# Mechanism of fatigue in micron-scale films of polycrystalline silicon for microelectromechanical systems

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Reported nearly a decade ago, cyclic fatigue failure in silicon thin films has remained a mystery. Silicon does not display the room-temperature plasticity or extrinsic toughening mechanisms necessary to cause fatigue in either ductile (e.g., metals) or brittle (e.g., ceramics and ordered intermetallics) materials. This letter presents experimental evidence for the cyclic fatigue of silicon via a conceptually different mechanism termed *reaction-layer fatigue*. Based on mechanical testing, electron microscopy, and self-assembled monolayers, we present direct observation of fatigue-crack initiation in polycrystalline silicon, the mechanism of crack initiation, and a method for altering fatigue damage accumulation. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1455142]

Over the past decade, microelectromechanical systems (MEMS) and the enabling technologies of surface micromachining have evolved from academic laboratory exercises to established commercial fabrication strategies. During this period of rapid innovation, a vast array of MEMS applications have emerged and many commercial products have entered the marketplace. The reliability of these products is critical for both their performance and safeguarding human life. Consequently, studies that characterize the failure modes that will ultimately dictate the long-term durability of MEMS are vital to this maturing field.

Fatigue, or the delayed failure of a material under cyclic loading conditions, is the most important and commonly encountered mode of failure in structural materials. After over a century of research, there are generally accepted mechanisms for the fatigue of ductile (e.g., metallic) and brittle (e.g., ceramic and ordered intermetallic) materials. Fatigue of ductile materials is generally attributed to cyclic plasticity involving dislocation motion that causes alternating blunting and resharpening of a pre-existing crack tip as it advances. In contrast, brittle materials invariably lack dislocation mobility at ambient temperatures; fatigue conversely occurs by cycle-dependent degradation of the (extrinsic) toughness of the material in the wake of the crack tip.[1] However, neither of these mechanisms appears to be active in silicon at ambient temperatures and thus to be relevant to fatigue of brittle silicon films.

Silicon is generally regarded as a prototypical brittle material; dislocation activity is generally not observed at low homologous temperatures and there is little evidence of alternative extrinsic toughening mechanisms, such as grain bridging or microcracking.[2] Moreover, silicon has not been found to be susceptible to environmentally induced cracking (e.g., stress corrosion cracking) in moist air or water.[3–5] *These observations strongly imply that silicon should not fatigue in air at room temperature*. Indeed, there has been no evidence to date of bulk silicon being susceptible to fatigue failure. However, thin films ($\sim$2 to 20 $\mu$m thick) of silicon are known to fail prematurely under cyclic fatigue loading in room-temperature air. First reported by Connally and Brown nearly a decade ago,[6] the fatigue of silicon has been confirmed in many studies by the present authors and others,[7–11] where it has been found that both mono and polycrystalline silicon films display failure stresses of approximately half their (single-cycle) fracture strength after fatigue lives in excess of $\sim 10^{11}$ cycles. Early studies established the importance of water vapor in fatigue-crack growth in silicon and speculations were made that the mechanism may be associated with static fatigue of the native silica layer.[6,11] However, there has been no direct experimental evidence to support any mechanism of silicon fatigue, by native oxide cracking (e.g., Refs. 6 and 11), room-temperature plasticity (e.g., Ref. 12), or any other phenomena. In this letter, we provide experimental evidence that establishes the mechanism of such silicon fatigue. Furthermore, this understanding is used to introduce a method that could be used to control fatigue damage in silicon-based MEMS devices.

If thin film silicon is fatigued at high enough stress amplitudes, cracks will initiate, grow, and the material will eventually fail. To characterize the mechanical properties of thin films such as these, an effective strategy is to manufacture the mechanical testing system directly from the material of interest. In our research, we have used a micron-scale fatigue characterization structure that is analogous to the specimen, load frame, and displacement transducer found in

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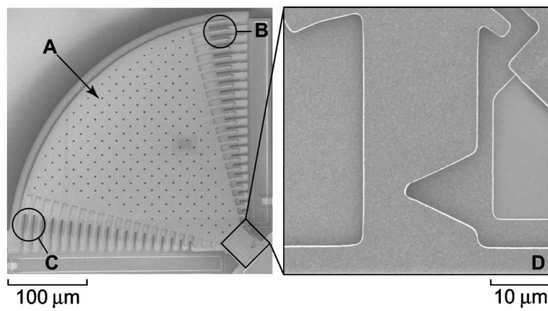


FIG. 1. Scanning electron micrographs of the fatigue life characterization structure. The (**A**) mass, (**B**) comb drive actuator, (**C**) capacitive displacement sensor, and (**D**) notched cantilever beam specimen (inset) are shown.

