Review of Traditional ASTM International Standards that have Miniaturization Potential

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ASTM standards run over a wide spectrum of topics and material classes.

Hundreds of standards for elastic deformation, plastic deformation (yield strength and hardness), fracture (strength and toughness), and thermal expansion have been developed over the last 109 years starting with irons and steels in Committee A01.

Nearly all have been developed for bulk sized specimens for engineering applications.

The suitability or adaptability of these standards to micro or nanoscale structures is briefly reviewed.

There are over 130 technical committees in ASTM.

Some are materials committees such as A01 Steel, Stainless Steel, and Related Alloys; C14 Glass and Glass Products; or C28 Advanced Ceramics (my committee), that have developed test standards tailored to a particular class of material.

Alternately, there are generic testing committees such as E28 Mechanical Testing or E08 Fracture and Fatigue.

Most of the latter focus on metals but they sometimes have subcommittees that deal with polymers or “advanced materials” or composites.

As a result of this dual track approach (materials committees and testing committees), there often is significant overlap and turf conflicts between committees.

The conflicts are usually sorted out without too much trouble.

There are literally hundreds of ASTM standards for elastic deformation, plastic deformation (yield strength and hardness), fracture (strength and toughness), and thermal expansion.

Some are master generic standards, such as E384, Microhardness of Materials which covers Knoop and Vickers hardness.

Some are material specific, such as C1326, Knoop Hardness of Advanced Ceramics.

C 1326 uses all the general requirements of E384 and references back to it, but adds specific requirements for ceramics such as best loads to use, loading rates, and how to deal with cracking.

Elastic Properties

Standard E111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus

Committee E28 Mechanical testing, *ASTM Annual Book of Standards*, Vol. 3.01

This classical standard dates back to 1955. It is intended to be used with classical engineering sized tension test specimens with a universal testing machine, dead weights, and extensometers. The general concepts may be applicable to sub millimeter miniaturization. It would be best to start with a fresh sheet of paper for miniature specimens.

Standard E 132 Test Method for Poisson's Ratio at Room Temperature

Committee E28 Mechanical Testing, Vol. 3.01

This classical standard dates back to 1958. It is intended to be used with classical engineering sized tension test specimens with a universal testing machine, dead weights, and extensometers. It recommends that a pair of extensometers be used, with special care for the transverse strain measurements. This standard is not suitable for sub millimeter specimens.

Standard E 143 Test Method for Shear Modulus at Room Temperature

Committee E28 Mechanical Testing, Vol. 3.01

This one dates back to 1959 and entails the torquing of a tube or solid cylinder and measurement of the angle of twist. It is not suitable for sub millimeter sized specimen testing since it requires the specimen be gripped in a testing machine. It is clearly intended to be used with engineering sized test specimens.

Standard E855 Test Methods for Bend Testing of Metallic Flat Materials for Spring Applications Involving Static Loading

Committee E28 Mechanical Testing, Volume 3.01

This is an interesting little test intended to measure the elastic modulus and the yield stress of very thin strips of metallic materials. The strips are loaded as cantilevers or in 3- or 4-point bending. The minimum recommended size thickness is 0.25 mm, but long spans are needed (> 35 mm). The method cannot be used for sub millimeter specimens, but some of the concepts might be applied to a new standard.

Standard E 1875 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Sonic Resonance
Committee E-28 Mechanical Testing, Vol. 3.01
Standard C 1198 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance Committee C-28 Advanced Ceramics, Vol. 15.01
(E 1875 is a clone of C 1198) (Authors: S. Gonczy, G. Quinn, J. Helfinstine)

This classical method measures the natural frequency of beams that vibrate in bending mode. It uses continuous forced excitation. It uses bend bar or larger sized specimens. The general principles are such that this methodology can be adapted to sub millimeter miniature specimens, but there may be some practicality problems. It would be easier to measure the natural frequency of a cantilever in bending, rather than the free standing bending mode as specified in E 1875.
(Standards **C 848** for Ceramic Whitewares and **C 623** for Glass are similar)

Standard E 1876 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Impulse Excitation of Vibration Committee E28 Mechanical Testing, Vol. 3.01
Standard C 1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration (Author: S. Gonczy with help from G., Quinn)
Committee C28 Advanced Ceramics, Vol. 15.01
(E1876 is a clone of C 1259)

Committee E28 Mechanical Testing, ASTM Annual Book of Standards, Vol. 3.01
This is almost identical to **C 1198** and **E 1875** listed above, with the exception that rather than use continuous forced excitation, a single impulse (a gentile tap) is used to start the specimen vibrating. It uses ordinary bend bar or larger sized specimens. The general principles are such that this methodology can be adapted to sub millimeter miniature specimens, but there may be some practicality problems. It would be easier to measure the natural frequency of a cantilever in bending, rather than the free standing bending mode as specified in E 1875.

