Reliability of Micromechanical Thin-Film Resonators
Reliability of Micromechanical Thin-Film Resonators

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# Table of Contents

1 Introduction

The structure of the book

References

2 Reliability Issues

2.1 Mechanical properties

Elastic properties
Strength
Bonding
Residual stress

2.1.1 Thin films in the submicron range

2.2 Failure mechanisms

Stiction and friction
Delamination
Creep

2.2.1 Fatigue

Cracking
Environmental corrosion

References

3 Thin-Film Resonators

3.1 Sample preparation

3.1.1 General comments

3.1.2 Silicon nitride cantilevers

3.1.3 Silicon carbide cantilevers

3.1.4 SiN/SiC multilayers

3.2 AFM measurements

3.2.1 Scanning probe microscopy

Utilizing the AFM
3.2.2 Quasi-static and fatigue tests ........................................ 38
  Spring constant ................................................................. 40
  Young’s modulus ............................................................... 42
3.2.3 Resonant tests ............................................................ 44
  Quality factor ................................................................. 46
  Air damping ................................................................. 47
  Young’s modulus ............................................................... 49
3.2.4 Large deflection and shock tests..............................  50
3.3 Stiffening effect ..........................................................  51
  3.3.1 Adsorption on solid surfaces ...................................  51
  3.3.2 Surface stress and related effects.............................  53
    Quality factor ...............................................................  56
    Resonance frequency ....................................................  57
    Experimental determination of the surface stress .... 63
  3.3.3 Measured resonance frequency shift .......................  64
    Surface oxidation ..........................................................  65
    Thickness dependence ..................................................  66
    Age dependence ...........................................................  68
    Analytical results ......................................................  70
    SiN vs. SiC .................................................................  71
    Surface passivation .....................................................  72
3.4 Shock response..........................................................  73
  3.4.1 Cyclic shock tests ....................................................  75
3.5 Environmental tests.......................................................  77
3.6 Numerical calculations - FEA ............................................  82
  3.6.1 SiN and SiC cantilevers...........................................  82
    Stiffening effect ..........................................................  82
    Undercut of Si (111) plane ...........................................  86

References ...........................................................................  87

4 Resonator Beams ........................................................................  93
  4.1 Sensor design .............................................................  95
    4.1.1 Process flow .........................................................  97
    4.1.2 The structures .......................................................  97
4.2 Theory ................................................................................. 99
4.2.1 Beam theory .................................................................... 99
4.2.2 Paddle beam theory ..................................................... 102
4.2.3 Beam vs. paddle beam ............................................. 102
4.3 Measurement techniques .................................................. 105
4.3.1 AFM measurements ................................................... 105
4.3.2 Piezoresistive measurements .................................... 106
4.3.3 Alpha-stepper bending tests .................................... 107
4.4 Measurement results ....................................................... 108
4.4.1 Quality factor ............................................................. 108
4.4.2 Resonance frequency ............................................... 108
4.4.3 Accelerometer calibration ....................................... 110
4.4.4 ‘Flycatcher’ effect ..................................................... 114
  Resonant air pollution detector .................................. 120

References ................................................................................. 125

5 Pressure sensors & Accelerometers ......................................... 129
5.1 Resonant microsensors .................................................... 129
  5.1.1 Resonant pressure sensors ................................... 133
  5.1.2 Resonant accelerometers .................................... 134
5.2 Sensor design .................................................................... 135
  5.2.1 Three-dimensional resonator bridge ................. 136
    Air damping and squeeze-film effects ..................... 139
  5.2.2 Reinforced membrane .................................. 143
  5.2.3 Excitation and detection .................................. 144
5.3 Sensor fabrication ............................................................ 144
5.4 Results ............................................................................. 147
5.5 Measurements .................................................................. 148

References ................................................................................. 149

6 Conclusions .............................................................................. 151
Chapter 1

Introduction

In the past few decades, the development of electronics reached a high level, changing our work and everyday life. The natural step that follows is interfacing our non-electronic world to the electronic one. Development of microsystems that can gather information from the environment, feed it to already available systems having vast computational power and/or share it through the worldwide network to distant units is crucial. The environment can be probed and controlled by means of sensors and actuators, which is called transduction. The actuators receive and execute the commands of the processing unit, which handles the input of the sensors. The further development of transduction systems should result in further penetration of electronics into our society.

Micro Electro Mechanical Systems (MEMS) combine integrated sensors, microactuators and low-power electronics. This field has recently been growing rapidly. MEMS technology is based on the already mature microelectronics technology, supplemented with special micromachining techniques. Owing to their fast and continuous development, these MEMS specific technologies are already partly standardized and available for industrial mass production. The mechanical structures, sensors, actuators and electronic circuitry are all formed on a common substrate, though advanced multi-layer MEMS devices are built of a stack of silicon and/or glass substrates. Emerging applications like accelerometers, gyros, inkjet print heads, projection displays, microphones and IR/thermal imagers are all based on MEMS technology. The extremely high reliability and accuracy of these microsystems are achieved at low cost thanks to the
embedded microprocessors employing self-testing and digital compensation [1.1].

MEMS is a relatively new field with impressive achievements and great potential. Numerous new characterization and testing methods have been developed, but still many more are required for better fundamental understanding of the failure mechanisms and for improvement of the stability and reliability of the MEMS devices. This is also of key importance for commercializing today’s advanced micromechanical devices. Material properties and degradation processes on the size scale of MEMS can essentially differ from those in bulk materials. Due to the small size and extreme aspect ratio of MEMS devices, crack-related mechanisms and surface effects can be much more significant in these devices. Stiction and friction of moving parts can build up very high local stress fields, for example when a MEMS gear starts rotating [1.2]. Therefore, materials and failure mechanisms that are fairly well characterized on the macro scale require further investigations using micromachined test structures. The fabrication process, size, and shape of the test structures should approach those of the actual MEMS device under examination. Development of highly sensitive, small-sized devices involves accelerated ageing tests, failure analysis and design optimization accordingly.

These considerations motivated us to study the failure mechanisms, long-term stability and reliability of and the environmental effects on thin-film resonators. Micromechanical test structures were designed and fabricated. Static and dynamic tests were carried out to characterize the material properties and to study their mechanical reliability with special emphasis on resonant test structures and resonant mode sensors.

The structure of the book

Chapter 1 introduces a general view of the motivation and circumstances of the study.

The reliability problems of micromechanical structures are described in chapter 2. Though many of the materials used in micromachining are fairly well characterized on the bulk level, MEMS designers encounter new issues. The material properties and failure mechanisms can be substantially different on the micro scale. The material properties depend on the fabrication conditions. Emphasized stress and surface-related
failure modes, cracking, aging and fatigue occur for micromechanical components. These considerations point out the necessity of using micromechanical test structures.

Depending on the dimensions, especially the thickness, thin-film structures suffer from different failure mechanisms. Therefore we would like to distinguish between thin films with thicknesses below and above 1 micrometer. Of course there is no clear dividing line: both groups suffer from all reliability problems, but to different degrees.

Chapter 3 discusses thin films with thicknesses up to 1 micrometer, where the surface-related failure mechanisms are the dominating ones. Particularly submicron-thick silicon nitride and silicon carbide resonators were tested. Both these materials are widely used in MEMS structures due to their outstanding mechanical and chemical properties. The quasi-static and resonant tests are carried out using an atomic force microscope head. The Young’s moduli of the thin films are calculated from the resonant measurements. The aging and the reliability are studied with cycling tests and shock tests in various environments. Adsorption- and surface-oxidation-induced reliability problems occur for the cantilever beams fabricated from the thin films. The stability of the resonators strongly depend on the working environment and on the applied mechanical shocks. The measured results are compared to analytical calculations and numerical FEA simulations.

Chapter 4 concentrates on resonator beams with thicknesses in the order of a few micrometers. Crystalline silicon beams and paddle beams with implanted piezoresistors are fabricated and characterized. The structures are implemented in applications such as vibration sensors and accelerometers. Though surface-related effects are less significant, other failure modes are present, which have an impact on the applications and determine the minimum requirements for packaging. The high-frequency vibration generates electrostatic charging resulting in adsorption of airborne particles from the environment. This so called ‘flycatcher’ effect leads to a long-term stability problem, false sensor output or eventually failure of the driving function. The ‘flycatcher’ effect is also exploited for sensing purposes. An air pollution sensor is demonstrated, where the sensing element is reset with an electrical signal.

What we learn from the resonant tests about the long-term stability of thin film resonators is implemented to build new, low-cost, resonant-mode pressure sensors and accelerometers, presented in chapter 5. The resonant
mode provides high sensitivity and convenient signal processing. The structures are based on locally reinforced silicon nitride membranes and three-dimensional silicon nitride bridges as sensing elements. This double mechanical structure allows us to optimize separately the membrane and the bridges for the workload and for the most efficient driving and sensing. Another advantage of the structures is that the membrane isolates the sensitive bridges from the working environment. The bridges are designed such, that they serve as mechanical amplifiers. The different sensors use thermal and external mechanical excitation combined with piezoresistive and optical readout. Reference resonators can be used for self-calibration. The bridges do not require vacuum packaging, only low-cost atmospheric packaging.

Finally, chapter 6 concludes the thesis.
References

Chapter 1 Introduction


Chapter 2

Reliability Issues

MEMS is a rapidly emerging field, for which an impact similar to that of microelectronics in the past few decades is expected. As MEMS is still at the beginning of its evolution, great effort is required for gaining a better understanding of the failure mechanisms and improving the performance and reliability. Due to the submicron to milimeter size scale of MEMS devices, their material properties can substantially differ from the ones found in bulk materials. Even not inherently scale-dependent properties like density and Young’s modulus can be altered from bulk values by creation of non-equilibrium structures. For instance, bulk silicon nitride is a sintered ceramic material with a grain structure. In contrast, the thin film silicon nitride is mostly formed by low-pressure or plasma-enhanced chemical vapor deposition (LPCVD, PECVD), and usually amorphous. Depending on different process conditions, such as the gas ratio, flow rate, or temperature, a wide variety in chemical composition, structure, residual stress, strain, Young’s modulus, refraction index and etch rate can be obtained [2-1]. Due to the different microscopic structures and scaling effects [2-2], the material, chemical and electrical properties of thin films (e.g. toughness, Yield strength, fracture strength, conductance, chemical inertness) can be substantially different from those of bulk materials. This can extend the application boundaries of micromechanical devices.

Owing to the small size and extreme aspect ratio of MEMS devices, new failure mechanisms, such as crack-related and surface effects can play a greater role. Environmental effects, like adsorption/desorption-induced surface stress, can change the mechanical, electrical and resonant...
properties of thin-film structures. On the other hand, this effect can also be exploited for sensing purposes. The microcantilever-based physical, chemical and biological sensors allow part-per-billion (ppb) to part-per-trillion (ppt) detection sensitivity.

2.1 Mechanical properties

Micromechanical and lifetime evaluation tests on micrometer-sized microelements help to establish design criteria for reliable micromachines. In conventional techniques such as X-ray diffraction [2-3] or wafer buckling [2-4] macroscopic specimens are used to measure mechanical properties. Raman spectroscopy is suitable for stress measurements on a microscopic scale [2-5] [2-6]. However, these techniques require previous knowledge of the elastic properties. Thus, accurate measurements of these parameters are necessary, which can only be achieved using miniaturized test probes approaching the film thickness and dimensions of the microstructures. One needs accurate data about the actual mechanical properties when designing micromechanical systems, e.g for realistic simulations, and numerical and analytical calculations. Micromechanical testing machines have been developed for quasi-static bending [2-7] and tensile [2-8] tests where piezo-electric loading and in situ monitoring are implemented in a scanning electron microscope. Other loading methods besides the piezo-electric one [2-9] [2-10] are DC motor [2-11] and electro-magnetic [2-12] [2-13] based methods.

Elastic properties

Elastic properties are mainly tested using microfabricated test structures: cantilever beams and diaphragms. The test structures can be statically deflected by electrostatic or external mechanical loading, or can vibrate at their resonance frequencies. These techniques are discussed in detail in chapter 3.
Mechanical properties

Strength

There are different approaches to characterize the strength as well. Nano-indentation is used for plastic materials [2-14], while deflection-induced fracture tests [2-15] [2-16] and tensile tests [2-17] are used to determine the fracture toughness. Simple deflection based in situ fracture tests can be carried out on cantilever beams in a scanning electron microscope [2-18]. These tests gave a number of fundamental fracture parameters. In tensile tests an inplane pulling force parallel to the sample beam is applied providing straightforward evaluation and reliable results for tensile strength and Young’s modulus. The inplane loading itself presents experimental difficulties. The load can be applied using electrostatic gripping [2-19] or special micromachined integrated test chips [2-20].

Bonding

Another key parameter is the bond strength of the multiple layers of which the MEMS devices consist. The adhesion of thin films is usually measured through bulge tests [2-21], peel tests and residual stress driven cohesion tests [2-22] carried out on micromachined test structures. An application used more specifically for MEMS is wafer bonding, where the most commonly used techniques are anodic and fusion bonding. The quality of the bond is characterized by non-destructive techniques such as infrared, X-ray and ultrasonic imaging. The bond strength can be studied by pressure burst testing, double cantilever beam specimens [2-23] and other mechanical loading tests applying tension and shear stresses on the bond.

Residual stress

Residual stress is a well-known and well characterized phenomenon in the electronics industry. As MEMS devices are based on multilayer structures, they suffer from residual stress, especially components with a higher film thickness (e.g. LIGA or CVD SiC). As a result of high residual stress the film can fracture. The source of the residual stress can be thermal expansion mismatch, lattice mismatch, residual gases in deposited materials, grain growth and size, point defects and sintering. By characterizing and controlling the residual stress in the structures, one can obtain devices with high performance and improved reliability. Several
test structures and methods have been developed for measuring residual stress that implement static or dynamic loading and resonance frequency analysis.

A large group of the characterization methods implement released, free-standing structures fabricated of the thin film. Measuring the deflection (rotation at the junction of the film and substrate) and curvature of a free-standing cantilever beam gives information on the mean and gradient components of the residual stress in the thin film [2-24]. Another way to determine the stress in the thin film is measuring the load-deflection behaviour of thin suspended membranes with a nano indenter [2-25] [2-26]. This method is suitable to measure tensile stresses, typical in silicon nitride films, and film stiffness. Besides the Young’s modulus, the residual stress can be determined by measuring the fundamental resonance frequencies of thin double-clamped plates [2-27]. As the thin film can interact with the substrate underneath, changing the mechanical behaviour, these tests are not perfectly representative of the thin coating well adhered to the substrate.

Other methods measure the bending of coated cantilevers, which allows realistic evaluation of the mechanical behaviour of multilayer systems. The evaluation theory often pre-assumes knowledge of the elastic properties of the thin films and/or the substrate [2-28], which is in practice not available or unreliable in case of the micromachined structures. A model was developed using only geometrical parameters and experimental load-deflection data. This model is based on equilibrium relationships for the forces and moments, avoiding the elastic constants, and works both in the plastic and elastic film-strain interval [2-29].

2.1.1 Thin films in the submicron range

As thin films are used more and more frequently in microelectronics, data storage and MEMS, their mechanical characterization becomes more and more important. The reliability of such devices is determined by the response of the thin film material to stresses developed during film deposition, device fabrication, or external loading due to operational and environmental conditions. Investigations of such thin films with typical
thicknesses of 100nm or less is important for device design, fabrication, and for understanding the fundamentals of nano-scale material science.

Uniaxial tensile tests provide the most suitable data for determining Young’s modulus, yield and ultimate strength, although when they are applied to nano-scale samples, certain difficulties occur [2-30]:

- The tensile tests require small forces, in the order of micro-Newton. The problem is usually avoided through the use of tougher freestanding structures with large cross sections [2-31], or thin film-substrate or multi-layer combinations [2-32] are tested. Thicker samples provide less interesting size scale effects, while increasing the width relative to the thickness makes it difficult to apply uniform tensile force across the width. Compliant substrate or multi-layers introduce substrate or interface effects [2-33].

- Fabrication of stress-free, free standing structures is difficult. Wet etching and peeling off of the substrate leaves significant pre-stress, which existing tensile testing techniques are not able to measure.

- Gripping and aligning the nano-scale freestanding structure contributes to pre-stress. Even slightly misaligned specimens may suffer from an unwanted bending moment, leading to premature failure.

2.2 Failure mechanisms

What are the main factors determining the performance and stability of MEMS devices? In most applications, mechanical robustness is not an issue, as the implemented structural materials (Si, poly-Si, SiN etc.) are well controlled and have outstanding mechanical properties. The long-term stability and reliability of MEMS devices can be monitored through the change of mechanical properties. The development of accelerated testing techniques and protocols have revealed new failure mechanisms of micromechanical components. The effects of the microscopic structure, fabrication and environment becomes significant on the micro scale.

Though relevant to MEMS, the reliability issues of the electronics and packaging are beyond the scope of this thesis.
Reliability Issues

Stiction and friction

The forces relevant to the behaviour of MEMS devices are different from those in macroscopic devices. Due to the high surface-to-volume ratio, gravitational forces are negligible, while surface-related forces associated with contact and rubbing are dominant. Stiction and friction have a great influence on the performance and reliability [2-34]. Besides mechanical wear, high local stress fields generated by rubbing surfaces have serious consequences on the lifetime of the device. Modifying the micromachined surfaces [2-35] or applying low-friction coatings [2-36] results in improved tribological characteristics. Another approach is the development of air bearings [2-37] and magnetic levitation.

Delamination

The fabrication process, thermal mismatch or epitaxial mismatch can introduce high stresses in multilayer structures. When the forces acting in the film overcome the adhesion between layers and/or substrate, delamination occurs. The delamination can initiate from a free surface, or above an intrinsic defect, reliving compressive stress with buckling [2-38]. Spalling accompanies the buckling and delamination in case of brittle films.

Creep

Creep is a stress-induced time-dependent mass transfer governed by dislocation and vacancy movements, glide and diffusion mechanisms. Creep can have an enormous impact on the MEMS scale, especially in metals where creep may occur at room temperature.

2.2.1 Fatigue

Cyclic mechanical deformations and loads can lead to failure of a mechanical component, even though the strains never exceed the ultimate ductility of the material. This failure mechanism called fatigue is a result of incremental damage accumulated during the load cycles [2-39]. Any
Failure mechanisms

process resulting in an irreversible repositioning of atoms in the material can contribute to fatigue.

Micromechanical devices may operate at high frequencies, subjecting them to a very high number of fatigue cycles during their lifetime. Fatigue can be a limiting factor for the allowable stress levels or useful lifetime. Fatigue processes can take place in both ductile and brittle microfabricated materials. Long cycling tests showed that dislocations pile up at crack tips in metals, which leads to long cycle fatigue. In contrary, polysilicon, the most frequently used mechanical material in surface micromachined structures, is a brittle material with no dislocation movement prior to fracture. The fatigue mechanism in polysilicon is based on the fatigue of the native oxide layer, as described below in the section “Environmental corrosion”. The fatigue strength of microelements and the influence of water on the fracture mechanism have been studied with quasi-static bending tests [2-40] [2-41]. The fracture surface is examined with scanning electron microscopes and scanning atomic force microscopes which enables nanoscopic 3-D damage evaluation [2-42].

Cracking

Fatigue is a wear out failure mechanism based on crack initiation and propagation. Cracks may develop in ductile materials at local stress and plastic strain sites, such as defects (e.g. grain boundaries) and other discontinuities [2-43]. The crack can propagate stably under cyclic stresses. The fatigue crack propagation can be modelled with continuum fracture-mechanics theory. Plastic deformation involves dislocation slip motion, while creep (static fatigue) is associated with dislocation climb, vacancy migration, diffusion and grain boundary sliding.

MEMS devices often operate at stress levels in the order of 1GPa, which are at least an order of magnitude higher than stress levels in macroscopic structures. The fabrication process (e.g. etching, polishing) and rough handling can introduce small defects, which act as critical cracks at these stress levels. Crack generation and growth can be monitored on micromechanical structures with resonant cyclic tests [2-44]. Stress concentration was introduced in a beam with the mask set to investigate the crack initiation. The crack propagation was studied on cracks introduced by a nanoindentor. Attention should be paid to the crack
Reliability Issues

initiation process, as the crack growth period is a fraction of the crack initiation period in most MEMS applications.

**Environmental corrosion**

MEMS devices, such as valves, sensors or pumps are designed to operate in various gases and fluids. Environmental effects can exacerbate the failure mechanisms and introduce new ones as well. Moisture has a particularly high impact on the long-term stability of thin-film resonators. Polysilicon resonators were tested in humid air [2-45]. Stress cycling in high relative humidity led to noticeable changes in the mechanical properties. Surface oxidation increased the stiffness and hence the resonance frequency of the resonator. Corrosion fatigue of the native oxide on the surface generated a time-dependent failure mode. Crack generation and propagation in the presence of moisture is believed to be the key to failure/fracture of the polysilicon resonators [2-44]. The same mechanism was found in silica [2-46]. Thus the failure mechanism of brittle materials is not a cyclic fatigue, but rather an environmentally assisted slow crack growth process. Fatigue and fracture of single-crystal silicon was investigated with resonant tests [2-40]. Propagation of cracks introduced by a nanoindenter was monitored by measuring the resonance frequency. Fatigue of the native surface silica layer is identified as the mechanism for crack growth. Increasing relative humidity greatly enhances the crack growth rate [2-47]. A similar surface oxidation-induced stiffening effect was found in amorphous silicon nitride and silicon carbide cantilever beams [2-48]. Surface oxidation resulted in an unstable resonance frequency; in a stiffening effect and a degrading shock response.
Failures mechanisms

References

Chapter 2 Reliability issues


References

2-30 M.A. Haque, M.T.A. Saif, “Application of MEMS force sensor for in-situ mechanical characterization of nano-scale thin films in SEM
and TEM”, to be published in Sensors and Actuators A, 2001


2-35 Z. Rymuza, Microsystems Technologies, 5, p. 173, 1999


Chapter 3

Thin-Film Resonators

Thin films are the basic building blocks of micromechanical structures. They serve as supporting elements, for example membranes, or can be perforated, forming any shape from simple beams to complicated surface-micromachined three-dimensional structures. One of the first applications of micromechanical sensors are pressure sensors based on thin, diffused, bulk micromachined membranes [3.1], which are widely used in the industry today. Thin beams and membranes are the basic components of optical components (optical switches, adaptive mirrors for wave front correction etc.), which gave a boost to the optical telecommunication industry recently. Resonant mode sensors implement membranes and beams vibrating on their fundamental resonance frequency. The resonant operating mode has several advantages, such as high accuracy and digital output, which is discussed in more detail in chapter 5.1. Integrated double-clamped beams can replace piezoresistive or piezoelectric strain gages in pressure sensors and accelerometers. Submicron-thick resonator beams are very sensitive to surface-related effects, which enables applications like functionalized probes and humidity sensors. Another big application field is scanning probe microscopy, where thin cantilever beams probe various properties of the sample surface. Several applications of resonating beams are discussed in chapter 4.

Besides the various applications, thin-film resonators are frequently used as test structures to characterize the thin films they are fabricated of. This is particularly important as the mechanical properties of thin films can essentially differ from the bulk properties due to their different
microscopical structure (stochiometry, grain size, boundary cracks, dislocations, voids etc.). A trivial example is crystalline silicon, where the thin film form has a lower dynamic Young’s moduli than the bulk material, which is attributed to the heavy doping levels [3.2]. The Young’s modulus, stress and strain of boron-doped silicon films were determined by the resonant method, implementing photothermal driving [3.3]. The material properties of thin films are strongly dependent on the fabrication process, and can be changed with different deposition parameters. Residual stress, optical parameters and etching properties are just a few of the parameters, which can be tailored according to the requirements. The tests carried out on thin-film resonators can be grouped under quasi-static tests and dynamic (resonant) tests. The first group determines for example the Young’s modulus, fracture toughness and tensile strength of the thin film, or the spring constant of the micromachined mechanical members. The second set of tests is suitable for calculating the Young’s modulus and for studying the aging, fatigue, long-term stability and reliability of micromechanical structures.

