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# Tensile testing of MEMS materials—recent progress

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Tension tests, while standardized for common structural materials, are currently being developed and used for MEMS materials by a small number of researchers. This paper presents recent progress at Hopkins in four areas:

- Comparison of the tensile test method with different approaches; agreement is found with Young's modulus measurements from membrane tests and with fracture strengths from other tensile tests.
- Tension-tension fatigue; increased life with decreased applied stress is measured, yielding S-N plots similar to those of metals.
- Stress versus axial and lateral strain of thick-film silicon carbide; Young's modulus = 420 GPa, Poisson's ratio = 0.21, fracture strength = 0.8 GPa.
- Polysilicon stress-strain behavior at high temperatures; it deforms inelastically at temperatures above 750°C

## 1. Introduction

Measuring mechanical properties of materials manufactured by processes used in MEMS is not easy. One must be able to: (1) obtain and mount a specimen, (2) measure its dimensions, (3) apply force or displacement to deform it, (4) measure the force, and (5) measure the displacement, or preferably, measure the strain. This is not always possible for MEMS materials; in fact, it is most often neither possible nor practical. It is then necessary to resort to inverse methods in which a model (simple or complex) is constructed of the test structure. Force is applied to the test structure and displacement is measured with the elastic, inelastic, or strength properties then extracted from the model.

One would prefer to determine mechanical properties by direct methods similar to the tensile test approaches of ASTM. A small number of researchers are developing and using tension test methods. In addition to work at Hopkins, Tsuchiya *et al.* pioneered electrostatic gripping for tensile tests [1], Greek *et al.* grip tensile specimens by hooking a ring on one end [2] as do LaVan *et al.* [3]. Read has developed novel approaches [4] as have Chasiotis and Knauss [5]. Bravman *et al.* test thin aluminum films in tension [6], and Kapels *et al.* elongate tensile specimens with thermal actuators [7]. More recent novel approaches have been presented by Haque and Saif [8] who test 100 nanometer thick aluminum, and by Yoshioka *et al.* [9] who have an on-chip scheme. Our approaches are initially presented in [10, 11]. For a comprehensive review of all test methods as well as results, the reader is referred to the chapter, Mechanical Properties of MEMS Materials, in [12].

This paper presents the latest results of ongoing research and testing at Hopkins. It was originally accepted as a short review article, but developments in the intervening ten months are more interesting. The paper is divided into four sections corresponding to the four topics listed in the abstract. Any new test method is naturally suspect and one needs to validate it against other ones if possible; recent 'exact' comparison of modulus measurements with those from membrane tests are comforting. Many versions of MEMS operate for billions to trillions of cycles so fatigue behavior is of prime interest. Initial tension-tension tests of polysilicon show a 'metal-like' behavior in contrast to expectations for ceramics. MEMS technology is constantly searching for new materials and manufacturing processes, and silicon carbide has some promising characteristics. Specimen preparation is different for each material, and we are pleased to present the first-ever stress-strain results for silicon carbide film. Finally, some polysilicon microdevices are thermally actuated, which leads one to question the material behavior at high temperature. We present the first-ever tensile results for polysilicon at high temperatures; it behaves like a ductile material.

## 2. Validation of test method

One means of evaluating completely new test methods is to compare various ones in hopes of getting agreement among the results. Eventually one method emerges as most generally useful, and a 'round robin' exercise is conducted in which several laboratories follow the same procedures to test the same material. If this is successful, then standardization of the test method can proceed. Mechanical testing of MEMS materials is only at the very first stage.

TABLE I Fracture strengths from [13]

| Participant–laboratory | Number of tests | Average strength (GPa) | Standard deviation (GPa) |
|---|---|---|---|
| Coles–Hopkins | 28 | 2.85 | 0.40 |
| Knauss–CalTech | 19 | 3.13 | 0.46 |
| LaVan–Sandia | 98 | 4.27 | 0.61 |
| Read–NIST | 5 | 2.87 | 0.41 |
| Tsuchiya–Toyota | 19 | 3.23 | 0.25 |

Fracture strengths have been measured for a common polysilicon material produced at Sandia; Dr. David LaVan arranged the exercise. The specimens were fabricated in the shape and sizes preferred by the participants and tested in their laboratories. Details are given in [13] and a partial summary of the results are presented in Table I.