Standard E 494 Practice for Measuring Ultrasonic Velocity in Materials
Committee E7, Nondestructive Testing, Vol. 3.03
Elastic properties are determined form the ultrasonic time of flight through a plate. This standard required specimens no less than 5 mm thick. Miniaturization is conceivable, but making very tiny transducers would be problematic.

Plastic Deformation (yield strength)

Standard E8 Test Methods for Tension Testing of Metallic Materials

Committee E28 Mechanical Testing, Vol. 3.01

This is an early standard (originally 1924) for determination of yield strength, yield point elongation, and tensile strength. This is a classic standard for direct tension tests whereby a specimen is loaded through pin holes, threaded grips, or shoulders. It is only suitable for engineering scale structures of the order on a fraction of an inch or several mm in cross section size or larger. It is not suitable for sub millimeter sized specimens.

Standard E 290 Test Methods for Bend Testing of Material for Ductility

Committee E28 Mechanical Testing, Vol. 3.01

This is a simple but interesting series of four alternative tests that entail bending thin slabs or cantilever specimens and simply observing whether the specimen develops a permanent set or not. A key step after the test is to examine the bent specimen's convex surface to see if there are any cracks present or not.

Although this test was designed to be used with engineering sized specimens, there is no reason the general concepts could be adapted to sub millimeter sized specimens, except that examining the curved surface for cracks would be very difficult.

Plastic Deformation (hardness)

There are many hardness standards in ASTM: Brinell for metals (**E 10**), Rockwell for metals (**E 18**), Vickers hardness for metals (**E 92**), etc. Many of these are intended for bulk specimens, but some are in the microhardness rage and go down to 1 gram (0.01 N).

The two advanced ceramic standards **C 1326 Knoop** and **C 1327 Vickers**, are for common-sized polished specimens with indentation loads of 19.6 N (2 kgf) and 9.8 N (1 kgf) respectively.

The instrumented indention work underway in **Committee E28** clearly is the way to go on hardness in the future. No one is advocating the use of the traditional tests for miniature specimens.

Fracture (Strength)

Standard **E8** Test Methods for Tension Testing of Metallic Materials

Committee E-28 Mechanical Testing, Vol. 3.01

This is an early standard (originally 1924) for determination of yield strength, yield point elongation, and tensile strength. This is a classic standard for direct tension tests whereby a specimen is loaded through pin holes, threaded grips, or shoulders. It is only suitable for engineering scale structures of the order on a fraction of an inch or several mm in cross section size or larger. It is not suitable for sub millimeter sized specimens.

Standard E 345 Test Method of Tension Testing of Metallic Foil

Committee E28 Mechanical Testing, Vol. 3.01

This is essentially a variant of ordinary engineering scale testing procedures with the exception that the specimens can be very thin, of the order or 50 microns in thickness. It still requires grips, strength testing machines, and specimens of the order of many millimeters or inches in length. **It is not suitable for sub millimeter sized specimens**.

Standard **E9** Test Methods for Compression Testing of Metallic Materials at Room Temperature

Committee E28 Mechanical Testing, Vol. 3.01

This also is an early (1924) test method for testing bulk cylindrical or block like specimens, a major fraction of an inch or larger. In principle it could be rewritten for miniature sub millimeter specimens, but it would be better to start with a fresh sheet of paper for such miniature specimens.

Standard **C 1161** Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature

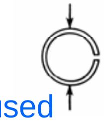
Committee C-28 Advanced Ceramics, ASTM Annual Book of Standards, Vol. 15.01

This one, which Quinn wrote, is for classic ceramic bend bars. It is not suitable for sub millimeter sized specimens and would be difficult to adapt to such.

Standard **C 1273** Test Method for Tensile Strength of Monolithic Advanced Ceramics

Committee C28 Advanced Ceramics, Vol. 15.01

This is for classic sized ceramic tension test specimens. Gripping and alignment are critical issues. This method could not be adapted to sub millimeter sized specimens.



