Vacuum-Sealed and Gas-Filled
Micromachined Devices

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The front cover shows two wafer stacks (10 cm in diameter), one having low-pressure encapsulated resonant density sensors, the other one having CO₂-filled chambers to be integrated in CO₂-gas sensors for medical applications. The other photos show a density sensor chip and its SEM cross-section, a SEM cross-section of a CO₂-filter chip, and glowing microfilaments placed in a ceramic holder above a CO₂-filter.

(Photos: Densitometer wafer stack, Boguslaw Rawinski; Densitometer cross-section, Peter Enoksson; CO₂-filter cross-section, Edvard Kälvesten; others, Tomas Asplund; Photomontage: the Author).

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Abstract

In the field of micromachining, microsensor packaging is one of the least investigated, although one of the most important and challenging, technology areas. In particular, hermetic packaging is a key aspect of many microelectromechanical systems (MEMS). By hermetically sealing Microsystems and protecting them from harmful environmental influences, their reliability and lifetime can be significantly increased. In addition, some MEMS need a specific gas or pressure environment within the package to function properly. In this thesis, techniques for forming low-pressure encapsulated and gas-filled micromachined devices using silicon fusion and anodic bonding are investigated. Particular attention is given to techniques that provide hermetically sealed electrical feedthrough conductors. The low-pressure encapsulation of electrically operated silicon resonators and the sealing of cavities with carbon dioxide (CO$_2$) are presented, both performed at the wafer level.

The silicon resonators were encapsulated in a low-pressure cavity between two glass lids with metal electrodes for electrostatic excitation and capacitive detection. The final cavity pressure obtained was 1 mbar. The effect of squeeze-film damping due to oscillation close to the lid walls was investigated and a theoretical model for damping was presented. The development of a novel electronic circuitry based on discontinuous, “burst” excitation eliminated the need for feedthrough conductors by placing the electrodes outside the low-pressure cavity and suppressed the crosstalk between excitation and detection. Using this technique, a fully low-pressure resonant fluid density sensor was fabricated. A large cavity recess of 100 µm could be formed in the glass to reduce squeeze-film damping. The feedback control together with the detection electronics enabled on-line density measurements. The encapsulated sensor showed high performance with density sensitivities of $-200$ ppm (kg m$^{-3}$)$^{-1}$, a high quality factor of 3400, low temperature sensitivities of $-29$ ppm °C$^{-1}$ in the range 20-100 °C and proven long-term stability.

The CO$_2$ chambers were filled with CO$_2$ during an anodic bonding procedure performed at overpressure up to 2 bar in a CO$_2$ atmosphere. The CO$_2$ chambers are used as optical gas filters. To increase the transmission, the silicon wafer was coated with silicon dioxide as an antireflective coating and the glass wafer was thinned down to 125 µm. The fusion and anodic bonding techniques led to very good air-tight gas cavity chambers with an internal pressure of 1 bar. These optical gas filters were integrated in an infrared gas analysis system to provide a reference signal for measuring the CO$_2$ concentration in patient airways during anesthesia or intensive care.

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To Ela and my family
They are generally small.
One can find them in large quantities.
Their individual lifetime can be very short but the species is almost immortal.
They are everywhere and can be found in many different forms.
Without them, the technical world would not be viable.

Who are they? You might find the answer in this thesis...
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1. **Gas damping of electrostatically excited resonators**
   Thierry Corman, Peter Enoksson and Göran Stemme

2. **Low-pressure-encapsulated resonant structures with integrated electrodes for electrostatic excitation and capacitive detection**
   Thierry Corman, Peter Enoksson and Göran Stemme

3. **Deep wet etching of borosilicate glass using an anodically bonded silicon substrate as mask**
   Thierry Corman, Peter Enoksson and Göran Stemme

4. **Dynamic simulation model for a vibrating fluid density sensor**
   Timo Veijola, Thierry Corman, Peter Enoksson and Göran Stemme
   Sensors and Actuators A (1999), accepted for publication.

5. **New CO₂-filters fabricated by anodic bonding at overpressure in CO₂ atmosphere**
   Thierry Corman, Edvard Kälvesten, Matti Huiku, Kurt Weckström, Pekka Meriläinen and Göran Stemme

6. **An optical IR-source and CO₂-chamber system for CO₂-measurements**
   Thierry Corman, Edvard Kälvesten, Matti Huiku, Kurt Weckström, Pekka Meriläinen and Göran Stemme
   Submitted for Journal publication.

7. **Novel “burst” technology for closed-loop detection and excitation of resonant silicon sensors**
   Thierry Corman, Kjell Norén, Peter Enoksson, Jessica Melin and Göran Stemme
   Submitted for Journal publication.

8. **A low-pressure encapsulated resonant fluid density sensor with feedback control electronics**
   Thierry Corman, Peter Enoksson, Kjell Norén and Göran Stemme
   Submitted for Journal publication.

The contributions of Thierry Corman to the different publications are:

1-3: All fabrication and experiments. Major part of writing.
4: All fabrication and experiments. Part of writing. The simulations were performed by Timo Veijola.
5: All fabrication and experiments. Major part of writing.
6: All filters fabrication and simulations. Part of experiments. Major part of writing.
7: All density sensor fabrication. Major part of experiments. Major part of writing. The electronics was built by Kjell Norén.
8: All fabrication and experiments. Major part of writing.
The work has also been presented at the following conferences:

1 **Gas damping of electrostatically excited resonators**  
   Thierry Corman, Peter Enoksson and Göran Stemme  

2 **Low pressure encapsulated resonant structures excited electrostatically**  
   Thierry Corman, Peter Enoksson and Göran Stemme  

3 **Deep wet etching of borosilicate glass using an anodically bonded silicon substrate as mask**  
   Thierry Corman, Peter Enoksson and Göran Stemme  

4 **Encapsulation of silicon resonant structures**  
   Thierry Corman, Peter Enoksson and Göran Stemme  

5 **Dynamic simulation model for a vibrating fluid density sensor**  
   Timo Veijola and Thierry Corman  

6 **A silicon IR-source and CO₂-chamber for CO₂ measurements**  
   Edvard Kälvesten, Thierry Corman, Matti Huiku, Kurt Weckström, Pekka Meriläinen and Göran Stemme  

7 **An optical IR-source and CO₂-chamber system for CO₂-measurements**  
   Thierry Corman, Edvard Kälvesten, Matti Huiku, Kurt Weckström, Pekka Meriläinen and Göran Stemme  

8 **Novel burst technology for closed-loop detection and excitation of resonant silicon sensors**  
   Thierry Corman, Peter Enoksson, Kjell Norén and Göran Stemme  

9 **A low-pressure encapsulated DRIE resonant pressure sensor electrically excited and detected using ‘burst’ technology**  
   Jessica Melin, Peter Enoksson, Thierry Corman and Göran Stemme  
1 Introduction

Originating from the integrated circuit (IC) industry, the field of micromachined sensors and actuators, often referred to as “MEMS” (microelectromechanical systems), has been growing rapidly and has attracted huge interest in recent years [1]. Micromachining and microfabrication advances have allowed the miniaturization of many types of sensors and actuators. Among the benefits of microsystem technology are its contributions to cost reduction due to batch fabrication possibilities, reliability and improved performance [1]. However, for many MEMS devices, the critical issue of packaging has often been neglected and has proven to be one of the biggest limitations for commercialization [2]. More than 70% of the sensor cost can be attributed to its packaging, and the performance of a micromachined sensor is often influenced by its package [3]. Therefore, microsensor packaging remains one of the primary challenges for the MEMS community.

A package must serve a wide range of purposes. It should protect the microsystem from its operating environment, while, somewhat in contradiction, enabling interaction with that environment in order to measure or affect the desired physical or chemical parameters [3]. Other requirements include reliable interconnections between the outside world and the sensing element, mechanical support, low fabrication cost, high device performance and long-term stability. The package must also provide an interior environment compatible with the device performance and reliability; for example, a high-\(Q\) resonator might need a good vacuum. In that sense, there is a great need for developing low-cost and reliable techniques to form hermetically sealed low-pressure and gas-filled cavities. Wafer level processes are particularly interesting since they can reduce the fabrication costs and open up possibilities to batch fabrication. Various wafer level methods for obtaining hermetically sealed cavities may be used, including wafer bonding, cavity sealing using thin-film deposition, and reactive sealing.

The present thesis discusses how to realize low-pressure encapsulated and gas-filled micromachined devices at the wafer level using silicon fusion and anodic bonding techniques. These techniques were chosen because of their well-known reliability, good bonding quality (long-term stability), and widespread use (equipment commercially available) [4, 5].

The thesis begins with an overview of wafer level hermetic sealing technology, followed by two specific sections focused on vacuum sealing and gas sealing technologies. A section is also attributed to hermetic sealing of electrical feedthrough conductors. Based on the described technologies, the encapsulation of two microsensor devices is realized. The first one is a silicon resonant fluid density sensor encapsulated in
a low-pressure cavity using fusion and anodic bonding techniques. The motivation for choosing a resonant device is that resonator encapsulation requirements are particularly demanding. Special attention must be paid to maintain a high quality factor. This is generally achieved by housing the resonator inside a low-pressure cavity to reduce gas-film damping losses due to the oscillation close to the package walls where electrodes are often placed for excitation and detection. In addition, the presented resonator must be in direct contact with the fluid to be sensed, and therefore its encapsulation becomes further complicated, as for many chemical or biological sensors. Lateral electrical feedthroughs conductors are also demonstrated for resonant test structures. The second device described is a CO\textsubscript{2}-optical gas filter consisting of a chamber filled with CO\textsubscript{2} during an anodic bonding performed at overpressure in a CO\textsubscript{2} atmosphere. The filters’s optical characteristics are extensively investigated. The eight papers at the end of the thesis give full details of the work.

2 Objective

The objective of the work is to investigate how to realize low-pressure encapsulated bulk silicon micromachined resonators and gas-filled micromachined devices using fusion and anodic bonding techniques. The encapsulation must be realized at wafer level and is limited to sealed micromachined devices. Particular attention is paid to techniques that provide hermetically sealed encapsulated devices with electrical feedthrough conductors.

