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Standard Test Method for Determining the X-Ray Elastic Constants for Use in the Measurement of Residual Stress Using X-Ray Diffraction Techniques[1]

This standard is issued under the fixed designation E1426; 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.

INTRODUCTION

When a crystalline material is strained, the spacing between parallel planes of atoms, ions, or molecules in the lattice changes. X-Ray diffraction techniques can measure these changes and, therefore, they constitute a powerful means for studying the residual stress state in a body. The calculation of macroscopic stresses using lattice strains requires the use of x-ray elastic constants (*XEC*) which must be empirically determined by x-ray diffraction techniques as described in this test method.

1. Scope

1.1 This test method covers a procedure for experimentally determining the x-ray elastic constants *(XEC)* for the evaluation of residual and applied stresses by x-ray diffraction techniques. The *XEC* relate macroscopic stress to the strain measured in a particular crystallographic direction in polycrystalline samples. The *XEC* are a function of the elastic modulus, Poisson's ratio of the material and the *hkl* plane selected for the measurement. There are two *XEC* that are referred to as $\frac{1}{2} S_2^{hkl}$ and S_1^{hkl}.

1.2 This test method is applicable to all x-ray diffraction instruments intended for measurements of macroscopic residual stress that use measurements of the positions of the diffraction peaks in the high back-reflection region to determine changes in lattice spacing.

1.3 This test method is applicable to all x-ray diffraction techniques for residual stress measurement, including single, double, and multiple exposure techniques.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E4 Practices for Force Verification of Testing Machines
E6 Terminology Relating to Methods of Mechanical Testing
E7 Terminology Relating to Metallography
E1237 Guide for Installing Bonded Resistance Strain Gages

3. Terminology

3.1 *Definitions:*

3.1.1 Many of the terms used in this test method are defined in Terminology E6 and Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *interplanar spacing*—the perpendicular distance between adjacent parallel lattice planes.

3.2.2 *macrostress*—an average stress acting over a region of the test specimen containing many crystals.

3.3 *Symbols:*

3.3.1 x = dummy parameter for $Sum(x)$ and $SD(x)$.

3.3.2 c = ordinate intercept of a graph of Δd versus stress.

3.3.3 d = interplanar spacing between crystallographic planes; also called d-spacing.

3.3.4 d_0 = interplanar spacing for unstressed material.

3.3.5 Δd = change in interplanar spacing caused by stress.

[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.

3.3.6 E = modulus of elasticity.
3.3.7 ν = Poisson's ratio.
3.3.8 XEC = x-ray elastic constants for residual stress measurements using x-ray diffraction.
3.3.9 hkl = Miller indices.
3.3.10 $\frac{1}{2} S_2^{hkl} = (1+\nu)/E$ for an elastically isotropic body.
3.3.11 $S_1^{hkl} = -\nu/E$ for an elastically isotropic body.
3.3.12 i = measurement index, $1 \leq i \leq n$.
3.3.13 m = slope of a plot of Δd versus stress.
3.3.14 n = number of measurements used to determine slope m.
3.3.15 $SD(x)$ = standard deviation of a set of quantities "x".
3.3.16 $Sum(x)$ = sum of a set of quantities "x".
3.3.17 $T_i = X_i$ minus mean of all X_i values.
3.3.18 X_i = i-th value of applied stress.
3.3.19 Y_i = measurement of Δd corresponding to X_i.
3.3.20 ψ = angle between the specimen surface normal and the normal to the diffracting crystallographic planes.
3.3.21 ϕ = the in-plane direction of stress measurement.
3.3.22 ij = in-plane directions of the sample reference frame.
3.3.23 σ_{ij} = calculated stress tensor terms.
3.3.24 $\varepsilon_{\phi\psi}^{hkl}$ = measured lattice strain tensor terms at a given $\phi\psi$ tilt angle.
3.3.25 σ^A = applied stress.
3.3.26 ε_{max} = maximum strain.
3.3.27 δ_{max} = maximum deflection.
3.3.28 h = specimen thickness.
3.3.29 b = width of specimen.
3.3.30 A_X = cross sectional area of specimen.
3.3.31 L = distance between outer rollers on four-point bend fixture.
3.3.32 a = distance between inner and outer rollers on four-point bend fixture.
3.3.33 F = known force applied to specimen.
3.3.34 $\varepsilon_{\phi\psi 0}$ = the intercept value for each applied force necessary for S_1 calculation.

