

TITLE:

# Mechanical reliability of silicon microstructures

AUTHOR(S):

Tsuchiya, Toshiyuki

#### CITATION:

Tsuchiya, Toshiyuki. Mechanical reliability of silicon microstructures. Journal of Micromechanics and Microengineering 2022, 32(1): 013003.

**ISSUE DATE:** 

2022-01

URL:

http://hdl.handle.net/2433/284806

#### RIGHT

This is the Accepted Manuscript version of an article accepted for publication in [Journal of Micromechanics and Microengineering]. IOP Publishing Ltd is not responsible for any errors or omissions in this version of the manuscript or any version derived from it. The Version of Record is available online at https://doi.org/10.1088/1361-6439/ac3cd6; CC BY-NC-ND licence; The full-text file will be made open to the public on 9 December 2022 in accordance with publisher's 'Terms and Conditions for Self-Archiving'; This is not the published version. Please cite only the published version. この論文は出版社版でありません。引用の際には出版社版をご確認ご利用ください。







# **Mechanical Reliability of Silicon Microstructures**

Toshiyuki Tsuchiya

Department of Micro Engineering, Kyoto University, Kyotodaigaku-Katsura C3, Nishikyo-ku, Kyoto 615-8540, Japan

Corresponding author: Toshiyuki TSUCHIYA

Department of Micro Engineering, Kyoto University

Kyotodaigaku-Katsura C3, Nishikyo-ku, Kyoto 615-8540, Japan

Tel./Fax: +81-75-383-3690

Email: tutti@me.kyoto-u.ac.jp





#### **Abstract**

In this article, an overview of the mechanical reliability of silicon microstructures for micro-electro-mechanical systems (MEMS) is given to clarify what we now know and what we still have to know about silicon as a high-performance mechanical material on the microscale. Focusing on the strength and fatigue properties of silicon, attempts to understand the reliability of silicon and to predict the device reliability of silicon-based microstructures are introduced. The effective parameters on the strength and the mechanism of fatigue failure are discussed with examples of measurement data to show the design guidelines for highly reliable silicon microstructures and devices.

## **Keywords**

silicon, mechanical reliability, strength, fatigue





#### 1 Introduction

Silicon is a fundamental material for both semiconductor and microelectromechanical devices. Since semiconductor devices, such as pn-junction diodes and transistors, were developed, the electrical properties of silicon have been investigated widely. Although the piezoresistive effect was also found and reported in the early stage of semiconductor device research [1], little attention has been given to the mechanical properties of silicon, especially its mechanical reliability. The reason was that semiconductor fabrication technologies were dedicated for small and planar structures, and were not suitable for fabricating mechanical components. Later, the excellent elastic properties and high piezoresistive coefficient of silicon were recognized, and sensor devices, such as strain gauges and pressure sensors using silicon, were developed. Then, a famous review article by Petersen [2], which pointed out the potential of silicon as a mechanical material on the microscale, boosted research on silicon micromechanical devices. Silicon, as an elastic material compared to steels, has several times higher yield point, similar Young's modulus and one-third of the density of steels, which helps realize a mechanical system with much smaller size and lower power consumption. Now, we can fabricate silicon gears, escapements and balance springs in wristwatches by deep-reactiveion etching technology [3,4]. Our basic understanding of silicon microstructures in the early stage of micro-electro-mechanical system (MEMS) research and development includes the risk of their sudden failure against shock; on the other hand, they show no fatigue failure in longtime operation.

Silicon has a diamond crystal structure with covalent bonds and shows brittleness on fracture similarly to glass materials. Unlike ductile metals, such as steels and aluminum alloys, silicon and glass structures may fail catastrophically, which means that devices consisting of silicon microstructures lose their functions suddenly without any pre-indications. This is critical if such devices are being used in important safety systems.

In terms of long-term reliability, silicon has been observed to show no fatigue failure because it did not show any dislocation motions or plastic and ductile deformations at room temperature, which was supported by some experimental results. Loading rate did not affect the bending of silicon wafers [5], and no failure was observed after a proof test by the cyclic loading on a cantilever-type piezoresistive accelerometer [6]. However, fatigue-like behaviors,





such as the dependence of strain rate on the bending strength of silicon beams [7] and the failure of precracked silicon beams after a number of cycles of constant-amplitude resonant vibrations [8], were observed on thin silicon device structures.

Therefore, the application of micro-electro-mechanical devices fabricated using silicon microstructures in harsh environments has often been questioned. There have been strong demands to evaluate the mechanical reliability of silicon and to investigate the mechanism underlying the reliability to expand the application area of silicon mechanical devices.

Under such circumstances, many research studies have been conducted to understand the fracture and fatigue failure of silicon microstructures. This article aims to clarify the mechanical reliability of silicon microstructures used in MEMS. The elastic properties of silicon have been reported in a previous articles [9], so the present article focuses on the strength and fatigue properties and mechanisms that ensure the mechanical reliability of MEMS devices.

### 2 Fracture Strength

#### 2.1 Theoretical strength

The theoretical tensile strength of single crystal silicon is evaluated as 18.8 GPa using Orowan's theory [10], in which the strength is determined from energy conservation, where the work of binding force on the fracture plane is converted to energy to create two opposing fracture surfaces. The theoretical shear strength is calculated as 13.7 GPa, which is derived from the energy required to cause a slip on the plane [11]. The ratio of the shear strength to the tensile strength indicates a rough tendency of the fracture mode of solid materials. Covalent bond materials show a ratio close to unity and also brittleness, which means that cleavage fracture occurs more easily than ductile deformation along shear directions. The ratio is also related to the motion of defects (dislocations). High shear strength means a high energy barrier of dislocation motion, so instead of generating and moving dislocations, fracture tends to occur in silicon.

