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Standard Test Method for Determining the Effective Elastic Parameter for X-Ray Diffraction Measurements of Residual Stress[1]

This standard is issued under the fixed designation E 1426; 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 spacings between parallel planes of atoms, ions, or molecules in the lattice change. X-ray diffraction techniques can measure these changes and, therefore, they constitute a powerful means for studying the residual stress state in a body. To calculate macroscopic stresses from lattice strains requires a material constant, E_{eff}, called the effective elastic parameter, that 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 effective elastic parameter, E_{eff}, for the evaluation of residual and applied stresses by X-ray diffraction techniques. The effective elastic parameter relates macroscopic stress to the strain measured in a particular crystallographic direction in polycrystalline samples. E_{eff} should not be confused with E, the modulus of elasticity. Rather, it is nominally equivalent to $E/(1 + \nu)$ for the particular crystallographic direction, where ν is Poisson's ratio. The effective elastic parameter is influenced by elastic anisotropy and preferred orientation of the sample material.

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 inch pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

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:*

E 4 Practices for Force Verification of Testing Machines[2]

E 6 Terminology Relating to Methods of Mechanical Testing[2]

E 7 Terminology Relating to Metallography[2]

E 1237 Guide for Installing Bonded Resistance Strain Gages[2]

3. Terminology

3.1 *Definitions:*

3.1.1 Many of the terms used in this test method are defined in Terminology E 6 and E 7.

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 a = dummy parameter for Sum(a) and SD(a).

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.

3.3.6 E = modulus of elasticity.

3.3.7 E_{eff} = effective elastic parameter for X-ray measurements.

3.3.8 i = measurement index, $1 \leq i \leq$ n.

3.3.9 m = slope of a graph of Δd versus stress.

3.3.10 n = number of measurements used to determine slope m.

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[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] *Annual Book of ASTM Standards*, Vol 03.01.

3.3.11 *SD(a)* = standard deviation of a set of quantities “a”.

3.3.12 *Sum(a)* = sum of a set of quantities “a”.

3.3.13 T_i = X_i minus mean of all X_i values.

3.3.14 X_i = *i*-th value of applied stress.

3.3.15 Y_i = measurement of Δd corresponding to X_i.

3.3.16 ν = Poisson’s ratio.

3.3.17 ψ = angle between the specimen surface normal and the normal to the diffracting crystallographic planes.

4. Summary of Test Method

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

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

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

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

4.4 The test specimen is mounted in a loading fixture in an X-ray diffraction apparatus, and sequentially loaded to several load levels.

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

4.5 The effective elastic parameter and its standard deviation are calculated from the test results.

5. Significance and Use

5.1 This test method provides standard procedures for experimentally determining the effective elastic parameter for X-ray diffraction measurement of residual and applied stresses. It also provides a standard means of reporting the precision of the parameter.

5.2 This test method is applicable to any crystalline material which exhibits a linear relationship between stress and strain in the elastic range.

5.3 This test method should be used whenever residual stresses are to be evaluated by an X-ray diffraction technique and the effective elastic parameter of the material is unknown.

6. Apparatus

6.1 Any X-ray diffraction instrument intended for measurements of residual macrostress that employs measurements of the diffraction peaks in the high back-reflection region may be used, including film camera types, diffractometers, and portable systems.

6.2 A loading fixture is required to apply loads to the test specimen while it is being irradiated in the X-ray diffraction instrument.

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

6.2.2 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 made.

6.2.3 The fixture may be designed to apply tensile or bending loads. A four-point bending technique such as that described by Prevey[3] is most commonly used.

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

7. Test Specimens

7.1 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.

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

7.2.1 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.

7.2.2 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 gages (see 7.5) and the width-to-thickness ratio should be greater than one and consistent with the method used to calculate the applied stresses in 8.1.

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

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

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

7.5 One or more electrical resistance strain gages is affixed to the test specimen in accordance with Guide E 1237. The gage(s) should 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 gage(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 load.

NOTE 3—In the case of four-point bending fixtures the gage(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.

8. Calibration

8.1 Calibrate the instrumented specimen using loads applied by dead weights or by a testing machine that has been verified according to Practices E 4. The loading configuration is such that the applied stresses, in the region where the strain gages are mounted and where X-ray diffraction measurements will be

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[3] 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.

made, are statically determinate (that is, may be calculated from the applied loads and the dimensions of the specimen and the fixture).

8.2 Prestress the specimen by loading to a level of approximately 75 % of the load 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 gages and creep in the strain gage adhesive during the subsequent testing procedure.

8.3 Apply loads in increasing sequence at levels of approximately 5, 15, and 25 % of the load that would produce a maximum applied stress equal to the nominal yield strength of the material.

8.4 At each load level calculate the applied stress and take strain gage readings.

8.5 Apply loads in decreasing sequence at levels of approximately 15 and 5 %, calculating the applied stress and taking strain gage readings at each level.

8.6 Repeat 8.3-8.5.

8.7 Examine the data for repeatability and linearity. Deviations from either may indicate the failure of a strain gage 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.

8.8 If the repeatability and the linearity of the data are acceptable, plot a graph of applied stress versus strain gage reading, 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 gage(s) differs in magnitude from the strain in the specimen surface because of the elevation of the strain gage grid above the surface. This does not affect the accuracy of the procedure.