4. Theory

4.1 The $\sin^2\psi$ method is widely used to measure stresses in materials using x-ray diffraction techniques. The governing equation can be written as follows:[3,4]

$$\varepsilon_{\phi\psi}^{hkl} = \frac{1}{2}S_2^{hkl}(\sigma_{11}\cos^2\phi + \sigma_{12}\sin 2\phi + \sigma_{22}\sin^2\phi - \sigma_{33})\sin^2\psi + \tag{1}$$

$$\frac{1}{2}S_2^{hkl}\sigma_{33} + S_1^{hkl}(\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1}{2}S_2^{hkl}(\sigma_{13}\cos\phi + \sigma_{23}\sin\phi)\sin 2\psi$$

where:

$\frac{1}{2} S_2^{hkl}$ *and* S_1^{hkl} = are the XEC.

For a body that is elastically isotropic on the microscopic scale, $\frac{1}{2} S_2^{hkl} = (1+\nu)/E$ and $S_1^{hkl} = -(\nu/E)$ where E and ν are the modulus of elasticity and Poisson's ratio respectively for the material for all hkl.

4.2 When a uniaxial force is applied along e.g. $\phi = 0$, Eq 1 becomes:

$$\varepsilon_{\phi\psi}^{hkl} = \frac{1}{2}S_2^{hkl}\sigma^A\sin^2\psi + S_1^{hkl}\sigma^A \tag{2}$$

where:

σ^A = the applied stress due to the uniaxial force.

Therefore:

$$\frac{1}{2}S_2^{hkl} = \frac{\partial^2\varepsilon_{\phi\psi}^{hkl}}{\partial(\sin^2\psi)\cdot\partial\sigma^A} \tag{3}$$

S_1^{hkl} is embedded in the intersection term for each applied force increment and is necessary when performing triaxial measurements.

5. Summary of Test Method

5.1 A test specimen is prepared from a material that is representative of the object in which residual stress measurements are to be performed.

NOTE 1—If a sample of the same material is available it should be used.

5.2 The test specimen is instrumented with an electrical resistance strain gauge, mounted in a location that experiences the same stress as the region that will be subsequently irradiated with x-rays.

5.3 The test specimen is calibrated by loading it in such a manner that the stress, where the strain gauge is mounted, is directly calculable, and a calibration curve relating the strain gauge reading to the applied stress is developed.

5.4 The test specimen is mounted in a loading fixture in an x-ray diffraction instrument and sequentially loaded to several force levels.

5.4.1 The change in interplanar spacing is measured for each force level and related to the corresponding stress that is determined from the strain gauge reading and the calibration curve.

5.5 The XEC and its standard deviations are calculated from the test results.

6. Significance and Use

6.1 This test method provides standard procedures for experimentally determining the XEC for use in the measurement of residual and applied stresses using x-ray diffraction techniques. It also provides a standard means of reporting the precision of the XEC.

6.2 This test method is applicable to any crystalline material that exhibits a linear relationship between stress and strain in the elastic range, that is, only applicable to elastic loading.

6.3 This test method should be used whenever residual stresses are to be evaluated by x-ray diffraction techniques and the XEC of the material are unknown.

7. Apparatus

7.1 Any x-ray diffraction instrument intended for the measurement of residual macrostress that employs measurements of the diffraction peaks that are, ideally and for best accuracy, in the high back-reflection region may be used, including film camera types, diffractometers, and portable systems.

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[3] Evenschor P.D., Hauk V. Z., *Metallkunde*, 1975, 66 pp. 167–168.
[4] Dölle H., *J. Appl. Cryst*, 1979, 12, pp. 489-501.

7.2 A loading fixture is required to apply a force to the test specimen while it is being irradiated in the x-ray diffraction instrument.

7.2.1 The fixture shall be designed such that the surface stress applied by the fixture shall be uniform over the irradiated area of the specimen.

7.2.2 The fixture shall maintain the irradiated surface of the specimen at the exact center of rotation of the x-ray diffraction instrument throughout the test with sufficient precision to provide the desired levels of precision and bias in the measurements to be performed.

