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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, also called an interferometric microscope. Measurements from cantilevers that are touching the underlying layer are not accepted.

1.2 This test method uses a non-contact optical interferometeric microscope 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~~ Test Method for Residual Strain Measurements of Thin, Reflecting Films Using an Optical Interferometer

E2444 Terminology Relating to Measurements Taken on Thin, Reflecting Films

E2530 Practice for Calibrating the *Z*-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si*(111)* Monatomic Steps

2.2 *SEMI Standard:*[3]

MS2 Test Method for Step Height Measurements of Thin Films

3. Terminology

3.1 *Definitions:*

~~3.1.1~~

3.1.1 The following terms can be found in Terminology E2444.

3.1.2 *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 interferomet~~er~~ic microscope.

~~3.1.2~~3.1.3 *3-D data set*, *n*—a three-dimensional group of points with a topographical *z*-value for each (*x*, *y*) pixel location within the interferometeric microscope's field of view.

~~3.1.3~~3.1.4 *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~~

3.1.5 *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~~

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

~~3.1.5~~

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

[3] For referenced Semiconductor Equipment and Materials International (SEMI) standards, visit the SEMI website, www.semi.org.

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

~~3.1.6~~

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

~~3.1.7~~

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

~~3.1.8~~

3.1.9 *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~~

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

~~3.1.9~~*-plane of the interferometric microscope).*

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

~~3.1.9.1~~

3.1.10.1 *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~~

3.1.11 *MEMS*, *adj*~~—microelectromechanical system.~~

~~3.1.11~~*—microelectromechanical systems.*

3.1.12 *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).~~ —in general, this term is used to describe micron-scale structures, sensors, actuators, and technologies used for their manufacture (such as, silicon process technologies), or combinations thereof.

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.

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~~ fixed-fixed beam or ~~a fixed-fixed beam)~~ cantilever) that is used to extract information (such as, the residual strain ~~gradient~~ or the ~~residual~~ strain gradient 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~~

σ_{6same} = the maximum of two uncalibrated values (σ_{same1} and σ_{same2}) where σ_{same1} is the standard deviation of the six step height measurements taken on the physical step height standard at the same location before the data session and σ_{same2} is the standard deviation of the six measurements taken at this same location after the data session

σ_{cert} = the certified one sigma uncertainty of the physical step height standard used for calibration

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

σ_{ycal} ~~= *the*~~ = the standard deviation in a ruler measurement in the interferomet~~e~~ric microscope'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*~~ = the *x*-calibration factor of the interferometeric microscope for the given combination of lenses

cal_y ~~= *the*~~ = the *y*-calibration factor of the interferometeric microscope 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~~

z = the *z*-calibration factor of the interferometric microscope for the given combination of lenses

cert = the certified (that is, calibrated) value of the physical step height standard

$ruler_x$ ~~= *the interferometer's*~~ = the interferometric microscope'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*~~ = the interferometric microscope'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~~

$scope_x$ = the interferometric microscope's maximum field of view in the *x*-direction for the given combination of lenses

$scope_y$ = the interferometric microscope's maximum field of view in the *y*-direction for the given combination of lenses

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~~*res* = the calibrated resolution of the interferometric microscope in the *x*-direction

$\bar{z}_{6same}$ = the uncalibrated average of the six calibration measurements from which σ_{6same} is found

z_{drift} = the uncalibrated positive difference between the average of the six calibration measurements taken before the data session (at the same location on the physical step height standard used for calibration) and the average of the six calibration measurements taken after the data session (at this same location)

z_{lin} = over the instrument's total scan range, the maximum relative deviation from linearity, as quoted by the instrument manufacturer (typically less than 3 %)

z_{res} = the calibrated resolution of the interferometric microscope in the *z*-direction

$\bar{z}_{ave}$ = the average of the calibration measurements taken along the physical step height standard before and after the data session

3.2.2 *For Strain Gradient Calculations*:

α = the misalignment angle

a ~~= *the*~~ = 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 that is measured with the interferometric microscope

b ~~= *the*~~ = 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*~~

int. An arc of this circle models the out-of-plane shape in the *z*-direction of the surface of the cantilever that is measured with the interferometric microscope

L = the in-plane length measurement of the cantilever

$n1_t$ = indicative of the data point uncertainty associated with the chosen value for $x1_{uppert}$, with the subscript "t" referring to the data trace. If it is easy to identify one point that accurately locates the upper corner of Edge 1, the maximum uncertainty associated with the identification of this point is $n1_t x_{res} cal_x$, where $n1_t$=1.

