

## Dynamic fatigue of silicon

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Received 3 November 2003; accepted 2 December 2003

### Abstract

Dynamic fatigue of silicon is a fairly recent area of materials research, having been detected for the first time in 1992. Within the past year, there have been several reports on silicon fatigue behavior describing novel phenomena and models for their origin. The findings include: an increase in high-cycle fatigue lifetime with a decrease in peak stress, the dependence of fatigue on the cyclic stress levels but not on the frequency of the cycle, and morphological changes in the surface silica during fatigue. Of the mechanisms proposed, the most likely candidate is one that involves mechanically induced damage in the silicon as a result of cyclic stresses.

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### 1. Introduction

As a brittle material at room temperature, silicon would not be expected to be susceptible to dynamic fatigue—delayed fracture under applied cyclic stresses. It is very difficult to test the fatigue resistance of bulk specimens of brittle materials. Under applied stress, brittle materials with uniform toughness fracture at the site of the highest stress concentration, which is the site of the “most dangerous” (largest) flaw. The strength-limiting flaws are invariably processing-induced and are randomly distributed, leading to scatter in measured strengths that can be modeled using Weibull statistics. Therefore, it is difficult to subject a bulk specimen to a stress level near its fracture strength, which is necessary for fatigue testing, since the strength of any individual specimen can vary significantly from the average. Also, bulk testing techniques generally require large increases in fatigue-induced crack lengths before they can be detected.

For all of these reasons, fatigue of silicon was unexpected when it was demonstrated in 1992 by Connally and Brown [\*1], using a specimen with micrometer-size dimensions. They created a pre-crack using indentation and applied cyclic stresses at the resonant frequency of

their specimen, about 12 kHz. Due to the small size of the specimen, and the claimed accuracy of their resonant frequency measurements, they reported crack extensions on the nanometer scale, and postulated crack growth and catastrophic fractures after cycling in humid air, but not in dry air. They claimed that the native surface silica (SiO<sub>2</sub>) layer on their silicon specimens underwent stress corrosion cracking in the humid ambient. (Stress corrosion of silica in humid air is well documented [2].) The cracking was presumed to expose fresh silicon deeper into the specimen, which subsequently formed a native oxide that underwent additional stress corrosion, and so on. However, Connally and Brown’s experimental technique was incapable of applying monotonic loads comparable to the cyclic loads achieved through resonance loading, and so they had no way to test their hypothesis under constant tensile stresses.

Recently, additional investigations have been reported on the fatigue of silicon. Like Connally and Brown, all of the researchers used specimens with micrometer-size dimensions. Both single crystal and polycrystalline silicon (polysilicon) have been examined. The inability to test specimens under both cyclic and monotonic loading is a limitation of several of the reported testing techniques.

### 2. Loading techniques

Silicon specimens used in fatigue experiments are invariably fabricated using silicon micromachining

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techniques that utilize silicon wafers as substrates. This technology allows two types of loading: external and integrated. External loading involves connecting the microfabricated specimens to a separate system that is used to apply forces. One simple external loading device is a nanoindenter. The tip of the nanoindenter was pushed downward on silicon cantilever beams [3,\*4] or clamped–clamped silicon beams [5], to generate bending stresses. A similar technique used a lateral-motion load cell to push on the end of a beam attached to both a torsional bar and a tensile bar, to create tensile stresses in the area of interest [\*6]. Another external loading technique used a piezoelectric-based load cell to pull on silicon tensile specimens [\*\*7,8,9]. Finally, the silicon substrate can be attached to a piezoelectric shaker; when this actuator vibrates at the resonance frequency of the silicon device, the device will experience large amplitudes of deflection and high stresses [10].

Since the silicon specimens are fabricated on silicon substrates, it is possible to integrate the specimens with wafer-level microactuators that can generate the fatigue stresses using only electrical connections. Most of these microactuators use electrostatic forces, created with applied voltages. Both comb-drive actuators (actuators that involve the capacitance increase that results from the increased overlap of two opposing sets of long straight fingers) [\*11,\*\*12,\*13,14,\*\*15,16,\*\*17,18–21,\*22,\*\*23,24–26] and parallel-plate actuators [27,28] have been used. Another integrated microactuator exploits the thermal expansion of current-induced locally heated silicon to generate the fatigue stresses [\*\*29].