7.2.3 The fixture may be designed to apply tensile or bending forces. A four-point bending technique such as that described by Prevey[5] is most commonly used.

7.3 Electrical resistance strain gauges are mounted upon the test specimen to enable it to be accurately stressed to known levels.

8. Test Specimens

8.1 Test specimens should be fabricated from material with microstructure as nearly the same as possible as that in the material in which residual stresses are to be evaluated. It is preferred for superior results to use the same material with a fine grain structure and minimum cold work on the surface to minimize measurement errors.

8.2 For use in tensile or four-point bending fixtures, specimens should be rectangular in shape.

8.2.1 The length of tensile specimens, between grips, shall be not less than four times the width, and the width-to-thickness ratio shall not exceed eight.

8.2.2 For use in four-point bending fixtures, specimens should have a length-to-width ratio of at least four. The specimen width should be sufficient to accommodate strain gauges (see 8.5) and the width-to-thickness ratio should be greater than one and consistent with the method used to calculate the applied stresses in 9.1.

NOTE 2—Nominal dimensions often used for specimens for four-point bending fixtures are 10.2 × 1.9 × 0.15 cm (4.0 × 0.75 × 0.06 in.).

8.3 Tapered specimens for use in cantilever bending fixtures, and split-ring samples, are also acceptable.

8.4 Specimen surfaces may be electropolished or as-rolled sheet or plate.

8.5 One or more electrical resistance strain gauges are affixed to the test specimen in accordance with Guide E1237. The gauge(s) shall be aligned parallel to the longitudinal axis of the specimen, and should be mounted on a region of the specimen that experiences the same strain as the region that is to be irradiated. The gauge(s) should be applied to the irradiated surface of the beam either adjacent to, or on either side of, the irradiated area in order to minimize errors due to the absence of a pure tensile or bending force.

NOTE 3—In the case of four-point bending fixtures the gauge(s) should be placed well inside the inner span of the specimen in order to minimize the stress concentration effects associated with the inner knife edges of the fixture.

9. Calibration

9.1 Calibrate the instrumented specimen using forces applied by dead weights or by a testing machine that has been verified according to Practices E4. Use a loading configuration that produces statistically determinate applied stresses in the region where the strain gauges are mounted and where x-ray diffraction measurements will be performed (that is, such that stresses may be calculated from the applied forces and the dimensions of the specimen and the fixture). In the case of pure bending using a four-point bending apparatus, the strain gauge may be calibrated by measurement of applied strains via deflection of the specimen and calculated using the following equation:

$$\varepsilon_{\max} = \frac{\delta_{\max}\, 12h}{3L^2 - 4a^2} \tag{4}$$

where:

ε_{max} = maximum applied strain to the strain gauge
δ_{max} = maximum applied deflection
h = specimen thickness
L = distance between outer rollers on four-point bend fixture
a = distance between inner and outer rollers on each side of the four-point bend fixture

If the modulus of elasticity E for the material being tested is known, the applied stress on the specimen may then be calculated using Hooke's law.

$$\sigma^A = E\varepsilon_{\max} \tag{5}$$

If the modulus of elasticity E for the material being tested is not known, the applied stress on the specimen may be calculated using known applied forces in the case where bending is being used:

$$\sigma^A = \frac{3Fa}{bh^2} \tag{6}$$

where:

b = the width of the specimen, and
F = known total force applied by the rollers to the specimen

For uniaxial loading, if the modulus of elasticity E for the material being tested is not known, the applied stress on the specimen may be calculated using known applied forces:

$$\sigma^A = F/A_x \tag{7}$$

where:

A_x = cross sectional area of specimen

9.2 Pre-stress the specimen by loading to a level of approximately 75 % of the force that is calculated to produce a maximum applied stress equal to the nominal yield strength of the material, then unload. This will minimize drift in the gauges and creep in the strain gauge adhesive during the subsequent testing procedure.

9.3 Apply forces in increasing sequence at increments of approximately 10, 20, 30, 40, 50, 60, and 70 % of the force that would produce a maximum applied stress equal to the nominal

[5] Prevey, P. S., "A Method of Determining the Elastic Properties of Alloys in Selected Crystallographic Directions for X-Ray Diffraction Residual Stress Measurement," *Advances in X-Ray Analysis 20*, 1977, pp. 345–354.