3.1 Sample preparation

All the samples used in this work were fabricated at the Delft Institute for Microelectronics and Submicron Technology (DIMES). Cantilever beams of different sizes, shapes and thicknesses were patterned in silicon nitride and silicon carbide thin films. Some of the samples were coated with passivation layers at the Kluyver Laboratory at the Delft University of Technology.

3.1.1 General comments

A few general aspects were considered concerning the sample preparation. The test structures need to be as simple as possible. This allows us to evaluate the reliability problems best. In complicated, multilayered structures different failure mechanisms can be superimposed, which makes it difficult to distinguish them. Therefore our approach was to build
Sample preparation

the test structures of simple building blocks, where the reliability of the blocks is studied separately. The simplest mechanical resonators are cantilever beams. These structures are used extensively for characterising the thin-film properties and in reliability studies. The driving and the readout of the sample needs to be realised externally. To obtain accurate results we applied external mechanical driving and optical readout methods to the thin-film resonators. The measurement technique is described in chapter 3.2.2 and chapter 3.2.3. The optical readout requires a reflective surface. Reflecting aluminium mirrors were deposited on the free end of the cantilever beams. Another constraint for the sample design comes from the measurement technique. The resonant measurements and tests were conducted in an AFM head. The sample substrate needs to be such that it fits in the AFM tip holder. The chip dimensions are 1.6mm x 4.5mm x 0.5mm. The maximum usable surface on a chip where test structures can be placed is approximately 1.4mm x 1mm.

3.1.2 Silicon nitride cantilevers

Owing to its excellent mechanical, electrical and thermal properties, silicon nitride thin films are widely used in the electronics and MEMS industry. They are mainly used for supporting mechanical elements, insulator layers and protective coating layers. The first group is particularly interesting for micromachining. Extreme aspect ratio structures such as large membranes, plates or cantilevers are fabricated of thin silicon nitride film. The film is usually deposited by Low-Pressure or Plasma Enhanced Chemical Vapor Deposition (LPCVD, PECVD) and processed with standard micromachining techniques.

Cantilever beams with different shapes and sizes were patterned in low-stress silicon-rich silicon nitride film [3.5], see figure 3-1. The process flow is illustrated in figure 3-2. The SiNₓ film was deposited on the silicon substrate from dichlorosilane (DCS) and ammonia (NH₃) gases with LPCVD. French and Sarro studied the deposition conditions in order to achieve low-stress silicon nitride films [3.6]. Based on their results we used a gas-flow ratio of NH₃/DCS=0.176, deposition pressure of 150mtorr and deposition temperature of 850°C. The Si/N ratio is 0.95 in the film, there is no detectable hydrogen content, and the residual tensile stress is...
125MPa [3.6]. X-ray diffraction studies proved that the SiN\textsubscript{x} film is amorphous. The film is a mixture of Si and SiN\textsubscript{x} clusters. The structural order in the SiN\textsubscript{x} cluster is described by the Radial Distribution Function (RDS). Calculations with the RDS function lead to the conclusion that the average cluster size is 10Å [3.7]. The energetically most favorable, thus the most probable formation of the SiN\textsubscript{x} inside the clusters is Si\textsubscript{3}N\textsubscript{4}. Even if subjected to 0.5-2 hours of post annealing at 900-1000°C, the SiN\textsubscript{x} clusters are unchanged, only the Si is recrystallized into poly-Si, forming 500-2000 angstrom Si clusters. Aluminium pads were deposited on the free end of the beam for the optical readout technique. The patterned SiN\textsubscript{x} cantilevers were etched free with topside wet etching in 25% 80°C TMAH. The aluminium was protected during etching with a silicon oxide layer, which was removed with HF. Finally the chips were diced while the cantilevers were protected with a thick resist layer filling up completely the well under the beams. The resist was removed with acetone.

![Fig. 3-1 Optical microscope picture of the SiN\textsubscript{x} cantilever beams.](image)

The different cantilever beams are 0.32-0.8µm thick, 15-40µm wide and 85-325µm long. The dimensions are checked with Scanning Electron Microscopy (SEM). The uniformity of the thickness is better than 5nm at
different positions on the wafer. 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, see chapter 3.6.1 section “Undercut of Si (111) plane”. This approximation holds for the first resonance mode of all the used beams. Therefore the effective length of the beam is the sum of the designed length and the undercut width.

Fig. 3-2 Silicon nitride cantilever beams process flow.
3.1.3 Silicon carbide cantilevers

Numerous new applications need to extend the boundaries of the conventional silicon-based electronics. Silicon carbide is a material with attractive properties to satisfy many of these demands [3.8]. Due to the excellent mechanical characteristics [3.9], such as good wear and friction properties, hardness, and a high Young’s modulus, silicon carbide is used in Microsystems as hard coating and also as a mechanical material. The outstanding chemical inertness of silicon carbide offers its application as a passivation layer for aggressive media and a masking layer for microelectronics processing and micromachining. Implementation of silicon carbide extends the working range of microelectronics to high-temperature, radioactive and chemically corrosive environments, and improves high-power and high-frequency electronics. Furthermore, the electronic band structure of silicon carbide is suitable for UV sensors and other optoelectronic applications, which were not feasible with silicon technology. As silicon carbide has natural and thermal oxides, a form of micromachining can already be applied to it similar to that for conventional silicon, although many technical difficulties still need to be solved.

There are several techniques to make crystalline, poly-crystalline and amorphous silicon carbide materials. The latter is particularly interesting, as amorphous silicon carbide films can be deposited at low temperatures with conventional PECVD, while many of its favourable properties are still preserved. Residual stress is a key problem in thin films in micromechanical systems. The compressive residual stress in silicon carbide films can be reduced by thermal annealing and by using the appropriate RF power and pressure in the PECVD process, or it can be compensated by using SiN/SiC multilayers [3.10]. The low-stress PECVD SiC film is an IC process compatible micromechanical material with great potential.

The 180nm thick low-stress PECVD silicon carbide film was deposited at 400°C using Sarro’s process [3.10]. Cantilever beams with different sizes and shapes (simple rectangular and triangular) were perforated from the SiC film. The typical width and length of the rectangular cantilevers were 20-40µm and 75-250µm, respectively. Reflective aluminium pads were deposited on the free ends of the cantilevers for optical readout in the
resonant AFM measurements. The silicon carbide cantilevers were etched and diced in the same way as the silicon nitride beams, described in chapter 3.1.2. An undercut of 1-2\(\mu\)m was observed at the clamping of the beams. This is due to the slow etching of the (111) plane of the Si substrate.

3.1.4 SiN/SiC multilayers

Silicon nitride and silicon carbide multilayered cantilevers were fabricated using the LPCVD and PECVD processes described in chapter 3.1.2 and chapter 3.1.3. The silicon nitride layer tends to have tensile residual stress, while the silicon carbide is compressed. The SiN/SiC multilayer have the advantage that one can minimize the residual stress in the structure by controlling the stress in the building layers.

3.2 AFM measurements

The cantilever beams were characterised using atomic force microscopy. Cycling tests and quasi-static bending tests were carried out in the AFM. Therefore the AFM and its basic operation modes are described in this section.

3.2.1 Scanning probe microscopy

The Scanning Probe Microscope (SPM) is an imaging tool with a vast dynamic range and unprecedented resolution. Applications of SPMs are very diverse and measure physical properties such as surface conductivity, static charge distribution, localized friction, magnetic fields, and elastic moduli [3.11]. They can operate in Ultra-High Vacuum (UHV), ambient air or even in liquids. The latter allows the Electro Chemical SPMs (EC SPM) to monitor chemical and electrochemical processes, phase
formation, adsorption, corrosion, and deposition of organic and biological molecules.

The first member of the SPM family was the Scanning Tunnelling Microscope (STM) invented in 1981 by Binnig and Rohrer [3.12]. Five years later they were awarded the Nobel prize in physics for its invention. An atomic sharp tip scans above the sample surface with a gap of approximately 10Å. A bias voltage is applied between the tip and the sample. The tunnelling current flowing between the closest atoms of the probe and the sample surfaces is measured. The tunnelling current can be visualized, resulting in a 2-D image of the electronic structure of the surface, hence the topography (called ‘constant-height mode’). The tunnelling current can be used as feedback to maintain a constant tip-to-sample spacing, and then plotting the tip controlling voltage gives the surface image (‘constant-current mode’). The key in STM is the exponential dependence of the tunnelling current on the tip-to-sample distance, which provides truly atomic resolution images of the surface topography. The STM can give analytical information about the electronic properties of the surface, which is called scanning tunnelling spectroscopy. One restriction of the STM technique is that both the sample and the tip must be conductors or semiconductors.

In contrast, Atomic Force Microscopes (AFM) use another principle and can probe all conductor, semiconductor and insulator surfaces. The AFM probe, a sharp tip attached to a usually triangular shaped cantilever - see figure 3-3; scans above the sample, following the surface corrugations. The interaction force between the tip and the sample surface makes the cantilever bend. The cantilever deflection is measured with a laser beam bouncing from the end of the cantilever onto a position-sensitive detector, see figure 3-4. The surface topography image is generated from the cantilever deflections. Similar to the STM, the AFM can operate in ‘constant-height mode’ or ‘constant-force mode’ (the latter corresponds to the ‘constant-current mode’ of the STM). The interatomic force acting between the probe tip and the sample, called the van der Waals force, is plotted as a function of the tip-to-sample distance in figure 3-5. The three operational modes of the AFM are defined according to the applied tip-to-sample spacing.

- **Contact mode**: physical contact between the tip and the sample; repulsive force acts. Constant-height and constant-force scanning modes,
AFM measurements

nanolithography and force-vs.-distance surface analysis modes are available.

- **Non-contact mode**: the AFM tip is vibrated near the surface. The force gradient changes the spring constant and hence the resonance frequency of the vibrating AFM cantilever. No tip or sample degradation upon scanning, no sample contamination.

- **Intermittent-contact mode**: the tip vibrates close to the sample, “tapping” the surface. The vibration amplitude varies according to the surface topography. Suitable for scanning large topography changes.

![Fig. 3-3 SEM photo of an AFM probe from TM Microscopes; top view (above) and close-up of the tip (below).](image)
Nowadays SPMs are used in a wide variety of disciplines, including fundamental surface science, routine surface roughness analysis, and three-dimensional imaging - from atoms to micron-sized protrusions on the surface of a living cell. All the other SPM modes and techniques developed from the STM and AFM are beyond the scope of this thesis, so they are just listed below:

- Magnetic Force Microscopy
- Lateral Force Microscopy
- Force Modulation Microscopy
- Phase Detection Microscopy
- Electrostatic Force Microscopy
• Scanning Capacitance Microscopy
• Scanning Thermal Microscopy
• Near-field Scanning Optical Microscopy
• Nanolithography
• Pulsed Force Mode
• Micro-Thermal Analysis
• Conductive AFM

Fig. 3-5 Van der Waals force plotted as a function of the tip-to-sample spacing.

Utilizing the AFM

The atomic force microscope is basically an imaging tool, which gives high-resolution information on the surface topography. Here the AFM is utilised in a different manner to study the reliability of our beams [3.13]. The relevant measurements and tests can be realised conveniently with the AFM; there is no need to construct complicated testing instruments. The
experiments in the present work were conducted with a NanoScope MultiMode SPM and an AutoProbe M5 SPM manufactured by Digital Instruments and Park Scientific Instruments, respectively. In one set of the experiments (chapter 3.2.2) the sample cantilever is loaded in the sample holder stage in the conventional way. In other experiments the sample cantilever is mounted in the AFM head replacing the AFM cantilever (chapter 3.2.3).

### 3.2.2 Quasi-static and fatigue tests

Beam-bending-based micromechanical tests were performed using an AFM. This technique combines very high resolution loading with nanometric precision measurement of the cantilever deflection [3.14]. As cantilever beams are fairly easy to fabricate from almost any material, the bending technique is a very simple and accurate measurement to determine the micromechanical properties. The Young’s modulus and the force or spring constant of thin films such as polysilicon and silicon carbide cantilevers can be measured with the bending technique [3.15]. In deflection loading and fatigue tests the sample cantilever is mounted conventionally on the AFM sample stage. The AFM tip is positioned on top of the sample cantilever to deflect it, see figure 3-6.

![Fig. 3-6 Bending test: the AFM tip deflects the sample cantilever](image)

The movement of the AFM tip is controlled by a high-precision piezo-electric stage, and its deflection is measured by the laser beam reflected on
the photodetector quad. The spring constant of the AFM cantilever is given by the manufacturer. The lateral position of the AFM cantilever is known as well by using surface features as reference points on the chip surface and exploiting the high-accuracy lateral movement of the piezoscanner. From all these data the deflection and the spring constant of the sample cantilever beam can be calculated.

Large deflections can be generated on the sample cantilever by using a stiffer AFM cantilever. The coarse approach mode of the AFM is controlled manually to apply the desired displacement. The creep behaviour of the sample cantilever beam can be studied through controlled static bending. With the same method the sample can be broken in order to characterise the fracture toughness. The bending method is suitable to characterise the mechanical long-term behaviour of multilayer structures as well. The building layers first need to be characterized separately. Delamination of the layers can be studied with the bending tests in combination with other techniques e.g. SEM or TEM.

Cyclic bending deflection of the sample cantilever serves as a reliability test. This is similar to the resonant method (chapter 3.2.3), but here the frequency and the amplitude of the deflection are arbitrary, and precisely maintained.

The Young’s modulus can be derived directly and accurately from Hooke’s law. This method requires true axial tensile force applied to the beam. Uniaxial tensile testing is the most direct way to characterise the yield strength and fatigue toughness. A rather complicated on-chip testing setup was developed by Yoshioka et al. [3.16] to overcome difficulties such as aligning, manipulating and fixing the brittle thin-film specimen, and measuring the elongation at reduced gauge length. The specimen and the loading mechanism, which translates the vertical deflecting load to in-plane uniaxial force, are fabricated on the same chip. The deflecting force is applied to a silicon paddle, which is supported by two torsion bars, and applies uniaxial tensile force to the sample beam. Silicon, silicon nitride and oxide films were tested with the uniaxial method. Another way to realize uniaxial tensile testing of thin films was demonstrated using a surface-micromachined testing-machine [3.17]. The advantage of the dry fabrication process is that it introduces the least stress in the structure. 3nN force and 58nm displacement was applied to the 110nm thick aluminium specimen by electrostatic comb-drive.
Spring constant

Figure 3-7 shows the force curve measured with the AFM. The force curve is obtained with a cyclical approach and retraction of the AFM tip on the beam surface. There are two overshoots in the negative region. The smaller triangle on the graph is the snap-in point occurring during approach, generated by the attractive van der Waals force between the tip and the cantilever. The larger triangle during retraction indicates the snap-back, which is due to the capillary force of the thin water layer. Solid surfaces are covered with a thin water layer in ambient air.

\[
\frac{k_b}{k} = \frac{k_A}{k_A - k}
\]

Fig. 3-7  Force curve measured on a SiN cantilever.

The total displacement of the AFM probe is the sum of its own deflection and the deflection of the sample beam. The bending tests are carried out in such a way that both the deflections are in the linear elastic domain and thus can be described by Hooke’s law. The beam-probe system can be considered as the combination of two springs in series, and the spring constant of the cantilever beam \(k_b\) can be calculated from the global spring constant \(k\) and the spring constant of the AFM probe \(k_A\).
When a deflecting force $F$ acts on the cantilever at a position $x$ ($x$ is the length between the base of the beam and the location where the bending force is applied), the deflection is given by

$$\delta(x) = \frac{Fx^3}{3EI}$$ (3-2)

where $E$ is the Young’s modulus and $I$ is the area moment of inertia of the beam cross section.

$$I = \int z^2 dA$$ (3-3)

where $z$ is the distance of the $dA$ cross section area unit from the neutral plane of the beam. The neutral plane is the plane which does not suffer from in-plane deformation during the deflection. The neutral plane is the mid-plane of an ideal beam with a rectangular cross section. For such a beam

$$I = \frac{\int \int z^2 dA dy}{\int \int w^2 dA dy} = \frac{wh^3}{12}$$ (3-4)

where $w$ and $h$ are the width and thickness of the beam.

The slope of the force curve gives the global spring constant

$$slope = \frac{k \delta_d}{z} = \frac{F}{z} = \frac{kz}{z} = k$$ (3-5)

where $\delta_d$ is the deflection of the AFM probe. The spring constant of the beam can be calculated from equation (3-2) as

$$k_b = \frac{3EI}{x^3} = \frac{Ewh^3}{4x^3}$$ (3-6)

or if the micro-beam is a wide beam [3.18]
Thin-Film Resonators

\[ k_b = \frac{3EI}{x^3(1 - \nu^2)} \]  

(3-7)

where \( \nu \) is the Poisson’s ratio.

The error of the calculated spring constant of the beam \( \frac{dk_b}{k_b} \) is mainly determined by the error of the thickness measurement, because this has the poorest relative accuracy, and the highest deviation due to non-uniformity of the thin film, depending on the processing technology:

\[
\left| \frac{dk_b}{k_b} \right| \leq \left| \frac{dE}{E} \right| + \left| \frac{dw}{w} \right| + \left| \frac{3dh}{h} \right| + \left| \frac{3dx}{x} \right| 
\]

(3-8)

If the Young’s modulus is calculated from the resonance frequency as described in section 3.2.3, the error of the spring constant is given by

\[
\left| \frac{dk_b}{k_b} \right| \leq \left| \frac{df}{f} \right| + \left| \frac{dm}{m} \right| = \left| \frac{df}{f} \right| + \left| \frac{d\rho}{\rho} \right| + \left| \frac{dL}{L} \right| + \left| \frac{dw}{w} \right| + \left| \frac{dh}{h} \right| 
\]

(3-9)

The length and width measurement accuracy is 0.1\( \mu \)m, and the thickness measurement accuracy is 0.01\( \mu \)m. Hence on a typical cantilever \( dw/w = 0.1\mu m/20\mu m \), \( dL/L = 0.1\mu m/100\mu m \), \( dh/h = 0.01\mu m/0.32\mu m \), \( df/f = 10\text{Hz}/30000\text{Hz} \) (no air damping, see section 3.2.3) and \( d\rho/\rho = (5\text{kg/m}^3)/(3000\text{kg/m}^3) \). The error of the spring constant measurement is 3.9% at most.

**Young’s modulus**

Several techniques have been used to measure the Young’s moduli of thin films used in micromechanical structures. They can be grouped as quasi-static and resonant methods. The latter will be discussed in the following section (*chapter 3.2.3 under “Young’s modulus”*). The quasi-static method is based on a small deflection of the micro cantilever or membrane prepared from the thin film (also referred to as bulge test) [3.19] [3.20]. If the dimensions of the beam are measured and the global spring constant is read from the slope of the force curve, the Young’s modulus of the beam can be calculated, combining equation (3-1) with equation (3-6)
The spring constant measurements have a reproducibility of 5%, which is determined by the readout accuracy. The spring constant of the sample should match that of the AFM cantilever to achieve higher measurement accuracy [3.21]. If $k_b$ is out of the $0.3k_A < k_b < 3k_A$ range, the error bars become increasingly large, because the total deflection is predominantly determined by the deflection of either the AFM or the sample cantilever beam. The measured results shown in figure 3-8 were obtained using an AFM cantilever with $k_A=0.4N/m$. The Young’s modulus was calculated from the measured spring constant: $E=185\pm17GPa$.

$$E = \frac{kk_A4x^3}{(k_A - k)3wh^3}$$ (3-10)

The spring constant measurements have a reproducibility of 5%, which is determined by the readout accuracy. The spring constant of the sample should match that of the AFM cantilever to achieve higher measurement accuracy [3.21]. If $k_b$ is out of the $0.3k_A < k_b < 3k_A$ range, the error bars become increasingly large, because the total deflection is predominantly determined by the deflection of either the AFM or the sample cantilever beam. The measured results shown in figure 3-8 were obtained using an AFM cantilever with $k_A=0.4N/m$. The Young’s modulus was calculated from the measured spring constant: $E=185\pm17GPa$.

**Fig. 3-8** Young’s modulus calculated from bending test.

When the AFM operates in the force-constant spectroscopic mode, the spring constant of the AFM probe needs to match the strength of local forces. Absolute measurements require absolute calibration of the AFM cantilever. Besides that, several areas using AFM need accurate calibration [3.21], such as quality assurance, measurement of surface forces [3.22] [3.23], determination of bond strengths [3.24], investigation of surface compliance of cells [3.25], rigidity and deformation of
biological materials [3.26], mapping of elastic properties [3.27] and dynamic micromechanical features of bio-systems [3.28]. The AFM probe spring constant value given by the manufacturer can have errors as large as 50% [3.29], [3.30], spoiling the accuracy of the Young’s modulus calculated from the bending test with equation (3-10). The spring constant can be determined accurately with a large-scale cantilever [3.31]. The spring constant of the latter is readily obtained when one measures the deflection and the mass of a weight attached to the end of the large-scale cantilever. The deflection is measured with an optical microscope.

Another quasi-static technique for measuring the Young’s modulus is the beam pull-in method. A double-clamped surface micromachined beam is deflected by an electrostatic force applied by the electrode under the beam. The Young’s modulus of the polysilicon beam is calculated from the pull-in voltage.

3.2.3 Resonant tests

Resonant tests are widely applied to determine the Young’s moduli of thin films and to characterize the long-term stability, aging and fatigue of thin-film resonators. Various thin films such as Si, GaAs, Glass, Kapton, PVF₂ and Quartz were characterized with resonant tests in a SEM [3.32]. The Young’s modulus was calculated from the resonance frequency, while the quality factor was determined from the time constant of the exponential decay of the vibrations after the excitation was switched off. Resonant measurements on double-clamped beams also give accurate results for the Young’s modulus and strain in thin films. The strain was calculated from the two lowest symmetric vibration modes of the 2µm thick silicon microresonators [3.33]. The resonance frequencies were measured in vacuum with the optical readout technique. The Young’s modulus was calculated including the effect of the strain, and showed good agreement with results obtained with other techniques. The relations describing the resonance frequency in classical continuum mechanics can be safely used down to submicron thicknesses [3.34]. Surface energy effects gain increasing importance when the thickness approaches atomic dimensions. The aging phenomena of heavily boron-doped p⁺-silicon film and Al-, Si₃N₄- and SiO₂-coated silicon films were studied [3.35]. The resonance
frequency of the cantilever beams prepared from the thin films shifted according to the 7 to 14 days cycling tests. The dynamic Young’s modulus and the internal energy dissipation was calculated from the resonance curves.

In our experiments, the sample chip with the cantilevers is mounted in the AFM head replacing the AFM cantilever. The cantilever is excited mechanically with a sweep, while the deflection of the free end is measured continuously with a bouncing laser beam, see figure 3-9. The measured peak, such as the one in figure 3-10, gives the resonant frequency of the cantilever beam.

![Fig. 3-9 Measuring the resonance frequency in the AFM head.](image)

This optical readout technique provides a very high resonance frequency measurement accuracy and a reproducibility of 10Hz. The air damping generates a frequency measurement inaccuracy of up to 0.25%, but it can be corrected as described below in the section “Air damping”. The disadvantage of the AFM resonant method is that multilayer cantilevers with severe stress-induced bending can deflect the laser beam to such an extent that it may not be reflected to the photo detector. This results in loss of signal. Another problem can arise in multilayer structures due to thermal effects. The laser diode heats the cantilever, generating parasitic deflection. Fortunately in most cases the system reaches thermal balance after a few minutes. These limitations can be avoided with capacitive [3.36], piezoresistive [3.37], electron tunnelling [3.12], or
piezoelectric [3.38] detection methods, but these methods introduce other difficulties.

