Designation: ~~E915 – 19~~ E915 – 21

Standard ~~Test Method~~ Practice for Verifying the Alignment of X-Ray Diffraction ~~Instrumentation~~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 test method covers the procedure for preparation and usage of a stress-free test specimen for the purpose of verifying the systematic error caused by instrument misalignment and/or sample positioning during X-ray diffraction residual stress measurement.~~

~~1.2 This test method is applicable to all X-ray diffraction instruments that measure diffracted X-rays from the crystal structure of a polycrystalline specimen and is applicable to single, double, and multiple exposure techniques. Through measurement of a high-angle back-reflection, these techniques are used to derive the interatomic spacing (d-spacing) and the macroscopic strain and to then calculate residual stress in which the θ, 2θ, and ψ rotation axes can be made to coincide (see Fig. 1).~~

~~1.3 This test method describes the use of iron powder for fabrication of the stress-free test specimen that is used to verify the systematic error for residual stress measurement of ferritic or martensitic steels. This test method is easily adapted to other alloys and ceramics through use of powder having a similar diffraction angle as the material to be measured.~~

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

~~1.5 *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~~

~~3. Terminology~~

~~3.1 The definitions of mechanical testing terms that appear in Terminology E6 apply to this test method.~~

[1] This ~~test method~~ 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.

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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.

The plane of diffraction is the plane of the figure

X = X-ray source
D = receiving slit and detector
C = diffractometer center (2θ, θ, ψ axes coincident)
O = diffractometer zero, 2θ = 0 (X, C, O collinear)
S = specimen
CN = specimen surface normal
XC = incident X-ray beam
CD = diffracted X-ray beam
CB = incident-diffracted beam bisector (scattering vector)

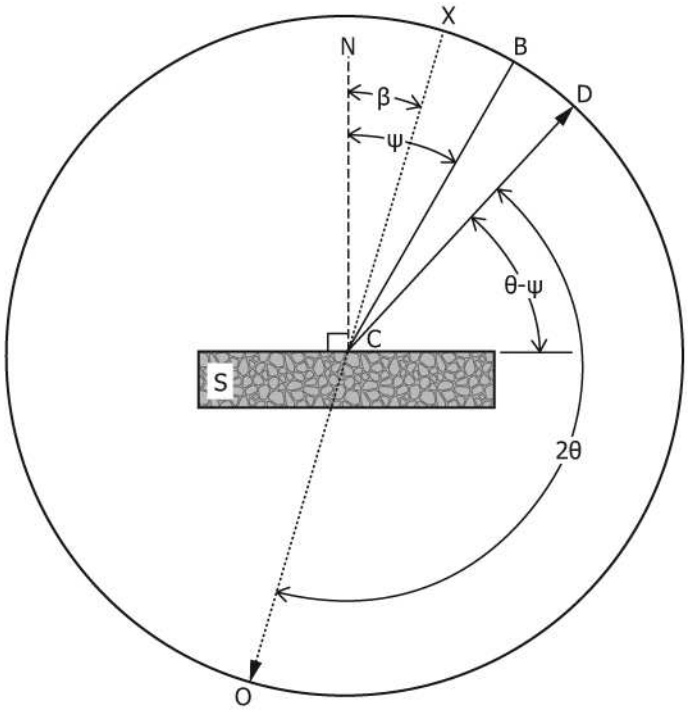

FIG. 1 X-Ray Diffraction Residual Stress Measurement Geometry and Angles Defined

3.1.1 In addition, the following common term from Terminology E6 is defined:

3.1.2 *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.

4. Significance and Use

4.1 This test method provides a means of verifying the alignment of an X-ray diffraction instrument in order to quantify and minimize systematic experimental error in residual stress measurement. This method is suitable for application to conventional diffractometers or to X-ray diffraction instrumentation designed for residual stress measurement of either the diverging or parallel beam types.[3,4]

4.2 Application of this test method 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 residual stress measurement. The specimen shall be sufficiently fine-grained, homogeneous, isotropic, and of sufficient depth such that incident X-rays interact with and diffract from an adequate number of individual coherent domains and or grains such that a near random grain orientation distribution is sampled. The crystals shall provide intense diffraction at all angles of tilt, ψ, which will be employed (see Note 1).