Although the theoretical tensile strength is high and there is no dislocation motion in a silicon crystal at room temperature, silicon fails under relatively low stress levels, from 500 MPa to 3 GPa [12,13]. The reason is the existence of non-idealities, such as defects, surface





roughness and cracks. In silicon microstructures, critical defects existing on the surface predominantly cause their failure and they are usually introduced during fabrication processes. Therefore, not only the average strength but also its distribution, which is caused by the deviation of the critical size of defects, should be measured and considered.

#### 2.2 Statistical analysis

The strength of silicon microstructures is usually analyzed statistically because failure is predominantly induced by flaws existing in the structure and the flaw size is not the same among specimens. The strength of brittle materials is expressed using Weibull statistics [14], which is based on the weakest link theory. The structure of the brittle material is modeled as a chain consisting of multiple rings with distributed strengths. The failure of the chain is predominantly caused by that of the weakest link in the chain. In the simplest model, where the links in the chain show the same failure mode and the strength of each link shows a normal distribution, the structural failure is expressed by the Weibull cumulative fracture probability, the probability of failure at the applied stress  $\sigma$  or lower:

$$F(\sigma) = 1 - e^{-\frac{\sigma^m}{\alpha}V_{\rm E}},\tag{1}$$

where m and  $\alpha$  are the shape and scale parameters, respectively. The former indicates scatterings, also called the Weibull modulus. The scale parameter represents the magnitude of strength and the average tensile strength is described as

$$\mu = \alpha V_E^{-1/m} \Gamma\left(\frac{m+1}{m}\right),\tag{2}$$

where  $\Gamma(x)$  is the gamma function and  $V_{\rm E}$  is the effective volume showing the size effect. The larger the structure, the higher the probability of the existence of a weaker link and the lower strength. When the stress on the specimen is distributed,  $V_{\rm E}$  is expressed as

$$V_E = \int_V \left(\frac{\sigma(V)}{\sigma}\right)^m dV. \tag{3}$$

The Weibull moduli of silicon microstructures are often reported to range from 5 to 20, which correspond to the standard deviations of about 23% and 6.2% of the average values. The size effect expressed with the equation may give an impression that a smaller specimen is stronger, which is correct if the fracture origin is the same. The size effect analysis may provide an estimation of the fracture origin location [15]. However, it is difficult to compare the Weibull





parameters among different reports because there are too many parameters that affect the measured strengths. Figure 1 shows the reported average tensile strength of various silicon microstructures as a function of their side surface area of the gauge part [15–26]. There are large variations in strength even when the tested specimens are of the same size. It is almost impossible to make the fabrication conditions the same and to control the failure modes identically.

#### 2.3 Test modes

Various test methods using different test modes for silicon microstructures have been developed. Table I lists the pros and cons of each test mode for the strength evaluation of silicon microstructures.

The uniaxial tensile test is the most preferred mode in the material strength test, but it is difficult to conduct it for microscale structures[27]. There are various tensile test methods that have been developed suitable for microstructures [12,16,21–23,28–30], but there is no universal test method. The reasons are variations in specimen fabrication and specimen chucking methods. On the basis of round-robin test results, it is concluded that there is no significant difference between the test methods as long as the specimens are properly fabricated and tested [19,26].

The cantilever bending test [13,20,31–34] and membrane bulge test [35,36] were conducted because the test system is versatile and easy. A nanoindenter machine is often utilized for pushing cantilever beam specimens and pneumatic or hydraulic pressures are used for deforming membrane specimens. One of the issues is the stress concentration at the edge where the maximum stress is applied. Another issue is the fracture mode. In most of the bending- and bulge-mode tests, the strength of a polished surface was measured, and the measured strength does not represent the strength of patterned microstructures.

Owing to the strong demand to improve the performance and reliability of optical scanner devices, in which torsional beam structures are utilized, torsional-mode tests have been conducted [37–44]. Because the maximum stress appears along the centerline of the wider side surface of beams with a rectangular cross section, the surface roughness of the beam is critically important and the scallops created by the Bosch process are often considered as a fracture origin.





For brittle materials, fracture toughness is a good measure for strength evaluation. In silicon microstructures, fracture toughness is measured with notched specimens in which the notch is introduced by either photolithographic patterns or cracks formed by nanoindentation. The measured values agreed well with these of bulk specimens (~1 MPa√m). However, it note that the fracture toughness of the specimen of thin film silicon should be treated to be in the plane stress state, whereas that of bulk specimens is usually in the plane strain state.

#### 2.4 Crystallinity

The crystallinity of silicon does not affect the strength significantly; the strengths of single crystals and polycrystalline silicon are not significantly different. However, amorphous silicon shows lower strength, which may be due to the damage caused by hydrofluoric acid used for sacrificial etching [35,45]. There are some reports that discussed the effects of deposition, annealing and grain size of polysilicon films [45,46]. They suggested an effect of microstructures on the strength.

The crystallographic orientation effect has been investigated [16,47,48]. The tensile strength does not show significant differences among specimens with three major in-plane orientations: <100>, <110> and <111> axial directions}. The <100>-direction specimen shows higher fracture strain because of the anisotropy of Young's modulus. There is a significant difference in the Weibull modulus of measured tensile strength among the three orientations. The <111>-direction specimen shows a smaller deviation than the <110>- and <100>-direction specimens, which may be due to the smooth fracture surface of the <111>-direction specimen [47].

It is also important to discuss the effect of internal defects, dopants and impurities in the structures, which may affect strength properties. However, there are only a few reports of these effects, because the defects on the surface dominate the strength and it is difficult to investigate bulk effects.

#### 2.5 Fabrication process

As discussed above, surface defects are main causes of failure, and their properties are dominant parameters of strength. Single-crystal silicon has polished surfaces both at the bottom and on top, and the surfaces have high strength, as shown by the bending test of cantilever





beam specimens [13]. However, the defects might be introduced on the surface during the bonding process for fabricating silicon-on-insulator (SOI) wafers and the thermal treatment process for annealing and oxidization. A deposited silicon film has a surface roughness caused by the non-uniformity of deposition and the grain boundary of polycrystalline films. The defect size and roughness are relatively small and are usually not the dominant parameters.

