Designation: E 2246 – 02

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

This standard is issued under the fixed designation E 2246; 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 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:*

E 2244 Test Method for In-Plane Length Measurements of Thin, Reflecting Films Using an Optical Interferometer[2]

E 2245 Test Method for Residual Strain Measurements of Thin, Reflecting Films Using an Optical Interferometer[2]

3. Terminology

3.1 *Definitions:*

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

3.1.1.1 *Discussion*—The height of the sample is measured along the *z*-axis of the interferometer. The interferometer's *x*-axis (as shown in Figs. 1-3) is typically aligned parallel or perpendicular to the transitional edges to be measured.

3.1.2 *3-D data set*, *n*—a three-dimensional data set with a topographical *z*-data 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 the mechanical layer makes contact with the underlying layer (see Figs. 1 and 2).

3.1.4 *anchor lip*, *n*—in a surface-micromachining process, the extension of the mechanical layer around the edges of the anchor (see Figs. 2 and 3).

3.1.5 *bulk micromachining*, *adj*—a MEMS fabrication process where the substrate is removed at specified locations, which can create structures suspended in air.

3.1.6 *cantilever*, *n*—a test structure that consists of a beam suspended in air and anchored or supported at one end (see Figs. 1-3, and Fig. X1.1).

3.1.7 *fixed-fixed beam*, *n*—a test structure that consists of a beam suspended in air and anchored or supported at both ends.

3.1.8 *in-plane length measurement*, *n*—a length (or deflection) measurement made parallel to the underlying layer (or the *xy*-plane).

3.1.9 *interferometer*, *n*—a non-contact optical instrument (such as shown in Fig. 4) used to obtain topographical 3-D data sets.

3.1.10 *mechanical layer*, *n*—in a surface-micromachining process, the patterned layer (as shown in Fig. 2) that is anchored to the underlying layer where cuts are designed in the sacrificial layer and that is suspended in air where no cuts are designed in the sacrificial layer.

3.1.11 *MEMS*, *adj*—microelectromechanical systems.

3.1.12 *out-of-plane*, *adj*—perpendicular (in the *z*-direction) to the underlying layer.

3.1.13 *out-of-plane measurements*, *n*—measurements taken on structures that are curved out-of-plane in the *z*-direction.

3.1.14 *residual strain*, *n*—in a surface-micromachining process, the strain present in the mechanical layer after fabrication yet before the sacrificial layer is removed. In a bulk-micromachining process, the strain present in the suspended layer after fabrication yet before the substrate is removed at specified locations.

3.1.15 *sacrificial layer*, *n*—in a surface-micromachining process, the layer fabricated between the mechanical layer and the underlying layer. This layer is removed after fabrication. If cuts are designed in this sacrificial layer (as shown in Fig. 2),

