Designation: E2246 – 05

Standard Test Method for Strain Gradient Measurements of Thin, Reflecting Films Using an Optical Interferometer[1]

This standard is issued under the fixed designation E2246; 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 a procedure for measuring the strain gradient in thin, reflecting films. It applies only to films, such as found in microelectromechanical systems (MEMS) materials, which can be imaged using an optical interferometer. Measurements from cantilevers that are touching the underlying layer are not accepted.

1.2 This test method uses a non-contact optical interferometer with the capability of obtaining topographical 3-D data sets. It is performed in the laboratory.

1.3 *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]

E2244 Test Method for In-Plane Length Measurements of Thin, Reflecting Films Using an Optical Interferometer

E2245 Test Method for Residual Strain Measurements of Thin, Reflecting Films Using an Optical Interferometer

3. Terminology

3.1 *Definitions:*

3.1.1 *2-D data trace*, *n*—a two-dimensional group of points that is extracted from a topographical 3-D data set and that is parallel to the *xz*- or *yz*-plane of the interferometer.

3.1.2 *3-D data set*, *n*—a three-dimensional group of points with a topographical *z*-value for each (*x*, *y*) pixel location within the interferometer's field of view.

3.1.3 *anchor*, *n*—in a surface-micromachining process, the portion of the test structure where a structural layer is intentionally attached to its underlying layer.

3.1.4 *anchor lip*, *n*—in a surface-micromachining process, the freestanding extension of the structural layer of interest around the edges of the anchor to its underlying layer.

3.1.4.1 *Discussion*—In some processes, the width of the anchor lip may be zero.

3.1.5 *bulk micromachining*, *adj*—a MEMS fabrication process where the substrate is removed at specified locations.

3.1.6 *cantilever*, *n*—a test structure that consists of a freestanding beam that is fixed at one end.

3.1.7 *fixed-fixed beam*, *n*—a test structure that consists of a freestanding beam that is fixed at both ends.

3.1.8 *in-plane length (or deflection) measurement*, *n*—the experimental determination of the straight-line distance between two transitional edges in a MEMS device.

3.1.8.1 *Discussion*—This length (or deflection) measurement is made parallel to the underlying layer (or the *xy*-plane of the interferometer).

3.1.9 *interferometer*, *n*—a non-contact optical instrument used to obtain topographical 3-D data sets.

3.1.9.1 *Discussion*—The height of the sample is measured along the *z*-axis of the interferometer. The interferometer's *x*-axis is typically aligned parallel or perpendicular to the transitional edges to be measured.

3.1.10 *MEMS*, *adj*—microelectromechanical system.

3.1.11 *microelectromechanical systems*, *adj*—in general, this term is used to describe micron-scale structures, sensors, actuators or the technologies used for their manufacture (such as, silicon process technologies), or combinations thereof.

3.1.12 *out-of-plane measurements*, *n*—experimental data taken on structures that are curved in the interferometer's *z*-direction (that is, perpendicular to the underlying layer).

3.1.13 *residual strain*, *n*—in a MEMS process, the amount of deformation (or displacement) per unit length constrained within the structural layer of interest after fabrication yet before the constraint of the sacrificial layer (or substrate) is removed (in whole or in part).

3.1.14 *sacrificial layer*, *n*—a single thickness of material that is intentionally deposited (or added) then removed (in whole or in part) during the micromachining process, to allow freestanding microstructures.

3.1.15 *stiction*, *n*—adhesion between the portion of a structural layer that is intended to be freestanding and its underlying layer.

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[1] This test method is under the jurisdiction of ASTM Committee E08 on Fatigue and Fracture and is the direct responsibility of Subcommittee E08.05 on Cyclic Deformation and Fatigue Crack Formation.

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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.

3.1.16 *(residual) strain gradient*, *n*—a through-thickness variation (of the residual strain) in the structural layer of interest before it is released.