With the exception of the Sandia values, all the strengths are very close to 3 GPa. The forces in the Sandia setup were measured with a nanoindenter inserted into a ring at the end of each specimen; the instrument pulled in its lateral mode. This is not a normal operation and although it had been calibrated for lateral forces, there may have been unknown friction involved. The Hopkins values agree with earlier fracture strengths of 3.09 ± 0.18 GPa measured on polysilicon from Sandia [14]. Strengths are relatively easy to measure and the fact that our results agree in general with those from other test methods gives one confidence in the test method.

Young's modulus is much more difficult to measure, and in addition to our work, only Greek *et al.* [2] and Chasiotis and Knauss [5] have measured it in tension. An interesting comparison of modulus measurements has recently been made in cooperation with Dr. Stuart Brown of Exponent, Inc. Tensile and membrane specimens of 0.5 micron thick silicon nitride were fabricated at Hopkins and tested there and at Exponent, respectively. The specimens are pictured in Fig. 1. The die in each case is one centimeter square.

The tensile specimens were tested following the usual procedures [10], and the membranes were pressurized on one side with the deflection measured interferometrically on the other. A finite element analysis of the membrane and the supporting die enable extraction of both the modulus and the residual stress. The comparison of results is shown in Table II.

TABLE II Compared results for silicon nitride

| | Number of tests | Modulus (GPa) | Poisson's ratio | Strength (GPa) | Residual stress (GPa) |
|---|---|---|---|---|---|
| Hopkins | 7 | 257 ± 5 | 0.22 ± 0.03 | 5.83 ± 0.25 | NA |
| Exponent | 4 | 258 ± 1 | NA | NA | 114–130 |

The almost exact agreement between the two modulus results is surprising, but shows the maturity of both test methods.

## 3. Fatigue of polysilicon

Fatigue testing of metals originated with rotating bending machines, and that continued to be the common method until the development of servo-controlled electrohydraulic test machines, which enable uniaxial tension-compression loading. It is easier to test MEMS materials in bending, and Brown *et al.* [15] and Kahn *et al.* [16] have developed specimens and procedures for high frequency cyclic in-plane bending of polysilicon. There is considerable merit to this approach because many microdevices that vibrate do so parallel to the substrate. However, bending tests inevitably mean stress and strain gradients, and we have developed test methods for uniaxial loading, albeit in tension-tension only.

The polysilicon specimens are 3.5 $\mu$m thick, 50 $\mu$m wide, and either 500 or 1000 $\mu$m long. One end remains fastened to the substrate and the other is gripped by gluing a thin silicon carbide fiber to it. A schematic of the fatigue test setup is shown in Fig. 2. The die with the specimens on it is glued to a metal block, which is fastened directly to a piezoelectric force cell. The fiber glued to the specimen is connected to a piezoelectric actuator, which has a peak force value of 1000 N and

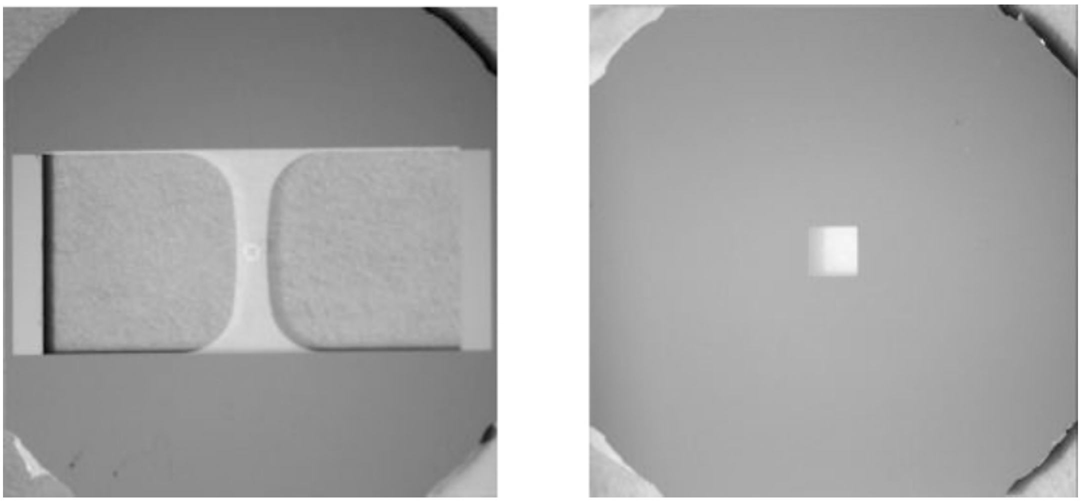

*Figure 1* Tensile specimen on the left and membrane specimen on the right.

