Environment-induced failure modes of thin film resonators

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ABSTRACT

Resonant mode micromechanical devices have great potentials due to their high sensitivity and easy signal processing. As they are also sensitive to environmental effects, vacuum packaging is often required, which largely increases the costs. The current study focuses on such environment induced reliability problems and degradation processes. Stiffening effect was observed on thin silicon nitride and silicon carbide cantilever beams in air. The resonance frequency gradually increases in time. When the cantilever is subjected to mechanical shock or large deflection, the resonance frequency suddenly drops, and then increases again. Air, increased humidity, argon rich and nitrogen rich atmosphere influence the stiffening and the shock response behavior. The effects are explained with the surface oxidation model. The oxide layer introduces stress in the structure increasing the overall stiffness, while mechanical shocks crack the layer. Silicon resonators gather airborne particles from the atmosphere due to electrostatic charging. The extra mass results in decrease of the resonant frequency. All these processes lead to unstable resonance frequency and thus to failure of the resonant mode device. Tests in inert environment suggest cheap atmospheric packaging solution to obtain reliable operation and yet good performance.

Keywords: resonance frequency, cantilever, beam, silicon, nitride, carbide, reliability, failure, cycling

1. INTRODUCTION

Material properties and degradation processes of micromechanical structures can differ essentially from macroscopic ones, therefore characterization of actual micromachined test structures is required. The materials used in MEMS are fairly well characterized, but the degradation processes leading to reliability problems leave many questions still unanswered. Surface-induced and crack related effects play important role in thin films implemented in micromechanical devices.

The resonant operational mode of micromachined sensors and actuators has several attractive features. The resonance frequency of the resonator structure is determined by the physical dimensions and the material properties. Furthermore, the resonance frequency is also strongly dependant on various environmental factors such as damping, temperature, pressure etc. These factors can introduce mechanical stress in the resonator shifting the resonance frequency. Butler et al. showed how sensitive a flexural silicon nitride resonator is on membrane stresses [1]. Stress is a main contributor to the resonance frequency. Silicon nitride films are usually in tensile stress on the silicon substrate. Large displacement enhances the membrane tension and leads to amplitude-induced stiffening. Heating the membrane reduces the tension and results in decreased resonance frequency. Hence the resonance frequency of the device can be tuned by controlling the internal stress. The shift of the resonance frequency is easily measurable and the frequency output provides easy signal processing. Unfortunately the resonance frequency is not only sensitive to the one property we want to measure, but to undesired effects as well. Marxer et al. found that humid environment oxidized the surface increasing the stiffness and so the resonance frequency of thin polysilicon membranes [2]. This mechanical drift results in failure of the resonant mode device. The surface oxidation generates high internal stress, and can lead to corrosion fatigue. No dislocation movement is present in polysilicon; therefore slow crack growth and rupture are initiated by the failure of the surface oxide layer. Conally and Brown measured the crack growth on pre-cracked polysilicon structures and found that humidity increased the crack growth [3]. One attempt to eliminate the environmental influence is first level vacuum packaging of the device, which largely enhances the costs. The vacuum packaging of the device can be particularly difficult or even impossible in applications where the sensor needs to be in contact with the media.

These considerations motivated us to study the degradation processes, which occur on both surface and bulk micromachined resonators. Silicon nitride, silicon carbide and crystalline silicon cantilever beams were used in the tests. The silicon nitride
and silicon carbide have outstanding mechanical properties, which make them particularly suitable as mechanical components in resonators. The silicon nitride film has excellent mechanical, electrical and thermal properties for various applications. The main fields of applications are supporting mechanical elements, insulator layers and protective coatings. Extreme aspect ratio structures such as large membranes, plates or cantilevers are fabricated of thin silicon nitride films. The mechanical characteristics of silicon carbide are even more excellent than silicon nitride. The carbide film is implemented in microsystems as hard coating and also as a mechanical material. The outstanding chemical inertness of SiC offers applications as passivation layers for aggressive media and masking layers for microelectronics processing and micromachining. Implementation of SiC extends the working range of microelectronics to high-temperature, radioactive and chemically corrosive environments, and improves high-power and high-frequency electronics. The electronic band structure of SiC is suitable for UV sensors and other optoelectronic applications, which were not feasible with conventional silicon technology.