The patterning process, including photolithography and etching, predominantly affects the strength. A poor lithography process causes ragged patterns and tapers on the sidewall of photoresists. These imperfections in the photoresist patterns are easily transferred to the sidewalls of etched silicon microstructures. "Scallops" generated during the cycle process consisting of SF<sub>6</sub> isotropic silicon etching and fluorocarbon passivation (Bosch process) are also identified as the source of surface defects[20,44,49,50]. One can imagine these defects might be caused by the plasma-induced damage in silicon crystals, as discussed with regard to semiconductor device properties, but they have not been identified as the direct fracture origin.

The wet process, not only for patterning silicon microstructures but also for sacrificial etching to release structures, affects the strength. The bending strength of single-crystal silicon cantilever beams prepared by crystallographic anisotropic etching using alkaline solutions has been investigated. The radius of curvature at the concave corner formed as an intersection of two crystal planes was controlled by the etching process and the bending strength was investigated [51]. A larger radius of curvature showed a higher strength. The effect of the chemical etching process on the tensile strength of single-crystal silicon has been investigated [17]. Three anisotropic etching solutions [potassium hydroxide (KOH), ethylene diamine pyrocatechol (EDP) and tetramethylammonium hydroxide (TMAH)] and isotropic etching gas (XeF<sub>2</sub>) were used. The surface roughness defined by the different etching processes predominantly affected the measured strength. The sacrificial etching process, by which silicon microstructures are exposed to HF solution, affects the strength. The etching-induced damage on polycrystalline silicon microstructures has been discussed [15,52]. Oxygen precipitation and grain boundaries will form pits on the surface where the fracture is initiated.

#### 2.6 Temperature effect

The effect of temperature on strength has been discussed in terms of its relationship with 8



the fracture modes. The brittle–ductile transition temperature (BDTT) of silicon depends on the microstructure size [53–60], which is summarized in Fig. 2. The BDTT of bulk silicon is about 600°C and slips and ductile deformation were observed at higher temperatures[53]. The creep deformation of a silicon wafer near melting point temperature during the oxidation process has been discussed [54] and no such ductile deformation or dislocation was observed in millimeter-order structures. On the other hand, the tensile test of 26-nm-diameter nanowires [60] and the bending test of 120-nm-thick beams [59] showed ductile deformation before failure at 500°C or lower. Fig. 3 shows the fracture surface of a 4-µm-thick single-crystal silicon tensile specimen tested at 500°C [56]. Cross slip lines and necking were observed. The fracture toughness also showed temperature and size dependences. The tensile test of photolithographically notched specimens of 4 µm thickness or less [57,58] and the bending test of cantilever beams notched by focused ion beam machining [61] showed increased fracture toughness, indicating the plastic zone generation at the notch tip. The increased toughness was also observed at lower temperatures when the structure size was smaller.

These results indicate that the BDTT depends on the specimen size. The smaller the size of microstructures, the lower the BDTT. The slip plane of silicon is {111} and there are two sets of dislocations in the {111} plane. The shuffle set dislocation occurs on the bonds perpendicular to the {111} plane and the glide set dislocation occurs on the tilted bonds, as shown in Fig. 4. The glide set dislocation occurs at high temperatures and is dominant in the ductile deformation. From the experimental observations [62,63] and molecular dynamics simulation [64], the shuffle set dislocation tends to generate at high stress, but it does not move at room temperature; thus, no ductile deformation occurs. The activation energy of the dislocation is too high to form slips and brittle fracture occurs at the flaws on the surface. At least microstructures as small as 3-5 µm are not susceptible to these ductile deformations, but high-temperature operation or further miniaturization would make the ductile deformations and dislocations more significant.

#### 2.7 Methods to improve strength of silicon microstructures

Several methods have been investigated to increase the strength by controlling the shape or dimensions of surface defects. Hydrogen annealing to smoothen the surface by reflowing the



silicon surface has been reported [37]. Corners were rounded and the scallops on the side surfaces were smoothen by hydrogen annealing at 1200°C, as shown in Fig. 5. The average torsional strength was increased from 1.0 GPa to 4.4 GPa. To localize the surface treatment area and reduce the thermal budget, KrF excimer annealing was conducted and the strength was improved by smoothing the surfaces, which also improved the strength [65,66]. Anisotropic etching using alkaline solutions was conducted to remove the scallops formed during the Bosch process [67]. The oxidation and removal of the grown silicon dioxide film smoothened the surface and improved the tensile strength [68]. However, the strength decreased because of the formation of etch pits during the oxide removal, where oxide precipitation formation occurred. Coating is another method to improve the strength by hindering crack formation and the crack enclosure effect of the internal stress of the coating film [48]. The plasma-enhanced chemical vapor deposition of an amorphous carbon film (a-C:H) improved the tensile strength significantly. The high compressive stress increased the strength and conformal coating by double-side deposition prevented the deformation and fracture of the structure.

Note that some of these methods improve the averages strength but not the deviation. When the dominant fracture mode is suppressed by these methods, another or other modes may become critical as the dominant parameters of the strength.

#### 3 Fatigue

Fatigue, which is a mechanical failure mode, is the weakening of a material caused by repetitive or cyclic loading. In brittle materials, one of the initially existing cracks, which dominate the strength of a material, propagates by the cyclic loadings and reaches to a certain critical size to cause failure. The fatigue failure of silicon microstructures has been observed widely in research experiments and commercial products, and the fatigue data are consistent with the empirical model.

