INFLUENCE OF THE ENVIRONMENT AND ALUMINA COATINGS ON THE FATIGUE DEGRADATION OF POLYCRYSTALLINE SILICON FILMS

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INFLUENCE OF THE ENVIRONMENT AND ALUMINA COATINGS ON THE FATIGUE DEGRADATION OF POLYCRYSTALLINE SILICON FILMS

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SUMMARY

Previous studies on very high-cycle fatigue behavior of thin silicon films suggest a strong environmental dependence of the degradation mechanism, the precise nature of which is still subject to debate. In the present study, 2-micron-thick polycrystalline silicon (polysilicon) notched cantilever beam structures were used to investigate fatigue degradation in a high-temperature (80°C), high-humidity (90%RH) environment. The specimens were subjected to fully reversed sinusoidal loading at resonance (\(\sim 40kHZ\)) with stress amplitudes ranging from 1.46 to 1.6GPa, resulting in life-spans between \(10^6\) and \(10^9\) cycles. Comparison to a reference set of S-N data obtained at moderate environmental conditions (30°C and 50%RH) reveals a strong tendency for faster degradation with increasing temperature and humidity. The critical influence of the environment is also unambiguously captured via the continuous monitoring of the change in resonant frequency, resulting from a change in compliance of the structure. The obtained damage accumulation rates in the 80°C, 90%RH environment exceed the reference by two orders of magnitude and tend to increase towards failure, as opposed to decreasing rates at 30°C, 50%RH. Transmission electron microscopy (TEM) on vertical through-thickness slices reveals oxide thickening in the highly stressed region of run-out specimens. The presence of highly localized, thick oxides along the specimens thickness explains the previously published contradicting data obtained from TEM imaging of horizontal slices.

The influence of \(\sim 20\text{nm }\text{Al}_2\text{O}_3\) deposited on the surface of the fatigue specimens using Atomic Layer Deposition (ALD) technique was also studied. The presence of the ALD-alumina coating results in a higher fatigue resistance at 30°C and 50%RH, as well as a drastically different frequency evolution behavior. The resonant frequency of
the coated devices remains constant after a fast initial decrease. This can potentially be exploited to increase the performance of resonator-based MEMS sensor devices. In contrast to the uncoated devices, no oxide thickening was observed in the TEM for coated run-out specimens. A simple model is proposed to explain the different degradation behavior of the ALD-alumina coated samples.

The TEM study provides convincing evidence for stress-assisted oxidation, although the nature of the mechanism remains elusive. Thickened oxides after cycling appear consistent with the reaction-layer fatigue mechanism. Finite element modal analysis incorporating surface oxide layers and full-thickness as well as localized cracking was employed to relate the damage observed in electron microscopy to the experimentally measured changes in resonant frequency. In conclusion, while the reaction-layer mechanism seems capable of describing the very high cycle fatigue behavior of polysilicon thin films, the details of the underlying nanometer-scale processes and their kinetics remain to be clearly established.
CHAPTER I

INTRODUCTION

1.1 Polysilicon as structural material for MEMS

The roots of microelectromechanical systems (MEMS) lie in the field of micro-electronics. Hence, it is only natural that the fabrication of most MEMS devices is based on the well established lithographic processes for silicon. This is particularly true for surface micro-machined devices, where the fabrication process involves deposition and patterning of polycrystalline silicon (hereafter polysilicon). Examples of surface micro-machined devices can be seen in Fig. 1. Regardless of the function of these MEMS devices (sensor, actuator), polysilicon is used as a structural material and may be subjected to various types of loading (transient, cyclic) during the lifetime of the device.

Apart from being readily available, micron-scale polysilicon in the thin film form exhibits excellent mechanical strength properties to recommend it for a structural material. Yet, it is very brittle and shows very low resistance to crack propagation due to the absence of extrinsic toughening mechanisms [12, 34]. MEMS designers must therefore optimize the device geometry to minimize the maximum stresses within the polysilicon structural film.

For long, fast fracture has been the only studied failure mode when addressing reliability issues for Si-based MEMS devices. Polysilicon thin films have indeed been considered immune to fatigue failure, based on the bulk mechanical properties of Si [3]. However, experimental evidence shows this assumption to be wrong for micro-machined silicon [3, 29]. In the prospect of MEMS devices serving as sensors in critical applications in the automotive industry or defense, where they are subject to periodic
loading, this aspect requires further investigation.

1.2 Reliability of Si-based MEMS devices - ”harsh” environments

The development of MEMS technologies (and sensors in particular) is driven mainly
by the needs of the automotive, defense and medical industries [26]. Apart from the
low price and small dimensions it is mainly their intrinsic capability of withstanding
harsh environments that makes MEMS attractive. 'Harsh environments' summarize
extreme conditions such as high temperature, humidity, vibration level, radiation or

Examples include accelerometers used in automotive airbag and ESP\(^1\) systems
[10] that have to function reliably in a wide range of temperatures and humidities,
smart ammunition sensors that have to withstand extreme gun-shock [26], biological
and blood-pressure sensors that have to function in liquid environments [61] or
accelerometers used in down hole drilling that experience extreme temperatures and

\(^{1}\)Electronic Stability Programme
vibrations [26]. High humidity is only crucial for devices that can not be hermetically packaged, because it is their purpose to interact with the environment, like in the case of chemical or biological sensors.

Most of these applications are critical, since failure may cause harm to human life. Hence, reliability of the structural materials used, i.e. mainly polysilicon, in the environments of interest must be assessed. Since sensors like pressure sensors, accelerometers and resonator-based biosensors accumulate a large number of cycles in a relatively short time, fatigue degradation in harsh environments is one of the main issues to address. To date, most of the fatigue data on polysilicon films have been generated in regular laboratory conditions [3, 29].

1.3 ALD coating for MEMS

As MEMS devices are advancing into harsh and/or sensitive environments, the necessity to control the interaction at the surface arises, or explicitly, the necessity for protective surface coatings.

'Harsh' in this context refers not only to what is mentioned in section 1.2, but also to situations with direct mechanical contact that might cause stiction, wear or simply electrical failure. Sensitive environments are found mostly in the field of BioMEMS, where devices need to operate in vivo.

Self-assembling monolayer techniques have been used to deposit anti-stiction coatings [41], and chemical vapor deposition has been employed to deposit protective surface coatings during the fabrication process. However, the first technique does not allow for variations in thickness, while the second approach requires relatively high deposition temperatures and lacks conformality.

Atomic layer deposition (ALD) is capable of depositing atomic-thin, conformal films with sub-nanometer thickness control. The process relies on a sequence of self-limiting binary surface reactions involving precursor gases, which deposit the
film atomic layer by atomic layer. For $\text{Al}_2\text{O}_3$ this process can be performed above 35°C [23], which allows for low-temperature depositions thus circumventing problems caused by thermal mismatch in composite materials. More details on the underlying process-chemistry and setup will be provided in 2.2.4.

The $\text{Al}_2\text{O}_3$ ALD coatings considered in this work are currently used in MEMS industry to prevent electrical shorting and reduce in-use stiction due to decreased surface energy.

### 1.4 Fatigue testing structures

Fatigue testing of bulk materials usually involves dedicated testing machines for external actuation and sensing. This approach is also possible for micro-machined silicon and was applied in [7, 22, 62]. One possible actuation device is the nano-indenteter; it can be used to apply a controlled displacement to a cantilever beam in order to generate bending stresses. Other possible actuation devices are piezo-electric actuators or loudspeakers [7]. External actuation requires the attachment as well as alignment of the actuator before every test, which might be rather cumbersome.

Taking advantage of the MEMS fabrication process, on-chip testing devices with integrated actuation and sensing capabilities can be fabricated. Electrostatic forces or thermally induced strains are used for actuation. Typically, electrostatically driven devices employ comb-drive actuators (arrays of interdigitated fingers that form a capacitor) for driving and in some cases motion sensing; less often, parallel plate electrostatic actuators are used [43, 44]. The resonating structure shown in Fig. 2a that was first introduced by Van Arsdell and Brown [66] features a (here notched) cantilever beam attached to a large perforated mass with actuation and sensing comb-drives. Since the electrostatic forces are often not large enough to achieve sufficient motion amplitudes away from resonance, these devices are usually driven at their natural frequency. This allows for a very fast cycle accumulation, while the loading
ratio\(^2\) is limited to \(R = -1\). Combdrives large enough for static loading can be build; however, the devices become relatively large - and expensive - with the typical linear dimension being 2mm (see Fig. 2b).

The frequency range covered by thermal actuators is limited by the cooling time to about 1Hz [36].

### 1.5 Existing fatigue data for polysilicon films

The literature on fatigue testing of polysilicon thin films was recently reviewed in [3, 29, 62]. An overview is given in the following, while the main results are summarized in section 1.6.2.

Van Arsdell and Brown [66] were the first to study fatigue of polysilicon thin films. They used an un-notched but pre-cracked version of the device type depicted in Fig. 2a made from 2\(\mu\)m-thick P-doped polysilicon, with the natural frequency being \(\sim 45\)kHz. Tests conducted in wet air showed decreasing resonant frequency over time, while tests in dry air did not. Control-samples without pre-crack tested in wet air displayed no change in resonant frequency. The decrease in resonant frequency was monitored and interpreted as damage accumulation in the form of crack extension.

Kahn et al. [28, 29, 30, 31] tested electrostatically actuated pre-cracked fracture mechanics specimens made from undoped Pd-coated 3\(\mu\)m-thick polysilicon. They observed increasing fatigue lives for decreasing stress amplitudes at \(R = -1\). Tests conducted in medium vacuum (10Pa) showed significantly prolonged lifetimes, while the fracture strength was found to be the same in both environments. However, low-cycle fatigue strength was found to increase with increasing loading ratio \(R\).

Kahn et al. also tested pre-cracked polysilicon beams in monotonic tension and found them not to exhibit any delayed failure [28]. In a more recent work they report that intentionally oxidized fracture mechanics specimens show fatigue under

---

\(^2\)The loading ratio is defined as the ratio of the minimal and the maximal applied stress \(R = \sigma_{\text{min}}/\sigma_{\text{max}}\)
(a) Structure for cyclic testing at resonance. Resonant frequency $\sim 40\text{kHz}$.

(b) MEMS structure for monotonic or cyclic testing (from [27])

Figure 2: Electrostatically actuated fatigue characterization structures
monotonic loading if the surface oxide thickness exceeds 45nm [27]. The same kind of
device (not intentionally oxidized) was cyclically fatigued using a maximum applied
stress of 3.6GPa and a loading ratio $R = -0.5$ at 7.6kHz; failure occurred after $2 \cdot 10^7$
cycles. A horizontal slice adjacent to the fracture was thinned using focused ion beam
(FIB) and viewed in a transmission electron microscope (TEM). The surface oxide
was measured to be $\sim 3$nm and no local oxide thickening in the highly stressed region
was found.

Kapels et al. [36] used thermally actuated silicon beams (4μm-thick). The actua-
tion frequency was 1Hz and the loading ratio $R = 0$. They also report increasing
fatigue lives with decreasing stress amplitudes.

Muhlstein et al. [46, 47, 48, 49] used the same device type as van Arsdell and
Brown but did not introduce pre-cracks. Instead the cantilever beam is notched to
produce a stress concentration. Tests were conducted in laboratory air and high vac-
umum at $\sim 40$kHz. A noticeable decrease in resonant frequency (as large as 50Hz)
was recorded when testing in air. Here as well, the fatigue life increased when the
applied stress amplitude was decreased. No delayed failure was reported in vac-
umum. High-voltage transmission electron microscopy (HVTEM) of control samples,
fatigued samples and samples interrupted prior to failure revealed a difference in ox-
ide thickness. The oxide at the notch root of control samples was measured to be
$\sim 30$nm, while fatigued samples showed oxides of up to 90nm. Interrupted samples
show thickened oxide layers and subcritical cracks within the oxide. [3]

Pierron and Muhlstein [54] tested the same devices in controlled environment
and measured the resonant frequency evolution. When switching the humidity from
50%RH to 25%RH at 30°C a falloff in the decrease rate of the resonant frequency was
observed; analogously, the rate increased when going back to 50%RH. The authors
concluded the degradation mechanism to be dependent on the humidity level. High-
spatial-resolution depth-dependent AES scans revealed thickened oxides (7-10nm)
Figure 3: Cumulative stress-life data for polysilicon films at room temperature. The Sharpe-data refers to 'dog-bone' shaped tensile specimens that exhibit lower strength due to an increase in highly-stressed volume.
after cycling in the areas that experienced high applied stress, while no thick oxide
was reported for a manually overloaded control sample.