2. THEORY

Cantilever beam and paddle-beam resonators were fabricated. The resonance frequency of a rectangular beam with one end clamped can be described with the following equation [4]:

\[ f_{res} = \frac{A}{2\pi} \sqrt{\frac{EI}{\rho_l L^3}} \]  

where \( f_{res} \) = resonance frequency  
\( E \) = Young’s modulus  
\( I \) = area moment of inertia of cross-section  
\( L \) = beam length  
\( \rho_l \) = mass per unit length of beam  
\( A = \) a coefficient related to end conditions, \( A=3.52 \) for the first natural mode of a clamped – free cantilever

Thus the Young’s modulus can be calculated from the resonance frequency. The accuracy of the method is determined by the measurement accuracy of the length, thickness and mass density. The effect of air damping on the resonance frequency can be calculated as [5]

\[ f_{res} = f_0 \sqrt{1 - \frac{1}{4Q^2}} \]  

Q is the quality factor, which describes the coupling of input energy to the output resonance energy. The quality factor is dependent on the acoustic radiation, internal friction and fatigue processes, but mainly determined by the damping of the environment. Q can be calculated as

\[ Q = \frac{w}{24\mu} \left( \frac{t}{L} \right)^2 \]  

or simply read from the resonance peak

\[ Q = \frac{f_{res}}{\Delta f_{FWHA}} \]  

where \( \Delta f_{FWHA} = \) full with at half amplitude, the 3dB point  
\( \mu = \) viscosity, \( \mu=1.8x10^{-15} \text{ Ns/m}^2 \) in air

The effect of air damping on the resonance frequency is negligible in our case as it generated less than 0.1% deviation in the resonance frequency, but determines the quality factor. The calculated results show good agreement with Finite Element Analysis (FEA) simulations.
3. EXPERIMENTAL

3.1. Measurement techniques

The resonance frequencies of cantilever beams were measured in order to characterise the Young’s modulus of the film. Monitoring the resonance frequency vs. time gives information about the long-term stability of the resonators. The samples were tested in different environments in order to characterise the influence of the environment on the resonant properties. The resonant tests followed two different concepts. On one hand the sensitivity of the samples was crucial, while on the other hand more robust samples were designed for long-term harsh experiments.

3.1.1. AFM technique

Optical readout technique and external mechanical excitation was implemented to achieve high measurement accuracy while keeping the samples simple. The later is very important in order to identify and characterise the different degradation processes on the samples. On-chip excitation and detection requires more complicated, multi-layer structures, which makes the evaluation more difficult. To combine optical readout and external mechanical driving techniques, atomic force microscopy (AFM) is implemented. The AFM utilized in the field of reliability is described in details in our previous study [6]. Park Scientific Instruments’ AutoProbe M5 and Digital Instruments’ MultiMode SPM is used. The sample chip with the cantilevers is mounted in the AFM head. The cantilever is excited mechanically at swept frequency; while the deflection of the free end is measured continuously with a deflected laser beam, see Fig. 1. The typical excitation amplitude of the sweep is a few nanometers. The maximum displacement of the cantilever’s free end in resonance in air is 4-5 times bigger than the excitation amplitude. This deflection is in the elastic range, as the thickness of the used cantilevers is 180-8000nm. The measured peak gives the resonant frequency of the cantilever beam. This resonance frequency measurement technique provides very high accuracy and reproducibility of 10Hz, which corresponds to measurement accuracy better than 0.05% in case of our samples. The samples were driven in resonance for a long time to characterize the long-term stability. Mechanical shock tests and large deflection test were also conducted in the AFM head. The built-in piezoelectric actuator in the AFM head applied mechanical shock to the sample. The large deflection was realized by resonant driving at higher excitation amplitudes or externally by an AFM cantilever. Repeated shock tests were conducted with 5sec resonant driving at a ten times higher excitation level than the one used for the resonance frequency determination. The resonance frequency is monitored constantly during the shock tests. The AFM operated in an environmental chamber filled with air, humid air, nitrogen rich and argon rich atmosphere to study the influence of environment on the resonant characteristics and on the shock response. All the silicon nitride, silicon carbide and crystalline silicon samples were tested with the AFM method.

