Designation: E81 – 96 (Reapproved 2024)

Standard Test Method for Preparing Quantitative Pole Figures[1]

This standard is issued under the fixed designation E81; 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 use of the X-ray diffractometer to prepare quantitative pole figures.

1.2 The test method consists of several experimental procedures. Some of the procedures (**1-5**)[2] permit preparation of a complete pole figure. Others must be used in combination to produce a complete pole figure.

1.3 Pole figures (**6**) and inverse pole figures (**7-10**) are two dimensional averages of the three-dimensional crystallite orientation distribution. Pole figures may be used to construct either inverse pole figures (**11-13**) or the crystallite orientation distribution (**14-21**). Development of series expansions of the crystallite orientation distribution from reflection pole figures (**22, 23**) makes it possible to obtain a series expansion of a complete pole figure from several incomplete pole figures. Pole figures or inverse pole figures derived by such methods shall be termed calculated. These techniques will not be described herein.

1.4 Provided the orientation is homogeneous through the thickness of the sheet, certain procedures (**1-3**) may be used to obtain a complete pole figure.

1.5 Provided the orientation has mirror symmetry with respect to planes perpendicular to the rolling, transverse, and normal directions, certain procedures (**4, 5, 24**) may be used to obtain a complete pole figure.

1.6 The test method emphasizes the Schulz reflection technique (**25**). Other techniques (**3, 4, 5, 24**) may be considered variants of the Schulz technique and are cited as options, but not described herein.

1.7 The test method also includes a description of the transmission technique of Decker, et al (**26**), which may be used in conjunction with the Schulz reflection technique to obtain a complete pole figure.

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Summary of Test Method

2.1 The test method consists of characterizing the distribution of orientations of selected lattice planes with respect to sample-fixed coordinates (**6**). The distribution will usually be obtained by measurement of the intensity of X rays diffracted by the sample. In such measurements the detector and associated limiting slits are fixed at twice the appropriate Bragg angle, and the diffracted intensity is recorded as the orientation of the sample is changed (**1-6, 25, 26, 27**). After the measured data have been corrected, as necessary, for background, defocusing, and absorption, and normalized to have an average value of unity, the results may be plotted in stereographic or equal-area projection.

2.2 The geometry of the Schulz (**25**) reflection method is illustrated in Fig. 1. Goniometers employing this geometry are commercially available. The source of X rays is indicated by *L*. Slit S1 limits divergence of the incident beam in the plane of projection. Slit S2 limits divergence perpendicular to the plane of projection. The sample, indicated by crosshatching, may be tilted about the axis *FF'*, which is perpendicular to the diffractometer axis and lies in the plane of the sample. The tilt angle was denoted φ by Schulz (**25**). The sample position shown in Fig. 1 corresponds to φ = 0 deg, for which approximate parafocusing conditions exist at the detector slit, S3. With the application of a defocusing correction, this method is useful over a range of colatitude φ from 0 deg to approximately 75 deg.

2.2.1 Tilting the sample about *FF* ', so as to reduce the distance between *L* and points in the sample surface above the plane of projection, causes X rays diffracted from these points to be displaced to the left of the center of S3, while X rays diffracted from points in the sample surface below the plane of

[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.

Current edition approved April 1, 2024. Published April 2024. Originally approved in 1949. Last previous edition approved in 2017 as E81 – 96 (2017). DOI: 10.1520/E0081-96R24.

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

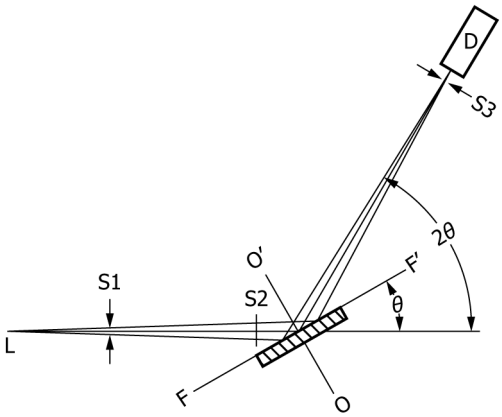

FIG. 1 Geometry of Reflection Method.

projection are displaced to the right of the center of S3. The displacement is equal to $2D \tan \varphi \cos \theta$, where D is the distance above or below the plane of projection. The integrated, or total, diffracted intensity is influenced only slightly by tilting the sample (**28**). Insofar as possible, the detector slit shall be of sufficient width to include the defocused line profile corresponding to the maximum sample tilt for which measurements are to be made. Because of interferences from neighboring diffraction peaks and physical limitations on sample size and detector slit width, it is necessary to limit vertical divergence of the incident beam. A widely used pole figure goniometer with a focal spot to the center of the sample distance of 172 mm employs a 0.5-mm slit located 30 mm from the center of the sample for this purpose. Measured intensities may be corrected for defocusing by comparison with intensities diffracted by a randomly oriented specimen of similar material, or byemploying the theoretically calculated corrections (**28**).