The chosen resonant sensor to be encapsulated in a low-pressure cavity is an earlier presented resonant silicon density sensor fabricated by silicon bulk micromachining and fusion bonding [6]. The ultimate goal is to obtain a fully low-pressure encapsulated device with integrated electrodes for electrostatic excitation and capacitive detection associated with an external feedback control circuit. The encapsulation process should preferably not degrade the performance of the initial device. Once encapsulated, the device should have a high quality factor with minimized damping and exhibit long-term stability.

To realize a gas-filled device, an optical gas filter consisting of micromachined chambers filled with CO\textsubscript{2} is fabricated. The encapsulation should be performed using fusion and anodic bonding techniques at a wafer level and at overpressure to encapsulate an optimum amount of gas for optimum absorption characteristics. The final goal is to integrate the optical filter in an infrared gas analysis system measuring the CO\textsubscript{2}-concentration in patient airways in anesthesia and ventilator equipment.
3 Hermetic sealing technology

3.1 Hermeticity

A hermetic package is defined as an enclosure with an internal cavity demonstrating acceptable gas-tightness. The stability of the enclosed gas (i.e. pressure or composition) is crucial to obtain reliable devices with long-term stability. The hermeticity of a package can be affected by different parameters such as stress, bond or seal quality and moisture permeation. Leakage rate detection methods can be used to control hermeticity.

When two different materials are combined, for example during a bonding process, a problem due to a mismatch of their thermal expansion coefficients may arise. This may lead to thermal stress as the temperature changes and break the bonded wafers. Using materials having sufficiently close thermal expansion coefficient can reduce thermal stress. Examples are silicon and silicon carbide, silicon and glasses specifically developed for this purpose (e.g. Hoya SD-2) and GaAs and sapphire. Lowering the bonding temperature, if possible, is a guaranteed method of reducing stress. Micromachined stress relief structures, such as V or U grooves, may also reduce the package stress [7, 8].

Several techniques exist to determine the quality of a seal. These include visual inspection (e.g. for anodically bonded wafers), imaging, cross-sectional analysis and bond strength measurements. The three dominant methods for imaging are infrared transmission [9, 10], ultrasonic transmission [11] and X-ray topography, all of which are nondestructive. The most common bond strength measurement techniques are illustrated in Fig. 3.1. The popular crack opening method (Fig. 3.1. (c)) consists of introducing a blade of defined thickness between the bonded wafers [12]. During an IR inspection, the length L of the crack gives a measure of the surface energy keeping the wafers together [13].

![Fig. 3.1. Illustration of three bond strength measurement methods.](image-url)
Moisture penetration is a common failure mode of microsensors [8]. Penetration of moisture inside the package occurs through diffusion and permeation, both of which can be accelerated by temperature and humidity [14]. Moisture ingress coupled with temperature variations may result in condensation leading to current leakage and corrosion [14]. Fig. 3.2 shows the permeability of various materials. Pure crystals and metals are the best materials as a moisture barrier. Glass (silicon dioxide) is an excellent moisture barrier. Organic polymers, such as epoxies and silicones are several orders of magnitude more permeable to moisture than glass.

The knowledge of the tightness of sealed cavity devices is essential for the evaluation of their long-term stability. Even an extremely small gas leakage along the bonding interface will lead to a significant pressure evolution inside the cavity, since the enclosed volume is usually very small. The pressure change per unit time in a device can be expressed as [16]

$$\frac{dP}{dr} = \frac{L}{V}$$  \hspace{1cm} (3.1)

where $L$ is the leak rate and $V$ the volume of the device. To determine the gas leakage, different methods are available such as the helium leak detection method, the radioisotope method or the Fourier-transform infrared spectroscopy (FTIR) method. Usually, the silicon package is exposed to a test gas at pressure $P_t$ and the corresponding internal partial pressure $P$ of the test gas inside the cavity is given by [17]
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\[ P = P_0 \left[ 1 - \exp\left( -\frac{L}{VP_0} T \right) \right] \]  

(3.2)

where \( L \) is the leak rate, \( T \) is the exposure time, \( V \) is the volume of the cavity and \( P_0 \) is equal to 1 atm.

The helium leak detection method is a common way to test the hermeticity of electronic devices. The package is subjected to helium gas under several atmospheres pressure. After pressurization, the package is introduced into a vacuum chamber attached to a helium sensitive mass spectrometer. Helium leaking out along fine leak paths is detected and converted into a standard leak rate. Helium is chosen since it efficiently penetrates along fine leak paths, has a high diffusion rate and is an inert gas (no reactions with the exposed materials are to be expected). The minimum detectable leak rate for the helium detection method is dependent on the cavity volume [18]. The ultimate sensitivity is given by the detection limit of the mass spectrometer [18]. The minimum detectable leak rate is in the order of \( 5 \times 10^{-11} \) to \( 5 \times 10^{-10} \) atm cm\(^3\) s\(^{-1}\) [16]. For cavity volumes in the order of \( 4 \times 10^{-4} \) to \( 4 \times 10^{-3} \) cm\(^3\), which would be realistic for a silicon sensor, a leak rate of \( 5 \times 10^{-11} \) atm cm\(^3\) s\(^{-1}\) corresponds to an increase in the pressure inside the sensor of 1-10 mbar/day from equation (3.1). This will clearly be unacceptable for many silicon sensor applications. Hence, the helium leak detection method is limited to relatively large volumes. For the radioisotope method, a tracer gas (e.g. Kr\(^{85}\)) is forced into the package by pressurization. The quantity of the gas that has penetrated the leak channel is then measured by an external gamma counter which determines the activity of the internal gas [17]. In the case of the FTIR method, the gas concentration and thereby the leakage into the sealed cavity after external pressure exposure is measured by FTIR absorbance. As for the radioisotope method, with the FTIR method, the amount of gas leaking into the cavity is measured directly and the minimum detectable leak rate is independent of the cavity volume [18].

3.2 Electrostatic bonding

Electrostatic bonding has become a key process in microsensor and microactuator technology since its introduction in 1969 [19]. Other terms commonly used for this technique are anodic bonding or field assisted bonding. This technique is nowadays widely used for the hermetic sealing of micromachined devices.

Electrostatic bonding is based on joining an electron conducting material (e.g. silicon) and a material with ion conductivity (e.g. alkali-containing glass). The bonding mechanism is assisted by heating at 180-500 °C and the application of an external
electric field in the range 200-1500 V. The glass used is normally a sodium glass, like Pyrex 7740 from Corning, TEMPAX from Schott or SD-2 from HOYA. It must be slightly conducting at the chosen bonding temperature. A standard pretreatment of the wafers to be bonded may be needed (e.g. 5 minutes in H₂O₂: H₂SO₄, 1:2.5 by volume). When the external electric field is applied at the elevated temperature, the positive ions in the glass (Na⁺, Li⁺) move and create a depletion layer in the glass near the silicon surface. The voltage drop over this depletion layer creates a large electric field that pulls the wafers into intimate contact. The voltage should be applied for a time-period long enough to allow the current to settle at a steady state minimized level. At the interface, oxygen from the Na₂O reacts with the silicon and generates an oxide layer [20]. The principle of anodic bonding is shown in Fig. 3.3.

![Fig. 3.3. Schematic set up for anodic bonding of (a) glass to silicon and (b) silicon to silicon with intermediate glass layer.](image)

The thermal expansion coefficient of the glass used should match the thermal expansion coefficient of silicon to prevent generation of thermal stress. To avoid this problem, anodic bonding of silicon to silicon with an intermediate glass layer on one of the silicon wafers can be used, see Fig. 3.3 (b). The intermediate glass layer can be deposited by sputtering [21, 22], evaporation [23, 24] or spin-coating [25]. With this method, wafers can be bonded at low temperatures (down to room temperature [22]) with applied voltages of only 50 V. However, the bond strength obtained is usually lower than for conventional bulk glass to silicon anodic bonding [25]. This can be explained by the dependence of the bond strength with the bonding temperature [26] and the quality of the glass layer surface.

Glass to silicon electrostatic bonding is also possible with intermediate layers. For instance, bonding with silicon dioxide, aluminium, silicon nitride and polysilicon as intermediate layers is possible [27, 28].

Anodic bonding usually leads to strong and hermetic bonds. When two bonded wafers are subjected to pull tests, fracture occurs either in the glass or in the silicon structure but not at the bond interface [14, 26, 29].
In the presented work, anodic bonding of glass to silicon with chromium, gold and silicon dioxide as intermediate layers was used to provide electrical feedthrough conductors to a hermetically sealed low-pressure cavity (paper 2). This technique was also used to enclose a gas inside a cavity (paper 5) and to form a resistant mask for deep glass etching (paper 3).

### 3.3 Direct bonding

When two mirror-polished wafers are bonded together without any adhesives or applied external forces it is called fusion, thermal or direct bonding. Wafer direct bonding usually involves a surface preparation step, a room temperature contacting step, and an annealing step to increase the bond strength. The surface preparation involves cleaning the mirror-smooth, flat surfaces to form hydrophilic wafers (or hydrophobic when a HF dip cleaning is used for silicon wafers). While hydrophobic wafers do not contact as easily as hydrophilic wafers, the final bond obtained has a higher strength [5]. Following this preparation, the wafers are contacted at room temperature and heat treated, at 600-1200 °C, to bring the bond to full strength [30]. Temperature rises causes the gas in the cavity to expand, building up a pressure which can separate the wafers or produce plastic deformation [31]. To avoid this problem, vent channels were used to create an air path between the inside of the cavity and the exterior (paper 8).

For the work presented silicon fusion bonding was used to bond micromachined silicon wafers (papers 5-8). A pretreatment of 20 minutes in H\textsubscript{2}O\textsubscript{2}:H\textsubscript{2}SO\textsubscript{4}, 1:2.5 by volume was used to enhance hydrophilicity. This treatment gave a fairly good bond at room temperature. After heat treatment for one hour at 1150 °C the bond strength is as high as that of bulk silicon [30]. In papers 5 and 6, fusion bonding with intermediate silicon dioxide was realized.

### 3.4 Eutectic bonding

Eutectic bonding uses the eutectic point in the metal-silicon phase diagrams to form silicides as intermediate layers [4, 32]. This technique has been used for silicon-to-silicon wafer bonding by depositing a thin gold film on the surface of one of the wafers. When the substrates are heated, gold and silicon form a eutectic melt already at 363 °C, well below the melting points of both silicon and gold. For the Pb/Sn system, the bonding temperature is only 183 °C. Advantages of this technique are the relatively low temperature process and the ability to bond relatively rough surfaces (the melt fills the surface irregularities). However, gold eutectic bonding is not advised for sensors integrated with CMOS electronics due to the potential of metal contamination. Another reported example is the bonding of silicon and glass wafers with Ti/Ni as an intermediate
The good adhesion obtained between the wafers is due to the formation of nickel silicide at 440 °C, and Ti has good adhesion to glass.