conventional mechanical testing systems (Fig. 1).[7–9] The notched cantilever beam specimen is subjected to fully reversed sinusoidal stresses at about 40 kHz as detailed in Refs. 8 and 9. The stress amplitude remains constant for a given test, but is varied across tests to characterize the behavior of the material. The natural frequency of the system is monitored; this frequency decreases as fatigue damage accumulates and eventually leads to failure of the specimen. If we plot the amplitude of the applied stress, $\sigma_a$, as a function of the number of cycles to failure, $N_f$, we generate what is referred to as a fatigue stress-life, or $S/N$, diagram, as shown for 2 $\mu$m thick films of polycrystalline silicon in Fig. 2.[9]

Our research suggests that fatigue of silicon thin films occurs through a process of sequential, mechanically induced oxidation and environmentally assisted cracking of the surface layer of material that forms upon reaction with the atmosphere (e.g., the $SiO_2$ "native oxide") that we term *reaction-layer fatigue*. This progressive accumulation of fatigue damage, depicted schematically in Fig. 3, was observed experimentally as a continuous decrease in the stiffness of the specimen during fatigue loading[9] and was visualized directly using high voltage transmission electron microscopy (HVTEM). In contrast to bulk ceramics where natural fatigue-crack initiation is not observed (all cracking initiates at pre-existing defects), reaction-layer fatigue is a process by which fatigue cracks can initiate in thin-film silicon. Initially, a native oxide forms on the exposed silicon surfaces, with thickness and composition dictated by the environment and

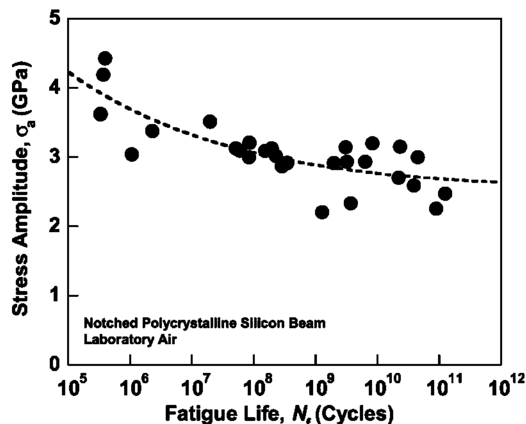


FIG. 2. Typical stress-life ($S/N$) fatigue behavior of the 2 $\mu$m thick, polycrystalline silicon at 40 kHz in moist room air under fully reversed, tension-compression loading (see Ref. 9). The dashed line is a fit of the stress-life power-law relation that is commonly applied to metals.

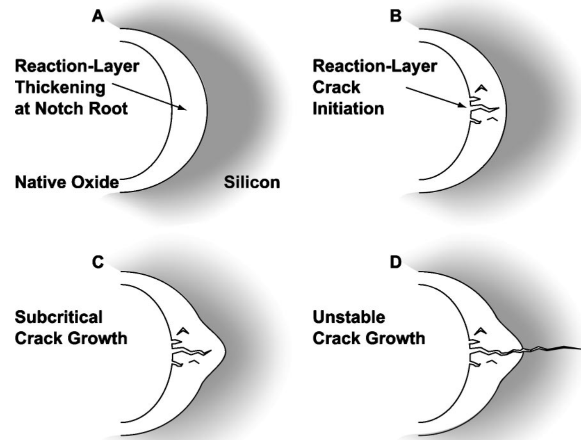


FIG. 3. Schematic of the reaction-layer fatigue mechanism at the notch of the polycrystalline silicon cantilever beam. (A) Localized oxide thickening at the notch root. (B) Environmentally assisted crack initiation in the native oxide at the notch root. (C) Additional thickening and cracking of reaction layer. (D) Unstable crack growth in the silicon film.

processing history of the thin film. In the case of the 2 $\mu$m thick, low-pressure chemical vapor deposited, phosphorous-doped ($n^+$-type) polycrystalline silicon used in this study, the initial oxide was about 30 nm thick. During subsequent fatigue loading, the oxide at the root of the notch thickened significantly. The oxide thickening process can cause up to a three-fold increase in amount of oxide at the notch, as documented using the HVTEM. Given the specimen heating usually observed in ultrasonic fatigue testing,[13] one might expect this phenomenon to be attributed to heating induced by the high frequency loading and to be further accelerated by the tensile stresses. However, high-resolution infrared imaging of the fatigue characterization structure showed that while the temperature of the resonating mass may have warmed slightly due to friction with the air during cycling, the silicon specimen remained within 1 K of ambient.[14] Thus, we believe that this cyclic oxide thickening process is *mechanical* in origin. Although the significance of cyclic loading is unclear, stress is already a key feature of silicon oxidation kinetics. The deformation of the $SiO_2$ reaction layer and the associated strain energy are thought to modify the diffusivity of oxygen and water and the kinetics of the interfacial reaction.[15,16] Cracks are then initiated in the thickened amorphous layer assisted by the well-known vulnerability of amorphous $SiO_2$ to stress-corrosion cracking (e.g., Ref. 17); indeed, these stable cracks were observed directly in the HVTEM (Fig. 4) by interrupting fatigue tests prior to failure. We believe that the fatigue of silicon proceeds via the propagation of these cracks by successive oxidation and environmentally assisted cracking of the reaction layer, until a critical crack size is reached when failure occurs in the underlying silicon structure. In addition to the experimental data and HVTEM observations, numerical models also support this reaction-layer fatigue mechanism. Finite element models of the fatigue characterization structure[9,14] show that the change in resonant frequency and crack lengths observed experimentally are consistent with each other. Given this understanding of the mechanism of silicon fatigue, it is now possible to pursue our ultimate objective of engineering sili-

