Designation: E915 – 10

StandardTest 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]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 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]

3.2 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.

4. Procedure

4.1 *Instrument Alignment:*

4.1.1 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):

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

4.1.1.2 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 5 and 6).

4.1.1.3 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.

4.1.1.4 The proper sample position shall be established, using whatever means are provided with the instrument, such

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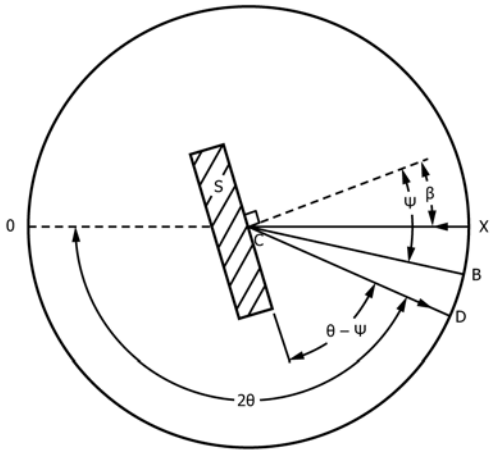
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[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

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 5 and 6).

4.1.1.5 The angle ψ must be determined accurately. (see Note 5)

4.2 *X-Ray Optics:*

4.2.1 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.

4.2.2 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.

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

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

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

4.3.3 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.

4.3.4 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.

4.4 *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.

4.4.1 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