3.5 Adhesive bonding

Adhesive bonding is used to join two substrate materials with an adhesive intermediate layer such as epoxies or polymers. After applying the adhesive layer (generally by spin-coating), the wafers are contacted and the bond is formed by a heat curing step and/or pressure force application. The advantages of adhesive coating are the low temperature process (often less than 450 °C) and possibility to join different materials. However, it is difficult to obtain humidity-insensitive, uniform and hermetic bonds [26, 34]. Problems of adhesive shrinkage due to temperature variation may occur as well.

3.6 Other sealing techniques

Sealing techniques other than the ones mentioned above may be used to form hermetically sealed devices at a wafer level. Sealing of micromachined cavities can be done by Chemical Vapor Deposition (CVD), evaporation and sputtering [35-37]. Reactive sealing can also be used [38]. It involves reacting the cavity’s structural material to form a seal (e.g. oxidizing a polysilicon structure). Polymer deposition by spin-coating [39] is another means to seal off cavity openings. A method using a HF-assisted bonding procedure and pressure loading, with proven air-tightness, was also presented [40].

3.7 Cavity pressure measurements

The knowledge of the internal pressure of sealed micromachined devices is essential for the evaluation of their behavior. A pressure variation over time may, for example, alter the frequency response of an accelerometer or the sensitivity of a pressure sensor.

Gas pressures inside hermetically sealed cavities can be determined by a differential measurement method [41, 42]. The device is placed inside a chamber and by varying the chamber pressure, \( P_1 \), and measuring the deflection at the center of a membrane, one can determine the internal cavity pressure, \( P_0 \), as illustrated in Fig. 3.4. When the membrane is flat, the pressure inside the chamber is assumed to be equal to that in the sealed cavity.

Fig. 3.4. Determination of the cavity pressure by membrane deflection measurement.
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The internal cavity pressure can also be calculated from the center deflection of a diaphragm as [43, 44]

$$P_0 = P_1 + Ah + Bh^3$$  \hspace{1cm} (3.3)

where $P_0$, $P_1$, and $h$ are the cavity pressure, the outside pressure and the diaphragm center deflection, respectively. $A$ and $B$ are constants depending on elastic properties, shape and residual stress of the diaphragm.

The internal cavity pressure may also be found indirectly, by measuring a pressure-dependent parameter associated with the device. In the case of resonators, the pressure-dependent parameter used is usually the quality factor or the resonant frequency [45, 46]. A leak is first introduced to the cavity on a test device and the quality factor is measured as a function of pressure. By matching the measured $Q$-factor of a sealed device with the reference curve, one can thus determine the internal cavity pressure. This technique was used in papers 1 and 2 to find the internal pressure of encapsulated resonators, as shown in Fig. 3.5, where the measured and theoretical $Q$-factor are plotted for various pressures. By reporting the $Q$-value measured for the low-pressure encapsulated structures ($Q=2350$) in the reference graph, the internal pressure was found to be 1 mbar.

Other methods such as integrating a pressure sensor in the cavity [46] and absorbance measurement [18] have been reported. A combination of several measurement techniques may also be used for increased accuracy.

![Fig. 3.5. Determination of the internal cavity pressure of encapsulated resonators indirectly by $Q$-factor measurements (papers 1 and 2).](image)
4 Vacuum sealing technology

4.1 Low-pressure cavity sealing

Various fabrication techniques can be used to form low-pressure cavities at the wafer level. These include low-pressure bonding, sealing of an opening having access to the cavity, and gettering.

Low pressure bonding

Bonding performed in vacuum is a widely used technique to produce low-pressure encapsulated devices. Low-pressure anodic bonding [41, 47] and low-pressure fusion bonding [48] are mainly used for this purpose. In this work, low-pressure anodic bonding was the adopted technique (papers 1, 2, 7 and 8).

Fig. 4.1 illustrates the low-pressure anodic bonding process used in paper 1 to encapsulate silicon resonators. To facilitate the achievement of a low pressure in the cavities between the wafers, spacers are usually placed between them while evacuating the chamber, see Fig. 4.1(a). Another alternative is to separate the wafers from each other using electrostatic clamping before bonding (Fig. 4.1(b)). The first solution was adopted in paper 1.

---

*Fig. 4.1. Illustration of low-pressure anodic bonding.*
The anodic bonding sequence used is illustrated in Fig. 4.2.

\[\text{Temperature} \quad 400^\circ \text{C} \quad \text{Elevated temperature: the glass becomes slightly conducting}\]

\[\text{Pressure} \quad 1000 \text{ mbar} \quad \text{Low pressure bonding} \quad 10^{-5} \text{ mbar}\]

\[\text{Voltage} \quad 800 \text{ Volts} \quad \text{High voltage: a large electric field pulls the wafers into intimate contact}\]

\[\text{Current} \quad 10 \text{ mA} \quad \text{Current stabilisation}\]

\[\text{Time} \quad 0 \text{ mA} \quad 1 \text{ mA}\]

\textit{Fig. 4.2. Low-pressure anodic bonding sequence.}

One drawback of low-pressure anodic bonding is that it results in gas desorption (oxygen) from the surfaces inside the cavity and the pressure of the sealed cavity increases \([41, 42]\). An initial bonding pressure of $10^{-5} \text{ mbar}$ resulted in a final internal cavity pressure of 1 mbar, which is the lowest pressure reported by low-pressure anodic bonding without getter materials (papers 1 and 2).
Cavity sealing under vacuum

A metal or nitride deposition [49], a selective epitaxial growth [50] or an LPCVD (Low Pressure Chemical Vapor Deposition) process performed in vacuum are other attractive methods for obtaining vacuum-sealed cavities.

The procedure is relatively simple. First, the resonator is encapsulated in a cavity (e.g. using a conventional anodic bonding procedure), leaving a small opening that will be sealed in a low-pressure deposition or growth process. The process is illustrated in Fig. 4.3 for a low-pressure metal deposition. The residual gases present in the cavity are first evacuated and the channel is then sealed off during the deposition process, yielding a low pressure cavity.

**Fig. 4.3. Illustration of vacuum packaging using a low-pressure metal deposition process.**

Pressures below $10^{-3}$ mbar can be expected in the best cases [36]. Henmi et al. [41] report on cavity sealing with Al and SiO carried out under a pressure of $10^{-6}$ mbar after a glass-silicon anodic bonding process. The gas inside the cavity was evacuated for 2 hours before Al sealing. The obtained final pressure inside the cavity was approximately 260 mbar. Alternatively, evacuation was carried out with substrate heating at 170 °C and the final pressure after SiO sealing was 80 mbar.

It is important to have good step coverage to obtain good sealing properties. This technique can be used to form metal interconnections between internal electrodes and the outside world at the same time. One possible drawback could be the deposition of a thin layer in the cavity itself.
**Gettering**

While efforts are made to effectively evacuate gases from the internal cavity before sealing, there will always be some generation of gases during device operation or fabrication [16]. For instance, if the final step of fabrication is an anodic bonding, the elevated temperature of the process will contribute negatively to the outgasing and consequently to a higher pressure inside the cavity. To avoid a build-up of pressure, a solution is to introduce a getter material into the cavity. These materials are generally used for vacuum lamps and electronic tubes. They have the ability to absorb gases when activated at a certain temperature. Different types of getter materials in powder or metal forms exist [16]. They consist of metal alloys containing materials like Ba, Ti, Fe or Al which chemically react with the residual gases to absorb them. Fig. 4.4 illustrates the absorbing effect of a getter introduced in the cavity.

![Gettering Diagram](image)

**Var. 4.4. The gettering effect.**

High vacuum cavities can be achieved with getters. Pressures lower than $10^{-5}$ mbar have been reported using a NEG (Non-Evaporable Getter) [41]. However, using a getter implies delicate processing steps in the fabrication process. Thus, it is seldom used in micromachining.

### 4.2 Resonator gas damping

A mechanical system with one degree of freedom, $x$, and harmonic excitation is described by the differential equation [51]

$$m\ddot{x} + c\dot{x} + kx = F_0 \sin \omega t$$  

(4.1)
where \( m \) is the mass, \( c \) is the viscous damping, \( k \) is the stiffness and \( F = F_0 \sin \omega t \) is a harmonic force driving viscous damping. The solution of equation (4.1) consists of two parts, the solution to the homogeneous equation \( m\ddot{x} + c\dot{x} + kx = 0 \) and the particular solution. The solution of the homogeneous part decays exponentially with time and is only initially significant. The particular solution is a steady state oscillation of the same frequency as the excitation and can be assumed to be of the form

\[
x = X \sin(\omega t - \phi) \tag{4.2}
\]

where \( X \) is the amplitude of oscillation and \( \phi \) is the phase of the displacement with respect to the exciting force. The amplitude and the phase can be expressed in nondimensional form by introducing the following parameters:

\[
\omega_n = \sqrt{\frac{k}{m}} \quad \text{natural frequency of the undamped oscillation}
\]
\[
c_c = 2m\omega_n \quad \text{critical damping}
\]
\[
\zeta = \frac{c}{c_c} \quad \text{damping factor.}
\]

The nondimensional expressions for the amplitude and phase then become

\[
\frac{Xk}{F_0} = \frac{1}{\sqrt{1 - \left(\frac{\omega}{\omega_n}\right)^2 + 2\zeta \left(\frac{\omega}{\omega_n}\right)^2}} \tag{4.3}
\]

and

\[
\phi = \tan^{-1} \left(\frac{2\zeta \left(\frac{\omega}{\omega_n}\right)}{1 - \left(\frac{\omega}{\omega_n}\right)^2}\right) \tag{4.4}
\]

Equations (4.3) and (4.4) are plotted in Fig. 4.5. For small damping, the amplitude becomes very large at resonance (i.e. \( \omega = \omega_n \)). The phase angle at this frequency is \(+90^\circ\) or \( -90^\circ \). The phase change between \(+90^\circ\) and \( -90^\circ \) was used to detect resonance in papers 7 and 8.
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The operation of a silicon resonant sensor is based on the principle that the resonance frequency is altered by the physical parameter of interest. This resonance frequency is thus the measured output of the sensor. The performance of such a device is related to its mechanical quality factor. The quality factor of a silicon resonant structure is usually defined as the total energy stored in the structure divided by the sum of energy losses from the vibrating element per cycle [52]:

\[
Q = \frac{2\pi \text{Total energy of the system}}{\text{Dissipated energy per cycle}}
\]  \hspace{1cm} (4.5)

The \( Q \)-value can also be calculated from the amplitude-frequency spectrum of the vibration by taking the resonance frequency, \( f_n \), divided by the width of the resonance peak at -3 dB:

\[
Q = \frac{f_n}{\Delta f_{n,-3\text{dB}}}
\]  \hspace{1cm} (4.6)

A high \( Q \)-factor is important as it implies better identification of the resonance frequency and low losses from the resonator [53]. A resonator acts as a bandpass filter rejecting noise at frequencies outside its bandwidth. A narrow resonance peak gives a high \( Q \)-factor with better rejection of external noise sources and high stability. A high mechanical \( Q \)-factor means that the sensor performance is almost entirely dependent on
the mechanical properties of the resonator element. This implies high accuracy and long term stability.

