Designation: E82/E82M – 14

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

This standard is issued under the fixed designation E82/E82M; 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 may be applied to macrograins and micrograins depending on the beam size within polycrystalline aggregates, as well as to single crystals of any size. This test method is described with reference to cubic crystals and other structures such as: 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) shall 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 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with 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:*[2]

E3 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[3,4] except that the detector is located between the X-ray source and the specimen or beside the X-ray source in the case of side reflection geometry. The incident beam of white X-radiation passes through a pinhole aperture, strikes the crystal, and is then diffracted back to the detector. White spots, which represent X-ray beams "diffracted" by the atomic planes within the crystalline specimen, appear on the digital picture collected by the detector. The indexation of the spots and their positions in space are calculated by simulation of the Laue pattern superimposed onto the digital image collected by the detector. Older techniques based on film technology can also be used to index the spots and to calculate the orientation of the crystal.

4. Significance and Use

4.1 The physical properties of metals and other materials are often anisotropic (for example: Young's modulus will typically vary in different crystallographic directions). As such, it is often desirable or necessary to determine the orientation of a single crystal to ascertain the relation of any pertinent physical properties with respect to different directions in the material.

4.2 This test method can be used commercially as a quality control test in production situations in which 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

[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] 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.

[3] Cullity, B. D., *Elements of X-ray Diffraction*, second edition, Addison-Wesley, Reading, MA, 1978.

[4] Barrett, C. S. and Massalski, T. B., *The Structure of Metals*, 3rd edition, McGraw-Hill Inc., New York, 1966, pp. 211–227.

material can be moved to a preferred orientation and subsequently sectioned, ground, or processed otherwise.

4.4 If the grains in a polycrystalline material are large enough, this test method can also be used to determine their orientations and differences in orientation can be documented or mapped or both.

5. Apparatus

5.1 *X-Ray Tube*—For exposure times be reduced to a minimum, the X-ray tube shall have a target that produces a high flux of white X-radiation and the detector shall be sensitive to the X-ray energies produced [Charge-Coupled Device (CCD)- and complementary metal–oxide–semiconductor (CMOS)-based detectors are normally suitable for this task]. Tungsten and molybdenum target X-ray tubes are typically used when collecting LAUE images. The X-ray tube power used is dependent on the detector sensitivity and the accelerating voltage normally varies from 20 to 50 kV depending on the saturation of the detector and the image quality.

5.2 *Back-Reflection Laue X-Ray Detector*—The Laue detectors can be of different types and should be sized such that a sufficient number of LAUE spots are collected in one contiguous image. The pinhole is usually sized to about 6 mm [about ¼ in.] in diameter or less when possible. The camera-to-sample distance should be adjustable to accommodate the application, the component geometry, and the detector window size; it is usually set to minimum of 30 mm [1.2 in.] and up to 60 mm [2.4 in.]. These parts may be assembled in various configurations depending upon the type of specimen being studied and the accuracy desired. For back-reflection systems, the main requirement for accurate results is that the pinhole system shall be precisely perpendicular to the detector. For side-reflection systems, the specimen surface shall be aligned precisely perpendicular to the bisector of the incident beam pinhole and the normal of the detector plane. Adjustment for accurate alignment of the specimen, incident beam pinhole, and the detector plane should be available on the instrument.

5.3 The acquired Laue images can be of different orientation depending on the sense of the projection. Two main Laue image orientations can be found on different instruments depending on the convention or the view direction selected; first view when looking at the detector from the sample and second view when looking at the sample from the detector. Some software allows the view to flip to accommodate any convention.

NOTE 1—Fig. 1 illustrates a back-reflection Laue camera constructed for use with single-crystal materials. The specimen-to-detector distance is fixed at 30 mm [1.2 in.] and the specimen surface is maintained perpendicular to the incident beam and parallel to the detector plane.

NOTE 2—Fig. 2 illustrates a side-reflection Laue camera for turbine blade single-crystal measurement. The measurements for this type of setting are limited to cubic symmetries.

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 etching 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 crystals of metals such as tin and zinc (or their solid solutions), which twin readily on being plastically deformed. For other applications that do not require high accuracy, the sample surface can be prepared by other means or left as-manufactured.

NOTE 3—Reference may be made to Methods E3, for procedures for polishing specimens.

