TECHNIQUES *by D.S. Gianola and W.N. Sharpe, Jr.*

TECHNIQUES FOR TESTING THIN FILMS IN TENSION

A review chapter[1] summarized the techniques and procedures for measuring the mechanical properties of freestanding films as of the end of 2000. Test methods can be characterized as direct or indirect. In the former, the property of interest is measured independently, e.g. Young's modulus from separate measurements of force, area, and strain on a tensile specimen. In the latter, a model of the test structure is used, e.g. Young's modulus determined from force, geometry, and displacement of a cantilever beam. This paper presents a tensile test method for directly measuring strength and indirectly measuring modulus of a thin film—silicon dioxide. These techniques are demonstrated in two tests on gold film. The approach is not new, but the detailed experimental techniques may be useful.

Silicon dioxide is often included in MEMS, although usually as a supporting layer rather than in a movable component, and its mechanical properties have been studied by various techniques. Jaccodine and Schlegel[2] furnished one of the earliest investigations of the Young's modulus of SiO_2 using an inflated "balloon" testing apparatus, much like a bulge test. In a study using thermally grown SiO_2 cantilever beams, Petersen and Guarnieri[3] employed a variable frequency oscillator to resonate the beams in an effort to measure Young's modulus. Weihs *et al*.[4] reported values for the modulus of cantilever microbeams of SiO_2 obtained using nanoindentation techniques. Several studies[5,6] have supplied values of Young's modulus via measurement of the thermal stresses in thin films. More recent investigations have measured the elastic constants of silicon dioxide using methods such as nanoindentation[7] and atomic force microscopy.[8]

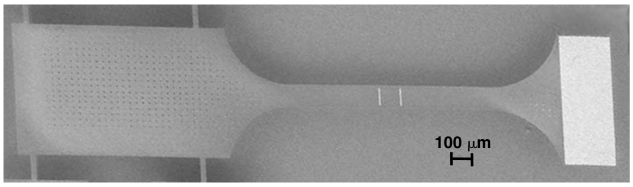

Fig. 1: Gold tensile specimen. Left end is the paddle for attaching the SiC fiber, while right end remains fixed to the substrate. Four support strips attach to fixed anchors, and are cut prior to testing.

A test method begins with the design and production of specimens; these are described in the next section. The test system for gripping, pulling, and measuring force and displacement is then presented. This approach yields direct measurements of strength, but one must determine Young's modulus by indirect means. Those are described, and results for silicon dioxide are presented. Silicon dioxide is a linear brittle material, and the test method is also demonstrated for ductile gold film. The paper ends with some concluding remarks.

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SPECIMEN SHAPE AND MATERIALS

At this size scale, it is simply impractical to pick up a specimen and place it in the grips of a test machine. MEMS are usually manufactured on a silicon wafer, which is then cut into dies, and one must arrange for part of the test structure to remain fixed to the die and the other part to be attached to the measurement system. A way of accomplishing this for a tensile specimen is shown in Fig. 1. A gold specimen is shown there because silicon dioxide is transparent and difficult to photograph. The large grip end at the left and the uniform gage section are freed from the substrate by etching the sacrificial layer underneath them. The 5 μm diameter holes in the grip allow the etchant to penetrate through to the sacrificial layer. The 110 μm wide gage section is released by the etchant penetrating from the sides. The right end remains fixed to the substrate because there are no holes in that end. There are four anchor strips on the grip to support it after etching; these are cut after the specimen is mounted.

Specimens of two materials—silicon dioxide and gold—were tested. These were manufactured, released, and provided by another organization using a proprietary process. The emphasis in this paper is on the test methods rather than the materials. These two materials are representative of thin films; silicon dioxide is linear and brittle, and gold is ductile. Only a few gold specimens have been tested; there are more results for the silicon dioxide.

TEST SYSTEM

A unique microsample tensile testing machine was employed in this research to study the mechanical properties of PECVD silicon dioxide and evaporated gold thin-film samples. Figure 2 shows the experimental apparatus, which consists of a picomotor to actuate displacement-controlled motion, a capacitance gage to precisely measure displacement, and a high-resolution load cell for force measurement. Uniaxial load is applied to the thin film samples by attaching a silicon carbide (SiC) fiber to the grip end of the specimen; the other end of the fiber is attached to the load cell.