The quality factor of an oscillating silicon resonator is often limited by damping. A high $Q$-factor is associated with low damping. There are several damping mechanisms that one should consider when designing a resonator. The total $Q$-factor of a resonant silicon structure may be divided into three components [52-54]: $Q_a$ which represents the acoustic and viscous dissipations, $Q_s$ which stands for the support dissipations and $Q_i$ which corresponds to the internal and material related dissipations. This can logically be written as:

$$\frac{1}{Q} = \frac{1}{Q_a} + \frac{1}{Q_s} + \frac{1}{Q_i} \quad (4.7)$$

This relationship indicates that the limitation of the total $Q$-factor comes from the lowest $Q$-factor of these three components. In an environment like air the lowest $Q$-factor is normally $Q_a$, the one related to acoustic and viscous dissipations. By definition, viscous damping is caused by the lateral displacement of the air surrounding the vibrating surface of the resonator and it corresponds to the squeeze-film damping while the origin of acoustic losses are due to the perpendicular displacement of the air surrounding the vibrating surface [52]. When the resonator material is made of single-crystalline silicon the contribution of the internal intrinsic $Q_i$-factor can be neglected in comparison to the other energy loss dissipations [52]. If we now consider $Q_s$, its contribution can also be neglected if the resonator oscillates in a balanced vibration mode. Thus the total $Q$-factor related to a balanced vibration system can be approximated by the quality factor $Q_a$, related to viscous and acoustic damping.

When the resonator oscillates close to a surface where the electrodes are usually placed for excitation and detection the air molecules must escape in order to enable the resonator to move closer. When the gap is small compared with the overlapping areas, the pressure between the resonator and the surface builds rapidly and results in extremely high resistance to motion and a much lower quality factor. Understanding this mechanism mainly due to squeeze film damping is of crucial importance in designing silicon resonators.

Squeeze-film damping has been investigated extensively [55-61]. The distribution of the gas pressure between two moving plates is governed by the Reynolds equation for a compressible gas film [59]. If the gas pressure is low, the molecular mean free path is not negligible compared to the gap between the plates and the effective viscosity is pressure dependent [62]. All these studies can be used to predict the influence of squeeze-film damping and the response of the resonator (amplitude and frequency responses). It
can also be very advantageous to make simulations which can help as a first step in the
design of the device. Such simulation models have been successfully utilized for
accelerometers [62-64] and angular rate sensors [65] for example. Simulation tools like
APLAC [66], which was used in paper 4, give very good results.

Fig. 4.6 illustrates the influence of gas-film damping on the $Q$-factor for different
pressures and cavity depths for the structures presented in paper 1. These measurements
show that the vibration damping is dominated by squeeze-film damping for small recess
depths (20 µm or less) and that a pressure below 1 mbar is needed to achieve $Q$-factors of
more than 3000. The same structures bonded to two glass lids with recess depths of
20 and 175 µm, had a $Q$-value of 5000 (paper 3).

![Fig. 4.6. Effect of gas-film damping on the Q-factor at different pressures and cavity
depths (paper 1).](image)

### 4.3 Thermal isolation

Vacuum sealing of micromachined cavities is an important technique to create thermal
isolation for thermal MEMS devices. For thermal radiation sensors, a vacuum seal
reduces heat dissipation [67]. The minimized thermal conductance between a sensing
element and its package may lead, for instance, to high sensitivity [41].

Thermal conduction is related to the propagation of heat from the encapsulated device
to its surroundings. Therefore, it is important to understand the influence of gases around
the encapsulated device. The process of heat transfer by gases is different in the case of
viscous state and in that of molecular state [16]. In the first case, the movement of a
large number of molecules is responsible for the heat transfer (by diffusion), while in the
second case each individual molecule carries the heat.

In the case of viscous state, the thermal conductivity of a gas is fairly independent of
pressure (since the viscosity is not a function of the pressure), although it varies with
temperature. For conduction between two parallel surfaces separated by a distance, \( d \), the heat transfer coefficient, \( G_{\text{dense \ gas}} \) (in \( \text{W K}^{-1} \text{ m}^{-2} \)), is given by [68]

\[
G_{\text{dense \ gas}} = \frac{\kappa}{d} \quad (4.8)
\]

where \( \kappa \) is the thermal conductivity (in \( \text{W K}^{-1} \text{ m}^{-1} \)).

The conductivities of various gases are listed in Table 4.1. When a gas is encapsulated in a sealed cavity, its thermal conductivity characteristics may influence the performance of the device. A high conductivity may introduce thermal losses and increase the power consumption. Consequently, the chosen gas and/or encapsulation process may be of great importance. For example, an anodic bonding encapsulation produces \( \text{O}_2 \) in the cavity, while a fusion bonding procedure generates \( \text{H}_2, \text{H}_2\text{O} \) and \( \text{N}_2 \) [42].

<table>
<thead>
<tr>
<th>Gas</th>
<th>Heat conductivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Helium</td>
<td>3.43×10^{-4}</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>4.19×10^{-4}</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>5.7×10^{-5}</td>
</tr>
<tr>
<td>Oxygen</td>
<td>5.8×10^{-5}</td>
</tr>
<tr>
<td>Air</td>
<td>5.8×10^{-5}</td>
</tr>
<tr>
<td>Carbon dioxide</td>
<td>3.4×10^{-5}</td>
</tr>
</tbody>
</table>

Table 4.1. Heat conductivity of gases at 0 °C (\( \text{cal cm}^{-1} \text{s}^{-1} \text{K}^{-1} \)), from [16].

At low pressure, when the mean free path of the molecules of the gas becomes large compared with the dimensions of the enclosure (molecular state), the heat transfer is carried out by individual molecules instead of by the gas as a whole. The heat transfer is directly proportional to the density of the molecules, \( \rho \) (in \( \text{kg m}^{-3} \)), i.e. to the pressure \( P \). The heat-transfer coefficient can then be approximated by [68]

\[
G_{\text{low pres.}} = G_0 \frac{P}{P_0} = \rho c_v u_{\text{mol}} \quad (4.9)
\]

where \( G_0 \) is the heat-transfer coefficient at the reference pressure \( P_0 \), \( c_v \) is the specific heat at constant volume, and \( u_{\text{mol}} \) is the average molecular velocity. For surface micromachined structures with 1 µm-gaps, the thermal conduction by the gas in the gap may be pressure-dependent up to almost atmospheric pressure [68].

Micromachined filaments are often encapsulated in a vacuum cavity to reduce heat losses by heat conduction through the surrounding gas. Such an encapsulation enables
low electrical power consumption to be achieved and may prevent oxidation of the filaments (e.g. made of polysilicon) during operation [69]. The surrounding pressure may also influence their response time. In [70], the influence of pressure on both power consumption and response time was evaluated. By vacuum encapsulation, both the power consumption and the time constant decreased. Another advantage of low-pressure encapsulation is that vacuum-sealed devices may not need individual temperature compensation [29].

4.4 Reference cavity pressure

Vacuum sealing is widely used to produce absolute pressure sensors. A zero pressure reference cavity is of great importance, not just for true pressure measurement but also for removing gas expansion effects that can be produced by large temperature changes. In a pressure sensor, a diaphragm generally deforms under the influence of pressure. One side of the diaphragm is exposed to the environment and the other side to a sealed cavity. Such a cavity must be hermetically sealed, preferably with vacuum inside, to provide a stable reference pressure and to prevent variations in the dielectric constant of the gas in the cavity (which may influence capacitive pressure measurements [71]).
5 Gas sealing technology

5.1 Gas encapsulation techniques

Various techniques can be used to encapsulate a gas in micromachined cavities at the wafer level. These include bonding of two wafers and sealing an opening under a gas atmosphere. Welding technology such as laser welding has also been used to form pressurized cavities [72, 73]. However, to the author’s knowledge, this technique has not been demonstrated at the wafer level.

**Bonding under gas atmosphere**

Encapsulating a gas at the wafer level using different bonding techniques has been reported, i.e. using polyimide or epoxy adhesive bonding [18, 74], anodic bonding [75] and silicon fusion bonding [5].

![Diagram of gas sealing by anodic bonding](image)

*Fig. 5.1. Gas sealing by anodic bonding (paper 5).*

In paper 5, anodic bonding at overpressure up to 2 bar was used to encapsulate CO$_2$ in micromachined sealed cavities. The procedure used is illustrated in Fig. 5.1 and the bonding parameters are displayed in Fig. 5.2. The wafers are first separated from each other using an electrostatic clamp and the chamber in which they are placed is evacuated of gas. To perform electrostatic clamping, the temperature of the wafer stack must be heated at about 200 °C, otherwise the glass is not conductive enough and cannot be clamped electrostatically. An alternative is to lift the silicon wafer, which has higher
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electrical conductivity than glass. Then, the CO₂ gas is introduced inside the chamber and its pressure is adjusted to the desired value. In paper 5, the chamber pressure could be adjusted up to 2 bar. The wafers are then put into contact, with the gas trapped into the cavities. The temperature is elevated to the bonding temperature (in our case 430 °C) and anodic bonding can be performed. The final cavity pressure was 1 bar.

Fig. 5.2. Sequence for sealing a gas using anodic bonding (paper 5).

