Designation: E915 – 21

Standard Practice for Verifying the Alignment of X-Ray Diffraction Instruments for Residual Stress Measurement[1]

This standard is issued under the fixed designation E915; 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 practice describes the procedure for verifying the alignment of X-ray diffraction instruments used for residual stress measurements.

1.2 This practice further describes the use of iron powder for fabrication of a stress-free test specimen to be used to quantify the systematic error that can occur in residual stress measurement of ferritic or martensitic steels. This practice is easily adapted to other alloys and ceramics by the use of a powder having a similar diffraction angle to the material to be measured.

1.3 This practice is applicable to all X-ray diffraction instruments that measure diffracted X-rays from the crystal structure of a polycrystalline specimen. It is applicable to the acceptable multiple exposure techniques of residual stress measurement as defined in Test Method E2860. Through measurement of a high-angle back-reflection set of planes, these techniques are used to derive the interatomic spacing (d-spacing) and the crystallographic strain, and then calculate residual stress in which the θ, 2θ, and ψ rotation axes can be made to coincide (see Fig. 1).

1.4 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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.*

1.6 *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. Referenced Documents

2.1 *ASTM Standards:*[2]

E6 Terminology Relating to Methods of Mechanical Testing

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E1426 Test Method for Determining the X-Ray Elastic Constants for Use in the Measurement of Residual Stress Using X-Ray Diffraction Techniques

E2860 Test Method for Residual Stress Measurement by X-Ray Diffraction for Bearing Steels

3. Terminology

3.1 The definitions of calibration and verification that appear in Terminology E6 apply to this practice.

3.1.1 In addition, the following common terms from Terminology E6 are defined:

3.1.2 *error, n*—for a measurement or reading, the amount it deviates from a known or reference value represented by a measurement standard.

3.1.2.1 *Discussion*—Mathematically, the error is calculated by subtracting the accepted value from the measurement or reading.

3.1.3 *residual stress [FL^{-2}], n*—stress in a body that is at rest and in equilibrium and at uniform temperature in the absence of external and mass forces.

3.2 The terms accuracy, bias, and precision are used as defined in Practice E177.

4. Significance and Use

4.1 This practice provides a means of verifying the alignment of an X-ray diffraction instrument so as to quantify and minimize systematic experimental error in residual stress measurements. This practice is suitable for application to

[1] This practice is under the jurisdiction of ASTM Committee E28 on Mechanical Testing and is the direct responsibility of Subcommittee E28.13 on Residual Stress Measurement.

Current edition approved Nov. 1, 2021. Published January 2022. Originally approved in 1983. Last previous edition approved in 2016 as E915 – 16. DOI: 10.1520/E0915-21.

[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.