7. Procedure

7.1 *Laue Instrument Calibration:*

7.1.1 It is necessary that the orientation relationships between the specimen and detector window be accurately known at the outset (a sketch of this relationship should be made) and preserved throughout the determinations. For example, this relationship is fixed if *(1)* the exposed specimen surface is parallel to the plane of the detector window, *(2)* a vertical line inscribed on the specimen surface is parallel to a vertical line on the detector, *(3)* the "top" of the detector corresponds with the "top" of the specimen, and *(4)* the exposed surface of the detector facing the specimen is definitely marked. To verify accurately the alignment of the apparatus, a Laue image should be collected on a known single-crystal reference before measuring the specimen of interest. This single-crystal reference can be considered as a standard reference and should be certified for use on all Laue instruments before testing. Many instruments include a calibration routine using a single-crystal silicon standard.

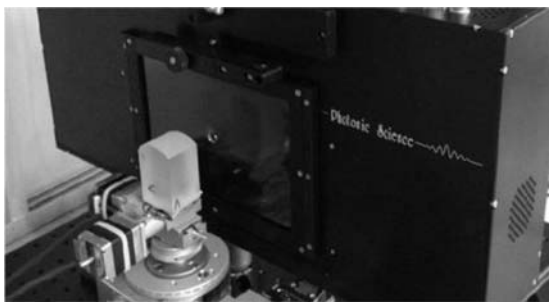

FIG. 1 Back-Reflection Laue Camera Measuring Sapphire Single Crystal Mounted on a Five-Axis Motorized Goniometer

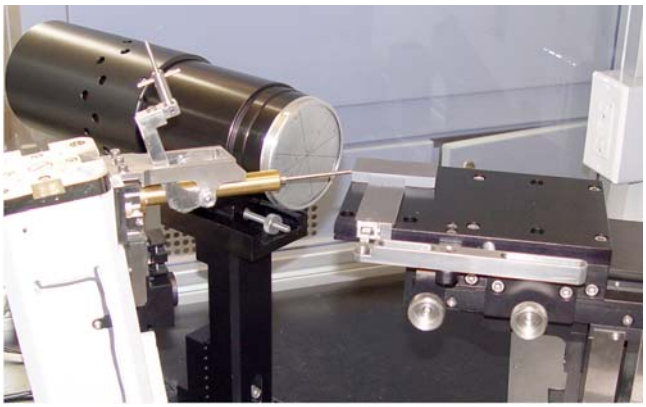

FIG. 2 Side-Reflection Laue Camera for Turbine Blade Single-Crystal Measurements

7.1.2 The calibration can be performed using a simulated pattern overlapped on top of the collected Laue image. The key spots should match accurately. If the pattern cannot be matched, a series of parameters can be adjusted to account for the offsets angles and distances. The parameters of interest are: the two main rotations (α,β), that is, up/down and right/left and the distance from the sample to the detector. This information should be sufficient if the detector is properly calibrated by the manufacturer.

7.1.3 To verify the accuracy of the measured angles (α,β,γ) and the direction, one can repeat the measurement on a known calibrated specimen tilted at different levels of (α,β,γ) using a three-way goniometer fixture. The measured values using the matching overlay pattern should match the manually set angles.

7.2 *Back-Reflection Laue Pattern:*

7.2.1 *Classical Method*—The back-reflection Laue pattern, properly prepared, will contain hundreds of diffraction spots. These spots represent "diffraction" of the X-ray beam from all important lattice planes of the crystal that are in positions suitable to diffract onto the detector. The subsequent diffraction pattern obtained will consist of many spots positioned on hyperbolic curves that represent crystallographic zones. 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-index planes. By using these observations and calculating the positions of the spots using the hyperbolic chart, one can determine the orientation of the crystal with respect to the specimen reference frame. Some programs still exist to help calculate the orientation using the zone hyperbolas but they are time-consuming to use.

7.2.2 *Simulation Method*—This method was developed in the last few decades and is being used more and more frequently; it would not be practical without the help of modern high-speed computers. The simulation method is both fast and accurate. The positions and intensities of the simulated Laue spots displayed in the overlay are calculated taking into account both the intensity and the extinction conditions calculated from the cell parameters input for the selected space group, the eleven Laue classes, or the Bravais symmetries.

7.2.2.1 Two approaches have been developed for analyzing Laue data using simulation:

(1) Manual matching or semi-automated matching in which the simulated pattern is generated on top of the experimentally obtained Laue image and the operator manually adjusts the position of the simulated Laue pattern to match the experimentally obtained Laue image by incrementing the rotation angles using a three-dimensional (3D) mouse. This technique can be very accurate and fast at execution and is sometimes preferred when the material condition in the specimen results in an unclear/less than ideal Laue image.