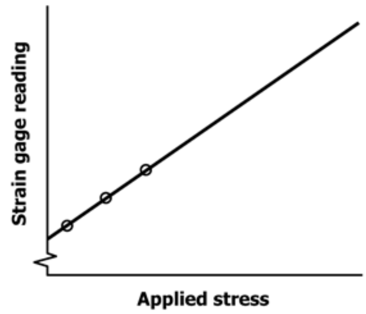


FIG. 1 Calibration Curve for Instrumented Test Specimen

yield strength of the material (a minimum of five (5) equally spaced points is recommended).

9.4 At each force level record strain gauge readings and calculate the applied stress.

9.5 Repeat measurement at 70, 60, 50, 40, 30, 20, and 10% of the yield strength, record strain gauge readings and calculate the applied stress at each level (a minimum of five (5) equally spaced points is recommended).

9.6 Repeat 9.3 – 9.5 at least once to ensure reproducibility of the measurement data.

9.7 Examine the data for repeatability and linearity. Deviations from either may indicate the failure of a strain gauge bond, stressing beyond the proportional limit of the material, or an imperfect loading configuration. If the deviations exceed the acceptable degree of uncertainty in the subsequent measurements of residual stress, the source of the deviations should be located and corrected before proceeding further.

9.8 If the repeatability and the linearity of the data are acceptable, plot a graph of strain gauge reading versus applied stress, draw a straight line through the data, and extrapolate it down to zero applied stress and up to 75 % of the nominal yield strength of the material. This is the calibration curve. (See Fig. 1.)

NOTE 4—For bending specimens, the strain indicated by the strain gauge(s) differs in magnitude from the strain in the specimen surface because of the elevation of the strain gauge grid above the surface. This does not affect the accuracy of the procedure.

10. Procedure

10.1 The x-ray diffraction technique, the crystallographic planes, the ψ angle(s), and the instrumentation that are used to determine the *XEC* of the material should be the same as those to be used subsequently to measure residual stresses in objects of the same material.

10.2 Mount the calibrated specimen in the loading fixture and mount the fixture in the x-ray diffraction instrument.

10.2.1 When using a bending fixture the arrangement should be such that the irradiated surface of the specimen may be stressed in either tension or compression.

10.3 Apply a force to the specimen with the loading fixture while monitoring the strain gauge readings. Using the calibration curve to convert strain gauge readings to applied stresses, apply stresses in increasing sequence at levels of approximately 10, 20, 30, 40, 50, 60, and 70 % of the nominal yield strength of the material (a minimum of five (5) equally spaced points is recommended). The pre-stress level, 75 %, should not be exceeded.

10.4 At each stress level, calculate the slope of the measured strain versus $\sin^2\psi$ values and the intercept of each curve for multiple ψ-angles, then calculate the slope of each set of values versus applied force. The slope of the first set is then $\frac{1}{2} S_2^{hkl}$ constant and the second slope is the S_1^{hkl} constant. Alternatively, if the double exposure technique is used, calculate the change in interplanar spacing, Δd, between the $\psi = 0$ and $\psi = \psi$ orientations for the particular crystallographic planes and ψ angles used. This approach may result in erroneous estimations of S_1^{hkl} depending on the accuracy of the intercepts obtained.

10.5 Apply stresses in decreasing sequence at levels of approximately 70, 60, 50, 40, 30, 20, and 10 %, measuring the change in the interplanar spacing at each level (a minimum of five (5) equally spaced points are recommended).

10.6 Repeat 10.3 – 10.5 at least once to ensure reproducibility of the measurement data.

10.7 Examine the data for repeatability and linearity, including the difference in the strain data between plus and minus ψ angles. Deviations from either may indicate the failure of a strain gauge bond, failure of the fixture to apply the correct force, or deviation from the proper x-ray geometry. The latter source of error, when present, is often caused by failure to maintain the irradiated area of the specimen surface at the exact center of the diffractometer while the specimen is under the applied force. If the deviations exceed the acceptable degree of uncertainty in the subsequent measurements of residual stress, the source of the deviations should be located and corrected before proceeding further.