The resonant method can be applied as a fatigue-ageing test, where the beam is driven at resonance for an arbitrarily long time. If the AFM operates in controlled environment, the air damping is constant, and the shift of the resonance frequency can be measured with 10Hz accuracy. The excitation amplitude can be set in the AFM. The resonance curve is measured frequently during the aging, and the driving frequency is set to the peak of the resonance curve.

**Quality factor**

The quality factor $Q$ of a resonator describes the coupling between the input energy and the output resonant energy. It can be defined as the total energy stored in the structure divided by the sum of energy losses from the vibrating element per cycle. $Q$ is mostly determined by the air damping, though acoustic radiation, internal friction, fatigue processes and surface effects lead to energy loss as well [3.32]. The quality factor can be calculated for a free standing vibrating beam with adequate space around as [3.39]

$$Q = \sqrt{\frac{E\rho}{24\mu}} \cdot \frac{w}{(h/L)^2}$$  \hspace{1cm} (3-11)
where $E$ is the Young’s modulus, $\rho$ is the mass density, $\mu$ is the viscosity (typical value is $\mu=1.8\times10^{-5}$ Ns/m$^2$ in ambient air at room temperature), $w$, $h$, and $L$ are the width, thickness and length of the beam. This calculation gives the quality factor value $Q=15$, which is lower than the measured value. $Q$ can be simply determined from the measured resonance peak, using the half-power-point method [3.40]

$$Q = \frac{f}{f_{FWHA}}$$

(3-12)

where $f$ is the resonance frequency, $f_{FWHA}$ is the full width at half amplitude, or the frequency bandwidth at the 3dB amplitude points (half-power point). The quality factor of the silicon nitride and silicon carbide vibrating beams was calculated to be $Q=45\pm15$ in ambient air, depending on the shape of the beams. The triangular shaped cantilever beams appeared to have higher $Q$ factors.

**Air damping**

The quality factor of the resonating beam is mainly determined by the air damping, while the internal damping and other mechanisms are practically negligible. Furthermore, the surrounding air has an effective mass, which affects the resonance frequency. The resonance frequency of the damped beam can be calculated from the undamped frequency as [3.41]

$$f_{damped} = f\sqrt{1 - \frac{1}{4Q^2}}$$

(3-13)

Even the worst case scenario where the calculated $Q=15$ value is substituted in equation (3-13) results in a resonance frequency decrease as low as 0.06%. This is in the order of the readout accuracy: 10Hz/30000Hz=0.033%. The standard error propagation analysis in the following section “Young’s modulus” shows that both the air damping and the readout error are negligible compared to other factors in the calculation of the Young’s modulus.

According to our measurements, the resistance of the media has a stronger influence than the calculated effect. The cantilever beam was
driven at different excitation amplitudes in the AFM head, and the resonance frequency was measured. The relative resonance frequency shift of a silicon nitride cantilever is plotted against the driving amplitude in figure 3-11. The driving amplitude is given as a percentage of the maximum amplitude, which is 8 µm. The resonance frequency decreases with increasing driving amplitude. This is explained with the energy loss due to drag force of the surrounding air. When the body is vibrating in air, the resisting force is proportional to the velocity in case of very small velocities [3.42]. For larger velocities it is assumed with sufficient accuracy to be proportional to the second power of the velocity. Thus the increasing vibration amplitude results in increasing energy loss of the system. As figure 3-11 shows, the resonance frequency can be corrected by linear regression of the measured curve and interpolating to zero driving amplitude. Thus the resonance frequency with no air damping effect can be determined.

Note that the AFM cantilever probe behaves differently. The AFM probe consists of two regular beams forming a triangle with the substrate base,
see figure 3-3. The thickness of the cantilever is 2µm. The vibrating structure exhibits the amplitude stiffening effect in air. The deflection of the cantilever is in the non-linear regime, therefore the increasing driving amplitude generates a positive resonance frequency shift. The effect of the increasing elastic modulus overcomes the energy loss due to the viscous damping. Above 50% driving amplitude, the deflection amplitude does not increase any more; only the resonant peak gets wider and shifts to higher values. The amplitude stiffening effect is described in detail for double-clamped beams in chapter 5.2.1.

**Young’s modulus**

The resonant method is a very simple way to measure the Young’s modulus [3.41]. Several excitation and readout techniques can be combined. Electrostatic excitation has the drawback of the metal coating, which can generate an error up to 20% [3.41]. Therefore photothermal, acoustic and mechanical excitations are preferred [3.43]. The AFM resonant test implements mechanical excitation and optical readout. The latter has the advantage that it requires only a small reflective mirror on the end of the cantilever. The reflective pad does not affect the deflection shape, but owing to the extra mass, lowers the resonance frequency. The pad can easily be deposited with one additional mask onto any of the thin films used in MEMS. Piezoresistive readout is only useful for silicon films, while piezoelectric techniques always require an additional layer at the base of the resonator structure, changing the resonant properties.

The Young’s modulus can be calculated from the measured resonance frequency. The resonance frequency of an undamped vibrating single-clamped cantilever beam can be given by equation (4-3), see chapter 4.4.2. If the beam thickness is very thin compared to the other two dimensions, as it is the case for our SiN and SiC cantilevers, the $1/(1-\nu^2)$ correction factor ($\nu$ is the Poisson ratio) should be applied as according to the plate theory. Hence $E$ should be substituted by $E/(1-\nu^2)$. When calculating the Young’s modulus from the resonance frequency, the mass loading of the reflective aluminium pad on the free end of the beam needs to be incorporated. For accurate results, we subtracted the Young’s modulus from finite-element models, which can account for the different aluminium pad geometries and shapes. The FEA model was based on two-
dimensional shell elements. The reliability of the models was tested and compared to analytical calculations of systems with simplified geometries.

The aluminium pad serves only as mass loading at the free end of the cantilever. Consequently the error of the Young’s modulus calculation can be approached with equation (3-14) derived from the equation of the free vibration.

\[ \left| \frac{dE}{E} \right| \leq \left| \frac{2dL}{L} \right| + \left| \frac{d\rho}{\rho} \right| + \left| \frac{4dL}{L} \right| + \left| 2\frac{dh}{h} \right| \]  

(3-14)

\[ d\rho/\rho = (5\text{kg/m}^3)/(3000\text{kg/m}^3), \quad dL/L = 0.1\mu\text{m}/100\mu\text{m}, \quad dh/h = 0.01\mu\text{m}/0.32\mu\text{m}. \]  

The only error in the resonance frequency is the readout inaccuracy, as the air-damping effect can be corrected; \( df/f = 10\text{Hz}/30000\text{Hz} \). After substitution into equation (3-14) we get \( dE/E = 6.8\% \). The Young’s moduli calculated with FEA are \( E = 230\pm16\ \text{GPa} \) for the silicon nitride and \( E = 320\pm22\ \text{GPa} \) for the silicon carbide thin films. The Young’s modulus of SiN is in good agreement with the data available in the literature [3.44], [3.45]. The value calculated for SiC is twice as high as the one reported in the literature [3.45], but it is strongly dependent on the film deposition conditions.

### 3.2.4 Large deflection and shock tests

Large deflections and mechanical shocks strongly influence the resonant characteristics of the thin cantilever beams. Controlled large deflections can be obtained by mounting the cantilevers in the AFM head, and driving the beam in resonance at high excitation levels. Arbitrarily large deflections can be applied by an AFM tip, where the sample beam is mounted conventionally on the sample stage. Other advantages of this method are the controllable cycle number and frequency. Mechanical shocks can be applied to the beam with the built-in piezoactuator of the AFM head, where the sample cantilever is mounted. An abrupt change in the driving voltage applied to the piezoactuator generates mechanical shock in the cantilever beams.
3.3 Stiffening effect

Surface-related effects can modify the resonant characteristics of vibrating thin-film cantilever beams. Surface stress and the adsorbate-induced change of the surface stress considerably affect the resonance frequency and the quality factor. In practice, if the beam surface interacts with the surrounding environment, instabilities in the resonant properties occur, as a result of which the operation of the resonant device becomes unstable. Eventually this can generate an error in the resonant sensor output, or in more extreme cause the driving function to fail. For our atmospheric resonator applications, the quality factor is less critical, but the shift in resonant frequency is a crucial stability issue. Long-term resonant cycling tests (see chapter 3.2.3) were carried out on silicon nitride and carbide thin-film resonators in various environments. A surface oxidation- and adsorption-induced stiffening effect was observed on the thin-film cantilevers in air and in a humid environment. Consequently the resonance frequency gradually increased.

3.3.1 Adsorption on solid surfaces

Solid surfaces adsorb various components from the surrounding media. The bond between the adsorbate and the surface atom can be different in type and/or strength, and thus the effect of adsorption on the surface can be different as well. Physisorbed adsorbates are bonded with second-order forces, while chemisorbed molecules establish chemical bonds. The presence of surface adsorbates can lead to variations in the surface stress and cause a transition from one surface reconstruction to another [3.46] [3.47]. Strong stress may induce surface defects [3.48]. Such point and line defects may serve to release surface stress. The adsorption-induced surface stress has three principle sources [3.49]. These microscopical origins are:

• Atomic size mismatch between the adsorbates and the substrate. If the size of the adsorbate increases, the compressive stress of the surface increases as well. The covalent radii, incorporating the bond length, need to be taken into consideration. Size mismatch is the most
straightforward source of surface stress, but not always the dominant one.

• The chemical nature of the adsorbates, which effects the hybridization of the surface atoms. The number of electrons available for bonds affects the bonding angle and thus the bond length, which can be substantially different from the one predicted from the covalent radii. A non-ideal bond length results in a change in the surface stress.

• Unusual bonding topology of the surface reconstruction. A relaxation process can change the bond angle stress and the separation between atoms. Different surface reconstruction mechanisms have a dominant effect on the surface stress of cleaved silicon surfaces. Tensile stresses are introduced on Si(111) and Si(100) surfaces due to π-bonded chain structure [3.46] and dimer reconstruction [3.50], respectively.

Solid surfaces subjected to ambient air adsorb hydrocarbons, water and hydrogen. Adsorption of oxygen often leads to surface oxidation, introducing interface stress between the substrate and the oxide layer. The forming native oxide layer is typically a few atomic layers thick. The growth rate decreases exponentially after the completion of the first layer. Water-vapor adsorption experiments have been performed by Sandler and Ibach on a Si(100)-1x2 surface [3.51]. Upon adsorption on the surface, the H₂O molecules dissociated to hydrogen atoms and hydroxyl groups. When all the Si atoms were covered with H or OH groups, the surface was saturated for further H₂O adsorption, while the surface reconstruction remained unchanged. The O to Si ratio of the saturated surface was measured with Auger electron spectroscopy. The results indicated that saturation occurs at 0.5 coverage on Si(100) and 0.43 coverage on Si(111) surfaces. It has been showed by Yang et al. that the different adsorbed components can be removed from the Si surface with Ultra-High Vacuum (UHV) treatment at high temperatures [3.52]. Some of the adsorbates can be removed from the silicon surface with UHV curing already at 600°C. Most of the adsorbates, including SiO₂ were removed in UHV at 900-1000°C, only SiC remained locally on the surface. The carbon can be removed above 1250°C.
3.3.2 Surface stress and related effects

The surface tension (surface free energy per unit area) of liquids can be measured by pulling a lamella of the liquid out of the surface. The required force per unit length is the surface stress, which is equal to the surface tension \([3.53]\). This last statement does not hold for solids, because unlike liquids, the number of surface atoms cannot increase upon expansion of the surface \([3.54]\). The definition of the surface stress is given in such a way that it explains the microscopic origins while retaining the correct continuum limit \([3.55]\). As according to the Hellmann-Feynman theorem, all the forces acting on the atomic nuclei originate from the Coulomb forces of the electronic charge density and the nuclear charges. Hypothetically, let’s cut a solid material along a plane with a right angle between the plane and the material surface, and remove all the atoms and the charge density on one side of the cutting plane. Consequently the atoms along the cutting plane have different neighbour configuration, so the force distribution in the plane is changed. To keep the position of these atoms unchanged, a force acting on each atom is required to counterbalance the mean forces acting from the other side. The surface stress is defined as the sum of these forces \(F^k\) minus the sum of the forces \(F^k_B\) required if there was no surface, per length \(L\) of the intersection of the cutting plane and the surface:

\[
\sigma = \frac{1}{L} \sum_k (F^k - F^k_B)
\]

Depending on the orientation of the intersecting plane, the components of the surface stress vector are converted into the components of a second-rank surface stress tensor \(\sigma_{ij}\). The infinitesimal reversible work \(\delta W\) from an infinitesimal strain \(\varepsilon_{ij}\) of the surface of the area \(A\) is

\[
\delta W = -A \sum_{i,j} \sigma_{ij} \delta \varepsilon_{ij}
\]

The reversible work generates a change in the surface free energy \(\delta E_s\), hence
\[ -\delta W = \delta E_s = \delta (\gamma A) \quad (3-17) \]

where \( \gamma \) is the surface free energy per surface area, the surface tension. The Shuttleworth equation [3.56] gives the relation between the surface stress, surface tension and surface strain as

\[ \sigma_{ij} = \gamma \delta_{ij} + \left[ \frac{\partial \gamma}{\partial e_{ij}} \right]_T \quad (3-18) \]

where \( \delta_{ij} \) is the Kronecker delta and the partial derivative is taken at a constant temperature [3.57]. Upon expansion of the surface of liquids, atoms are free to flow onto the surface, thus \( \gamma \) remains constant and the second term in equation (3-18) is zero. This is not the case for solid surfaces, and therefore the second term can reach substantial values, even in the order of \( \gamma \) itself. Theoretical calculations indicated that the second term of the equation (3-18) is always positive for a solid surface [3.58] [3.59], and the surface stress is a tensile stress. Note that the mathematical formulation and physical interpretation of the surface effects, which govern the equilibrium shapes of surfaces and gain crucial importance on the nanometer scale, are still a cause of controversy. The insufficiently precise definitions of and distinctions between surface energy, surface tension and surface stress lead to inconsistencies [3.60]. The validity of the Shuttleworth equation was questioned recently and the equivalence of the surface stress and surface tension was proposed instead [3.61].

When new surface is created, the electrons respond to the missing atoms and the electronic charge rearranges itself as shown in figure 3-12. The electrons originally connecting to atoms on the other side of the virtual cutting plane now occupy orbitals between the surface atoms. This increased charge density between the surface atoms has a symmetric distribution, and hence it does not generate a net force acting on the atoms. Some of the electrons flow into the space between the surface atoms and the first layer underneath. This effect does result in a net force acting on the surface atoms, leading to contraction of the first interlayer spacing. Thus the electron redistribution results in a charge depletion effect in the surface layer, which can also be predicted from the boundary condition imposed on the wave functions in the Fermi gas model [3.62].
What happens if adsorbates are present on the solid surface? Regarding the substrate surface atoms, the charge redistributes in a more bulk-like configuration, and the tensile surface stress is modified, or eventually disappears. However, tensile stress can now build up in the adsorbed layer. For submonolayer quantities of electronegative adsorbate, the charge is removed from the space between the substrate surface atoms, and the tensile stress within the surface layer can be substantially reduced, or even turned into a compressive stress. Besides this charge transfer mechanism, the direct attractive or repulsive interaction between the adsorbates needs to be taken into account [3.55]. Coulomb repulsion of the dipole moments is a weak effect compared to the overlapping of the wave functions of antibonding orbitals at small distances. As overlapping depends exponentially on the distance, the induced stress depends on the adsorbate coverage $\Theta$ as

$$\Delta\sigma \propto e^{-c/(\sqrt{\Theta})} \quad (3-19)$$

Besides the overlapping of the wave functions of different adsorbate atoms, the electronic charge distribution is rearranged around each well-separated adsorbate atom. This effect contributes to the surface stress.

---

**Fig. 3-12** Redistribution of the charge from dangling bonds into back bonds: electrons flow into the space between surface atoms and into the first interlayer spacing [3.55].
which is linear in coverage. Hence the adsorbate-induced surface stress has a coverage dependence as

$$\Delta \sigma = a \theta + b e^{-c/(\sqrt{\theta})}$$  \hspace{1cm} (3-20)

where $a$, $b$ and $c$ are constants. This coverage dependence was experimentally verified [3.55].

The surface stress of solids depends significantly on the crystallographic orientation of the surface and on the electronic structure of the material [3.58] [3.63] [3.64]. Surface stress is of key importance in fundamental effects like forces acting on a surface layer, surface morphology, surface reconstruction, surface diffusion and adsorbate-induced changes of forces between surface atoms. The issue of surface stress is crucial in epitaxial growth. Substantial strain can be imposed onto the epitaxial overlayers, modifying the electronic and structural properties. The desired properties can be tailored accordingly.

**Quality factor**

The ultrahigh sensitivity of scanning probe microscopy is attributed to the sensitivity of the micromachined cantilever probe. The cantilever beam needs to have a high quality factor, high resonance frequency and small spring constant. Miniaturization of the probe improves the latter two factors, but recent research shows that the $Q$ factor decreases with the size [3.68], [3.69]. For larger-scale beams the $Q$ is mainly determined by acoustic radiation and internal friction if no air damping is present. When the cantilever thickness scales down to the submicron range, the surface-to-volume ratio increases, and the surface loss becomes the dominant energy dissipation mechanism. The surface loss is generated by the surface stress, which is greatly modified by surface adsorbates and surface defects [3.55], [3.70]. The adsorbates interact with the surface atoms via charge transfer, Coulomb repulsion of the dipole moments, and overlapping of the atomic wave functions. These interactions modify the surface stress, and the modification is dependent on the crystallographic orientation [3.55]. The surface dissipation is proportional to the square of the maximum strain integrated over the surface area of the beam. For flexural vibrations, $Q$ is proportional to the thickness, thus for high surface-to-volume ratio structures the surface effect poses a crucial
restriction to the device miniaturization. Despite its significance, little knowledge is available on the surface loss mechanism of such ultrathin resonators. The effects of surface adsorbates, UHV treatment and exposure to atomic hydrogen on crystalline silicon cantilevers were investigated [3.52]. The $Q$ factor of Si (100) cantilevers with thickness 60-500nm was increased after removal of the native SiO$_2$ and adsorbates from the surface with 1000°C annealing in a UHV chamber. Hydrogen termination of the surface inhibits the surface oxidation and adsorption processes, and further $Q$ factor improvement was achieved. Exposure to atomic hydrogen modifies the surface structure to a 2x1:H monohydride configuration with high stability and less stress, which can also contribute to the increase of the $Q$ factor. Because the (110) oriented cantilevers have a different surface structure than the (100) oriented ones, their $Q$ factor had improved less after they had been heated and exposed to atomic hydrogen.

Thermal effects do not play a role in the change of $Q$. The Si-H bond was broken by an excimer lamp, then the structure was exposed to atmosphere for a day, and $Q$ returned to a similar value as it was before the treatment. Thus the improvement of $Q$ factor mainly originates from the surface modification.

Resonance frequency

The resonance frequency of a single-clamped cantilever beam is determined by the elastic properties and mass density of the beam material, by the dimensions of the beam, and by the surface effects in case of thin cantilever beams. The thinner the beam, the more significant the contribution of surface effects becomes because the surface-to-volume ratio is larger [3.71] [3.72]. The normal mode of vibration of a cantilever beam without the surface effects can be calculated conveniently by implementing an approximate method, namely the Rayleigh analysis [3.73]. The shape of the deflection during the vibration is assumed to be the same as one produced by a uniformly distributed load which represents the weight of the beam. The resonance frequency is obtained from the energy-conservation (balance) equation: the maximal kinetic energy of the vibration equals the potential energy of bending. The deflection curve $y$ of the cantilever under the action of its weight per unit length $q$ is approximated as [3.74]
where \( E \) is the Young’s modulus, \( I \) is the area moment of inertia of the beam cross section, \( L \) is the beam length and \( x \) is the coordinate along the length \( (x=0 \text{ at the clamping}) \). The potential energy of the bending is

\[
V = \frac{EI}{2} \int_0^L \left( \frac{d^2y}{dx^2} \right)^2 dx = \frac{q^2L^5}{40EI} \tag{3-22}
\]

The maximum kinetic energy during the harmonic vibration will be

\[
T = \frac{q\omega^2}{2g} \int_0^L y^2 dx = \frac{q^3\omega^2L^9}{E^2I^2g} \cdot \frac{13}{8 \cdot 9 \cdot 90} \tag{3-23}
\]

where \( g \) is the acceleration due to gravity and \( \omega \) is the angular frequency. The natural frequency \( f_0 \) of the vibration is calculated as \( V = T \) from equation (3-22) and equation (3-23)

\[
f_0 = \frac{\omega}{2\pi} = \frac{1}{2\pi} \sqrt{\frac{90 \cdot 9}{65}} \frac{EIg}{qL^4} \tag{3-24}
\]

The error of this approximate solution for the resonance frequency is less than 0.5%. More accurate results can be obtained from the elastic theory.

How can the surface stress be incorporated in the Rayleigh method? According to Müller and Kern, the surface stress generates two moments; one depending on the difference of the surface stresses of the two sides of the cantilever (this differential surface stress is exploited in functionalized probes, see the section “Experimental determination of the surface stress” below), and the other moment depending on the sum of the surface stresses [3.75]. First-order approximation allows us to assume that the deflection curve is not changed by the surface stress. Then the latter moment is equivalent to an axial force acting on the mid-plane of the cantilever at its free end, and results in contraction or expansion according to the sign of the total surface stress. According to Chen et al., this axial
Stiffening effect

force changes the resonance frequency, similar to a guitar string under tension [3.78]. We question this explanation. One end of the beam is free and the beam can deform freely, corresponding to the stresses. The symmetric surface stresses on the two sides on the cantilever beam are counterbalanced by the bulk stresses and thus the resultant stress is zero. A double-clamped beam would be indeed similar to the guitar string, exhibiting non-zero resultant stress. In our view the resonance frequency is affected rather by the change of the surface stresses upon bending of the cantilever. The effect of the adsorption-induced change of the surface stress can be described as follows. The surface adsorbates interact with each other and affect the local electron structure of the surface atoms, as explained above. During bending of the cantilever beam, these effects (e.g. the repulsive force from the overlapping of the wave functions of antibonding orbitals, which depends exponentially on the distance) change the surface stress. This non-zero surface stress depends on the direction and magnitude of the bending. The result is a restoring force different on the two sides of the deflected cantilever beam. Hence the neutral plane of the cantilever shifts and the resonance frequency increases. We can conclude that the resonance frequency shift is not attributed to the symmetric surface stresses, but to the change of the surface stress upon deflection of the cantilever, which delivers differential restoring forces. Unfortunately the interpretation of tensile and compressive surface stress contributions is still controversial in the literature [3.60]. Better understanding of the surface stress and its influence on the resonance frequency requires further experimental and theoretical work.

However, we give the equations below for the beam under axial tensile stress for two reasons. One is that it can be useful when calculating centrifugal effects for instance. The other reason is that the surface stress change-induced restoring force developing during cantilever deflection can be represented by a tensile axial force $S$ acting on the free end of the cantilever (at $x=L$). But this force is not correlated to the absolute value of the total surface stresses, but to the developed differential surface stress. This corresponding tensile force can be incorporated in the Rayleigh analysis. The work done against the axial tensile force during bending should be subtracted from the potential energy of bending. The work done by the tensile force is given by [3.74]
Due to the increase of the potential energy, the natural frequency \( f_0 \) is modified to \( f \) as

\[
f = f_0 \sqrt{1 + \frac{5 S L^2}{14 EI}}
\]  

(3-26)

If the tensile forces \( s \) are uniformly distributed along the length, like in case of surface stress, then the following term needs to be subtracted from the potential energy

\[
-\frac{1}{2} \int_{0}^{L} \left( \frac{dy}{dx} \right)^2 s(L-x) dx = -\frac{q^2 L^7}{14 EI} \frac{7sL}{20}
\]

(3-27)

Note that the effect of the uniformly distributed forces on the resonance frequency is the same as if 7/20 of the sum of these forces was applied at the free end of the cantilever.