3.1.16.1 *Discussion*—If the variation through the thickness in the structural layer is assumed to be linear, it is calculated to be the positive difference in the residual strain between the top and bottom of a cantilever divided by its thickness. Directional information is assigned to the value of "s."

3.1.17 *structural layer*, *n*—a single thickness of material present in the final MEMS device.

3.1.18 *substrate*, *n*—the thick, starting material (often single crystal silicon or glass) in a fabrication process that can be used to build MEMS devices.

3.1.19 *support region*, *n*—in a bulk-micromachining process, the area that marks the end of the suspended structure.

3.1.20 *surface micromachining*, *adj*—a MEMS fabrication process where micron-scale components are formed on a substrate by the deposition (or addition) and removal (in whole or in part) of structural and sacrificial layers.

3.1.21 *test structure*, *n*—a component (such as, a cantilever or a fixed-fixed beam) that is used to extract information (such as, the strain gradient or the residual strain of a layer) about a fabrication process.

3.1.22 *transitional edge*, *n*—the side of a MEMS structure that is characterized by a distinctive out-of-plane vertical displacement as seen in an interferometric 2-D data trace.

3.1.23 *underlying layer*, *n*—the single thickness of material directly beneath the material of interest.

3.1.23.1 *Discussion*—This layer could be the substrate.

3.2 *Symbols:*

3.2.1 *For Calibration*:

σ_{xcal} = the standard deviation in a ruler measurement in the interferometer's x-direction for the given combination of lenses

σ_{ycal} = the standard deviation in a ruler measurement in the interferometer's y-direction for the given combination of lenses

σ_{zcal} = the standard deviation of the step height measurements on the double-sided step height standard

cal_x = the x-calibration factor of the interferometer for the given combination of lenses

cal_y = the y-calibration factor of the interferometer for the given combination of lenses

cal_z = the z-calibration factor of the interferometer for the given combination of lenses

$cert$ = the certified value of the double-sided step height standard

$inter_x$ = the interferometer's maximum field of view in the x-direction for the given combination of lenses

$inter_y$ = the interferometer's maximum field of view in the y-direction for the given combination of lenses

$mean$ = the mean value of the step-height measurements (on the double-sided step height standard) used to calculate cal_z

$ruler_x$ = the interferometer's maximum field of view in the x-direction for the given combination of lenses as measured with a 10-μm grid (or finer grid) ruler

$ruler_y$ = the interferometer's maximum field of view in the y-direction for the given combination of lenses as measured with a 10-μm grid (or finer grid) ruler

3.2.2 *For Alignment*:

L = the in-plane length measurement of the cantilever

$x1_{lower}$ = the x-data value along Edge "1" locating the lower part of the transitional edge

$x1_{upper}$ = the x-data value along Edge "1" locating the upper part of the transitional edge

$x3_{lower}$ = the x-data value along Edge "3" locating the lower part of the transitional edge

$x3_{upper}$ = the x-data value along Edge "3" locating the upper part of the transitional edge

$x4_{lower}$ = the x-data value along Edge "4" locating the lower part of the transitional edge

$x4_{upper}$ = the x-data value along Edge "4" locating the upper part of the transitional edge

x_{lower} = the x-data value along the transitional edge of interest locating the lower part of the transition

x_{upper} = the x-data value along the transitional edge of interest locating the upper part of the transition

3.2.3 *For Strain Gradient Calculations*:

a = the x- (or y-) coordinate of the origin of the circle of radius R_{int}. An arc of this circle models the out-of-plane shape in the z-direction of the topmost surface of the cantilever

b = the z-coordinate of the origin of the circle of radius R_{int}. An arc of this circle models the out-of-plane shape in the z-direction of the topmost surface of the cantilever