Fig. 1 Resonance frequency measurement in the AFM head
3.1.2. Piezoresistive technique

To investigate environmental effects, some of the tests need to be independent from the AFM and from clean room conditions. The chips are glued on a multi-layer piezo actuator, and mechanically excited at swept frequency, see Fig. 2. The deflection of the cantilevers is detected with implanted piezoresistors. The piezoresistors form half-Wheatstone-bridges with reference resistors on the substrate. The function generator and the detecting oscilloscope are controlled with a computer. The resonance frequency is measured regularly, and the sample is driven in resonance for weeks. Cycling tests were conducted on the crystalline silicon beams and paddle-beams in ambient air and in “dusty” environment.

Fig. 2 Si resonators glued on piezo-actuators in package

3.2. Sample preparation

The samples were fabricated in Delft Institute for Microelectronics and Submicron Technology (DIMES). Cantilever beams with different shape and size were patterned in low-stress silicon rich silicon nitride and low-stress silicon carbide films, see Fig. 3. The 0.225GPa low-stress amorphous SiN film was deposited from DCS and NH₃ gases with LPCVD on 850°C [7]. The film thickness is 320nm, 500nm and 800nm. The SiC cantilever beams were patterned in 180nm thick low-stress PECVD SiC film deposited on 400°C. The typical width and length of the rectangular cantilevers were 15-40μm and 75-250μm respectively. Reflective aluminium pads were deposited on the free ends of the cantilevers for optical readout in the AFM measurements. The structures were etched free with topside wet etching in 25% 80°C TMAH. The aluminium was protected during etching with a silicon dioxide layer. There is a 1-2μm wide undercut at the clamping of the beams as the silicon is etched slowly in the (111)-direction as well. The width of the undercut region depends on the etching time. Finite element simulations (FEA) showed that the resonance frequency of an undercut beam agrees to a close approximation with the resonant frequency of an ideally clamped but longer beam. This approximation holds for the first resonance mode of all the used beams. Therefore the effective length of the beam is the designed length plus the undercut width. The more robust, crystalline, n-type silicon beams and paddle-beams were fabricated by topside etching, see Fig. 3. The applied contactless galvanic electrochemical etching technique is described in reference [8]. Implanted piezoresistor strain-gage stripes detect the deflection of the beam. The beams are 7μm thick and 300-600μm long; the paddles are 300μm wide. The gold metalization for electronic interconnects and reflective pads was deposited on top of chromium layer for better adhesion. The substrate size of all chips is such that they can be mounted in the AFM head.
4. RESULTS AND DISCUSSION

4.1. Resonator characterization

The silicon nitride and silicon carbide cantilever beams are characterized in details elsewhere [9, 10]. The cantilever beams were tested in the AFM head. The quality factor in air was determined from the resonant peak with Eq. 3 and 4. \( Q = 30 \, \text{–} \, 60 \) in air depending on the shape and thickness. The Si resonators have the highest quality factor. The Young’s modulus was calculated from the resonance frequency considering the extra mass of the reflective pad on the free end of the beams. The Young’s modulus is 230GPa for SiN and 320GPa for SiC. These values accord with the literature. The air damping was calculated with Eq. 2. The damping effect was neglected, as it generated less than 0.1% deviation in \( f_{\text{res}} \). The calculated results show good agreement with FEA simulations.

![Optical microscope picture of SiN and SiC cantilever beams (left) and Si paddle-beams (right).](image)

**Fig. 3** Optical microscope picture of SiN and SiC cantilever beams (left) and Si paddle-beams (right).

4.2. Long-term stability

4.2.1. Stiffening effect

Monitoring the resonance frequency gives information about the long-term stability of the resonator. The silicon nitride and silicon carbide cantilever beams showed a stiffening effect during the long-term AFM resonant tests in air. The resonance frequency increased gradually in air, see Fig. 4. This behavior is independent from the mechanical driving; the samples that stood still between the measurement points showed the same characteristics. The shapes of the curves are consistent, while curves measured on samples with different shape and size have different slopes. The two dominating factors, which determine the curve, are the thickness of the cantilever and the age of the cantilever. Figure 4 shows the behavior of SiN cantilever beams with different thickness. The 2µm thick SiN and the 7µm Si cantilevers did not perform the stiffening effect at all. The stiffening problem can be overcome by designing more robust, thicker resonators. Figure 5 shows the stiffening behavior on a several months old sample and after a buffered hydrogen fluoride (BHF) dip. The BHF removed the native oxide from the surface creating fresh sample again. Old samples have weaker stiffening effect than fresh ones. The stiffening effect is a long-term degradation process. The resonant mode device needs frequent recalibration. The resonator is considerably stable after a few months burn-in time; so accelerated burn-in techniques can be a solution.
4.2.2. Shock response