R_{int} ~~= *the*~~ = the radius of the circle with an arc that models the shape of the ~~topmost~~ surface of the cantilever that is measured with the interferometeric microscope

s ~~= *equals*~~ = equals 1 for cantilevers deflected in the minus *z*-direction of the interferometeric microscope, and equals ~~−1~~ –1 for cantilevers deflected in the plus *z*-direction

s_g ~~= *the*~~ = 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~~

g0 = the strain gradient when the residual strain equals zero

$s_{gcorrection}$ = the strain gradient correction term for the given design length

t = the thickness of the suspended, structural layer

$x1$~~$_{ave}$~~ = the average of *ave* = the calibrated average of $x1$~~$_{lower}$~~ and *uppera* and $x1$~~$_{upper}$~~
uppere

$x1_{uppert}$ = the calibrated x-value along Edge 1 locating the upper corner of the transitional edge using Trace t

$x2$~~$_{ave}$~~ = 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~~*uppert* = the calibrated x-value along Edge 2 locating the upper corner of the transitional edge using Trace t

y_t = the calibrated y-value associated with Trace t

3.2.3 *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~~

$\sigma_{repeat(samp)}$ = the relative strain gradient repeatability standard deviation as obtained from cantilevers fabricated in a process similar to that used to fabricate the sample

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~~

ave = the calibrated surface roughness of a flat and leveled surface of the sample material calculated to be the average of three or more measurements, each measurement taken from a different 2-D data trace

R_{tave} = the calibrated 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 taken from a different 2-D data trace

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

U_{sg} = the expanded uncertainty of a strain gradient measurement

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

cert = the component in the combined standard uncertainty calculation for strain gradient that is due to the uncertainty of the value of the physical step height standard used for calibration

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~~

correction = the component in the combined standard uncertainty calculation for strain gradient that is due to the uncertainty of the correction term

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~~

csg = the combined standard uncertainty of a strain gradient measurement

u~~$_{xcal}$~~ = the component in the combined standard uncertainty calculation for strain gradient that is due to the uncertainty of the ~~calibration in the~~ *drift* = the component in the combined standard uncertainty calculation for strain gradient that is due to the amount of drift during the data session

u_{linear} = the component in the combined standard uncertainty calculation for strain gradient that is due to the deviation from linearity of the data scan

u_{noise} = the component in the combined standard uncertainty calculation for strain gradient that is due to interferometric noise

u_{Rave} = the component in the combined standard uncertainty calculation for strain gradient that is due to the sample’s surface roughness

$u_{repeat(samp)}$ = the component in the combined standard uncertainty calculation for strain gradient that is due to the repeatability of measurements taken on cantilevers processed similarly to the one being measured

$u_{repeat(shs)}$ = the component in the combined standard uncertainty calculation for strain gradient that is due to the repeatability of measurements taken on the physical step height standard

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*~~ = the component in the combined standard uncertainty calculation for strain gradient that is due to the resolution of the interferometeric microscope in the x-direction

u~~$_{zcal}$~~$_{zres}$ ~~= *the*~~ = the component in the combined standard uncertainty calculation for strain gradient that is due to the ~~uncertainty~~resolution of the ~~calibration~~interferometric microscope in the z-direction ~~$\mathrm{u}_{\mathrm{zres}}$ = 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~~

3.2.4 *For Round Robin Measurements*:

L_{des} ~~= *the*~~ = the design length of the cantilever

n ~~= *the*~~ = the number of ~~reproducibility~~repeatability or ~~repeatability~~reproducibility measurements

s_{gave} ~~= *the*~~ = the average strain gradient value for the ~~reproducibility~~repeatability or ~~repeatability measurements. It~~reproducibility measurements that 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~~ *cgave* = the average combined standard uncertainty value for the strain gradient measurements that is equal to the sum of the u_c csg values divided by *n* ~~.~~

~~3.2.6~~3.2.5 *For Adherence to the Top of the Underlying Layer*::

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

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

j_b ~~= *in*~~ = 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*~~ = in a surface micromachining process, the height in the interferometeric microscope'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*~~ = 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*~~ = 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*~~ = 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~~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 (or the subscript of the symbols) above that start with the letter "*x*."

4. Summary of Test Method

4.1 A surface-micromachined cantilever is shown in Figs. 1-3. After fabrication, this cantilever bends in the out-of-plane *z*-direction. An optical interferometeric microscope (such as shown in Fig. 4) is used to obtain a topographical 3-D data set. ~~Two-D~~2-D data traces beside the cantilever (such as Traces a and e shown in Fig. 3 and Fig. 5~~) and along the top of the cantilever (such as shown in~~) and along the top of the cantilever (such as Traces b, c, and d shown in Fig. 3 and Fig. 6) are extracted from this 3-D data set for the strain gradient analysis.

~~4.2A circular arc models the out-of-plane shape of cantilevers. Three data points (such as shown in~~

4.2 Traces a and e are used to a) determine the misalignment angle, α, and b) ensure that the *x*-values obtained along the cantilever in Traces b, c, and d are indeed along the cantilever and not on the anchor lip.

4.3 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.~~

~~4.3To 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~~) define the circular function for each data trace (b, c, and d) after the data points have been modified for any misalignment. The strain gradient for each data trace is calculated from the radius of this circle. The strain gradient is the average of the strain gradient values calculated from Traces b, c, and d.

