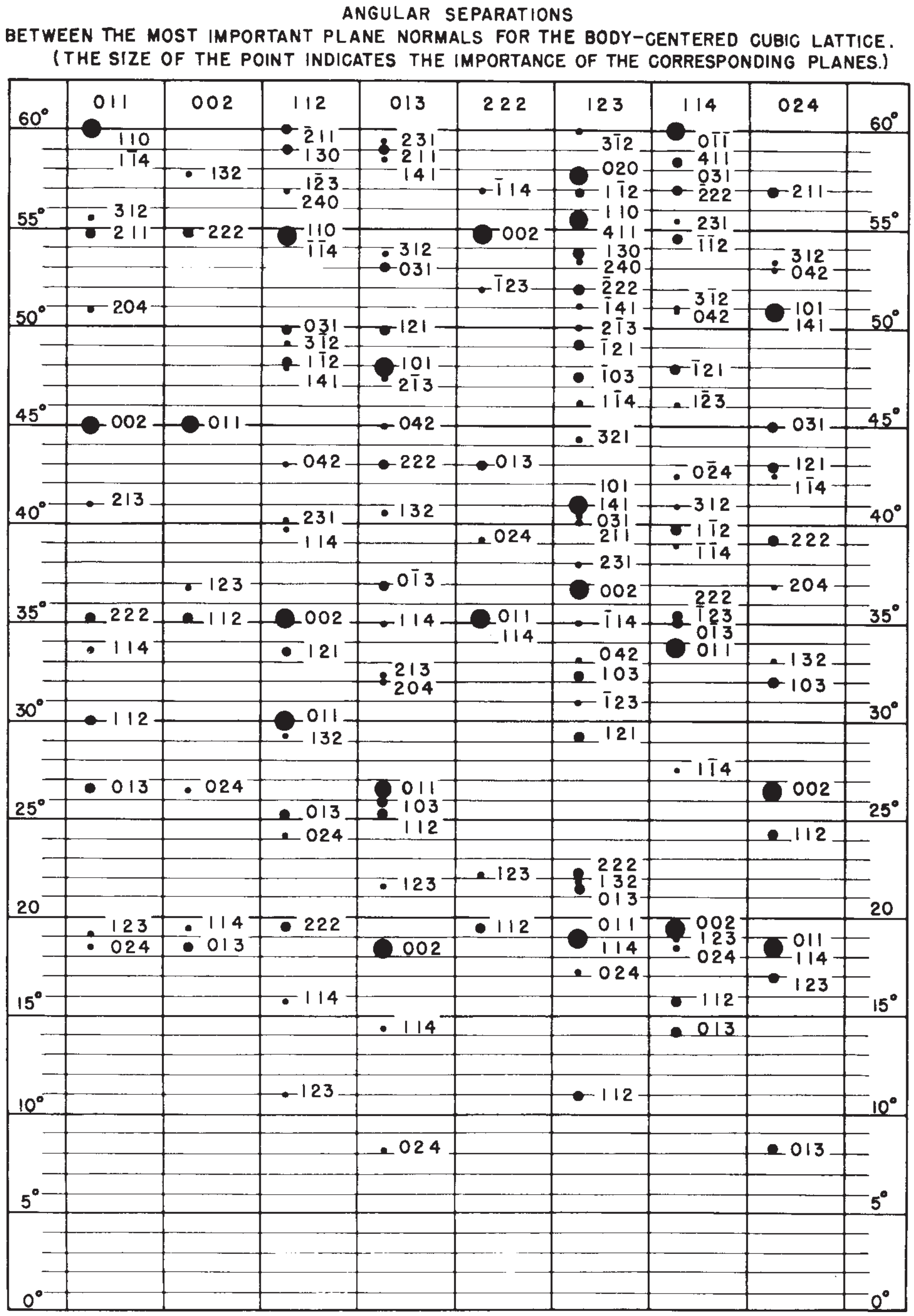

FIG. 9 Angular Separations Between the Most Important Plane Normals for the Body-Centered Cubic Lattice (Point Size Indicates Importance of Corresponding Plane)