Another interesting phenomenon is the shock and large-deflection response of the thin SiN and SiC cantilevers. The shock and the large deflection generates an abrupt drop in the resonance frequency, up to a few percent, see Fig. 6. Following the mechanical shock, the resonance frequency increases again, as according to the stiffening effect. The shock response is stronger in case of thin cantilevers. The shock sensitivity is a major stability problem of the resonant mode devices. Robust design decreases the shock sensitivity, but loses measurement sensitivity as well.
4.2.3. **Environmental influence**

The height of the negative frequency step is proportional to the magnitude of the shock or large deflection. Small shocks generate resonance frequency drops, which fully recover in a few minutes. Repeated shock tests were conducted with the AFM in various environments. The thin silicon carbide and silicon nitride cantilevers behaved in a similar manner \[9, 10\]. The curves in Fig. 7 show the results of repeated shock tests conducted on a SiN cantilever beam in various environments. Each curve is an average of 8-10 shock tests. The deviation of the measurement points is 10-20%. We can conclude from the tests that the resonance frequency drop and recovery effects are stronger in humid environment, than in air. On the other hand, nitrogen and argon rich environments weaken the effects. The results suggest atmospheric packaging solution to overcome the shock response problem. Note, that atmospheric packaging is much cheaper than vacuum packaging. Packaging in pure argon or nitrogen atmosphere is satisfactory, if vacuum packaging is not required to obtain high quality factor for instance. The shock response is stronger on thinner cantilevers. Samples with thickness higher than 2µm did not show considerable response to the shocks applied in the tests.

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**Fig. 6** Stiffening effect on a SiN cantilever. Mechanical shocks generate negative step in the resonance frequency.

**Fig. 7** Repeated shock tests on a SiN cantilever in various environments.
4.3. Model

The following model can explain the stiffening and shock response effects. There are two parallel processes taking place on the surface: oxidation and adsorption. The surface of the silicon nitride and silicon carbide oxidizes when it is exposed to air or water vapor [11]. The formed graded native oxide layer is in compressive stress introducing tensile stress in the structure. The beam surface exposed to the environment adsorbs atoms and molecules, typically oxygen, carbides and different organic molecules. The adsorbed layer changes the surface stress of the cantilever [12], [13], [14]. As the surface stress can turn to compressive, the spring constant (K) and the resonance frequency increase. The mass of the system also increases, which leads to decrease of the resonance frequency. The two effects together determine the resonance frequency of the resonator:

\[
f_{\text{res}} = \frac{1}{2\pi} \sqrt{\frac{K + \delta K}{m^* + \delta m^*}}
\]  

(5)

where \(m^* = nm\) is the effective mass of the beam. The effect of the surface stress introduced by the surface oxidation and adsorption can be considered as force acting on the free end of the cantilever beam. Incorporating it in the Rayleigh method, the maximum kinetic energy of the vibration equals the potential energy of bending increased with the work done by the tensile force during bending [15]. From these calculations we obtained a compressive stress of 3MPa in the surface layer formed in a week on silicon nitride cantilevers.

When shock or large deflection is applied to the sample, the surface layer cracks, as this is the most strained part of the structure during bending of the cantilever. Accordingly, the resonance frequency drops. The cracks in the oxide layer leave fresh nitride or carbide surface exposed. After the shock the surface layer and thus the resonance frequency recovers. The recovery is logarithmical in time, which is a typical behavior of coverage dependant processes like adsorption. The several months old samples having thicker native oxide on their surface showed weaker stiffening effect and less sensitivity to shocks. This consideration offers the possibility of thicker oxide layer deposition on the surface, which would withstand the mechanical shocks. The reproducible shock response during the repeated shock tests suggests that a statistical large number of microcracks are generated on the surface, instead of a few macrocracks. In case of larger shock or deflections, more severe damage occurs, which cannot recover even in a few days (see Fig. 6). Probably not just the adsorbed and the native oxide layers are cracked, but also the cantilever itself. The various environments used in the tests influence the adsorption and oxidation rate. Higher humidity increases the oxidation rate; therefore the shock recovery effect is faster. The initial negative step of the resonance frequency is larger as well, because the surface layer is thicker. Argon and nitrogen are inert gases in respect of oxidation, so the shock recovery effect is slower.