Aligning and Gripping

Other researchers[9–11] have gripped the specimen by means of electrostatic forces, while Chasiotis and Knauss[12] used an adhesive to attach the fiber. In the current study, contact cement and UV-curable glue were used to fasten the SiC

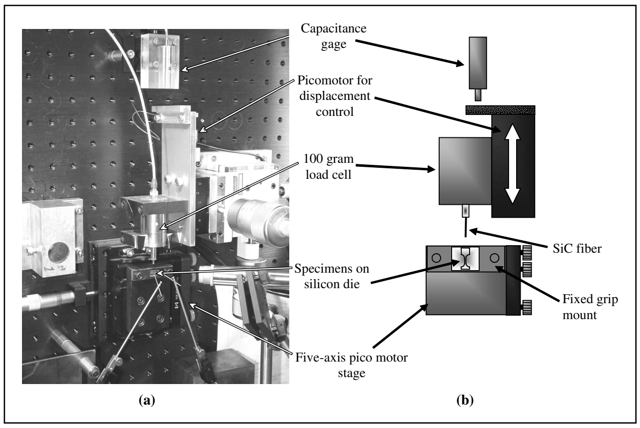

Fig. 2: Experimental apparatus (a) used for tensile testing of SiO_2 and (b) schematic showing individual components of setup. Stereo microscope used for manipulation of specimens is not shown.

fiber to the top paddle of the specimen. Figure 3 shows a specimen that has been glued to a SiO_2 specimen using UV adhesive prior to tensile testing. In the particular case of the UV adhesive, a cure time of 2 min. was used. Alignment of the SiC fiber to the tensile axis of the thin films samples is crucial for precise measurements.

To circumvent any uncertainty associated with misalignment (i.e. non-uniaxial normal loading such as bending or eccentric loading), a New Focus, Inc. 5-axis picostage (model #8801), coupled with stereo and telescoping microscopes, was utilized to align the tensile axis of the specimen. The stereo microscope (offering a generous depth of view) was used for alignment in the plane of the sample, while the telescoping microscope assisted with the out-of-plane alignment. In addition, a 20 mW HeNe laser system was employed during testing to ensure proper alignment. Due to the small width of the specimens in comparison to the beam size of the laser, a diffraction pattern was reflected from the edges of the gage section of the tensile specimens. The observed pattern is analogous to diffraction created by illumination on a rectangular slit, projected in reflection as opposed to transmission. A schematic showing the effect of incorrect alignment of the sample on the resulting diffraction pattern is given in Fig. 4. From this illustration it is shown that the reflected pattern will move some amount during motion in the tensile direction if the fiber is misaligned. Careful observation of any movement of this reflected pattern on a screen during testing would give indication of misalignment, which could be corrected during the initial stages of loading.

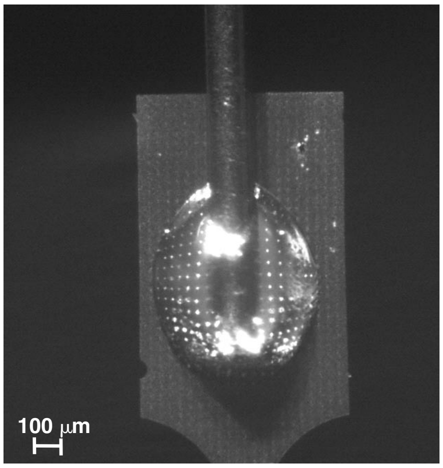

Fig. 3: Silicon dioxide specimen that has been glued to the SiC fiber using UV-curable adhesive. Fiber orientation indicates direction of tensile axis. Note that all four support strips that anchor the top paddle of the specimen to the silicon die have been cut in this image.

The attachment of the fiber causes a small eccentricity of loading due to the asymmetry that arises from gripping on only one side of the specimen. However, Saint Venant's principle can be used to show that any localized out-of-plane bending that occurs as a result of this eccentricity will not have a significant effect on the stress field in the gage section. The region of uniform stress in the sample gage is at a distance of up to 1000 times the thickness (0.5 μm, 1 μm) from the point of loading, and should not be affected by the asymmetry of loading.