The surface stress is affected by adsorption on the surface, and hence the ambient atmosphere changes the resonance frequency of the vibration. Single-crystal GaAs beams were tested implementing optical readout by means of a bouncing laser beam and mechanical excitation with chopped white light exploiting the photopiezoelectric effect [3.76]. Upon rapid exposure to room atmosphere, the natural frequency of (111) GaAs cantilevers did not change instantaneously, but increased logarithmically with time before reaching a steady-state value [3.77]. This type of logarithmic transient is typical for adsorption on semiconductor surfaces. Monitoring the resonance frequency gives information on the adsorption-desorption kinetics. The measurements indicated that the decrease in surface stress is a logarithmic function in time. Assuming that the number of adsorbed molecules on the semiconductor surface is logarithmic in time [3.77], one comes to the conclusion that the adsorption-induced decrease in surface stress is proportional to the number of adsorbed molecules.
The resonance frequency of vibrating cantilever beams can be affected both by the mass loading of the adsorbed molecules and by the adsorption-induced change in the surface stress [3.78]. The altered resonance frequency $f_2$ can be described as

$$f_2 = \frac{1}{2\pi} \sqrt{\frac{K + \delta K}{m_{\text{eff}} + n\delta m}}$$  \hspace{1cm} (3-28)

where $K$ is the spring constant of the beam, $\delta K$ is the change of the spring constant due to the adsorption-induced change of the surface stress, $m_{\text{eff}}$ is the effective mass of the cantilever and $\delta m$ is the mass increase of the cantilever beam upon adsorption. The effective mass $m_{\text{eff}} = nm$, where $m$ is the mass of the cantilever and $n$ is a factor depending on the shape of the cantilever beam. In most cases the adsorption-induced changes in $K$ and $m$ are very small, and the resonance frequency after adsorption can be approximated with the following linear equation [3.78]:

$$f_2 = f_1 \left( 1 + \frac{1}{2} \left( \frac{\delta K}{K} - \frac{\delta m_{\text{eff}}}{m_{\text{eff}}} \right) \right)$$  \hspace{1cm} (3-29)

This equation can be used safely as long as $\delta m << m$ and $\delta K << K$.

In practice, there are four typical scenarios arising from adsorption of molecules on the cantilever surface:

- The change of the resonance frequency is governed by the mass loading, while the change of the spring constant is negligible.

- The change of resonance frequency due to mass loading is negligible, but static deformation of the cantilever occurs as a result of differential surface stress on the two surfaces.

- The resonance frequency change is determined by change of the spring constant, and the mass loading is negligible.

- The resonance frequency changes as result of the combination of adsorption-induced change of the spring constant and mass loading effects.

In fortunate situations, one of the two effects, either the mass loading or the introduced surface stress, is much stronger than the other. In the first
scenario, where the mass loading determines the resonance frequency shift, the extra mass on the cantilever can be determined from the natural frequency shift, derived from equation (3-28).

\[
\delta m = \frac{K}{4\pi^2n} \left(\frac{1}{f_2^2} - \frac{1}{f_1^2}\right)
\]  

(3-30)

If this is the case, the new resonance frequency \(f_2\) is always smaller than the original one \(f_1\). Such mass loading effect was measured on crystalline silicon vibrating beams, and is described in chapter 4.4.4. The mass change can be calculated more easily using equation (3-29), where the term containing the spring constant is zero, as \(\delta K=0\).

The surface effects are more emphasized and the mass loading can be neglected for thin structures with thicknesses in the submicron range. Scenario (2) and (3) are observed depending on the exposure time and configuration, and on the material of the cantilever and the media which it is exposed to. The resonance frequency increases over time, which is the opposite of that expected due to mass loading. Thus the resonance frequency is mainly determined by the variation in surface stress. The change of the spring constant can be calculated from equation (3-29), where \(\delta m=0\) this time. This scenario was found for thin silicon nitride and carbide cantilevers vibrating in clean room environment, and is presented below in the section “Measured resonance frequency shift”. Submicron-thick (100) oriented single-crystal silicon cantilevers exhibited a similar resonance frequency decrease after removal of the surface adsorbates by heating in a UHV chamber and hydrogen termination [3.52].

Chen et al. calculated the adsorption rate of mercury vapor on a gold-coated silicon cantilever by measuring the resonance frequency shift: rate\(=553\)pg/min [3.78]. The spring constant increased at a rate of \(0.001\)N/m/min. The resonance frequency in this case was determined by variations in both the mass loading and the spring constant. If the first or the second term dominates, the resonance frequency shift is negative or positive, respectively. The second term can be eliminated for submicron-thick cantilevers as well if only the last section of the beam is coated with the active material.
Experimental determination of the surface stress

Experimental determination of the absolute surface stress is problematic, but the variation of the surface stress upon the deposition of another material can be experimentally measured with the bending method, also known as the cantilever method [3.65]. If the surface stress on one side of a thin plate of a crystal changes, the plate bends. The radius R of the bending is given by Stoney’s formula [3.66]

\[ R = \frac{Eh^2}{6\Delta\sigma(1-\nu)} \] (3-31)

where \( E \) is Young’s modulus, \( h \) is the thickness, \( \Delta\sigma \) is the change of the surface stress and \( \nu \) is Poisson’s ratio. For elastically anisotropic materials \( E \) and \( \nu \) are replaced by the appropriate combinations of the elastic compliance tensor. The bending of crystalline Si substrates was measured capacitively in a UHV chamber, and orientation dependent surface stress was found [3.51]. The room-temperature adsorption of oxygen resulted in breaking of the Si-Si bonds, forming Si-O-Si bonds. The O-Si(111) system showed a compressive surface stress of -7.2N/m per monolayer coverage, while a tensile stress of +0.26N/m was measured for the Si(100) surface. The measured surface stresses are larger than the ones expected from the mismatch of bond lengths alone. The absolute amount of stress is determined by the local geometry as well as by the surface-induced changes in the chemical bonds. The experimental results for surface stress induced by adsorption of oxygen on silicon surface was sufficiently explained by the valence force model using bulk valence forces and bulk structural parameters. For more accurate results, first-principal total energy calculations are required, incorporating the dangling bonds and the reconstruction. Gelatin-coated silicon cantilevers allow the measurement of relative humidity [3.78]. Stoney’s formula, see equation (3-31), was used to determine differential surface stress of 0.38N/m by measuring the static bending of the cantilever.

The major part of the surface stress is introduced by the first layer, or even sub-monolayer. It was demonstrated that the cantilever method is sensitive enough to stresses introduced by submonolayer quantities of adsorbates [3.55] [3.65]. Orientation-dependent anisotropic surface stress was measured on (110) crystalline wolfram cantilevers [3.67]. Half a
monolayer of adsorbed Fe induced compressive stress along [001] and tensile stress along [110]. The biaxial curvature of W (110) was measured using the cantilever bending method. The surface stresses was derived from equation (3-31) as

\[
\sigma_i = \frac{E h^2}{6(1-v)} \left( \frac{1}{R_i} + \frac{1}{R_j} \right)
\]

(3-32)

where \(i,j=[001],[\bar{1}10]\). As there are limitations to the size of single-crystal for metals that can be realized, a 3-segment composite cantilever was built to measure adsorption-induced variations of the surface stress on crystalline nickel [3.55]. The nickel sample was bolted between two metal sheets of molybdenum. The adsorption of oxygen, sulphur and carbon on the nickel surface was investigated.

The surface stress can be derived from the resonance frequency measurements, as described above in the section “Resonance frequency”. The resonance frequencies of thin cantilever beams are sensitive to the change of surface stress. The adsorption-induced change in surface stress, and thus the adsorption-desorption kinetics and various environmental effects can be studied by monitoring the resonance frequency over time. Besides affecting the surface stress, the adsorbates on the beam surface act as mass loading as well. To interpret the resonance frequency change, one should measure the cantilever bending and natural frequency change simultaneously to decouple the effects of spring constant variation and mass change.

### 3.3.3 Measured resonance frequency shift

The AFM resonant method, described in chapter 3.2.3, was used to study the long-term stability of thin silicon nitride and silicon carbide cantilever beams. Cycling tests of several days were conducted in various environments. The cantilevers vibrated at their fundamental resonance frequencies, which were measured hourly during the cycling test. The absolute frequency measurement accuracy of 10Hz corresponds to a relative error less than 0.03%. A stiffening effect was observed both for silicon nitride [3.79] and silicon carbide [3.80] cantilever beams in
ambient air. The resonance frequency increased over time, as shown in figure 3-13, figure 3-16 and figure 3-19. Note that the stiffening effect is independent of the mechanical driving. The same logarithmic behaviour was observed for samples which stood still in ambient air, and vibrated only during the resonance frequency measurements. This eliminates the possibility of a vibration-induced intrinsic material property change. The measured logarithmic behaviour is typical for surface adsorption. The adsorbed molecules modify the surface stress and consequently the stiffness of the resonator, as described in chapter 3.3.2. The typical molecules adsorbed on the surface from ambient air are oxygen (or water) hydrocarbons and hydrogen molecules. Oxygen seems to have a dominant effect on the resonant behaviour and can lead to surface oxidation.

Surface oxidation

Environmental effects can change the properties of thin films. Silicon nitride is a chemically stable material, but the surface incorporates oxygen when exposed to air or water vapor. This results in increased surface conductivity [3.81] and in change of mechanical properties. It was described in chapter 3.1.2 that the low-stress LPCVD silicon nitride film is a two-component amorphous material, a mixture of Si and Si3N4 nano-clusters. As is expected, the Si clusters oxidize, forming SiO2. More interestingly, Si3N4 also reacts with oxygen. The Si-N bonds oxidizes when subjected to air or water, and the reaction is described with the following equations [3.82]:

\[
\text{Si}_3\text{N}_4(s) + 3\text{O}_2(g) \rightarrow 3\text{SiO}_2(s) + 2\text{N}_2(g) \tag{3-33}
\]

\[
\text{Si}_3\text{N}_4(s) + 6\text{H}_2\text{O}(g) \rightarrow 3\text{SiO}_2(s) + 4\text{NH}_3(g) \tag{3-34}
\]

The free-energy change of the reactions are \(\Delta F_{298^\circ C} = -304\text{kcal/mole}\) and \(\Delta F_{298^\circ C} = -147\text{kcal/mole}\), respectively. The surface oxidation of silicon nitride at room temperature is thermodynamically feasible. The Si atoms at the surface bond to the more electronegative O atoms instead of to the N atoms. The surface oxidation of silicon nitride at room temperature and at an elevated temperature was examined with X-ray Photoelectron Spectroscopy (XPS) [3.82]. The silicon nitride is rapidly oxidized in air at
room temperature. The oxidant can be water vapor or oxygen, but their relative reactivity is not known. The surface of silicon nitride is likely to be a graded oxynitride, which is oxygen rich and nitrogen deficient at the film surface. The O atom is chemically bound to the Si atom. The silazane (Si-N-Si) bridge of silicon nitride is changed to siloxane (Si-O-Si), which is present in silicon oxynitride films. According to the XPS measurements, the formed SiO\textsubscript{x}N\textsubscript{y} is not a mixture of SiO\textsubscript{2} and Si\textsubscript{3}N\textsubscript{4} clusters, but the siloxane and silazane groups are blended on the molecular scale. On elevated temperatures, first oxynitride, then SiO\textsubscript{2} is formed on the silicon nitride surface.

The surface of silicon carbide oxidizes as well when subjected to ambient air or water vapor. XPS and infrared spectroscopy showed that the near-surface region consists of different phases: Si, Si oxides (mainly SiO\textsubscript{2}) and SiC and Si oxicarbides (SiO\textsubscript{x}C\textsubscript{y}) \cite{3.83}. As the SiC is more resistant to oxidation than elemental Si, most of the incorporated oxygen in the SiC layer is not associated with silicon oxicarbide formation \cite{3.84}. The oxidation process is governed by the formation of SiO\textsubscript{2}, CO and CO\textsubscript{2} and loss of the carbon atoms. The SiC samples with a higher unreacted Si content showed stronger oxidation.

**Thickness dependence**

The stiffening effect strongly depends on the film thickness. It is plausible that because of their lower surface-to-volume ratio, the thicker resonators are less sensitive to surface-related effects. This explains why no stiffening effect was found for 1-2\textmu m thick silicon nitride membranes \cite{3.85} \cite{3.86}, while a similar effect was reported for thin polysilicon membranes \cite{3.87}. The thickness dependence was studied by Ultra-High Vacuum (UHV) heat treatment at 1000°C and H termination of crystalline silicon surfaces \cite{3.52}. The Q-factors of 60-500nm thick silicon cantilevers were compared before and after the various treatments. It was shown that the thicker structures are not so sensitive to short-time exposure to air.

To confirm the thickness dependence we have carried out 2-3 days long cycling tests on silicon nitride cantilevers with thicknesses of 320, 500 and 800 nm. It is shown in figure 3-13 that a weaker stiffening effect was measured for thicker SiN cantilever beams.
Fig. 3-13 Stiffening effect for SiN cantilevers with different thicknesses.

Fig. 3-14 Cycling test of 1µm thick crystalline Si cantilever (contact AFM probe) - no stiffening effect.
We also tested 1µm thick crystalline silicon contact AFM probes. The native oxide layer was first removed with buffered hydrogen fluoride (BHF), and then the cantilevers were cleaned with acetone. The cycling tests showed no measurable stiffening effect in ambient air. The deviation of the resonance frequency remained within the readout accuracy interval, as shown in figure 3-14.

**Age dependence**

The resonant behaviour depends on the thickness of the oxide layer. The logarithmic section of the curve measured on SiN cantilevers is followed by a linear part, see figure 3-13. This linear slope can be attributed to the increase in the overall thickness. At this point the surface stress introduced by further oxidation and adsorption is negligible as the surface is already fully oxidized and covered with adsorbed layers. The oxidation rate is very slow, close to linear. The resonance frequency of the SiN cantilever is calculated to increase with increasing thickness at a rate of 0.2% per 1nm grown oxide. The surface stress effects are neglected in this calculation. The Young’s modulus of the silicon oxide is at least three times lower than that of the silicon nitride. Consequently the resonance frequency increase is lower upon oxide growth than it would be if the thickness of the silicon nitride itself would increase, see figure 3-15.

The stiffening effect depends on the age of the cantilever beam. The surface of several months old SiN cantilevers is covered by a few nanometers thick native oxide layer, which makes them less sensitive to further oxidation and adsorption. Figure 3-16 compares the measured stiffening effect for a several months old SiN cantilever and a fresh one. The fresh cantilever was obtained by removal of the surface oxide and other adsorbates with BHF etching and subsequent cleaning with acetone. The fresh cantilever follows the usual logarithmic behaviour, which is due to the adsorbate-induced surface stress change. In contrast, the old cantilever beam is rather stable. Small changes of the environment can account for the slight fluctuations of the resonance frequency. Both the adsorption/oxidation-induced surface stress changes and the oxidation rate (and hence the thickness increase) are much smaller than in case of the fresh surface.
Stiffening effect

Fig. 3-15 Calculated relative resonance frequency shift of SiN cantilever upon increase of the thickness.

Fig. 3-16 Stiffening effect for a several months old 320nm thick SiN cantilever before (old) and after (fresh) BHF etch.
Handling, mounting and dismounting the sample generates large deflections, which “shake off” most of the loosely bounded surface adsorbates. As no substantial logarithmic increase is observed in the resonance frequency of the old beam, we can conclude that the most relevant adsorbate components of the ambient air are oxygen and water vapor, which are less effective on top of the oxidized surface.

**Analytical results**

The surface adsorption-induced stiffening effect can be quantitatively described to some extent by the Rayleigh method, as explained in chapter 3.3.2 section “Resonance frequency”. Let’s take a fresh SiN cantilever with dimensions 104x20x0.32 µm³ as an example, where a 0.96% resonance frequency shift was measured in 4 days. The corresponding change of the differential surface stress on the two sides during bending was 1.7·10⁻³N/m.

![Diagram](image.png)

*Fig. 3-17* The calculated relative shift of the resonance frequency of a SiN cantilever beam is plotted as function of the beam thickness (h<x<3h).
The spring constant shift of the cantilever beam was 1.9%. The effective thickness increase of the native oxide in 4 days is estimated to be 0.25 nm on both sides of the cantilever. The surface oxide is a graded structure, which is most oxygen rich on the surface, and the absolute thickness is difficult to determine experimentally.

The thickness dependence was studied with analytical calculations. The plot in figure 3-17 shows that the $1.7 \times 10^{-3} \text{N/m}$ surface stress has a much stronger influence on the resonance frequency than the 0.5 nm total thickness increase upon surface oxide formation. Furthermore, it is also clear from the plot that the 500 and 800 nanometer thick SiN cantilever beams are much less sensitive to the adsorption/oxidation-induced effects, than the $h=320$ nm thick beams. This is comparable to the stiffening effects measured for the beams with different thicknesses, shown in figure 3-13.

**SiN vs. SiC**

Silicon carbide cantilevers of the same geometry behaved slightly different from the silicon nitride cantilevers due to the differences in material and chemical properties. Although the 180 nm thick SiC cantilever beams are almost twice as thin as the thinnest SiN beams tested, they appeared to be more resistant to environmental effects. The stiffening effect in ambient air was weaker than that measured on the SiN cantilevers. The slope of the linear part also appeared to be practically zero, see figure 3-18, which indicates that after a fast initial increase the resonance frequency is stable. In other words, short exposure to air initiates an adsorption/oxidation-induced stiffening effect, but further oxidation is limited. This explains why the resonant behaviour of SiC cantilevers remains unchanged after BHF etching, see figure 3-19, unlike that of SiN cantilevers, see figure 3-16. All the three curves measured on the SiC cantilevers reach their maximum after 30-40 hours, where the resonance frequency eventually stabilizes. The surface adsorbates have the same effect on fresh and ‘oxidized’ SiC surfaces. Handling, mounting and dismounting the sample subjects the beam to mechanical shock, which damages the adsorbed surface layer, as explained in chapter 3.4. Therefore the three weeks old sample also exhibits the initial logarithmic stiffening effect.
Surface passivation

The silicon nitride and silicon carbide cantilever beams were coated with tin nitride and gold with thicknesses in the range of a few nanometers. These coating materials tend to oxidize rapidly in ambient air, but further oxidation practically stops after completion of the first atomic layer. No substantial improvement of the resonant properties of the coated beams was achieved, because the major part of the surface stress shift is generated by the first monolayer of the oxide. However, the long-term linear component of the resonance frequency shift was slightly improved, as expected from the inhibited oxidation mechanism of the coating material. Furthermore, the coating layers with uneven thicknesses on the two sides of the beams introduced static deflection and thermal instabilities in the resonant behaviour due to differential surface stresses and different thermal expansion coefficients of the substrate and the coating materials.

Fig. 3-18 Stiffening effect on SiC cantilever following severe mechanical shock
3.4 Shock response

The shock sensitivity is a major stability problem in resonant mode devices. It was observed from thin cantilever beams that the resonance frequency is strongly influenced by mechanical shocks. This can eventually lead to false measurement results of the resonant sensor, or to failure of the resonant driving function. The response of the thin SiN and SiC cantilevers to mechanical shocks and large deflections was studied. Mechanical shocks were generated with the AFM’s built-in piezo actuator, and large deflections were realized by high-amplitude resonant excitation and cyclic bending tests using an AFM cantilever probe as described in chapter 3.2.4. Shocks and large deflections generate an abrupt drop in the resonance frequency up to a few percent, see figure 3-20. The height of the negative step depends on the magnitude of the shock or large deflection. Following the mechanical shock, the resonance frequency
increases again as according to the stiffening effect. A stronger shock response was observed for thinner cantilevers. Robust design decreases the shock sensitivity, but leads to loss of measurement sensitivity as well.

![Graph showing the relationship between aging time and relative resonance frequency shift.](image)

*Fig. 3-20 Mechanical shock generates negative step in the resonance frequency curve.*

This abrupt shift of the resonance frequency upon mechanical shock can be explained with the surface adsorption/oxidation theory. The shocks and large deflections damage the oxide layer and the surface film consisting of other adsorbates. The typical forms of the damage are microcrack generation in the surface layer and shake-off of the surface adsorbates. The discontinuities in the surface layer weaken the stiffening effect, so the overall stiffness of the structure drops. After the applied shock or deflection, the surface re-oxidizes and adsorbates cover the ‘fresh’ areas, hence the stiffness and the resonance frequency increase again. Small shocks generate small resonance frequency drops, which recover in a few minutes, and the microscopical surface damage is difficult to examine. Generation and propagation of cracks are likely to occur in the surface oxide layer. Large shocks, like the one applied to the sample in figure 3-20, can generate severe damage, which can be visualized. Nano- and micro-cracks were observed with AFM on the SiN
beam surface at the clamping site, where the highest stress occurs, see figure 3-21.

3.4.1 Cyclic shock tests

Repeated shock tests were performed on the thin silicon nitride and silicon carbide cantilever beams. Reproducible mechanical shocks were applied in the form of 5sec long high-amplitude resonant excitation. The deflections of the free end of the beam were in the order of 20-30nm. Note that this large deflection is still in the elastic range of the 320-800nm thick silicon nitride beams. “Large” indicates that the deflection is approximately 10 times bigger than those used in the resonance frequency measurements. Following the 5sec large deflections, the resonance frequency was measured every 30 second for 2.5 minutes. The tests were repeated every 3 minutes. As a response to the mechanical shocks, both

Fig. 3-21 Micro-crack on a SiN cantilever surface generated by mechanical shock.
the resonance frequency and the vibration amplitude dropped. These large deflections generated only small negative steps in the resonance frequency, which totally recover in 2 minutes, unlike the large deflections and shocks described above in chapter 3.4. The first few large deflection tests on SiN cantilevers showed hysteresis. Here the deflection resulted in higher resonance frequency drop which could not recover fully in a short time. This is due to the cracking of the thicker oxynitride layer, which had grown previously on the samples in air. Then the sample reached a dynamic balance with the environment, where the repeated large deflection tests had a reproducibility better than 20%. The results of 8-12 tests are averaged and the mean resonance frequency shift values are plotted vs. time in figure 3-22. After the 5sec large deflections, the resonance frequency increased, following a logarithmic characteristic curve. The resonance frequency shift was positive after 1-2 minutes, indicating that a reasonably fast stiffening effect took place. It can be explained by comparing the results to the severe shock tests (chapter 3.4) and stiffening effect (chapter 3.3.3). Regarding the time scale, a strong stiffening effect was observed for 10-20 hours following severe mechanical shocks. Afterwards the frequency increase was slower, linear in time. Translating this to the current cyclic shock test, the first few shock cycles result in severe damaging of the thick oxynitride and adsorbed surface layers which grew previously in weeks or months time in ambient air. The beam responds with a large resonance frequency drop. Therefore the beam response in the first 2-3 shock cycles is considered as transient behaviour and will be neglected from further data evaluation. The relatively fast resonance frequency increase following the transient cycles would continue for 10-20 hours, till the damages would be recovered. Consequently it applies for each shock test cycle that the resonance frequency is higher at the end of the cycle than it was before the shock was applied. Upon mechanical shocks the surface adsorbates are partly ‘shaken off’ and a statistically large number of nano- and micro-cracks (see figure 3-21) are assumed to be generated on the surface. For that reason the cycling shock tests are reproducible. Subsequent shock test cycles generate small resonance frequency drops, which recover fast in ambient air. The crack generation and “healing” effects are in dynamic balance, which is determined by the magnitude of the shock (or duration and magnitude of the high-amplitude excitation), by the time interval between the shock tests and by the surrounding media.
Environmental tests

3.5 Environmental tests

Cycling shock test were carried out in various environments in order to study the stiffening effect and crack generation and healing mechanisms. The tests were identical to the cyclic shock tests described in chapter 3.4.1, but now the AFM was placed in an environmental chamber. The influence of the surrounding environment on the resonant characteristics and on the shock response was investigated. Silicon nitride, silicon carbide and single-crystal silicon cantilevers were tested in ambient air, humid air and in a nitrogen- and argon-rich atmosphere. As is expected from the surface adsorption/oxidation theory, the adsorbates were partly released from the surface of the SiN cantilever in a nitrogen-rich environment, hence the resonance frequency decreased. It did not make any difference whether the beam was driven at resonance (on
moderate excitation levels) or stood still; the resonance frequency followed the curve shown in figure 3-23. This indicates that no cracking of the surface oxynitride, but rather a “shake off” of the surface adsorbates took place. When the chamber was ventilated and filled with ambient air, the resonance frequency increased, following a mirrored logarithmic curve. Again, exciting the sample at resonance had no effect on the curve shape. This experiment shows that oxygen, either gas or vapor, has dominant effect on the surface stress and hence on the resonance frequency.