R_{int} = the radius of the circle with an arc that models the shape of the topmost surface of the cantilever as measured with the interferometer

s = equals 1 for cantilevers deflected in the minus z-direction of the interferometer, and equals −1 for cantilevers deflected in the plus z-direction

s_g = the strain gradient as calculated from three data points

s_{g0} = the strain gradient when the residual strain equals zero

t = the thickness of the suspended, structural layer

$t_{support}$ = in a bulk-micromachining process, the thickness of the support region where it is intersected by the interferometric 2-D data trace of interest

$x1_{ave}$ = the average of $x1_{lower}$ and $x1_{upper}$

$x2_{ave}$ = the average of $x2_{lower}$ and $x2_{upper}$

$x2_{lower}$ = the x-data value along Edge "2" locating the lower part of the transitional edge

$x2_{upper}$ = the x-data value along Edge "2" locating the upper part of the transitional edge

z_{upper} = the z-data value associated with x_{upper}

$z_{upper\text{-}t}$ = in a bulk-micromachining process, the value for z when the thickness of the support region, $t_{support}$, is subtracted from z_{upper}

3.2.4 *For Combined Standard Uncertainty Calculations*:

σ_{sample} = the standard deviation in a height measurement due to the sample's peak-to-valley surface roughness as measured with the interferometer

R_{tave} = the peak-to-valley roughness of a flat and leveled surface of the sample material calculated to be the average of three or more measurements, each measurement of which is taken from a different 2-D data trace

$s_{g\text{-}high}$ = in determining the combined standard uncertainty value for the strain gradient measurement, the highest value for s_g given the specified variations

$s_{g\text{-}low}$ = in determining the combined standard uncertainty value for the strain gradient measurement, the lowest value for s_g given the specified variations

u_c = the combined standard uncertainty value (that is, the estimated standard deviation of the result)

u_{samp} = the component in the combined standard uncertainty calculation for strain gradient that is due to the sample's peak-to-valley surface roughness as measured with the interferometer

u_W = the component in the combined standard uncertainty calculation for strain gradient that is due to the measurement uncertainty across the width of the cantilever

u_{xcal} = the component in the combined standard uncertainty calculation for strain gradient that is due to the uncertainty of the calibration in the *x*-direction

u_{xres} = the component in the combined standard uncertainty calculation for strain gradient that is due to the resolution of the interferometer in the *x*-direction

u_{zcal} = the component in the combined standard uncertainty calculation for strain gradient that is due to the uncertainty of the calibration in the *z*-direction

u_{zres} = the component in the combined standard uncertainty calculation for strain gradient that is due to the resolution of the interferometer in the *z*-direction

$w_{1/2}$ = the half width of the interval from $s_{g\text{-}low}$ to $s_{g\text{-}high}$

x_{res} = the resolution of the interferometer in the *x*-direction

z_{res} = the resolution of the interferometer in the *z*-direction

3.2.5 *For Round Robin Measurements*:

L_{des} = the design length of the cantilever

n = the number of reproducibility or repeatability measurements

s_{gave} = the average strain gradient value for the reproducibility or repeatability measurements. It is equal to the sum of the s_g values divided by n.

u_{cave} = the average combined standard uncertainty value for the reproducibility or repeatability measurements. It is equal to the sum of the u_c values divided by n.

3.2.6 *For Adherence to the Top of the Underlying Layer*:

A = in a surface micromachining process, the minimum thickness of the structural layer of interest as measured from the top of the structural layer in the anchor area to the top of the underlying layer

H = in a surface micromachining process, the anchor etch depth, which is the amount the underlying layer is etched away in the interferometer's minus *z*-direction during the patterning of the sacrificial layer

J = in a surface micromachining process, the positive distance (equal to the sum of j_a, j_b, j_c, and j_d) between the bottom of the suspended, structural layer and the top of the underlying layer

j_a = in a surface micromachining process, half the peak-to-peak value of the roughness of the underside of the suspended, structural layer in the interferometer's *z*-direction. This is due to the roughness of the topside of the sacrificial layer.