Force Application and Measurement

A displacement-controlled modality was employed to apply a uniform uniaxial load on the thin film specimens. A screw-driven New Focus, Inc. picomotor (model #8701), with a resolution of less than 40 nm, induced a load on the sample. The single-axis picomotor operates by employing a piezoelectric element that turns a screw with a pitch of 80 turns /inch. A constant displacement rate was applied by means of a computer-controlled data acquisition system, and was set to $\dot{\delta} = 0.02$ μm/sec (corresponds to an approximate strain rate of $\dot{\varepsilon} = 1 \times 10^{-5}$ and 2×10^{-5} for the long and short samples, respectively) for all tests. Measurement of

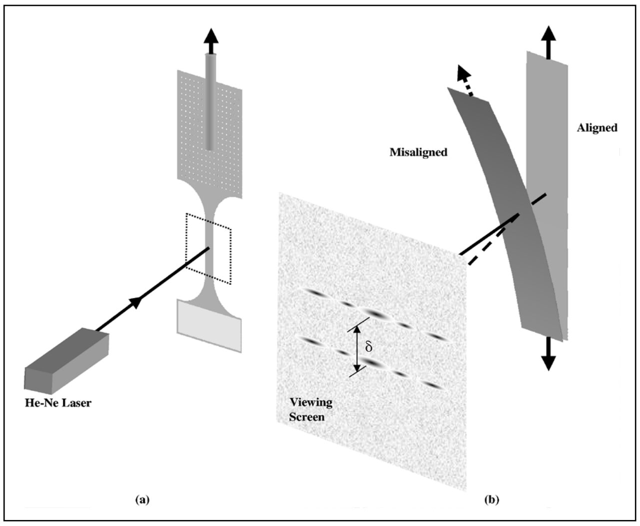

Fig. 4: Schematic of the laser alignment technique. (a) Optical setup for alignment and (b) section view of sample gage showing both aligned and misaligned configurations, along with resulting diffraction pattern. Sample is properly aligned when displacement (δ) of pattern is minimized.

load was captured using a Cooper Instruments LPM 620 100 gf (~1 N) load cell, offering a resolution of 0.1 gf.

Displacement Measurement and Test Procedure

An extremely precise method of measuring displacement is requisite to microsample testing. Typical techniques for crosshead motion do not supply the needed displacement resolution for this type of testing. To overcome this challenge, a capacitive-based probe was employed to precisely measure displacement of the test system. The HPC-40 probe, manufactured by Capacitec with a resolution of approximately 0.1 μm, is positioned near a flat metal plate, and the voltage change between the two surfaces is measured during testing. A simple calibration can restore this voltage value to displacement.

Control and acquisition of data were performed using a Pentium computer. A program coded in FORTRAN allowed for simple user input and output. Parameters such as maximum force and displacement rate are entered, and a signal is sent to the picomotor to begin the test. Concurrently, the analog output of the load cell and capacitance gage are converted to a digital output and logged in an ASCII data file. A data sampling rate of approximately 4 Hz was employed for all tests presented herein.

A typical tensile test using the technique described is completed as follows. The die of the specimens is attached to a fixed mount on the five-axis picostage. Then the SiC fiber is positioned close, while not touching, to the tensile axis of the specimen using the two microscopes. Subsequently, the picostage is positioned precisely so that the tensile axis is parallel with the fiber. The fiber is then moved to the desired location on the top paddle of the sample, and then pulled directly outward for gluing. A small amount of adhesive is applied to a fine wire, and then to the SiC fiber tip using the stereo microscope. A precision translation stage is utilized to place the fiber back onto the specimen until contact is made (observed with the telescoping microscope). The proper curing time is allowed, dependent on the type of adhesive used, and subsequently the four support strips (which connect the samples to the anchors) are cut using a micromanipulator viewed via the stereoscope. The microscope must then be removed to allow for the placement of an optical mirror for the laser alignment system. Finally, the movement of the picomotor is initiated by the computer.

