Designation: E 82 – 91 (Reapproved 2001)

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

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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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.

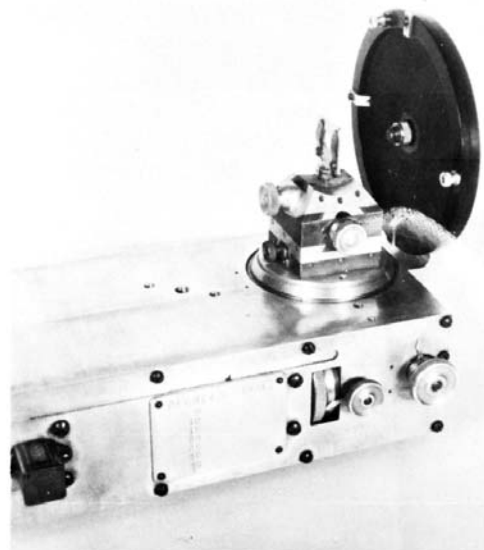

FIG. 2 Universal Camera With Goniometer Head for Back-Reflection Laue Studies

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.

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

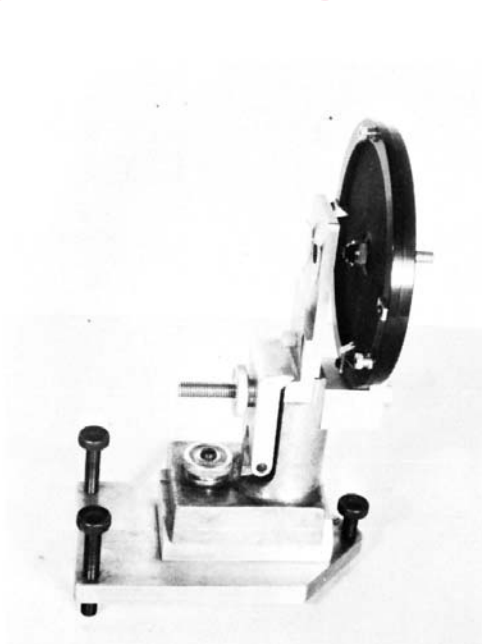

FIG. 1 Back-Reflection Laue Camera for Metallic Sheet Specimens

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).