j_b = in a surface micromachining process, the tilting component of the suspended, structural layer that accounts for the deviation in the distance between the bottom of the suspended, structural layer and the top of the underlying layer that is not due to residue or the roughness of the surfaces. This component can be positive or negative.

j_c = in a surface micromachining process, the height in the interferometer's z-direction of any residue present between the bottom of the suspended, structural layer and the top of the underlying layer

j_d = in a surface micromachining process, half the peak-to-peak value of the surface roughness of the topside of the underlying layer

$z_{reg\#1}$ = in a surface micromachining process, the interferometric *z* value of the point of maximum deflection along the cantilever with respect to the anchor lip

$z_{reg\#2}$ = in a surface micromachining process, a representative interferometric *z* value of the group of points within the large anchor area

3.2.7 *Discussion*—The symbols above are used throughout this test method. However, when referring to *y* values, the letter "*y*" can replace the first letter in the symbols above that start with the letter "*x*."

4. Summary of Test Method

4.1 A cantilever is shown in Figs. 1-3. After fabrication, this cantilever bends in the out-of-plane *z*-direction. An optical interferometer (such as shown in Fig. 4) is used to obtain a topographical 3-D data set. Two-D data traces beside the cantilever (such as shown in Fig. 5) and along the top of the cantilever (such as shown in Fig. 6) are extracted from this 3-D data set for the strain gradient analysis.

4.2 A circular arc models the out-of-plane shape of cantilevers. Three data points (such as shown in Fig. 6) define the circular function. The strain gradient is calculated from the radius of this circle.

4.3 To obtain three data points representative of the shape of a surface-micromachined cantilever: (*1*) select two transitional edges, (*2*) obtain a 3-D data set, (*3*) ensure alignment, and (*4*) select three data points. This procedure is presented in Appendix X1 for a bulk-micromachined cantilever or a surface-micromachined cantilever with transitional edges greater than 8 µm in height.

4.4 To determine the strain gradient: (*1*) solve three equations for three unknowns, (*2*) plot the function with the data, and (*3*) calculate the strain gradient.

5. Significance and Use

5.1 Strain gradient values are an aid in the design and fabrication of MEMS devices.

6. Interferences

6.1 Measurements from cantilevers that are touching the underlying layer (as ascertained in Appendix X2) are not accepted.

7. Apparatus [3] (1)[4]

7.1 *Non-contact Optical Interferometer*, capable of obtaining a topographical 3-D data set and exporting a 2-D data trace.

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[3] The same apparatus is used as in Test Method E2244 and Test Method E2245.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

