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Mechanism of Fatigue in Micron-Scale Films of Polycrystalline Silicon for MEMS Applications

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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 paper 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 the first direct observation of fatigue-crack initiation in polycrystalline silicon, the mechanism of crack initiation, and a method for altering fatigue damage accumulation.

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. These applications range from memory, mass storage, and video displays for personal computing to sensors such as pressure transducers in medical devices and inertial sensors for passive restraint systems (e.g. airbags) and active automotive suspensions. The reliability of these components is

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critical for both product 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. Owing to the use of fabrication technologies learned from the microelectronics industry, silicon-based films are currently the dominant structural material for such micromachines. However, information on the long-term structural integrity of such micron-scale films is sadly lacking.

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. This understanding of material behavior and engineering fracture mechanics has allowed for the reliable design and operation of macro-scale structures such as aircraft, engines, buildings, orthopedic implants, etc. 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 have led researchers to believe 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, fatigue cracks are known to initiate and grow in silicon thin films (~ 2 to 20 μm thick) in room temperature air. First reported by Connally and Brown in this journal nearly a decade ago (*6*), the fatigue of silicon has been confirmed in many studies by the authors and others around the world (*7-13*); 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. Although static fatigue of the native silica layer has been suggested (*6*), until now the mechanism of fatigue in silicon thin films has essentially remained a mystery. In this paper, we provide experimental and theoretical 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 conventional mechanical testing systems (Fig. 1) (*7-11*). This structure is reminiscent of the geometry used in commercially available inertial sensors and filters and therefore provides insight into their durability and reliability. The fatigue specimen is a notched cantilever beam that is in turn attached to a large (300 μm

square) perforated plate that serves as a resonant mass. This simple harmonic oscillator is excited at resonance using electrostatic forces applied via one set of interdigitated "fingers," or a "comb drive," located on the edges of the mass. The opposing comb drive is used to measure the resulting motion. This tiny mechanical testing system applies large, fully reversed sinusoidal stresses at about 40 kHz to the material. 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, σ_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-μm thick films of polycrystalline silicon in Fig. 2 (*11*). Note how the films can fail at stresses approximately one half of their (single cycle) fracture stress after large numbers of fatigue cycles. This information can be used for component design and life prediction, and for evaluating different forms of the material.

Our research suggests that fatigue of silicon thin films occurs through a novel process of sequential, mechanically induced oxidation and environmentally-assisted cracking 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 (*11*) and was visualized directly using high voltage, transmission electron microscopy (HVTEM). Initially, a native oxide forms on the exposed silicon surfaces, with thickness and composition dictated by the environment and processing history of the thin film. In the case of the 2 μm thick, low pressure chemical vapor deposited (LPCVD), 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. 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 (Fig. 4). Thus we believe that this oxide thickening process is *mechanical* in origin. Cracks are then initiated in the thickened amorphous layer assisted by the well-known vulnerability of amorphous SiO_2 to stress-corrosion cracking (*14-16*); indeed, these stable cracks were observed directly in the HVTEM (Fig. 5) 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 (*11*) 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 silicon films with superior resistance to fatigue failure.

Resistance to fatigue is generally improved by carefully engineering material composition, microstructure, and surface condition (e.g., roughness and residual stresses). However, major improvements in fatigue performance by material/microstructural design are often quite modest as it is difficult, if not impossible, to target and suppress a specific fatigue mechanism in bulk materials. However, the unique nature of the reaction-layer fatigue phenomenon avails a novel approach. Since reaction-layer fatigue in silicon is confined to the native oxide, alteration 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 SAM in a reactor containing a solution of one part 1-octadecene in nine parts hexadecane (*17*). This hydrophobic, self-assembled monolayer (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 (*17*). We hypothesized that if the silicon does, in fact, fatigue by the reaction-layer mechanism, preventing formation and stress corrosion 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. Indeed, as anticipated, damage accumulation was dramatically altered and fatigue lives considerably extended in the coated samples (Fig. 6); this provides additional support for the proposed mechanism. 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). Such degradation in strength was not observed in previous testing of perfluorodecyltrichlorosilane (PFTS) and octadecyltrichlorosilane (OTS) SAM coatings on polysilicon (*18*). In principle, there are two methods for avoiding this problem. First, the processing of the film may be refined to improve its surface roughness. A more pragmatic approach, however, may be to use a hydrophobic layer on the native oxide. This would act to smooth the film surface through the presence of the initial oxide layer, yet at the same time restrict further access of the environment, thereby suppressing the formation of stress-corrosion cracks and hence inhibiting fatigue damage in the silicon film.