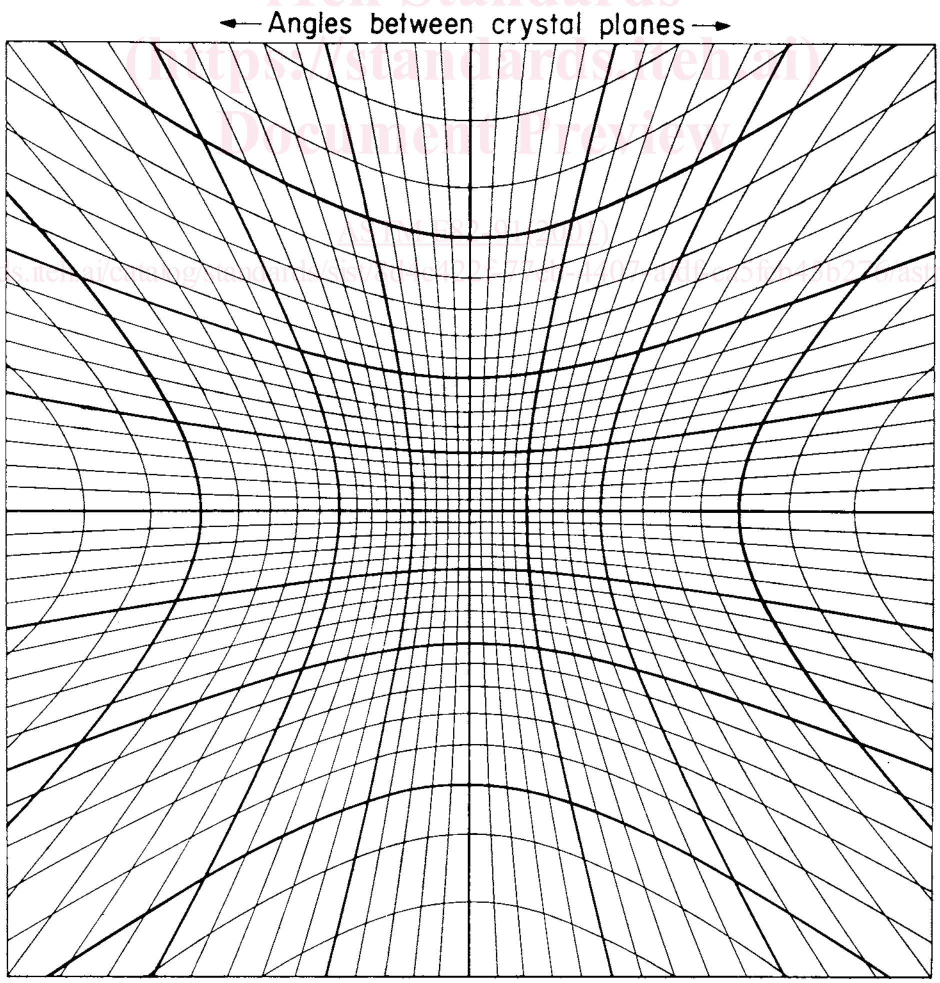

FIG. 3 Hyperbolic Chart for Solution of Laue-Back-Reflection Patterns

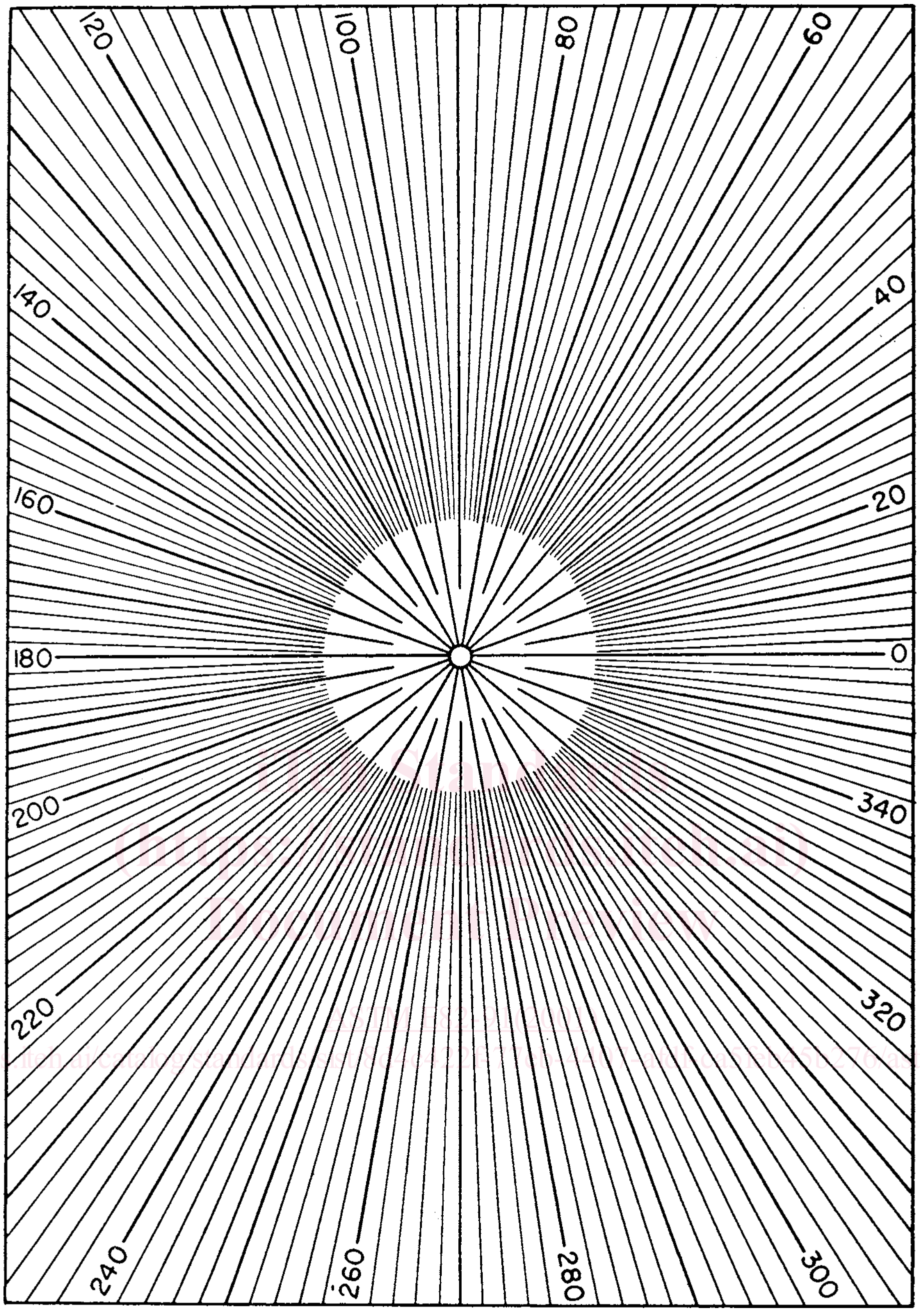

FIG. 4 Polar Chart for Solution of Laue Back-Reflection Patterns

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 $\langle 110\rangle$ and $\langle 100\rangle$; for the body-centered cubic lattice these are $\langle 111\rangle$, $\langle 100\rangle$, and $\langle 110\rangle$. 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 $\langle 111\rangle$, 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) $\langle 110\rangle$ 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:

$ab = 45°$
$ac = 35\frac{1}{2}°$
$bc = 30°$

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

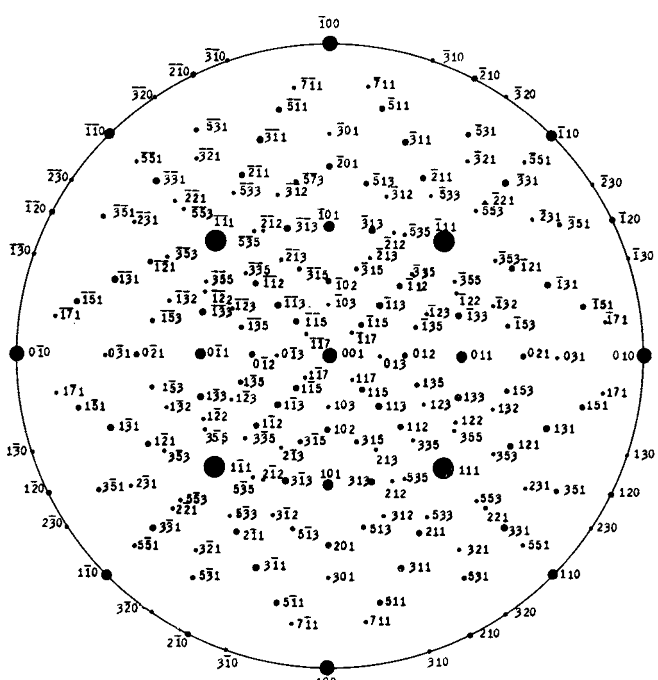

FIG. 5 Standard {001} Projection for a Cubic Crystal

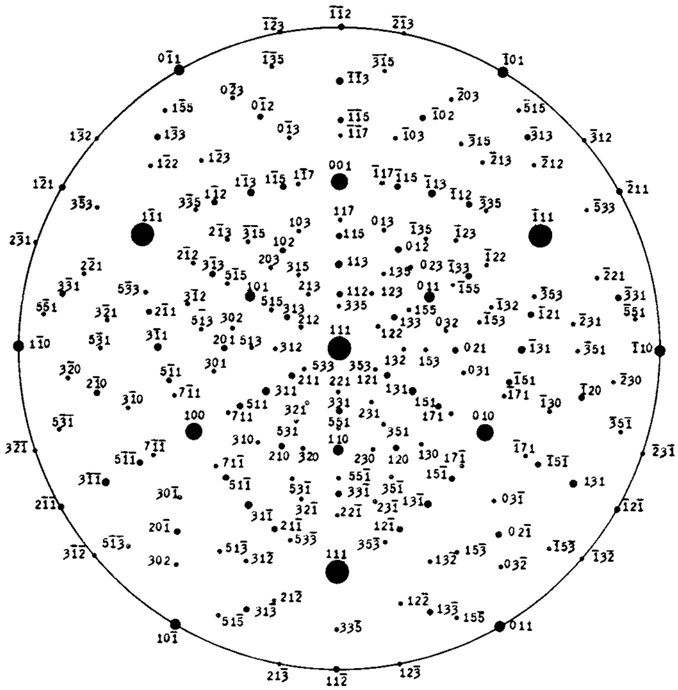

FIG. 6 Standard {111} Projection for a Cubic Crystal

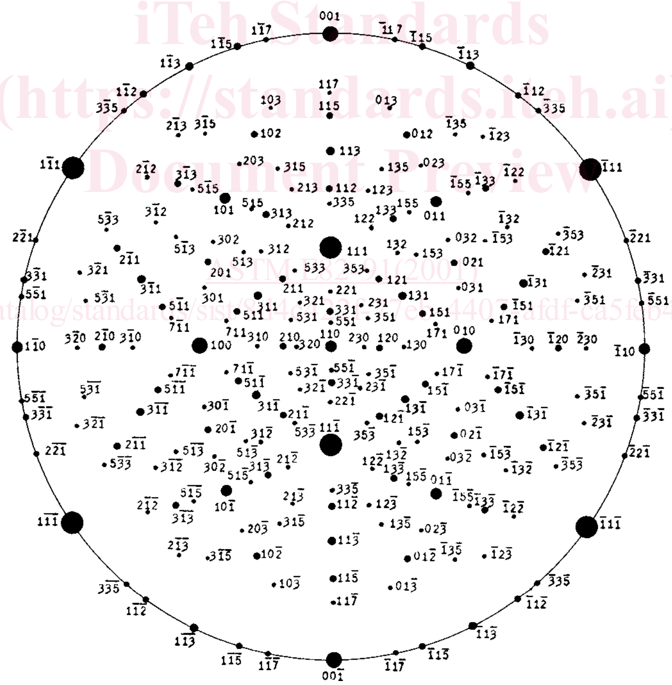

FIG. 7 Standard {110} Projection for a Cubic Crystal

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