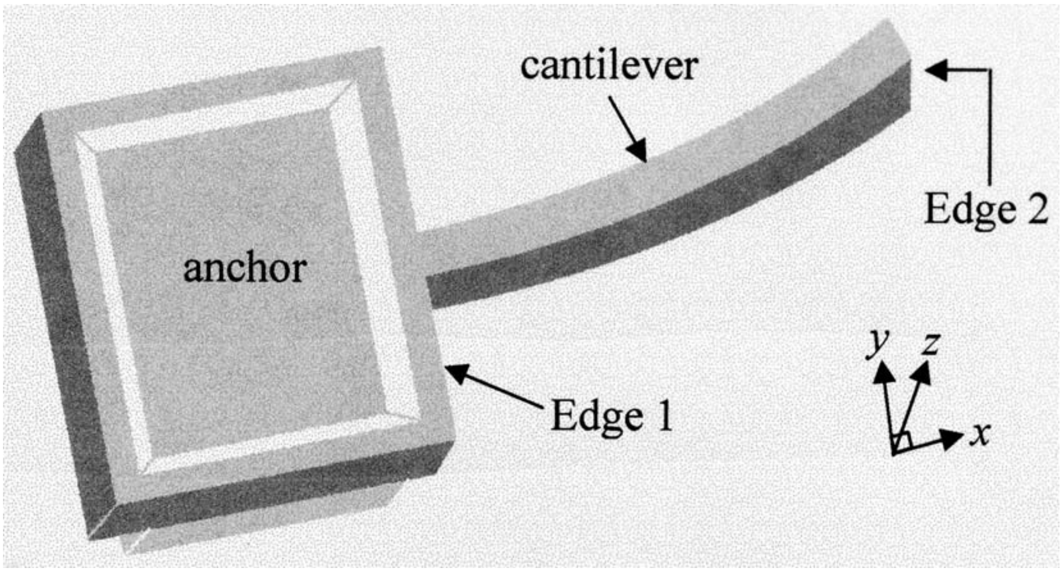
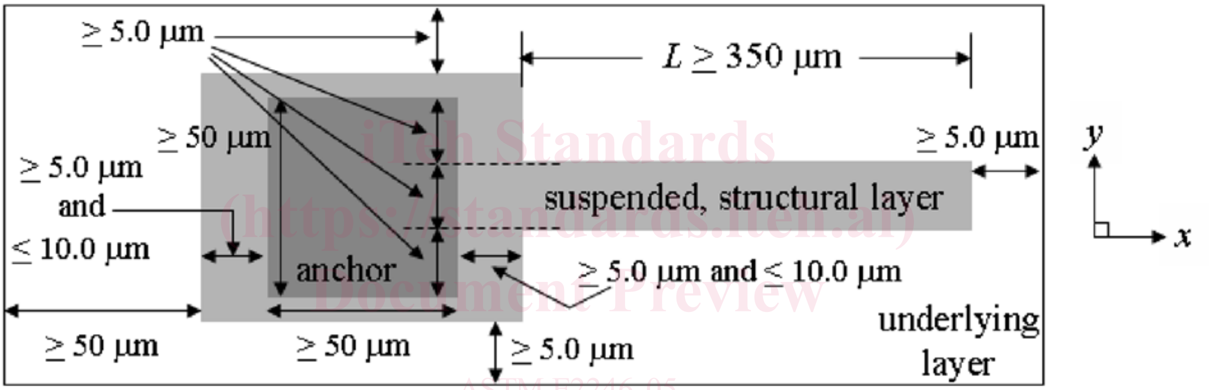


FIG. 1 Three-Dimensional View of Surface-micromachined Cantilever



NOTE 1—The underlying layer is beneath the entire test structure.

NOTE 2—The structural layer of interest is included in both the light and dark gray areas.

NOTE 3—The light gray area is suspended in air after fabrication.

NOTE 4—The dark gray area (the anchor) is the designed cut in the sacrificial layer. This is where the structural layer contacts the underlying layer.

FIG. 2 Design Dimensions for Cantilever in Fig. 1

Fig. 4 is a schematic of such an interferometer. However, any non-contact optical interferometer that has pixel-to-pixel spacings as specified in Table 1 and that is capable of performing the test procedure with a vertical resolution less than 1 nm is permitted. The interferometer must be capable of measuring step heights to at least 5 µm higher than the step height to be measured.

NOTE 1—Table 1 does not include magnifications at or less than 2.5× because the pixel-to-pixel spacings will be too large for this work or the possible introduction of a second set of interferometric fringes in the data set at these magnifications can adversely affect the data, or both. Therefore, magnifications at or less than 2.5× shall not be used.

7.2 *A 10-µm-grid (or finer grid) Ruler*, for calibrating the interferometer in the *xy*-plane. This ruler should be longer than the maximum field of view at the lowest magnification.

7.3 *Double-sided Step Height Standard*, for calibrating the interferometer in the out-of-plane *z*-direction.