ILLUSTRATIVE RESULTS

Young's Modulus of Silicon Dioxide

Silicon dioxide is a brittle material and exhibits a linear response to mechanical stimuli. As a result, one can extract the Young's modulus (E) of a material by measuring the overall displacement of the system, including that of the load cell and the gripping system. Subsequently, the stiffnesses of the two latter components can be "subtracted" to yield a value of E. Greek and colleagues[13] have developed the "differential stiffness" technique, in which two specimens with identical cross-sections, but with different lengths can be tested to result in a measurement of the Elastic modulus. This process eliminates the stiffnesses of the load cell and grips, and results in a calculation to give the simple expression

$$E = \frac{L_1/A_1 - L_2/A_2}{1/S_1 - 1/S_2}, \tag{1}$$

where S_1 and S_2 are the slopes of the load displacement plots for both short and long samples, and A_1, A_2, L_1, and L_2 are the geometrical properties of both samples. This relation holds true under the assumption that the geometries are chosen so that numbers of similar size are not subtracted in the denominator of equation 1, which can be aided by choosing a load cell that is much stiffer than the specimens. Measurements of Young's modulus of polysilicon have been conducted[13–15] using the "differential stiffness" technique, and the results agree well with accepted values of E.

A plot illustrating representative results for the load/stress displacement tests for both the short and long samples of silicon dioxide is shown in Fig. 5. The slopes of the least-squares linear regression fit of the load displacement data quantify the stiffness of the material. As expected, the shorter samples exhibit a larger value of stiffness in comparison to their long counterpart specimens. The test results confirm that SiO_2 indeed displays a linear response, and no evidence of macroscopic plasticity was observed. Table 1 shows the results of the calculations of Young's modulus for the material studied using the "differential stiffness" technique, along with other published values of E for SiO_2 for comparison.

Two single crystalline silicon dies (cleaved from the same wafer) were used as the substrate for the mechanical testing, accounting for a total of 20 specimens (10 per die). As presented in Table 1, the measured values for the Elastic modulus of SiO_2 for the die 1 and die 2 specimens are 68 ± 5

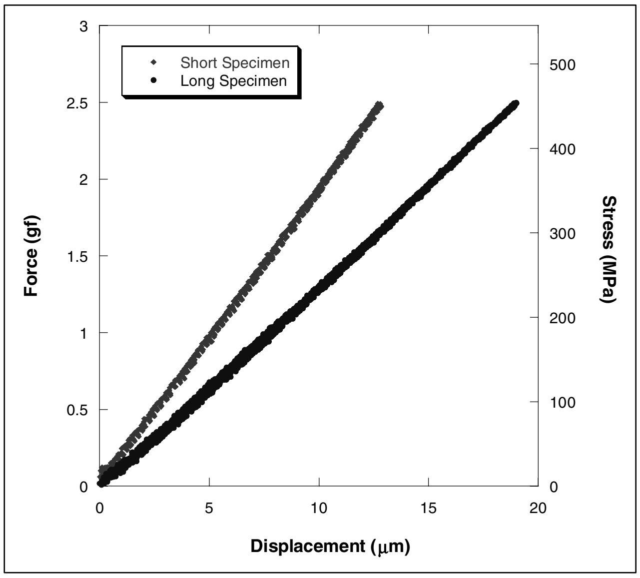

Fig. 5: Plot showing force/stress displacement data for both one short and one long specimen of silicon dioxide. Force/stress displacement plot illustrates linear brittle behavior of SiO_2.

Table 1—Results of measurement of Young's modulus of SiO_2 for this study, along with other published values of E.

MATERIAL	SAMPLE THICKNESS	E (GPa)	TEST METHOD	COMMENTS	REFERENCE
Die 1	0.5 µm	68 ± 5	Tensile	Differential Stiffness	current study
Die 2	0.5 µm	60 ± 9	Tensile	Differential Stiffness	current study
Thermal SiO_2	< 2 mm	66	Bulge	Assumed $\nu = 0.18$	[2]
Thermal-wet, thermal-dry, and sputtered SiO_2	0.3–0.4 µm	57–92 ± 20%	Resonance	Various depositions	[3]
Thermal SiO_2	1 µm:	64	Beam bending		[4]
Thermal SiO_2	1 µm	83	Nanoindentation		[4]
PECVD, LPCVD	1–3 µm	64 ± 2 61 ± 2	Brillouin light scattering	Calculated from thermal stresses	[5]
PECVD	0.5 µm	59	Beam bending	Calculated from thermal stresses	[6]
PECVD	0.5 µm, 2 µm	59–82	Nanoindentation	Various dielectric interlevels	[7]
Thermal SiO_2	0.4 µm	85 ± 13	Atomic force microscopy	Beam bending	[8]
Bulk SiO_2		73			[17]