NOTE 1—Complete freedom from preferred orientation in the stress free specimen is, however, 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. In general, this alignment shall achieve the following, whether the θ, 2θ, and ψ axes are variable or fixed (see Fig. 1):

[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).

5.1.1.1 The θ, 2θ, and ψ axes shall coincide.

5.1.1.2 The incident X-ray beam shall be centered on the ψ and 2θ axes, within a focusing range, which will conform to the desired error and precision tolerances (see Sections 6 and 7).

5.1.1.3 The X-ray tube focal spot, the ψ and 2θ axes, and the receiving slit positioned at 2θ equals zero degrees shall be on a line in the plane of diffraction. Alternatively, for instrumentation limited to the back reflection region, the diffraction angle 2θ shall be calibrated.

5.1.1.4 The proper sample position shall be established, such that the surface of the sample is positioned at the θ and ψ axes, within the focal distance range which will conform to the desired error and precision tolerances (see Sections 6 and 7).

5.1.1.5 The angle ψ shall be determined accurately. Values for accuracy and precision of the various angles and displacements are not specified herein. These may be considered to be met collectively when overall measurement errors and tolerances are within those specified in Sections 6 and 7.

5.2 *X-Ray Optics:*

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

5.2.2 For the purpose of verifying systematic errors, 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 actual stress measurements where the Kα characteristic radiation doublet is used to improve accuracy. Kβ peaks may also be used; although they are generally less intense than Kα lines they are not doublet peaks, so subsequent analysis on test articles is simplified.

5.2.3 It is desirable to select incident and receiving X-ray beam optics that will produce maximum separation of the $K\alpha_1 - K\alpha_2$ doublet. Because resolution of the Kα doublet may vary with the angle ψ, and because some instrumentation may 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.

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

5.3.1 Use iron powder with a particle size greater than 1 µm (4×10^{-5} in.) (See Note 2.)

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

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

5.3.3 The reporting of crystallographic strain instead of stress circumvents the necessity of establishing applicable elastic constants and serves to eliminate a source of uncertainty. It should be noted that crystallographic strains differ from macroscopic strains in most materials since the single crystal elastic properties in most materials are anisotropic.

5.3.4 The iron (or other) powder under vacuum may be annealed so as to reduce the diffraction peak width, and thereby increase the diffracted intensity and subsequently, the peak position resolution. This is generally desirable (see Note 3). Powders in the form of filings may be used, but may produce broader diffraction peaks due to plastic deformation; the broadening can persist even after annealing.

NOTE 3—It can be advantageous to anneal an oxide-forming powder in a reducing atmosphere rather than in vacuum to avoid problems from surface contamination. I may not be necessary to anneal ceramic powders since these materials do not tend to exhibit line broadening due to plastic deformation.

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

5.3.6 Extremely fine powders have also been shown to produce line 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 or partially resolved or unresolved, doublet to a single equation defined by the sum of the $K\alpha_1$ and $K\alpha_2$ contributions; then the peak position is given by that of 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 (strain-free) specimen may be prepared by mounting the powder on the face of a microscope slide or in a shallow powder tray (of the type used for powder diffraction work on a diffractometer) using a 10 % solution of nitrocellulose cement diluted with acetone. The binder should not diffract in the high-angle back-reflection region and interfere with the diffraction peaks of interest. Place several drops of the solution on a clean microscope slide or in a sample tray, and sprinkle the powder into the binder. The powder may be spread and leveled 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. Excess amounts of the binder may cause it to peel away from the surface of the microscope slide. Rewetting of the surface with acetone and redrying may eliminate this difficulty. 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 A temporary specimen may be rapidly prepared using petroleum jelly as an amorphous binder. 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. Remove the second microscope slide with a wiping action taking care to keep the surface layer of petroleum jelly thin and flat. Holding the petroleum jelly-coated slide at a steep angle to a vertical line, 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 sample during the measurement of residual stress, a binder may not be necessary.

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). (See Note 4.)

NOTE 4—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 which 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, and adjust the positioning to account for the inclusion of the shim in front of the gage such that the surface of the powder is at the correct distance from the reference point of the gage for residual stress measurement.