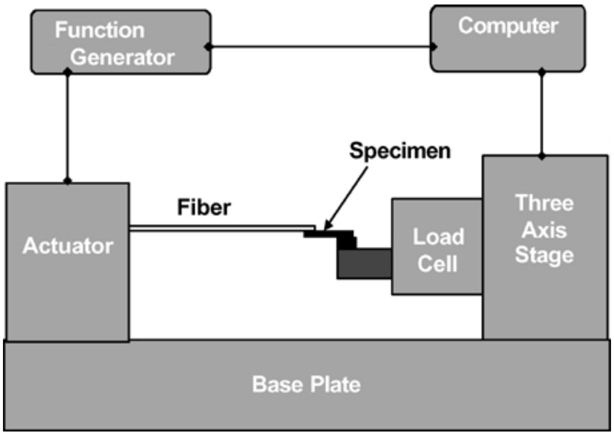

*Figure 2* Schematic of the tension-tension fatigue setup.

maximum displacement of 40 $\mu$m with a maximum frequency of $\sim$1 kHz. The load cell has a range of ±50 N with a frequency response of $\sim$1 kHz. A sinusoidal waveform is generated using a digital function generator. A PC controls the test using a program written in Agilent VEE, which displays the waveform from the load cell and counts the cycles until the sample breaks.

The initial results from what is to be an extensive test program are shown in Fig. 3. The static strengths were measured by pulling the specimen slowly with the piezoelectric actuator. The results from ten tests yield 1.10 ± 0.01 GPa with no dependence upon the length of the specimen.

The overall shape of the S-N plot is similar to what one would expect for a metal. This increase in life with decrease in applied stress has also been observed by Brown *et al.* [15] and Kahn *et al.* [16]. Note also that the cycling frequency has no effect on the results—at least in this range.

## 4. Silicon carbide

Bulk silicon carbide has a high Young's modulus ($\sim$430 GPa), good mechanical properties at high temperature, and high resistance to chemical attack. These attributes make it attractive for MEMS, and researchers are developing processes for manufacturing thin-film silicon carbide microdevices by methods similar to those used for polysilicon, etc. We are collaborating with Dr. Chris Zorman at Case Western Reserve University and with Professor Marc Spearing at MIT who are providing specimens.

Our goal is to measure stress versus axial and lateral strain on specimens that are the same planar shape as the tension one shown in Fig. 1. Our collaborators provide us with one-centimeter-square dies with the silicon carbide patterned onto or into them. Processing these to apply reflective markers for interferometric strain measurement and to etch away the substrate underneath the specimen has turned out to be quite a challenge. The specimens from MIT are relatively thick at 25 microns, and the silicon carbide is deposited into a mold that is cut into the silicon wafer by the Deep Reactive Ion Etch (DRIE) method. Silicon carbide is then deposited into the mold (and onto both sides of the wafer) by vapor deposition. The overcoating on the specimen side and on the back side of each die must be removed. This is not easy since silicon carbide is a hard material. The back side can be cleaned in a lapping machine, but the front must be hand-polished to avoid damaging the specimen. The window in the back side is etched in the silicon wafer by patterning it with photoresist and etching with xenon diflouride. Fig. 4 shows a specimen as viewed from the back side.

The first tensile stress versus axial and lateral strains ever recorded for silicon carbide film are plotted below in Fig. 5.

The stress-strain curve does not start at zero because one must put some initial tension in the specimen in order to straighten and align it so that strains can be measured by interferometry. The modulus of 417 GPa in this case is similar to the common handbook value of 430 GPa, but the strength is less than for bulk material—0.8 GPa versus 2 GPa. The Poisson's ratio is typical for