## 3.1 Lifetime modeling

After the report by Connally and Brown [8], a number of experimental reports showed that failure occurred after a number of cycles of constant-amplitude cyclic loading [20,24,31,69–74], but there is no standard theory about its underlying mechanism. It can be explained from the experimental data that a defect, from where fracture initiation occurs, is propagated by the





cyclic loading and the growth is modeled with the well-known theory for brittle materials. However, it is almost impossible to observe the defect before the fracture initiation and there is no obvious evidence of defect propagation. In addition, silicon microstructures have relatively high resonant frequency because of their size, and more than ten billion cycles of loading might be applied during their operation.

The fatigue failure of silicon microstructures is understood as a slow crack growth induced by the constant or cyclic load, expressed by Paris' law [75]. The propagation of the flaw of size a at the number of cycles, N, is expressed as

$$\frac{da}{dN} = C(\Delta K)^n,\tag{4}$$

where  $\Delta K$  is the amplitude of the stress intensity factor and n and C are constants. The parameter n is called the fatigue exponent. When the fracture initiation flaw in a microstructure has the initial size of  $a_0$  and the applied cyclic stress amplitude of  $\sigma$ , the number of cycles to failure, N, is calculated as [76,77]

$$N - 1 = \frac{a_0}{C'} \frac{2}{2 - n} \left(\frac{\sigma}{\sigma_0}\right)^{-2} \left[1 - \left(\frac{\sigma}{\sigma_0}\right)^{2 - n}\right],\tag{5}$$

where  $\sigma_0$  is the initial strength and C' is a constant. An S-N curve obtained by solving the equation is drawn in Fig. 6 by plotting the test results from different methods [57,72,74,78–80] by normalizing with their initial strength [77]. There is an initial flat region and then the strength decreases with increasing the number of cycles. However, there are large deviations of the plots, and it is difficult to fit the plots to the theory, which is caused by the deviation of their initial strength. Ikehara and Tsuchiya [76] conducted a resonant vibration test using a fanshaped resonator made of single-crystal silicon. The fabrication process was carefully tuned to make the surface roughness small. By the amplitude-ramping and constant-amplitude tests, a wide range of fatigue plots from  $10^4$  to  $10^{10}$  cycles and the matching of fatigue characteristics to the theory in Eq. 5 were obtained, as shown in Fig. 7 [76].

Assuming high cycle loading and an applied stress smaller than the initial strength, the S-N relationship is simply described as

$$N\sigma^n = \text{const.}$$
 (6)

The equation corresponds to the straight decreasing line in the  $\log \sigma - N$  plot and the slope indicates the fatigue exponent. However, the initial strength  $\sigma_0$  shows scattering and statistical





analysis is required. By combining the Weibull distribution of strength and crack propagation law, we can obtain the following relationship:

$$\log(-\log(1-F))F = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^m \left(1 + \frac{n-2}{2}C''\sigma^2N\right)^{\frac{m}{n-2}}\right). \tag{7}$$

By fitting fatigue test results at several stress levels, the parameters required for reliability assessment are obtained. As an example, Fig. 8 shows the results of a resonant fatigue testing of a polycrystalline silicon membrane [81]. The membrane was exposed to different humidity conditions and the fatigue test was conducted at three stress levels. The specimens were ranked according to the number of cycles to fatigue failure and each cumulative fracture probability F was estimated using the median rank method. The results were fitted to Eq. 7 and both the Weibull and fatigue exponent parameters were obtained and are plotted as a function of humidity in Fig. 9. The Weibull modulus of the same thickness was constant against humidity, which indicated the same fracture mode. The fatigue exponent decreased with increasing ambient humidity and the silicon structure degraded more rapidly in a humid environment. These parameters are useful for predicting the device reliability with a certain safety factor.

#### 3.2 Mechanisms

There is no generalized mechanism that explains the fatigue failure of silicon. The experimental findings of previous studies on the fatigue of silicon microstructures are as follows.

- Strength weakening by cyclic loading: There is no fatigue limit.
- Environmental effect: In particular, there is a significant effect of humidity, and no fatigue failure of single-crystal silicon is observed in an ultra-high-vacuum environment [82]. However, polycrystalline silicon shows fatigue failure in the same environment [83].
- Stiffness change is observed as a resonant frequency shift during cyclic loading, but this is not associated with fatigue failure occurrence.
- The empirical crack propagation model (Paris' law) describes well the experimental results.

These findings suggest the following fatigue model: silicon reacts with water at the crack tip to form an oxide, which accelerates the growth of the crack. However, there is no direct evidence indicating that some existing cracks propagate during the fatigue test. The reason is that the initial crack is too small to be observed and identified. There have been many studies





to clarify the details of the fatigue process. Table II shows the proposed models of how the fatigue failure occurs in silicon microstructures.

Muhlstein et al. proposed the "Reaction layer fatigue" model in which the fatigue fracture of polycrystalline silicon is due to the interaction between surface oxidation and crack growth in the oxide film [84]. A native oxide film forms on the surface of polycrystalline silicon. Cracks initiate and grow while cyclic stress is applied to the native oxide film. Furthermore, the oxide film is thickened through the grown crack and this process recursively occurs to propagate the crack to its critical size. This model well explains the decrease in fatigue strength in the presence of water and oxygen, but there is no evidence indicating that the thickened oxide film observed grows through the proposed process.

Kahn et al. proposed "Mechanically induced subcritical cracking" [85]. In the bending fatigue test of polycrystalline silicon, it is shown that the fatigue life is shortened when the stress ratio is small (negative), that is, when the compressive stress is increased (Fig. 10). It is considered that when compressive stress is applied near small irregularities, damage due to fatigue accumulates and cracks grow. However, the effect of the environment and the occurrence of failure at a positive stress ratio cannot be explained fully with this theory.