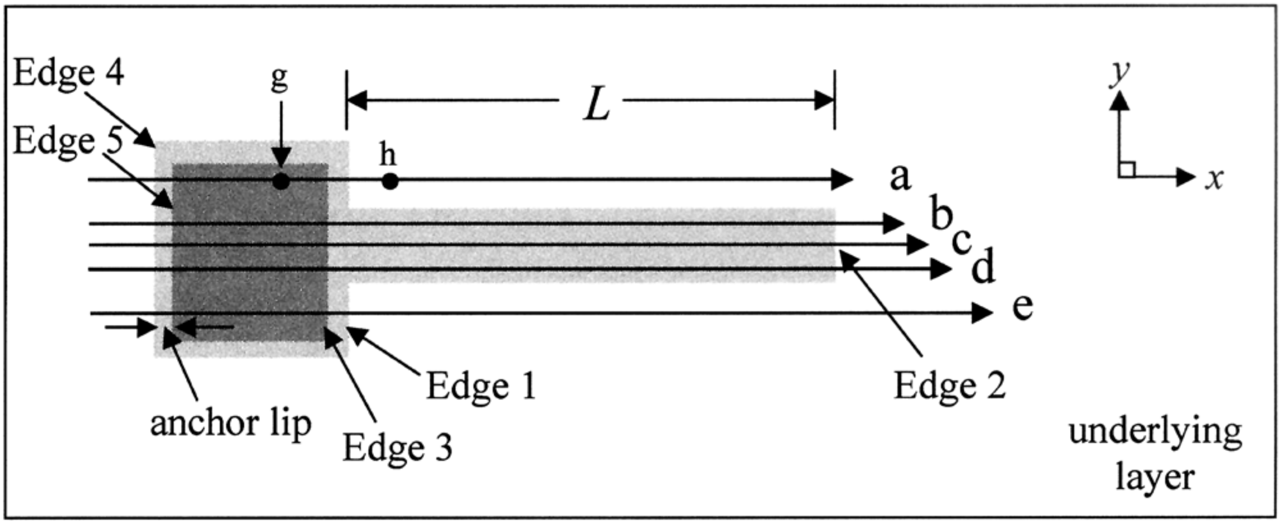
8. Test Units

8.1 *Cantilever Test Structures Fabricated in Either a Surface-micromachining or Bulk-micromachining Process*—The design of a representative surface-micromachined cantilever is specified below.

8.1.1 The cantilever shall be wide enough (for example, 5-µm wide, as shown in Fig. 2) such that obtaining a 2-D data trace (such as Trace "c" in Fig. 3) along its length is not a difficult task.

8.1.2 The cantilever shall be long enough (for example, $L \geq 350$ µm, as shown in Fig. 2) such that it exhibits out-of-plane curvature in the *z*-direction (as shown in Fig. 1 and Fig. 6).

8.1.3 The anchor lip between Edges "1" and "3" in Fig. 3 and between Edges "4" and "5" shall be wide enough to include at least three data points. If the pixel-to-pixel spacing is 1.56 µm, then this anchor lip should be at least 3.2 times



NOTE 1—The 2-D data traces (“a” and “e”) are used to ensure alignment.
NOTE 2—Trace “c” is used to determine the strain gradient and ascertain if the cantilever is adhered to the top of the underlying layer.
NOTE 3—Traces “b,” “c,” and “d” can be used in the calculation of u_W.