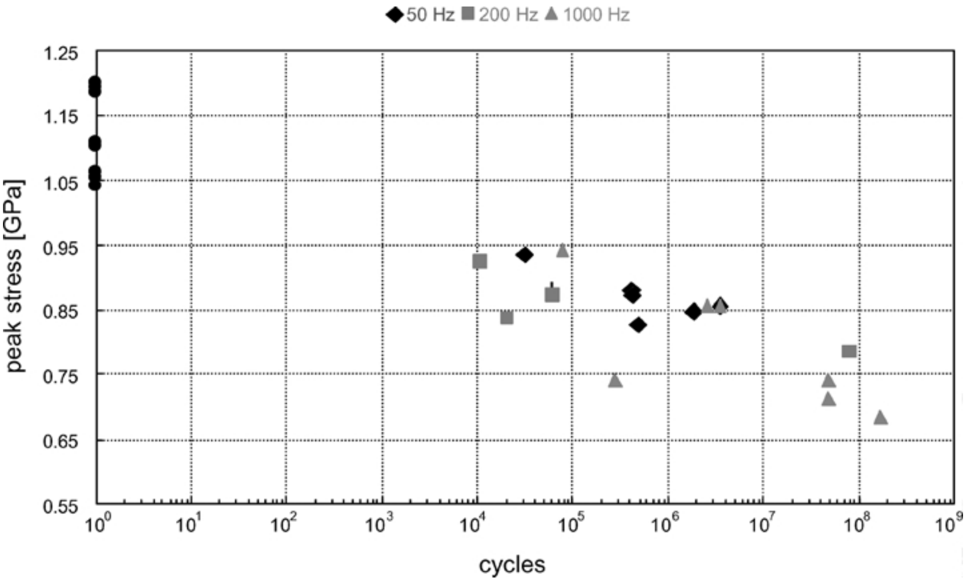

*Figure 3* Static and fatigue results for polysilicon.

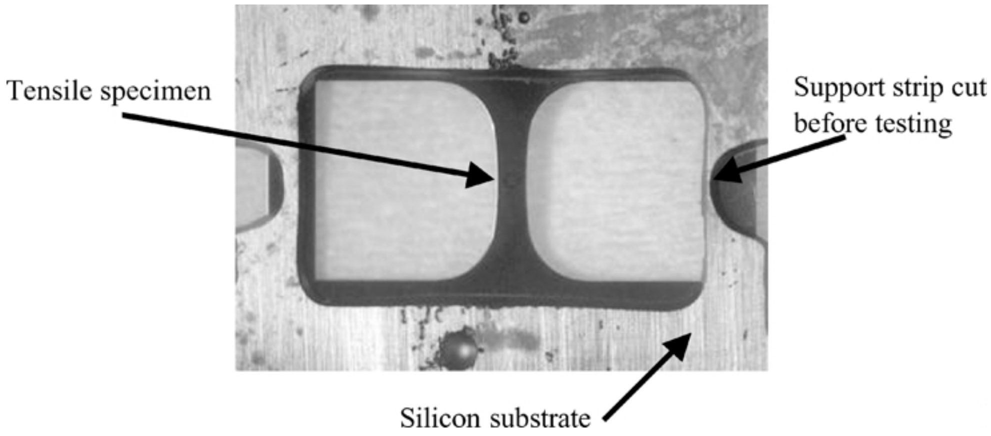

*Figure 4* A silicon carbide specimen in its silicon support frame.

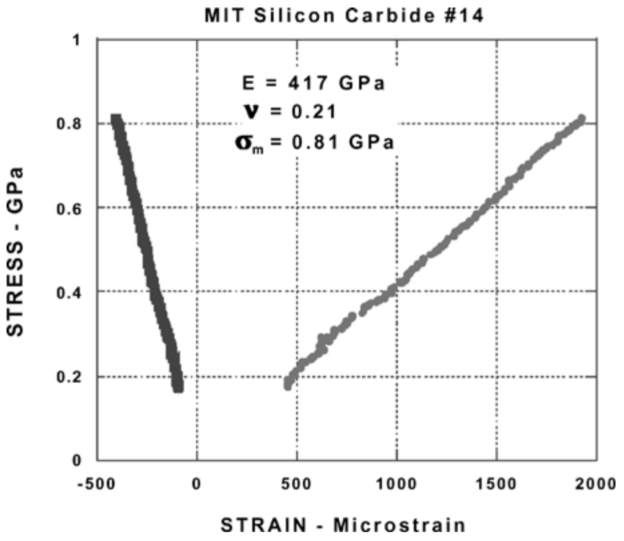

*Figure 5* Stress–strain for silicon carbide.