Alsem et al. [5, 6] used the same device type as Muhlstein et al. and also showed
delayed failure in laboratory air, but not in vacuum. Devices fabricated in the MUMPs
and SUMMIT V™ processes with initial oxide thicknesses of ∼4nm (SUMMIT V™)
and ∼20nm (MUMPs) were tested. EFTEM oxygen maps show oxide thickening after
cycling in air but not in vacuum and no oxide thickening after single-cycle fracture
for devices from both foundries.

Allameh et al. [1] investigated the surface evolution during cycling using the
same device as Muhlstein. Atomic force microscopy (AFM) was used to measure
the evolution of the top surface of the beam near the notch, since the actual notch
root was inaccessible to AFM investigation. Roughening of the surface due to cyclic
loading was reported; peak-to-valley heights increased from ∼ 20nm before cycling to
about 80nm after cycling.

Bagdahn and Sharpe [7, 62] tested 3.5µm-thick tensile specimens externally ac-
tuated at 50Hz, 200Hz, 1kHz and 6kHz with a loading ratio $R = 0$. Delayed failure
was reported with fatigue lives independent of the actuation frequency.

In a recent study Boroch, Bagdahn et al. [10] tested structures similar to Fig. 2a
featuring an un-notched, 10µm-thick cantilever beam attached to a perforated mass
driven at its resonant frequency of ∼ 90kHz. Tests were conducted in low vacuum at
a pressure of approximately 100-200Pa. They report delayed fracture after cycling as
well as an increase in fracture strength after pre-cycling by up to 700MPa (18% of
the initial strength).

Cumulative S-N data is presented in Fig. 3. Note that the data was digitized
from plots in the respective publications.
1.6 Fatigue mechanisms

1.6.1 Bulk silicon

As a solid with covalently bonded diamond structure, bulk silicon is believed not to be susceptible to mechanical fatigue. This belief is supported by experimental studies not showing any time or cycle dependent crack extension under tension/compression loading in either air or water environments [14, 17, 40, 59] and atomistic simulations showing no susceptibility to stress corrosion cracking [69].

However, recent results for bulk single crystal silicon indicate its ability to undergo crystalline-amorphous as well as amorphous-crystalline phase transformations [70] and 'fatigue' under cyclic indentation loading. The mechanical response to cyclic indentation in air and water was found to differ [71], indicating an environmental influence; the authors suggest this behavior to be due to infringement of oxygen in the transformation zone when in aqueous environment, but do not exclude a different mechanism.

Bhowmick et al. [9] find ring cracks developing in single crystal silicon under cycling indentation loading at what was determined to be less than one-half the critical load after about $10^3$ cycles. Scanning electron microscopy revealed platelets and particles being dislodged from the crack walls and ejected on the surface to further the degradation. The authors attribute crack nucleation to damage from micro-contacts and continuing degradation to frictional sliding of the crack faces subjected to a non-negligible shear stress. The same mechanism is suggested to work for micromachined silicon. However, it can be argued [2] that the completely different loading geometry makes this case "an entirely distinct phenomenon of questionable relevance to the fatigue cracking of thin films of silicon in bending or tension/compression loading conditions".
1.6.2 Polysilicon thin films

Fatigue of silicon thin films has been known for over 15 years; however, its origin is still subject to debate. To the present date two explanations dominate the literature: the mechanism proposed by Kahn et al. assumes subcritical crack growth in silicon itself, assisted through wedging by asperities on the crack faces [28] and a form of grain boundary plasticity [31]. The explanation proposed by Muhlstein et al. [48] involves stress-assisted oxide thickening and moisture-induced subcritical cracking therein. This mechanism is referred to as 'reaction-layer fatigue'.

A successful mechanism should consistently explain the experimental evidence detailed out in section 1.5. Summarizing, the main effects and observations are:

1. Thin silicon films exhibit fatigue in bending and tension/compression loading, while bulk silicon does not (e.g. [2])

2. Both single crystal- as well as polysilicon exhibit fatigue\(^3\) (e.g. [3])

3. The fatigue degradation depends on the environment [2, 53]\(^4\) and is faster in high-humidity environments [53]

4. No fatigue in high vacuum [2, 5, 53] (results on fatigue in medium vacuum presented in [10, 28])

5. Thick oxides in the highly stressed region were observed after cycling [48, 49] (independent verification in [6]; contrary results presented in [27])

6. No frequency dependence of the fatigue damage was observed [7]

7. No fatigue under monotonic loading [28]

---

\(^3\)This study is concerned with polysilicon thin films. However, both single crystal- as well as polysilicon exhibit very similar fatigue properties. This is not very surprising, since the critical flaw size (e.g. [27]) is typically smaller than the grain size (more on grain sizes in section 3.2). Therefore, a unifying mechanism appears feasible.

\(^4\)Study [53] deals with single crystal silicon.
8. Fatigue after as low as $10^6$ cycles in moderate environments observed (see [3] or Fig. 3)

9. The low cycle fatigue strength is strongly dependent on the loading ratio $R$ [28]

10. Strengthening effect after pre-cycling [10, 31]

None of the two approaches is capable of explaining all of the points listed above.

Observations (3), (4) and (5) clearly support the environmental explanation. Sub-critical crack growth in silica would also explain the absence of bulk fatigue under the conditions mentioned in (1); crack length would always be limited by the oxide thickness, i.e. the crack would never reach the critical size for the whole structure necessary to induce catastrophic failure. Fatigue of thin silicon films is typically observed for stress amplitudes between 2 and 5GPa; the corresponding range of oxide thicknesses necessary to activate the reaction layer fatigue mechanism was calculated to be between 15 and 75nm [56]. Problems occur trying to explain the lack of frequency dependence (6) and absence of fatigue under monotonic tension (7), i.e. the absence of the time dependence present in typical stress corrosion cracking [67]. However, it can be argued [2] that the rate limiting factor may lie in the stress-assisted oxide thickening, which, so it seems, is a function of the number of cycles rather than time. Still, Alsem, Muhlstein et al. are at a loss to explain the observed strengthening effect (10). Also, it is unlikely that thick enough oxide layers could form at room temperature in $10^6$ cycles or less, assuming an initial native oxide of 2-3nm [56].

The initial oxide thickness is crucial for the reaction-layer mechanism. Pierron et al. [56] show that oxide-layers as thin as 15nm are sufficient to activate the mechanism, if the interfacial failure-mode is considered. However, the typical native oxide

---

5 Subcritical crack growth is more extensive at lower $R$ values
6 Pierron et al. do not account for compressive residual stresses in the $SiO_2$ in their interfacial fracture mechanics analysis. Therefore, the values given should be understood as approximations.
thickness for silicon is considered to be 2-3nm, thus necessitating oxide thickening prior to the activation of reaction-layer mechanism, which is unlikely for low-cycle fatigue.

Larger initial oxide thicknesses of 15-30nm have been observed [4, 48]; the current understanding of this phenomenon is galvanic oxidation of silicon during the HF-release process due to the presence of gold on the chip [35, 55].

Supporting evidence for the mechanical damage-mechanism is found in (6), (7), (8), (9) and (10). These observations indicate a loading ratio and cycle dependent mechanism. Indeed, a grain-boundary plasticity model implemented into a finite element analysis [31] showed residual compressive stresses at the notch that would lead to an increase in strength upon subsequent loading. However, this model cannot explain fatigue of single crystal silicon.

The asperity "wedging" model for crack extension in silicon proposed by Kahn et al. [28] follows the ideas of Suresh for brittle solids [64] claiming a "cantilever effect" during the compressive part of the cycle. This idea was recently revisited by Pierron et al. [57]; using a FE-model for wedged cracks they show that for fully reversed loading the maximal driving force for crack advance is reached during the tensile part of the cycle. Hence, the wedging effect is unlikely to explain the observed fatigue effects. Moreover, fatigue of silicon thin films is observed under tension-tension loading conditions ($R > 0$) [28, 29, 30, 31, 59].

It has to be emphasized that, while stress corrosion cracking of $SiO_2$ is relatively well understood [42], no specific physical mechanisms have been suggested to explain either stress assisted oxide thickening nor subcritical cracking in silicon. As Kahn et al. [27] put it: "The mechanism(s) responsible for silicon fatigue will remain a disputed topic until a comprehensive explanation is developed."
1.7 Motivation for study

It can be inferred from the literature survey that one of the core issues in understanding fatigue of silicon thin films lies in the understanding of the role of the environment. Regarding the reaction layer model in particular, this means understanding the rate-limiting step of the process. However, no conclusive study on degradation rates in high-humidity environments, which should favor oxidation, exists in the literature.

Another issue involves the clarification of the existence or nonexistence of thickened oxide layers after cycling, since contradicting data is presented [27]. It is true that the HVTEM approach to evaluating oxide thickness is unconventional and potentially problematic, since it gives no way of determining the degree of localization of the surface oxide and is more prone to focusing, edge and tilting artifacts due to the large sample thickness.

The FIB sample fabrication chosen by Kahn et al. [27] and Alsem et al. [4] simplifies oxide thickness determinations due to the smaller thickness of the sample. However, by choosing a horizontal slice the authors implicitly assume homogeneity of the surface layer, since every slice is subject to essentially the same stress. This assumption seems not justified, since SEM analysis of the fracture surfaces indicates localized defects [28, 54], i.e. highly localized oxides might be sufficient to create a critical flaw.

The present work intends to address these issues by conducting tests with the same kind of specimen geometry in a moderate reference environment (30°C, 50%RH)\(^7\) as well as a harsh environment (80°C, 90%RH)\(^8\) and record, evaluate and compare damage accumulation rates accessible from the change in resonant frequency during testing. Also, TEM investigations of vertical slices taken at the notch root, i.e. the

\(^{7}\)RH \(\equiv \) Relative Humidity  
\(^{8}\)When increasing the temperature from 30°C to 80°C the vapor pressure of water changes from 4.243kPa to 47.34kPa. The ratio in partial pressures between the two environments is 20.08.
region with the highest applied stress, of a control specimen and specimen fatigued (and interrupted) in each of the testing environments are carried out. The degree of localization of eventually existing post-cycling oxides should become accessible using this sample geometry.

As described in part 1.3, ALD coatings present an attractive way of improving the mechanical and electrical properties of MEMS devices. However, to date, no studies on their effect on reliability are available in the literature. The present work intends to fill this gap and present data on the fatigue behavior of polysilicon films coated with ALD-deposited alumina.

The alumina layer will prevent the silicon surface from interaction with the environment. Hence, if the reaction-layer fatigue mechanism is correct, the fatigue behavior should change drastically. The obtained fatigue lives and degradation rates as well as TEM investigations of fatigued devices should give further insight in the mechanisms underlying the fatigue of micro-machined silicon.
CHAPTER II

EXPERIMENTAL / NUMERICAL PROCEDURES

The present chapter includes details on the fatigue characterization device used in this study as well as the associated laboratory setup and testing protocols. Also, a full description of the electron imaging techniques employed for damage characterization and information on sample preparation is given.

Finally, a finite element (FE) model of a pre-cracked fatigue resonator is introduced and detailed out.

2.1 Testing principles

A mechanical structure (see Fig. 4) is driven at resonance by application of an electrostatic force. The motion results in a periodic change in capacitance causing an induced current. This current is proportional to the amplitude of motion and is experimentally measured as an output voltage. The resonant frequency is periodically assessed by sweeping over a frequency range and adjusted at the maximal amplitude of output current, corresponding to the maximal amplitude of motion.