In general, the use of external actuation makes the silicon specimen fabrication much simpler, since the integrated microactuator is absent. However, the problems of attachment and alignment of external loading sources cause these experimental procedures to be cumbersome. For fatigue testing, it is advantageous to stress at relatively high frequencies. This is possible using external piezoelectric-based actuators, and especially for integrated electrostatic actuation. Thermally induced loading suffers from long cooling times, which limit its testing frequencies to about 1 Hz [\*\*29]. As discussed above, it is also desirable that the loading system be able to apply monotonic as well as cyclic loads, be able to measure the monotonic strength of the specimens, and be able to test the specimens for long-term durability under constant stresses. Since the external loading schemes are relatively large, they all display this capability, except for the shaking technique. For the integrated microactuators, the thermally induced stresses are large enough to test the monotonic strength, but the thermal stability of the device may not be high enough for long-term testing under constant loads. Electrostatic actuators typically do not generate sufficient force to study monotonic fracture. However, electrostatic comb-drive actuators can be

made with enough comb fingers to cause both monotonic fracture at stress concentrations, as well as fatigue-induced fracture [\*11,\*\*12,\*13]. The disadvantage is that the device is relatively very large (about 2 mm×2 mm), which decreases the yield of useful devices on a wafer processed in a semiconductor “foundry.”

### 3. Single crystal silicon

Following from Connally and Brown [\*1], Mino-shima et al. [3] created initial notches in single crystal silicon cantilever beams using a focused ion beam. They applied zero/tension stress cycles ( $R = 0$ ) to notched and unnotched beams using a nanoindenter at 0.1 Hz. (For fatigue cycling, the load ratio,  $R$ , is the ratio of the minimum stress to the maximum stress in the cycle. Tension is taken as positive, compression as negative.) Fatiguing in air had no effect for up to  $5 \times 10^4$  cycles. When tested under water, the fatigue lives were shorter, and the number of cycles to failure decreased as the immersion time in water before the start of the test increased, from about  $3 \times 10^4$  cycles for an immersion time less than one hour to about  $7 \times 10^3$  cycles for an immersion time of 400 h. Fracture surfaces followed  $\{111\}$  planes. Li and Bhushan [5] used a nanoindenter to cyclically stress ( $R = 0$ ) clamped–clamped silicon beams. They found that the stiffness of the beams decreased with increasing cycles, which they interpreted as accumulated damage in the form of crack propagation. Their fracture surfaces revealed  $\{111\}$  steps. Ando et al. [\*6] used their external loading source to create a region of uniaxial tension, and reported a decrease in the cycles to failure with decreasing tensile strain. Tsuchiya et al. [10] used a piezoelectric resonator to vibrate their single crystal silicon specimens at a frequency of about 9 kHz, with full reversed loading ( $R = -1$ ). They found that cycling in humid air (78% relative humidity) led to failures in fewer cycles than cycling in dry air (11% relative humidity). Muhlstein et al. [\*\*15] tested notched single crystal silicon specimens with an integrated electrostatic comb-drive actuator at frequencies of 40 and 50 kHz ( $R = -1$ ). Their results showed increasing cycles to fracture with decreasing peak stresses, with lifetimes of  $10^9$ – $10^{11}$  cycles for peak stresses of 50% of the values required for essentially immediate fracture. They also detected a continuous decrease in resonant frequency with number of cycles, which they attributed to damage accumulation. (A decrease in resonant frequency corresponds to either a decrease in stiffness or an increase in mass.) The fracture surface of a specimen that failed in less than  $10^6$  cycles revealed multiple cracking on  $\{111\}$  planes, while the fracture surface of a specimen that took over  $10^9$  cycles to fail had a predominantly  $\{110\}$  crack path.