![Resonance frequency shift on a SiN cantilever in nitrogen-rich environment and in ambient air.](image)

*Fig. 3-23 Resonance frequency shift on a SiN cantilever in nitrogen-rich environment and in ambient air.*

The environmental influence on the resonant properties can be demonstrated by cyclic shock tests on a silicon nitride cantilever beam, see figure 3-24. When shock or large deflections were applied, the adsorbed layer and the grown oxynitride layer were damaged. The actual microscopic structure of the adsorbed layer as well as the nature of the damage are difficult to identify experimentally. The weakly bonded molecules, clusters can be shaken off the surface, while the more solid layers can suffer from cracking. In air, the damages generated after the large deflection all healed within 2 minutes. The resulting resonance frequency was higher than the initial one due to the stiffening effect.
Environmental tests

When the same tests were repeated in a nitrogen-rich environment, the average resonance frequency was lower, 30kHz instead of the 31kHz measured in air, and the resonance peak was substantially narrower. Hence the quality factor of the resonating cantilever is higher in a nitrogen-rich environment. The recovery of the resonance frequency after the large deflections was much slower than in air, and after 2.5 minutes the resonance frequency was lower than the initial one, see figure 3-24. The lower oxygen and water vapor content of the nitrogen-rich environment explains this behaviour, as the surface oxidation/adsorption process is slower here. A further increased nitrogen content led to an even weaker resonance frequency recovery effect. The negative shift of the resonance frequency was also lower in a nitrogen-rich environment. As the resonance frequency did not recover fully during the 3 minutes between the applied large deflections, the surface film remained damaged. Consequently, large deflections of the same magnitude could not create such a resonance frequency drop as in air. Of course, the transient response of the beam during the first 2-3 shock cycles are disregarded here as explained in the repeated shock tests in chapter 3.4.1.

Fig. 3-24 Repeated shock tests on SiN cantilevers in different environments I.

When the same tests were repeated in a nitrogen-rich environment, the average resonance frequency was lower, 30kHz instead of the 31kHz measured in air, and the resonance peak was substantially narrower. Hence the quality factor of the resonating cantilever is higher in a nitrogen-rich environment. The recovery of the resonance frequency after the large deflections was much slower than in air, and after 2.5 minutes the resonance frequency was lower than the initial one, see figure 3-24. The lower oxygen and water vapor content of the nitrogen-rich environment explains this behaviour, as the surface oxidation/adsorption process is slower here. A further increased nitrogen content led to an even weaker resonance frequency recovery effect. The negative shift of the resonance frequency was also lower in a nitrogen-rich environment. As the resonance frequency did not recover fully during the 3 minutes between the applied large deflections, the surface film remained damaged. Consequently, large deflections of the same magnitude could not create such a resonance frequency drop as in air. Of course, the transient response of the beam during the first 2-3 shock cycles are disregarded here as explained in the repeated shock tests in chapter 3.4.1.
The nitrogen-rich atmosphere is inert in respect of the surface oxidation, but it might interact with the silicon nitride surface, changing the mechanical properties of the cantilever. To eliminate this possibility, we conducted large-deflection tests in argon-rich environment as well, see figure 3-25. The damage healing and the stiffening of the cantilever were slower than in air, and the negative resonance frequency shift due to the large deflections was smaller as well. The resonant behaviour of the SiN cantilever was identical to that measured in the nitrogen-rich environment.

While the nitrogen- and argon-rich environments inhibit the surface oxidation, a humid environment, being an oxidizing medium, is bound to have the opposite effect on the resonant properties. The large deflection tests were repeated in air with an increased humidity. The resonance peak became wider and shifted to higher values in seconds when the sample was introduced to the humid environment. This indicates that surface oxidation and oxygen adsorption took place rapidly. If the humidity was further increased, the response curve became noisier and the resonance peak became yet wider. The stiffening effect was stronger than in ambient air. The resonance frequency drop upon large deflections was higher, and
Environmental tests

the following recovery of the frequency was faster than in air, see figure 3-24 and figure 3-25. This is explained by the presence of a thicker oxynitride and adsorbed surface layer and by a faster damage healing process.

![Graph showing frequency shift over time in different environments](image)

**Fig. 3-26 Repeated shock tests on SiC cantilevers in different environments.**

The same tests were carried out on silicon carbide cantilevers, see figure 3-26. The characteristic behaviour of the beams was comparable to that of the SiN cantilevers, but these beams proved to be less sensitive to the environmental effects. The curve slopes of the recovery phase measured in air, humid air, nitrogen- and argon-rich environments did not deviate as much as those of the SiN cantilevers. This can be explained with the outstanding chemical inertness of the SiC, where the oxidation of and adsorption on the surface are limited. Consequently, the stiffening and the crack healing mechanisms are slower.

Several micrometer thick crystalline silicon cantilevers did not show a measurable resonance frequency shift when the same mechanical shock was applied. The vibration amplitude slightly decreased and recovered just like in the case of the thin SiN cantilevers, but the resonance frequency was stable within 10Hz.
3.6 Numerical calculations - FEA

Finite-Element Analysis (FEA) [3.88,3.89] of the vibrating cantilever beams was performed using ANSYS and FEMAP in combination with various FEA solvers. FEA modelling was implemented as a design aid tool to predict and to optimize the mechanical and resonant behaviour of the structures.

3.6.1 SiN and SiC cantilevers

The resonating SiN and SiC cantilevers with different geometries, described in chapter 3.1.2 and chapter 3.1.3, were modelled with FEA. The resonance frequencies and the Young’s moduli of the beams supplied by the FEA models showed good agreement with the measured and analytically calculated results. Taking into account the air damping, we calculated the Rayleigh damping coefficients $\alpha$ and $\beta$ as

$$2d_i = \frac{\alpha}{2\pi f_i} + \beta 2\pi f_i$$  \hspace{1cm} (3-35)

where $f_i$ are the natural vibrating modes, and $d_i$ are the modal damping factors. The $f_{1,2}$ values of the first two vibrating modes were taken from the AFM measurements.

Stiffening effect

The adsorption- and surface oxidation-induced stiffening effect was modelled with FEA. The SiN cantilever beam was built up as a sandwich structure: a 320 nm thick SiN cantilever was covered with a 0.5nm thick oxide layer on both sides. All layers are represented by plate elements. The laminate beam was attached to a silicon substrate, just like the real structure. The oxide layer is under compressive stress, which results in expansion of the cantilever beam. The oxide layers have an asymmetric configuration, as shown in figure 3-27, generating static bending. The
Numerical calculations - FEA

FEA model of the stretched and bent cantilever beam is shown in figure 3-28.

![Schematic model of a SiN cantilever coated with native oxide on both sides.](image)

In the real structure there are several effects influencing the resonance frequency of the beam upon surface adsorption and oxidation. Not all of them can be easily described analytically, so FEA is a useful tool to handle the problems numerically.

- The dominant effect is the introduced surface stress, which was calculated by the Rayleigh method. The analysis is described in detail in chapter 3.3.2 in section “Resonance frequency”, and an example is given in chapter 3.3.3 under “Analytical results”. This effect can be modelled by redistributing the tensile forces on the nodes of the SiN beam. The modelled resonance frequency shift was in good agreement with the measured values. Typical values range up to 1%, depending on the beam material, the exposure time and the surrounding environment.

- The second effect incorporated in the model is the thickness increase of the beam. The effective thickness of the native oxide varied between 0.1 and 2nm, depending on the age of the sample and on the surrounding media. As expected from the analytical formulas, the resonance frequency has a linear dependence on the thickness. A relative resonance frequency increase of 0.2% per 1nm grown oxide
was calculated, see chapter 3.3.3 under “Age dependence” and showed good agreement with the FEA results. The effect of the thickness increase is approximately an order of magnitude smaller than the effect of the surface stress.

• The third effect is the corner effect, which cannot be simply calculated analytically. There is stress accumulation in the concave corners at the clamping, which affects the resonance frequency. The meshing was refined at the corners until the results converged. The effect is similar to the stress accumulation upon beam deflection. As the peak stress occurs at these sites, fatigue effects like crack generation start here first. The corner effect strongly depends on the corner configuration. The effect can be reduced with rounded corners, as shown in figure 3-29. Depending on the angle between the beam and the substrate and on the roundness of the corner, the resonance frequency shift due to the corner effect is in the order of 0.01%. Thus the geometry effects of the clamping are negligible compared to the first two effects.

All effects become more dominant with decreasing beam thickness.

Fig. 3-28 FEA model of a SiN cantilever, stretched and deflected by the asymmetric compressive surface stresses.
Fig. 3-29 Stress distribution on SiN cantilever due to surface oxidation - corner effect.
Undercut of Si (111) plane

The SiN and SiC cantilever beams have an undercut at the clamping due to the slow etching of the (111) plane, see figure 3-28 and figure 3-29. The width of this under-etched region is 1-2µm, depending on the etching time. FEA simulations showed that the beam with undercut can be handled as a regular beam whose beam length is increased with the width of the undercut. This approximation can be used safely for the first natural vibration mode, see table 3-1.

<table>
<thead>
<tr>
<th>Natural modes</th>
<th>Original ( F_{\text{res}} ) [kHz]</th>
<th>2µm longer</th>
<th>2µm undercut</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>18.9</td>
<td>-1.6%</td>
<td>-1.6%</td>
</tr>
<tr>
<td>2.</td>
<td>118</td>
<td>-1.7%</td>
<td>-1.7%</td>
</tr>
<tr>
<td>3.</td>
<td>315</td>
<td>-1.6%</td>
<td>-1.0%</td>
</tr>
<tr>
<td>4.</td>
<td>333</td>
<td>-2.1%</td>
<td>-1.5%</td>
</tr>
</tbody>
</table>

Tab. 3-1 FEA simulations compare \( F_{\text{res}} \) of a 200µm long beam with 2µm length increase vs. 2µm undercut
References

Chapter 3 Resonators

3.11 R. Wiesendanger, “Scanning probe microscopy and spectroscopy; methods and applications”, Cambridge University Press,
References

Cambridge, UK, 1994
3.26 N.J. Tao, S. M. Lindsay, S. Lees, *Biophys.*, 63, pp. 1165-1169, 1992
Numerical calculations - FEA

References

3.68 J.L. Yang, T. Ono, M. Esashi, Proc. of Transducers ’99, Vol. 2,
Numerical calculations - FEA

Sendai, Japan, pp. 1140-1143, 1999
3.72 W.W. Millins, in “Metal Surfaces”, American Society for Metals, Cleveland, Ohio, 1963
References


Chapter 4

Resonator Beams

Resonant beams have a wide range of applications in micromechanical devices. Single- and double-clamped beams are implemented as sensing elements in microsensors. The resonance frequency of a double-clamped beam, also referred to as a bridge, is strongly dependent on the stress in the structure. This phenomenon is exploited in force sensors, where an externally applied force generates axial forces at the clamping of the beam. The silicon bridge is excited and detected by means of a ZnO piezoelectric layer [4.1]. Resonant microbeams are used as strain-sensitive elements to replace silicon piezoresistors in conventional pressure sensors and accelerometers. The double-clamped beam is integrated in the diaphragm of the pressure sensor or in the flexure of the accelerometer. The deformation in the diaphragm or flexure generates strain in the beam, and therefore the resonance frequency of the beam shifts. The pressure or acceleration is converted directly into a frequency output. Such vacuum-enclosed, double-clamped, resonant polysilicon microbeams exhibit extremely high quality factors, typically over 25000 [4.2]. Gage factors above 1000 were obtained, while the gage factors of metal piezoresistors are typically less than 5, while for single-crystal silicon it is less than 125. Highly sensitive angular rate sensor was reported with a single-clamped silicon cantilever beam as vibrating mass [4.3]. Linear output, low fabrication costs and suitability for mass-production were obtained with the fairly simple microstructures. These silicon/glass multilayer gyroscopes implement 2-axis resonant operation of the beam. Electromagnetic or piezoelectric excitation can be used in the different
sensor configurations. Tuning the geometry of the vibrating beam, feedback control on the reference vibration and reduction of the zero-point offset has resulted in a device that is 50 times more sensitive than comparable conventional ones [4.4]. Various industries implement Vibration Signature Analysis (VSA) for health monitoring of mechanical and electromechanical systems [4.5]. It is an effective tool that is widely used in condition-based maintenance of rotating machinery such as turbines, generators, etc. Tracking the vibration signature of critical components helps to detect wear and pending failure and allows maintenance before actual failure or performance degradation occurs. The relevant frequencies range from a few tens to several hundreds of hertz, and are caused by surface irregularities and defects of moving parts [4.6]. Conventional VSA systems extract the health-state information from a frequency spectrum. The spectrum is obtained using time-domain accelerometers, A/D converters and Fast Fourier Transform (FFT) hardware and software. Substantial performance improvement and cost reduction can be achieved by implementing MEMS vibration sensors, which allow a wider set of applications. These frequency-resolved sensors work directly in the frequency domain, providing easy processing of the digital signal, and they can be tuned for a specific frequency band [4.7] [4.8]. The most frequently used approach is the ‘tuning-fork’ set-up, which consists of a parallel array of uniform, high quality factor cantilever beams with different lengths [4.9]. Each beam is tuned for a specific resonance frequency, and detects the acceleration of a component of the vibration. The key is that one beam is only sensitive to one very narrow frequency range, where the width depends on the quality factor of the beam. Such micromechanical cantilever beams can reach very high quality factors (10<Q<25000 depending on packaging), comparable to those of high-end electronic resonators. The vibration spectrum is reconstructed by the discrete components measured by the large number of beams with closely spaced resonance frequencies. The multiplexer and other signal conditioning electronics can be integrated on-chip, resulting in improved sensitivity and reduction of size, power consumption and cost.

This chapter introduces resonant microbeams and paddle beams, forming tuning-fork-type vibration sensors. The paddle beams can operate as accelerometers as well. The main goal of the work was to study the long-term stability of and environmental effects on single-crystal silicon
beam resonators. The electrostatic charging-induced adsorption (‘flycatcher’ effect) was first encountered in the form of a stability problem; later on, it was exploited to detect air pollution.

### 4.1 Sensor design

The vibration sensors implement crystalline silicon beam and paddle beam sensing elements. To detect mechanical resonance, the corresponding stress in the cantilevers and paddle beams is converted into an electrical resistance change using implanted piezoresistors. The designed beams are 200-600\(\mu\)m long, 20\(\mu\)m wide and 4.7\(\mu\)m thick. The paddle beams have a paddle that is 110-210\(\mu\)m wide and 150-300\(\mu\)m long attached to the free end of the beam to lower the resonance frequency. This configuration provides more mass without affecting the flexibility of the suspending beam. The paddle beams suspended on two beams provide easy separation of bending and torsional modes by comparing the resonant behaviour of the two suspending beams. Similar paddles that are supported by two arms were used for flow detection, where a gas velocity as low as 1-3cm/s could be measured [4.10]. To obtain high efficiency, the piezoresistors are placed at the clamp of the beams, which is the most stressed region. Finite-element simulations showed that this region takes up 5-10% of the beam length, beginning at the base of the beam. The sensing resistors are connected to reference resistors on the substrate in a half Wheatstone bridge configuration for easy readout. The vibration-induced periodical resistance change is converted to an AC voltage. The Wheatstone bridge configuration is usually implemented for temperature compensation [4.11], which is important as the piezoresistive effect has a temperature dependence in the range of a few tenth of a percent at room temperature [4.12] [4.15]. The temperature dependence of the piezoresistors is not a problem in case of our microbeam resonators, because the temperature-induced resistivity drift is negligible during one resonance frequency measurement. As the aim of this work is to study the reliability and long-term stability of the sensor structure itself, the readout electronics is not integrated on-chip. Besides the piezoresistive readout, optical readout of the sensors is available as well [4.13], [4.14].
Reflective pads are deposited onto the free end of the beams and paddles. Some of the structures are coated with metal all along for electrostatic driving. The substrate of the sensor is designed such that it fits in the AFM head. The structures can be characterised with optical readout using the AFM method described in chapter 3.2. All the masks are patterned and the structures are etched from the frontside. As no double-sided alignment is required, the alignment accuracy could improve from 10µm to 0.2µm.

Fig. 4-1  Process flow of the Si vibration sensors
4.1.1 Process flow

A 4µm thick n-type silicon epi layer was deposited on the p-type silicon wafer, see figure 4-1. The beams and paddle beams were defined with deep boron diffusion into the n-type silicon layer. The piezoresistors were formed with p+ implantation. 100nm thick LPCVD silicon nitride was deposited, where contact holes were opened to the resistors and etch holes for exposing the diffused p-type silicon. On top of a thin chromium layer for better adhesion, gold metalization was deposited. The gold layer served both as electric interconnects and as a cathode for the electrochemical etching. Ashraf’s contactless electrochemical etching technique was implemented using a deposited gold cathode on the passive areas of the chip surface [4.15]. The cathode was in contact with the n-type epi, where the contact resistance was lowered by shallow n+ implantation. Following a 30sec HF dip, the wafers were etched in 25% TMAH at 80°C. The free-standing structures were protected with a thick photoresist coating during dicing of the chips.

4.1.2 The structures

The silicon beams and paddle beams are shown on figure 4-2. Besides by the piezoresistive readout, the resonators are characterized in an AFM head using optical readout. Therefore reflective pads are deposited onto the free end of the beams. Some beams and paddle beams are coated with gold all along the beam length for electrostatic driving.

The contactless galvanic etching technique has the advantage of being simple [4.15]. The wafer is immersed in the etching solution, and after the calculated etching time it is rinsed in water and dried. The drawback of the method is the poor uniformity. The beams are not perfectly underetched. Figure 4-3 shows the initial etching profile. After 4 hours of etching, the structures are free standing, but not ideally underetched. Some p-type silicon remains under the beams even after a longer etching time. This incomplete etching is an artifact of the electrochemical etch-stop technique.
Another difficulty is the remaining debris after the lift-off patterning of the gold/chromium metalization. It cannot be removed with simple rinsing in acetone or water.
The gold metalization can suffer fatigue failure due to poor adhesion. It is standard practice in IC and MEMS technology to deposit a chromium or titanium intermediate layer between the gold and the silicon to improve the adhesion. However, insufficient adhesion was found on some structures. Though the gold metalization is at least 2µm away from the moving beams, the lateral shear stress damaged some of the structures. The metalization peeled off at the highly stressed regions, see figure 4-4.

![Au/Cr metalization peels off due to shear strain.](image)

4.2 Theory

4.2.1 Beam theory

The free-standing cantilever beam with one clamped end is the simplest resonator. The analytical description of the bending and resonant behaviour is given in this chapter. Fairly accurate results are achieved assuming the following: there is a

- a uniform prismatic beam with a rectangular cross section
- uniform mass distribution
- infinitely stiff clamping
- no internal damping.
As the damping in the beam material is neglected, the quality factor is only determined by the air damping. Integration of the linear stress-strain relationship (Hooke’s law $\sigma = \varepsilon E$) gives the equations used in the analytical calculations [4.11]. These equations are shown in table 4-1, where

- $L/ w/ h =$ length / width / thickness
- $E =$ Young’s modulus
- $\rho =$ mass density
- $x =$ running position coordinate along the beam’s length
  \hspace{1cm} x = 0$ at clamped end
- $P =$ inertial pressure (from acceleration)
- $a =$ applied acceleration
- $Q =$ quality factor
### Theory

<table>
<thead>
<tr>
<th><strong>Stress distribution:</strong></th>
<th>( \sigma_m(x) = \frac{3P(L-x)^2}{h^2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Elastic line:</strong></td>
<td>( z(x) = \frac{K}{12} (L-x)^4 + \frac{K}{3} L^3 x - \frac{K}{12} L^4 )</td>
</tr>
<tr>
<td></td>
<td>( K = \frac{6P}{E h^3} )</td>
</tr>
<tr>
<td><strong>Max. stress @ x=0:</strong></td>
<td>( \sigma_m(0) = \frac{3P L^3}{h^2} )</td>
</tr>
<tr>
<td><strong>End deflection @ x=L:</strong></td>
<td>( z(L) = \frac{3P L^4}{2E h^3} )</td>
</tr>
<tr>
<td><strong>Center of gravity coordinate:</strong></td>
<td>( x_m = \frac{L}{2} )</td>
</tr>
<tr>
<td><strong>Deflection @ x_m:</strong></td>
<td>( z(x_m) = \frac{17P L^4}{32E h^3} )</td>
</tr>
<tr>
<td><strong>Integrated force F:</strong></td>
<td>( F = P \cdot w \cdot L )</td>
</tr>
<tr>
<td><strong>Mass m:</strong></td>
<td>( m = \rho \cdot w \cdot h \cdot L )</td>
</tr>
<tr>
<td><strong>Stiffness constant k:</strong></td>
<td>( k = \frac{F}{z(x_m)} = \frac{32E w h^3}{17L^2} )</td>
</tr>
<tr>
<td><strong>1\textsuperscript{st} natural frequency:</strong></td>
<td>( \omega_n = \sqrt{\frac{32E}{17p}} \left( \frac{h}{L^2} \right) )</td>
</tr>
<tr>
<td><strong>Static Sensitivity S\textsubscript{0}:</strong></td>
<td>( S_0 = 3\rho \left( \frac{L^2}{h} \right) \left[ \frac{Pa}{m \cdot s^2} \right] )</td>
</tr>
<tr>
<td><strong>Dynamic Sensitivity S:</strong></td>
<td>( S = Q \cdot S_0 )</td>
</tr>
</tbody>
</table>

*Tab. 4.1 Beam theory [4.11]*.
4.2.2 Paddle beam theory

Similar to the beam theory, the paddle beams are described with equations resulting from integration of Hooke’s law, see table 4-2 [4.11]. The same formulation, assumptions and boundary conditions were used as in the beam theory above, see chapter 4.2.1. As the damping in the beam material is neglected, the quality factor is determined by only the air damping. The stress-strain relations are integrated along the length of the paddle beam. Note that the width has a step at the beam/paddle transition at $x = \gamma L$, while the width was uniform along the simple beams.