TABLE 1 Angles Between Important Reflecting Planes of the Face-Centered Cubic Lattice

Angle between Normals		Types of Planes	Angle between Normals		Types of Planes	Angle between Normals		Types of Planes
deg	min		deg	min		deg	min	
5	00	211-533	31	20	110-311	46	50	111-153
6	10	331-221	31	30	511-301	46	50	153-115
8	10	210-310	31	40	511-511	47	10	210-513
9	30	311-511	31	40	153-335	47	10	211-212
10	00	211-311	32	00	210-301	47	30	533-151
10	40	210-531	32	20	153-212	47	30	533-103
13	10	110-331	32	20	100-531	47	40	210-131
13	10	511-301	32	30	221-310	48	10	211-211
14	10	133-153	32	40	331-212	48	10	100-221
14	20	111-533	33	00	133-151	48	30	311-531
14	30	311-531	33	30	511-221	48	30	111-133
15	00	211-531	33	30	211-121	48	40	133-531
15	00	533-221	33	50	111-511	48	40	533-212
15	00	311-533	33	50	210-351	48	50	110-301
15	50	100-511	34	00	153-135	49	00	153-531
15	50	111-221	34	10	211-315	49	30	110-133
15	50	153-031	35	10	311-311	49	40	110-335
16	40	153-122	35	20	110-111	49	50	211-130
17	00	110-531	35	20	110-221	50	10	153-031
17	30	153-353	35	20	110-511	50	30	311-113
17	30	311-310	35	20	211-122	50	50	110-201
17	40	211-221	35	30	133-315	51	00	211-151
18	30	210-110	36	30	133-533	51	30	211-533
18	30	100-301	36	50	301-301	51	30	311-133
18	50	210-511	36	50	210-120	51	40	153-221
19	10	133-355	37	00	153-353	51	50	153-115
19	20	153-151	37	50	331-313	52	10	210-335
19	20	210-311	38	10	211-511	53	00	210-151
19	30	111-211	38	30	311-335	53	10	301-103
19	30	211-511	39	00	221-221	53	10	210-210
19	30	153-153	39	10	210-511	53	20	210-132
19	30	110-221	39	10	210-111	54	30	331-301
20	30	211-331	40	10	311-315	54	30	133-315
22	00	111-331	40	20	100-533	54	30	533-533
22	10	511-511	40	20	311-310	54	40	111-100
22	40	210-331	41	00	311-511	54	40	111-122
24	10	210-211	41	20	210-353	54	40	511-122
24	30	533-511	41	20	133-151	54	40	533-013
25	00	533-353	41	30	153-013	54	40	011-211
25	10	100-311	41	30	153-151	55	00	133-115
25	10	311-221	41	30	211-133	55	10	311-130
25	20	211-310	41	40	153-533	55	50	311-153
25	50	301-310	41	50	210-212	56	00	153-103
26	00	311-331	42	20	211-113	56	10	533-511
26	30	100-210	42	30	221-301	56	20	111-151
26	30	331-331	42	40	133-335	56	30	211-531
26	30	210-221	42	50	533-511	56	50	210-211
26	30	110-310	42	50	153-212	57	00	110-511
26	30	133-153	43	00	111-310	57	10	153-513
27	20	221-212	43	00	210-121	57	40	331-212
27	30	210-033	43	30	331-301	58	00	210-153
27	40	153-351	44	00	210-313	58	10	221-013
28	00	153-151	44	10	110-153	58	30	311-151
28	30	111-153	44	30	153-351	58	30	111-113
29	29	211-355	45	00	001-011	58	50	211-013
29	30	311-511	45	00	110-212	59	00	210-133
29	30	331-310	45	00	210-130	59	30	100-315
29	30	111-113	45	10	511-212	59	30	153-212
29	50	533-301	45	20	311-122	59	40	311-335
30	00	011-121	45	50	153-335	59	50	311-221
30	10	533-122	46	20	211-153	60	00	101–011
30	20	110-533	46	20	311-533	60	00	211-112
31	10	153-130	46	30	001-133	60	00	533-155