2.2 Description of Fatigue Characterization Structures

2.2.1 Geometry

The 2µm-thick fatigue characterization structure shown in Fig. 4 is comprised of a notched cantilever beam attached to a fan-shaped mass. This type of device was first introduced by van Arsdell and Brown [66] and since then successfully used in various studies on fatigue of thin films [1, 5, 6, 45, 47, 48, 49].

The design incorporates two arrays of interdigitated finger-pairs referred to as comb-drives, one of which is used for electrostatic actuation at resonance while the
Figure 4: Fatigue characterization device overview and close-up of the notch. The out-of-plane thickness is 2\(\mu\)m.

Other provides on-chip electrical motion-sensing capabilities.

A sinusoidal electrostatic force is applied to excite the first in-plane bending mode of the structure with the center of the remaining ligament being the center of rotation (see Figs. 4 and 5).

Reasons for the success of this device are its high linearity over a wide range of amplitudes, the well defined stress concentration at the notch and the excellent control of the testing frequency via the geometry of the mass. However, it can only be actuated at resonance, which limits the loading ratio to \(R = -1\).

2.2.2 Fabrication Process

The devices used in this study were fabricated in the polysilicon surface micromachining process PolyMUMPs\textsuperscript{TM} (run 78) by MEMSCAP. This process encompasses eight lithography levels to create seven physical layers. A cross-sectional overview over the present device is given in Fig. 5b. The numbers below refer to this figure.

The wafer (1) is heavily doped with phosphorus to minimize charge build-up at the silicon nitride layer (2) deposited to isolate the electrical surface layers from
Figure 5: Schematic of the fatigue characterization structure (see text for the labeling of the layers). Layer 5 (2μm polysilicon) is the structural layer.

The substrate. Then, an initial polysilicon layer (3) (POLY0) is deposited using Chemical Vapor Deposition (CVD) and patterned with Reactive Ion Etching (RIE). The first 2μm-oxide layer (4) is deposited using low temperature CVD and patterned and etched with a combination of RIE and Buffered Oxide Etch (BOE). In this patterning step holes in the oxide are created that will later become stand-offs to prevent the plate from sticking when the oxide is removed. A 2μm-polysilicon layer (5) (POLY1) is deposited, doped with phosphorus and patterned; this layer includes the plate as well as the comb-drives. The plate is perforated with 2μm holes, referred to as release holes, to expedite the etching process of the oxide underneath. A last oxide layer (0.75μm) is deposited and etched, followed by a final polysilicon layer (6) (POLY2). In a last step 20nm of Cr (7) and 500nm of Au (8) are deposited using e-beam evaporation.

To prevent damage during transportation the fatigue characterization devices are not free-standing when delivered. Hence, before testing the sacrificial oxide layer (see 4 in Fig. 5b) needs to be removed. This procedure encompasses several steps.

Firstly, the dies have to be cleaned from remaining photo resist and fabrication
debris:

1. acetone bath (10min)
2. acetone bath (10min)
3. IPA rinse
4. visual inspection (using an optical microscope)

Secondly, the sacrificial oxide is etched in 49% hydrofluoric (HF), which is then washed off in DI water:

5. HF etch (49% HF, 2.5min)
6. DI water bath (10min)
7. DI water bath (5min)

Then, water is replaced with IPA:

8. IPA bath (5min)
9. IPA bath (25min)

Finally, the dies are dried at the critical point using the Tousimis Autosamdri-815B supercritical drier.

2.2.3 Supercritical drying

During a conventional drying process a liquid-gas interface establishes, which gives rise to capillary forces large enough to pull a free-standing film down to the substrate. Since the vertical stiffness of thin films is very small, van-der-Waals interaction between the film and the substrate will cause the device to adhere and lose functionality. This is referred to as stiction.
Supercritical drying is used to circumvent stiction. This process goes around the liquid-gas transition by going beyond the critical point, which for $CO_2$ is $73.9\text{bar}$ at $31.1\degree\text{C}$ (see Fig. 6). No liquid-gas interface and therefore no capillary forces are present.

During the drying process the supercritical dryer cycles through several steps:

1. The chamber containing the sample submerged in IPA is cooled to $-5\degree\text{C}$

In the next three steps IPA is replaced by liquid $CO_2$:

2. The chamber is filled with liquid $CO_2$ for 8min

3. IPA is purged out of the chamber for 15min

4. The chamber is refilled with liquid $CO_2$ for an additional 4min

Now, the drying process begins:

5. The pressure is raised to $93.1\text{bar}\pm5\%$ and the chamber heated to $34\degree\text{C}$

6. This state is maintained for 4min
7. The $CO_2$ is bleeded out

8. When the pressure reaches 25-27.5bar the chamber is vented

### 2.2.4 ALD coating

To study the influence of coatings on the fatigue behavior of polysilicon films in different environments, an atomic thin layer of alumina was deposited on selected dies using the atomic layer deposition (ALD) technique. The process relies on a binary reaction sequence using the gas precursors trimethylaluminum ($Al(CH_3)_3$) and water vapor ($H_2O$)

\[
A : \quad AlOH^* + Al(CH_3)_3 \rightarrow AlOAl(CH_3)_2^* + CH_4 \quad (1)
\]

\[
B : \quad AlCH_3^* + H_2O \rightarrow AlOH^* + CH_4 \quad (2)
\]

where $^*$ designates the surface species. The surface functionality is changed in each half-reaction. Exposure to a precursor-gas results in a self-limiting reaction with the surface, thus forming the reactant for the next half-reaction. More information on the underlying chemistry can be found in [19] and [39].

One cycle $AB$ deposits one atomic layer of $Al_2O_3$. The film thickness can be controlled by the number of cycles in the sequence $ABAB\ldots$ with an expected 1.4Å-increment per cycle (at 100°C) [21]. For depositions at this temperature tensile residual stresses of $\sim 500$MPa are reported in the ALD-alumina layer [58].

An in-house ALD tool was used to deposit 100 cycles of alumina at 100°C. The layer thickness was controlled in SEM (Fig. 7a) and TEM (Fig. 7b) and found to be 23nm, indicating a lower density of the deposited material.
2.3 Dynamic Behavior of Fatigue Structures

2.3.1 Governing Equations

The employed fatigue testing structure is by design a linear oscillator, the motion of which is governed by the following equation

\[ J\ddot{\theta} + b\dot{\theta} + k\theta = f(t), \tag{3} \]

where
\( \theta \) is the angle of rotation,
\( J \) the moment of inertia,
\( b \) the damping constant,
\( k \) the stiffness and
\( f \) the generalized electrostatic force.

The expression for the generalized electrostatic force (which in this case can be interpreted as moment of force) is easily found as the derivative of the electrostatic
energy with respect to the angular displacement

\[ f = \frac{\partial}{\partial \theta} \left( \frac{1}{2} CV^2 \right) = \frac{1}{2} \frac{\partial C}{\partial \theta} V^2 \]  

(4)

with \( C \) being the capacitance of one comb-drive and \( V \) the applied driving voltage.

An applied sinusoidal voltage \( V = \hat{V} \sin \frac{\omega}{2} t \) at half the natural frequency \( \omega = \sqrt{\frac{k}{J}} \) will result in a periodic moment

\[ f = \frac{1}{2} \frac{\partial C}{\partial \theta} \hat{V}^2 \sin^2 \frac{\omega}{2} t = \frac{1}{2} \frac{\partial C}{\partial \theta} \hat{V}^2 \left( \frac{1}{2} - \frac{1}{2} \cos \omega t \right) \]  

(5)

at the natural frequency. This is often referred to as frequency doubling.

The change in capacitance with respect to angular rotation is given by the sum over all comb-pairs

\[ \frac{\partial C}{\partial \theta} = \varepsilon h \sum_{j=1}^{32} \frac{1}{\ln(r_{o,j}/r_{i,j})} \]  

(6)

with \( \varepsilon \) being the dielectric permittivity, \( h \) the electrical thickness of the structure and \( r_{i,j} \) and \( r_{o,j} \) the inner and outer radii of the \( j \)th capacitor.

Equation (3) has the well known solution for the amplitude at resonance\(^1\)

\[ \hat{\theta} = \frac{\hat{f}}{b \omega_0} \]  

(7)

where \( \hat{f} = \frac{1}{4} \frac{\partial C}{\partial \theta} \hat{V}^2 \) is the forcing amplitude and \( \omega_0 = \sqrt{k/J} \) the natural frequency of the system. Introducing \( Q = k/b \omega_0 \) this can be alternatively written as

\[ \hat{\theta} = \frac{\hat{f}Q}{k}, \]  

(8)

or, using equations (5) and (6),

\[ \hat{\theta} = \frac{1}{4} \varepsilon h \sum_{j=1}^{32} \frac{1}{\ln(r_{o,j}/r_{i,j})} \frac{Q}{k} \hat{V}^2. \]  

(9)

---

\(^1\)What is referred to as 'resonance' here and from here on does not mean driving at the natural frequency of the system \( \omega_0 \). Rather, the resonant frequency is defined as the frequency of maximal amplitude of motion \( \hat{\omega} \) and 'driving at resonance' means driving at the frequency of maximal amplitude. The reader will be reminded of this circumstance further in the text where appropriate.

The corresponding maximal amplitude is given by \( \hat{\theta} = \frac{\hat{f}}{b \sqrt{\frac{\omega_0^2 - \omega^2}{\omega^2}}} \). However, the \( b^2 \)-correction proves to be of second order and is neglected. Alternatively, (7) can be seen as a lower-bound approximation.
This representation will gain importance, since the quality factor can be obtained directly from experiment.

### 2.3.2 Optical Calibration

While being a consistent measure for changes in amplitude of motion for one device, the measured output-signal (proportional to amplitude of motion, see section 2.1) does neither allow for a comparison between devices nor does it give the amplitude itself. The absolute value might be influenced by parasitic capacitance and/or non-ohmic contacts. Therefore, a different technique must be used to directly assess the amplitude of rotation, which dictates the stress applied at the notch root.

The method of choice is the optical measurement of motion-blur coupled with a model to extract the displacement therefrom. The measurement is performed using a setup of a digital camera equipped with a 50x objective mounted on a vibration free stage.

For each calibration two images at different motion amplitudes and one rest image are obtained. The images of the device in motion are aligned to the rest image and pixel values \( I_{\text{rest}}^i \) and \( I_{\text{motion}}^i \) are obtained along the same given path (see Fig. 8).

To extract the amplitude of motion a model similar to [11] is chosen. It can be derived from the sinusoidal motion of an essentially one-dimensional structure with a prescribed intensity distribution \( \hat{I} \) along its length as depicted in Fig. 9a. Using the coordinate \( x \) to track the motion of the structure as shown in Fig. 9b, the intensity observed at an arbitrary coordinate \( x_0 \) at a time \( t \) is \( \hat{I} (x(t) - x_0) \), while the intensity of the motion-blur at this point is proportional to the time average over the half-period of motion \( \tau \)

\[
I(x_0) \sim \int_0^\tau \hat{I} (x(t) - x_0) \, dt,
\]

(10)
Figure 8: Rest- and motion-images and their profiles.
Figure 9: Basic model for the pixel intensity distribution in motion-blur.

with

\[ x(t) = \hat{x} \sin \omega t , \]  
\[ \tau = \frac{\pi}{\omega} . \]  

Using (11) and introducing \( \xi = x/\hat{x} \), expression (10) can be written as an integral over a normalized spatial coordinate

\[ I(x_0) \sim \int_{0}^{1} \hat{I} (\hat{x} - x_0) \, d \arcsin \xi . \]  

Pixel images introduce a discretization to the intensity distribution \( \hat{I} \), which is mainly dictated by the pixel size, i.e. the intensity distribution can be written as

\[ \hat{I} = \sum_{j} \hat{I}_j \left[ \theta(x_j) - \theta(x_{j+1}) \right] \]  

with \( x_{j+1} - x_j \) being the effective pixel size and \( \theta \) the unit-step function. Using this and switching summation and integration simplifies (13) to

\[ I(x_0) \sim \sum_{j} \hat{I}_j \left[ \arcsin(x/\hat{x}) \right]_{x_j}^{x_{j+1}} . \]
For a given $\hat{I}$ formula (15) predicts the intensity distribution in the motion blur.

