Designation: E915 – 16

Standard Test Method for Verifying the Alignment of X-Ray Diffraction Instrumentation 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 preparation and use of a flat stress-free test specimen for the purpose of checking the systematic error caused by instrument misalignment or sample positioning in X-ray diffraction residual stress measurement, or both.

1.2 This test method is applicable to apparatus intended for X-ray diffraction macroscopic residual stress measurement in polycrystalline samples employing measurement of a diffraction peak position in the high-back reflection region, and 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 which has been investigated in round-robin studies for the purpose of verifying the alignment of instrumentation intended for stress measurement in ferritic or martensitic steels. To verify instrument alignment prior to stress measurement in other metallic alloys and ceramics, powder having the same or lower diffraction angle as the material to be measured should be prepared in similar fashion and used to check instrument alignment.

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]

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.

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

4.2 Application of this test method requires the use of a flat specimen of stress-free material that produces diffraction in the angular region of the diffraction peak to be used for stress measurement. The specimen must be sufficiently fine-grained and isotropic so that large numbers of individual crystals contribute to the diffraction peak produced. The crystals must 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 stressfree specimen is, however, not critical in the application of the technique.

5. Procedure

5.1 *Instrument Alignment:*

5.1.1 Align the X-ray diffraction instrumentation to be used for residual stress measurement in accordance with the instructions supplied by the manufacturer. In general, this alignment must achieve the following, whether the θ, 2θ, and ψ axes are variable or fixed (see Fig. 1):

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

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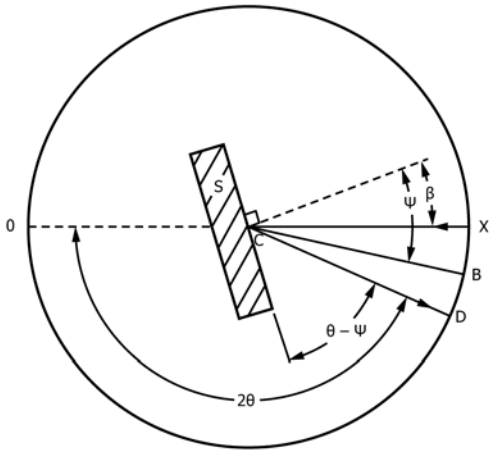
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Hilley, M. E., Larson, J. A., Jatczak, C. F., and Ricklefs, R. E., eds., Residual Stress Measurement by X-ray Diffraction, *SAE J784a*, Society of Automotive Engrs., Inc., Warrendale, PA (1971).

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



NOTE— The plane of diffraction is the plane of the figure, and:
X = x-ray source,
D = receiving slit and detector,
C = diffractometer center (2*θ*, *θ*, *ψ* axes coincident),
O = diffractometer zero, 2*θ* = 0 (O, C, X colinear),
S = sample,
CN = sample surface normal,
XC = incident x-ray beam,
CD = diffracted x-ray beam, and
CB = incident-diffracted beam bisector.

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

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, using whatever means are provided with the instrument, 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 must be determined accurately. (see Note 5)

5.2 *X-Ray Optics:*

5.2.1 Appropriate X-ray peak selection should be made at the highest diffraction angle possible, consistent with peak intensity, and this may include selection of the x-radiation to be used.

5.2.2 When the Kα characteristic radiation doublet is used for stress measurement, 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. Perform stress measurements on the stress-free specimen employing the $K\alpha_1$ diffraction peak at all ψ angles investigated. 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 must 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.)

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

5.3.3 The reporting of strain instead of stress circumvents the necessity of establishing applicable elastic constants and serves to eliminate a source of uncertainty.

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

5.3.4 Annealing of the powder in vacuum reduces diffraction peak width, thereby increasing diffraction peak resolution. This is generally desirable (see Note 3). Powders in the form of plastically deformed filings may be used, but will produce broader diffraction peaks. In the event that an instrument incapable of resolution of the $K\alpha_1 - K\alpha_2$ doublet is being employed, it may be desirable to deliberately obtain plastically deformed powders which insure that partial resolution of the Kα doublet does not occur. Extremely fine powders have also been shown to produce line broadening, sufficient to suppress resolution of the Kα doublet.

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

5.4 *Stress-Free Specimen Preparation*—Preparation methods other than those described below are permissible providing that no residual stress (strain) is sustained in the binder that might 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 as a suitable amorphous binder. 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.

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.

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

5.5 *Instrument Alignment Check:*

5.5.1 Position the stress-free (strain-free) specimen on the X-ray diffraction apparatus (see 5.1.1.4). 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 stress measurement.

NOTE 4—Failure to place the powder surface directly over the center of rotation of the ψ and 2θ axes induces a systematic specimen displacement error.

5.5.2 Without adjusting the specimen position, perform five successive stress measurements using the method and correction procedures normally employed for the instrument, including positive and negative psi tilts when applicable. Psi splitting is a symptom of misalignment where psi is the angle between the specimrn surface normal and the diffracting plane normal.[5] The strain differential between the split linear portions of the least square fit sin-square-psi plots should be equivalent to less than 14 Mpa (2ksi). To avoid systematic error in the verification process when Kα radiation is being used, care must be taken to either completely split or blend the $K\alpha_1 - K\alpha_2$ doublet (see 5.2).

NOTE 5—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] SAE, "*Residual Stress Measurement by X-ray Diffraction*", 2003 Edition, HS-784, p. 17.

6. Calculations and Interpretation of Results

6.1 *Systematic Error*—All methods leading to the calculation of both in-plane and shear stresses can be employed. These methods are based on the calculation of the slope and the opening of the d-spacing versus sin-square-psi values.

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 instrumentation 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 may 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 and specimen-positioning gage can be considered to be 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 strain mean would be 100 ppm of the stress-free (strain-free) d-spacing; 50 ppm for shear strain.

6.2 *Random Error:*

6.2.1 Experience has shown that the standard deviation of the five measurements should be within approximately 6.9 MPa (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 should be investigated for sources of random error affecting the measurement precision. Random error due to counting statistics may result from failure to take sufficient time during the measurement to obtain accurate intensity information, and thus to accurately determine the diffraction peak positions. Methods are available[3] for estimating the standard deviation of the measured stress due to the errors involved in counting and curve fitting to determine peak positions. Mechanical sources of error such as loose bearings and ways in the apparatus may result in significant random error.

6.2.2 When strain values are reported the standard deviation of the five measurements should be within 100 ppm; 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 stress measurement. The preliminary results of round-robin investigations using this method indicate that instrument alignment within 14 MPa (2.0 ksi) (see 6.1) can be achieved for both standard diffractometers and two types of X-ray diffraction instrumentation designed for stress measurement in the back reflection region only. Instrumental precision measured by this method (see 6.2) has been found to be less than $\pm$6.9 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 in performing this method, provided the specimen has

been properly prepared and maintained, can be attributed to the instrumentation under investigation.

7.3 Other sources of error can be related to different factors, such as the quality of the diffracted X-ray peaks (background and noise). In some cases, depending on the material, the average stress (strain) precision may not be achievable. Thus, users may need to investigate the issue or choose a different stress-free (strain-free) material.

8. Keywords

8.1 alignment; residual stress; x-ray diffraction