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

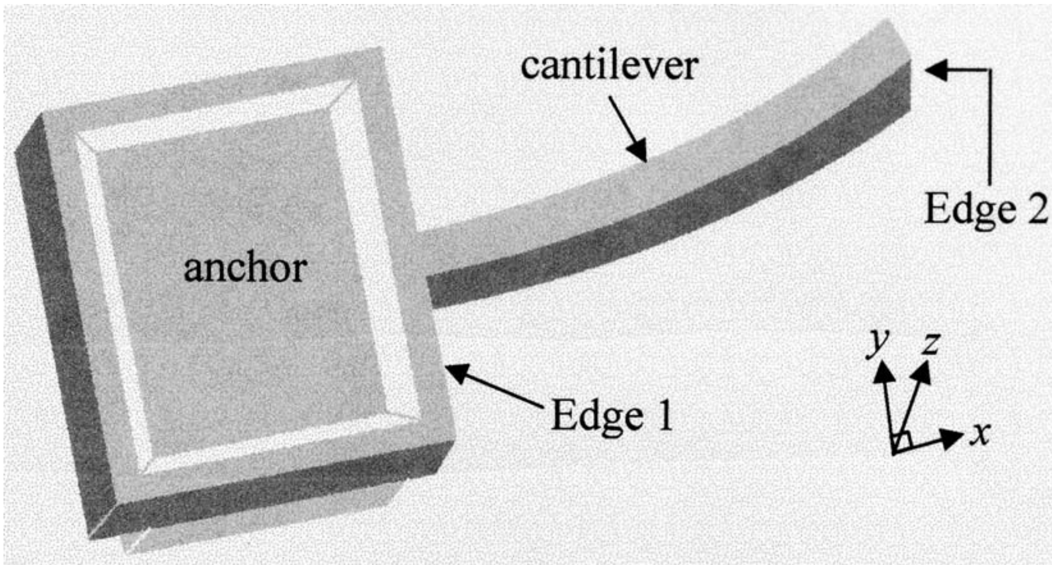
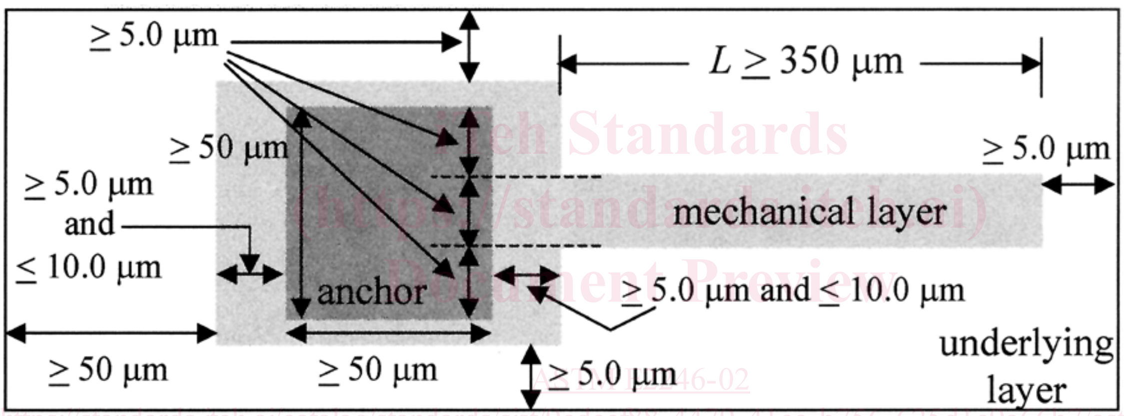


FIG. 1 Three-Dimensional View of Surface-micromachined Cantilever



NOTE 1—The underlying layer is beneath the entire test structure.
NOTE 2—The mechanical layer is included in both the light and dark gray areas.
NOTE 3—The dark gray area (the anchor) is the designed cut in the sacrificial layer. This is where the mechanical layer contacts the underlying layer.
NOTE 4—The light gray area is suspended in air after fabrication.

FIG. 2 Design Dimensions for Cantilever in Fig. 1

an anchor is created allowing the mechanical layer to contact the underlying layer in that region.

3.1.16 *stiction*, *n*—in a surface-micromachining process, a structure exhibits this when a non-anchored portion of the mechanical layer adheres to the top of the underlying layer.

3.1.17 *strain gradient*, *n*—the positive difference in the strain between the top and bottom of a cantilever divided by its thickness.

3.1.17.1 *Discussion*—Consider a surface-micromachining process. The strain gradient is present in the cantilever before the sacrificial layer is removed. After the sacrificial layer is removed, the cantilever bows out-of-plane in the plus or minus *z*-direction (as shown in Fig. 1). The strain gradient in this cantilever is zero. Examining the out-of-plane measurements of the cantilever after the sacrificial layer is removed allows for the calculation of the strain gradient present in the cantilever before the sacrificial layer is removed.