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Figures

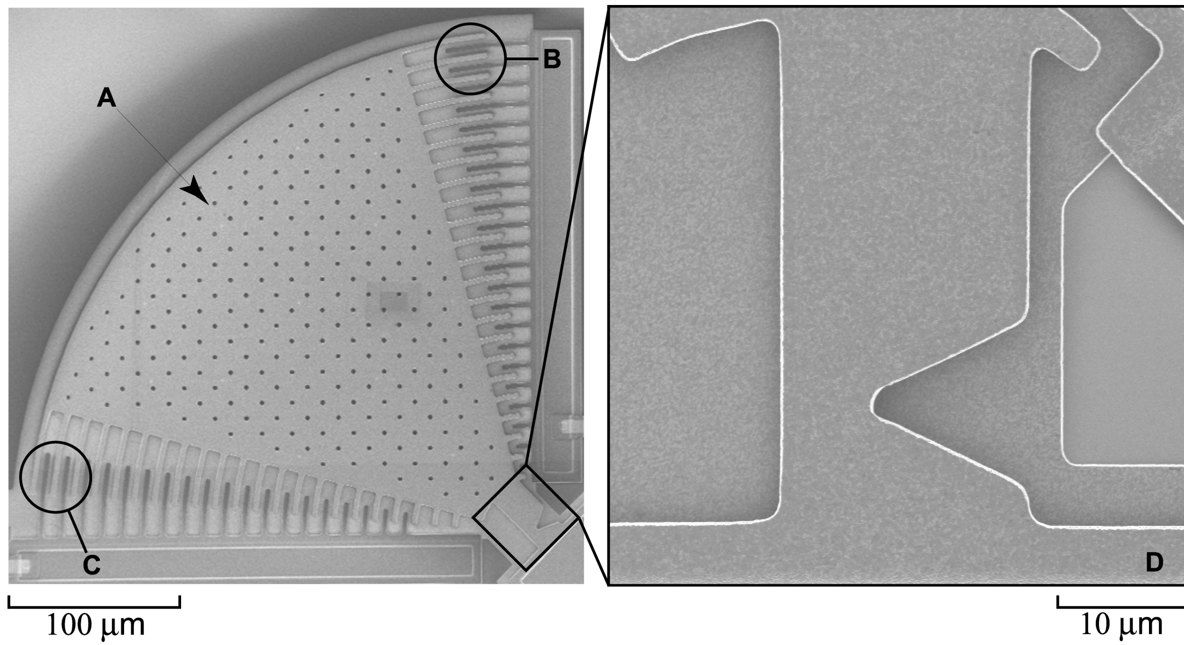

Figure 1 Scanning electron micrographs of the fatigue life characterization structure used in this investigation. The (**A**) mass, (**B**) comb drive actuator, (**C**) capacitive displacement sensor, and (**D**) notched cantilever beam specimen (inset) are shown. This 2 μm thick, surface micromachined structure was fabricated from LPCVD, n^+-type, polycrystalline silicon.