(2) Automatic matching—The simulated Laue pattern is calculated from the positions of the spots defined in space and the optimal solution is determined on the basis of the orientation angles calculated from a "best match" to the experimentally obtained Laue image. These algorithms require extensive calculation and the analysis can sometimes be slow, even with modern high-speed computers.

7.2.3 The pattern rotation is programmed such that the axes of rotation are with respect to the sample holder reference frame (*X*, *Y*, *Z*). Alternatively, the rotations can be referenced with respect to the [001] axes of the crystal reference frame. Additional rotation axes can be defined if required.

NOTE 4—Fig. 3 shows a simulated Laue pattern on top of a Laue image collected on a sample.

7.3 *Indexation of Back-Reflection Laue Patterns:*

7.3.1 Most commercially available programs are capable of calculating and displaying indices of Laue spots. This is done using routines developed according to the crystal symmetry of the specimen. While some programs can display the directions of the <uvw> planes, others can display the (hkl) planes for each spot. The indices are automatically displayed for the main directions such as: <100>, <100>, and <111> and, if the crystal is face-centered cubic, the projection shall include all the spots of the forms {100}, {110}, {111}, and {113}; if body-centered cubic, the projection shall include {100}, {110}, {111}, and {112}. Other directions are user selectable.

7.3.2 After some experience has been gained, it is possible to solve a Laue pattern by visual inspection of the image displayed alone. The following remarks should be of assistance

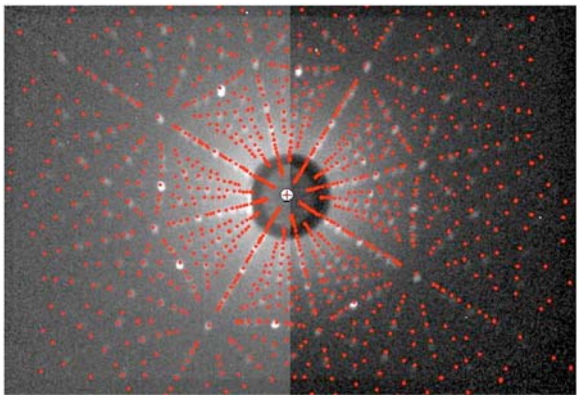

FIG. 3 Laue Back Reflection Showing Hundreds of Spots Simulated on Top of the Collected Image

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 the <100>, <110>, and <111> zones, and if the crystal is face-centered cubic, the projection shall include all of the spots 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 spots and their angular separations, the symmetry characteristics of each spot, the important zonal curves passing through each spot and so forth.

7.3.3 In Figs. 4-7, the standard stereographic projections of a cubic crystal with the {100}, {110}, {111}, and {112} spots at the center are reproduced. 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 structure or {100} projection is made of 24 identical triangular areas. Only 12 can be represented on a stereographic projection. The equation governing the indexation is highly geometrical with rotation matrices to step from one position to another one. For a known symmetry, we consider here the cubic system because is one that is widely measured; the equations are executed in a Cartesian coordinate system with consideration of the conditions of reflection to select the appropriate (hkl) planes. For a face-centered cubic (FCC) example, the list of planes begins from $Q = (h^2 + k^2 + l^2) = 4$, in other words the first possible plane is (002) corresponding to the [001] direction. All the equivalent planes in (00h) will have the same solution and are presented as [001]. The precise index of h can be varied depending on the parameters of the crystal.

[5] Marin, C. and Dieguez, E., *Orientation of Single Crystals by Back Reflection Laue Pattern Simulation*, World Scientific, 1999.