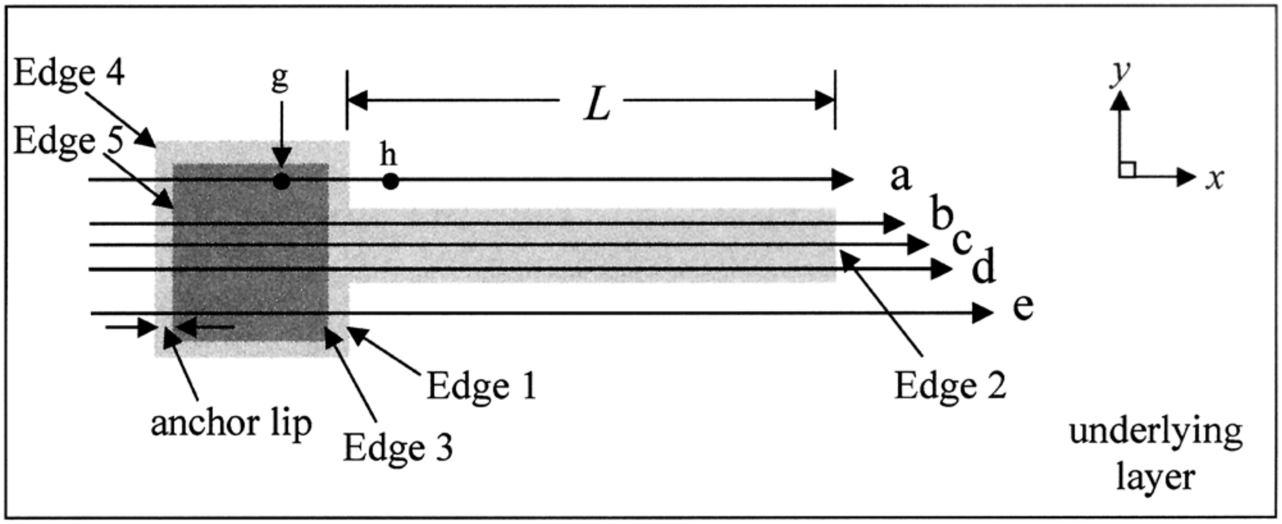
3.1.18 *substrate*, *n*—the thick, starting material in a MEMS fabrication process.

3.1.19 *support region*, *n*—in a bulk-micromachining process, the region that marks the end of the suspended structure. This region is suspended in air, attached to the substrate, or both.

3.1.20 *surface micromachining*, *adj*—a MEMS fabrication process where thin, sacrificial layers are removed, which can create structures suspended in air.

3.1.21 *test structure*, *n*—a structure (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*—an edge of a MEMS structure (such as Edge "1" in Fig. 3) that is characterized by a distinctive out-of-plane vertical displacement (as shown in Fig. 5).



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" are used in the calculation of u_W.