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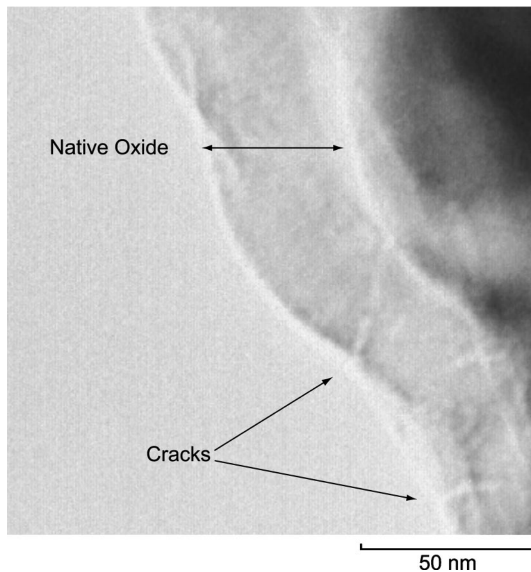


FIG. 4. HVTEM (800 keV) showing stable cracks in the native oxide of a notched, polycrystalline silicon beam during cyclic loading. Testing was interrupted after $N = 3.7\times10^9$ cycles at a stress amplitude, $\sigma_a$, of 2.58 GPa. Image intentionally defocused to facilitate the observation of the cracks.

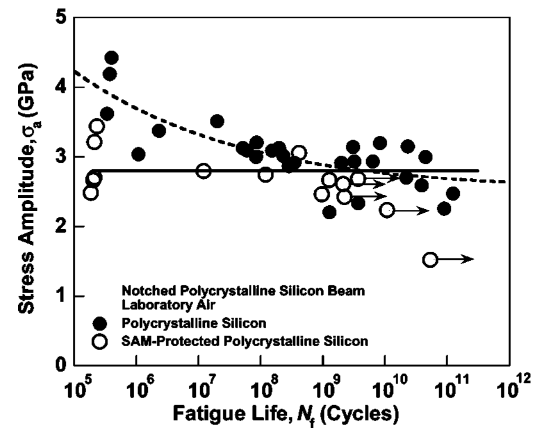


FIG. 5. Comparison of the fatigue behavior of uncoated and SAM-coated polycrystalline silicon thin films, showing the respective $S/N$ curves. Note the reduced susceptibility of the coated polysilicon films to fatigue failure compared to uncoated polysilicon. Horizontal arrows indicate samples that had not failed.

con films with superior resistance to fatigue failure.

The unique nature of the reaction-layer fatigue phenomenon permits a different approach to fatigue prevention. Since reaction-layer fatigue in silicon is confined to the native oxides, modification of the composition of this surface and/or its interaction with the environment should alter the fatigue resistance.

In order to test this theory, an alkene-based monolayer (1-octadecene, $C_{16}H_{33}CH{=}CH_2$) was applied to the polycrystalline silicon fatigue characterization structure before the native oxide formed. This was achieved by creating a hydrogen terminated surface using aqueous HF and applying the self-assembled monolayer (SAM) in a reactor containing a solution of one part 1-octadecene in nine parts hexadecane.[18] This hydrophobic SAM bonds directly to the (hydrogen-terminated) surface atoms of the silicon film (no oxide is allowed to form) and acts as an effective barrier to oxygen and water.[18] We hypothesized that if the silicon does, in fact, fatigue by the reaction-layer mechanism, preventing the formation and stress-corrosion cracking of the native oxide should suppress fatigue failures. Initial results on SAM-protected polycrystalline silicon confirmed that lifetimes were not affected by the applied cyclic stresses (Fig. 5). One should note, however, that there is an unexpected penalty for using the SAM for suppressing fatigue failures. Although the coated films do not appear to be as susceptible to premature failure by fatigue, their (single-cycle) fracture strength is degraded. For example, the average fracture strength of the SAM coated polysilicon was 2.6 GPa compared to 3.9 GPa for the uncoated polysilicon. It is believed that the native oxide smoothes the surface of the film, whereas its suppression leaves extremely thin, sharp features that lower the strength due to stress concentration.

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