4.3.1 For Traces b, c, and d, to obtain three data points representative of the shape of a surface-micromachined cantilever: *(1)* select a transitional edge, *(2)* align the transitional edge in the field of view, *(3)* obtain a 3-D data set, *(4)* determine the attachment location of the cantilever, and *(5)* for Traces b, c, and d, obtain three data points representative of the shape of the cantilever. (This procedure may need to be modified for a bulk-micromachined cantilever.)

4.3.2 To determine the strain gradient for each data trace (b, c, and d): *(1)* account for any misalignment, *(2)* solve three equations for three unknowns for Trace c, *(3)* plot the function with the data from Trace c, *(4)* calculate the strain gradient for Trace

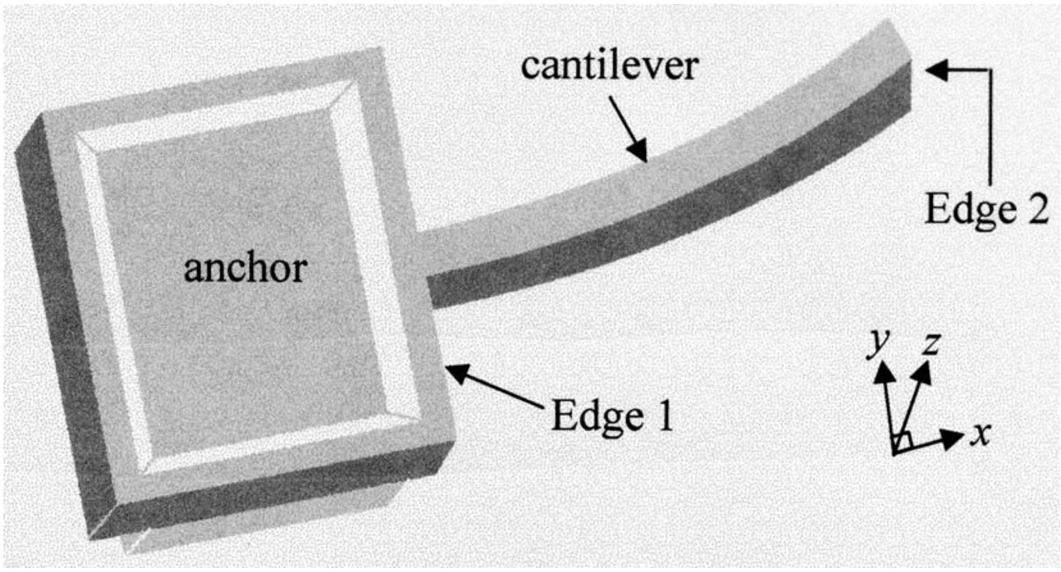
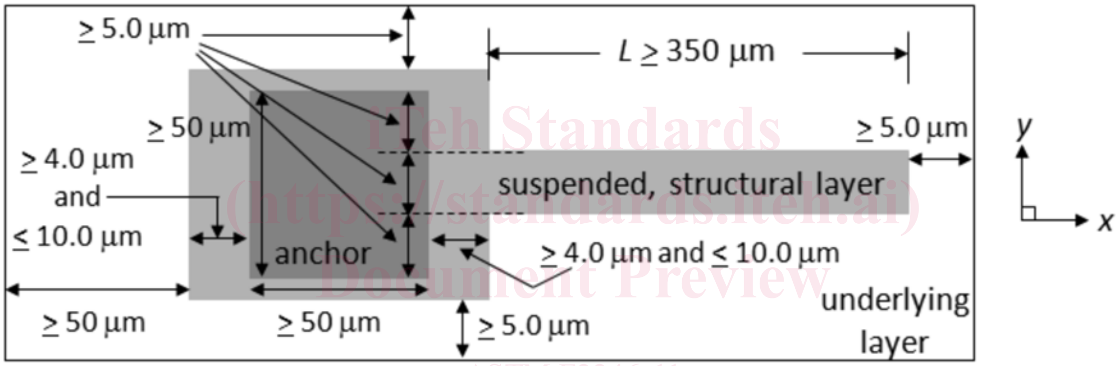


FIG. 1 Three-Dimensional View of Surface-micromachined Cantilever



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

c, and *(5)* repeat steps 2 through 4 for Traces b and d. The strain gradient is calculated as the average of the three strain gradient values obtained from the three data traces.

4.4 The equations used to find the combined standard uncertainty are given in Annex A1.

4.5 Appendix X1 ~~for a bulk-micromachined cantilever or a surface-micromachined cantilever with transitional edges greater than 8 µm in height.~~

~~4.4To determine the strain gradient: (*1*) solve three equations for three unknowns, (*2*) plot the function with the data, and (*3*) calculate the strain gradient.~~ is used to determine if the cantilever has adhered to the top of the underlying layer.

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~~X1) are not accepted.

7. Apparatus [4] (~~1~~1-3)[5]

7.1 ~~*Non-contact Optical Interferometer*~~Non-contact Optical Interferometric Microscope, capable of obtaining a topographical

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

[4] The same apparatus is used (or can be used) in Test Method E2244, Test Method E2245, and SEMI Test Method MS2.

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