4.2.3 Beam vs. paddle beam

A regular beam and a paddle beam are compared using the equations described above. The length of the beam and of the paddle beam is 300$\mu$m. The paddle beam’s suspension arm is 150$\mu$m and the paddle is 150$\mu$m long (thus $\gamma=0.5$). The width of the paddle is 110$\mu$m. The resonance frequency of the paddle beam is approximately half of the resonance frequency of the regular beam. The resonance frequency of a resonator with a given length can be tuned with the size of the paddle. This is useful in applications where the total length of the structure is limited. Furthermore, not only the resonance frequency of the paddle beam is different, but the stress on its piezoresistors as well. The same acceleration generates a four times higher stress on the piezoresistor of the paddle beam than on that of the regular beam. The sensitivity of the resonator is proportional to the stress at the clamping site ($x=0$). Consequently the sensors implementing paddle beams are more sensitive to acceleration and vibration.
RELIABILITY OF MICROMECHANICAL RESONATORS

Theory

Stress distribution:

\[ \sigma_w(x) = \frac{3P(\gamma L - x)^2}{h^2} + \frac{3P(w_2/w_1)}{h^2} L (1-\gamma) \left[ L (1+\gamma) - 2x \right] \]

\[ \sigma_w(x) = \frac{3P(L-x)^2}{h^2} \]

Elastic line:

\[ z(x) = \frac{K}{12} (\gamma L - x)^2 - \frac{K}{3} \left( \frac{w_2}{w_1} \right) L (1-\gamma) x^3 + \]

\[ \frac{K}{2} \left( \frac{w_2}{w_1} \right) L^2 (1-\gamma^2) x^2 + \frac{K}{3} \gamma^2 L^3 x - \]

\[ \frac{K}{12} \gamma^4 L^4 \]

\[ z(x) = \frac{K}{12} (L-x)^2 + K L^3 x \left[ \gamma \left( \frac{w_2}{w_1} \right) (1-\gamma) + \frac{\gamma^3}{3} \left( \frac{1-\gamma}{3} \right) \right] \]

\[ - \frac{K L^3}{12} \left[ \gamma (1+2w_2/w_1) + 2 \gamma^2 \left( \frac{w_2}{w_1} \right) (3-4\gamma) + (1-\gamma)^2 (1+3\gamma) \right] \]

\[ K = \frac{6P}{E h^3} \]

Max. stress at x=0:

\[ \sigma_w(0) = -\frac{3P L^2}{h^2} \left[ \gamma^2 + \left( \frac{w_2}{w_1} \right) (1-\gamma^2) \right] \]
Resonator Beams

End deflection @ x=L:

\[ z(L) = K \cdot L^4 \left[ \frac{\gamma^2}{12} \left( 1 + 2 \frac{w_z}{w_1} \right) (4 - \gamma) + \frac{1}{4} (1 - \gamma)^3 + \gamma \cdot \frac{w_z}{w_1} \left( 1 - \frac{3 \gamma}{2} \right) \right] \]

\[ K = \frac{6P}{Eh^3} \]

Center-of-gravity Coordinate:

\[ x_m = \frac{L}{2} \frac{\left( \frac{w_z}{w_1} \right) (1 - \gamma^3) + \gamma^2}{\left( \frac{w_z}{w_1} \right) (1 - \gamma) + \gamma} \]

Deflection @ x_m:

\[ z(x_m) = \text{Substitution} \]

Integrated force F:

\[ F = P \cdot L \left[ \gamma \cdot \frac{w_z}{w_1} + (1 - \gamma) \cdot w_z \right] \]

Mass m:

\[ m = \rho \cdot h \cdot L \left[ \gamma \cdot \frac{w_z}{w_1} + (1 - \gamma) \cdot w_z \right] \]

Stiffness constant k:

\[ k = \frac{F}{z(x_m)} = \text{Substitution} \]

1st Natural frequency:

\[ \omega_n = \sqrt{\frac{k}{m}} = \text{Substitution} \]

Static Sensitivity \( S_z \):

\[ S_z = 3P \left( \frac{L^3}{h} \right) \left[ \gamma^2 + \left( \frac{w_z}{w_1} \right) (1 - \gamma^3) \right] \left[ \frac{P\rho}{m/s^2} \right] \]

Dynamic Sensitivity S:

\[ S = Q \cdot S_0 \]

*Tab. 4-2 paddle beam theory [4.11]*

RELIABILITY OF MICROMECHANICAL RESONATORS
4.3 Measurement techniques

4.3.1 AFM measurements

The resonant properties of the vibration sensors, such as their resonance frequency and quality factor, can be characterized with the optical readout technique in an AFM head, see chapter 3.2.3. The driving frequency sweeps through the whole range to map the resonant response of the cantilever. The peaks give the frequencies of the resonant modes. Figure 4-5 shows a typical resonance peak measured on a paddle beam.

![Fig. 4-5  Resonance peak of a paddle beam measured with AFM](image)

The AFM is implemented for quasi-static bending tests as well chapter 3.2.2. The resonator chip is mounted on the sample stage, and the AFM tip applies a deflecting force on the beams and the paddle beams, one at a time. The deflection of the beam can be calculated, and the resistance of the piezoresistor can be measured in situ if the chip is wire bonded in a package. The spring constant of the beam and the sensitivity of the piezoresistors can be characterised with this technique.
4.3.2 Piezoresistive measurements

In order to investigate environmental effects, the tests need to be independent from the AFM and from clean room conditions. The vibration sensor chips are glued on multi-layer piezoactuators, see figure 4-6, and mechanically excited with a frequency sweep, similar to the AFM method. The deflection of the cantilevers is detected with implanted piezoresistors.

The relative resistance change \( \frac{dR}{R} \) of a piezoresistor upon beam deflection can be calculated as

\[
\frac{dR}{R} = \frac{d\rho}{\rho} = \pi_{||}\sigma_{||} + \pi_{\perp}\sigma_{\perp}
\]  

(4-1)

where \( \rho \) is the resistivity, \( \sigma \) is the deflection-induced stress and \( \pi \) is the piezoresistive coefficient, which depends on the crystallographic orientation. The indexes \( || \) and \( \perp \) denote the components parallel and
perpendicular to the resistor. The piezoresistors form half Wheatstone bridges with reference resistors on the substrate. $U_0$ voltage is connected to the half-bridge, which is distributed on the sensing and reference resistors according to their resistance. The measured voltage on the sensing resistor is given as

$$U = U_0 \frac{R_{\text{sens}}}{R_{\text{sens}} + R_{\text{ref}}}$$  \hspace{1cm} (4-2)

The function generator and the detecting oscilloscope are controlled with a computer running HP-VEE software. Long-term cycling test are conducted conveniently with the PC controlled measurement setup. The resonator is driven at the resonance frequency, which is measured and re-adjusted accordingly every 1 hour. Another advantage of the method is that the packaged chip is small, and therefore, fits in any environmental chamber, while the controlling and data acquisition components are outside the chamber. Cycling tests were conducted in ambient air and in environments with various filtered dust and smoke contents.

### 4.3.3 Alpha-stepper bending tests

This test is similar to the quasi-static tests conducted in the AFM (chapter 3.2.2), but in this case the deflecting force is applied by the $\alpha$-stepper tip instead of the AFM tip. This tip is much larger, and the applied force can be much larger too. The advantage of the $\alpha$-stepper test is that the applied force is measured accurately, while the force calculated from the spring constant of the AFM cantilever can have an error up to 50\% [4.16] [4.17]. This means that the $\alpha$-stepper test is appropriate for calibration purposes, because the deflecting force acting on the sample beam is known accurately. The tip scans the surface following the topography. The paddle beam deflects under the force, and the curvature of the deflected beam is measured by the tip of the $\alpha$-stepper. The tests can be carried out on wafer level or on packaged chips that are open on the top. In the latter case, the resistance of the piezoresistors can be measured in situ during the bending test. This allows simultaneous characterization of the bending stiffness and the sensor output.
4.4 Measurement results

4.4.1 Quality factor

The quality factor $Q$ of a resonating beam is calculated from the resonance peak as described in chapter 3.2.3; $Q=200-300$. These values are 2-3 times higher than the ones obtained by Benecke et al. [4.9]. The difference is explained by the difference of the beam size: their beams were twice as long and 4 times wider with a 4 times larger paddle at the free end, which involves higher air damping. The quality factor of the crystalline silicon resonator is higher than that of the silicon nitride and silicon carbide cantilevers, measured under the same conditions.

4.4.2 Resonance frequency

The resonance frequencies were measured with both the AFM and the piezoresistive techniques. The silicon resonators are stable, show no stiffening effect (chapter 3.3) or degrading shock response (chapter 3.4), unlike the SiN and SiC cantilevers. The reason is not the different material of which the resonators are made, but the different thickness. The silicon resonators are 10-40 times thicker than the different SiN and SiC structures. Other groups measured the crystallographic orientation dependent resonance frequency shift on submicron thin crystalline silicon cantilevers [4.18].

The measured resonance frequencies of beams and paddle beams differ from the simulated and calculated results. The resonance frequency of a regular cantilever beam is given by

$$f_{res} = \frac{A}{2\pi \sqrt{EI/\rho L^4}}$$

(4-3)

where $f_{res}$ is the resonance frequency, $A$ is a coefficient ($A=3.52$ for the first natural mode of a single-clamped cantilever), $E$ is the Young’s modulus, $I$ is the area moment of the inertia of the beam cross section, $L$ is
the beam length, and $\rho L$ is the mass per unit length of the beam [4.19]. The calculated resonance frequencies are in good agreement with finite-element simulations, but differ substantially from the measured results, see table 4-3. A beam thickness of 4.7\(\mu\)m was used in the calculations. The thickness of the fabricated structures is non-uniform and much greater than the designed 4.7\(\mu\)m. This is due to the incomplete silicon removal under the beams during the electrochemical etching. The effective beam thickness can be calculated from the measured resonance frequencies of the different beams. The bending stiffness of our beams with non-rectangular cross section (V-shaped bottom) and non-uniform thickness compares to that of a 8\(\mu\)m thick ideal beam, thus the effective beam thickness is 8\(\mu\)m.

The relative deviation of resonance frequencies of well-defined beams is determined by the deviation of their thickness. The resonance frequency is sensitive to the shape and dimensions of the resonator. Identical resonator beams from different locations on the wafer were compared. The non-uniformity of the etch-stop technique resulted in a scattering of the measured resonance frequencies within 5%. The non-uniformity of the contactless electrochemical top-side etching was studied by Ashruf [4.15]. Similar incomplete p-type silicon removal was reported for the use of the electrochemical p-n etch-stop [4.20], [4.21], [4.23], and for the fabrication of silicon membranes [4.15].

### Tab. 4-3 Calculated and measured resonance frequencies of regular Si cantilever beams. Width = 20\(\mu\)m.

<table>
<thead>
<tr>
<th>Beam Length [(\mu)m]</th>
<th>Calculated $F_0$ [kHz]</th>
<th>Mean Measured $F_0$ [kHz]</th>
<th>Rel. Max. deviation [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>161.9</td>
<td>281.7</td>
<td>3.9</td>
</tr>
<tr>
<td>300</td>
<td>71.9</td>
<td>135.1</td>
<td>3.6</td>
</tr>
<tr>
<td>400</td>
<td>40.5</td>
<td>82.9</td>
<td>4.2</td>
</tr>
<tr>
<td>500</td>
<td>25.9</td>
<td>57.3</td>
<td>1.6</td>
</tr>
<tr>
<td>600</td>
<td>18.0</td>
<td>46.0</td>
<td>2.8</td>
</tr>
</tbody>
</table>
Resonator Beams

The resonance frequencies of the paddle beams measured with the AFM technique are shown in table 4-4.

Tab. 4-4 Measured resonance frequencies of Si paddle beams. Suspension width = 20µm.

<table>
<thead>
<tr>
<th>Suspension length [µm]</th>
<th>Pad. width [µm]</th>
<th>Pad. length [µm]</th>
<th>Gold coating</th>
<th>Res. freq. [Hz]</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>210</td>
<td>300</td>
<td>end</td>
<td>21330</td>
</tr>
<tr>
<td>350</td>
<td>110</td>
<td>150</td>
<td>end</td>
<td>32970</td>
</tr>
<tr>
<td>350</td>
<td>110</td>
<td>150</td>
<td>end</td>
<td>36080</td>
</tr>
<tr>
<td>150</td>
<td>110</td>
<td>150</td>
<td>end</td>
<td>81810</td>
</tr>
<tr>
<td>300</td>
<td>210</td>
<td>300</td>
<td>full</td>
<td>18640</td>
</tr>
<tr>
<td>350</td>
<td>110</td>
<td>150</td>
<td>full</td>
<td>29980</td>
</tr>
<tr>
<td>350</td>
<td>110</td>
<td>150</td>
<td>full</td>
<td>32490</td>
</tr>
<tr>
<td>150</td>
<td>110</td>
<td>150</td>
<td>full</td>
<td>73990</td>
</tr>
</tbody>
</table>

4.4.3 Accelerometer calibration

The paddle beam can be considered as a seismic mass (the paddle) attached to a low-spring-constant suspension arm (the beam). This is the basic structure of bulk micromachined accelerometers. The paddle beams were characterised as accelerometers. The paddle beam is mounted on the AFM sample stage, and the AFM tip applies the load on the beam, see chapter 3.2.2. It was also explained in that chapter that the spring constant of the sample should match that of the AFM cantilever to achieve a higher measurement accuracy. The position \( x_c \) along the length of the paddle beam which fulfils this criteria can be found by simply reading out the spring constant from the measured force curve. At the critical \( x_c \) site, the measured global spring constant is half of the spring constant measured on the substrate. The latter is purely the spring constant of the AFM tip.
(supposing that the substrate surface is much harder than the cantilever), while the global spring constant is the combination of the spring constants of the AFM tip and the sample beam connected in series, see chapter 3.2.2. Consequently, at site $x$, the vertical displacement of the AFM tip $z=d_A+d_s(x_c)$ and $d_A=d_s(x_c)$, where $d_A$ and $d_s(x_c)$ are the deflections of the AFM and sample cantilever beams, respectively. The vertical displacement of the AFM cantilever is controlled by the $z$-piezoactuator and the stepper motor with $1\mu$m step size.

![Graph showing the relationship between deflection of beam and relative resistance change.](image)

**Fig. 4-7 Quasi-static deflection test on Si paddle beam.**

Figure 4-7 shows the characteristic behaviour of a paddle beam. The deflection was obtained with several back and forth steps of both the $z$-piezoactuator and the stepper motor. The resistance change of the piezoresistor has a linear dependence on the deflecting force. Thus the accelerometer has linear response to linear acceleration change. At higher deflections ($>16\mu$m) the curve flattens, indicating that the beams are in the non-linear region.

The applied force on the cantilever can be calculated from the spring constants. As the spring constants of the AFM cantilevers given by the manufacturer can have errors up to 50% [4.16] [4.17], this data cannot be used for absolute calibration of the device. Therefore another calibration test was carried out on the paddle beam by means of an $\alpha$-stepper. The tip
Resonator Beams

scanned along the beam with an $F = g \cdot 15.5 mg$ constant force. The obtained deflection curve is shown in figure 4-8; it was measured on the smallest paddle beam (length = 150µm beam + 150µm paddle). The deflection $h$ of a beam with a rectangular cross section can be calculated as

$$h(x) = \frac{Fx^3}{3EI}$$

(4-4)

where $E$ is the Young's modulus, $I$ is the area moment of inertia of the beam's cross section and $x$ is the running coordinate along the beam, where $x = 0$ at the clamping. The calculated curve is plotted on figure 4-8. The area moment of inertia of the beam's cross section is

$$I = \int z^2 dA$$

(4-5)

where $z$ is the distance of the $dA$ cross section area unit from the neutral plane of the beam. The neutral plane is the plane which does not suffer from deformation during the deflection. The neutral plane is the mid-plane of an ideal beam with a rectangular cross section. For such a beam

$$I = \int \int z^2 \; dz \; dy = \frac{wh^3}{12}$$

(4-6)

Our beams have non-ideal cross sections, which results from the contactless electrochemical etching process: a small triangle is added to the rectangle. Integrating on this cross section, $I$ is 3.6 times higher than that of the rectangular cross section. Furthermore, the thickness is not uniform along the beam; the beam is thicker at the clamping. This non-uniform thickness and the non-ideal cross section together are responsible for the deviation of the measured results from the calculated curve. The beam width has a step at $x = 150 µm$, where the paddle is jointed to the suspending beam. The paddle region acts as rigid body because its bending stiffness ($EI$) is 7.5 times higher than that of the suspension beam. Loading the paddle can be considered as a bending momentum applied on a spring. The suspending beam serves as the spring, and the
momentum is proportional to the distance where the loading force acts. Consequently the measured deflection curve is linear above 150 µm.

![Deflection curve](image)

**Fig. 4-8**  α-stepper loading test on Si paddle beam, calculated and measured deflection curves

The resistance of the piezoresistor was measured during the α-stepper deflection tests. Figure 4-9 shows the relative resistance change of the 932 Ohm piezoresistor measured on the same paddle beam. The centre of gravity coordinate of the beam calculated from table 4-2 is $x = 202 \mu m$. The resistance change calculated from the measured resistance shift curve is 2.54% if a $g \cdot 15.5$ mg force acts at the centre of gravity coordinate. The corresponding beam deflection at this site is $-2.73 \mu m$. The resistance shift is 3.76% and the deflection is $-4.88 \mu m$ when the same force is applied at the end of the beam ($x = 300 \mu m$). These values are used for calibration. The data at the centre of gravity coordinate is particularly important, because this is used in calculations of vibrating beams and deflections due to acceleration. From calculations based on the resonant measurements in air and the equations in table 4-2, the following results were obtained for the 300 µm long paddle beam. The measured maximum resistance change during resonant vibration is 1.27%, which corresponds to a 1.37 µm maximum deflection of the free end. The Q-factor is 250. The maximum stress generated at the clamping of the beam is 37.4 MPa. The Yield
strength of silicon is around 7GPa, so no failure or fatigue is expected, and the vibration can be safely described applying the elastic theory.

4.4.4 ‘Flycatcher’ effect

The long-term stability of the vibrating beams was tested. Several weeks long cycling tests were conducted in ambient air as described in chapter 4.3.2. No stiffening effect (see chapter 3.3) or degrading shock response (see chapter 3.4) was observed on these robust crystalline silicon beams and paddle beams. These 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, which we called the ‘flycatcher’ effect [4.22]. The mass loading of the adsorbates lowers the resonance frequency. The resonance frequency decreases constantly during the weeks-long tests, as shown in figure 4-10.
The effect of adsorbates on the resonance frequency was discussed in chapter 3.3.2 section “Resonance frequency”. The ‘flycatcher’ effect is governed by the mass loading, while the spring constant change due to adsorption is negligible. Therefore the resonance frequency shift can be described as [4.26]

\[
f_2 = f_1 \left(1 - \frac{1}{2} \frac{\delta m}{m_{\text{eff}}} \right)
\]

(4-7)

where \( m_{\text{eff}} = n m \) is the effective mass of the beam, \( m \) is the mass and \( n \) is a constant depending on the geometry of the beam. The adsorption rate can be defined as the mass increase due to adsorption on 1\( \text{m}^2 \) surface area in 1 sec. The adsorption rate in ambient air measured on a 300\( \mu \text{m} \) long vibrating silicon paddle beam was \( 3.6 \cdot 10^{-13} \ \text{kgm}^{-2}\text{s}^{-1} \), where the deflection of its free end was calculated to be 1.37\( \mu \text{m} \) (see chapter 4.4.3). The adsorption rate depends on the environment (particle size and density) and on the amplitude of the vibration.
Resonator Beams

Note that the resonance frequency shift is linear in time, unlike the logarithmic shift of thin cantilevers caused by the environment-induced stiffening effect. This is due to the difference between the two adsorption mechanisms. The adsorption of atoms and molecules depends on the surface coverage and scales logarithmically in time. The change of surface stress governs the resonance frequency shift of the submicron thin SiN and SiC cantilevers. The mass loading of the few atomic layers is negligible, and because the experiments are done in clean room environment, no large airborne particles are present. However, the robust Si beams are tested in ambient air. The surface stress-related effects are negligible due to the lower surface-to-volume ratio. The mass loading determines the resonance frequency shift, which is linear in time. Saturation effect was observed in extreme cases though, when the beam was vibrating in cigarette smoke and the surface was fully contaminated with large particles, see below.

Remarkably, the beam at resonance gathers dust particles, while the neighbours stay clean. The key to this is the higher vibration amplitude. Figure 4-11 shows a tested beam and two neighbouring beams on the same chip, which were subjected to the same environment during the test. The chip was excited at the resonance frequency of the shorter paddle beam. The resonance frequency shift of the shorter paddle is plotted in figure 4-10. The neighbouring beams remained uncontaminated. The piezoresistive readout technique does not introduce charging of the beam. This was confirmed with experiments, where the piezoresistors were disconnected during the one-hour interval between two measurement points, and similar resonance frequency shift was obtained. We can conclude, that the charge built up on the resonating beam is a result of the interaction between the vibrating beam and the airborne particles.

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/photoresist clusters remaining from the lift-off process could not be completely removed with acetone, but came off the surface during the vibration, and stuck on the vibrating beam. The contamination pattern showed that this is not a migration along the surface. The particles are adsorbed from the surrounding media, and the contamination pattern is correlated to the motion of the beam.
In order to further characterize the electrostatic charging-induced mass loading effect, we placed the chip with the silicon paddle beam in an environmental chamber. The previous experiment was continued for another 90 minutes. At this point, filtered cigarette smoke was introduced in the chamber. Consequently, the resonance frequency dropped abruptly, see figure 4-12. The adsorption rate calculated from the linear region of the curve using equation (4.7) is $3.8 \times 10^{-10} \text{kg m}^{-2} \text{s}^{-1}$. The surface started to saturate after 8 hours. No further adsorption was observed after 12 hours, so the resonance frequency stabilized. It is clear from figure 4-12, that the resonance frequency shift is proportional to the particle content of the environment. The relative resonance frequency shift due to the smoke is 17 times larger than the shift in ambient air in 3 weeks.
In another experiment a gold-coated paddle beam was first driven at its resonance frequency in an open chamber, so in ambient air. A slow linear frequency decrease was measured, similar to the previous experiment shown in figure 4-10. 22 hours later, fine smoke was introduced in the measurement chamber with two matches. Consequently the resonance frequency dropped, see figure 4-13. The adsorption rate calculated from the linear drop using equation (4-7) is $2.3 \times 10^{-9}$ kgm$^{-2}$s$^{-1}$. The smoke settled in 2 hours on the walls of the chamber and the resonance frequency stopped decreasing. Note that the smoke from the matches settled much faster than the filtered cigarette smoke particles. As the airborne particle content of the chamber dropped, the paddle beam started to release some of the adsorbed particles, hence the resonance frequency increased. Again, only the resonating beam got contaminated; the rest of the chip remained clean, as can be seen in figure 4-14.
Fig. 4-13 ‘flycatcher’ effect measured on a gold-coated paddle beam. The resonance frequency dropped when smoke was introduced in the chamber. Some of the adsorbed particles were released after the smoke had settled.