Shrotriya et al. proposed the "Stress-assisted surface oxide dissolution" model in which the stable growth of cracks in the oxide film is the cause of fatigue fracture [86]. The change in the surface roughness of the test part in the fatigue test of polycrystalline silicon (the same device structure as that used by Muhlstein et al. [84]) is observed by atomic force microscopy (AFM), and the increase in surface roughness after the fatigue test is observed. Komai et al. observed a similar phenomenon in the underwater bending fatigue test of a single-crystal silicon cantilever [31]. The increase in surface roughness is considered the evidence of crack growth and the cause of fatigue fracture, but the growth model of the oxide film is not clear.

Kamiya et al. [87] observed the effect of hydrogen on silicon fatigue lifetime in the cyclic bending test in four different environments. Fatigue failure was observed only in lab air with 35-45% relative humidity (RH) and in the hydrogen environment, and no failure was observed in the oxygen and nitrogen environments. They hypothesized that hydrogen plays an important role in fatigue damage accumulation in silicon. Kamiya et al. also observed damage accumulation in silicon by electron beam-induced current (EBIC) imaging [88]. The silicon





beam specimen with a notch fabricated by DRIE was bent by cyclic load application. The notch tips had p-n junctions and the current generated by the electron beam was measured through the p-n junction. After bending for more than one million cycles at 50°C and 75%RH, island contrasts and line-shaped dark contrasts extending from the notch tips were observed: such contrasts are considered to indicate defect and slip generation, respectively.

## 4 Summary and Outlook

Silicon microstructures have been and will continue to be widely used as basic MEMS structures, and high reliability is desired as their application range expands. A large amount of testing data on fracture and fatigue has been collected, but there is no acceptable model. Therefore, a reliability design based on experimental data for individual device structures is still required. In device development, efforts should be made to prevent failure by designing for a high safety factor, packaging in vacuum or nitrogen atmosphere, or preventing surface reactions by surface treatment.

Furthermore, from the viewpoint of quality control, it is necessary to have a measurement method at the wafer level as a process monitor, which has been inevitably conducted for electrical characterizations. In recent years, data-driven material research has been widely conducted, and there is a need for research to develop appropriate methods for material reliability data and data collection, and analysis based on the collected data.

#### References

- [1] Smith C S 1954 Piezoresistance Effect in Germanium and Silicon *Phys. Rev.* **94** 42–9
- [2] Petersen K E 1982 Silicon as a mechanical material *Proc. Ieee* **70** 420–57
- [3] Noell W, Clerc P-, Jeanneret S, Hoogerwerf A, Niedermann P, Perret A and Rooij N F de 2004 MEMS for a watches 17th IEEE International Conference on Micro Electro Mechanical Systems. Maastricht MEMS 2004 Technical Digest pp 1–4
- [4] 2008 A New Heart [Internet]. [cited 2021 Mar 26]. Available from: https://www.omegawatches.com/chronicle/2008-a-new-heart





- [5] Chen C P and Leipold M H 1985 Stress Rate and Proof-Testing of Silicon Wafers J. Am. Ceram. Soc. 68 C-54-C-55
- [6] Mutoh M, Iyoda M, Fujita K, Mizuno C, Kondo M and Imai M 1990 Development of integrated semiconductor-type acceleration *Electronic Applications in Transportation*, 1990., IEEE Workshop on pp 35–8
- [7] Goto H, Ikeda M, Sakata M and Imanaka K 1996 Development of miniature two-dimensional optical scanner (2nd report, improvement of two-dimensional leaf spring resonator for low-voltage drive and wide scanning angle) Nippon Kikai Gakkai Ronbunshu C HenTransactions Jpn. Soc. Mech. Eng. Part C 62 3202-9
- [8] Connally J A and Brown S B 1992 Slow Crack Growth in Single-Crystal Silicon *Science* **256** 1537–9
- [9] Tabata O and Tsuchiya T 2006 Material properties: Measurement and data MEMS: A Practical Guide of Design, Analysis, and Applications pp 53–92
- [10] Orowan E 1949 Fracture and strength of solids *Rep. Prog. Phys.* **12** 185–232
- [11] Frenkel J 1926 Zur Theorie der Elastizitätsgrenze und der Festigkeit kristallinischer Körper Z. Für Phys. 37 572–609
- [12] Greek S, Ericson F, Johansson S and Schweitz J A 1997 In situ tensile strength measurement and Weibull analysis of thick film and thin film micromachined polysilicon structures *Thin Solid Films* **292** 247–54
- [13] Ericson F and Schweitz J 1990 Micromechanical fracture strength of silicon J. Appl. Phys. 68 5840–4
- [14] Weibull W 1951 A Statistical Distribution Function of Wide Applicability J. Appl. Mech. 18 293-7
- [15] Tsuchiya T, Tabata O, Sakata J and Taga Y 1998 Specimen size effect of tensile strength of surface-micromachined polycrystalline silicon thin films J. Microelectromechanical Syst. 7 106–13
- [16] Sato K, Yoshioka T, Ando T, Shikida M and Kawabata T 1998 Tensile testing of silicon film having different crystallographic orientations carried out on a silicon chip *Sens. Actuators -Phys.* **70** 148–52
- [17] Yi T C, Li L and Kim C J 2000 Microscale material testing of single crystalline silicon: process effects on surface morphology and tensile strength *Sens*. *Actuators -Phys.* **83** 172–8