#### 4. Polysilicon

Muhlstein et al. [15,16,17,18–21] used the single crystal device design to fabricate electrostatically actuated devices from P-doped polysilicon and found results very similar to those obtained for single crystal silicon—lifetimes of  $10^{11}$  cycles for peak stresses of 65% of the values required for short-term fracture. Using transmission electron microscopy (TEM), they measured the thickness of the surface silica layers on their unstressed samples to be about 30 nm. (Typical native oxides on silicon are about 2 nm thick [30].) After fatigue cycling to failure, three different specimens revealed surface oxide thicknesses up to three times greater in the highly stressed regions than in the non-stressed regions; no heating occurred during the test. They also reported TEM studies of one specimen that was cyclically stressed but not to failure, which showed cracks in the surface oxide layer. Some of the polysilicon samples were treated with a self-assembled monolayer (SAM) coating during fabrication; this layer prevented surface oxide formation. Fatigue tests on these specimens revealed a smaller dependence of lifetime on the peak stress amplitude—lifetimes of  $10^9$  cycles for peak stresses 90% of the values required for essentially immediate fracture. However, the scatter in the data was large enough to call into question any dependence of lifetime on peak stresses, although Muhlstein et al. claimed that the lifetimes are essentially unaffected by cyclic stresses [17].

Van Arsdell and Brown [22] conducted fatigue tests on the same devices used by Muhlstein et al. [15, 16, 17, 18–21], except that a pre-crack was introduced before testing using indentation. They reported that the resonant frequency of the device decreased when fatigued in humid air (50% or 75% relative humidity), but remained unchanged when fatigued in dry air. They interpreted the data to indicate crack extension during cycling in humid air and reported apparent crack growth rates. Allameh and co-workers [23,24–26] also used the same device designs. Their experiments were mainly concerned with the evolution of the surface topography during fatigue. Though the device moves laterally, and the surface of highest stress occurs on the vertical sidewall, they were unable to investigate this surface using atomic force microscopy (AFM), and instead measured the morphology of the top surface next to the sidewall. They found that deep grooves developed on this surface during fatigue loading, with peak-to-valley heights increasing from about 20 nm before cycling to as high as 80 nm after cycling.

Kahn et al. [11,12,13,14] tested a similar polysilicon device, except that the electrostatic comb-drive actuator was much larger, and they used undoped polysilicon. (They sputtered a thin Pd layer onto the fabricated devices to allow sufficient conductivity for

electrostatic actuation.) They found a similar trend for decreasing fatigue lives with decreasing peak stresses ( $R = -1$ ) [13,14]. They also investigated delayed failures under monotonic tensile loads. For a load corresponding to 90% of the average monotonic strength, they found no delayed fractures in ten specimens after 3000 h in 90% relative humidity air. However, when specimens were thermally oxidized to form surface silicon dioxide layers ranging from 45 to 140 nm thick, delayed failures were observed in humid air within 200 h [11]. (They measured the native surface oxide layers on their specimens to be about 2 nm thick.) With the large forces possible with their actuator, they were also able to perform tests with varying  $R$ . They reported that for low-cycle fatigue tests—tests where the cyclic stress amplitude was slowly ramped up—the fatigue strength decreased by a factor of about 2.5 as  $R$  was decreased from 0.5 to  $-3$  [12]. The fracture surfaces revealed that the lower fatigue strength was accompanied by an increase in the size of the fracture-initiating flaw, which was interpreted as evidence of subcritical crack growth during cycling. They also found essentially identical fatigue strengths in air ( $10^5$  Pa) and in vacuum (10 Pa). However, for standard high-cycle fatigue tests (with a fixed  $R = -0.5$ ), the lifetimes in vacuum significantly exceeded those in air.

Kapels et al. [29] used a thermal expansion-based actuator to fatigue polysilicon specimens with cyclic tensile loads ( $R = 0$ ) at 1 Hz, and reported the typical increase in lifetime with decreasing peak stress. Bagdahn and co-workers [7,8,9] performed similar cyclic tensile tests ( $R = 0$ ) on polysilicon specimens, with the additional feature that they were able to vary the frequency of their loading cycle. For four different frequencies between 50 and 6000 Hz, they reported the same trend in increasing cycles to failure for decreasing peak stress. In fact, they combined their data with those of Muhlstein et al., Kahn et al., and Kapels et al. into a single plot of normalized peak stress versus fatigue lifetime and found that all the data followed the same curve. They concluded that fatigue lifetime depends only on the number of cycles, not on the total time or the frequency of the test, and fit the results to the equation

$$\sigma_f / \sigma_c = N_f^{-0.02} \quad (1)$$

where  $\sigma_f$  is the peak stress in the fatigue cycle,  $\sigma_c$  is the monotonic strength, and  $N_f$  is the number of cycles to failure. They also noted an increase in surface roughness of the top surface of their specimens in the vicinity of the fatigue fracture [7].