FIG. 3 Top View of Surface-micromachined Cantilever

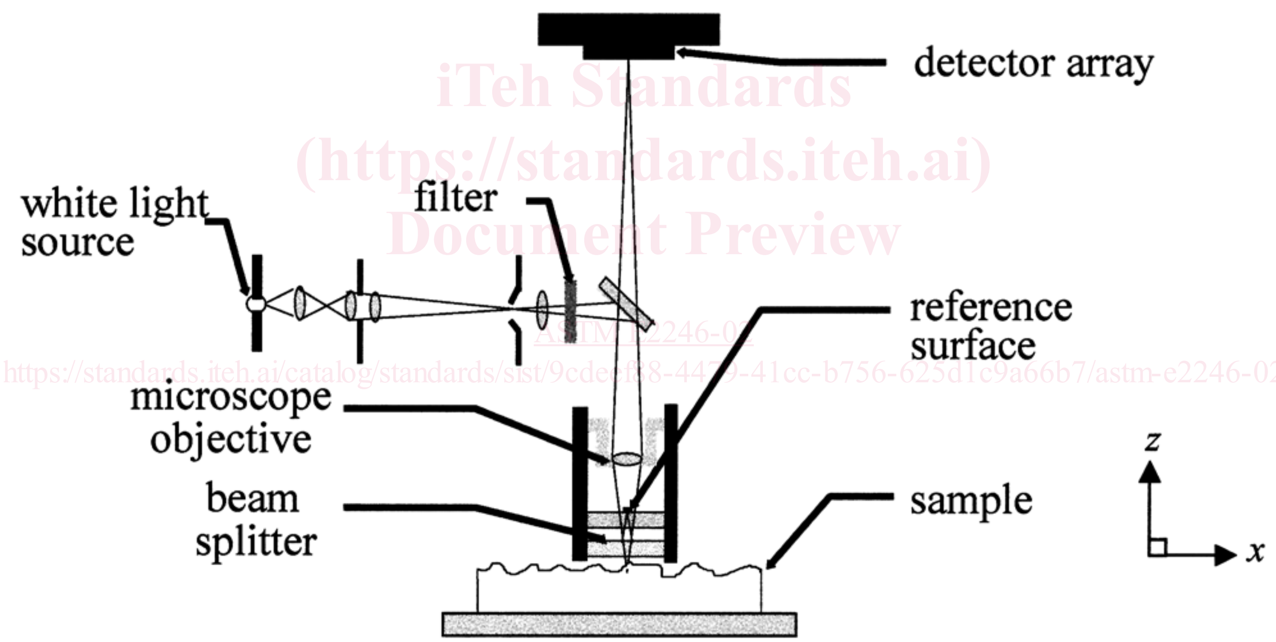


FIG. 4 Sketch of Optical Interferometer

3.1.23 *underlying layer*, *n*—in a surface-micromachining process, the layer directly beneath the mechanical layer after the sacrificial layer is removed.

3.2 *Symbols:*

3.2.1 *For Calibration*:

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 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 ruler

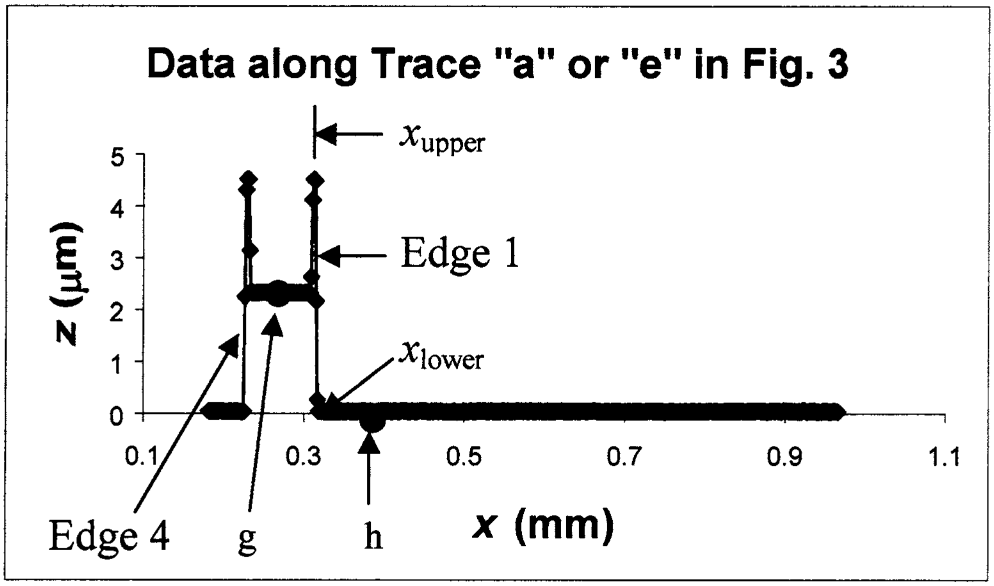


FIG. 5 2-D Data Trace Used to Find $x1_{lower}$, $x1_{upper}$, $x4_{lower}$, and $x4_{upper}$

3.2.2 *For Alignment*:

L = the in-plane length measurement of the cantilever (see Fig. 2 or Fig. 3)

$x1_{lower}$ = the x-data value along Edge "1" (such as shown in Fig. 5) locating the lower part of the transition

$x1_{upper}$ = the x-data value along Edge "1" (such as shown in Fig. 5) locating the upper part of the transition

$x3_{lower}$ = the x-data value along Edge "3" (such as shown in Fig. X1.2) locating the lower part of the transition

$x3_{upper}$ = the x-data value along Edge "3" (such as shown in Fig. X1.2) locating the upper part of the transition

$x4_{lower}$ = the x-data value along Edge "4" (such as shown in Fig. 5) locating the lower part of the transition