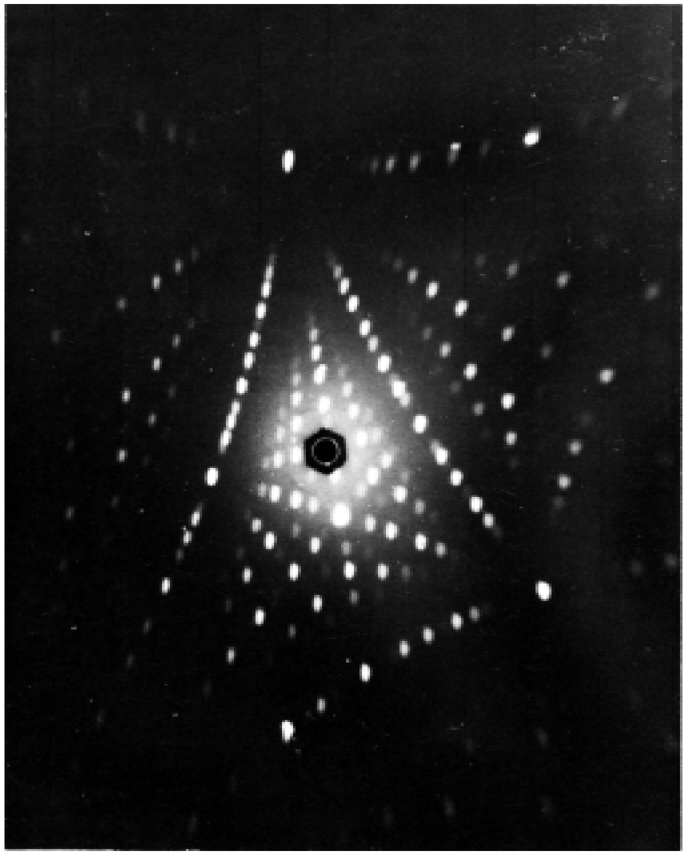

FIG. 10 Unsymmetrical Back-Reflection Laue Pattern of Tungsten Crystal (Body-Centered Cubic)

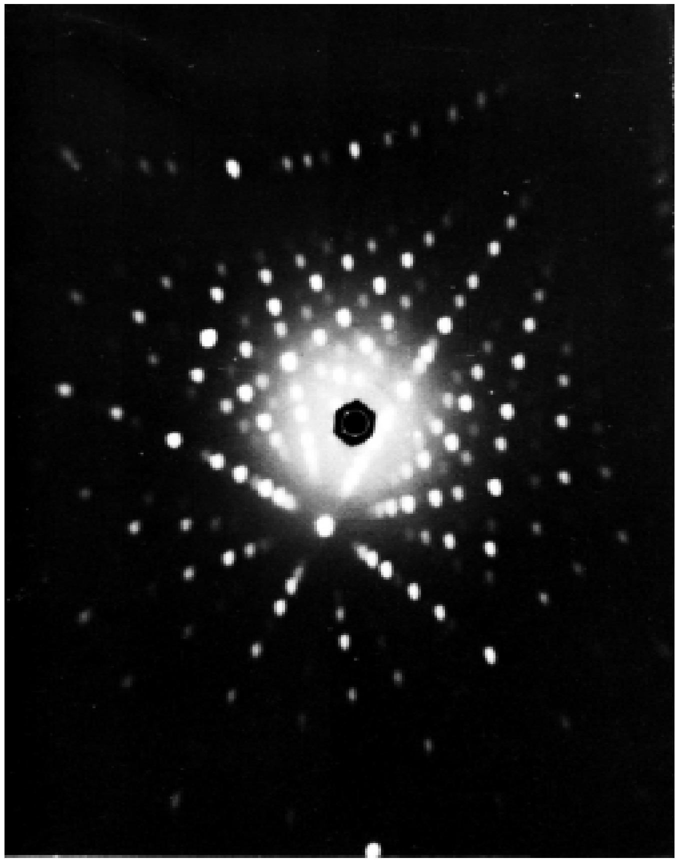

FIG. 11 Unsymmetrical Back-Reflection Laue Pattern of Tungsten Crystal (Body-Centered Cubic)