2.3 The geometry of the transmission technique of Decker, et al (**26**) is shown in Fig. 2. In contrast to the reflection method, X rays diffracted from different points in the sample diverge, making the resolution of adjacent peaks more difficult. The ratio of the diffracted intensity at $\alpha = -5, -10, \cdots, -70$ deg, to the diffracted intensity at $\alpha = 0$ deg, calculated in accordance with the expression given by Decker, et al (**26**) for linear absorption thickness product, μt, = 1.0, 1.4, ···, 3.0, and, for $\theta = 5, 10, \cdots, 25$ deg is given in Table 1. These data may be used as a guide to determine the useful range of α for a given

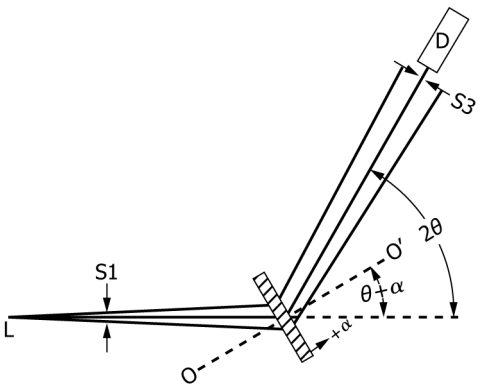

FIG. 2 Geometry of Transmission Method.

μt and θ. If, for example, I_α / I_0 is restricted to values ≥ 0.5, one arrives at the series of curves shown in Fig. 3.

3. Significance and Use

3.1 Pole figures are two-dimensional graphic representations, on polar coordinate paper, of the average distribution of crystallite orientations in three dimensions. Data for constructing pole figures are obtained with X-ray diffractometers, using reflection and transmission techniques.

3.2 Several alternative procedures may be used. Some produce complete pole figures. Others yield partial pole figures, which may be combined to produce a complete figure.

4. Apparatus

4.1 *Source of X Rays*—A beam of characteristic X rays of substantially constant intensity is required. Characteristic Kalpha radiation of chromium, iron, cobalt, nickel, copper, molybdenum, and silver have all been used successfully, depending on the chemical composition of the specimen. Insofar as possible, the radiation selected shall provide sufficient angular dispersion to permit the resolution of peaks to be measured, and shall not produce excessive fluorescence in the sample. Linear absorption coefficients (**29**) for selected elements are given in Table 2. Lower energy radiation (Cr, Fe, Co, Ni, Cu) is generally preferred for reflection pole figure measurements as it provides greater angular dispersion. Higher energy radiation (Mo, Ag) is generally preferred for transmission measurements.

4.2 *Slits*—Suitable slits shall be provided to limit horizon-tal (in the plane of projection of Figs. 1 and 2) and vertical (perpendicular to the plane of projection of Figs. 1 and 2) divergence of the incident beam. Horizontal divergences of 1 to 3 deg for reflection and 0.5 deg for transmission are typical. Vertical divergences of 0.2 deg for reflection and 1 deg for transmission are typical. Insofar as possible, the receiving slit shall be of sufficient width to include the diffracted peak. Receiving slits corresponding to 1 deg 2–theta are typical.

4.3 *Specimen Holder—Reflection Method:*

4.3.1 The specimen holder for the reflection method shall preferably employ the Schulz reflection geometry illustrated in Fig. 1 and described in 2.2. It is desirable that the specimen holder be equipped with a means for oscillating the sample in the plane of its surface without changing the orientation of the sample. It is also desirable that the magnitude of the oscillation be variable. The specimen holder shall preferably be provided with automatic means for changing colatitude and longitude of the sample.

4.3.2 Alternative reflection geometries include those of Bakarian (**1**), Field and Marchant (**27**), and Jetter and Borie (**2**). The method of Bakarian requires machining a number of cylindrical specimens whose axes are perpendicular to the sheet normal direction. Each specimen provides intensity data along one parallel of longitude. The method of Jetter and Borie entails the preparation of a spherical specimen. In the methods of Bakarian and of Jetter and Borie, the sample shall, insofar as possible, be prepared from homogeneous material. These methods have the advantage that intensity data need not be corrected for absorption or defocusing. They do not permit