- [18] Schweitz J-A and Ericson F 1999 Evaluation of mechanical materials properties by means of surface micromachined structures *Sens. Actuators Phys.* **74** 126–33
- [19] Tsuchiya T, Hirata M, Chiba N, Udo R, Yoshitomi Y, Ando T, Sato K, Takashima K, Higo Y, Saotome Y, Ogawa H and Ozaki K 2005 Cross comparison of thin-film tensile-testing methods examined using single-crystal silicon, polysilicon, nickel, and titanium films *J. Microelectromechanical Syst.* **14** 1178–86
- [20] Namazu T and Isono Y 2009 Fatigue Life Prediction Criterion for Micro-Nanoscale Single-Crystal Silicon Structures *J. Microelectromechanical Syst.* **18** 129–37
- [21] Sharpe W N, Turner K T and Edwards R L 1999 Tensile testing of polysilicon *Exp. Mech.* **39** 162–70
- [22] Koskinen J, Steinwall J E, Soave R and Johnson H H 1993 Microtensile testing of free-standing polysilicon fibers of various grain sizes J. Micromechanics Microengineering 3 13-7
- [23] Greek S, Ericsont F, Johansson S, Fürtsch M and Rump A 1999 Mechanical characterization of thick polysilicon films: Young's modulus and fracture strength evaluated with microstructures *J. Micromechanics Microengineering* 9 245–51
- [24] Kapels H, Aigner R and Binder J 2000 Fracture strength and fatigue of polysilicon determined by a novel thermal actuator *Ieee Trans. Electron Devices* 47 1522–8
- [25] Chasiotis I and Knauss W G 2003 The mechanical strength of polysilicon films: Part 1. The influence of fabrication governed surface conditions *J. Mech. Phys. Solids* **51** 1533-50
- [26] LaVan D A, Tsuchiya T, Coles G, Knauss W G, Chasiotis I and Read D 2001 Cross comparison of direct strength testing techniques on polysilicon films *ASTM Special Technical Publication* vol 1413, ed C Muhlstein and S B Brown (West Conshohocken: American Society for Testing and Materials) pp 16–27
- [27] Srikar V T and Spearing S M 2003 A critical review of microscale mechanical testing methods used in the design of microelectromechanical systems *Exp. Mech.* **43** 238–47
- [28] Tsuchiya T and Sakata J 1999 Tensile Testing of Insulating Thin Films Using Electrostatic Force Grip *IEEJ Trans. Sens. Micromachines* **119** 290–4
- [29] Ogawa H, Suzuki K, Kaneko S, Nakano Y, Ishikawa Y and Kitahara





- T 1997 Tensile testing of microfabricated thin films Microsyst. Technol. 3 117–21
- [30] Tai Y C and Muller R S 1988 Fracture strain of LPCVD polysilicon. pp 88-91
- [31] Komai K, Minoshima K and Inoue S 1998 Fracture and fatigue behavior of single crystal silicon microelements and nanoscopic AFM damage evaluation *Microsyst. Technol.* **5** 30–7
- [32] Johansson S, Schweitz J-A, Tenerz L and Tirén J 1988 Fracture testing of silicon microelements in situ in a scanning electron microscope *J. Appl. Phys.* **63** 4799–803
- [33] Wilson C J, Ormeggi A and Narbutovskih M 1996 Fracture testing of silicon microcantilever beams *J. Appl. Phys.* **79** 2386–93
- [34] Najafi K and Suzuki K 1989 Measurement of fracture stress, young's modulus, and intrinsic stress of heavily boron-doped silicon microstructures *Thin Solid Films* **181** 251–8
- [35] Gaspar J, Paul O, Chu V and Joao P C 2008 Mechanical Properties and Reliability of Amorphous vs. Polycrystalline Silicon Thin Films *MRS Online Proc. Libr.* **1066** 10661504
- [36] Tanemura T, Yamashita S, Wado H, Takeuchi Y, Tsuchiya T and Tabata O 2012 Fatigue Testing of Polycrystalline Silicon Thin-Film Membrane Using Out-of-Plane Bending Vibration *Jpn. J. Appl. Phys.* **51** 11PA02
- [37] Hajika R, Yoshida S, Makishi W, Kanamori Y, Tanaka S and Esashi M 2013 Mechanical strengthening of silicon torsion bar of MEMS scanning mirror by hydrogen anneal 2013 IEEE 26th International Conference on Micro Electro Mechanical Systems (MEMS) (IEEE) pp 425–8
- [38] Fujii T, Yamagiwa H, Inoue S and Namazu T 2013 Tension-torsion-bending combined loading test technique for the reliability of MEMS structures 2013 Transducers & Eurosensors XXVII: The 17th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS & EUROSENSORS XXVII) (IEEE) pp 1954-7
- [39] Namazu T, Yamagiwa H and Inoue S 2013 Tension-Torsion Combined Loading Test Equipment for a Minute Beam Specimen *J. Eng. Mater. Technol.-Trans. Asme* 135 011004
- [40] Rezazadeh G, Khatami F and Tahmasebi A 2007 Investigation of the torsion and bending effects on static stability of electrostatic torsional micromirrors *Microsyst. Technol.-Micro-Nanosyst.-Inf. Storage Process. Syst.* 13 715–22





- [41] Wolter A, Schenk H, Korth H and Lakner H 2004 Torsional stress, fatigue and fracture strength in silicon hinges of a micro scanning mirror *SPIE* 5343, Reliability, Testing, and Characterization of MEMS/ MOEMS III ed D M Tanner and R Ramesham pp 176–85
- [42] Zhang X M, Chau F S, Quan C, Lam Y L and Liu A Q 2001 A study of the static characteristics of a torsional micromirror *Sens. Actuators -Phys.* **90** 73–81
- [43] Zhang W, Obitani K, Hirai Y, Tsuchiya T and Tabata O 2019 Fracture strength of silicon torsional mirror resonators fully coated with submicrometer-thick PECVD DLC film *Sens. Actuators Phys.* **286** 28–34
- [44] Izumi S, Ping C W, Yamaguchi M, Sakai S, Suzuki A and Ueda Y 2005 Strength analysis of MEMS micromirror devices Effects of loading mode and etching damage *Advances in Fracture and Strength*, *Pts 1- 4* vol 297–300 (Zurich-Uetikon: Trans Tech Publications Ltd) pp 527–32
- [45] Tsuchiya T, SAKATA J and Taga Y 1998 Tensile strength and fracture toughness of surface micromachined polycrystalline silicon thin films prepared under various conditions *Materials Research Society Symposium Proceedings* vol 505 pp 285–90
- [46] Sharpe Jr. W N, Jackson K and Coles G 2000 Mechanical properties of different polyslilicons *Proc ASME MEMS* 1 255–9
- [47] Uesugi A, Tsuchiya T, Tabata O, Sugano K and Hirai Y 2015 Effect of crystallographic orientation on tensile fractures of (100) and (110) silicon microstructures fabricated from silicon-on-insulator wafers *Micro Nano Lett.* **10** 678–82
- [48] Johansson S, Ericson F and Schweitz J 1989 Influence of surface coatings on elasticity, residual stresses, and fracture properties of silicon microelements J. Appl. Phys. 65 122–8
- [49] Fitzgerald A M, Dauskardt R H and Kenny T W 2000 Fracture toughness and crack growth phenomena of plasma-etched single crystal silicon Sens. Actuators Phys. 83 194–9
- [50] Wilson C J and Beck P A 1996 Fracture testing of bulk silicon microcantilever beams subjected to a side load *J. Microelectromechanical Syst.* **5** 142–50
- [51] Pourahmadi F, Gee D and Petersen K 1991 The effect of corner radius of curvature on the mechanical strength of micromachined single-crystal silicon structures Transducers '91 pp 197–200