Standard C 1323 Test Method for Ultimate Strength of advanced Ceramics with Diametrally Compressed C-Ring Specimens, Committee C28 Advanced Ceramics, Vol. 15.01

Originally written in 1996 by Segal, Quinn is in the process or revising this standard which was intended to be used with slotted tube or ring shaped specimens of the order of many millimeters diameter. The standard has some deficiencies. A small task group is working on it this summer. Originally intended to be used with engineering scale specimens, I see no reason this method cannot be scaled down to very small sizes.

Standard C 1424 Test Method for Monotonic Compression Strength of Advanced Ceramics

Committee C28 Advanced Ceramics, Vol. 15.01

This standard compresses a small, but engineering sized dumbbell shaped specimen. It is not readily adaptable to sub millimeter sized specimens.

Standard C 1499 Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics

Committee C28 Advanced Ceramics, Vol. 15.01

This is ring-on-ring testing of disk shaped specimens. It is intended for engineering sized specimens and would be difficult to miniaturize due to alignment problems.

Standard C 1557 Test Method for Tensile Strength and Young's Modulus of Fibers

Committee C28 Advanced Ceramics, Vol. 15.01

Single fibers are gripped and/or mounted on pull tabs and loaded to fracture. It would be extremely difficult to adapt this to sub millimeter sized specimens, due to gripping or mounting problems.

Standard C 158 Test Methods for Strength of Glass by Flexure

Committee C14 on Glass and Glass Products, Vol. 15.02

Engineering sized glass rods or plates are loaded in flexure. The specimens are much larger than what we use for ceramics and this test method is not adaptable for sub millimeter sized specimens.

Standard C 674 Test Method for Flexural Properties of Ceramic Whitewares

Committee C21 on Whitewares, Vol. 15.02

This is a bend bar standard for large whiteware specimens. It is not suitable for sub millimeter specimens.

Standard C 773 Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials

Committee C21 on Whitewares, Vol. 15.02

Crush right circular cylinders between platens. This is a crude engineering test meant for moderate sized specimens. It is not suitable for sub millimeter sized specimens. The general concepts could be applied, but substantial forces are needed to actually cause compression fractures. Usually one gets localized tensile stress fractures in such rudimentary set ups.

Fracture Toughness

Standard E23 Test Methods for Notched Bar Impact Testing of Metallic Materials

Committee E28, Mechanical Testing, Vol. 3.01

This is the classic Charpy or Izod type test originating in 1933. It uses very specific test specimens that are larger than one inch in size. It is not suitable for miniaturization. There are also considerable problems in interpretation. How does fracture toughness correlate with Charpy impact resistance? An analogous miniature impact type test for sub millimeter type bars or cantilevers could be devised, but would be unwise. The Charpy test is very susceptible to artifacts in specimen preparation, notch tip radius, etc. Severe problems with these would be expected with miniature specimen testing.

Standard E 399 Test Method for Plane Strain Fracture Toughness of Metallic Materials

Committee E08 Fatigue and Fracture, Vol. 3.01

This is the master fracture toughness standard developed for metals and dates back to 1970. It has evolved over the years and now has many specimen configurations including notched bars in bending, compact tension specimens, arc shaped specimens, and even disk shaped compact tension specimens. Fatigue precracking is required and the specimens are engineering sized. This standard is not suitable for sub millimeter sized specimens.

Standard E 740 Practice for Fracture Testing with Surface-Crack Tension Specimens

Committee E08 Fatigue and Fracture, Vol. 3.01

This method, indented for metals, has engineering sized tensile test plates with machined semielliptical cracks are loaded to fracture. A conceptually similar approach for ceramic test coupons (small bend bars) is in the ceramic fracture toughness standard C 1421, but that method also is for specimens of the order of millimeters or greater in size and cannot be miniaturized.

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Standard E1304 Test Method for Plane Strain (Chevron Notch) Fracture Toughness of Metallic Materials

Committee E-8 Fatigue and Fracture, Vol. 3.01

This is an alternative test method for getting KIc of metals. It uses engineering sized specimens with machined notches. The specimen must be gripped and crack opening displacement measured. It is not suitable for sub millimeter sized specimens.

Standard E 1820 Test method for Measurement of Fracture Toughness

Committee E08 Fatigue and Fracture, Vol. 3.01

This is a new standard for metallic materials that is intended to give not only KIc, but also J and CTOD. Either a point value of toughness or an R curve may be evaluated. Notched beams, compact tension, or disk shaped compact tension specimens with machined and fatigued notches are used. Load and crack mouth opening displacement must be monitored. The method is intended for engineering sized specimens.

