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 standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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:*[3]

E 3 Guide for Preparation of Metallographic Specimens

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.

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[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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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 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.

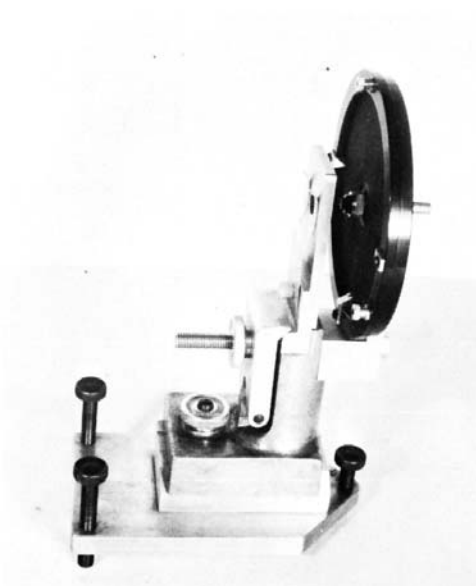

FIG. 1 Back-Reflection Laue Camera for Metallic Sheet Specimens

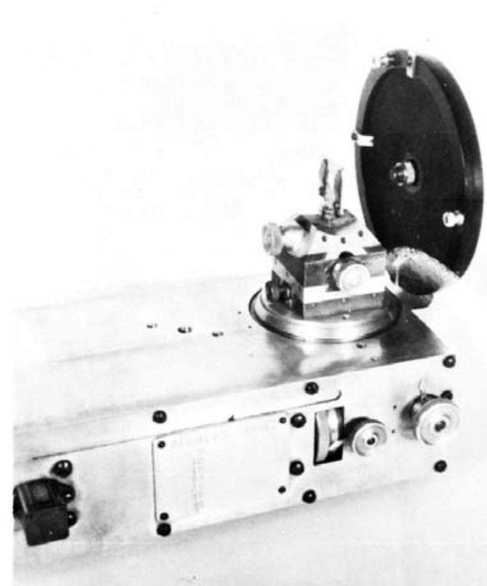

FIG. 2 Universal Camera With Goniometer Head for Back-Reflection Laue Studies

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, 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

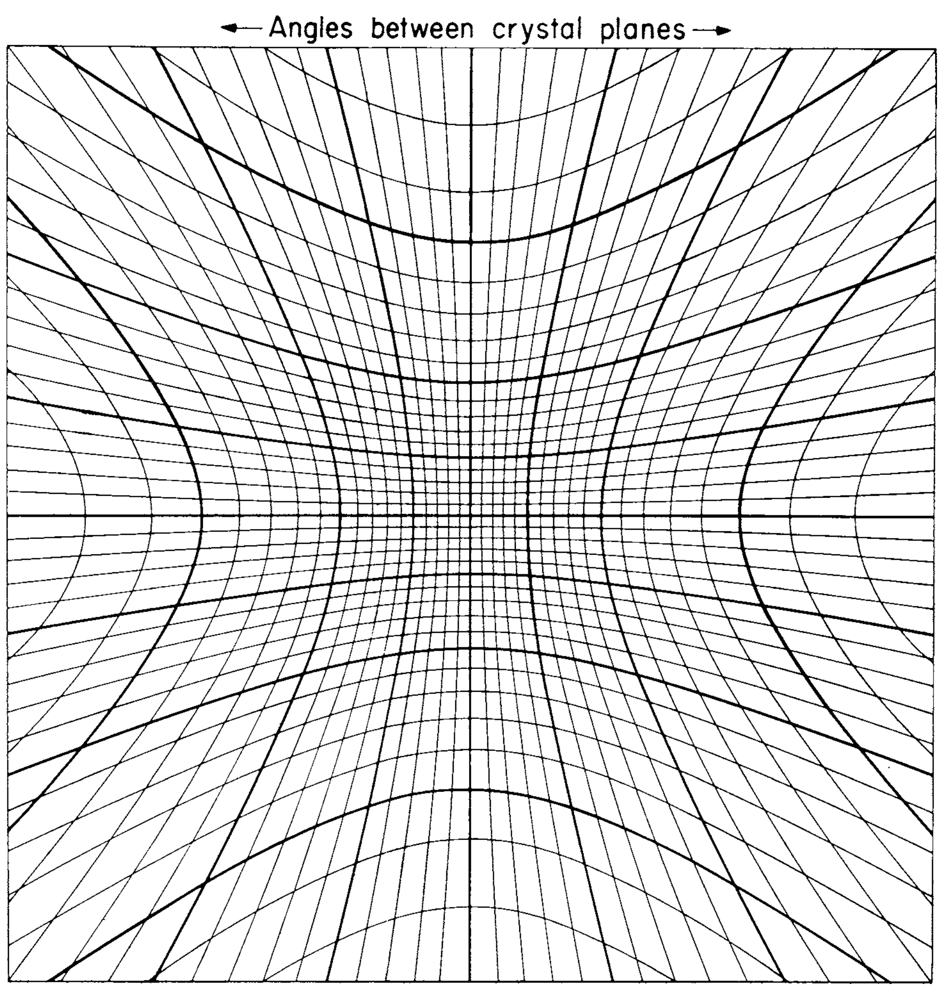

FIG. 3 Hyperbolic Chart for Solution of Laue-Back-Reflection Patterns