Using $\hat{I}_{i}^{\text{rest}}$ as initial intensity distribution, different motion-blur distributions are predicted for different amplitudes $\hat{x}$, while the correct amplitude will provide the best fit to $I_{i}^{\text{motion}}$. This value is found using the method of least squares. The rest image does not contain any intensity information on the part of the substrate underneath the resonator-plate, which is only uncovered during motion. Therefore, the above approach assumes equal pixel intensity for the whole substrate. This approximation is partly responsible for the deviation of the model’s prediction from the measured motion profile. Overall, the method is limited by the pixel-size of the camera (94nm at 50x magnification), corresponding to an angular uncertainty of $3.2 \cdot 10^{-4}\text{rad}$.

### 2.3.3 Stress and stiffness calculations

To relate stress at the notch to the measured angular rotation, a 2D finite element model was employed (see Fig. 10). The cantilever beam was clamped (zero displacement boundary condition) and a pressure on the left side of the mass was applied; the first principal stress at the notch as well as the angle of rotation around the center of the ligament were extracted. The commercial software ANSYS v11 was used for computation. The geometry was defined parametrically by keypoints in the internal preprocessor. The meshing was performed using 8-node quadriliteral elements (PLANE82) with the mesh-density locally increasing at the notch. Small displacements and plane stress state were assumed for the structural analysis.

The mesh density was varied until convergence of the natural frequency, which is the frequency corresponding to the first eigen-mode of the system, could be verified.

The underlying material model was chosen to be isotropic linear-elastic with the Young’s modulus $E = 163\text{GPa}$ and the Poisson ratio $\nu = 0.23$ calculated as the mean of the Hashin-Shtrickman bounds for polycrystalline silicon [17]. However, in the given case the grain-size is relatively large compared to the dimensions of the
sample; van Arsdell and Brown [66] refer to this as 'multi-crystalline' rather than polycrystalline. Here the isotropy assumption is only approximate and scattering of the real stiffness and stress values around the model’s prediction must be expected.

The model was used to calculate the constant of proportionality $\lambda$ between the first principal stress at the notch $\sigma$ and the angular rotation $\theta$ in the relation

$$\sigma = \lambda \dot{\theta}$$

(16)

The stiffness of the structure $k$, which relates angular rotation to the applied moment $M$

$$\dot{\theta} = \frac{M}{k}$$

(17)

is found accordingly. Its value highly depends on the geometry of the cantilever beam; to illustrate the effect of a slight change in the dimensions, calculations were carried out for both the geometry as drawn on the fabrication masks and the real geometry measured using scanning electron microscopy (SEM). The dimensions for both cases are given in Fig. 11. Since the analysis is linear, one simulation is sufficient to obtain the required information.
as drawn: as measured:
\[ l = 39.8\mu m \quad l = 40.0\mu m \]
\[ w = 20\mu m \quad w = 19.25\mu m \]
\[ d = 9.8\mu m \quad d = 10.0\mu m \]
\[ r = 1\mu m \quad r = 1.35\mu m \]
\[ \lambda = 6.67\mu m \quad \lambda = 6.13\mu m \]

Figure 11: Cantilever beam dimensions.

2.4 Fatigue Testing

2.4.1 Description of overall system

Fatigue tests using the structure described in section 2.2.1 were performed in controlled environment. The environmental chamber ESPEC SH-241 was used to control temperature and humidity levels with a tolerance of \( \pm 0.1^\circ C \) and \( \pm 1\% \text{RH} \) respectively.

A sinusoidal voltage at half the natural frequency of the resonator is applied to one of the device’s pads while the plate is attached to ground, thus forcing the device at resonance. To achieve the required stress level at the notch root, a relatively high voltage (\( \sim 115 - 120V \)) must be applied; the signal is generated using the function generator Agilent 33220A and then amplified to high voltage (HV) using the HV-amplifier AVTECH AV-110G with the nominal gain set to 100x. The signal amplitude and harmonic distortion of the function generator were verified to be within its specifications. The gain of the HV-amplifier was assessed through both, direct measurement and the output signal of a resonator designated for calibration. The real gain of the different amplifiers used was found to lie between 95\% and 105\% of the nominal gain; it does not vary over time. This result is consistent for both measurement techniques.
Figure 12: Current to voltage converter for output pre-amplification.

A bias voltage $V_{\text{bias}} = +100\text{V}$ is applied to the remaining pad of the testing structure; the periodic change in capacitance of the attached comb drive caused by motion of the plate results in an induced current

$$i_{\text{out}} = \omega_0 \dot{\theta} V_{\text{bias}} \frac{\partial C}{\partial \theta}.$$  \hspace{1cm} (18)

The expected current amplitude is $\leq 10\text{nA}$; to get a conveniently measurable signal this current is pre-amplified by means of a current-to-voltage converter as depicted in Fig. 12. Thus, an output voltage in the mV1-range is achieved. To prevent noise induced by long cable connections, the pre-amplifier needs to be positioned as close as possible to the resonator; therefore, it is located inside the environmental chamber. The transfer function of the converter was measured in the different testing environments (see Fig. 13) and long term stability was verified.

Due to the small size of the on-chip structure and the relatively high driving voltage, various parasitic electrical effects and feed-through in particular have to be
Theoretical Output (mV) | Frequency (kHz)
--- | ---
30C 50%RH | 0
80C 90%RH | 10

Figure 13: Transfer functions measured in the different environments and the theoretical transfer function for 50nA of input-current. The current was generated by supplying a 50mV voltage (calibrated) over a 1MΩ resistor (not calibrated).
dealt with. The output-signal is overlaid by feed-through from the driving side of the device with the noise amplitude being typically two orders of magnitude larger than the amplitude of the useful signal. However, due to the frequency doubling effect mentioned in section 2.3.1 this noise is at half the driving frequency $\omega$ and hence can be separated.

For this purpose a lock-in amplifier (Stanford Research SR 830) is employed. This device uses the TTL-sync output of the function generator as a reference to isolate and measure signal amplitudes of its higher harmonics.

The full setup is schematically pictured in Fig. 14. While being able to test two devices simultaneously in its current configuration, it is designed to be fully extensible by cascading units consisting of one function generator, one HV-amplifier and one current-to-voltage converter. Only one control computer and one lock-in amplifier are required, since input only needs to be acquired discretely and multiplexers can be used to switch the output and reference signals between different sources.

The test is fully automated and controlled by a program created using the commercial software LabVIEW. It keeps track of the resonant frequency\(^2\) evolution by periodically (every 50s) sweeping over a small frequency range (60Hz). For each sweep, the maximum is found by fitting a 2\(^{nd}\)-order polynomial of the form

$$p(f) = af^2 + bf + c$$

(19)
to the acquired data and determining the location of its center

$$f_{\text{max}} = -\frac{b}{2a}.\quad (20)$$

The driving frequency is adjusted accordingly.

Amplitude and phase of the output signal, as well as the temperature and humidity values inside the environmental chamber are acquired every 15s, while a large range

\(^2\)For this purpose, what is referred to as resonant frequency is not the natural frequency of the system $f_0 = \omega_0/2\pi$, but rather the frequency at maximum amplitude of motion.
Figure 14: Schematic overview over the testing setup. The blue square signifies the environmental chamber.
sweep is performed every 30min in order to track the quality factor evolution.

2.4.2 Testing Protocol

For consistency, a common protocol for all tests was employed:

1. set environmental conditions to 30°C
2. determine resonant frequency
3. drive at resonance at low voltage corresponding to ca. 0.6GPa of stress at the notch root for 30min or until stable response
4. change environment to desired testing conditions
5. increase voltage in several steps until final testing voltage is reached
6. perform test until either catastrophic failure or $10^{10}$ cycles

In step 4 temperature and humidity are typically increased. To preserve electrical functionality it is important to prevent condensation. To this end, the following procedure is adopted:

1. increase temperature to desired value and wait for 10min
2. increase humidity in 10%RH-steps and wait 15min after completion
3. wait for 30min after final humidity value is reached

2.5 Failure Analysis / Electron Microscopy

2.5.1 SEM sample prep

Fractographic analysis was conducted using scanning electron microscopy (SEM) on failed devices. The FEI Nova Nanolab 200 and Zeiss Ultra 60 field emission SEMs were used for imaging at 10kV and 5kV accelerating voltage, respectively. A micro-manipulator was used to lift out the resonator plates, taking advantage of the predominant adhesive forces at small scale. The plates were then attached to a standard 90° SEM sample-holder using conductive epoxy, which was cured for 5min at 120°C. No conductive coating was applied to the samples.
2.5.2 TEM sample prep (FIB)

To quantitatively assess microstructural changes due to fatigue degradation, transmission electron microscopy (TEM) on full vertical cross-sections taken at the notch root was employed. For imaging the HRTEM JEOL 4000EX was used, while the analytical TEM Hitachi HF-2000 equipped with a field emission gun (FEG) as well as a thin window energy dispersive X-ray spectrometer (EDS) was used for elemental analysis.

For this purpose electron transparent slices with a uniform thickness < 200 nm were prepared using the focused ion beam (FIB) method. The preparation was carried out in the FIB workstation FEI Nova Nanolab 200, which is equipped with a nano manipulator, thus allowing for in-situ lift-out procedures.

Figs. 15 and 16 show the sample preparation step by step. First a Pt layer is deposited over the notch root to protect the sample area of interest (about 3 µm wide), which is exposed to the ion beam at normal incidence, from damage (Fig. 15c). Then, the cantilever beam is cut on the mass-side (Fig. 15e). In the next step the micro-manipulator needle is welded with Pt to the sample (Fig. 16a) and the sample is cut free. The 3 µm thick slice is moved to the TEM half-grid (Fig. 16c) and welded to the top of the grid. In several subsequent steps the slice is thinned down to the desired thickness, while the ion beam current is reduced in each step to minimize redeposition with the final step carried out at 30 pA. The last milling steps are carried out at lower accelerating voltage (20 kV) to reduce sidewall-amorphisation. Fig. 16d shows the final result in side view; this is the area normal to the electron beam in TEM. In this image the notch is pointing to the right. It should be noted, that the dark material is platinum, which was deposited (top) or redeposited (bottom).
Figure 15: TEM sample preparation using FIB before lift-off step by step.
Figure 16: TEM sample preparation using FIB after lift-off step by step.
2.6 Modal analysis of oxidized, cracked and coated fatigue characterization structures.

The influence of damage in the form of full-thickness and localized semi-elliptical cracks and full-thickness oxides on the resonant frequency of the resonators was investigated numerically. The introduction of a crack creates a system with bilinear stiffness (crack open/closed). In the linear approximation, the effective resonant frequency is the arithmetic mean of the cracked and uncracked resonant frequencies

$$\omega_{eff} = \frac{1}{2} (\omega_0 + \omega_cr) \ .$$

Accounting for the bilinear stiffness effect yields changes in resonant frequency that differ by a factor of $\frac{1}{2}$ from the previously published [45].