Cavity sealing under gas atmosphere

A gas can also be inserted in a cavity by sealing a small opening in a gas atmosphere. This can be realized for example by CVD [38], sputtering [76] and epitaxial growth [50]. The principle is shown in Fig. 5.3 for a growth process (i.e. reactive sealing). It consists of exposing the substrate to a gas atmosphere, which causes growth of material in the channels sufficient to close them off. As mentioned in [38], a layer builds up on all surfaces exposed to the gas, including the interior surfaces of the cavity, and the active gas trapped in the cavity may be consumed. Therefore, the gas to be encapsulated is
rarely the active gas, but rather an additional gas which does not react with the substrate. For instance, if the substrate is exposed to an oxidizing ambient as in the figure, the exposed silicon surfaces will form silicon dioxide to seal off the channel opening. The remaining oxidizing gas trapped in the cavity will continue to oxidize the surfaces of the cavity until the oxygen in the cavity is exhausted. In the case of sputtering, the deposition is carried out in the presence of the gas to be introduced into the cavity [76]. The channels to be sealed must be small enough to be sealed off in a reasonably short period of time while being large enough to allow the cavity to be filled with the gas to be encapsulated. Good step coverage is favourable for good sealing properties.

![Illustration of gas encapsulation using reactive sealing.](image)

**5.2 Pressure controlled cavity**

The final pressure inside the cavity after sealing is of great importance to predict the device behavior and performance. For example, the frequency response of a mechanical accelerometer inside a cavity is strongly dependent on the gas pressure. A pressure lower than the estimated value may result in a fragile accelerometer with insufficient mechanical overload protection. In contrast, a pressure higher than the predicted value may result in a device insensitive to accelerations because of too much damping. A precise control of the internal pressure is therefore required for optimum damping characteristics of the seismic mass [77]. It is thus important to know which parameters influence the final cavity pressure.

According to Boyle’s law for an ideal gas, the final cavity pressure, $P_{\text{cavity}}$, can be expressed as [45]
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\[ P_{\text{cavity}} = P_{\text{process}} \frac{V_{\text{unsealed}}}{V_{\text{sealed}}} \frac{T_0}{T_{\text{process}}} \frac{n_{\text{products}}}{n_{\text{reactants}}} \]  

(5.1)

where \( T_0 \) and \( T_{\text{process}} \) denote room and processing temperatures, respectively, \( V_{\text{unsealed}}/V_{\text{sealed}} \) denotes the ratio of the volume of the cavity prior to and after sealing, \( P_{\text{process}} \) denotes the processing pressure and \( n_{\text{products}}/n_{\text{reactants}} \) denotes the mole ratio of gaseous products and reactants.

A slight volume variation, e.g. due to a membrane deflection, may be induced as a result of the gas sealing, but generally the cavity volume remains approximately the same, resulting in a volume ratio \( V_{\text{unsealed}}/V_{\text{sealed}} \) close to unity. It was demonstrated that the mole ratio is not the dominant factor in determining the final cavity pressure in the presence of no reactions [45]. The most important parameters for the final cavity pressure in the presence of no reactions (e.g. chemical reaction or diffusion) are the processing temperature and the processing pressure [45, 78].

In paper 5, CO\(_2\)-filled cavities were fabricated for use as optical filters. The desired cavity pressure for optimum optical performance of the filters was 1 bar. Using the Boyle’s law, the process parameters to build the CO\(_2\)-filled chambers were determined. With a bonding temperature of 430 °C, the pressure to obtain a final cavity pressure of 1 bar was 2 bar. Measurements clearly showed the influence of the bonding pressure on the filter’s final cavity pressure and absorption characteristics, see paper 5.

The pressure in the cavity can be controlled by selecting the partial pressures of inert gas during the sealing process [31, 38, 79]. For example, when wafers are contacted in air, and subsequently annealed at high temperature, the oxygen within the cavity can be completely consumed to form an oxide on the interior walls of the cavity. This results in a pressure inside the cavity of 0.8 atm, consistent with the consumption of the 20 % oxygen in air [13]. Wafers bonded in pure oxygen may show results close to those bonded in vacuum [80].

The final cavity pressure can also be influenced by the sealing technique used, the annealing time and treatment, the surface pretreatment, and the bonded area surrounding each cavity [5, 42, 48, 80]. Fusion bonding two silicon wafers in vacuum results in the generation of mainly H\(_2\), H\(_2\)O and N\(_2\) gases inside the cavities, the total gas pressure being primarily determined by the H\(_2\) component [42, 81]. In that case, the gas generation takes place during annealing and is strongly temperature dependent [48]. It also depends on the bonding area surrounding the cavity, a large bonding area corresponding to a higher cavity pressure [5]. Cavities sealed by anodic bonding contain mainly O\(_2\) originating from mobile oxygen ions inside the bonding glass [42]. In contrast to silicon fusion bonding, the residual gas pressure inside anodically bonded cavities is nearly independent of the bonded area surrounding the cavity and the bonding
voltage does not have any significance either [42, 48]. For silicon direct bonding under vacuum conditions, hydrophobic bonding leads to about 50% lower residual pressure compared to hydrophilic bonding, where a slow saturation of the gas pressure after annealing is observed [48]. Pressure within sealed cavities can further be controlled by the use of getters. For example, in [43] a getter was introduced in the cavity to absorb the residual gas (O₂) produced during anodic bonding. The cavity pressure could be controlled by introducing an inert gas (argon) under appropriate pressure during anodic bonding.

5.3 Light absorption

A sensor containing a special gas in its chamber shows a characteristic spectral sensitivity in the wavelength range of the infrared light that is absorbed by the filling gas. The gas-filled device can therefore be used as an optical filter absorbing specific wavelengths of the incoming radiation. Such filters are widely used in medical respiratory applications as wavelength selection devices.

According to the Beer-Lambert’s law, the absorption increases exponentially with length. The intensity, I, drops exponentially along the coordinates of propagation, say z,

\[ I = I_0 \exp(-\alpha z) \]  \hspace{1cm} (5.2)

where \( I_0 \) is the intensity at \( z=0 \), and \( \alpha \) the attenuation constant. After propagation for one “decay length”, \( z=\alpha^{-1} \), the intensity drops to \( e^{-1} \), i.e., to 37%, after two decay lengths to 13%, and after four to 2%.

In paper 5, CO₂ optical gas-filters fabricated by anodic bonding in CO₂-atmosphere were realized. The anodic bonding was carried out at overpressure to encapsulate more gas in the chamber to obtain optimized optical absorption characteristics. A cross section of the filter and the transmission spectrum are shown in Fig. 5.4. One can clearly notice the strong peak absorption of CO₂ at 4.23 μm and the influence of the bonding pressure on transmission. Numerical simulations based on the Beer-Lambert’s law indicating the degree of light absorption in each layer and the light reflection at each interface were presented in paper 5.
Fig. 5.4. SEM cross-section and optical transmission as a function of wavelength for different bonding pressures of the CO$_2$-filter presented in paper 5. The cavity length of absorption is 1 mm and the same chamber filled with air was used as reference. The absorption peaks correspond to the absorption characteristics of CO$_2$.

5.4 Thermal gas expansion

In many micromechanical structures having a sealed cavity, one of the wafers is thinned down to form a membrane. When a gas is present in the cavity and the system is heated, the gas will expand and the membrane will deflect. This principle can be used for IR sensor systems (e.g. based on Golay cell) as illustrated in Fig. 5.5. IR light is pre-absorbed by the ambient gas and then heats the gas inside the chamber. The deflection of the diaphragm induced by expansion of the trapped gas is detected as a capacitance change. The chamber can be filled with various gases. The sensor containing a certain gas shows a specific response due to the intrinsic spectrum of absorption of ambient gas. Ambient gas detection can thus be realized.

![Light source](image1)

![Light source](image2)

Fig. 5.5. Golay cell using the thermal gas expansion principle [74].
6 Electrical feedthrough technology

Electrical feedthrough conductors are generally needed to connect an active area of the sensor, situated inside the sealed cavity, to the outside world. They supply electrical power to the sealed region (e.g. needed for actuation) and collect electrical output signals from the encapsulated sensor. A challenge in the realization of sealed cavities is to provide hermetically sealed electrical feedthrough conductors. When designing hermetically sealed electrical feedthroughs, different aspects must be considered, as discussed below for lateral, vertical and diffused electrical feedthrough conductors.

6.1 Lateral metallic electrical feedthroughs

Lateral electrical feedthrough metal conductors are commonly used [71, 83]. The use of standard batch fabrication steps makes the lateral electrical feedthrough technique very attractive.

*Step height*

The step height of the electrical feedthrough is one of the major causes of hermetic failure since a small air leakage channel may remain after bonding. This is illustrated in Fig. 6.1(b), showing a seal with air leakage, produced by anodically bonding a silicon substrate (coated with a thin oxide for isolation) to a glass substrate with an electrical feedthrough metal conductor. During such a bonding process, a hermetic seal can be produced along the step at the edge of the feedthrough provided that the metal height does not exceed 50 to 100 nm [71], see Fig. 6.1(a). With metal step heights above this level, a phenomenon known as “tenting” occurs. This is characterized by a small opening channel formed along the edge of the feedthrough. In paper 2, where we used chromium and gold as feedthrough metal conductors, we demonstrated that the maximum possible metal thickness to obtain a hermetic seal was approximately 50 nm. It should be noted that this limit probably varies slightly from metal to metal, depending on the metal’s ability to deform. In [84], a hermetic seal was obtained using a 60 nm-thick layer of TiW-Au.
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Fig. 6.1. Cross-section illustration of (a) a hermetic seal and (b) a seal with air leakage, obtained by anodic bonding of a silicon substrate to a glass substrate with electrical feedthrough metal conductors. For metal thicknesses larger than 50 nm [71], the resulting bond presents air leakage.

In a seal as the one shown in Fig. 6.1 (a), one employs the glass-to-metal compression effect. At the bonding temperature, the metal expands as the glass deforms and fills the created void at the edge of the conductor. When the wafers are cooled, the metal contracts at a higher rate than the glass. Since the glass can withstand and transfer these compressive loads, a hermetic compression seal is formed.