FIG. 3 Top View of Surface-micromachined Cantilever

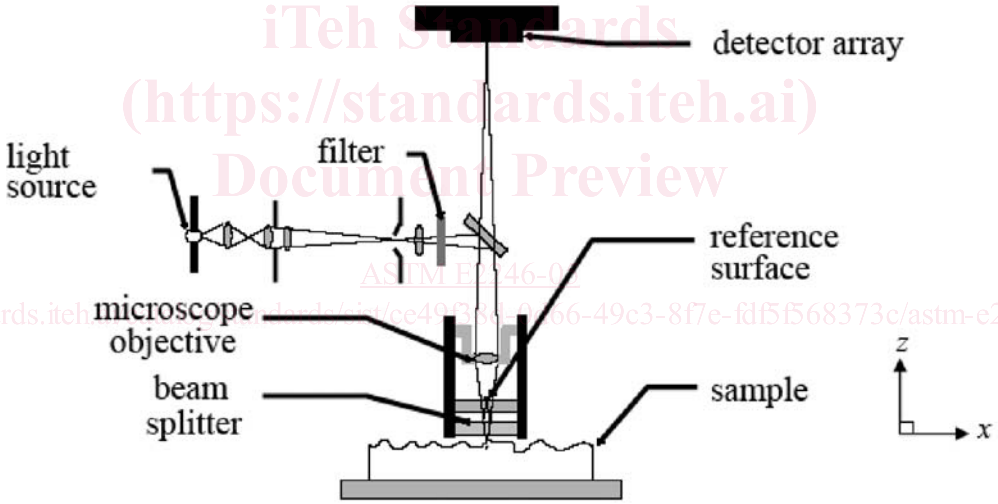


FIG. 4 Schematic of an Optical Interferometer

greater (or 5.0 µm, as shown in Fig. 2). At the same time, it should be less than or equal to 10.0-µm wide.

8.1.4 The cut in the sacrificial layer that defines the anchor should be at least 50 by 50 µm (as shown in Fig. 2) to determine if the cantilever has adhered to the top of the underlying layer as ascertained in Appendix X2.

NOTE 2—If one or more “posts” are used in the anchor area, a post layer is not considered the underlying layer. The post or posts connect the underlying layer to the sample material, in which case replace the words “cut in the sacrificial layer” with the words “post or posts.”

8.1.5 The anchor shall extend beyond the width of the cantilever in the ±*y*-directions (for example, at least 5.0 µm, as shown in Fig. 2) such that obtaining Traces “a” and “e” in Fig. 3 is not a difficult task.

8.1.6 There should be only one cantilever for each anchor (as shown in Fig. 2).

8.1.7 The underlying layer shall be un-patterned beneath the structural layer of interest and should extend at least 5.0 µm beyond the outermost edges of this patterned, structural layer (as shown in Fig. 2). However, the underlying layer should extend at least 50 µm beyond the anchor lip in the minus *x*-direction (as shown in this figure) to ascertain if the cantilever has adhered to the top of the underlying layer, if necessary.

NOTE 3—Any tilt in the sample (or the sample data) is eliminated by leveling the interferometric optics (or the 3-D data set) with respect to the top of the exposed underlying layer. The exposed underlying layer straddling the cantilever in Fig. 2 is used for this purpose. Therefore, no other structures should be designed in these areas.