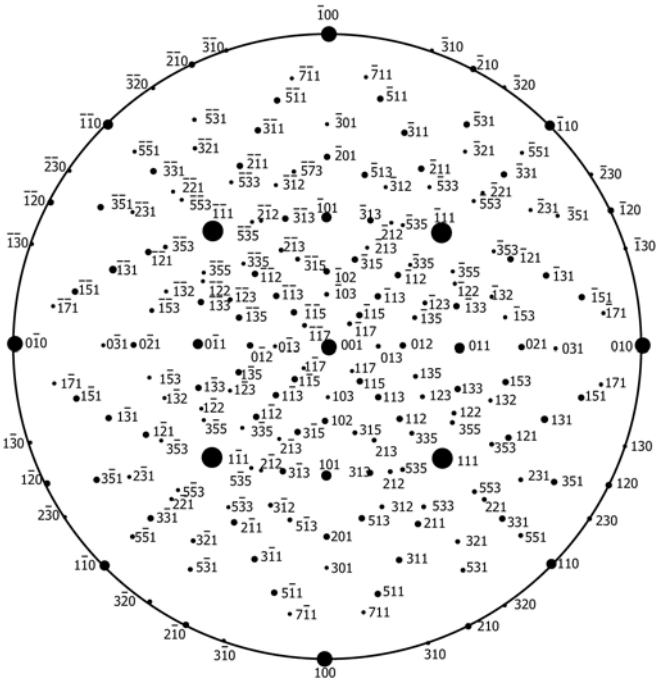

FIG. 4 Standard {001} Projection for a Cubic Crystal

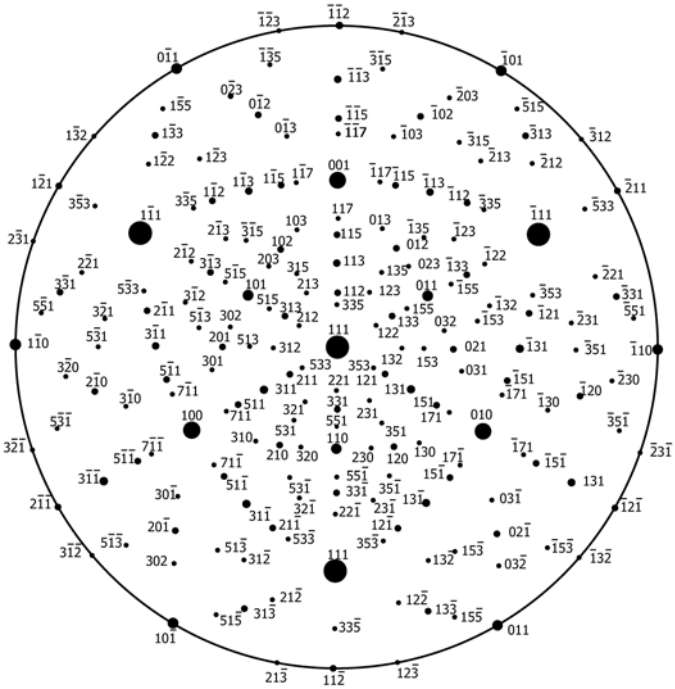

FIG. 5 Standard {111} Projection for a Cubic Crystal

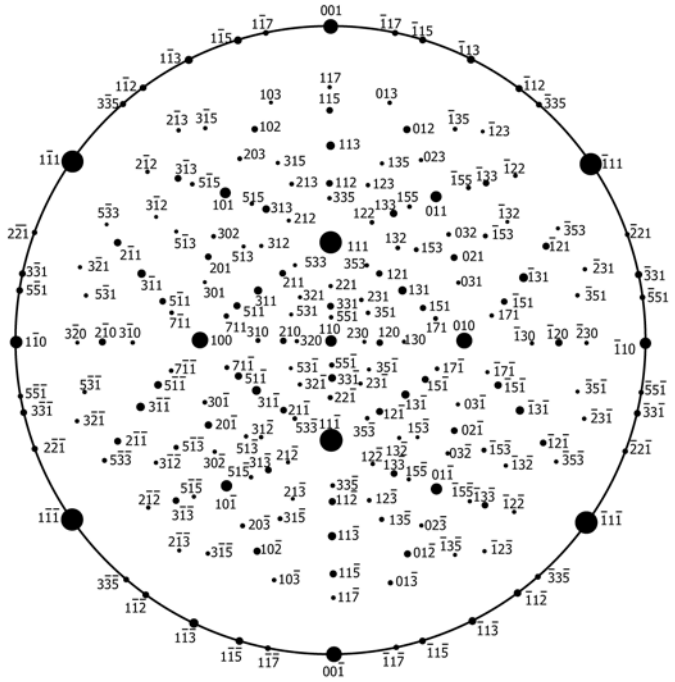

FIG. 6 Standard {110} Projection for a Cubic Crystal

7.3.4 *Basic Theory and Simulation*—In a cubic symmetry, the angles between different directions can be calculated using the basic dot product as follows: if plane one is oriented in the direction [u,v,w] and plane two is oriented in the direction [u',v',w'], then the subtended angle between these two directions θ is:

$$\theta = ArcCos\left(\frac{u.u'+v.v'+w.w'}{\sqrt{u^2+v^2+w^2}.\sqrt{u'^2v'^2+w'^2}}\right) \quad (1)$$