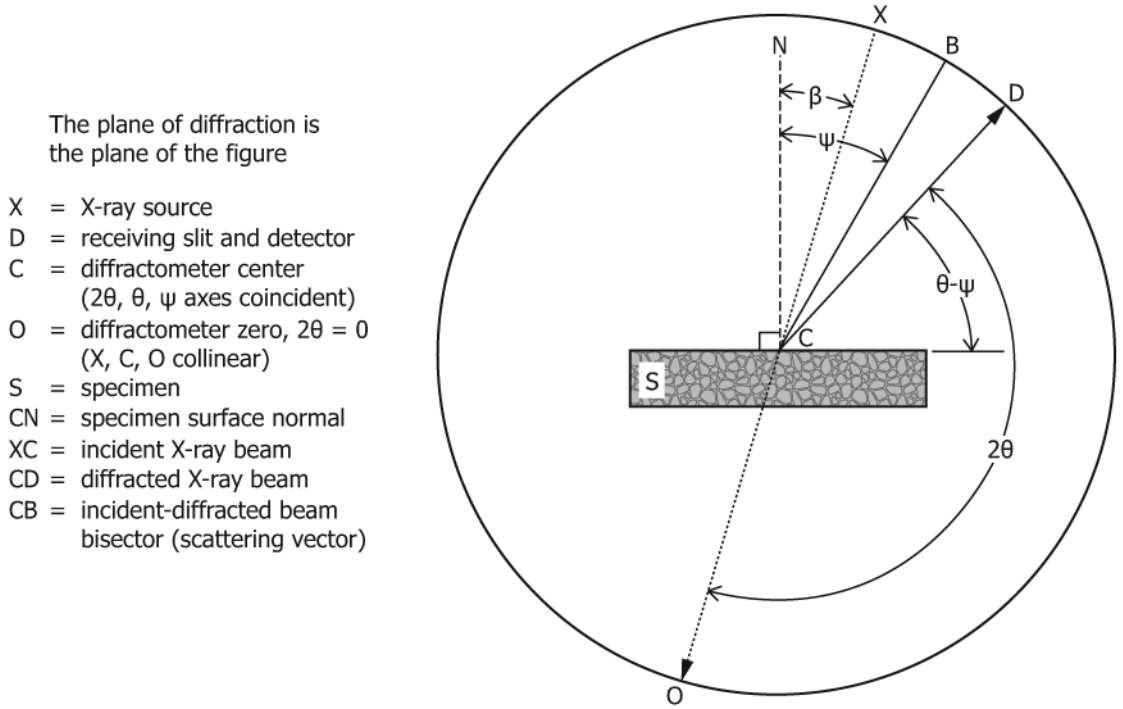

FIG. 1 X-Ray Diffraction Residual Stress Measurement Geometry and Angles Defined

conventional X-ray diffraction instruments or to X-ray diffraction residual stress measurement instruments of either the diverging or parallel beam types[3,4]

4.2 Application of this practice requires the use of a flat stress-free specimen that diffracts X-rays within the angular range of the diffraction peak to be used for subsequent residual stress measurements. The specimen shall have sufficiently small coherent domains or grains, be quasi-homogeneous, quasi-isotropic, and be of sufficient thickness such that incident X-rays interact with and diffract from an adequate number of individual coherent domains or grains such that a near-random grain orientation distribution is sampled. The crystals shall provide intense diffraction at all tilt angles ψ that will be employed.

NOTE 1—A major bias in crystal structure orientation is undesirable, but complete freedom from preferred orientation in the stress-free specimen is not critical in the application of the technique.

5. Procedure

5.1 *Instrument Alignment:*

5.1.1 The X-ray diffraction instrument shall be aligned in accordance with the instructions supplied by the instrument manufacturer. For variable or fixed θ, 2θ, and ψ axes (see Fig. 1), this alignment shall achieve the following:

5.1.1.1 The θ, 2θ, and ψ axes shall coincide at the diffraction center in Fig. 1.

5.1.1.2 The incident X-ray beam shall be centered on the ψ and 2θ axes within a chosen focal distance range. This focal distance range shall conform to the desired error and precision tolerances (see Section 6).

5.1.1.3 The X-ray tube focal spot, the ψ and 2θ axes, and the receiving slit positioned at 2θ = 0° shall lie along a line in the plane of diffraction. Alternatively, for instruments limited to the back-reflection region, the diffraction angle 2θ shall be calibrated in accordance with the instrument manufacturer specifications.

5.1.1.4 The specimen position shall be established, such that the surface of the specimen is positioned at the θ and ψ axes within a chosen focal distance range. This focal distance range shall conform to the desired error and precision tolerances (see Section 6).

5.1.1.5 Values for accuracy and precision of the various ψ angles and displacements may be considered to be met collectively when overall measurement errors and tolerances are within those specified in Section 6.

5.2 *X-Ray Optics:*

5.2.1 Selection of an appropriate X-ray peak should be made at the highest diffraction angle possible, consistent with peak intensity, Bragg reflection multiplicity, and the X-ray wavelength used.

5.2.2 For the purpose of verifying alignment, residual stress measurements on the stress-free specimen shall employ the $K\alpha_1$ diffraction peak at all ψ angles of interest. This is in contrast to residual stress measurements where the Kα characteristic radiation doublet is used to improve accuracy. Alternatively, Kβ peaks may also be used. Although Kβ peaks are generally less intense than Kα peaks they are not doublet peaks, so subsequent analysis on test articles is simplified.

[3] Ahmad, A., Prevéy, P, Ruud, C.O., , eds., *Residual Stress Measurement by X-ray Diffraction*, SAE HS-784, Society of Automotive Engrs., Inc., Warrendale, PA (2003).

[4] "Standard Method for X-Ray Stress Measurement," *Committee on Mechanical Behavior of Materials*, The Society of Materials Science, Japan, (20 April 1973).

5.2.3 The incident and receiving X-ray beam optics should be selected to produce maximum separation of the $K\alpha_1 - K\alpha_2$ doublet. Because resolution of the Kα doublet can vary with the angle ψ, and because some instruments can be incapable (due to fixed X-ray optics) of obtaining resolution of the doublet, care shall be taken not to resolve the doublet at some ψ angles while blending the doublet into a single peak at other ψ angles.

5.2.4 To avoid systematic error in the verification process when Kα radiation is being used, care shall be taken either to split completely or to blend the $K\alpha_1$–$K\alpha_2$ doublet (see 5.2.3 and 5.3.7), or consistently define the peak position by fitting to a single equation summing the $K\alpha_1$ and $K\alpha_2$ contributions.

5.3 *Selection of Powder for a Stress-Free Iron Specimen:*

5.3.1 Iron powder with a particle size greater than 1 µm (4×10^{-5} in.) shall be used whenever possible.

NOTE 2—Annealed iron powder of <45 µm (325 mesh) has been found suitable when using Cr Kα X-ray radiation.