2.6.1 Full-thickness cracks and oxide

To investigate the influence of full-thickness oxides and cracks on the resonant frequency of the testing structure, finite element (FE) modal analysis was performed. The model used in section 2.3.3 was modified by skewing the notch geometry to create an oxidized region (see Fig. 17). The $SiO_2 - Si$ interface was moved 46% of the nominal oxide thickness into the silicon to account for the volumetric expansion during oxidation\(^3\). A crack was placed in the oxide region. Crack meshing is discussed in section 2.6.2. To account for the effect of pure oxidation the crack faces were closed using contact-target element pairs (CONTA172, TARGE169); the multi-point-constraint (MPC) contact approach was applied. It was verified that the contact elements do not alter the uncracked resonant frequency, by moving the silica-silicon interface 100% of the oxide thickness into the silicon and changing the material properties of the oxide ($E = 60GPa$ and $\nu = 0.2$)\(^4\) to those of silicon ($E = 163GPa$

\(^3\)The volume is changing from 12$cm^3/mol$ for $Si$ to 26$cm^3/mol$ for $SiO_2$ [25]

\(^4\)Values are close to what is reported for thermally grown silica [50].
2.6.2 Semi-elliptical surface cracks

SEM fractography (see 3.3.1) suggests localized rather than full-thickness flaws. To account for their effect on the resonant frequency modal analysis in the presence of semi-elliptical surface cracks is performed. The crack geometry is schematically shown in Fig. 18. Geometric modeling was carried in the ANSYS v11 preprocessor by keypoints, resulting in a fully parametrized geometry. The PCG Lanczos method was used for modal extraction. The material model was chosen as in section 2.3.3 to be linear-isotropic with the elastic constants $E = 163\,\text{GPa}$ and $\nu = 0.23$. The model was meshed with 20-node brick elements (SOLID186) (see Fig. 19); the overall mesh-density and the mesh-density around the crack could be varied independently by means of two parameters. In FE-analysis cracks are typically meshed using quarter-point crack-tip elements to implement the $1/\sqrt{r}$ stress singularity. In the present case
however, the focus is on the global behavior of the structure rather than the local stress state; therefore, following the observations of Chati et al. [13] on the influence of crack-tip meshing in modal analysis, crack-tip elements were not used. To verify the validity of this approach, a convergence study was performed by subsequently increasing the mesh-density around the crack. For the small cracks of interest, it could be verified that no change in calculated resonant frequency\(^5\) occurs above a certain threshold value for the mesh-density parameter.

2.6.3 ALD-alumina coated structures

A further modification of the model used in section 2.3.3 allows to investigate the effect of cracking in an ALD-alumina surface coating. A 20nm-surface layer of linear-isotropic \(Al_2O_3\) \((E = 155\) and \(\nu = 0.24\) [65]) is added to the model; the employed geometry is shown in Fig. 20.

Since relatively large tensile residual stresses of \(\sim 500\)MPa are reported in the ALD layer [58], their effect on the resonant frequency needs to be considered. Static analysis was performed allowing the silicon to expand isotropically until the desired

\(^5\)The in-plane resonant frequency is the eigenfrequency corresponding to the third eigenmode. The first two eigenmodes are out-of-plane bending and torsion respectively.
Figure 19: 3D-model for cracked modal analysis.
stress in the ALD-layer was reached. The calculated prestress-matrix was used for subsequent modal analysis.
CHAPTER III

EXPERIMENTAL AND NUMERICAL RESULTS

3.1 Fatigue Testing

3.1.1 Stress measurements

The stiffness of the fatigue testing device and the relation between the stress at the notch root and angular rotation are found according to section 2.3.3 using FEM. As mentioned earlier, the stiffness is very sensitive to the geometrical parameters of the structure, thus necessitating exact post-fabrication measurements. This fact is illustrated in Fig. 21; here the different dimensions refer to Fig. 11. For the real geometry the stiffness was calculated to be \( k = 0.6385 \mu \text{N/rad} \). The constant of proportionality \( \lambda \) between the stress at the notch root \( \hat{\sigma} \) and the angle of rotation \( \hat{\theta} \) was found to be \( \lambda = 98.6 \text{GPa} \), i.e.

\[
\hat{\sigma} = 98.6 \text{GPa} \cdot \hat{\theta}.
\]  \( (22) \)

The corresponding values obtained for the as-drawn geometry are \( k = 0.7956 \mu \text{N/rad} \) and \( \lambda = 120.7 \text{GPa} \). The stress is given in [GPa] and angle of rotation in [rad].

Optical measurements according to the procedure outlined in section 2.3.2 were performed on 10 uncoated devices and 5 devices coated with 20nm ALD-alumina for two different voltages. The amplitude of rotation \( \theta \) is found to be proportional to the square of the voltage amplitude \( V^2 \) (see Fig. 22) such that

\[
\frac{\hat{\theta}}{V^2} = 1.293 \cdot 10^{-6} \left[ 1/V^2 \right] \quad \text{(uncoated)},
\]  \( (23) \)

\[
\frac{\hat{\theta}}{V^2} = 1.377 \cdot 10^{-6} \left[ 1/V^2 \right] \quad \text{(coated)}.
\]  \( (24) \)

Fig. 23 shows a plot of these normalized amplitudes over the corresponding average quality-factor \( Q \), which was obtained as described in section 2.4.1 by sweeping over a
Figure 21: The dependence of the angle of rotation $\hat{\theta}$ on the applied moment.

Large frequency range (see Fig. 24); it suggests that the linear proportionality between $\hat{\theta}$ and $Q$ predicted in (9) holds. For the amplitude of motion the relation

$$\hat{\theta} = \kappa Q \hat{V}^2$$

with $\kappa = (1 \pm 0.05) \cdot 10^{-8} [1/V^2]$, $Q$ being the quality factor and $\hat{V}$ the amplitude of the driving voltage is established. Equation (25) holds in the coated and uncoated case with the same\(^1\) constant of proportionality $\kappa$. As predicted in section 2.3.1 the amplitude of motion $\hat{\theta}$ is proportional to $\hat{V}^2$, which translates using equation (18) into a linear relation between the output voltage and $\hat{V}^2$; this could be verified experimentally (see Fig. 25).

\(^1\)The ALD-alumina coating does not alter the capacitance of the structure, since it is very thin (20nm) with respect to the air-gap (3.3µm) and has a relatively low dielectric constant ($6 \div 7$) [20, 37]. Indeed, assuming $\varepsilon_{ALD} = 7$ basic calculations give an increase in capacitance of only 1%.
Figure 22: Dependence of the amplitude of rotation on the applied voltage.

Figure 23: The dependence of the amplitude of rotation on the quality-factor.
Figure 24: Large range frequency sweep to obtain the quality-factor $Q$.

Figure 25: The dependence of the output voltage $V_{out}$ on the input voltage $V_{in}$.
3.1.2 Low-cycle fatigue strength

Ramp-up tests were performed to determine the low-cycle fatigue strength of the devices as an indicator of the strength of the material. The applied voltage was increased until failure occurred within \( \sim 20 \) sec; for these tests, the motion of the resonators was optically recorded at 20x magnification with a frame-rate of 27fps. After failure the angle of rotation was extracted from the motion blur and stresses calculated using equation (22). The strength was found to be 3.17 ± 0.5 GPa as a result of 8 measurements. Alsem et al. [4] report a higher strength of 3.8 ± 0.3 GPa for devices fabricated in the same foundry; a possible explanation is the presence of vertical "nano-grooves" observed on the sidewalls of the present devices, which might act as nano-notches creating local stress concentrations. Such severe nano-grooves have not been observed in previous runs of the same fabrication process (more information on the sidewall morphology can be found in section 3.3.1). Hence, all stress values given in the following should be understood as nominal values.

3.1.3 Fatigue Results

Fatigue data were obtained for 16 devices tested in moderate (30°C, 50%RH) environment, 11 devices tested in harsh (80°C, 90%RH) environment as well as 11 devices coated with 20nm ALD-alumina tested in moderate environment. To this date, no comprehensive data on the fatigue behavior of polysilicon thin films in harsh environments is available in the literature. Similarly, no investigations on the influence of ALD-alumina coatings on (poly)silicon fatigue were published so far.

3.1.3.1 Uncoated devices

The stress-life data obtained for uncoated 2\( \mu \)m-thick polysilicon devices in the respective environments is shown in Fig. 26. Delayed failure is observed in the range between \( 5 \cdot 10^5 \) and \( 1.2 \cdot 10^{10} \) cycles for stresses significantly lower than the low-cycle fatigue strength. The squares denote 'run-out' tests, i.e. devices that did not
The brittleness of silicon is reflected on the $S - N$ curve in the form of large scatter. In agreement with previously published results [4, 30, 45, 54, 62] (see Fig. 3), the fatigue life varies over three orders of magnitude for a given level of applied stress. This can be understood by considering that the critical flaw size is determined by the local stress state and the local fracture toughness of the material, which both depend on the grain orientation [12]. Similarly, the life to failure depends on the initial flaw size.

Comparison of Figs. 3 and 26 reveals that in the present study fatigue tends to occur at lower stress levels. A possible explanation is the different sidewall morphology already mentioned in section 3.1.2. Grooves in the sidewall can cause local stress concentrations resulting in a higher stress acting on the flaw that initiates failure.

Regardless of the initial flaw distribution that may be more severe than in previous
investigations on similar devices [4, 30, 45, 54], the present study clearly highlights the critical influence of the environment on the fatigue behavior. Indeed, 10 out of 11 devices tested between 1.46 and 1.6GPa failed before $10^9$ cycles at $80^\circ$C, 90% RH, while 6 devices tested at same stress levels or higher did not fail before $10^{10}$ cycles in $30^\circ$C, 50% RH environment.

The critical influence of the environment is also unambiguously captured via the monitoring of the resonant frequency for each fatigue test. The resonant frequency was obtained as detailed out in section 2.4.1 by sweeping over a frequency range and finding the frequency of maximum output from a second-order polynomial fit (see Fig. 27). It was first verified that the resonant frequency remains stable ($\pm 0.5Hz$) over a large number of cycles for small applied stresses ($\sigma \leq 1.3$GPa). Hence, change in resonant frequency is associated with change in stiffness of the structure caused by damage accumulation and is used as a measure thereof.
The frequency decrease of failed specimens over the total device-life is shown in Fig. 28. The total decrease in resonant frequency tends to increase with increasing life-time, which is interpreted as an increase in damage accumulation necessary to grow a crack to its critical size. This result is consistent with previous investigations [6, 45]. However, the current results also highlight the critical influence of the environment on the damage accumulation rate, as larger decreases in resonant frequency occur over a much shorter fatigue life in the 80°C, 90%RH environment than the 30°C, 50%RH environment. This result is better captured in Fig. 29, where the evolution of the decrease rate $df/dN$ of the resonant frequency during the specimen’s life is shown. The rate was calculated by fitting a 6th-order polynomial to the frequency evolution data and differentiating analytically; the obtained rates were sampled at $10^i$ ($i = 4, ..., 10$) cycles for readability. It is apparent that the frequency decrease rates for the different environments lie in separate 'bands' with the low-humidity band ranging from $10^{-10}$ to $10^{-8}$Hz/cycle and the high-humidity band ranging from $10^{-8}$ to $10^{-6}$Hz/cycle. In addition, in the 30°C, 50%RH environment the decrease-rate in resonant frequency, which is associated with the damage accumulation rate, tends to decrease towards the end of life, while damage acceleration is observed in the high-humidity environment. This result can also be observed in Figs. 30 to 32 showing the frequency evolution over the device-life for the tested devices.

It should be noted that Fig. 29 has no notion of stress; this is particularly remarkable, since the devices that failed in the high-humidity environment were typically subjected to stress-amplitudes that do not lead to failure within $10^{10}$ cycles in moderate environment.

3.1.3.2 ALD-alumina coated devices

According to the previous section, a harsh environment significantly accelerates the fatigue degradation process for polysilicon thin films. Therefore, the presence of a
conformal surface coating should prevent any interaction between the silicon surface and the surrounding environment, provided the coating remains intact during cyclic loading. The stress-life data for ALD-alumina coated resonators at 30°C, 50%RH is presented in Fig. 33. The presence of the alumina coating does not appear to significantly affect the $S - N$ curve. However, the coated devices show a drastically different frequency evolution behavior compared to the uncoated devices. While for uncoated resonators the total loss in resonant frequency increases with increasing fatigue life, this trend is not observed for ALD-alumina coated devices (see Fig. 34), indicating a different degradation mechanism. This deduction is further supported by the different trends observed for the resonant frequency evolution over the device life shown in Figs. 30 to 32 and 35. ALD-alumina coated devices exhibit a very fast decrease in resonant frequency over the first $\sim 10^9$ cycles, followed by either fatal failure or a plateau of relatively constant resonant frequency until the tests
Figure 29: The frequency decrease rate plotted over the life ($N$).