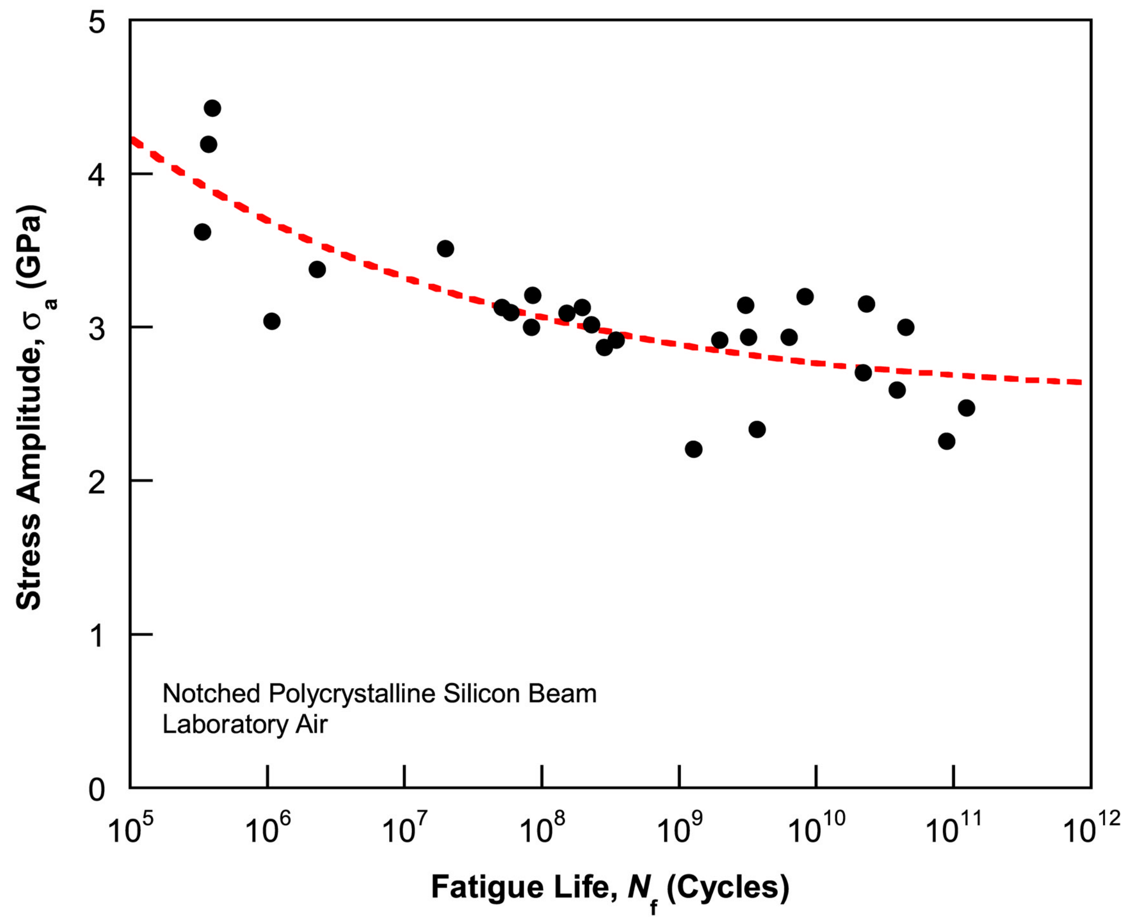

Figure 2 Typical stress-life (*S/N*) fatigue behavior of the 2 µm-thick, polycrystalline silicon at 40 kHz in moist room air under fully reversed, tension-compression loading (*9,11*). The dashed line is a fit of the stress-life power-law relation that is commonly applied to metals.

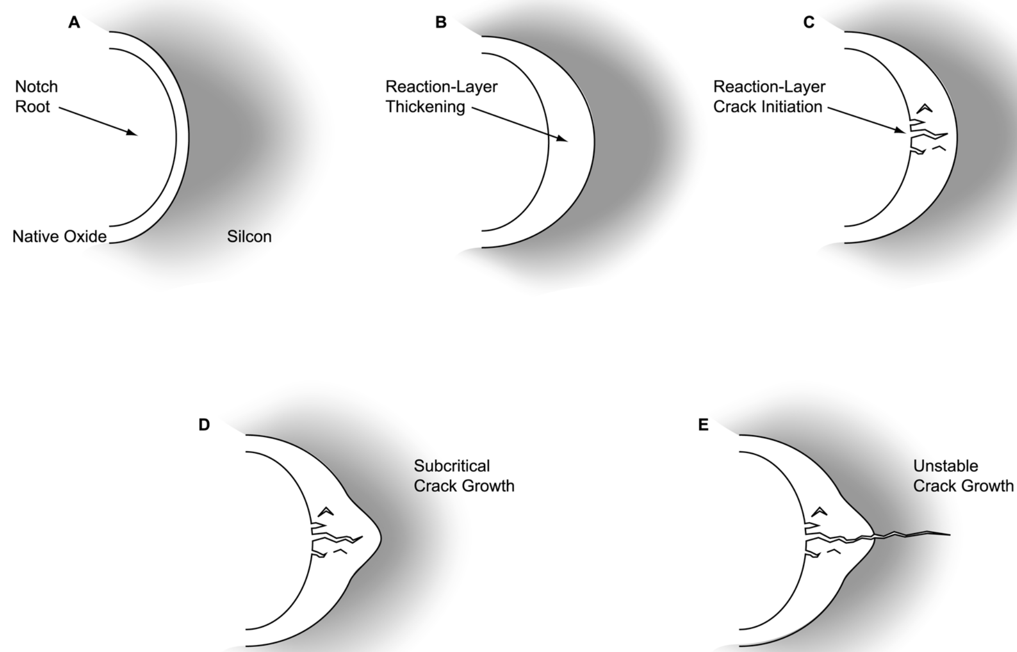

Figure 3 Schematic of the reaction-layer fatigue mechanism at the notch of the polycrystalline silicon cantilever beam. (**A**) Reaction layer (native oxide) on surface of the silicon. (**B**) Localized oxide thickening at the notch root. (**C**) environmentally-assisted crack initiation in the native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