The resonance frequency shift was simulated with finite element analysis (FEA) software (ANSYS and FEMAP) using the calculated surface stress. The results were in good agreement with the measured frequency shift.

4.4. “Flycatcher” effect

Cycling tests were conducted on the crystalline silicon paddle-beams and cantilever beams in air. No stiffening or shock response effects were found on these robust structures, but the resonators showed another failure process. Electrostatic charging occurs, because of the high frequency vibration in air (30-80kHz). The beams gather airborne particles from the ambient air, as a flycatcher. The extra mass lowers the resonance frequency, see Fig. 8. The mass loading can be calculated from the resonance frequency shift:

\[
f_2 = f_1 \left(1 - \frac{1}{2} \frac{\delta m^*}{m}\right)
\]  

(6)

The adsorption rate was \(3.6 \times 10^{13}\) kg/sm², depending on the driving amplitude and on the content and size of the particles. Figure 9 shows that only the beam in resonance gathers dust particles, because this has much higher vibration amplitude than the neighboring beams. Proper cleaning of the chips is crucial before packaging, because most of the contamination on the resonating beam comes from the chip itself. The sticking gold/photo-resist clusters remaining from the lift-off process could not be removed with acetone, but come off the surface during the vibration, and stick on the vibrating beam, see Fig 9 left.
A gold-coated paddle-beam was driven in resonance in air. Twenty hours later fine smoke particles were introduced in the measurement chamber. The resonance frequency dropped abruptly, see Fig. 10. The adsorption rate calculated from the steep linear region of the frequency response curve was $2.3 \times 10^{-9}$ kg/sm². The dust particles settled in the chamber in approximately 10 hours; the resonance frequency reached its minimum at this time. Then the resonance frequency increased again indicating that the beam releases slowly some of the dust particles. Again, only the resonating beam collected the airborne particles, the rest of the chip remained clean (Fig 9 right). These results point out that even if vacuum package is not necessary, protective package is required against dust for ambient applications to maintain stable resonance frequency.

![Graph](image.png)

**Fig. 8** Resonance frequency decrease during cycling test on two Si paddle beams

![Images](image1.png)

**Fig. 9** Si paddle-beam after few hundred hours vibrating in air (left), gold coated paddle-beam vibrating in dusty environment (right).
5. CONCLUSIONS

The goal of this work is to characterize the reliability and stability problems related to resonant mode micromechanical devices operating in ambient environment. Using these results we try to set up design rules for building low cost devices. The resonant operating mode offers outstanding sensitivity and convenient signal processing. Unfortunately the resonant structures based on thin membranes, beams or bridges are very sensitive to undesired environmental effects as well. Vacuum packaging largely increases the fabrication costs, and not applicable in applications, where the resonant structure is in direct contact with the medium. Therefore it is necessary to study the undergoing degradation processes on resonators exposed to the environment. Thin silicon nitride and silicon carbide films show a gradual stiffening effect in air. The resonance frequencies of the cantilever beams increase in time, which would require frequent recalibration of the device. This behavior is explained with adsorption and oxide formation on the surface, introducing surface stress in the structure and increasing the overall stiffness. FEA modeling supports this theory. The surface layer is very sensitive to mechanical shocks and large deflections. Cracks are generated in the layer resulting in an abrupt drop of the resonance frequency. The cracks recover in air after the shock, and the resonance frequency increases again. This unstable resonant behavior is a major reliability problem of the thin resonators. Repeated shock tests in various environments proved that humidity increases, while inert gases like argon and nitrogen decrease the stiffening and shock response effects. Atmospheric packaging in inert gases is one solution to the problem. More robust design eliminates the problem, losing measurement sensitivity though. No degrading effects were observed on films thicker than 2µm. The third solution is growing thicker oxide on the surface, which withstands the shocks applied to the device. Another stability problem was observed on silicon paddle-beam resonators in ambient air. Electrostatic charging occurs on the vibrating beam and airborne particles stick to the surface. The extra mass lowers the resonance frequency. As the floating particle content of the surrounding environment changes, the beam can gather more particles, or slowly release some of it. This effect can result in huge fluctuations of the resonance frequency and points out the necessity of dust free packaging. Special care is required to eliminate the electrostatic charging on functionalized probe tips, which exploit the mass increase – resonance frequency shift relation.

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