Figure 30: Frequency decrease over the lifetime of devices failed at 30°C, 50%RH.
Figure 31: Frequency decrease over the lifetime of run-out devices at 30°C, 50%RH.

Figure 32: Frequency decrease over the lifetime of devices failed at 80°C, 90%RH.
Figure 33: Stress-life curve for ALD-alumina coated specimens tested in 30°C, 50%RH environment.

were interrupted after $\sim 10^{10}$ cycles. No continuous damage accumulation as for the uncoated case is observed. This behavior may be consistent with mechanical damage in the alumina-coating without activating the mechanism responsible for silicon fatigue.

3.1.3.3 Monocrystalline silicon devices

To investigate a possible grain-boundary effect on the resonant frequency evolution, two single-crystal silicon (beam oriented along [100] in (100) plane) fatigue resonators of similar geometry\(^2\) were subjected to cyclic loading in the 30°C, 50%RH environment. The same testing setup as for the polysilicon resonators was used. The applied stress level was chosen in the 2.2-2.6GPa range in order to allow comparison with the

\(^2\)The in-plane shape and dimensions are identical with the polysilicon devices; however, the out-of-plane thickness is 10µm rather than 2µm.
Figure 34: Frequency decrease over total device life for ALD-alumina coated specimens tested in 30°C, 50%RH environment.

Data obtained for the polysilicon material while accounting for the higher strength of the SOI devices. The change in resonant frequency over the test-time is illustrated in Fig. 36 and shows trends similar to those observed in Fig. 31 for polysilicon. While one device (blue curve) exhibits the typical decrease in damaging rate over the testing time, the second device shows this effect less pronounced. No damage acceleration is observed, which is consistent with the behavior of polycrystalline devices in the same environment.

3.2 Microstructure and sidewall morphology

Fig. 37a shows a TEM micrograph of a vertical cross-section of a beam taken at the notch root. Several grain-boundaries are highlighted. In Fig. 37b a top-view of the beam is shown. Both images show a variety of grain shapes and sizes (≈50 – 500nm) making it hard to speak of a characteristic grain size of ≈350nm or
Figure 35: Frequency decrease over the lifetime of ALD-alumina coated devices. Arrows denominate run-out tests.

Figure 36: Frequency evolution of SOI devices tested at 30°C, 50%RH.
distinct crystallographic texture (vertical columnar grains) as in [4].

Figs. 38, 39 and 44 show the morphology of the sidewall near the notch. The typical horizontal etching line but also sharp vertical grooves so far unmentioned in the literature can be seen. These grooves tend either to go through the full thickness of the beam or to be located below the etching line; the full-thickness grooves have a typical width of ~100nm, while the smaller grooves below the etching line show widths of about 10-20nm.

In the context of fatigue, full-thickness features can be interpreted as nano-notches causing highly localized stress concentrations, while the sharper and smaller grooves can act as fracture initiating flaws. This observation is consistent with the fact that for manually overloaded resonators the lines on the fracture surface indicate a fracture origin below the etching line (see Fig. 40).

The sidewalls of ALD-alumina coated devices show less smaller grooves indicating that the deposited layer is (at least partially) filling up the present flaws.

3.3 Fractography

Post testing electron imaging was performed to assess the forms of degradation due to cyclic loading. This includes fractographic analysis by means of scanning electron microscopy (SEM) of failed devices as well as transmission electron imaging of vertical cross-sections of devices interrupted prior to failure.

3.3.1 SEM

Fracture surfaces of a manually overloaded device as well as devices fatigued in the 30°C, 50%RH and 80°C 90%RH environments respectively can be seen in Figs. 40 to 43, while Fig. 45 shows the fracture surface of an ALD-alumina coated device. In all cases the topography is typical for brittle fracture, not always showing mirror-like surfaces as reported e.g. in [28]. However, the absence of mirrors might be attributed to damage of the fracture surface due to electrical arcing during failure (Fig. 43 near
(a) TEM micrograph of a vertical cross-section taken at the notch root.

(b) SEM top-view of the polysilicon layer.

Figure 37: Electron images of the microstructure of the polysilicon layer.
Figure 38: Full-thickness nano-notches at the notch root of a device.
Figure 39: Smaller flaws below the etching-line at the notch of a device.
the bottom of the beam).

It is remarkable that the fracture surfaces of manually overloaded devices show a clear initiation point and a small mirror-surface (see Fig. 40). These features were reported not to be present on corresponding fracture surfaces from previous Poly-MUMPs runs [54]. Images of the edge of the fracture surface indicate that fracture initiates inside the nano-notches mentioned in section 3.2 (see Fig. 44 lower image). ALD-alumina coated devices do not show any initiation sites on the fracture surface, implying fracture initiation inside the surface coating (see Fig. 45).

The observed localization of the fracture origin for uncoated devices suggests vertical rather than horizontal (as used in [4] and [27]) slices for subsequent TEM studies.

3.3.2 TEM

3.3.2.1 Imaging

TEM imaging was carried out on two reference samples (Fig. 46), two samples fatigued in 30°C, 50%RH (Fig. 47) and 80°C, 90%RH (Figs. 48 and 49) environments respectively and one ALD-alumina coated sample (Fig. 50). The fatigued samples were manually interrupted after \( \sim 10^{10} \) cycles. Thicknesses of surface layers were measured from either interfaces visible as lines, or if possible, crystalline-amorphous interfaces found by tracing the silicon lattice fringes to their end; however, for the second method the lattice needs to be in the correct orientation, which is not always the case in a polycrystal. The results are summarized in Table 1.

For all samples the full sidewall\(^3\) was imaged; selected images are shown in the following.

The samples chosen for reference were fabricated from (1) a device within 24h after the HF release and (2) a device stored for 4 weeks in ambient air. Both samples

---

\(^3\)Sample thinning in FIB results in thickness variations over the sample height with the top-part being the thinnest. In an attempt of fabricating samples that are electron-transparent over the full height, some of the samples suffered damage in the top-part; however, at least 90% of the sample height were available for TEM imaging.
Figure 40: SEM micrograph of the fracture surface of a manually overloaded device.
Figure 41: SEM micrograph of the fracture surface of a device fatigued at 30°C, 50%RH. Failure occurred after $1.15 \cdot 10^{10}$ cycles at 1.57GPa and a total decrease in resonant frequency of 21.8Hz.
Figure 42: SEM micrograph of the fracture surface of a device fatigued at 80°C, 90%RH. Failure occurred after $6.4 \cdot 10^7$ cycles at 1.48GPa and a total decrease in resonant frequency of 8Hz.
Figure 43: SEM micrograph of the fracture surface of a device fatigued at 80°C, 90%RH. Failure occurred after $5.2 \cdot 10^7$ cycles at 1.45GPa and a total decrease in resonant frequency of 28.1Hz.
Figure 44: SEM micrograph of the notch sidewall of device fatigued at 80°C, 90%RH (for fracture-surface see Fig. 43).
Figure 45: SEM micrograph of a fatigued ALD-alumina coated device. Failure occurred after $2.44 \cdot 10^7$ cycles at 1.75GPa and a total decrease in resonant frequency of 43Hz.
Table 1: Results of the TEM fractographic analysis

<table>
<thead>
<tr>
<th>sample type</th>
<th>testing history</th>
<th>observations in TEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>reference</td>
<td>none</td>
<td>4-17nm of oxide at the notch, no thick oxide at the bottom (away from notch)</td>
</tr>
<tr>
<td>30°C, 50%RH</td>
<td>1.4 \cdot 10^{10} cycles at 1.39GPa; 18Hz frequency decrease</td>
<td>12-20nm of oxide at the notch, no thick oxide at the bottom (away from notch)</td>
</tr>
<tr>
<td>80°C, 90%RH</td>
<td>7.5 \cdot 10^{9} cycles at 1.48GPa; 54Hz frequency decrease</td>
<td>8-15nm of oxide at the notch, highly localized region with \sim 50nm of oxide, no thick oxide at the bottom</td>
</tr>
<tr>
<td>ALD-alumina coated</td>
<td>9.8 \cdot 10^{9} cycles at 1.62GPa; 28.7Hz frequency decrease</td>
<td>no thick oxide</td>
</tr>
</tbody>
</table>

show variations in the thickness of the oxide layer at the notch from 4 to 17nm; no thick oxide > 4nm can be seen at the bottom of the samples.

The sample fatigued in moderate environment was interrupted after 1.4 \cdot 10^{10} cycles with a decrease in resonant frequency of 18Hz. While no cracks in the notch region could be observed, Fig. 47 shows a thickened oxide layer of about 20nm; over the height of the notch the layer thickness varies between \sim 12-20nm. No thick oxides could be observed at the bottom part of the cross-section that is not exposed to high stress.

The 80°C, 90%RH-sample was interrupted after 7.5 \cdot 10^{9} cycles. The measured decrease in resonant frequency amounted to 54Hz. Like the specimen fatigued in moderate environment, this sample is showing a thickened oxide layer at the notch but not at the bottom of the cross-section, away from the highly stressed area. In addition, a highly localized amorphous region of about 50nm depth is located at the upper half of the notch (see Fig. 48). The amorphous material is verified to be silica (see section 3.3.2.2 for EDS results).

The ALD-coated sample, which was interrupted after 9.8 \cdot 10^{9} cycles in moderate
environment, shows an undamaged alumina layer and less that 8nm of \( \text{SiO}_2 \) underneath.

### 3.3.2.2 Elemental analysis

Elemental analysis using energy dispersive X-ray spectroscopy (EDS) was performed to verify the composition of the localized amorphous region mentioned in 3.3.2.1. Spectra were obtained (a) in the silicon, (b) in the surface oxide above the area of interest, (c) in the platinum, (d) in the amorphous region and (e) in the surface oxide below the area of interest. Fig. 51 shows the spectra and the position of the locations chosen for analysis; a live bright-field TEM-image was available for precise positioning of the spot.

Contamination with C (most likely from the precursor-gas Trimethyl \( [(1,2,3,4,5\text{-ETA.})-1 \text{ Methyl 2,4-Cyclopentadienyl}] \text{ Platinum} \) used for Pt deposition), Cu (from the TEM half-grid) as well as Ga, Zr and Sn were detected. For the deposited Pt layer (c) the mass composition was found to be 57.9\% Pt, 30.9\% Cu and 11.2\% Ga. The copper is redeposited material from the TEM half-grid, while the gallium can be considered contamination from the ion beam.

The \( O \) to \( Si \) ratios in atomic number are summarized in Table 2.

<table>
<thead>
<tr>
<th>location number</th>
<th>description</th>
<th>( #O/#Si )</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>in the silicon</td>
<td>0.1</td>
</tr>
<tr>
<td>(b)</td>
<td>surface oxide above the area of interest</td>
<td>0.9</td>
</tr>
<tr>
<td>(d)</td>
<td>in the amorphous region</td>
<td>2.1</td>
</tr>
<tr>
<td>(e)</td>
<td>surface oxide below the area of interest</td>
<td>0.5</td>
</tr>
</tbody>
</table>

The spot-size of the electron beam used for excitation is \( \sim 30 - 35 \text{nm} \), i.e. for the thinner oxide layers, part of the spot will be on the oxide and part on the silicon; hence, the atomic number ratio should be shifted from the stoichiometric ratio for
Figure 46: TEM micrograph of a vertical cross-section taken at the notch root of a released resonator used for reference. The oxide thickness along the notch varies between 4 and 17nm.
Figure 47: TEM micrograph of a vertical cross-section taken at the notch root of a resonator fatigued in 30°C, 50%RH environment. The notch is showing 12-20nm of oxide, while no thick oxide can be found in the bottom part of the beam.
Figure 48: TEM micrograph of a vertical cross-section taken at the notch root of a resonator fatigued in 80°C, 90%RH environment (small image). The notch is showing 8-15nm overall oxide, and an about 35-50nm deep oxide region localized over ~200nm.
Figure 49: TEM micrograph of a vertical cross-section taken at the notch root of a resonator fatigued in 80°C, 90%RH environment (continued).
Figure 50: TEM micrograph of a vertical cross-section taken at the notch root of a fatigued ALD-alumina coated resonator. No damage in the alumina layer due to cyclic loading could be found. The layer is shown exemplarily at two different locations along the notch and one location at the bottom of the sample. The silicon-alumina interface was found by tracing the lattice fringes on the silicon side (see image in lower-right corner).
$SiO_2$ towards silicon. For thicker oxides the measured ratio should approach the stoichiometric ratio $#O/#Si = 2$ and coincide with it (within the precision of the technique) if the spot is fully contained in silica.