Fracture Toughness – continued

Standard E561 Practice for R-curve Determination

Committee E08 Fatigue and Fracture, Vol. 3.01

This is a test method designed for engineering sized test coupons. It uses several configurations from the fracture toughness standard E 399. Crack extension as a function of load is monitored either optically, by crack opening displacement gages, by eddy current, or by electrical resistance measurements.

Fatigue precracking is required. These methods are not amenable to miniaturization to sub millimeter sizes.

Standard C 1421 Test Methods for Fracture Toughness of Advanced Ceramics

Committee C28 Advanced Ceramics, Vol. 15.01

This standard features single-edged precracked beam, surface crack in flexure, and **chevron-notched beam** methods. All use classically-sized bend bars. Jenkins, Salem, Bar-On, Quinn, authors. None of these are suitable for sub millimeter sized specimens.

Thermal Expansion

Standard C 372 Test Method for Thermal Expansion of Porcelain Enamel and Glaze Frits and Fired Ceramic Whiteware Products by the Dilatometer Method Committee C21 Whitewares, Vol. 15.02

Originally prepared in 1955, this is a classical test method based on either pushrod-transducer or optical measurements for length changes inside a furnace. It is intended for small, but bulk sized specimens of the order of 5 mm in size or greater. Thermocouples are required. (A miniature specimen that we might be interested in might actually be smaller than the size of a thermocouple bead!) I do not know whether the procedure in this could be adapted to much smaller specimens. Conceptually it can, but I do not know what the practical problems would be.

Standard C 1300 Test Method for Linear thermal Expansion of Glaze Frits and Ceramic Whiteware Materials by the Interferometric Method Committee C21 Whitewares, Vol. 15.02

This standard requires the use of specimen 5 mm or longer inside a furnace with a thermocouple. This seems to be a newer version of C 372, but using an interferometer with a monochromatic light source (but not necessarily a laser) to measure displacement. It references an apparatus at the National Bureau of Standards (here) going back to 1933?!

Standard E 289 Test method for Linear Thermal Expansion of Rigid Solids with Interferometry

Committee E37 Thermal Measurements, Vol. 14.02

This test method covers the determination of linear thermal expansion of rigid solids using either a Michelson or Fizeau interferometer. It appears to be a replacement for the more conventional push rod dilatometry standard E 228 that was dropped in 2005. E 289 says it is applicable to very small specimens with improved accuracy. One option in the method (Michelson double pass) enables very thin sheets or test pieces to be tested provided that they are mounted on the ends of longer fused silica pieces. The combination is put into the rig which is intended to be used with 5 -20 mm long specimens. NIST SRM's 731, 738, and 739 (glasses) were prepared by the Fizeau technique described in E 289.

Standard E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Push-Rod Dilatometer

Committee E37 Thermal Measurements, Vol. 14.02

This was a standard using classical vitreous silica push rod dilatometry and bulk sized specimens. It was dropped from the books in 2005 and apparently replaced by E 289

Standard E 381 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis

Committee E37, Thermal Properties, Vol. 14.02

This method uses a different apparatus and smaller specimens than the older E 228. Nevertheless, they recommend lengths between 2 and 10 mm. It is difficult to understand the difference between this rig and a conventional push rod dilatometer apparatus. Apparently a fixed base is used with E 381 and a furnace is opened or closed around the base and specimen. It does not seem like this method is suitable for sub millimeter sized specimens.

So, the vast majority of ASTM standards for fracture strength, yield strength, fracture toughness, elastic moduli and thermal expansion that are on the books are not suitable for miniaturization.

New standards need to be devised.

What other activities are underway in ASTM?

Ongoing ASTM Standardization Activities

Committee E 56

Nanotechnology

- Terminology
- Particle Size

Committee E 28

Mechanical Testing

- E 2546-07 Instrumented Indentation Practice
  (data for E and H)

Dylan Morris will cover this.

ISO

NIST SRM's

- 5-in-one
- Micro chevron notch bond strength

Committee E 08

Fracture and Fatigue

- Symposium 2000 Orlando STP 1413
- 3 MEMS thin film standards:
  in plane length
  residual strain and
  strain gradient

SEMI

- 3 MEMS standards
- Step height, E, wafer "bond strength"