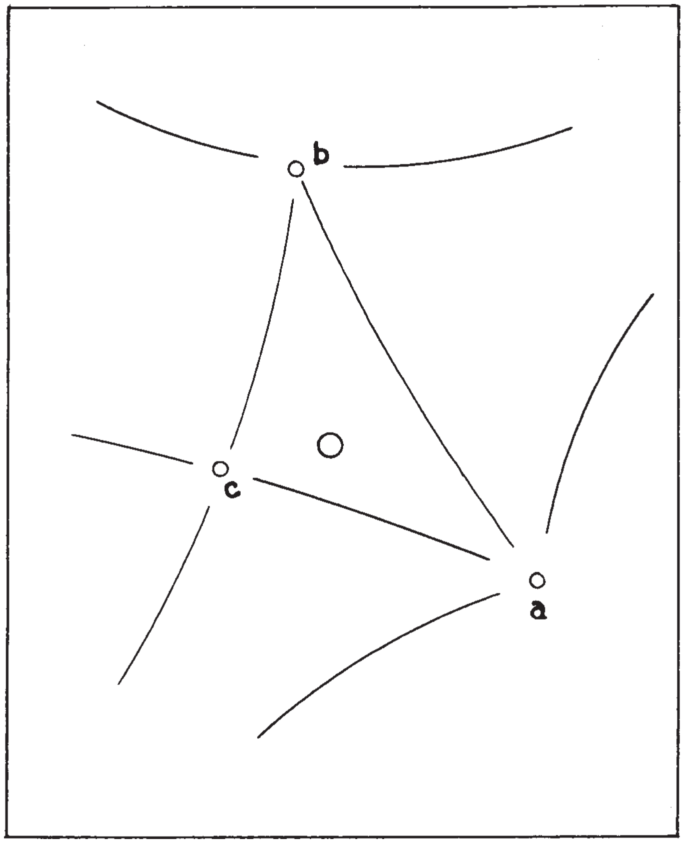

FIG. 12 Tracing of Important Zones of Fig. 10

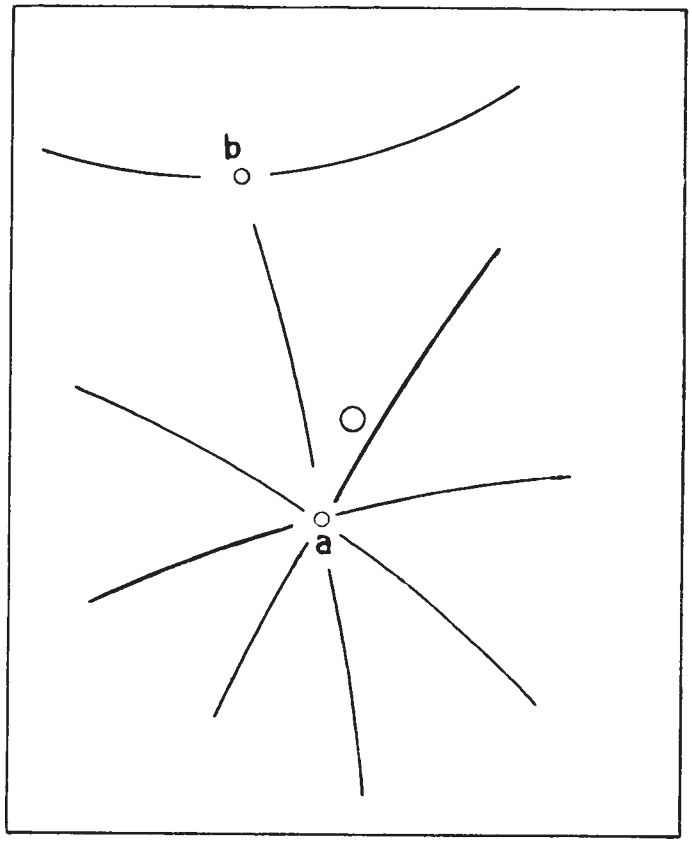

FIG. 13 Tracing of Important Zones of Fig. 11

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(**7**) Compiled by H. L. Burghoff from data available at Hammond Laboratory of Yale University.

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