These results point out that even if vacuum package is not necessary, dust-free protective packaging is required for ambient applications to maintain a stable resonance frequency. There are numerous microbeam resonator based applications, functionalized probes, exploiting the mass loading similar to the ‘flycatcher’ effect, see next paragraph for details. A localized region of the resonator is coated with an active layer which selectively adsorbes the desired components from the environment. The mass increase results in a resonance frequency shift, which can be measured directly. In this type of sensors the resonator structures are in direct contact with the surrounding media, so packaging is not an option. Special care needs to be taken to prevent electrostatic charging and the consequent catching of undesired particles. The ‘flycatcher’ effect results in an environment-dependent drift of the sensor and/or leads to false measurement results.
Resonator Beams

Resonant air pollution detector

Resonant microcantilever probes, such as those designed for atomic force microscopy, are recently used in a wide variety of applications. Microcantilever based structures proved to be a universal platform for a spectrum of physical, chemical and biological sensors providing real-time in-situ measurements and parts-per-billion (ppb) to parts-per-trillion (ppt) detection sensitivity [4.24] [4.25]. The adsorption-induced surface stress and mass loading generate deflection and variations of the resonance frequency (see chapter 3.3 and chapter 4.4.4). Microcantilevers coated with metal on one side implement the bimetallic effect, and undergo static bending as a result of minute temperature variations [4.26] [4.27]. Exothermic adsorption of chemicals on the surface leads to measurable thermal effects as well. Coating the cantilevers with hygroscopic materials allows one to measure the relative humidity with the static bending method with high efficiency [4.28]. The concept can be extended to different functionalized probes for the detection of various chemical vapors.
It was shown above that the ‘flycatcher’ effect is strongly dependent on the particle size and density of the surrounding environment, and the effect is reversible. This suggests that the electrostatic charging-induced adsorption can be exploited for sensing purposes, to build a smoke sensor or airborne contamination detector. Measuring the change of the resonance frequency of the vibrating structure over time, one can conveniently obtain information on the particle size and density. Such a sensor can monitor the air pollution with a simple resonating structure providing a digital frequency output. Regular beams and paddle beams were tested, and the regular beams proved to be more sensitive sensors. This is not surprising, as the resonance frequency of these beams is a few times higher than that of the paddle beams. The smaller surface does not affect the sensitivity, because the mass of the beam is smaller as well. Thus the relative mass increase of a regular beam and that of a paddle beam would be the same if the resonance frequencies were equal. Of course the thickness of the beams, the measurand media and the spatial distribution pattern of the adsorbed particles are considered to be the same as well.

How can we reset the sensor? This is a key issue in chemical sensors and functionalized probes. These structures have an active area, which selectively adsorbes desired components of the surrounding media [4.29]. The quantity of the adsorbed molecules can be translated to output signals such as resistivity, conductance, or capacitance change, static bending of the supporting cantilever, or resonance frequency shift of a vibrating structure. When the active area is saturated, the sensitivity drops. This was found in our experiments as well, see above in figure 4-12. Conventional chemical sensors and probes implementing the adsorption phenomenon often require high-temperature treatment or other tricks to reset the sensor. The treatment can involve dismounting the sensor, or replacing the sensing element with a new one. This is not only an expensive resetting solution, it disturbs the measurand system as well. In case of conventional systems, simply turning the sensor on and off can be a difficult task. When the sensor is in contact with the measurand environment, it reacts with the media, even if no results are read out. The lifetime of the sensor could be increased substantially, if the sensor could be switched off and stay passive without being removed from the measurand media. Our vibrating system exploiting the electrostatic ‘flycatcher’ effect offers convenient on/off switching and resetting. The sensor can stand by inactively in the
measurand media if it is not driven at resonance. Without resonant vibration, no electrostatic charging occurs, and thus no airborne particles are adsorbed on the beam surface. The sensor is turned on for the desired period of the measurement. When the slope of the resonance frequency shift is determined, which is directly proportional to the mass of the adsorbed particles, the sensor can be switched off and stand by till the next measurement. To obtain reliable and reproducible measurements one should reset the sensor before each measurement. In other words, the adsorbed particles need to be removed from the surface of the beam. In order to develop a resetting procedure, we carried out tests on regular beams and paddle beams coated with a thin metal electrode on the frontside. No extra mask was needed, but the same gold metalization was used, which served as electronic interconnects and as cathode for the contactless electrochemical etching. For a real application, the resonator beam needs to be coated on both sides with metal electrodes.

The following test gave evidence that the adsorption of the airborne particles is driven by electrostatic charging, and not by high-energy collision of the particles with the beam surface. The electrode on the frontside of a regular cantilever beam was connected to ground potential, and the beam was driven at resonance in ambient air. After 67 hours, the gold electrode was disconnected. Figure 4-15 shows that the resonance frequency shift is linear in time in both cases, but the slope is steeper for the free beam than for the grounded one. At first glance one would expect the slope measured with the disconnected surface electrode to be twice as steep as the one measured with the grounded electrode. In fact the ground potential of the surface electrode affects the backside as well, through the metal-semiconductor interface. Plus the small air gap under the vibrating beam can make the adsorption on the backside less effective. Consequently, the free beam has a 4.5 times higher adsorption rate than the one with a grounded surface electrode. We can conclude that the ‘flycatcher’ effect occurs due to electrostatic charging, and that it can be eliminated by keeping the surface of the resonator at ground potential. The grounding can serve as an on/off switch for our sensor, but more importantly, it improves the stability of functionalized resonant probes. If the beam surface is grounded, then only the functionalized area will be active, adsorbing only the desired particles.
The experiment was continued with a resetting test. A high-frequency electrical signal was connected to the surface electrode while driving the beam at resonance in ambient air. The AC signal prevents electrostatic charging. The beam released the particles from the frontside of the resonating cantilever, and thus the resonance frequency increased, see figure 4-16. The resonance frequency settled at the value that the system would have reached if the surface electrode had been grounded along all the measurements. This indicates that the frontside released the adsorbed particles, while the backside remained unaffected. This test demonstrates that the electrostatic resonant pollution detector can be conveniently reset by means of a purely electronic signal. Both sides of the resonator should be grounded or connected to an AC signal between two measurements to refresh the surfaces and to obtain repeatable, reliable results. Note that the increasing region of the measured curve shows no scattering. Thus the scattering of the measurements in the first two regions are not artifacts due to the measurement technique, but real fluctuations of the mass of the adsorbed layer. The adsorption and desorption on the surface are dynamic.

Fig. 4-15 Cycling test of gold coated Si beam in air. The gold electrode is at ground potential, then free.
processes, and are in balance according to the surrounding media and driving conditions. Even if the response time of the sensor is in the order of hours, there can be numerous applications found (e.g. environment-monitoring stations, space applications), where the easy switching and resetting of the sensor is of great importance. A system based on the ‘flycatcher’ effect can operate for a very long time without having the measurements interrupted by resetting or replacing the sensing element.

Fig. 4-16 Cycling test on Si beam in air. The surface electrode is a.) connected to ground, b.) not connected, c.) connected to AC signal.
References

Chapter 4 Vibration sensors


References


Measurement results


The reliability of silicon nitride thin-film resonators was studied and presented in Chapter 3. The environment-induced reliability problems can be minimized by proper design and/or cheap atmospheric packaging. To demonstrate this, resonant mode pressure sensors and accelerometers were fabricated, where thin silicon nitride double-clamped beams were implemented as sensing elements. These sensors also serve as test structures for further environmental tests that are independent from the AFM. These tests are not included in the thesis yet.

5.1 Resonant microsensors

The resonant sensor is a device with an element vibrating at resonance, which changes its resonance frequency as a function of a physical or chemical measurand parameter. By means of a change in the stresses, mass or form of the resonator, the measurand quantity is translated to a resonance frequency shift. Resonant sensors offer the highest resolution, transduction sensitivity, performance, long-term stability and reliability available today. The stability of the sensor is not affected by the stability of the electronics, but determined by the mechanical properties of the
resonator material. It is well accepted that resonant sensors offer significant advantages for integrating the sensing and digitization in the same monolithic structure by providing a frequency output. This output is basically independent of analog levels and it can be readily connected to digital circuitry and converted to a digital sensor output [5.1]. As no generation of analog output and its conversion to digital form are required, the inaccuracies are minimized. The frequency output is a high-level signal and is less sensitive to spurious or parasitic influences than capacitive or piezoresistive devices. The quartz wrist watch is a perfect example to show that the cost of batch-fabricated resonant structures together with their associated oscillators and digital electronic circuits can be very low and their performance high. The first resonant transducers implemented different metals as resonant element. As the quality of the resonator is closely related to its mechanical properties, crystalline silicon and quartz became the dominant resonator materials. Resonators developed rapidly, their quality factor increased and various shapes became available, exploiting different vibration modes. Nowadays resonant micromechanical devices play an important role in numerous application fields. Gyro-sensors [5.2] [5.3], pressure sensors [5.4], accelerometers [5.5] [5.6], functionalized probes [5.7] and probes for scanning probe microscopy [5.8] [5.9] are just a few. The most recent devices implement VLSI-compatible surface-micromachined laterally resonating structures, actuated by comb drives [5.10]. The typical setup of a resonant sensor system is illustrated in figure 5-1 [5.11]. Owing to the compatibility of MEMS processing with IC technology, the excitation unit and the detection unit can be integrated on the mechanical resonator structure. Note that in some applications - typically SPMs and vibration characterization techniques implementing optical readout, this integration does not take place, resulting in a more bulky system, but providing more application-specific freedom. The digital output is processed, forming the driving signal for the resonator, and connected via the feedback loop to the excitation unit. Even higher-level integration is realized in smart micromechanical systems, where on-chip signal conditioning, self-calibration, self-testing or other electronic functions are integrated on the same die.
Resonant microsensors

Fig. 5-1  Resonant microsensor system with feedback.

<table>
<thead>
<tr>
<th>Excitation</th>
<th>Detection</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrostatic</td>
<td>Capacitive</td>
<td>Requires large electrodes with small distance. Effective in low air damping.</td>
</tr>
<tr>
<td>Dielectric</td>
<td>Capacitive (dielectric)</td>
<td>Diel. sandwich on beam generates bending. Capacitance change upon change of dielectric thickness is measured.</td>
</tr>
<tr>
<td>Piezoelectric</td>
<td>Piezoelectric</td>
<td>Good perform., but special deposition needed, not IC compatible, introduces stress, lower Q, temp. sensitivity.</td>
</tr>
<tr>
<td>Resistive heating</td>
<td>Piezoresistive</td>
<td>Heat pulse creates local expansion and beam deflection. Easy IC compatible processing, but strong temp. drift.</td>
</tr>
<tr>
<td>Optical heating</td>
<td>Optical</td>
<td>Laser heating. Interferometric or amplitude modulated detection, no interference with vibration, media or EM field.</td>
</tr>
<tr>
<td>Magnetic</td>
<td>Magnetic</td>
<td>Force = interaction of electric current in resonator &amp; M field. Voltage induced by vibration of conductor is detected.</td>
</tr>
</tbody>
</table>
The most frequently used excitation and detection combinations are listed in table 5-1. These principles are illustrated in figure 5-2 on the cantilever beam, where possible vibration types are longitudinal, flexural and torsional vibrations, and their combinations. Microbeam resonators are widely used in resonant MEMS devices, substituting piezoresistors, or supporting vibrating components.

This chapter focuses on developing low-cost resonant pressure sensors and accelerometers. Therefore, these two applications are discussed in more detail.
5.1.1 Resonant pressure sensors

Pressure sensors play a crucial importance in the measurement of the pressure, flow rate, level and density of fluids in the process industry. Pressure sensors have a wide range of applications in numerous fields such as the automotive industry, medicine, or process control in power plants. A more specific example is their use in the barometer in meteorological observation systems. The pressure sensor is based on a diaphragm, which is deformed due to the differential pressure on its two sides. The diaphragm strain is translated into an electrical signal. The conventional pressure sensors are capacitive or resistive. The first group is based on an electrostatic capacitance change measured from a deflected diaphragm, while the second group measures the resistance change of diffused resistors in a strained diaphragm. The ever increasing requirements in the past decades led to further improved accuracy and stability and to the development of resonant pressure sensors. Frequency output is obtained if the diaphragm is excited at its fundamental resonance frequency. The resonance frequency of a diaphragm is strongly dependent on the membrane stress [5.12], thus the pressure-induced strain can be measured as a resonance frequency shift. Sensitivity of at least a factor 100 higher was achieved with the resonant diaphragm method. Although having excellent resolution, repeatability and stability [5.13], conventional resonant pressure sensors implementing mechanically machined resonators were limited to specific application fields. The first digital pressure sensor in 1970 combined a resonant Cu-Be membrane with digital electronics [5.14]. The drawback of these structures is that the resonance frequency of the resonating structure is strongly affected by the surrounding fluid, and severe energy dissipation occurs during oscillation, especially in liquid [5.15]. Double mechanical structures were developed, implementing mechanical resonators apart from the membrane. A simple, resonant pressure sensor was realized with a double-clamped beam attached to a diaphragm on top of two pillars [5.4]. The resonator is driven by an intensity modulated laser beam, and the vibration is detected using an optical heterodyne interferometer. In today’s micromechanical resonant pressure sensors, the resonator is encapsulated in the vacuum cavity, so it is not affected by the surrounding media. The resonant characteristics of such structural- or geometric-type sensors are
determined by the material, geometric dimensions and initial strain. The four corners of a “H” shaped resonator have been fixed onto a diaphragm in a vacuum cavity [5.16]. The deflection of the membrane upon the applied differential pressure is translated into a resonance frequency shift of the resonator. This digital pressure sensor chip was applied in a differential pressure transmitter, digital manometer, barometer and liquid-level meter. A similar resonant pressure sensor was reported which implements a vacuum-encapsulated double clamped beam as resonator structure integrated in the silicon diaphragm [5.17]. The sensor provides high accuracy and a wide working range. Various combinations of optical, electrostatic and piezoelectric readout and driving techniques have been applied in wafer level testing. A high quality factor was observed, and the Young’s modulus and the strain of the beam were calculated.

5.1.2 Resonant accelerometers

Micromechanical accelerometers were first applied in the automotive industry as airbag triggers. Nowadays they are widely used in the automotive industry, inertial navigation systems, avionics, unmanned aerial vehicles, satellites etc. Resonant accelerometers translate the displacement of a seismic mass into stretching of the supporting beams. Pure bulk micromachining can be used to fabricate resonant accelerometers, where the seismic mass is supported by two beams [5.6]. A third, thinner beam is driven at resonance by electrothermal excitation, and the vibration is detected piezoresistively. The seismic mass is often suspended by double or triple beams to achieve balanced vibration modes and a high quality factor [5.18]. In advanced multi-wafer accelerometers, polysilicon resonators encapsulated in vacuum cavities detect the displacement of the proof mass suspended by biplane flexures [5.5]. The system had a high gauge factor on a wide acceleration range. Polysilicon surface-micromachined differential resonant accelerometers (DRXL) with torsional beam structures exploited the gap sensitive electrostatic stiffness changing effect [5.19]. State-of-the-art designs use surface micromachined lateral resonators with electrostatic comb drives [5.20] [5.21].
5.2 Sensor design

We designed low-cost pressure sensors and accelerometers, implementing three-dimensional thin film microbeam resonators as sensing elements attached to a locally reinforced diaphragm [5.22]. The term “three-dimensional” indicates that the double-clamped beams do not lie in the plane of the diaphragm. The double mechanical structure allows separate optimization of the diaphragm and the bridges for the workload and for the most efficient driving and sensing. The three-dimensional beams work as mechanical amplifiers, resulting in a higher detection efficiency. The reinforced diaphragm localizes the deflection, thus further increasing the detection efficiency. A silicon proof mass in the middle of the silicon nitride diaphragm is implemented in the accelerometers. External mechanical and thermal excitation combined with piezoresistive and optical detection methods are implemented in the different sensors. Differential detection using reference resonators allows compensation for thermal, environmental and aging-induced stresses.

Both the resonators and the membrane are fabricated of low-stress silicon nitride thin film. 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 can be fabricated of thin silicon nitride films. Fabricating the membrane and the sensing beams of the same material solves many of the typical problems related to thermally induced stresses. The long-term stability of the silicon nitride resonators and the effects of the environment on their mechanical and resonant properties are presented in chapter 3.3, chapter 3.4 and chapter 3.5. Surface oxidation and the adsorption-induced stiffening effect and degrading shock response were found for the thin silicon nitride cantilevers. Both effects depend on the thickness of the beam and on the surrounding media. Based on these results, we made certain trade-offs between simple design, low-cost fabrication process, stable operation and high performance. Consequently, we implemented thicker (1µm thick) resonators in the resonant sensors, which are less sensitive to surface-related degradation processes. Though some measurement sensitivity is lost when thicker beams are used, the signal-to-noise ratio can be substantially improved. A substantial cost
reduction can be achieved if the resonators are not encapsulated in a vacuum cavity, which would require multiple-wafer design. The environmental tests indicated that cheap, atmospheric packaging of the resonators is sufficient if no particularly high Q-factor is desired. Packaging filled with inert media such as nitrogen or argon provides stable resonant operation. The resonator geometry can be optimized in correspondence to the expected pressure or acceleration loading. Hence the relatively low quality factor does not degrade the sensitivity and resolution of the sensors.

5.2.1 Three-dimensional resonator bridge

In resonant pressure sensors and accelerometers the sensing double-clamped beam is often integrated in the membrane or in the suspending arm. The beam lies in the surface plane, released either by topside etching [5.5], or backside etching [5.6]. We took a different approach. The resonators are not integrated in the plane of the membrane, but form a three-dimensional bridge above it, see figure 5-3. The three-dimensional bridge can be considered as a cantilever beam attached to two pillars.

![Fig. 5-3 Schematic drawing of a 3-D bridge resonator with piezoresistors and metalization, on top of a membrane.](image)

The resonance frequency $f_0$ of the first transversal mode of a double-clamped beam is given by [5.23]
where \( h \) and \( L \) are the height and length of the beam, respectively, \( E \) is the Young’s modulus and \( \rho \) is the density. When the beam is subjected to axial stress \( \sigma \), the resonance frequency of the 1st vibrating mode is modified as [5.24]

\[
f = f_0 \sqrt{1 - \sigma / \sigma_0}
\]

(5-2)

where \( \sigma_0 \) is the buckling threshold [5.25], which can be expressed as

\[
\sigma_0 = \frac{\pi^2 E h^2}{3 L^2}
\]

(5-3)

Several effects can generate axial stress in the beam. Residual stress occurs from the processing. Our silicon nitride film has a tensile residual stress of 125MPa when deposited onto a silicon substrate. The different thermal expansion coefficients of the beam and the substrate can cause thermal stresses in the beam. A third, often significant component is the curvature shortening, which comes from the vibration itself [5.29]. When the beam deflects, the effective length contracts. The length of the double-clamped beam cannot change, hence tensile stress builds up in the beam upon deflection. The effect is determined by the amplitude of the deflection, and therefore also referred to as amplitude-induced stiffening effect. The deflection curve \( z(x) \) of the double-clamped beam under a uniformly distributed load \( q_0 \) can be approximated as

\[
z(x) = \frac{q_0}{24EI} (x^4 - 2Lx^3 + L^2x^2)
\]

(5-4)

where \( E \) is the Young’s modulus and \( I \) is the moment of inertia of the cross sectional area. The elongation \( \Delta \) due to the beam deflection is a function of the maximum deflection \( z_{\text{max}} \) in the middle of the beam \( (x=L/2) \), and can be approximated as [5.26]
The axial tensile stress resulting from the amplitude-induced stiffening $\sigma_\lambda$ is given as

$$\sigma_\lambda = \frac{E_\lambda}{L} = \frac{256 E z_{max}^2}{105 L^2}$$

The maximum deflection is $1 \mu m$, limited by the gap under the beam. The maximum value of the curvature shortening-induced tensile stress is calculated to be $14 MPa$. This is much smaller than the $125 MPa$ residual tensile stress, and can be neglected in most cases.

Fig. 5-4 3-D bridge sensing elements on top of diaphragm with backside reinforcement

Geometric considerations prove that this configuration behaves as a mechanical amplifier. The three-dimensional structure generates a considerably larger axial strain in the beam, and hence provides higher sensitivity than the inplane double-clamped configuration does. Let’s take the geometrical dimensions given in figure 5-4 as an example. A membrane deflection of $1 \mu m$ results in an inplane strain of $4 \cdot 10^{-5}$, while the rotation of the support of the bridge results in a bridge strain of $1 \cdot 10^{-4}$. 
The total strain induced in the bridge is the sum of the two components, which is $1.4 \times 10^{-4}$ in this example. A 3.5X mechanical amplification was achieved with the 3-D bridges.

Bridges with different size, shape and clamping configuration were designed. The bridges were typically 200-400$\mu$m long, 20-60$\mu$m wide, 1$\mu$m high and the thickness of the beams was 1-2$\mu$m. Many of the beams are formed such, that the middle section behaves as a rigid body, and the deformation is concentrated in the region where the sensing elements are positioned. This method detects the vibration more efficiently. The wider beams have holes in the middle, as shown in figure 5-8, which provide better exposure to the etching solution during sacrificial etching, and allow a better airflow during vibration, decreasing the air damping and squeeze-film effects. The perforating of the beam is extremely advantageous when the gap between the bridge and the substrate is in the order of a few micrometers, which is typically the case in structures with capacitive excitation. Perforation is useful even if the electrode surface is reduced, because it can reduce the air damping and the squeezed-film effect substantially. Several simulations and calculations can be found in the literature comparing the dynamic responses of vibrating plates with and without holes [5.27].

**Air damping and squeeze-film effects**

The air surrounding the vibrating beam applies a drag force and acts as extra mass, thus reducing its quality factor and slightly the resonance frequency. The squeeze-film flow results in a spring-like force acting on the beam due to the compressibility of the fluid in the gap, and thus increases the resonance frequency. The closely spaced vibrating plate can be modelled as a spring-mass-damper system, see figure 5-5 [5.28].

The extent to which air damping and the squeeze-film effect occur depends on the geometry of the resonator and its resonance frequency, and the surrounding media. For a vibrating plate with a narrow air gap underneath, the damping force and the squeeze-film spring force are plotted as a function of the vibration frequency in figure 5-6. Note that this is a generic curve, and the axis scales with the width/length ratio of the double-clamped beam.
Pressure sensors & Accelerometers

Fig. 5-5 Spring-mass-damper model of vibrating beam in ambient air above substrate.

Fig. 5-6 Damping force (air damping) and spring force (squeeze-film effect) characteristics as a function of the vibration frequency.
At low frequencies the system behaves as a normal damper with a constant damping coefficient acting on the vibrating beam, and the damping force is linearly proportional to the driving frequency. With increasing frequency, the damping force decreases, while the spring force increases. At high frequencies the spring force is independent of the frequency, indicating a normal spring with constant spring coefficient. The point where the two curves intersect is the cut-off frequency $\omega_c$ [5.29].

$$\omega_c = \frac{\pi^2 p_0 d^2}{12 \mu_{\text{eff}} \left( \frac{1}{w^2} + \frac{1}{L^2} \right)}$$  \hspace{1cm} (5-7)

where $p_0$ is the air pressure, $d$ is the air gap above and under the beam, $w$ is the width and $L$ is the length of the beam. $\mu_{\text{eff}}$ is used instead of the viscosity of air $\mu_\nu$ when slip-flow becomes significant. The Knudsen number $K_n$ is defined as the ratio of the mean free-molecular path $\lambda$ and the beam thickness $h$; $K_n = \lambda/h$. For $K_n > 0.1$ the flow cannot be treated as a continuum, but slip-flow becomes important. This is often the case with thin-film resonators with thicknesses in the submicron range, because for air at atmospheric pressure $\lambda = 63$nm.

$$\mu_{\text{eff}} = \frac{\mu_\nu}{1 + f(K_n)}$$  \hspace{1cm} (5-8)

$\mu_\nu = 1.8 \cdot 10^{-5}$ Pa/s in air at atmospheric pressure, and $f(K_n) = 9.638 \cdot K_n^{1.159}$. The squeeze number $\sigma$ is an important parameter for describing the air damping and squeeze-film effects.

$$\sigma = \frac{12 \mu_{\text{eff}} w^2}{p_0 d^2 \omega}$$  \hspace{1cm} (5-9)

At low squeeze numbers and frequencies, the damping force is linearly proportional to $\omega d^3$ [5.29]. At high squeeze numbers and frequencies the spring force converges to a frequency-independent value $F_{\text{max}}^s$, proportional to $1/d$. 
where $A$ is the plate area $A=L\cdot w$, and $|d_d|$ is the half vibration amplitude. The spring constant of the air film $k_g$ can be calculated, which is defined as $k_g=F_s/d_d$. We calculated the maximum value of the air spring constant of our double-clamped resonator system: $k_g=800$N/m. This is a considerably high value. In order to evaluate this result, we need to obtain the mechanical spring constant of the resonating beam.