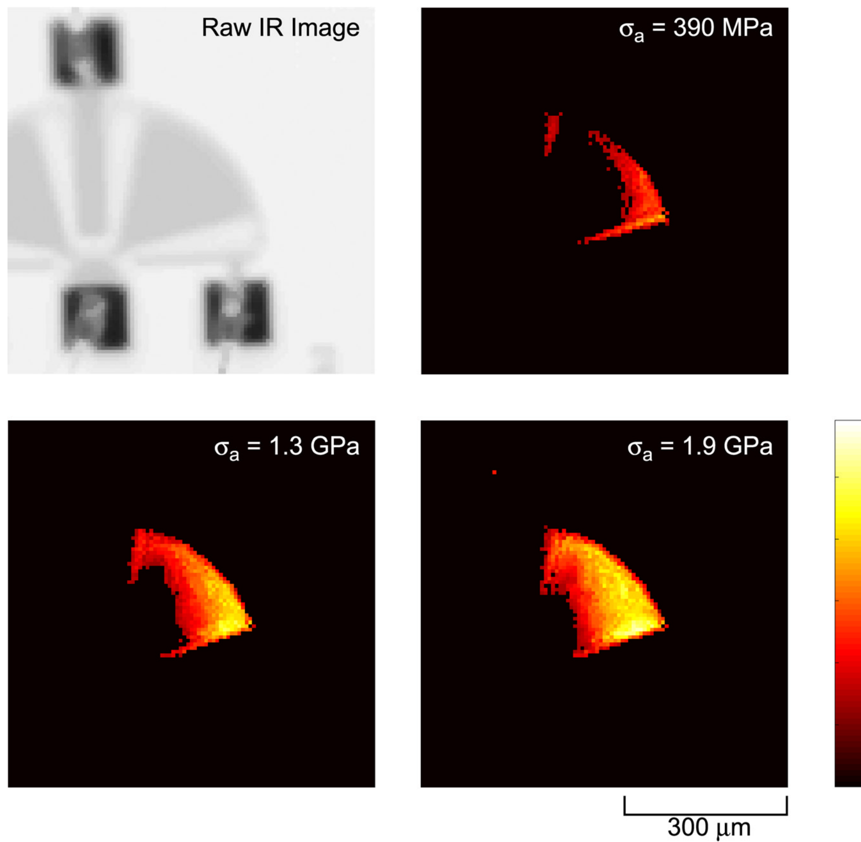

Figure 4 High resolution (20 mK) infrared (IR) images of the fatigue characterization structure. Each image was created by plotting the difference between IR images collected while the structure was resonated at the indicated stress amplitude, σ_a, and at rest. Individual IR images were collected by averaging over 2 seconds at an acquisition rate of 50 Hz. The absence of a difference in the IR images in the vicinity of the cantilever beam specimen indicates that the notch root oxidation process during reaction-layer fatigue is mechanical in origin. The slight warming of the mass is believed to originate from friction of the silicon with air as the structure resonates at ~40 kHz and has not been quantified. The vertical scale represents temperature changes less than 1 K.