GPa and 60 ± 9 GPa, respectively. These values were obtained by averaging the stiffnesses of each set of long and short samples and substituting the result in Equation 1. The coefficients of variation were computed by summing the individual standard deviations resulting from the short and long sets. All of the tested specimens were originally deposited on the same substrate, but the silicon die was cleaved into two halves for ease of testing. As a result, the specimens were all from the same "batch", and any variance resulting from changes in deposition parameters can be neglected. The increase in the scatter for the measurements conducted on die 2 can be attributed to a change in adhesive that was used to attach the SiC fiber to the sample. A more viscous UV-curable glue was used for die 2, while a liquid contact cement was used for die 1. This change in adhesive was performed to eliminate any stress concentrations on the top paddle of the sample resulting from the contraction of the glue upon curing. However, it was much more difficult to control the size of the UV glue bead, which has a direct effect on the measured stiffness values, and in turn induced a larger variance for the values of E.

The fracture strengths of the SiO_2 specimens were also determined to be 377 ± 53 MPa and 352 ± 80 MPa for dies 1 and 2, respectively. The results demonstrate typical scatter for linear brittle materials, but a standard t-test shows that the strengths between dies are not statistically different within a 5% confidence interval.

Testing of Gold Thin Films

Gold thin film specimens on a Si die identical to the silicon dioxide samples mentioned previously were tested to confirm the feasibility of the discussed technique. The SiC fiber was attached to the Au sample in the same fashion, where the contact cement was employed as the adhesive for all testing. Figure 6 shows a representative load/stress displacement plot for both a short and long Au sample. The nonlinearity shown at the beginning of the test is associated with the straightening of the sample, and is not an intrinsic material response. By determining the stiffness from the linear regime of the curves, Young's modulus can be estimated from the differential stiffness method. An elastic modulus of 86 GPa was calculated from load-displacement plots of a short and long sample, which agrees well with a recent study of Au thin films.[16]

Some indication of macroscopic plasticity was observed, and it can be seen that it is difficult to discern the linear elastic regime from the onset of plastic deformation. As a result, some amount of uncertainty is inherent in the calculation of Young's modulus. However, it has been shown that the differential stiffness technique is feasible for ductile as well as linear brittle materials.

CONCLUDING REMARKS

Bending tests require low forces and generate large displacements; tensile tests are just the opposite. A source of error in bending tests at this size scale arises from the boundary conditions, which can be imprecise because of the etching process. Tensile tests require precise alignment, which can be difficult, but the laser reflection procedure is effective for these small specimens.

Determination of Young's modulus by the differential approach requires precise measurements of the slopes to avoid propagation of uncertainties. These results demonstrate that this can be accomplished with the relatively simple techniques and procedures presented. A more sophisticated approach in which strain is measured directly on the tensile gage section[15] is preferred, but may not be worth the added

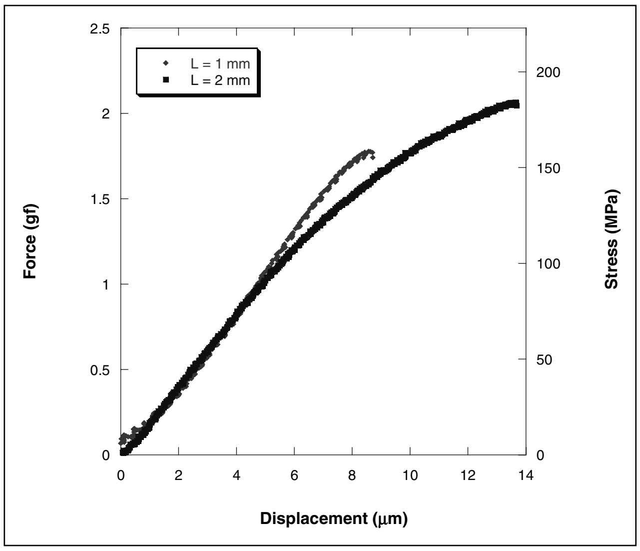

Fig. 6: Plot showing force/stress displacement data for both one short and one long specimen of gold.

effort. For example, experiments measuring the Young's modulus of polysilicon samples[14] give a coefficient of variation of ±13 percent using the differential stiffness technique, while reporting ±6 percent using a direct interferometric strain measurement system.