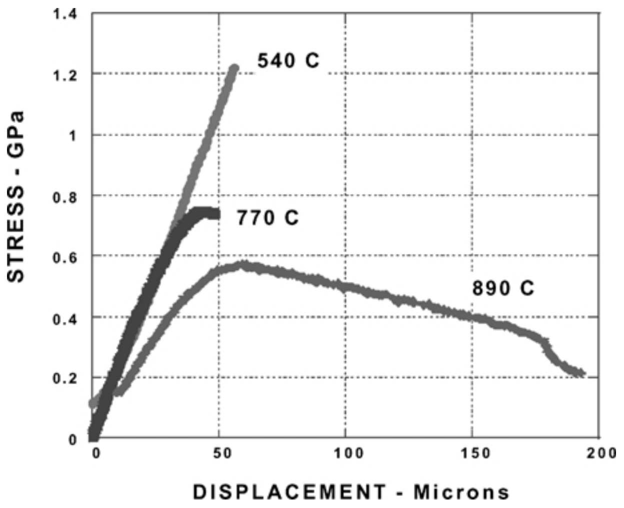

*Figure 6* Polysilicon at high temperatures.

ceramics. Clearly, many more tests must be conducted to develop a reliable data set, but it is encouraging to get these first results.

## 5. Polysilicon at high temperature

Thermal actuators can be made from polysilicon, which can be heated resistively. The temperatures reached can be quite high; one can observe the glow of the actuating arm. It is therefore important to determine the properties of the material at elevated temperatures. Previous tensile tests over the range 0°C to 250°C showed a modest decrease in the modulus and no significant change in the strength of polysilicon [17]. Furthermore, the behavior was linear and brittle in this temperature range.

We have long been interested in whether polysilicon would deform inelastically at higher temperatures and have finally been able to show that it does. Fig. 6 is a plot of stress versus displacement for polysilicon specimens the same shape as the silicon carbide one in Fig. 4. They were heated by passing a current through the specimen via lead wires attached to the wide grip ends of the specimens. Note that these are not stress-strain plots. Gold reflective markers diffuse into the polysilicon around 250°C. Initial attempts with platinum markers have not worked at these high temperatures. Measuring strain will be more of a challenge than we prefer.

Polysilicon continues to behave in a linear, brittle manner at 540°C, but begins to deform nonlinearly before breaking in a brittle fracture at 770°C. At the higher temperature, it exhibits large ductility, and Fig. 7 shows a photograph of the specimen that was tested at 890°C. The necked region is similar in appearance to that of a ductile metal.

## 6. Concluding remarks

New technologies usually begin with an emphasis on new products, materials, and processes. More fundamental research follows. That is the case with MEMS where microdevices were on the market well before extensive measurement of material properties were begun. There has been ever-expanding activity in mechanical testing of materials used in MEMS over the past five years with various types of on-chip and off-chip test methods introduced.

Tensile testing as conducted at Hopkins is one approach that has the advantage of direct stress and strain measurement and the disadvantage of large specimens. This test method complements others and contributes to the overall measurement of mechanical

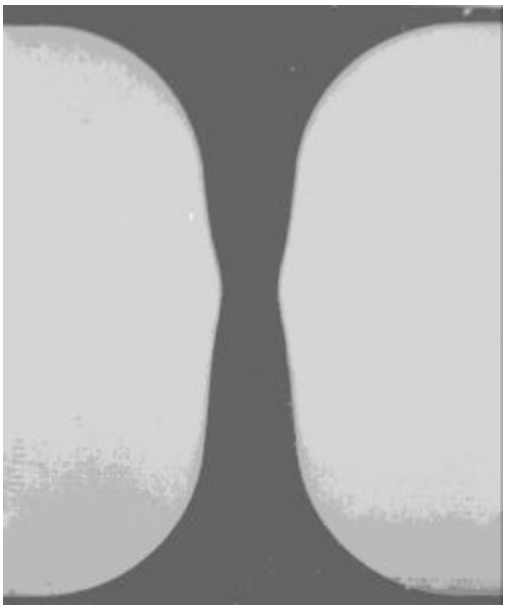

*Figure 7* A polysilicon tensile specimen after tensile testing at 890°C.

properties of materials at this scale. The comparison of results from tensile and membrane tests is an example of this synergy. Each method has its own features and the agreement of a common property—Young's modulus—enhances the validity of both.

As this tensile test approach has developed, it is natural to use it in different loadings (fatigue), for different materials (silicon carbide), and in different environments (high temperature). Initial results in these three directions have been presented here, and work is continuing to both refine the techniques and procedures and to acquire a substantial database of properties.

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