5.5.3 Without adjusting the specimen position, perform five successive residual stress measurements using the method and correction procedures normally employed for the instrument, including positive and negative ψ tilts when applicable, where ψ is the angle between the specimen surface normal and the diffracting plane normal.[5] ψ splitting is a symptom of misalignment.

5.5.4 The differential between the split portions of the least squares fit d-spacing versus $\sin^2(\psi)$ data shall result in a shear stress equivalent to or less than ±14 MPa (±2.0 ksi). To avoid systematic error in the verification process when Kα radiation is being used, care shall be taken to either completely split or blend the $K\alpha_1 - K\alpha_2$ doublet (see 5.2), or consistently define the peak position by fitting to a single equation summing the $K\alpha_1$ and $K\alpha_2$ contributions.

6. Calculations and Interpretation of Results

6.1 *Systematic Error*—All methods leading to the calculation of both in-plane and shear stresses may be employed. These methods are based on the calculation of the slope and the opening of the d-spacing versus $\sin^2(\psi)$ values or fitting to an ellipse.

6.1.1 Reduce the X-ray diffraction data obtained from the five measurements in whatever manner is normally employed for the

X-ray diffraction instrument in use, and include all corrections normally applied to raw X-ray diffraction data. Application of the X-ray elastic constants appropriate for the stressed material to be measured is important. It can be advantageous to report strain values, rather than stress, to avoid the uncertainty of specifying elastic constants. Calculate the simple arithmetic mean and standard deviation about the mean for the five measurements. If the mean value is within ±14 MPa (±2.0 ksi) of zero, the instrument is properly aligned. In the event that the mean differs from zero by more than ±14 MPa (±2.0 ksi), repeat 5.1 and 5.5.

6.1.2 Alternatively, strain values may be used. This avoids error due to selection of inappropriate elastic constants. The acceptable mean normal strain ±100 ppm and the acceptable mean shear strain is ±50 ppm with respect to the stress-free (strain-free) d-spacing.

6.2 *Random Error:*

6.2.1 Experience has shown that the standard deviation of the five measurements using a well aligned goniometer is ≤ 7MPa (±1.0 ksi). In the event that the standard deviation of the five measurements exceeds ±14 MPa (±2.0 ksi), the stress-measurement technique employed and the instrumentation shall be investigated for other sources of error affecting the measurement precision. Error due to counting statistics can result from a failure to take sufficient time during the measurement to obtain precise intensity information, and to subsequently determine the precise diffraction peak positions

6.2.2 Methods are available[5] for estimating the standard deviation of the measured stress that is related to counting statistics, and fitted peak positions. Mechanical sources of error such as loose bearings and ways within the X-ray diffraction instrument can result in significant random error

6.2.3 When strain values are reported, the standard deviation of the five measurements shall be ≤100 ppm and ≤50 ppm for shear stress.

7. Precision and Bias

7.1 The precision of this method will be dependent upon the type of X-ray diffraction instrumentation employed and the methods of data reduction used in residual stress measurement. The preliminary results of round-robin investigations using this method indicate that instrument alignment resulting in measured stress of stress-free powders of zero ±14 MPa (±2.0 ksi) (see 6.1) was achieved for both standard diffractometers and X-ray diffraction instrumentation designed for residual stress measurement in the back reflection region only. Instrumental precision measured by this method (see 6.2) has been found to be less than ±7 MPa (±1.0 ksi).

7.2 The accuracy of this method is considered to be absolute because the specimen is stress-free. Deviation of results obtained when using this method, provided the specimen has been properly prepared and maintained, are attributed to the instrumentation under investigation.

7.3 Additional sources of error may be related to other factors, including the quality of the diffracted X-ray peaks (background and noise). They may be classified as either instrumental/equipment or specimen-related factors. If other errors are suspected, the use of a high-strain standard is recommended.

7.4 In some cases, depending on the material being used, the specified average stress (strain) precision may not be achievable. Thus, users should investigate the issue or choose a different stress-free (strain-free) material.

8. Keywords

8.1 alignment; residual stress; stress-free specimen; systematic error; x-ray diffraction

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 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