7.3.4.1 In general, to simplify the mathematical equations, the rotation angles around the (X,Y,Z) axes between the specimen and the crystal are defined in a Cartesian coordinates

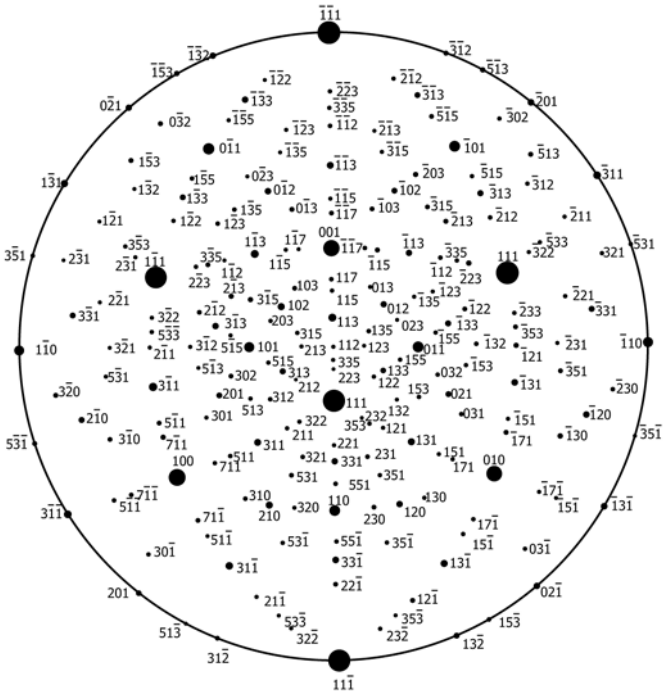

FIG. 7 Standard {112} Projection for a Cubic Crystal

as: α, β, and γ. The rotation matrix between the two reference frames is thus expressed as follows:

$$A = A_x A_y A_z = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\alpha & -\sin\alpha \\ 0 & \sin\alpha & \cos\alpha \end{pmatrix} \begin{pmatrix} \cos\beta & 0 & -\sin\beta \\ 0 & 1 & 0 \\ \sin\alpha & 0 & \cos\beta \end{pmatrix} \begin{pmatrix} \cos\gamma & -\sin\alpha & 0 \\ \sin\gamma & \cos\gamma & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (2)$$

7.3.4.2 The representation of the simulated LAUE pattern is a direct projection onto the detector plane. For side reflection, the simulated LAUE pattern is projected onto the plane whose normal is symmetrical with the incident beam. The sample surface normal in this case is the bisector of the incident beam and the diffracted beam.

7.3.4.3 Each $(h_i k_i l_i)$ in the crystal is calculated in the specimen coordinate system (x_i, y_i, z_i) as:

$$X_i = A_s A . H_j \; or \begin{pmatrix} x_i \\ y_i \\ z_i \end{pmatrix} = A_s A . \begin{pmatrix} u_i \\ v_i \\ w_i \end{pmatrix} \quad (3)$$

A_s is the rotation matrix for the side reflection setup.

7.3.4.4 The coordinates of the spots on the detector are calculated using the geometry detailed in Sands.[6]

7.3.4.5 The orientation matrix can be calculated using at least 3 spots. The angles of the three spots in spherical coordinates are expressed as follows:

$$\varphi_i = -ArcTan\left(\frac{y_i}{x_i}\right), \psi_i = -ArcTan\left(\frac{z_i}{\sqrt{x_i^2 + y_i^2}}\right) \quad (4)$$

7.3.4.6 Knowing the spherical coordinates φ, ψ, of at least three spots, the simulated pattern and the angular relation between the spots are combined to select the potential solutions. Among all of the solutions, the criteria based on the lowest error and the highest number of Laue spots matched are accepted as a solution. Other criteria can be added to the calculation to help find the solution quickly. The operator can refer to reference section of this document and published papers for further clarification.