For small amplitudes, the stress-free double-clamped beam resonator can be considered as a simple harmonic oscillator with $m=\rho whL$ effective mass, and the mechanical spring constant $k_b$ is given as [5.30]

$$k_b = \frac{41.7Ewh^3}{L^3}$$

where $E$ is the Young’s modulus and $w$, $h$ and $L$ are the width, thickness and length, respectively. The calculated mechanical stress-free spring constants of our resonators are in the order of 3 to 48 N/m. This indicates that at high frequencies, the spring constant of the resonator system is determined by the spring effect of the gas film. This makes the system stiffer and increases the resonance frequency. If axial stress is present in the system, the model of the stress-free beam is adjusted for the decreased or increased stiffness of the pre-stressed beam [5.29]. The spring constant of the pre-stressed double-clamped beam $k_{ps}$ is a combination of the stress-free spring constant $k_b$ and the effect of the stress $k_s$

$$k_{ps} = k_b + k_s$$

The $k_s$ term can be defined directly by a correction factor $c$, which is a function of the force $S$ resulting from the internal axial stress $\sigma, S=\sigma A$

$$k_s = k_bc(S)$$

By definition, $S>0$ for tensile stress, and $S<0$ for compressive stress. The $c(S)$ correction factor can be written in linearized form as
The effect of axial stress is very significant in long, thin beams, e.g. guitar strings. We estimated the maximum correction factor \( c(S)_{\text{max}} = 24 \) for our structures, indicating that the stiffening can be quite considerable. An estimated value of 125MPa tensile stress was applied in the calculations, which would be the case if the silicon nitride was deposited onto the silicon substrate. There is no measurement data available about the stress in silicon nitride deposited on PSG. Thermal effects do not substantially influence the free-standing silicon nitride bridges, as they are anchored onto a silicon nitride layer. In view of the maximum values, the stiffest pre-stressed structure has a spring constant of \( k_{ps} = 1152 \text{N/m} \), which is of the same order as the maximum air spring constant \( k_g = 800 \text{N/m} \). This indicates that the squeeze-film effect needs to be taken into consideration when one describes the behaviour of high-frequency resonator bridges.

\[ c(S) = \left( \frac{L}{h} \right)^2 \sigma E \]  
(5-14)

5.2.2 Reinforced membrane

Large aspect ratio structures can be realized with thin silicon nitride films. Silicon nitride membranes are often used as mechanical components for mirrors in adaptive optics and optical switches, pressure sensors or as mechanical suspension in accelerometers. The silicon nitride is usually in tensile stress when deposited on silicon, but the residual stress can be tailored with proper deposition conditions. When corrugations are introduced in the membrane, the tensile stress, which governs the behaviour, is reduced by a factor of \( 10^2 - 10^5 \), and the sensitivity is increased up to 100 times [5.31]. Symmetrical corrugations made by wet etching gave the best performance. In order to further increase the detection efficiency of our sensors, we reinforced the middle of the membrane. The membrane reinforcement is realized either on the backside by a remaining part of the silicon substrate - as shown in figure 5-4, or on the frontside with polysilicon, protected PSG, metalization, or anything else implemented in the fabrication process, see figure 5-9. The middle section of the membrane can be considered as a rigid plate with a large area, supported by a small, flexible ring. When differential pressure
is applied on the membrane, the reinforcement concentrates the membrane deflection to the narrow flexible ring. Depending on the sensor configuration, the edges of the sensing bridges are attached onto the flexible ring and onto the rigid part, which can be either the substrate or the reinforced middle section of the membrane. The reinforced membrane with localized deflection applies a higher rotation to the pillar of the resonator bridge than regular membranes.

5.2.3 Excitation and detection

The most frequently used excitation and detection combinations in beam-based resonant sensors are described in table 5-1. For comparison, we implemented several excitation and detection methods in the recent design. Some of the chips are glued onto multilayer piezoactuators, which apply external mechanical excitation to the resonators. The vibration is detected either optically or by the integrated piezoresistors. A modified atomic force microscope (AFM) is used for the optical detection, which is capable of detecting deflections in the nanometer range. The differential mode piezoresistive readout technique is applied using reference resonators on the substrate. On-chip excitation and detection are implemented as well, using multiple resistor loops at both edges of the beams. Vibration detection and resistive heating excitation using current pulses are realized with separate resistor loops. Furthermore, some of the bridges have integrated piezoresistors all along the length from edge to edge, in order to test a new resonant heating method. The same resistor loop is used for heating and detection, where the mechanical resonance of the bridge has positive feedback on the measured current, increasing the measurement efficiency.

5.3 Sensor fabrication

A fairly simple fabrication method combining bulk and surface micromachining was developed to obtain low-cost resonant devices. All the structures were fabricated at Delft Institute for Microelectronics and
Sensor fabrication

Submicron Technology (DIMES). The three-dimensional bridge resonators and the membranes were fabricated from low-stress, silicon-rich, LPCVD silicon nitride thin films deposited under the same processing conditions as the cantilever beams discussed in chapter 3.1.2. The general process flow of the pressure- and g-sensors is shown in figure 5-7. The fabrication process requires only 6 masks. The 3-D resonator bridges are deposited onto thick PSG. Geometric constraints are applied to the bridges by the maximum thickness of the PSG layer, which was around 1µm. A higher bridge results in higher mechanical amplification and thus higher sensitivity. Polysilicon piezoresistor loops are deposited for vibration detection, local resistive heating and novel resonant heating. Gold metalization is deposited on top of a thin chromium layer for better adhesion, and patterned with a lift-off process. The metalization forms interconnects on the chip, and serves as a reflective mirror for optical readout. Most of the diaphragms are reinforced in order to concentrate the deflection in the area where the sensing elements are positioned, see chapter 5.2.2. Protected PSG, polysilicon, and gold layers were used for the topside reinforcement, while on other chips a chunk of the silicon substrate is left under the diaphragm as back reinforcement, also forming the proof mass of the accelerometers. Another advantage of this design is the convenient etching, which does not involve any electrochemical or other etch-stop techniques. The diaphragm is formed with anisotropic back etching in KOH, where the etching stops automatically when the etch profile reaches the silicon nitride. After the wafer is diced, the bridge resonators are released with sacrificial etching of the PSG. Timing is not critical in the etching processes, because the silicon nitride acts as etching mask in both cases. Sticking is a serious problem in surface micromachining, when wet etching is applied to structures with a very thin gap between the etched surfaces. When the etching solution is drying, the capillary forces pull the surfaces towards each other, which can lead to sticking. Several rinsing and drying methods can be applied to avoid sticking [5.32]. However, we implemented a second PSG mask, and formed small bumps on the sides of the bridges in the middle section, which reduces the contact area while the etchant is drying.
Low-stress SiN deposited on Si wafer.

3-D SiN bridges deposited on SiN membrane using PSG sacrificial interlayer.

Poly-Si piezoresistors and Au/Cr metalization deposited.

Forming membrane and releasing resonator bridges with back and sacrificial etching.

Fig. 5-7 Process flow
5.4 Results

Two of the test chips with different topside reinforcements are shown in figure 5-8 before etching, thus the membrane is not distinguishable. Figure 5-9 shows bridges on top of an etched, topside reinforced membrane. Reference bridges with the same geometry are positioned on the substrate for differential readout. By comparing the resonance frequencies of the sensing and reference bridges, one can accurately measure the deflection of the membrane underneath. The differential setup compensates for thermal, environment- and aging-induced stresses, as both resonators are subjected to the same environment. The sensing element is loaded purely by the measurand pressure or acceleration. The holes in the wider beams serve as etching holes for the sacrificial etching, and reduce the air damping and squeezed-film effects [5.33] during the high-frequency vibration.

*Fig. 5-8* Top view of pressure sensor test chips with different 3D bridges, before etching.
5.5 Measurements

Due to the time frame available the measurement results of the pressure sensors and accelerometers are not yet included in this thesis. The bridges are characterized using the optical detection method of the atomic force microscope combined with external piezoelectric driving similar to that of the silicon beams and paddle beams see chapter 4.3.2. Using the piezoresistive readout the pressure sensors can be characterized in a pressure chamber independent from the AFM. Owing to the rigid middle section of the accelerometers, they can be characterized applying loads by an indenter or alpha-stepper probe while the resonator bridges are driven and detected with the piezoresistive method.
References

Chapter 5  Pressure sensors & Accelerometers

5.9  A. Boisen, O. Hansen, S. Bouwstra, J. Micromech. Microeng., 6, p. 58, 1996
5.14  R. Frische, “Vibratory pressure sensors”, Scientific Honeywell, pp. 79-84, Fall 1987
References

Chapter 6

Conclusions

Silicon, silicon nitride and silicon carbide thin-film cantilever beams were characterized with special emphasis on their mechanical and resonant properties. The long-term stability of the resonators was studied in various environments. The following conclusions can be drawn:

• Micromechanical components are typically on the nanometer to millimetre size scale. The material properties of the microstructures are strongly dependent on the fabrication conditions and can be substantially different from those of bulk materials. Therefore fairly well characterised materials must be investigated further by means of micromachined test structures. The fabrication process, size and shape of the test structures should approach those of the actual MEMS device under examination. The residual stress of the silicon nitride and silicon carbide thin films was tailored by using the proper deposition parameters. The Young’s modulus was calculated from the resonance frequencies of the cantilever beams. The Young’s modulus of silicon nitride \( E = 230 \text{GPa} \) is in good agreement with the data available in the literature [6.1], [6.2]. The value calculated for SiC \( E = 320 \text{GPa} \) is twice as high as the one reported in the literature [6.2], which can be explained by the different deposition conditions.

• A studying of the failure mechanisms of micromechanical structures is of great importance if one wants to improve the reliability and the commercialization of advanced MEMS devices. Owing to the small size and extreme aspect ratio of micromechanical structures, surface effects
Conclusions

and crack-related mechanisms play a more significant role. The surface stress has a measurable influence on the mechanical properties of cantilever beams with a high surface-to-volume ratio.

- Adsorbed molecules change the surface stress and hence the stiffness and the resonance frequency of the submicron-thick beams. This effect is exploited in functionalized probes where the static deflection due to the asymmetric surface stresses or the resonance frequency shift of the cantilever beam is measured. We tested the submicron-thick cantilever beams in various environments. Both the silicon nitride and the silicon carbide cantilever surfaces were oxidized in ambient air. The native oxide layer and the surface adsorbates changed the surface stress on both sides of the cantilever beam, which influenced the restoring forces upon bending. A more detailed interpretation of this effect requires further experimental and theoretical work. A stiffening effect was observed: the resonance frequency increased logarithmically in time. Water vapour is an oxidising agent that is more reactive than oxygen gas. Increased humidity led to a stronger stiffening effect and to a higher resonance frequency. Non-oxidizing media such as nitrogen and argon improved the stability of the beams, diminishing the stiffening effect. Surface adsorption and desorption is a dynamic effect. Consequently the resonance frequency of beams previously exposed to air decreased in argon- and nitrogen-rich environments. The gradual shift in the resonance frequency corresponding to the operating environment leads to false measurement results and drift in case of the resonant mode MEMS devices.

- Another important failure mechanism of the submicron-thick resonators is the shock response. Mechanical shocks and large deflections generate nano- and micro-cracks in the surface oxide and shake off the surface adsorbates. The stiffening effect is diminished, resulting in an abrupt resonance frequency drop. Following the mechanical disturbance the surface layer recovers in air and the resonance frequency increases again. The magnitude of the resonance frequency drop and the rate of the recovery depend on the surrounding media. Inert atmosphere prevent (re)oxidation of the surface, resulting in stable resonant operation (following an initial ‘burn in’ period). The same mechanical shocks or large deflections generate higher resonance frequency deviations in a humid environment. These environment-dependent abrupt changes in the
resonance frequency upon shocks and large deflections present a failure mechanism of resonant micromechanical devices.

• Concerning the stiffening effect and the shock response, the various environments can be ranked according to their adsorption and oxidising capability as follows:

1) inert environment (argon, nitrogen, vacuum) $\Rightarrow$ stable resonator
2) ambient air $\Rightarrow$ slight stiffening effect, resonance frequency drop and recovery upon mechanical shocks and large deflections
3) humid environment $\Rightarrow$ strong stiffening effect and shock response

• The environmental tests showed that inert atmospheric packaging can present a low-cost solution for devices for which low quality factors ($\approx 50$) are sufficient. To demonstrate the versatility and wide application range of microbeam resonators, we fabricated resonant-mode pressure sensors and accelerometers. The piezoresistive sensing elements of the conventional sensor structures were replaced by double-clamped silicon nitride microbeam resonators. The beams are 1 micrometer thick, so they are less sensitive to environmental effects, but still compliant and easy to drive and read out. Packaging in nitrogen or argon can provide sufficiently stable resonators, but this should still be verified experimentally.

• Electrostatic charging-induced adsorption of airborne particles was observed on the surface of single-crystal silicon beams vibrating in ambient air. The mass loading applied by the adsorbates resulted in a long-term failure mode of the microbeam-based resonant sensors. Proper grounding of the resonator inhibits this so called ‘flycatcher’ effect. If no metalization on the resonator surface is feasible, the electrostatic charging-induced adsorption effect determines the minimum requirements for the packaging of the device. The ‘flycatcher’ effect was also exploited for sensing purposes. A novel resonant mode air pollution detector was demonstrated where the sensor can be switched on/off and reset conveniently with a purely electrical signal. This is a major advantage compared to conventional chemical and biological sensors, where the sensing element needs to be replaced or heat-cycled in order to refresh it after saturation. The new sensor is desirable for, for instance, space applications and environment-monitoring stations.
References

Chapter 6


Summary

Thin-film resonators form the fundamental mechanical component of many micromechanical devices. This thesis addresses their relevant reliability problems.

The nanometer to millimeter size scale of MEMS results in material properties, reliability issues and failure mechanisms different from those of bulk materials. Therefore the mechanical material properties and the reliability of the thin-film resonators have been studied using dedicated micromechanical test structures. Submicron-thick silicon nitride and silicon carbide resonators were tested using an atomic force microscope. The Young’s modulus and quality factor of the cantilever beams were obtained from the resonant measurements. The spring constants of the beams were determined by quasi-static bending tests.

The long-term stability of the cantilever beam resonators was studied with resonant cycling tests in various environments. Due to the high surface-to-volume ratio, surface stress effects contributed to the resonance frequency of the thin-film resonators. Adsorption of various components of the surrounding media and surface oxidation modified the surface stress, generating a measurable resonance frequency shift. The resonance frequency of the silicon nitride and silicon carbide cantilever beams increased logarithmically with time in ambient air, causing an environment-induced failure mode. The changes in the spring constant and surface stress were calculated from the resonance frequency shift. Oxynitride formation on the surface increased the stability of the silicon nitride beams. This indicates that the deposition of a thin oxide layer onto the silicon nitride beams can inhibit the surface-oxidation-induced stiffening effect. No substantial oxide formation was observed on silicon carbide surfaces in ambient air, though adsorption slightly increased the resonance frequency of silicon carbide beams. The silicon carbide surface is practically passivated by the first layer of oxide.

The shock response of the microbeams was studied in various environments. The resonance frequency dropped abruptly due to nano-
and micro-crack formation in the surface oxide layer and “shake-off” of the surface adsorbates. Following the mechanical shocks or large deflections the resonance frequency recovered logarithmically in air. These fluctuations in the resonance frequency can lead to false measurement results or eventually failure of the driving function of the resonant sensor. The initial resonance frequency drop and the recovery rate depend on the environment. Humidity enhances and argon- and nitrogen-rich environments diminish the effects, offering a cheap atmospheric packaging solution for micromechanical resonators. The silicon carbide resonators proved to be less sensitive to the environmental effects than the silicon nitride resonators.

The surface adsorbates change not only the surface stress, but the mass of the micromechanical resonators as well. The mass loading effect was observed on single-crystal silicon cantilever beams. Electrostatic charging-induced adsorption (also referred to as the ‘flycatcher’ effect) resulted in a gradual decrease of the resonance frequency in ambient air. This drift can be avoided by proper grounding or packaging of the vibrating beams. The ‘flycatcher’ effect can be exploited to construct air-pollution and other airborne-particle detectors. The major advantage of such systems compared to conventional ones is that they can be switched on/off and reset conveniently with a purely electrical signal. This is preferred for instance in environment-monitoring stations or space applications. The sensing, switching and resetting principles were demonstrated for silicon cantilever beams with gold surface electrodes.

The environment-induced reliability problems of submicron-thin resonators were well characterized and can be circumvented by proper design and/or low-cost, inert atmospheric packaging. To demonstrate this, we designed and fabricated resonant-mode pressure sensors and accelerometers. The sensors consist of FEA-optimized double mechanical structures. Double-clamped microbeam resonators sense the deflection of a reinforced membrane. External piezoelectric and resistive heating excitation methods were implemented in combination with piezoresistive and optical detection.

We experimentally characterized, and explained many of the reliability issues of thin-film resonators. A surface adsorption/oxidation model was proposed to describe the failure mechanisms. Further experiments and theoretical work are required to refine and extend the model for future nano devices.
Samenvatting

Dunne-film resonators worden tegenwoordig door veel onderzoekers bestudeerd, omdat ze de belangrijkste mechanische onderdelen vormen van veel micromechanische chips. Dit proefschrift beschrijft de relevante betrouwbaarheidsproblemen van deze resonators.

Door de zeer kleine afmetingen hebben deze micromechanische structuren andere materiaaleigenschappen, betrouwbaarheidsaspecten en foutmechanismes dan het overeenkomstige bulkmateriaal. Daarom zijn de materiaaleigenschappen en de betrouwbaarheid bestudeerd met behulp van micromechanische teststructuren.

Siliciumnitride en siliciumcarbide resonators met een dikte van minder dan een micrometer zijn getest met behulp van een atoomkracht-microscoop. De Young's modulus en de kwaliteitsfactor van de balken zijn verkregen d.m.v. resonantiemetingen. De veerconstanten van de balken zijn bepaald door middel van quasi-statische buigtests.

De lange-termijnstabiliteit van de resonators is bestudeerd met cyclische resonantietests in verschillende media. Door de hoge oppervlakte/volume-verhouding van de resonators werd de resonantiefrequentie in hoge mate bepaald door mechanische spanning effects van het oppervlak. Adsorptie van verschillende componenten uit de omringende media en oppervlakte-oxidatie beïnvloedden de oppervlaktespanning en genereerden zo een meetbare verschuiving in de resonantiefrequentie. De resonantiefrequentie van de siliciumnitride en siliciumcarbide balken nam in lucht logarithmisch toe met de tijd. De veranderingen in de veerconstante en de oppervlaktespanning werden afgeleid uit de verschuiving van de resonantiefrequentie. Oxi-nitridevorming aan het oppervlak vergrootte de stabiliteit van de siliciumnitride balken. Dit wijst ertop dat depositie van een dunne oxidelaag op de siliciumnitride balken de oxidatie-geïnduceerde verstijving tegengaat. Er werd geen oxidevorming waargenomen op siliciumcarbide oppervlakken in lucht, alhoewel de adsorptie de resonantiefrequentie van de carbide
balken enigszins toenam. Het siliciumcarbide-oppervlak werd praktisch gepassiveerd door de eerste laag van natuurlijkoxide.

De schokresponsie van de microbalken is bestudeerd in verschillende media. De resonantiefrequentie viel abrupt terug door nano- en micro-scheuren in de oxide-oppervlakslag en het afschudden van geadsorbeerde deeltjes op het oppervlak. Ook bij deze tests herstelde de resonantiefrequentie in lucht zich logaritmisch. Deze fluctuaties in de resonantiefrequentie kunnen er toeleiden dat de sensor verkeerde resultaten meet en uiteindelijk tot het falen van de excitatie van de resonators. De initiële terugval van de frequentie en de herstelsnelheid waren afhankelijk van de omgeving. Vocht vergrootte dit effect terwijl argon en stikstof het effect juist verkleinde. Dit levert de mogelijkheid op micromechanische resonators goedkoop te verpakken onder atmosferische druk. De siliciumcarbide resonators waren minder gevoelig voor omgevingsfactoren dan de siliciumnitride resonators.

De geadsorbeerde deeltjes veranderden niet alleen de oppervlakte-spanning, maar ook de massa van de micromechanische resonators. Dit effect is waargenomen bij balken van mono-kristallijn silicium. Elektrostatische ladingsgeïnduceerde adsorptie (ook wel het ‘vliegenvanger effect’ genoemd) resulteerde in een geleidelijke afname van de resonantiefrequentie in lucht. Dit drift-probleem kan voorkomen worden door de vibrerende elementen goed te aarden of te verpakken. Het vliegenvanger-effect kan benut worden voor systemen die luchtverontreiniging of andere door de lucht verspreide deeltjes detecteren. Het grote voordeel dat zulke systemen hebben ten opzichte van conventionele systemen is dat ze gemakkelijk aan- en uitgezet en gereset kunnen worden met een elektrisch signaal, waardoor ze makkelijker zijn te gebruiken in bijvoorbeeld milieubewakingsstations of ruimte-toepassingen. De meet-, schakel- en resetprincipes zijn gedemonstreerd aan de hand van silicium balken met gouden elektroden.

De omgevingsgerelateerde betrouwbaarheidsproblemen van submicron-dunne resonators zijn gekarakteriseerd en kunnen worden voorkomen door speciale ontwerpen en/ of goedkope inerte atmosferische verpakkingstechnieken. Dit is gedemonstreerd door het ontwerp en de fabricage van resonantiemodus druk- en versnellingsensoren. Deze sensoren bestaan uit geoptimaliseerde dubbele mechanische structuren. Balken opgehangen aan twee punten op een membraan detecteerden de uitwijking van dit membraan. Voor de excitatie werden piezo-elektrische
en resistieve (verwarmings)technieken gebruikt. Voor de detectie werden piezoresistieve en optische technieken gebruikt.

De belangrijkste betrouwbaarheidsaspecten van dunne-film resonators zijn experimenteel gekarakteriseerd en verklaard. De foutmechanismen werden beschreven door middel van een oppervlakte-adsorptie/oxidatie-model. Dit proefschrift laat nog ruimte voor verder onderzoek om het voorgestelde model te verfijnen en uit te breiden met name voor toekomstige nano-structuren.
Samenvatting
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List of publications


List of publications


164 RELIABILITY OF MICROMECHANICAL RESONATORS
Biography

Róbert Kazinczi was born in Tatabánya, Hungary, in 1973. After finishing the Bólyai János Secondary School in Salgótarján, Hungary, he joined the Technical University of Budapest, Hungary in 1992. His specialisation was material science, and he worked in the field of scanning tunnelling microscopy (STM). He developed a fast and reliable EC STM tip preparation method. He received his M.Sc. degree in applied physics at the TU Budapest in 1997. In 1996 and 1997 he worked part-time at the Central Research Institute for Chemistry of the Hungarian Academy of Science on scanning electron microscopy and X-ray microanalysator systems. From 1997 he is a Ph.D. student at the Electronic Instrumentation Laboratory at Delft University of Technology, the Netherlands. His research project focuses on the reliability of micromechanical structures, and is founded by the Dutch Technology Foundation (STW). He is engaged in mechanical studies of thin-film resonators for micro-electromechanical systems (MEMS). After his graduation in February 2002, he will start as a MEMS Design/Process Engineer at Cavendish Kinetics, Den Bosch.
Biography