A solution to reduce the tenting effect in the glass and to improve the bond quality has been proposed [71]. As shown in Fig. 6.2, it consists of having sharp planar feedthrough projections in order to form a compression bond that eliminates tenting regions that would otherwise form at the edges of the conductor. A thin oxide for isolation is also introduced along the path of the feedthrough, extending out on each side of the path, as illustrated in Fig. 6.2. The sharp planar metal projections, sharp edges, and deformable thin film topographical structures enable a hermetic seal to be formed during electrostatic bonding of a silicon substrate to a glass substrate. The preferred feedthrough conductor material should be somewhat ductile, such as titanium. During the bonding process, the glass (the softest material used here) is deformed along the planar projections, the tenting gaps are closed at the sharpest metal regions and a compression seal is formed. In the case of titanium, an electrostatic bond with the surface of the glass substrate is also created, increasing the bond strength. Note that several sharp planar projections are preferable to increase the reliability of the resulting seal. In [71], the thickness of the titanium layer was 100 nm. A drawback with this method is the introduction of a relatively large parasitic capacitance between the metal conductor and silicon, as the required conductor area is relatively large.
Parasitic capacitance

One important parameter to consider when designing electrical feedthrough conductors across a sealed boundary is the parasitic capacitance. The feedthrough conductor must be isolated from the silicon by an insulating layer, e.g. oxide, to eliminate any conductive path between the feedthrough conductor and the underlying silicon substrate. The parasitic capacitance depends on the thickness and dielectric constant of the insulating layer, and on the contact area between the electrical feedthrough and the underlying silicon. This can be expressed by the following equation:

\[ C_{\text{parasitic}} = \frac{A \varepsilon_e \varepsilon_r}{t} \]  

(6.1)

where  
- \( A \) is the contact area between the electrical feedthrough and the underlying silicon,  
- \( \varepsilon_e \) is the vacuum permittivity (or vacuum dielectric constant),  
- \( \varepsilon_r \) is the relative permittivity of the insulating layer, and  
- \( t \) is the thickness of the insulating layer.

Parasitic capacitance can also be introduced between the electrical feedthroughs themselves, which can be a problem when a large number of feedthroughs are needed [14]. Grounding every other line or connecting many lines in parallel provides an easy way to reduce this crosstalk [14].
When anodic bonding is performed between glass and an oxide-coated silicon substrate, the thickness of the oxide used to obtain a good bond should not exceed 460 nm [4]. Thus, a relatively large parasitic capacitance between the feedthrough conductor and silicon is introduced in parallel with the capacitance one wants to measure, as illustrated in Fig. 6.3. This parasitic capacitance is, in most cases, of the same order of magnitude as the capacitance one wants to measure, which leads to difficulties in sensing very small capacitive signals. To reduce the parasitic capacitance, narrow feedthroughs are generally used, which minimize the contact area between the feedthrough and the isolation layer at the sealed interface. For example, in [84], 10 µm-wide conductor lines were formed.

![Diagram of parasitic capacitance and electrical equivalent](image)

**Fig. 6.3. Illustration of parasitic capacitance and the electrical equivalent.**

In paper 2, the external bonding pads and the internal cavity electrodes were connected by a narrow 20 µm-wide metal conductor through the bonded area to minimize the parallel capacitance coupling due to the presence of oxide, as illustrated in Fig. 6.4. A thermal oxide was grown on the silicon resonators to isolate the electrical metal conductors from the silicon. Different oxide thicknesses were tested. Results showed that a good bond was obtained for thicknesses of less than 460 nm, which is in good agreement with previously published results [4]. The oxide thickness used was 430 nm. The calculated and measured resting capacitance between one contact pad and the silicon was 3 pF for a 30 µm deep cavity. Calculations showed that the parasitic capacitance was approximately 1.77 pF, which is about half the capacitance at rest. The calculated capacitance variations due to the structure oscillation were less than 40 fF. Consequently, the detection sensitivity of the capacitance variations is reduced by a factor of approximately three.
Damages during bonding and over time can degrade the feedthrough quality. Therefore, the choice of the feedthrough conductor material is of great importance. At elevated temperatures (e.g. during anodic bonding), the metallic conductors (e.g. Al or Ti) can become oxidized, and hence exhibit high resistance properties [85]. Corrosion of the thin films used as feedthroughs can be a cause of failure. Corrosion-resistance materials such as TiW-Au are preferably used.

**Thick metallization and embedded feedthroughs**

Methods of achieving a hermetic seal with thicker metallisation have been proposed [29, 86]. The metal feedthroughs can be recessed into the silicon or glass surfaces. As explained in [86], when using recessed metal feedthrough conductors, hermeticity can be difficult to obtain, for different reasons. Not only does the thickness of the metal fill have to be kept within tight limits, but, as is known to happen for pyrex etching with metal masks, long and flat slopes are formed on the sides of the trench due to mask underetching. Consequently, unfilled areas may remain on these slopes, leading to air leakage. In addition, due to mask adjustment errors, metal feedthroughs may not be placed in the centers of the trenches. To overcome these difficulties, Hiltmann et al. [86] proposed the solution presented in Fig. 6.5. The feedthrough layout is modified in order to form a small overlap of the concave metal corners with the glass trench, which seals the trench slope tightly, see Fig. 6.5.
Another means of forming thick hermetically sealed electrical feedthroughs conductors is to encapsulate the feedthroughs themselves inside isolating layers before cavity sealing, as illustrated in Fig. 6.6. Such examples have been reported [14, 87]. The metal feedthrough is first deposited and patterned on an isolation layer such as thermal oxide. One or several insulators are then deposited on the wafer to encapsulate the feedthroughs. The top layer deposited over the feedthroughs is bonded to a wafer lid to seal the cavity. A critical issue for this type of feedthrough is to obtain a planar top layer surface suitable for bonding. Any non-planarity (more than 100 Å as stated in [14]) or surface irregularities (roughness, voids, defects, etc.) may compromise the hermeticity of the seal. Therefore, a planarization step is necessary. Standard chemical mechanical polishing (CMP) or high-temperature annealing processes can be used for planarization.

For vertical feedthrough conductors, contacting can be done either through silicon [88-90] or through glass [91-93].

In the case of contacting through silicon, an opening is formed by anisotropic etching. In that case, an insulating layer is needed and a parasitic capacitance is introduced.
as previously described for lateral feedthrough conductors. Electrical contact can be made using a low-pressure deposition process of a conductive layer, as illustrated in Fig. 6.7 (a).

Fig. 6.7. Vertical electrical feedthrough connections (a) through silicon and (b) through glass [67].

To eliminate the problem of parasitic capacitance, vertical contacting through glass is an attractive solution. No insulating layer is necessary. However, for contacting through glass, the process is more complicated; it requires a deep etching step in a silicon-bondable glass, which can be very difficult (paper 3). To form a hole in glass, methods like laser structuring [94, 95], electrochemical discharge drilling [96, 97] or sand blasting have been reported. These methods make it possible to produce straight walls in glass, although they do not meet the desired standard batch process requirements and introduce accuracy problems. The best way to form a hole through glass with micromachining compatibility is by wet etching. The principle of wet etching is illustrated in Fig. 6.8. Several wet etching methods are available, including deep etching using an LPCVD polysilicon deposited mask [98] (contamination problems may arise (paper 3)). Another method presented in paper 3 uses an anodically bonded silicon substrate as a mask for glass etching. Using HOYA-SD2 glass with a conventional chromium and gold mask is another interesting alternative since this glass etches faster than Pyrex 7740 and is especially suited for anodic bonding to silicon [47]. In paper 8, we used this glass to wet etch cavity depths of 100 µm.
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Fig. 6.8. Illustration of glass wet etching and important factors to obtain deep etching.

To avoid any air leakage due to the tenting effect as previously seen in the case of lateral feedthroughs, Esashi et al. [99] proposed a solution where a vertical feedthrough structure is formed, see Fig. 6.9. This vertical structure has no air leakage through the channel formed by the glass tenting because the glass and silicon are anodically bonded around the glass hole.

Fig. 6.9. Diffused feedthrough conductor sealing by vertical contacting [99].

6.3 Diffused electrical feedthroughs

To avoid structural steps on the bonding surface, one can use diffused feedthrough conductors across the sealed boundary [67, 93, 100]. The electrical signals are transmitted through p-type (or n-type) diffused lines in an n-type (or p-type) silicon substrate. A
significant problem with this technique is the induced surface electrical leakage between
the n and p layers as a result of the electrostatic bonding process, which tends to degrade
the pn-junction [71, 100], as illustrated in Fig. 6.10. The difficulty of avoiding mobile
ions to drift from the glass to the pn-junction causes electrical instability and reliability
problems [100]. Electrical leakage between the n and p layers also occurs in the silicon
substrate and is dependent on the contact area of the diffused layer with the silicon
substrate. Such diffused feedthroughs have a fairly high resistance which limits the
amount of current supplied to the hermetically sealed region [29, 101]. The parasitic
capacitance between the electrodes and silicon is relatively high using this method,
making it unsuitable for capacitive detection.

![Illustration of current leakage in diffused feedthrough conductors.](image)

To overcome the problem of surface electrical leakage, the diffused feedthrough may
be buried under an epitaxial layer [100]. The buried conductors are connected to the metal
(aluminium) via doped polysilicon contacts and contact holes in a passivation layer, see
Fig. 6.11. The use of buried conductors isolates the pn-junction under the epitaxial layer
and away from the influence of the ions from the glass. During the electrostatic bonding
process, the ions from the glass will affect the surface of the epitaxial layer only where
no pn-junctions are located, thus preserving the reliability of the buried pn-junction
[100].
The resistance of the feedthrough conductor is often of great importance and is dependent on the layer resistivity and dimensions as \( \rho l/A \), where \( \rho \) is the resistivity of the conductor, \( l \) its length and \( A \) its cross-section area. In the case of doped feedthroughs, a heavily doped layer may reduce the resistance. However, a diffused layer feedthrough has some resistance and is generally used for low voltages. Connecting several feedthrough lines in parallel can be used to further reduce the resistance.