$x4_{upper}$ = the x-data value along Edge "4" (such as shown in Fig. 5) locating the upper part of the transition

x_{lower} = the x-data value along the transitional edge of interest locating the lower part of the transition (see Fig. 5)

x_{upper} = the x-data value along the transitional edge of interest locating the upper part of the transition (see Fig. 5)

3.2.3 *For Strain Gradient Calculations*:

a = the x- (or y-) coordinate of the origin of the circle of radius R_{int}. 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}. 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 modeling the shape of the topmost surface of the cantilever as measured with the interferometer **(1)**[3]

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

s_g = the strain gradient. Three data points (such as shown in Fig. 6) are used for this calculation

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

t = the thickness of the suspended layer, such as shown in Fig. X2.1 **(2-4)** for a surface-micromachining process

$t_{support}$ = in a bulk-micromachining process, the thickness of the support region where it is intersected by the 2-D data trace of interest (such as, Trace "a" or "e" in Fig. X1.1, as shown in Fig. X1.2)

$x1_{ave}$ = 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" (as shown in Fig. 6) locating the lower part of the transition

$x2_{upper}$ = the x-data value along Edge "2" (as shown in Fig. 6) locating the upper part of the transition

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

$z_{upper-t}$ = 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*:

$s_{g\text{-}high}$ = 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_{1pt} = the component in the combined standard uncertainty calculation that is due to the measurement uncertainty of one data point

u_c = the combined standard uncertainty value (that is, the estimated standard deviation of the result) **(5)**.

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

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

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

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

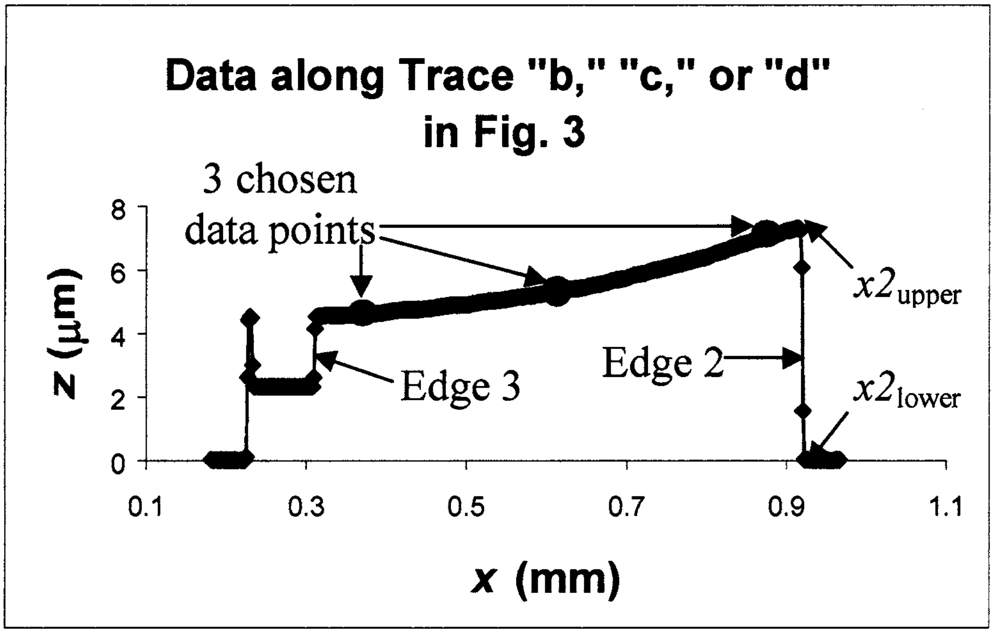


FIG. 6 2-D Data Trace Used to Find $x2_{lower}$, $x2_{upper}$, and the Three Data Points

A = the minimum thickness of the mechanical layer as measured from the top of the mechanical layer in the anchor area (or region #2 in Fig. X2.2) to the top of the underlying layer (as shown in Fig. X2.1) and as specified in the reference **(4)**

H = the anchor etch depth (as shown in Fig. X2.1). The amount the underlying layer is etched away in the *z*-direction during the patterning of the sacrificial layer.