Location (a) shows a ratio of 0.1 indicating a small amount of post-FIB oxidation. Surface oxides in locations (b) and (e) (oxides in both locations are too thin to contain the full spot) show ratios of 0.9 and 0.5 respectively suggesting thicker oxide at (b), which is consistent with bright-field TEM observations. From TEM, the thick amorphous region should be wide enough to contain the full spot, and indeed, shows a ratio of 2.1. Hence, it can be concluded that the localized amorphous region is comprised of $SiO_2$.

### 3.4 FE analysis

#### 3.4.1 The influence of full-thickness cracks and oxides

The 2D-FE model described in section 2.6.1 was used to investigate the influence of full-thickness oxide, full-thickness cracks and cracked oxide on the resonant frequency of the devices. Volumetric expansion during oxidation was accounted for.

To investigate the influence of cracks in silicon, the volumetric expansion was switched off and the elastic properties in the 'oxide'-region set to those of silicon$^4$.

Fig. 52 shows the frequency decrease due to cracking, oxidation and cracking in the oxide. For cracking in the oxide the crack-length was chosen to be 5nm less than the depth of the oxide, thus assuming the limiting case of (almost) fully cracked oxide. For up to 20nm of crack/oxide extension the decrease in frequency is dominated by the oxidation. For larger flaws and thicker oxides the effect of cracking becomes predominant. It is intuitively clear that the effects of cracking and oxidation do not additively superpose, since the elastic modulus of $SiO_2$ (60GPa) is much lower than that of silicon (163GPa) resulting in a smaller change in effective stiffness due to

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$^4$See Fig. 17 for geometry.
Figure 51: EDS spectra obtained around the localized thick oxide on the 80°C, 90%RH TEM-sample. The number ratio of oxygen to silicon atoms is given where relevant.
3.4.2 The influence of semi-elliptical surface cracks

The FE model described in section 2.6.2 was used to calculate the change in resonant frequency of the testing structure due to semi-elliptical cracks. Calculations were carried out for crack-length between 20 and 150nm with the dimensionless shape parameter $c/a$ ranging from 1 to 3.8. The results are presented in Fig. 53 along with the 2D limiting case of a through-thickness crack ($c/a = \infty$). As expected, the contribution of localized defects to the change in resonant frequency is negligible compared to full-thickness flaws.

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$^5$See Fig. 18 for explanation of the crack geometry.
Figure 53: Change in resonant frequency due to semi-elliptical and through-thickness cracks at the notch root.

3.4.3 The influence of residual stress on cracks in the ALD-alumina coating

Using FE-analysis, it was found that 500MPa of tensile residual stress in the ALD-alumina layer causes an increase in resonant frequency of $\sim 6$Hz for an uncracked device. From this result it can be anticipated that the influence of residual stress on the loss of resonant frequency is negligible. Indeed, Fig. 54 shows the decrease in resonant frequency for crack-extensions between 5 and 18nm in a 20nm ALD-alumina layer. It has to be noted that the change in frequency $\Delta f$ is calculated as arithmetic average between the cracked and uncracked frequencies (see section 2.6). However, due to the tensile residual stress, the crack will be open for a larger part of the cycle. Hence, the values given in Fig. 54 should be understood as a lower bound. The real decrease value lies in-between $\Delta f$ and $2\Delta f$. 
Figure 54: Change in resonant frequency due to cracking in the ALD-alumina surface layer.
CHAPTER IV

ANALYSIS OF RESULTS

4.1 Process-dependent geometry effects

In section 3.2 it has been shown that the sidewall morphology of the fatigue resonators is dominated by nano-grooves with widths ranging between 10 and 100nm. The presence of these features is strongly dependent on the details of the etching process, local geometry as well as nonlocal influences from as far as neighboring dies. Therefore, characterization of the sidewall morphology is needed to talk about "a" stress-life curve for a given process. This explains the large differences between previous $S – N$ curves (see Fig. 3) and the present study. Different nominal values of the stress are caused by different local stress concentrations and a different initial flaw population.

The notches can be subdivided into two groups according to their influence on the reliability of MEMS devices. The larger grooves in the size range of $\sim 50 – 100$nm create local stress concentrations, resulting in higher stresses acting on the smaller flaws. To estimate the magnitude of the resulting geometrical stress concentration factor, it is convenient to refer to the concept of a semi-elliptical notch in an infinite thin element; its stress concentration factor can be well approximated by the stress concentration of the corresponding elliptical hole with the error being as small as 10% even for deep notches [52]. Thus,

$$K_t = 1 + 2\sqrt{\frac{t}{r}},$$

with $t$ being the depth of the notch and $r$ the notch root radius. For a semi-circular notch the stress concentration is $K_t = 3$. This purely geometric effect can explain fatigue at stress values lower than previously published, which is observed in this study. It is also worth mentioning that the stress field of a notch is highly local;
hence, degradation will experience a slowdown once the flaw grows out of the sphere of influence of the notch. Fig. 55 shows the stress ahead of a circular notch\(^1\) normalized with the applied stress \(\sigma/\sigma_\infty\) over the distance from the notch root normalized with the notch radius \(d/r\). This is consistent with the decrease in degradation rate observed in the \(30^\circ\)C, 50\%RH environment. In the framework of reaction layer fatigue this effect was previously explained with the presence of the silica-silicon interface [47] (also see [8]).

The second group is comprised of the small and sharp notches found prominently in the lower part of the sidewall. These features can be considered initiating flaws rather than geometrical stress concentrations\(^2\). As such, they would contribute to the initial flaw distribution instead of the local stress state. Supporting evidence for a flaw distribution different from the previous PolyMUMPs runs can be drawn from the fractographic analysis of manually overloaded devices. All fracture surfaces show a clear initiation point indicated by ray-like lines converging into it and small mirror-like surfaces (see Fig. 40). Literature on the fatigue properties of devices from previous runs clearly states the absence of such features for single-cycle failure [54].

\(^1\)Again, the approximation of a circular hole in an infinite plate is used [38]
\(^2\)Clearly, all kinds and sizes of notches cause stress concentrations. The distinction is being made between grooves that create stress fields that act on other evolving flaws and grooves that are such flaws.
The size of mirror region is proportional to the initiating flaw size [28]; hence, the presence of a well-defined initiation point usually implies a larger flaw.

4.2 Implications for the reliability of Si-based MEMS devices

It has been unambiguously shown that harsh environments accelerate fatigue damage accumulation and shorten the life to failure. This must be accounted for, when designing MEMS devices that need to interact with the environment and can not be hermetically packaged. An example for such a device pose chemical and biological sensors. In essence, these sensors are resonators coated with a reactive polymer, which exhibit a high quality factor $Q$. If a chemical reaction takes place (sensing event), the oscillator shifts of resonance due to a change in mass, resulting in a decreased amplitude of motion, which can be detected. By design, these devices rely upon stability of the resonant frequency over time and can suffer loss of functionality caused by fatigue degradation long before fatal failure occurs.

For such an application, the alterations in the fatigue behavior introduced by the ALD-alumina coating can be advantageous. Instead of a continuing decrease in resonant frequency over the lifetime of the device, the coated specimens show a large initial decrease followed by a plateau of stable resonant frequency. A sensor can be pre-cycled until the plateau is reached; a frequency calibration obtained at this point can be expected to be valid for the rest of the device’s life.

4.3 Fatigue mechanism

The presence of thickened oxides after cycling was unambiguously determined. However, the oxide thickness was found to vary over the height of the sample; also, a highly localized thick region (see 3.3.2.2) was found. This inhomogeneity can explain why no oxide thickening after cycling was reported by Kahn et al. [27]; in fact, horizontal slices taken at different locations at the notch root would reveal vastly different
oxide thicknesses, presenting a 'hit-or-miss' situation.

Localized oxide thickening implies that the oxidation process depends not only on the environment, but also on the local stress state. This has been postulated as part of the reaction-layer fatigue mechanism by Muhlstein et al. [48] and is supported by recent evidence obtained from first-principles molecular dynamics simulations [15].

Thickened post-cycling oxides along with accelerated degradation at higher temperatures and humidities as well as changed fatigue behavior after application of an ALD-alumina coating are consistent with a surface degradation mechanism. Reaction-layer fatigue is such a mechanism that postulates the observed oxide thickening; its consistence with the experimentally obtained evidence is discussed in the following.

4.3.1 Experimentally observed changes in resonant frequency

From bright-field TEM, the observed oxide thickening is up to $3 \times 20 \text{nm}$ overall and up to $50 \text{nm}$ localized. This would result in a frequency decrease of up to $10 \text{Hz}$ based on Fig. 52.

The absence of large flaws means that even assuming a worst case scenario of $35 \text{nm}$ of almost fully cracked full-thickness oxide, which is much more than experimentally observed, the predicted decrease would be less than $20 \text{Hz}$ (see Fig. 52). This is not enough to explain the loss in resonant frequency for all tested specimens (see Fig. 56). Instead, it indicates the presence of multiple evolving flaws, one of which becomes critical and leads to fatal failure. This is not unlikely, since the area exposed to high stress is large enough to contain several flaws (see Fig. 57).

4.3.2 Fatal failure

The absence of thick full-thickness oxides indicates that the reaction-layer fatigue mechanism can only be active in the localized oxidation sites (see Fig. 58). The most quantitative assessment of the reaction-layer mechanism to date, was first presented

\[\text{In the sense of an upper bound.}\]
Figure 56: Experimentally obtained total decreases in resonant frequency. Ranges covered by different damage mechanisms indicated in shades of gray.

Figure 57: Notch root of a resonator. Area exposed to > 85% of the nominal stress is highlighted in blue. A semi-circular crack with 50nm radius is symbolically indicated in red.
Figure 58: Activity map for reaction layer fatigue [56]. Superposed (in grey) is the range of observed oxide thicknesses.

by Muhlstein et al. [47] and later revisited by Pierron et al. [56]; the authors developed a ‘failure map’ showing the stress and oxide thickness conditions necessary for the activity of reaction-layer fatigue. Since the stress intensity factor for semi-elliptical cracks approaches the 2D-case for aspect-ratios $c/a > 3$, the same map can be used for localized flaws.

It is clear that reaction-layer fatigue cannot be active at the nominal applied stress of 1.5-1.6GPa, necessitating instead large (local) stresses\(^4\) which can be caused by the

\(^4\)The same is true for cracking in silicon itself. As a numerical example, assuming a fracture strength of $K_{IC} = 1MPa\sqrt{m}$ and a semi-elliptical crack with an aspect-ratio $c/a = 2$, the critical crack length for an applied stress of 1.6GPa would be $a_c = 128nm$, much larger than experimentally
local geometry as discussed in 4.1.

Assuming a geometric stress concentration factor of \( K_t = 3 \), a nominal applied stress of 1.5GPa would result in a local stress of 4.5GPa acting at the crack tip. This stress value is sufficient to activate reaction-layer fatigue for some of the observed oxide thicknesses (see grey region in Fig. 58). The TEM study was performed on run-out samples, i.e. devices that did not fail, but were interrupted manually. Hence, it may be assumed that the oxides have not reached their final thickness. Considering an extended range of 'likely attainable' oxide thicknesses, smaller stress concentrations caused by shallower nano-notches would be sufficient to activate the mechanism\(^5\).