Committee C-28

Advanced Ceramics

- Strength Theta and C ring?
- Nanoindentation mechanics?
- This workshop

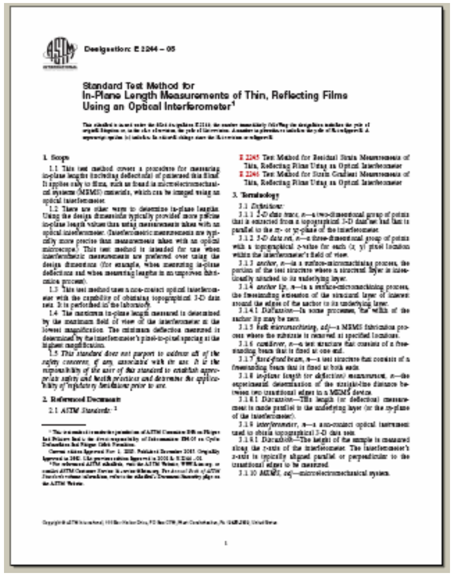
NIST SRM's

- MEMS size cantilever spring constants

The 3 ASTM E-08 MEMS Test Methods

Slide courtesy Janet Marshall, EEEL , NIST, Gaithersburg

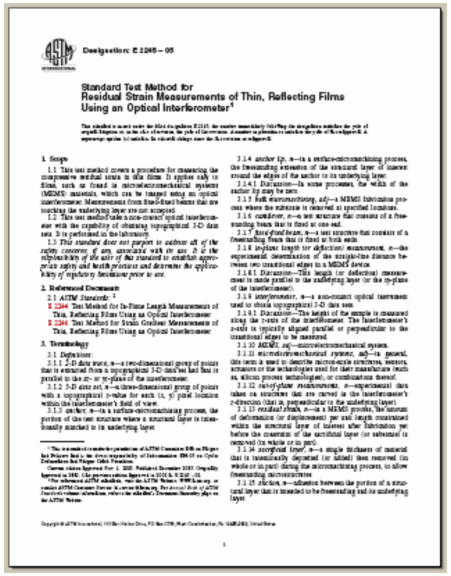
- These standards include precision and bias data (so considered validated standards)



Designation: E 2244 – 05

Standard Test Method for In-Plane Length Measurements of Thin, Reflecting Films Using an Optical Interferometer[1]

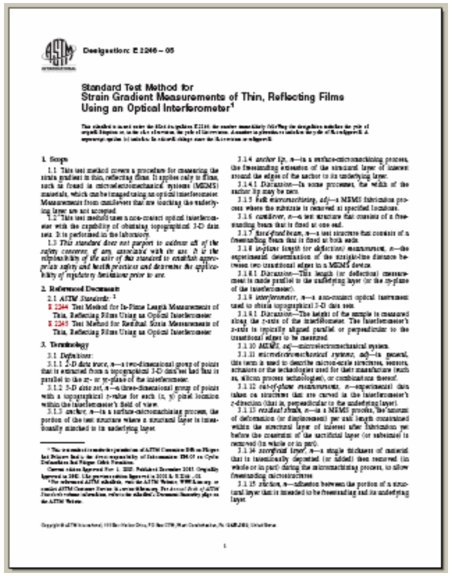
ASTM E 2244–05
in-plane length



Designation: E 2245 – 05

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

ASTM E 2245–05
residual strain



Designation: E 2246 – 05

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

ASTM E 2246–05
strain gradient

These three are closely connected and there is considerable overlap and redundancy in them. Note their consecutive numbers.

Standard E 2244 Test Method for In Plane Length Measurements of Thin Reflecting Films using and Optical Interferometer

Committee E08, Fracture and Fatigue, Vol. 3.01

An optical interferometer is used to measure *in-plane lengths* and the 3-D topography of thin film beams anchored at both ends. It can be used to measure in-plane deflections. A particular fixed-fixed beam specimen is used. This is like a classic tensile specimen with end flats with a web section. In this case, however, the end flats are fixed to a substrate. The mid section is free floating.

Standard E 2245 Test Method for Residual Strain Measurements of Thin Reflecting Films Using an Optical Interferometer

This standard uses the non contact optical interferometer to measure 3-D topographical plots of a thin film to measure the *compressive residual strain* in films. The same fixed-fixed beam specimen configuration listed in E 2244 (above) is used. Any 3-D curvature or warpage is interpreted to get a residual compression strain measurement.