11. Calculation

11.1 From Eq 2 and experimental data, $\frac{1}{2} S_2^{hkl}$ is obtained by fitting the data and estimating the uncertainty using a linear least squares regression (see Fig. 2) using:

$$\frac{\partial \varepsilon_{\phi\psi}^{hkl}}{\partial (\sin^2 \psi)} = m = \frac{1}{2} S_2^{hkl} \sigma^A \tag{8}$$

$$\frac{1}{2} S_2^{hkl} = \frac{\partial \left(\frac{1}{2} S_2^{hkl} \sigma^A \right)}{\partial \sigma^A} \tag{9}$$

11.2 Alternatively, $\frac{1}{2} S_2^{hkl}$ may be calculated using the steps described in 11.2.1 – 11.2.5.

11.2.1 For each measurement described in 10.3 – 10.6 of this test method, calculate the change in interplanar spacing and the corresponding applied stress.

11.2.2 Plot the data on a graph of change in interplanar spacing versus applied stress, and check for apparent errors. (See Fig. 3.)

11.2.3 Calculate the slope, m, of the best-fit straight line using least-squares linear regression. The following formulae[6] may be used:

[6] Press, W. H., et al., "Numerical Recipes: The Art of Scientific Computing." Cambridge, 1986, Art. 14.2.

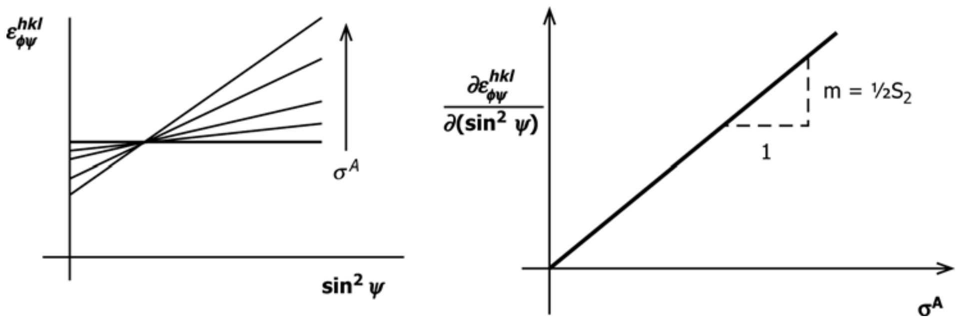
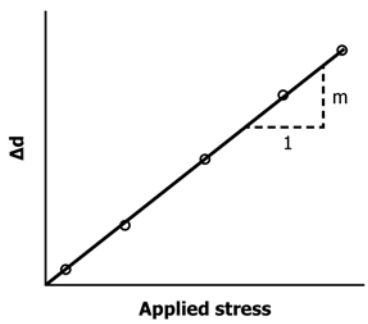


FIG. 2 Change in Strain with ψ and Applied Force



For clarity, only one point is shown for each applied stress level

FIG. 3 Change in Interplanar Spacing Versus Applied Stress

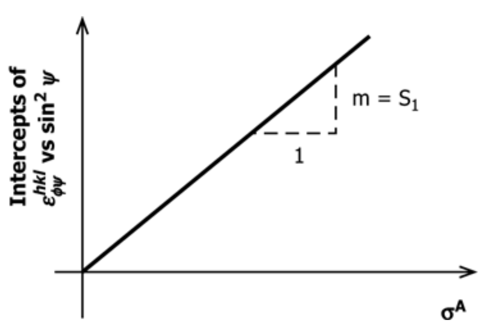


FIG. 4 Change in Intercepts with Applied Force

$$\mathrm{Sum}(X) = \sum_{i=1}^{n} X_i \tag{10}$$

$$T_i = X_i - \frac{\mathrm{Sum}(X)}{n} \tag{11}$$

$$\mathrm{Sum}(T^2) = \sum_{i=1}^{n} T_i^{\,2} \tag{12}$$

$$\mathrm{Sum}(TY) = \sum_{i=1}^{n} T_i \; Y_i \tag{13}$$

$$m = \frac{\mathrm{Sum}(TY)}{\mathrm{Sum}(T^2)} \tag{14}$$

In Eq 10-14, X_i represents the i-th value of the applied stress, and Y_i represents the measured value of Δd that corresponds to X_i.