Fig. 6.11. Illustration of a buried diffused feedthrough conductor to avoid degradation of the pn-junction during anodic bonding [100].
7 A low-pressure encapsulated resonant silicon density sensor with integrated electrodes

7.1 The silicon resonant density sensor

The sensor to be encapsulated was presented in [6, 102, 103]. It consists of a planar double-loop system with dimension $8.6 \times 17.7 \times 1$ mm, as illustrated in Fig. 7.1. The tube structure is connected at the center to a surrounding support frame having external dimension of $12 \times 21 \times 1$ mm. This frame is bonded to two glass covers to form a hermetic seal, thereby encapsulating the sensor inside a low-pressure cavity. The tube inlets and outlets are situated at the side of the frame, see Fig. 7.1. With an effective length of 61 mm, a silicon wall thickness of 100 µm and a cross-sectional fluid area of $0.57 \text{ mm}^2$, the tube contains a sample fluid volume of 0.035 ml [6].

Fig. 7.1. The resonant fluid density sensor vibrating in the anti-phase torsion mode.

Fig. 7.2 shows the four different out-of-plane vibration modes of the resonator. The anti-phase torsion mode is the interesting mode, since it leads to a balanced vibration; the torsion and bending moments of the two loops cancel out throughout the oscillation period, minimizing the mechanical losses due to the oscillating movement and thus maintaining a high $Q$-factor [103].
Fig. 7.2. The four investigated vibration modes of the resonant structure.

The sensor operation is based on the vibration of the tube system in the selected anti-phase torsion mode. The structure is excited electrostatically and the vibrations are detected capacitively. A change in density of the fluid in the tube changes the mass loading of the tube system and consequently changes the resonance frequency of the vibration. Thus the resonance frequency is a measure of the fluid density. The resonance frequency, $f_0$, of the vibrating tube is related to the fluid density $\rho_f$ by [6]:

$$\frac{1}{f_0^2} = \frac{1}{C^2 E} \left( \frac{A_f}{A_f + A_t} \rho_f + \frac{A_t}{A_f + A_t} \rho_t \right)$$

(7.1)

where $C$ is a constant that depends on the loop dimensions and the vibration mode, and $\rho_f$ and $\rho_t$ are the densities of the fluid and the tube material (Si), respectively. $A_f$ and $A_t$ are the cross-sectional areas of the fluid and the tube wall. $E$ is Young’s modulus of the tube material. The numerical values of these parameters can be found in [6].

A simplified resonant test structure, having the same design layout and mechanical properties as the densitometer, was used to investigate and develop the presented encapsulation concept. This structure is much easier to fabricate than the densitometer since only one KOH-etching step is necessary to form the double-loop shaped structure.

7.2 Gas film damping

The test structures were anodically bonded to glass lids having various recess depths to study the influence of the cavity depth on our resonator mechanical $Q$-factor. A theoretical $Q$-factor related to squeeze-film losses was derived. The final expression
obtained for two glass lids is (paper 1):

\[
Q_{\text{squeeze}} = \frac{4\pi \rho_s}{\eta \left( b^3 + b'^3 \right) \left( 1 + 9.638 \left( \frac{P_0 \lambda_0}{Pd} \right)^{1.159} \right) d^3}
\]  

(7.2)

where \(d\) is the distance between the lid wall and the resonator, \(P\) the internal cavity pressure, \(f\) the resonance frequency, \(\eta\) the viscosity of air at atmospheric pressure, \(\lambda_0\) the mean free path at pressure \(P_0\), \(b\) and \(b'\) the widths of the resonator in front of glass, \(\rho_s\) the silicon density and \(A\) the cross-section area of the resonator.

Measurement results are presented in Fig. 7.3 which also includes the theoretical model for the squeeze-film \(Q\)-factor. For these tests, the structures were excited electrostatically with an external electrode and the vibrations were detected optically (paper 1). The results clearly show that vibration damping is completely dominated by the squeeze-film effect for small recess depths at atmospheric pressure. For larger depths, the squeeze-film effect is less dominant and acoustic losses become more important.

![Fig. 7.3. Theoretical squeeze-film and measured Q-values versus glass lid recess depth at 1000 mbar for the anti-phase torsion mode (one glass lid).](image)

7.3 Simulations

A dynamic simulation model for the vibrating silicon resonator was investigated (paper 4). The model includes electrostatic excitation, two torsional and two bending vibration modes and the influence of gas-film damping. It predicts the \(Q\)-factors and amplitudes of resonance from the device dimensions. It also includes the change in resonance frequency caused by the DC bias voltage applied during electrostatic excitation. The model is implemented as an electrical equivalent circuit and simulated with the
circuit analysis program APLAC [66].

In the model, three energy domains (electrical, mechanical, and fluidic) are described by means of electrical equivalents. Fig. 7.4 shows the complete block model of the sensor. Each vibration mode is represented by one block which consists of an excitation source, a resonator and separate squeezed-film damping blocks for the parallel and transverse parts of the silicon loop structure. The electrical actuation voltage is converted into twisting moments, $\tau_1\ldots\tau_4$, which act as input for each resonance circuit. The displacement at an arbitrary position $(x, y)$ in the structure is the superposition of the tilting angles, $\theta_1\ldots\theta_4$, of the various modes.

Measurements were performed and compared to the simulation results for different pressures. The structures vibrate close to the glass lid surface situated 16 $\mu$m away from the resonator. The measurement set-up is similar to that described in paper 1; the excitation is electrostatic and the detection is optical. The results illustrated in Fig. 7.5 show good agreement between measured and simulated $Q$-factors and frequency responses, which confirms the validity of the model.
Fig. 7.5. Simulated (—) and measured (□) frequency responses of the test structure at 100 mbar and 1000 mbar. The model is fitted to the measured resonance frequencies and Q-values in vacuum, but not to the Q-values at high pressures (i.e. 100 or 1000 mbar).

7.4 Low-pressure encapsulation

The test resonators were encapsulated using a final anodic bonding procedure performed at $10^{-5}$ mbar in a Karl Suss SB6 bonder (paper 1). The device consists of a triple-stack sandwich; the resonator in the middle and two Pyrex glass lids forming a hermetically sealed low-pressure cavity, see Fig. 3.5. We did not use any getter material or gas evacuation procedure after the bonding. Reference structures where the ambient air leaks into the cavity were also fabricated. These structures were used to determine the internal pressure of the encapsulated structures by indirect Q-value measurements (see section 3.7). The final internal pressure obtained was 1 mbar.

In Fig. 7.6, where the vibration amplitudes are plotted in the frequency domain, the influence of pressure on damping is demonstrated. At low pressures, the resonance peak is much higher and sharper than at high pressures, meaning a higher Q-factor.
Vacuum-sealed and gas-filled micromachined devices

In Fig. 7.7, the $Q$-values and resonance frequency dependence on air pressure for a structure without a glass lid are reported. From this graph it can be seen that at 0.1 mbar $Q$-values of 20 000 are measured for the non-encapsulated structures, while $Q$-values up to 30 000 were obtained for structures encapsulated between two glass lids (see Fig. 3.5). The structures without glass lids have higher support losses than the structures with glass lids since the glass-lid arrangement is stiffer than the structure without glass lids.

From Figs. 7.6 and 7.7, we notice a slight frequency shift from 1000 mbar to 100 mbar where lower air mass is involved in the oscillation.

Fig. 7.6. Measured frequency spectrum at different pressures for a structure with one glass lid of 16 µm recess depth.
Thierry Corman

Fig. 7.7. Q-values and resonance frequencies vs. air pressure for the structure without a glass lid.

7.5 Lateral electrical feedthrough conductors

The next step was to introduce electrodes for electrostatic excitation and capacitive detection. The major design issue in this step was to provide hermetically sealed electrical feedthrough conductors from the external environment, while preserving the low pressure cavity which contributes to a high quality factor.

Fig. 7.8(a) illustrates the fabrication sequence for the low-pressure encapsulated resonator with hermetic lateral electrical feedthrough conductors. Metal conductors were deposited and patterned on the previously microstructured glass lids (paper 2). The final encapsulation was performed at wafer level during a low-pressure anodic bonding procedure. The final encapsulated device consists of a triple-stack wafer sandwich made up of the resonator in the middle and two surrounding Pyrex glass wafers to define the hermetic seal of the resonator inside the low-pressure cavity. Lateral electrical feedthrough metal conductors connect the electrodes situated on the glass inside the hermetically sealed cavity across the sealed boundary to the external bonding pads. The metal conductors’ thickness was 44 nm and the maximum oxide thickness used for isolation was 430 nm. The different parts of one encapsulated device with integrated electrodes are shown in the exploded view of Fig. 7.8(b).
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Glass etching
Metallization
KOH-etching
Oxidation
Anodic bonding
Spacers
Gas evacuation
Anodic bonding
Cr/Au/resist
Cr/Au (44 nm)
Si
Glass
Si
Oxide (430 nm)

(a)
(b)
Top glass lid
Silicon resonator
Metal electrodes
Bottom glass lid

Fig. 7.8. (a) Fabrication sequence and (b) exploded view of the silicon resonator encapsulated at wafer level between two glass lids with electrical feedthrough metal conductors for electrostatic excitation and capacitive detection (paper 2).

Fig. 7.9 shows a photo of the encapsulated resonators with integrated electrodes before dicing. The special arrangement of the electrodes makes it possible to saw the triple stack wafer in only three steps (paper 2). The final device is thus fabricated using only standard micromachining techniques.

Fig. 7.9. Photo of the low-pressure encapsulated resonators with integrated electrodes before dicing. The wafer diameter is 100 mm.
An alternating voltage of approximately 5 V \text{r.m.s.} was sufficient when using one of the integrated electrodes for excitation.

The internal cavity pressure of the low-pressure encapsulated structures with integrated electrodes could be determined indirectly by measuring the \(Q\)-factor. The internal cavity pressure was the same as that obtained for the encapsulated structures without encapsulated electrodes, i.e., 1 mbar. This means that the seal obtained with the electrical feedthrough conductors is as good as the seal formed without integrated electrodes.

A special electronic circuit was developed to detect the small capacitance variations due to the oscillations (paper 2). These variations are about 40 fF per \(\mu\)m displacement around a value of about 3 pF at rest. We could measure the resonance frequency of the vibrating structures (~ 9175 Hz) but the circuit was not accurate enough to measure the \(Q\)-factor.

A long-term stability test shows that after storage for two years, no leakage has been observed.

### 7.6 Burst technology and elimination of feedthroughs

A novel method for excitation and detection of resonant sensors based on discontinuous, "burst" excitation was developed (paper 7). The solution eliminates the crosstalk between excitation and detection by separating them in time. It also eliminates electrical feedthrough conductors for electrostatically excited and capacitively detected resonators.