Standard E 2246 Standard Test Method for Strain Gradient Measurements of Thin, Reflecting Films Using an Optical Interferometer

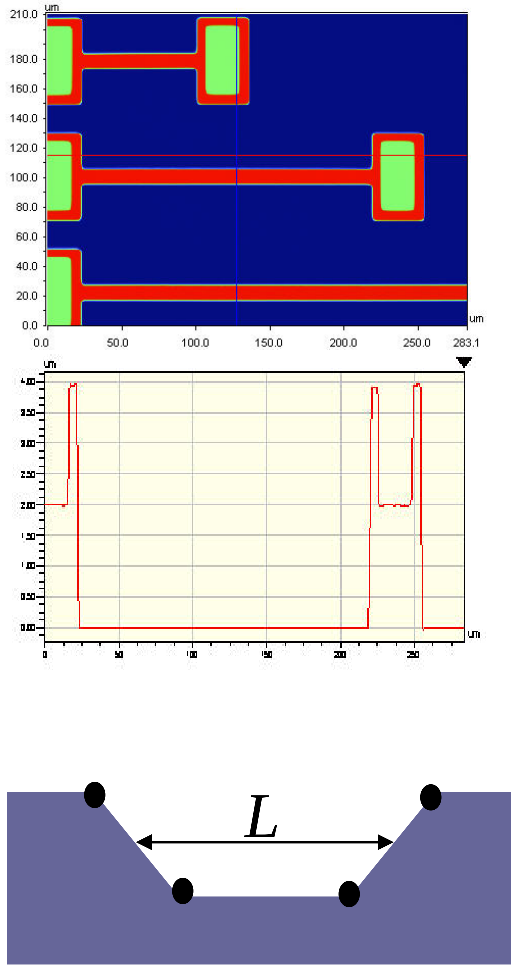
Committee E08, Fracture and Fatigue, Vol. 3.01

This new ASTM standard uses non contact optical interferometry to measure the 3-D topography of a cantilever specimen. It is only applicable to thin film type specimens and requires that a cantilever be fabricated. Deflections of the end of the cantilever are measured and interpreted to get *strain gradients*.