This is an efficient test method for freestanding thin films. Several specimens (14 in this case) can be produced on a single die. Attaching the fiber to the grip goes quickly with the UV curing adhesive. The components of the test setup are readily available at reasonable cost, and the data acquisition/analysis program is simple. One can conduct tensile tests in a fashion similar to the ASTM standard with relative ease.

References

1. Sharpe, W.N., Jr., *The MEMS Handbook,* CRC Press, 3.1–3.33, (2001).

2. Jaccodine, R.J., and Schlegel, W.A., "Measurement of Strains at Si-SiO$_2$ Interface," J. Appl. Phys. **37** (6), 2429–2433 (1966).

3. Petersen, K.E., and Guarnieri, C.R., "Young's Modulus Measurements of Thin Films Using Micromechanics," J. Appl. Phys. **50** (11), 6761–6766 (1979).

4. Weihs, T.P., Hong, S., Bravman, J.C., and Nix, W.D. "Mechanical Deflection of Cantilever Microbeams: A New Technique for Testing the Mechanical Properties of Thin Films," J. Mater. Res. **3** (5), 931–942 (1988).

5. Carlotti, G., Doucet, L., and Dupeux, M., "Elastic Properties of Silicon Dioxide Films Deposited by Chemical Vapour Deposition from Tetraethylorthosilicate," Thin Solid Films, **296**, 102–105 (1997).

6. Zhao, J.H., Ryan, T., and Ho, P.S., "Measurement of Elastic Modulus, Poisson Ratio, and Coefficient of Thermal Expansion of On-wafer Submicron Films," J. Appl. Phys., **85** (9), 6421–6424 (1999).

7. Chen, F., Li, B., Sullivan, T.D., Gonzalez, C.L., Muzzy, C.D., Lee, H.K., Levy, M.D., Dashiell, M.W., and Kolodzey, J., "Influence of Underlying Interlevel Dielectric Films on Extrusion Formation in Aluminum Interconnects," J. Vac. Sci. Technol. B, **18** (6), 2826–2834 (2000).

8. Sundararajan, S., Bhushan, B., Namazu, T., and Isono, Y., "Mechanical Property Measurements of Nanoscale Structures Using an Atomic Force Microscope," Ultramicroscopy, **91**, 111–118 (2002).

9. Tsuchiya, T., Sakata, J., and Taga, Y., *MEMS Structures for Materials Research,* MRS Symposium, **505**, 285–290 (1998).

10. Sharpe, W.N., Jr., Turner, K., and Edwards, R.L., *MEMS Structures for Materials Research,* MRS Symposium, **518**, 191–196 (1998).

11. Sharpe, W.N., Jr., and Jackson, K., *Microscale Systems: Mechanics and Measurements Symposium,* Society for Experimental Mechanics, ix–xiv (2000).

12. Chasiotis, I., and Knauss, W.G., *Microscale Systems: Mechanics and Measurements Symposium,* Society for Experimental Mechanics, 56–61 (2000).

13. Greek, S., and Johansson, S., "Tensile Testing of Thin Film Microstructures," Proceedings of SPIE, **3224**, 344–351 (1997).

14. Sharpe, W.N., Jr., Turner, K.T., and Edwards, R.L., "Tensile Testing of Polysilicon," EXPERIMENTAL MECHANICS, **39** (3), 162–170 (1999).

15. Sharpe, W.N., Jackson, K.M., Hemker, K.J., and Xie, Z., "Effect of Specimen Size on Young's Modulus and Fracture Strength of Polysilicon," Journal of Microelectromechanical Systems, **10** (3), 317–326 (2001).

16. Emery, R.D., and Povirk, G.L., "Tensile Behavior of Freestanding Gold Films, Part I. Coarse-grained Films," Acta Materialia, **51**, 2067–2078 (2003).

17. Spearing, S.M., "Materials Issues in Microelectromechanical Systems (MEMS)," Acta Materialia, **48**, 179–196 (2000). ■