5.3.2 This practice may be applied to other metallic alloys and ceramics (see 1.3).

5.3.3 Crystallographic strain may be reported instead of stress to circumvent the necessity of establishing applicable elastic constants and thereby eliminate a source of uncertainty.

NOTE 3—Crystallographic strains differ from macroscopic strains in most materials since the single crystal elastic properties in most materials are anisotropic. (See Test Method E1426.)

5.3.4 The iron (or other) powder may be annealed under vacuum so as to reduce the diffraction peak width, increase the diffracted intensity, and subsequently, improve the peak position resolution. This is generally desirable.

5.3.4.1 An oxide-forming powder may be annealed in a reducing atmosphere rather than in vacuum to avoid problems from surface contamination.

5.3.4.2 Annealing ceramic powders may be omitted, since these materials do not tend to exhibit peak broadening due to plastic deformation.

5.3.5 Although less ideal, annealed powders in the form of filings may be used.

NOTE 4—Use of filings can produce broader diffraction peaks due to plastic deformation; the broadening can persist even after annealing.

5.3.6 In the event that an instrument incapable of resolving the $K\alpha_1 - K\alpha_2$ doublet is being employed, deliberately plastically deformed powders may be used to ensure that partial resolution of the Kα doublet does not occur.

5.3.6.1 Extremely fine powders with a particle size less than 1 µm (4×10^{-5} in.) may also be used because they have been shown to produce peak broadening sufficient to suppress resolution of the Kα doublet.

5.3.7 The need for broader peaks or full resolution may be eliminated by fitting the observed, either fully resolved, partially resolved, or unresolved, doublet to a single equation defined by the sum of the $K\alpha_1$ and $K\alpha_2$ contributions. The peak position is then given by the fitted $K\alpha_1$ position.

5.4 *Stress-Free Specimen Preparation*—Preparation methods other than those described below may be used providing that no residual stress (strain) is sustained in the binder that may be used to hold the crystalline particles together.

5.4.1 A permanent stress-free specimen may be prepared by mounting the chosen powder on the face of a microscope slide or in a shallow powder tray (of the type used for powder diffraction work) using a binder.

NOTE 5—A commonly suitable choice of binder is a 10 % solution of nitrocellulose cement diluted with acetone.

5.4.1.1 Place several drops of the binder solution on a clean microscope slide or in a powder tray, and sprinkle the powder into the binder solution.

5.4.1.2 The binder shall not diffract in the high-angle back-reflection region such that it interferes with the diffraction peaks of interest. If peak interference occurs, another binder shall be chosen such that it does not interfere with the diffraction peaks of interest.

5.4.1.3 Spread and level the powder with a second microscope slide. When a uniform flat surface has been produced by alternately wetting with the binder solution and wiping with a second slide, set the specimen aside and allow it to dry for several hours.

NOTE 6—Excess amounts of the binder solution can cause the powder layer to peel away from the surface of the microscope slide. Rewetting of the surface with acetone and redrying often eliminates this difficulty

5.4.1.4 Make the surface of the specimen as flat as possible so that the specimen surface is clearly defined and consistent across the surface.

5.4.2 Alternatively, a specimen may be prepared as follows:

5.4.2.1 Place a small quantity of petroleum jelly on the face of one microscope slide and press it against a second slide to extrude the petroleum jelly into a uniform flat film.

5.4.2.2 Remove the second microscope slide with a wiping action taking care to keep the surface layer of petroleum jelly thin and flat.

5.4.2.3 Holding the petroleum jelly-coated slide so that its surface is near to vertical, sprinkle the iron powder from a sufficient height above the slide so that the powder strikes the coated surface and either adheres or is deflected away. Do not allow the powder to pack and build up on the surface. For instruments that do not tilt or rapidly rotate the specimen during the measurement of residual stress, the petroleum jelly binder may be omitted.

5.4.3 The surface area of the powder shall be of sufficient size to intersect the entire incident X-ray beam at all ψ angles to be used during the measurement of residual stress.

5.5 *X-ray Diffraction Instrument Alignment Verification:*

5.5.1 The X-ray diffraction instrument shall be aligned in accordance with the instructions supplied by the instrument manufacturer. Position the stress-free (strain-free) specimen in line with the X-ray aperture of the X-ray diffraction instrument (see 5.1.1.4).

NOTE 7—Failure to place the powder surface on the center of rotation of the ψ and 2θ axes induces a systematic specimen displacement error.

5.5.2 In the event that a mechanical gage that contacts the surface of the specimen is used for specimen positioning, a thin metal shim may be placed in front of the powder surface to protect it. Place this gage against the face of the metal shim,