J = this dimension (as shown in Fig. X2.1) incorporates j_a, j_b, j_c, and j_d, as shown in Figs. X2.3 and X2.4 **(4)**

j_a = the roughness of the underside of the suspended, mechanical layer in the *z*-direction (as shown in Figs. X2.3 and X2.4). This is due to the roughness of the topside of the sacrificial layer.

j_b = the tilting component of the suspended, mechanical layer (as shown in Figs. X2.3 and X2.4)

j_c = the height in the *z*-direction of any residue present between the bottom of the suspended, mechanical layer and the top of the underlying layer (as shown in Figs. X2.3 and X2.4)

j_d = the roughness of the topside of the underlying layer (as shown in Figs. X2.3 and X2.4)

$z_{reg\#1}$ = the *z* value (as shown in Fig. X2.2) of the point of maximum deflection along the cantilever beam with respect to the anchor lip

$z_{reg\#2}$ = a representative *z* value (as shown in Fig. X2.2) of the group of points in region #2 within the large anchor area

3.2.6 *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 The circular function 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.

4.2 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.

4.3 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 [4]

7.1 *Non-contact Optical Interferometer*, capable of obtaining a topographical 3-D data set and has software that can export a 2-D data trace. Fig. 4 is a sketch of a suitable non-contact optical 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 from 0.1 nm to at least 10 µm higher than the step height to be measured.

7.2 *A 10-*µm*-grid Ruler*, for calibrating the interferometer in the *xy*-plane.

[4] The same apparatus is used as in Test Method E 2244 and Test Method E 2245 and reference (**1**).

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

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).

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 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.

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 mechanical layer and should extend at least 5.0 µm beyond the outermost edges of the patterned, mechanical 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 1—Any tilt in the sample is eliminated by leveling the interferometric optics 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.

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]

9.1 Calibrate the interferometer in the *x*- and *y*-directions using a 10-µm-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.

9.1.1.2 Orient the ruler in the *y*-direction using crosshairs, if available. Record *ruler-y* as measured on the interferometer's screen.

9.1.1.3 Determine the *x*- and *y*-calibration factors using the following equations:

$$cal\text{-}x = ruler\text{-}x \,/\, inter\text{-}x \quad (1)$$

$$cal\text{-}y = ruler\text{-}y \,/\, inter\text{-}y \quad (2)$$

NOTE 2—Multiply the *x*- and *y*-data values obtained during the data session by the appropriate calibration factor to obtain calibrated *x*- and *y*-data values.

9.1.2 *For Reflective Rulers*:

9.1.2.1 Orient the ruler in the *x*-direction along the bottom edge of the field of view using crosshairs (if available).

9.1.2.2 Select the detector array size that achieves the best lateral resolution.

9.1.2.3 Adjust the intensity with respect to the brightest layer of interest.

9.1.2.4 Eliminate any tilt in the sample by nulling the fringes on the top of the flattest region of the ruler.

9.1.2.5 Recheck the sample alignment.

9.1.2.6 Take an average of at least three measurements to comprise one 3-D data set.

9.1.2.7 Move the ruler slightly in the *y*-direction and obtain another 3-D data set.

9.1.2.8 Continue until the ruler is out of the field of view.

NOTE 3—Obtain at least five data sets representative of the field of view.

9.1.2.9 For each 3-D data set, extract a 2-D data trace in the *xz*-plane at the same location on the ruler, if possible.

9.1.2.10 Record in tabular form the ruler measurements versus *x* for each *y*.

9.1.2.11 Orient the ruler in the *y*-direction along the left-hand edge of the field of view. Repeat the above steps in a similar manner.

NOTE 4—This step can be skipped if the in-plane measurements are restricted to the *x*-direction due to a smaller pixel-to-pixel spacing in that direction.

9.1.2.12 By interpolating or extrapolating, or both, use the newly created calibrated lookup table(s) to find the calibrated *x* (or *y*, or both) values for pertinent pixels within the field of view.

9.2 Calibrate the interferometer in the out-of-plane *z*-direction using the certified value of a double-sided step

[5] The same calibration procedure is used as in Test Method E 2244 and Test Method E 2245 and reference (**1**).