9. Procedure

9.1 The X-ray diffraction technique, the crystallographic planes, the ψ angle, and the instrumentation that are used to determine the effective elastic parameter of the material should be the same as those to be used subsequently to measure residual stresses in objects of the same material.

9.2 Mount the calibrated specimen in the loading fixture and mount the fixture in the X-ray diffraction instrument.

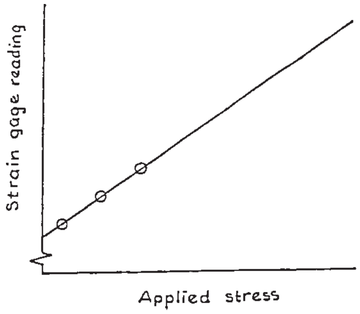


FIG. 1 Calibration Curve for Instrumented Test Specimen

9.2.1 When using a bending fixture the arrangement should be such that the irradiated surface of the specimen is stressed in tension.

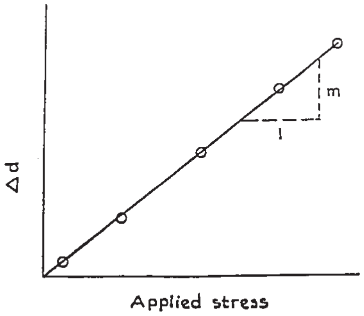
9.3 Load the specimen with the loading fixture while monitoring the strain gage readings. Using the calibration curve to convert strain gage readings to applied stresses, apply stresses in increasing sequence at levels of approximately 5, 20, 40, 60, and 70 % of the nominal yield strength of the material. The prestress level, 75 %, must not be exceeded.

9.4 At each stress level calculate the change in interplanar spacing, Δ*d*, between the ψ = 0 and ψ = ψ orientations for the particular crystallographic planes and ψ angles used.

9.5 Apply stresses in decreasing sequence at levels of approximately 60, 40, 20, and 5 %, measuring the change in the interplanar spacing at each level.

9.6 Repeat 9.3-9.5.

9.7 Examine the data for repeatability and linearity. Deviations from either may indicate the failure of a strain gage bond, failure of the fixture to apply the correct loading, 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 load. 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.



NOTE 1—For clarity, only one point is shown for each applied stress level

FIG. 2 Change in Interplanar Spacing Versus Applied Stress

10. Calculation

10.1 For each measurement described in Sections 9.3-9.6 of this test method, calculate the change in interplanar spacing and the corresponding applied stress.

10.2 Plot the data on a graph of change in interplanar spacing vs. applied stress, and check for apparent errors. (See Fig. 2.)

10.3 Calculate the slope, m, of the best-fit straight line using least-squares linear regression. The following formulas[4] may be used:

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

$$\mathrm{Sum(X)} = \sum_{i=1}^{n} X_i \qquad T_i = X_i - \frac{\mathrm{Sum(X)}}{n} \tag{1}$$

$$\mathrm{Sum(T^2)} = \sum_{i=1}^{n} T_i^2 \qquad \mathrm{Sum(TY)} = \sum T_i \; Y_i$$

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

In these equations, 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.

10.4 For the procedure described in 9.3-9.6 of this test method, n = 18 in the above equations. This number corresponds to 2 repetitions of a sequence of 5 measurements at ascending loads and 4 measurements at descending loads.

NOTE 5—There is no particular significance to using 18 data points. Any number equal to or greater than 14 (4 ascending, 3 descending) would be acceptable.

10.5 Calculate the X-ray effective elastic constant using the following equation:

$$E_{\mathrm{eff}} = \frac{1}{m} d_o \sin^2 \psi \tag{2}$$

where:

d_o = the interplanar spacing for unstressed material, is usually approximated by the interplanar spacing for $\psi = 0$.

11. Precision and Bias

11.1 Precision in the context of this test method is the uncertainty of the calculated X-ray elastic parameter, E_{eff}, 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 X-ray elastic parameter lies in the range of the calculated value of E_{eff}, plus or minus the standard deviation of E_{eff}. This standard deviation is designated $\mathrm{SD}(E_{\mathrm{eff}})$.

11.1.1 The precision of the calculated X-ray elastic parameter is dependent on the precision of the measured quantities used to determine its value. 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.

11.1.2 Calculate the standard deviation of the slope, m, of the best-fit straight line plotted in paragraph 10.2 of this test method. The following formulas[4] may be used:

$$\mathrm{Sum(Y)} = \sum_{i=1}^{n} Y_i \qquad c = \frac{\mathrm{Sum(Y)} - m\,\mathrm{Sum(X)}}{n} \tag{3}$$

$$\mathrm{Sum((Y-mX-c)^2)} = \sum_{i=1}^{n} (Y_i - m\,X_i - c)^2$$

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

11.1.3 Calculate the standard deviation of the X-ray effective elastic constant. This value provides an estimate of the precision of the result calculated in Section 10.5 of this test method. The following formula may be used:

$$\mathrm{SD}(E_{\mathrm{eff}}) = \frac{\mathrm{SD}(m)}{m^2} d_o \;\; \sin^2 \psi \tag{4}$$

11.1.4 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.

11.2 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.

11.2.1 The bias of this test method cannot be evaluated on the basis of the round-robin test program in 11.1.4 because there is no accepted reference value available for the effective elastic parameter of the material. However, the results from all three of the participating laboratories agreed within approximately two sigma.

12. Keywords

12.1 elastic parameter; residual stress; x-ray diffraction