7.3.4.7 In the case of a noncubic symmetry, the calculations will require orthogonalization of the reference frame. The main method uses a tensor β characteristic of each symmetry in either real or reciprocal space. In this case, the orientation matrix becomes:

$$A' = \beta A \quad (5)$$

NOTE 5—*Examples of Solutions for Back-Reflection Laue Patterns; Simulation Method*—Tourmaline- and nickel-base single-crystal Laue images with overlay simulated pattern are shown in Figs. 8 and 9. Data orientation angles are instantaneously calculated. Asymmetric back-reflection Laue patterns for tungsten crystals (body-centered cubic) are reproduced in Fig. 10 with a tracing of the important zones and spots. A measurement of the angles between the spots can measured using the old technique.

8. Precision and Bias

8.1 *Precision*—With reasonable care, the orientation of a crystal can be determined with an uncertainty of about ±½ °. With considerable care, the uncertainty may be improved to one or two tenths of a degree. It is recommended that the orientation be overdetermined (with many spots measured) because of the number and proximity of interplanar angles.

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

[6] Sands, Donald E., *Vectors and Tensors in Crystallography*, Dover Publication, New York, 1995.

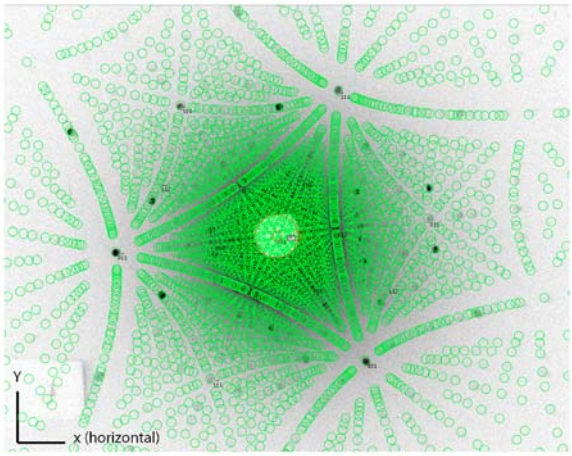

FIG. 8 Asymmetric Back-Reflection Laue Pattern of Tourmaline Crystal (Trigonal)

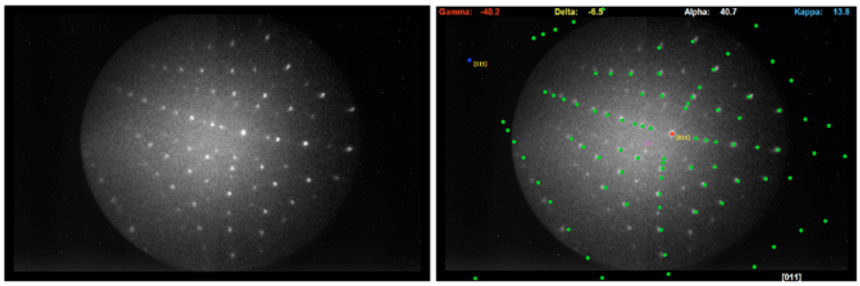

FIG. 9 Asymmetric Back-Reflection Laue Pattern of Nickel-Base Single Crystal (Face-Centered Cubic) with Overlay Simulated Pattern

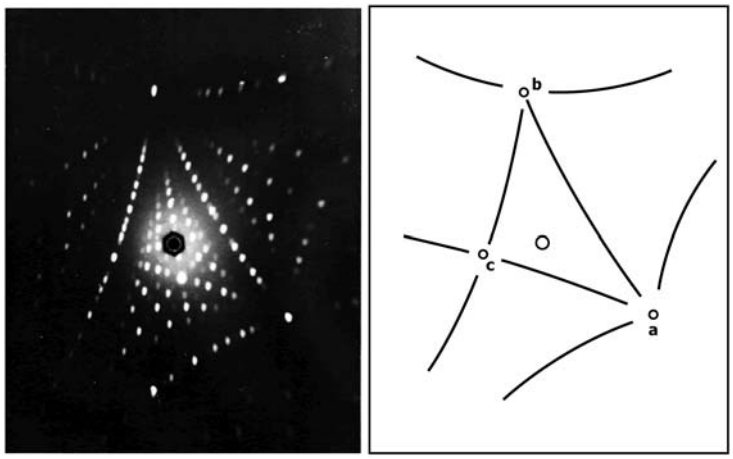

FIG. 10 Asymmetric Back-Reflection Laue Pattern of Tungsten Crystal (Body-Centered Cubic) and Tracing of Important Zones

9. Keywords

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