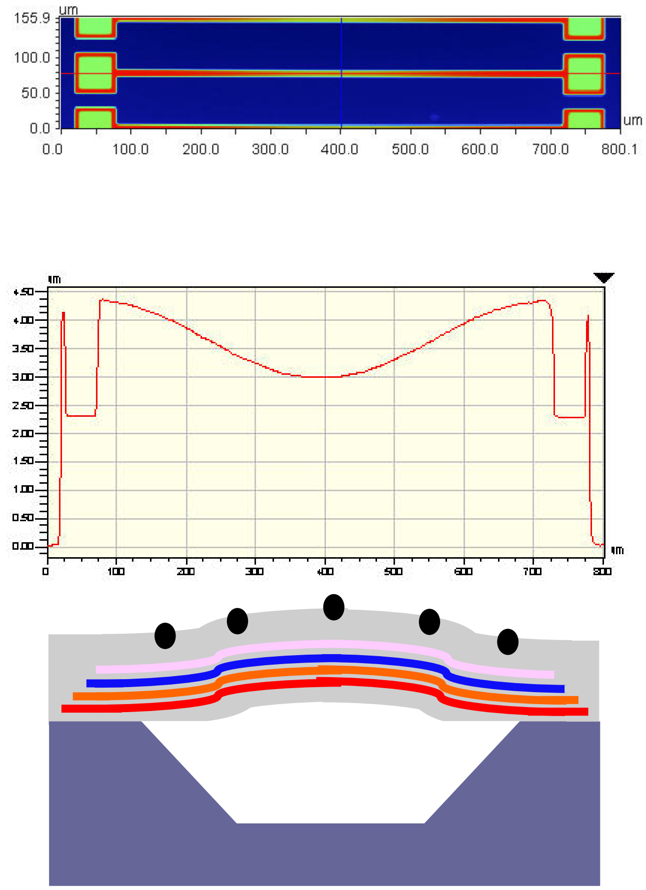
Snapshot of the 3 ASTM Standards

Slide courtesy Janet Marshall

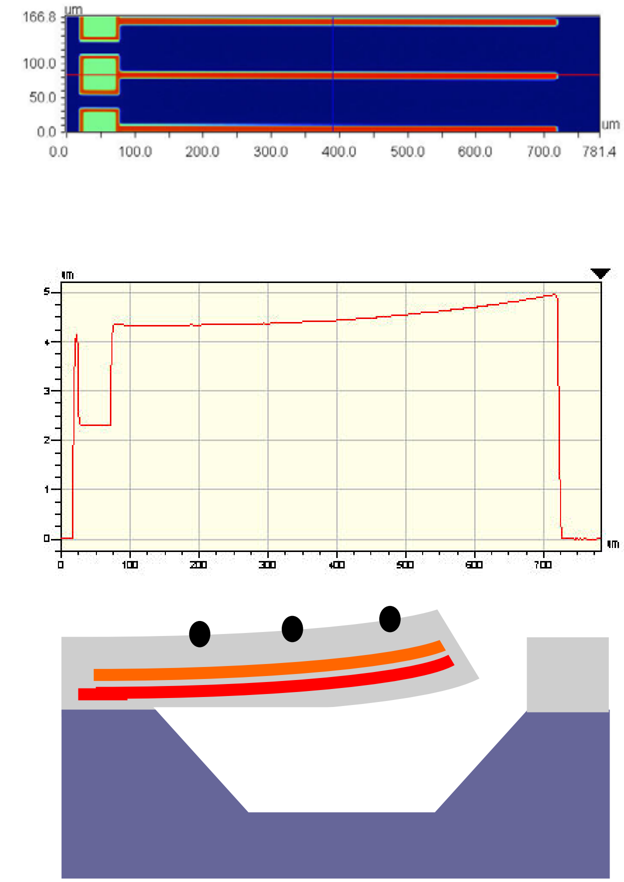
in-plane length



residual strain



strain gradient

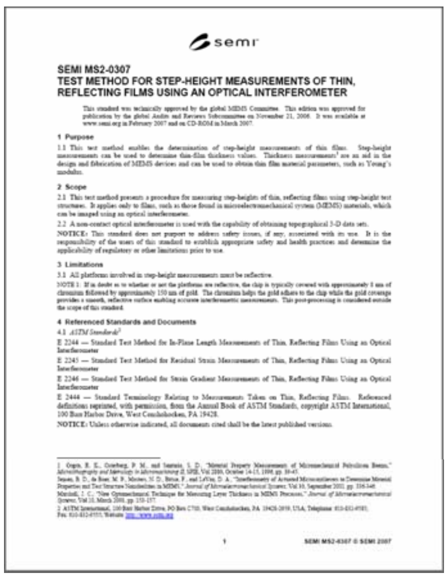


Uses an optical interferometer

The 3 SEMI MEMS Test Methods

Slide courtesy Janet Marshall

- These standards do not yet include precision and bias data (so not yet validated)



SEMI MS2-0307
step height

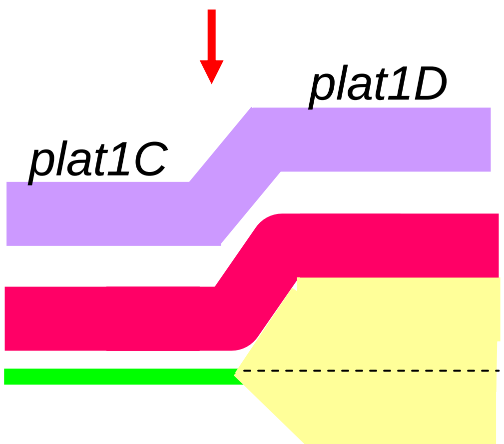
SEMI MS4-1107
Young's modulus

SEMI MS5-1107
wafer bond strength

Snapshot of the 3 SEMI Standards

Slide courtesy Janet Marshall

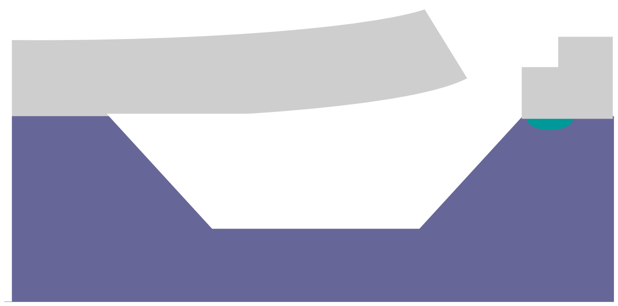
step-height



$$step1_{CD} = plat1D - plat1C$$

Uses an optical interferometer

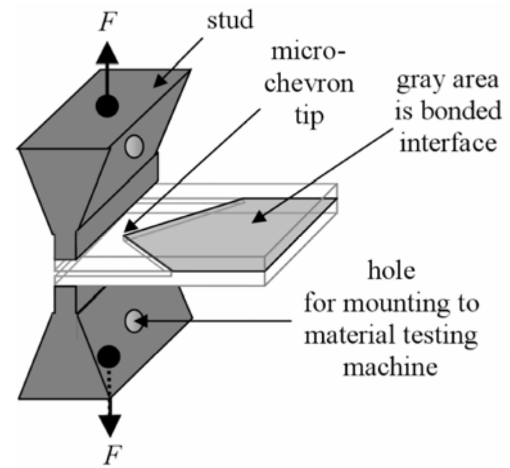
Young's modulus



$$E = 38.330\left[\frac{\rho f^2 L^4}{t^2}\right]$$

Uses an optical vibrometer

wafer bond strength



$$G_{cwb} = \frac{K_c^2}{\overline{E}}$$

where $K_C = \dfrac{F_{max} Y_{min}}{B\sqrt{w}}$

Uses a material test machine

The MNT 5-in-1 SRM

(What is it ?)