Millet et al. [27,28] used parallel-plate electrostatic actuators to subject polysilicon beams to cyclic torsional stresses, and noted microcrack formation in the vicinity of the anchor points where the polysilicon beam was thinner. The stresses generated in this test are complicated and make fatigue analysis difficult.

## 5. Fatigue mechanisms

Since Muhlstein et al. obtained qualitatively similar results for both single crystal silicon and polysilicon [\*\*15,\*\*17], it is likely that the origin of fatigue in these materials is similar. It is also clear, particularly in light of the analysis by Bagdahn and Sharpe [\*\*7], that undoped polysilicon with a 2 nm surface oxide and a sputtered Pd film behaves the same under cyclic loading as P-doped polysilicon with a 30 nm surface oxide. Therefore, it is reasonable to expect that any proposed fatigue mechanism for silicon must explain all of the observed phenomena. These phenomena include: an increase in high-cycle fatigue lifetime with a decrease in peak stress, a decrease in the resonant frequency of specimens during a fatigue test, a decrease in low-cycle fatigue strength with decreasing  $R$ , an increase in fracture-producing flaw size with decreasing fatigue strength, no dependence on testing ambient for low-cycle fatigue, dependence on ambient for high-cycle fatigue, and morphological changes including an apparent oxide thickness increase and surface roughness increase in the area of high stress. Three mechanisms have been proposed: stress-assisted surface oxide dissolution by Allameh et al. [\*\*23,24], reaction-layer fatigue by Muhlstein et al. [\*\*17], and mechanically induced subcritical cracking by Kahn et al. [\*\*12].

The stress-assisted surface oxide dissolution model [\*\*23,24] involves cyclic stress enhanced oxidation, followed by uneven “dissolution” of the surface oxide such that deep grooves are formed where the dissolution is fastest; these grooves are then sites for crack nucleation. One drawback of this model is that cyclic stress enhanced oxidation has never previously been observed. Though Allameh and co-workers [\*\*23,24] report that their specimens had an initial surface oxide of 2–4 nm, they used the same structures as Muhlstein et al. [\*\*17] who showed surface oxides of about 30 nm. (Their devices, as well as those of Bagdahn and co-workers [\*\*7,8,9] and Van Arsdell and Brown [\*\*22], were fabricated by the MEMSCAP (formerly JDS Uniphase, Cronos, and MCNC) Multi-User MEMS Processes (MUMPs) program.) Allameh and co-workers [\*\*23,24–26] used only AFM to investigate their devices, and inferred the oxide thicknesses from the AFM results. Still, to achieve the deep grooves reported by Allameh et al., oxide growth, oxide redistribution, or some other mechanism of morphological change must have occurred. Another complication with this model is that it is based on the stress-dependent surface reaction model of Yu and Suo [31], which involves monotonic stresses, not the cyclic stresses that Allameh et al. used exclusively. Also, this model has a time dependence, which Bagdahn and Sharpe [\*\*7] have shown is not a feature of silicon fatigue.

The reaction-layer fatigue model [\*\*17] also involves cyclic stress enhanced surface oxide thickening, which

then undergoes environmentally assisted stress corrosion cracking. The process repeats until a critical crack size is reached, and the silicon itself fractures catastrophically. Thus, the fatigue damage occurs only in the surface oxide. While stress corrosion has both a time dependence and a monotonic stress dependence, if the rate controlling step is the oxide thickening which depends only on the cyclic stresses, the time dependence of fatigue failure is avoided. To support their model, Muhlstein et al. [\*\*17] present TEM top view images showing surface layers that contain cracks and that are thicker in the regions of highest applied stress. However, using TEM to determine the thickness of a surface layer on a sidewall is both unconventional and quite difficult, since the microfabricated sidewalls of the polysilicon specimens are neither perfectly flat nor perfectly vertical. Also, this type of analysis cannot determine if the thickness of the surface oxide is uniform through the 2000 nm height of the polysilicon device, or if the observed thickness increase is due to redistribution of the initial oxide. Another concern with this model involves the SAM-coated results. Muhlstein et al. [\*\*17] report failures at  $10^7$  and  $10^8$  cycles (4.2 and 42 min), for specimens with peak stresses of 2.8 GPa. For these devices to fail via the reaction-layer mechanism the SAM must first break down, then a surface oxide of sufficient thickness (20 nm) must grow, followed by stress corrosion cracking of the oxide layer, all within the time span of a few minutes.