It should be mentioned, that the failure-map in Fig. 58 does not account for the influence of compressive residual stress in the oxide. Values as high as 400MPa are reported for thermally grown SiO\(_2\) [24, 33] and are likely to be even higher for the very-low-temperature oxides assumed in the present case. Compressive residual stress would 'shift' the failure map towards higher stresses. Therefore, the stresses and/or oxide-thicknesses required to activate reaction-layer fatigue might be higher than presented here.

The above discussion was carried out in terms of the classical fracture-mechanics concepts 'stress-intensity factor' and/or 'strain-energy release rate'. This approach was used throughout the literature on the fatigue of polysilicon thin films and its applicability has not been questioned. However, fracture mechanics was developed for bulk materials, where the continuum assumptions can be assumed to hold. This is not necessarily true for the thin-film case. In fact, assuming an interatomic spacing\(^6\)

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\(^5\)However, when using Fig. 58 with assumed stress concentrations caused by nano-notches, the decrease of the stress ahead of the notch needs to be accounted for. As an example, a semicircular notch with a radius of 80nm is considered; crack-extension from 5 to 40 nm ahead of the notch will reduce the stress acting on the crack tip by \( \sim 40\% \).

\(^6\)Based on the lattice spacing \( d^{(220)} = 192015\text{fm} \) for WASO 4.2 A high purity silicon. The \( d^{(100)} \) spacing for the same material is \( d^{(100)} = 543102\text{fm} \). [68]
of \( b = 2\text{Å} \), a 20nm crack extends only over 100 atomic distances; the zone of interest at the crack tip is even smaller. At this level, the validity of continuum-based theories is not guaranteed.

### 4.4 The effect of ALD-alumina coatings

ALD-alumina surface coatings change the fatigue behavior of the polysilicon structures. The \( S - N \) curve does indicate a slightly higher fatigue resistance. This can be understood as an implication of atomic layer deposited alumina filling up the smaller flaws and creating a smoother surface. Since the elastic properties of \( \text{Al}_2\text{O}_3 \) are reported to be very similar to those of polysilicon \((E = 150 - 155\text{GPa}, \nu = 0.24)\) [65], the jump in stress values when crossing the interface of the resulting composite will be small, thus practically removing stress concentrations ahead of sharp flaws.

Since no thick silica layer underneath the ALD-alumina coating was visible in TEM, an environmental degradation mechanism seems effectively disabled. However, the large initial decrease in resonant frequency indicates damage in the alumina layer. Stress corrosion cracking is a known damaging mechanism for alumina. A flaw can propagate driven by the applied and residual stresses in the ALD-layer, if the stress-intensity factor \( K_I \) exceeds the threshold for stress corrosion \( K_{I,\text{sc}} \). If the stress-intensity factor reaches the fracture toughness \( K_{IC,\text{Al}_2\text{O}_3} \) during propagation in alumina, fatal failure will occur (see Fig. 59 case (a)); however, if the crack can reach the alumina-silicon interface before becoming critical, it will be arrested (see Fig. 59 case (b)), since stress corrosion cracking is not active in silicon [69].

This mechanism explains why fatigue degradation is only observed during a short part of the device’s life, namely the time required to grow a flaw to the alumina-silicon interface, and why failure (almost) exclusively occurs during the same portion of life. For this to be valid, it has to be assumed that the fatigue degradation mechanism for silicon itself remains inactive even after the crack reaches the interface and provides
Figure 59: Stress corrosion cracking of ALD-alumina coated MEMS.
some (though very limited) exposure to the environment\textsuperscript{7}. Further evidence for damage constrained to the alumina layer is found in SEM-fractography; fracture surfaces of fatigued ALD-alumina coated devices reveal brittle fracture with no well defined fracture origin in the silicon (see Fig. 45).

However, even a full-thickness crack through the whole alumina layer will cause a decrease in resonant frequency of less than 6Hz (see Fig. 54). This is not consistent with the experimentally observed decreases of more than 20Hz (Fig. 35). Additional damage in the form of several cracks is necessary to explain the experimental observations.

Also, the evolution of a single crack is not compatible with the observed degradation kinetics. Stress corrosion cracking can be described by a law of the form

\[ \frac{da}{dt} = AK_I^n, \] (27)

with \( a \) being the crack extension, \( K_I \) the stress intensity factor and \( A \) and \( n \) phenomenological parameters of the material and environment [18]. The stress-intensity factor can be obtained (without assuming any particular geometry) from

\[ K_I = Y \sigma \sqrt{a}, \] (28)

with \( Y \) being a geometric form factor and \( \sigma \) being the stress acting on the crack tip. It can be inferred that the stress-intensity factor and thus (as per Eq. (27)) the crack propagation rate will increase with increasing crack length. This is not consistent with decreasing frequency-decrease rates observed in experiment (see Fig.

\textsuperscript{7}Referring to the reaction layer fatigue mechanism, the question of why stress assisted oxidation should work underneath a silica layer, but is not observed underneath an ALD-alumina layer, arises. A possible explanation lies in the presence of intrinsic residual stresses in the layers. Thermally grown SiO\textsubscript{2} is reported to have an intrinsic (i.e. corrected for thermal stresses) residual compressive stress of up to \( \sim 400\text{MPa} \) [24, 33]. This stress is attributed to incomplete fulfillment of the molar volume requirement at lower temperatures [25]. The compressive stress causes a similar tensile stress in the silicon, which favors oxidation as a way to increase the volume. Similarly the \( \sim 500\text{MPa} \) tensile residual stress in ALD-alumina [58] would superpose the stress applied to the silicon substrate with a compressive stress and thus slow down or inhibit oxidation.
Figure 60: Schematic of the cracking-starvation mechanism. Surface layer thickness not to scale.

Moreover, a crack will advance towards the alumina-silicon interface whenever $K_I > K_{I_{sc}}$; hence, the measured final decrease in frequency should be independent of stress. The contrary is observed in experiment (see graph in Fig. 60).

A much more feasible scenario presents a system of multiple cracks (see Fig. 61). While the kinetics of every single crack follow relations (27) and (28) the cumulative behavior is different. Application of an external load will create a ‘process-volume’ in which $K_I > K_{I_{sc}}$ for a class of flaws; all of these flaws will evolve until they reach the alumina-silicon interface (or one of them causes fatal failure). However, there will be always cracks that are growing faster than others due to a higher local stress acting on the crack tip; these cracks will reach the alumina-silicon interface first and be arrested. The same is true in the presence of crack-channeling, since some cracks will reach the free surface faster than others and stop expanding further (see Fig. 61 second row). This effects would decrease the cumulative damage rate due to ‘cracking-starvation’. Also, the size of the process-volume, i.e., the volume exposed
Figure 61: Schematic of the 'cracking-starvation' mechanism. Evolution of a crack-system is shown in the top-row; the corresponding steps in the evolution of a single crack (in the presence of crack-channeling) can be seen underneath. Surface layer thickness not to scale.
to a high enough stress to activate stress corrosion cracking, and thereby the number of evolving flaws, depend on the applied stress (see Fig. 60); additionally, classes of crack-sizes previously unaffected would be subject to stress corrosion if a higher stress is applied, thereby further increasing the number of evolving flaws. This can explain the experimentally observed larger decreases in resonant frequency for larger applied stresses.

Crack-tip shielding would result in an additional decrease in degradation rate with crack advance. The effect would be less pronounced than might be suggested by the schematic in Fig. 61 (top row), if the flaws tend to be localized rather than full-thickness cracks. However, if crack channeling is present, this mechanism might become very effective.
CHAPTER V

CONCLUSION AND DIRECTIONS FOR FUTURE WORK

This study investigated the influence of the environment as well as ALD-alumina surface coatings on the fatigue of thin polysilicon films. The results of both the fatigue testing as well as the electron microscopy characterization strongly suggest an environmental degradation mechanism.

5.1 Influence of the environment

Comparison of stress-life data obtained at 80°C, 90%RH to a reference set of S − N data gathered at moderate environmental conditions (30°C and 50% RH) reveals a strong tendency for faster degradation with increasing temperature and humidity. Indeed, 10 out 11 devices tested between 1.46 and 1.6GPa failed before $10^9$ cycles at 80°C, 90%RH, while 6 devices tested at same stress levels or higher did not fail before $10^{10}$ cycles in the 30°C, 50%RH environment. The measured damage accumulation rates in the high-humidity environment\(^1\) exceed the rates at 30°C, 50%RH by up to two orders of magnitude.

TEM studies of control and fatigued samples reveal thickened oxide layers in the highly stressed region after cycling as well as highly localized oxides of up to 50nm thickness. The presence of highly localized, thick oxides along the specimen’s thickness explains the previously published contradicting data obtained from TEM imaging of horizontal slices; a horizontal slice is not suitable to capture the oxide thickness distribution along the height of the sample’s sidewall.

\(^1\)When switching from the 30°C, 50%RH environment to 80°C and 90%RH, the temperature increases only by a factor of \(\sim 1.16\), while the partial pressure of water increases by a factor of \(\sim 20\).
These findings are consistent with the reaction-layer fatigue mechanism that pos-
tulates stress-assisted oxide thickening and stress corrosion cracking within the oxide. This mechanism, if active in localized oxidation sites, can qualitatively describe the fatigue degradation process in silicon and is free of contradictions with experimental observations. However, the experimentally measured decreases in resonant frequency and SEM fractography indicate that an extension to multiple cracks may be necessary.

5.2 Influence of ALD-alumina surface coatings

This work shows that the resonant frequency evolution over the lifetime of a MEMS device can be drastically altered by the application of an ALD-alumina surface coating. Indeed, as opposed to uncoated devices that exhibit steady decrease in resonant frequency over the lifetime, the resonant frequency of $\text{Al}_2\text{O}_3$-coated devices experiences a rapid decrease over an initial period of typically $< 10^9$ cycles before it reaches a plateau and remains stable over the remaining life. This behavior can be exploited for resonator based MEMS sensors and other devices that rely on frequency-stability over the lifetime. TEM imaging shows no thickened oxides underneath the alumina layer, indicating that an environmental degradation mechanism of silicon itself is disabled. No fracture origin is visible in SE-micrographs of the fracture surface, suggesting damage localization in the alumina layer.

A mechanism explaining the different behavior of the coated (with respect to the uncoated) devices is proposed, based on stress corrosion cracking$^2$ in $\text{Al}_2\text{O}_3$ and successive crack arrest at the alumina-silicon interface, since silicon is immune to stress corrosion. After crack arrest the resonant frequency would remain stable. If multiple cracks are considered, this mechanism is capable of explaining the observed decreases in resonant frequency as well as the stress dependence of the amount of

$^2$Alumina is known to be susceptible to stress corrosion caking in aqueous environment. ALD-deposited alumina is reported to contain $\text{OH}^{-}$-groups from excess surface species remaining during deposition [37], which would favor stress corrosion.
decrease and the kinetics of the process.

5.3 **Directions for future work**

Experimental evidence, such as strengthening after pre-cycling, suggests that the reaction-layer fatigue mechanism as it is presented to date might not be complete. Indeed, further research is required to understand the physical processes underlying the postulated stress-assisted oxide thickening, which is the core part and, drawing from experimental evidence, the rate limiting step of the mechanism. These efforts should target the problem on its own scale and account for discontinuum effects or, alternatively, verify the validity of continuum-based theories on the nanometer-scale.

From the experimental point of view, the present work investigated the evolution of nanometer-scale flaws using micron-scale devices. Future efforts should by directed towards true nano-scale testing with device-thicknesses being less than 200nm, which would allow for direct observation of crack-evolution and/or oxide thickening using in-situ environmental TEM.

For a more quantitative understanding of the degradation processes in ALD-alumina coated MEMS devices, more research on the properties of the ALD-deposited alumina coating is necessary. To date, no information on fracture toughness or the stress corrosion threshold are available in the literature. However, precise understanding of these properties and their dependence on the deposition process (temperature, purge time) and layer-thickness is required to design reliable MEMS devices.
REFERENCES