Slide courtesy Janet Marshall

MNT = Micro Nano Technology

SRM = Standard Reference Material

5-in-1 = five parameters measured at NIST on one physical test chip

1. in-plane length (ASTM E 2244)
2. residual strain (ASTM E 2245)
3. strain gradient (ASTM E 2246)
4. step height (SEMI Doc 4331A)
5. Young's modulus (SEMI Doc 4330)

Therefore, with the MNT 5-in-1 SRM, companies will be able to compare their own in-house measurements with NIST's measurements.

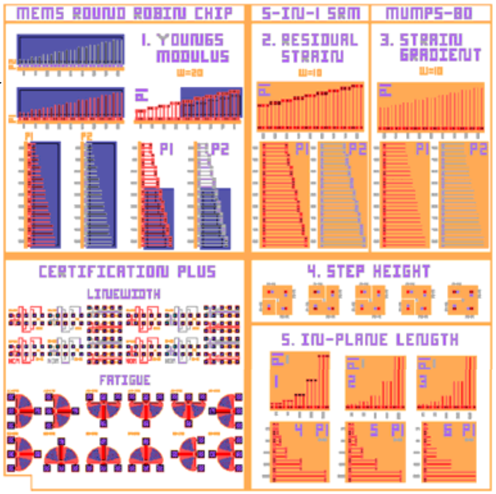
Received statements/letters of support for this work from (Veeco Metrology, Applied Materials, MOSIS, MEMSCAP, Klaros Corp, ASTM, and SEMI)

These parameters map into those specified in a USMS MN.

The MNT 5-in-1 SRM

Slide courtesy Janet Marshall

– Two or three SRM contenders:
1. MUMPs test chip (10 mm by 10 mm)
   – Can dice into 4 subdie
   – Submitted for fabrication without backside etch
   – Backside etch recommended ($10k)
   – Also, waiting for backside etch customer
2. CMOS-MEMS test chip (4.6 mm by 4.7 mm)
   – Submitted for fabrication
   – On an AMIS 1.5 μm CMOS process
3. TSMC test chip
   – Considering a design for this process



– Each SRM has six sections
1. Young's modulus (SEMI MS4)
2. Residual strain (ASTM E 2245)
3. Strain gradient (ASTM E 2246)
4. Step height (SEMI MS2)
5. In-plane length (ASTM E 2244)
6. Certification Plus

Vision for MEMS Standards

Slide courtesy Janet Marshall

ASTM E08.05.03

- (E 2244-05) In-plane length – available for purchase from ASTM
- (E 2245-05) Residual strain – available for purchase from ASTM
- (E 2246-05) Strain gradient – available for purchase from ASTM
- (E 2444-05$^{\varepsilon 1}$) Terminology – available for purchase from ASTM

SEMI: MEMS Materials Characterization TF

- (MS3-0307) Terminology – available for purchase
- (MS2-0307) Step-Height – available for purchase
- (MS4-1107) Young's modulus – available for purchase
- (MS5-1107) Wafer bond strength – available for purchase
- Stiction – work in progress
- EMM – work in progress
- Etc.

ASTM E08.05.03

- Young's modulus – work in progress
- Ultimate strength – work in progress
- Fatigue – work in progress

Ongoing ASTM Standardization Activities

Committee E 56

Nanotechnology

- Terminology
- Particle Size

Committee E 28

Mechanical Testing

- Instrumented Indentation for E and H

Committee E 08

Fracture and Fatigue

- Symposium 2000 Orlando
- 3 MEMS thin film standards:
  in plane length
  residual strain and
  strain gradient

Committee C-28

Advanced Ceramics

- Strength Theta and C ring
- Nanoindentation mechanics
- This workshop

NIST SRM's

- 5-in-one
- Micro chevron notch bond strength

SEMI

- 3 MEMS standards
- Step height, E, wafe
  "bond strength"

Daytona Workshop:

New activity in C 28?
Find a Niche?

Coordinate and cooperate with E-8 and E-28?

Joint Projects and Standards?