The mechanically induced subcritical cracking model states that subcritical cracking occurs in the silicon itself when subjected to cyclic loads, particularly cyclic loads with large compressive components [\*\*12]. Fatigue damage of bulk brittle materials under cyclic compression has been well established [32]. Thus, this model explains the fatigue results that depend on stress levels but not on ambient, while the models that involve surface oxide growth better explain the fatigue results that have an ambient dependence. To explain the ambient effects on high-cycle fatigue, Kahn et al. [\*\*12] postulated that in air, surface oxide formation on mechanically induced subcritical cracks caused wedging effects that increased the applied stress intensity at the crack tips. There is another explanation for enhanced fatigue behavior in humidity that does not involve chemical reactions with the environment. Increasing the relative humidity in operating air greatly reduces the amount of wear debris formed by contacting polysilicon components [\*\*33]. In fatigue of bulk ceramics, wear debris decreases crack growth by preventing crack closure and reducing the effective driving force for crack advance [34]. Similar wear debris formed in dry air or in vacuum could reduce the fatigue effects in polysilicon operated in these ambients. Therefore, the only feature of fatigue behavior not accounted for by this model is the surface roughness increase reported by Allameh and co-workers

[\*\*23,24] and Bagdahn and Sharpe [\*\*7]. It is possible that this phenomenon is restricted to silicon specimens with thick (30 nm) surface oxides, such as those used by these researchers, and that it is not a general feature of silicon fatigue.

To summarize the discussion on fatigue mechanisms, the stress-assisted surface oxide dissolution model [\*\*23,24] and the reaction-layer fatigue model [\*\*17] are based on phenomena (stress-assisted dissolution and stress corrosion, respectively) that are time-dependent. To adhere to the time independence established by Bagdahn and Sharpe [\*\*7], these two mechanisms must incorporate a rate-limiting step that is cyclic stress-dependent, namely, cyclic stress-induced surface oxidation. This phenomenon must depend only on the applied stresses, and not on time. It must also be active only for cyclic stresses and not for monotonic stresses. In addition, to reconcile the results of Kahn et al. [\*\*12] regarding essentially identical fatigue behavior in air ( $10^5$  Pa) and in vacuum (10 Pa) for low-cycle fatigue, the cyclic stress-induced oxidation must be independent of pressure, at least over four orders of magnitude. Also, as stated above, it must be very fast—20 nm must grow within 4.2 min at room temperature. It is also noted that there have been no confirming investigations on the effects of cyclic stresses on surface oxide thicknesses. Therefore, cyclic stress-induced surface oxidation seems a doubtful component of silicon fatigue.

On the other hand, mechanically induced subcritical cracking can rationalize all of the fatigue life data for silicon, and is therefore a more likely explanation for silicon fatigue. It is noted, however, that the exact mechanism of irreversible deformation leading to fatigue damage at the crack tip that results in subcritical crack growth is still in question. Presumably, stress concentrations at asperities or other surface flaws result in microcrack formation, irreversible shear deformation at grain boundaries, or irreversible pressure-induced silicon phase transformations.

## 6. Conclusions

Dynamic fatigue of silicon is an unexpected phenomenon that was first detected in 1992. The exact mechanisms for its occurrence are still not fully understood, but it is likely that mechanically induced damage plays a central role. Successful experiments have only been achieved using micrometer-sized specimens, and a number of important results have recently been reported. These include the fatigue of silicon specimens under varying loading and ambient conditions, and the behavior of the surface oxide layers on the silicon specimens. The fatigue behavior of silicon when tested in air is complicated by the chemical reactivity of silicon with oxygen and water vapor, and by the fact that

polysilicon fabricated in different facilities displays significantly different surface oxide thicknesses.

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The papers of particular interest have been highlighted as:

\* of special interest;

\*\* of very special interest.

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