- [52] Walker J A, Gabriel K J and Mehregany M 1991 Mechanical integrity of polysilicon films exposed to hydrofluoric acid solutions *J. Electron. Mater.* **20** 665–70
- [53] Taylor T A and Barrett C R 1972 Creep and recovery of silicon single crystals *Mater. Sci. Eng.* **10** 93–102
- [54] Walters D S and Spearing S M 2000 On the flexural creep of single-crystal silicon *Scr. Mater.* **42** 769–74
- [55] Uesugi A, Yasutomi T, Hirai Y, Tsuchiya T and Tabata O 2015 High-temperature tensile testing machine for investigation of brittle-ductile transition behavior of single crystal silicon microstructure *Jpn. J. Appl. Phys.* **54** 06FP04
- [56] Nakao S, Ando T, Shikida M and Sato K 2006 Mechanical properties of a micron-sized SCS film in a high-temperature environment *J. Micromechanics Microengineering* **16** 715–20
- [57] Nakao S, Ando T, Shikida M and Sato K 2008 Effect of temperature on fracture toughness in a single-crystal-silicon film and transition in its fracture mode *J. Micromechanics Microengineering* **18** 015026
- [58] Ando T, Takumi T, Nozue S and Sato K 2011 Fracture toughness of si thin film at very low temperatures by tensile test 2011 IEEE 24th International Conference on Micro Electro Mechanical Systems 2011 IEEE 24th International Conference on Micro Electro Mechanical Systems pp 436–9
- [59] Namazu T, Isono Y and Tanaka T 2002 Plastic deformation of nanometric single crystal silicon wire in AFM bending test at intermediate temperatures *Microelectromechanical Syst. J. Of* 11 125–35
- [60] Han X D, Zheng K, Zhang Y F, Zhang X N, Zhang Z and Wang Z L 2007 Low-Temperature In Situ Large-Strain Plasticity of Silicon Nanowires *Adv. Mater.* **19** 2112–8
- [61] Jaya B N, Wheeler J M, Wehrs J, Best J P, Soler R, Michler J, Kirchlechner C and Dehm G 2016 Microscale Fracture Behavior of Single Crystal Silicon Beams at Elevated Temperatures *Nano Lett.* **16** 7597–603
- [62] Rabier J and Demenet J L 2000 Low Temperature, High Stress Plastic Deformation of Semiconductors: The Silicon Case *Phys. Status Solidi B* **222** 63–74
- [63] Rabier J 2007 High-stress plasticity and the core structures of dislocations in silicon *Phys. Status Solidi A* **204** 2248–55
  - [64] Shima K, Izumi S and Sakai S 2010 Reaction pathway analysis for





dislocation nucleation from a sharp corner in silicon: Glide set versus shuffle set *J. Appl. Phys.* **108** 063504

- [65] Mitwally M E, Tsuchiya T, Tabata O and Sedky S 2014 Improvement of tensile strength of freestanding single crystal silicon microstructures using localized harsh laser treatment *Jpn. J. Appl. Phys.* **53** 06JM03
- [66] Mitwally M E, Tsuchiya T, Tabata O and Sedky S 2016 Effect of localized KrF excimer laser treatment on fracture behaviors of freestanding <110> and <100> single crystal silicon beams *Microsyst. Technol.* 22 379–86
- [67] Shikida M, Hasegawa T, Hamaguchi K and Sato K 2013 Mechanical strengthening of Si cantilevers by chemical wet etching *Microsyst. Technol.-Micro-Nanosyst.-Inf. Storage Process. Syst.* **19** 547–53
- [68] Tsuchiya T, Miyamoto K, Sugano K and Tabata O 2016 Fracture behavior of single crystal silicon with thermal oxide layer *Eng. Fract. Mech.* **163** 523–32
- [69] Brown S B, Arsdell W van and Muhlstein C L 1997 Materials reliability in MEMS devices *Proceedings of International Solid State Sensors and Actuators Conference (Transducers '97)* vol 1 pp 591-3
- [70] Izumi S, Ping C W, Yamaguchi M, Sakai S, Ueda Y and Suzuki A 2005 Development of specimen and test method for strength analysis of MEMS micromirror *Eng. Fract. Mech.* **72** 2672–85
- [71] Ando T, Shikida M and Sato K 2001 Tensile-mode fatigue testing of silicon films as structural materials for MEMS Sens. Actuators Phys. 93 70-5
- [72] Kwak K, Otsu M and Takashima K 2010 Resonant Bending Fatigue Tests on Thin Films Sens. Mater. 22 51–9
- [73] Kapels H, Urscher J, Aigner R, Sattler R, Wachutka G and Binder J 1999 Measuring fracture strength and long-term stability of polysilicon with a novel surface-micromachined thermal actuator by electrical wafer-level testing *Eurosensors XIII* 379–82
- [74] Tsuchiya T, Yamaji Y, Sugano K and Tabata O 2010 Tensile and Tensile-Mode Fatigue Testing of Microscale Specimens in Constant Humidity Environment *Exp. Mech.* **50** 509–16
- [75] Paris P and Erdogan F 1963 A critical analysis of crack propagation laws J. Fluids Eng. Trans. ASME 85 528-33
- [76] Ikehara T and Tsuchiya T 2012 Low-cycle to ultrahigh-cycle fatigue lifetime measurement of single-crystal-silicon specimens using a microresonator