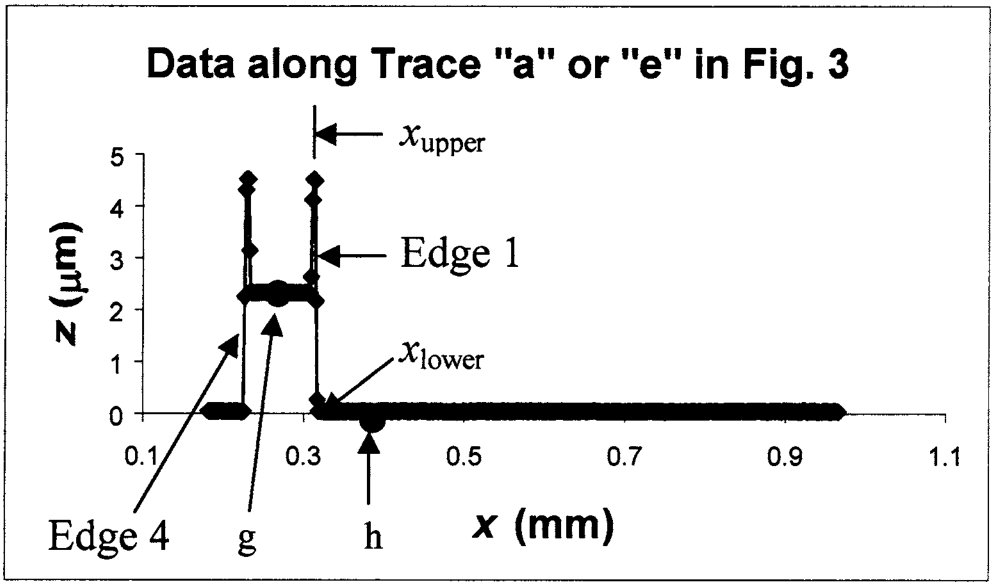


FIG. 5 2-D Data Trace Used to Find $x1_{lower}$, $x1_{upper}$, $x4_{lower}$, and $x4_{upper}$

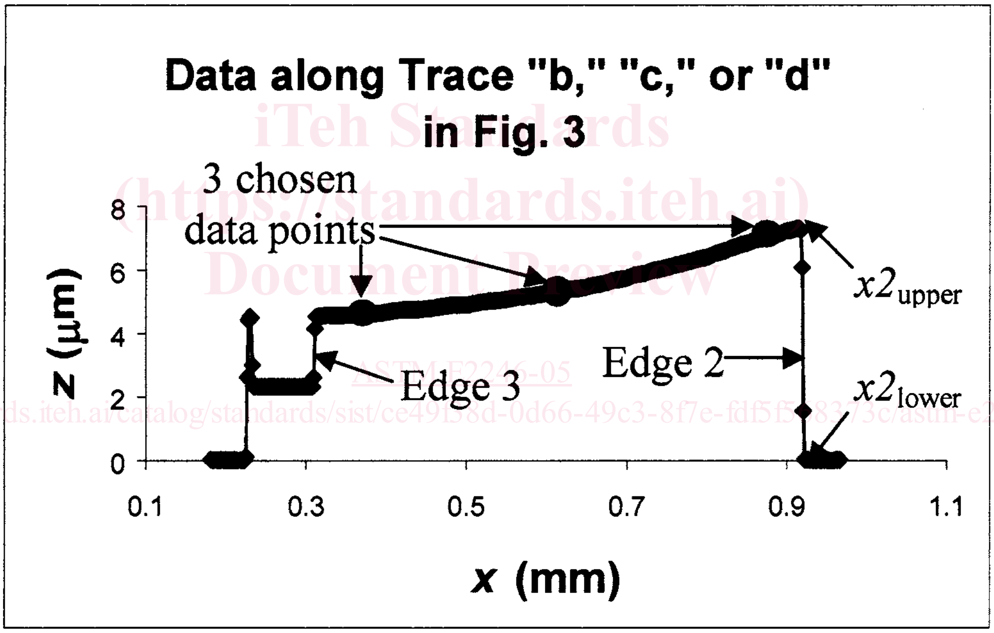


FIG. 6 2-D Data Trace Used to Find $x2_{lower}$, $x2_{upper}$, and the Three Data Points

TABLE 1 Interferometer Pixel-to-Pixel Spacing Requirements

Magnification, ×	Pixel-to-pixel spacing, µm
5	< 1.57
10	< 0.83
20	< 0.39
40	< 0.21
80	< 0.11

8.1.8 A sufficient number of cantilevers (preferably of different lengths) should be fabricated in order to obtain at least one cantilever after fabrication, which exhibits out-of-plane curvature in the *z*-direction and which has not adhered to the top of the underlying layer.

9. Calibration [5] (1)

9.1 Calibrate the interferometer in the *x*- and *y*-directions using a 10-µm-grid (or finer grid) ruler. Do this for each combination of lenses used for the measurements. Calibrate in the *xy*-plane on a yearly basis.

9.1.1 *For Non-reflective Rulers*:

9.1.1.1 Orient the ruler in the *x*-direction using crosshairs, if available. Record $ruler_x$ as measured on the interferometer's screen. Determine σ_{xcal}.

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[5] The same calibration procedure is used as in Test Method E2244 and Test Method E2245.