11.2.4 For the procedure described in 10.3 – 10.6 of this test method, n = 28 in the above equations. This number corresponds to two (2) repetitions of a sequence of seven (7) measurements using ascending forces and seven (7) measurements using descending forces.

NOTE 5—There is no particular significance to using 28 data points. Any number equal to or greater than 14 (four (4) ascending, and three (3) descending repeated once) would be acceptable.

11.2.5 Calculate the ½ S_2^{hkl} using the following equation:

$$\frac{1}{2} S_2^{hkl} = \frac{1}{m} \, d_o \; \sin^2 \psi \tag{15}$$

where:

d_o = the interplanar spacing for unstressed material, is usually approximated by the interplanar spacing for ψ = 0.

11.3 For S_1^{hkl}, the calculation is performed as follows: Plot the y-intercepts $\varepsilon_{\phi\psi 0}$ of the $\sin^2\psi$ curves versus the applied forces (see Fig. 4):

$$S_1^{hkl} = \frac{\partial \varepsilon_{\phi\psi 0}}{\partial \sigma^A} \tag{16}$$

12. Precision and Bias

12.1 Precision in the context of this test method is the uncertainty of the calculated *XEC* ½ S_2^{hkl} and S_1^{hkl} due to uncertainties in the experimental measurements of the d-spacing and the corresponding applied stresses. If these measurement uncertainties are normally distributed with zero mean, there is a 68 % probability that the true *XEC* lie in the range of the calculated value plus or minus the standard deviation. This standard deviation is designated SD(½ S_2^{hkl}).

12.1.1 The precision of the calculated *XEC* is dependent on the precision of the measured quantities used to determine their values. These quantities are the slope m and interplanar spacing for unstressed material, d_o. This test method assumes that the slope m is the major source of uncertainty, and that the uncertainty in d_o is sufficiently small in comparison that it can be considered negligible.

12.1.2 Calculate the standard deviation of the slope, m, of the best-fit straight line plotted in 11.2 of this test method. The following formulae[6] may be used:

$$\mathrm{Sum}(Y) = \sum_{i=1}^{n} Y_i \tag{17}$$

$$c = \frac{\mathrm{Sum}(Y) - m\,\mathrm{Sum}(X)}{n} \tag{18}$$

$$\mathrm{Sum}((Y - mX - c)^2) = \sum_{i=1}^{n} (Y_i - \; mX_i - c)^2 \tag{19}$$

$$\mathrm{SD}(m) = \frac{\mathrm{Sum}((Y - mX - c)^2)}{\sqrt{(n-2)} \; \mathrm{Sum}(T^2)} \tag{20}$$

12.1.3 Calculate the standard deviation of the *XEC*. This value provides an estimate of the precision of the result calculated in 11.2 of this test method. The following formula may be used:

$$SD\left(\frac{1}{2}S_2^{hkl}\right) = \frac{SD(m)}{m^2} d_o \quad \sin^2 \psi \tag{21}$$

12.1.4 A round-robin test program was carried out to assess the precision of the test method. Three laboratories participated, using this test method and nominally identical test specimens. Three repeat runs were made on each of three specimens at one of the laboratories. The other two laboratories carried out one run on each of three specimens. The methods of loading to establish the applied stresses were somewhat different for each of the laboratories. The precisions reported by the three laboratories were comparable, on the order of 2 % to 3 %. The random error appears to be due primarily to the underlying precision of lattice spacing measurement.

12.2 Bias will exist if the irradiated surface is not maintained precisely at the center of the goniometer circle during testing. Also, bias error will occur if there is a residual stress gradient on the specimen, and the irradiated surface was caused to traverse along this gradient during loading.

12.2.1 The bias of this test method cannot be evaluated on the basis of the round-robin test program in 12.1.4 because there are no accepted reference values available for the x-ray elastic constants of the material. However, the results obtained for $\frac{1}{2} S_2^{hkl}$ from all three of the participating laboratories agreed within approximately two standard deviations for a normal distribution.

13. Keywords

13.1 residual stress; *XEC*; x-ray diffraction; x-ray elastic constant