test device J. Microelectromechanical Syst. 21 830–9

- [77] Kamiya S, Tsuchiya T, Ikehara T, Sato K, Ando T, Namazu T and Takashima K 2011 Cross comparison of fatigue lifetime testing on silicon thin film specimens 2011 IEEE 24th International Conference on Micro Electro Mechanical Systems (IEEE) pp 404–7
- [78] Ikehara T and Tsuchiya T 2008 Crystal orientation dependence of fatigue characteristics in single crystal silicon measured using a rotating micro resonator 2008 IEEE 21st International Conference on Micro Electro Mechanical Systems (IEEE) pp 435-8
- [79] Kamiya S, Amaki S, Kawai T, Honda N, Ruther P, Gaspar J and Paul O 2008 Seamless interpretation of the strength and fatigue lifetime of polycrystalline silicon thin films *J. Micromechanics Microengineering* **18** 095023
- [80] Tanaka M, Goto D, Namazu T and Inoue S 2008 Temperature dependency on single crystal silicon fatigue life under cyclic stresses *Asia-Pacific Conference on Transducers and Micro-Nano Technology (APCOT 2008)* (Tainan, TAIWAN) p 1S87
- [81] Tanemura T, Yamashita S, Wado H, Takeuchi Y, Tsuchiya T and Tabata O 2013 Fatigue characteristics of polycrystalline silicon thin-film membrane and its dependence on humidity *J. Micromechanics Microengineering* 23 035032
- [82] Hong V A, Yoneoka S, Messana M W, Graham A B, Salvia J C, Branchflower T T, Ng E J and Kenny T W 2015 Fatigue experiments on single crystal silicon in an oxygen-free environment *J. Microelectromechanical Syst.* **24** 351–9
- [83] Alter A L, Flader I B, Chen Y, Ortiz L C, Shin D D and Kenny T W 2020 Characterization of Accelerated Fatigue in Thick Epi-Polysilicon Vacuum Encapsulated MEMS Resonators *J. Microelectromechanical Syst.* **29** 1483–92
- [84] Muhlstein C L, Brown S B and Ritchie R O 2001 High-cycle fatigue and durability of polycrystalline silicon thin films in ambient air *Sens. Actuators Phys.* **94** 177–88
- [85] Kahn H, Ballarini R, Bellante J J and Heuer A H 2002 Fatigue failure in polysilicon not due to simple stress corrosion cracking *Science* **298** 1215–8
- [86] Shrotriya P, Allameh S, Butterwick A, Brown S and Soboyejo W O 2002 Mechanisms of fatigue in polysilicon MEMS structures *Mater. Sci. Microelectromechanical Syst. Mems Devices Iv* **687** 29–34
- [87] Izumi H, Udhayakumar A and Kamiya S 2015 Hydrogen enhanced





mechanical fatigue in single crystal silicon Mater. Lett. 142 130-2

[88] Kamiya S, Kongo A, Sugiyama H and Izumi H 2018 Electronic imaging of subcritical defect accumulation in single crystal silicon under fatigue loading Sens. Actuators Phys. 279 705–11





# **Table captions**

Table I: Modes for fracture strength test of silicon microstructures.

Table II: Fatigue models of silicon microstructures.

# Figure captions

- Figure 1: Reported average tensile strength of single-crystal and polycrystalline silicon plotted as a function of side surface area.
- Figure 2: Side surface roughness and corner shape of silicon dry-etched by DRIE. Non-annealed (Fig. 6 of Ref. 37) and hydrogen annealed (Fig. 7 of Ref. 37). © 2013 IEEE. Reproduced, with permission, from [37].
- Figure 3: Size- and temperature- dependent fracture mode transition between brittle and ductile modes. Different color plot points indicate different testing modes.
- Figure 4: Scanning electron micrograph of the tensile specimen fractured at 500°C. [56] Reproduced from Ref. 56. © IOP Publishing Ltd. All rights reserved.
- Figure 5: Silicon projected along [101] showing two slip planes. A, B and C locations for perfect screw dislocations. [63] Reprinted from Ref. 63, Copyright (2007), with permission from John Wiley & Sons, Inc..
- Figure 6: Fatigue test results of silicon microstructures tested with different specimen and methods. The stress amplitude is normalized with the initial strength. [77] © [2011] IEEE. Reprinted, with permission, from Ref. 77.
- Figure 7: S-N plot of single-crystal silicon in-plane fan-shaped resonator. CA, constant-amplitude test; SRA, slow-ramping-amplitude test; and RRA, rapid-ramping-amplitude 23





test. [76] © [2012] IEEE. Reprinted, with permission, from Ref. 76.

Figure 8: Weibull plots of fatigue lifetime of polysilicon membrane in out-of-plane bending test. [81]. Reproduced from Ref. 81. © IOP Publishing Ltd. All rights reserved.

Figure 9: Weibull modulus and fatigue index of polysilicon membrane as a function of humidity.

[81]. Reproduced from Ref. 81. © IOP Publishing Ltd. All rights reserved.

Figure 10: Fatigue strength of polysilicon as a function of load ratio. [85] © [2002] The American Association for the Advancement of Science. Reproduced, with permission from Ref. 85.