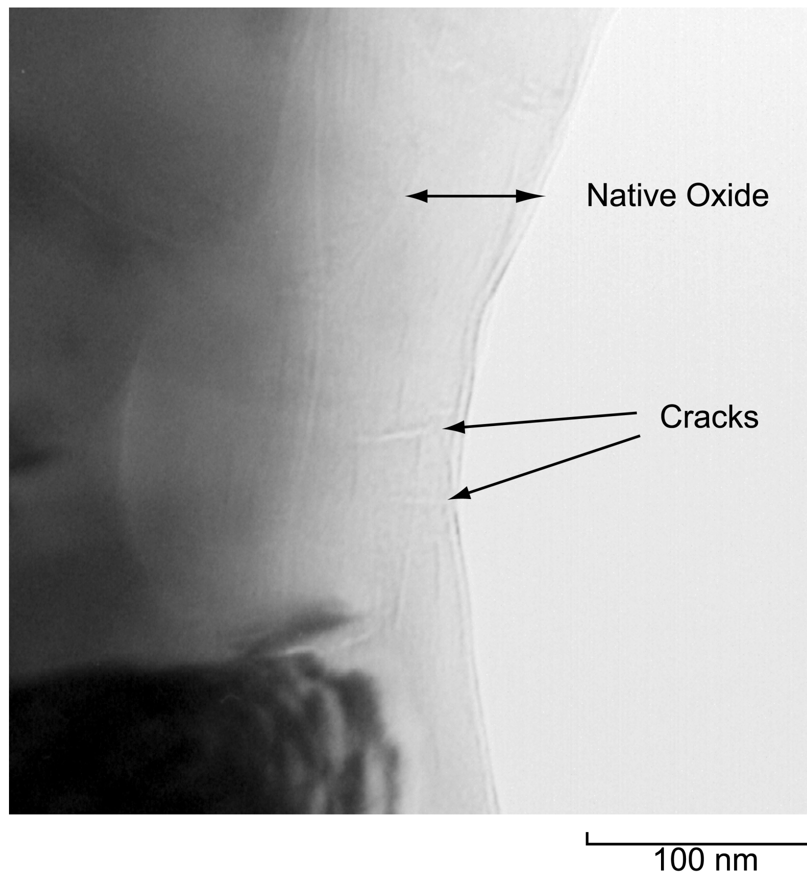

Figure 5 High voltage transmission electron micrograph (HVTEM) showing stable cracks in the native oxide of a notched, polycrystalline silicon beam formed during cyclic loading. Testing of this sample was interrupted after N = 3.56 x 10^9 cycles at a stress amplitude, σ_a, of 2.51 GPa. The micrograph was taken at 800 keV without thinning or other modification of the sample prior to imaging. The presence of cracks was confirmed by observation of the Fresnel fringes as the image was moved through focus, indicating an absence of material at the cracks. Furthermore, the size of the cracks is consistent with those predicted by numerical models of the experimentally observed damage accumulation. Prolonged exposure of the cracks to the 800 keV electron beam leads to healing of the cracks in a matter of minutes. This image was intentionally defocused to facilitate the observation of the cracks.

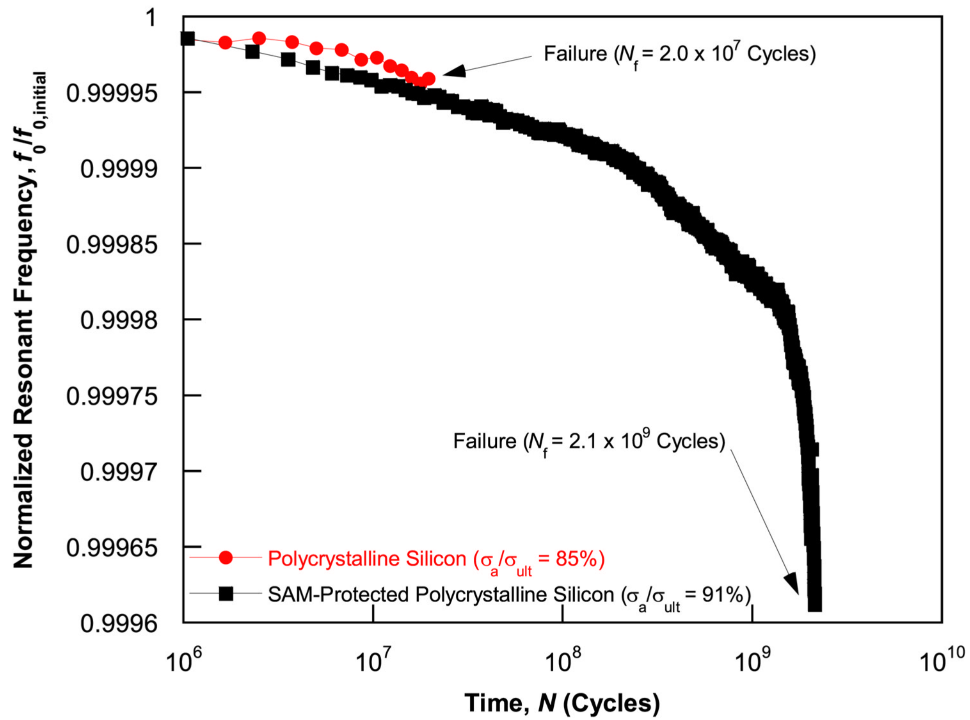

Figure 6 Damage accumulation in polycrystalline silicon and SAM-protected polycrystalline silicon thin films. The accumulated fatigue damage, i.e., notch-root oxidation and cracking, changes the natural frequency of the structure, f_0, as cycles, N, accumulate during the test. The accumulated damage with respect to the natural frequency of the structure at the start of the test, $f_{0,\mathrm{initial}}$, is shown as a function of cycles, N, for both SAM coated and uncoated samples. The fatigue life of the SAM-protected silicon was improved by two orders of magnitude compared to uncoated silicon, despite being subjected to more severe fatigue loading conditions.