The principle illustrated in Figs. 7.10 and 7.11 can be described as follows. The resonator is excited during a certain time by an alternating signal at a selected frequency corresponding to the device resonance frequency of interest. The excitation is then switched off and the detection starts on the now freely oscillating resonator. In this way, the excitation and detection are separated in time and do not interfere with each other. A short time delay is inserted between the excitation and the detection to allow stable oscillation of the structure without direct influence of the excitation signal. After the detection period, the excitation is once again switched on and the cycle is thus completed. The excitation is phase-locked to the detected signal, thus keeping the resonator at resonance.
In the “burst” technology solution, the excitation consists of a burst of pulses. This allows a specific resonance frequency to be selected, not only the resonance frequency of the dominant mode that one would automatically select when a pulse excitation is used, as in [104]. To find the desired resonance frequency, the oscillator frequency is ramped up or down from an initial frequency $f_{ini}$, until the detection circuit locks on the frequency at which resonance occurs.

![Diagram of the excitation and detection time separation](image)

**Fig. 7.11. Illustration of the excitation and detection time separation (not to scale).**

The “burst” technology was used to encapsulate the silicon density sensor (paper 8). The electrodes for excitation and detection could be placed outside the internal cavity on top of the glass lid, at a distance of 400 µm from the resonator. As neither electrical
feedthroughs nor oxide were needed, the fabrication of the encapsulated sensor was simplified, with an increased yield and less risk of leakage from the low-pressure cavity. A large resonator-to-lid wall distance of 100 µm could be formed, reducing squeeze-film damping losses and consequently increasing the quality factor. The measured $Q$-value was 3400 with air in the tube. As a comparison, a similar unencapsulated densitometer gave a $Q$-factor of 3000 [103]. A schematic view and a SEM cross-section of the encapsulated density sensor are shown in Fig. 7.12.

Fig. 7.12. Schematic drawing and cross-section of the encapsulated resonant fluid density sensor. The electrodes used for excitation and detection are placed on top of the glass lid. No electrical feedthroughs are needed. The distance between the inner glass surface and the silicon tube is approximately 100 µm.

The density sensitivity was measured for air and five different solutions of 2-propanol in water. The feedback control loop of the “burst” electronics allows the variations of the resonance frequency to be followed while changing the fluid density in the tube. The density measurements could thus be done “on-line”. High excitation voltages alternating between 0 V and 150 V could be applied to the resonator with measured electronic sensitivities of 0.4 fF. A high signal-to-noise ratio of 100 was obtained using the “burst” electronics. The density measurement results are reported in Fig. 7.13, showing that the linear relationship of equation (7.1) between the density and the squared inverse of the frequency is respected. The results show a high density sensitivity of $-200$ ppm (kg m$^{-3}$)$^{-1}$. Temperature dependence measurements were also performed on the encapsulated device. Low temperature sensitivities of $-29$ ppm °C$^{-1}$ were obtained over the range 20-100 °C (paper 8).
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Fig. 7.13. (a) Photo of the low-pressure encapsulated density sensor and (b) the squared inverse of the measured resonance frequency plotted as a function of fluid density of air and five different water/2-propanol mixtures. A straight line fit is shown. These measurements are made at 23 °C using the closed-loop “burst” electronics.

As illustrated in Fig. 7.12, a glass dielectric medium was placed between the resonator and the metal electrodes. As explained in [105], this degraded the device reliability as a result of localized charge storage on the glass surface. The electrostatic field effect is locally reduced in the air gap, which decreases the detection voltage during capacitive detection. To avoid this problem, we proposed a solution (paper 7) consisting of alternately switching the polarity of the detection bias voltage. Thus, the accumulation of charges at the dielectric surface was eliminated by discharging the capacitance formed between the resonator and the metal electrodes.

As described in paper 7, the “burst” electronics were evaluated with electrostatic excitation and capacitive detection. It may also be applied to other types of excitation and detection principles, such as piezoresistive, piezoelectric, optical or thermal principles. The “burst” technology was also tested with an encapsulated resonant pressure sensor (paper 7), which demonstrated its high adaptability to different sensors. The same electrodes could be used for both excitation and detection. The electronic evaluation revealed that the “burst” duty cycle (i.e. the excitation time relative to the free oscillation time, or \( t_1/(t_1+t_2) \)) had a strong influence on the output detection voltage. The time interval \( t_1 \) corresponds to the excitation period, and \( t_2 \) to the free oscillation period, see Fig. 7.11. It was also shown that only two excitation periods at a burst duty cycle of 1 % and a burst frequency \((1/(t_1+t_2))\) of 115 Hz were sufficient to select and lock the resonance frequency for the tested pressure sensor (paper 7).
8 An integrated micromechanical IR gas analysis system

In papers 5 and 6, the design, fabrication and characterization of a miniaturized IR-sensor system to measure CO₂-concentration was presented. This work is the result of a medical technology research collaboration with Datex-Ohmeda, Instrumentarium Corp., Finland. This miniaturized IR-sensor system is especially designed for CO₂-concentration measurements in patient airways during anesthesia or intensive care.

8.1 Principle of operation

The sensor unit is located outside the patient tube as illustrated in Fig. 8.1. As in any conventional IR sensor systems for respiratory gases such as CO₂, the miniaturized IR sensor system consists of a light source, a filter and a detector. The light source is used for emission of IR light. The filter is necessary to produce a reference signal used for long-term stability and for compensation against cuvette contamination. The detector sends the measured output signal (corresponding to the detected CO₂-concentration) to the patient monitor, which subsequently interprets the results and sends back the appropriate amount of oxygen to the patient.

![Fig. 8.1. The infrared CO₂-gas sensor unit is placed outside the patient airway.](image)

The principle of operation of the developed sensor is illustrated in Fig. 8.2. The system comprises an IR-source chip with two pairs of diagonally arranged IR-sources generating two sample and reference beams. The filter consists of a chip arranged with two CO₂-filled chambers placed directly beneath the two reference sources, as shown in Fig. 8.2. These chambers are used to produce the reference beam by “pre-absorption” at the CO₂-wavelength absorption band around 4.23 µm. By using exactly the same gas in the reference chamber as the gas one wants to measure, the filter presents the same
Vacuum-sealed and gas-filled micromachined devices

absorption properties as the gas in the airway. The sample signal is absorbed by the CO$_2$ in the patient airway, giving an indication of the gas concentration. A higher CO$_2$-concentration in the tube increases the absorption of the sample beam. By electrically switching between the sample and the reference sources, one sample and one reference signals are gathered at the detector. The ratio between the sample and reference signals indicates the degree of light absorption in the patient airway, and thus gives the gas concentration.

![Infrared sensor system](image)

**Fig. 8.2.** The infrared sensor system for measurement of CO$_2$-concentration.

The developed CO$_2$-sensor system has several advantages compared to conventional systems. The IR-source and CO$_2$-filter chips are fabricated using silicon micromachining batch fabrication techniques. This enables a small sensor size, relatively low-fabrication costs, low power consumption and a high performance device with fast electrical modulation time of the IR-sources. The special CO$_2$-chambers arrangement, together with the principle based on the two electrically switched sources, eliminates any moving parts, contrary to conventional systems using rotating wheels for light modulation. Another advantage of this IR sensor system is its ability to measure the gas concentration without direct contact to the gas.
8.2 IR-polysilicon microfilament sources

As shown in Fig. 8.3, the IR-source chip is made up of four groups of IR-sources, which generate the two switched sample and reference beams. The sources are connected diagonally two by two to produce a more uniform light at the detector. The fabrication sequence of the source chip is illustrated in Fig. 8.3. Basically, the IR-sources consist of phosphorus-doped polysilicon filaments encapsulated inside low-stress silicon nitride. They are suspended across KOH-etched cavities and electrically connected to aluminium bonding pads. A more detailed fabrication description can be found in paper 6.

![Fabrication process and SEM-photo of the micromachined IR-source chip.](image)

The filament length is 1 mm which is a compromise between the mechanical strength and the thermal characteristics of the IR-source. Shorter filaments would result in higher mechanical strength with the drawback of an uneven temperature distribution over the filament areas. The cavity depth under the filaments has a direct influence on power consumption and thermal response time. As shown by the theoretical calculations of Fig. 8.4(a), the larger the cavity depth, the lower the power consumption and the higher the thermal response time. A cavity depth between 100 and 300 µm results in a relatively fast modulation time (< 10 ms) necessary for fast switching between the sample and reference beams, and relatively low power consumption (~1 W for two sources).
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For characterization of the sources, a PbSe light detector was placed at a distance of approximately 19 mm away. The filament temperature could be measured using a thermo-camera. The peak (maximum) temperature was measured for different power consumptions, see Fig. 8.4(b). The sources normally operate at 700 °C, which corresponds to a power consumption of about 0.5 W for each source. The corresponding 10-90 % light emission response time was 4 ms.

In Fig. 8.5(a), a thermographic photo of an IR-source chip with a 100 µm deep cavity having a peak temperature of about 615 °C is shown. From this diagram, the temperature distributions along two lines ("Line 1" and "Line 3") in Fig. 8.5(b) were obtained. Line 1 is aligned perpendicular to the filament wires. This results in a relatively good thermal isolation and a correspondingly uniform temperature distribution, with only minor irregularities due to the openings between the wires. Line 3 is aligned parallel to the wires. The temperature distribution is less uniform as a result of thermal conduction along the wires to the cavity edge support. However, the temperature is relatively constant over the filament area due to the dominating heat transfer through this 100 µm-deep cavity air gap.
8.3 The CO₂-filter

For an optimal CO₂-filter, the beam transmission through the glass and silicon wafers should be as high as possible. Therefore, the silicon surface is preferably coated with an antireflection coating (e.g. silicon nitride or silicon dioxide) and the glass wafer should be as thin as possible to minimize absorption losses.

Fig. 8.6 shows a photo of a complete wafer stack of CO₂-optical filters, before dicing.

The filter fabrication is described in Fig. 8.7(a). The chamber is formed by double-side KOH-etching through two 500 µm-thick silicon wafers. Note that if the etching is prolonged just after having etched through the wafers, the inclined walls are eliminated.
and straight walls are formed instead, as can be seen by the dashed lines in Fig. 8.7(a). These wafers are fusion bonded together to form 1 mm long chamber holes. Then, these two wafers are fusion bonded to a third silicon wafer lid coated with silicon dioxide as an antireflective coating. A glass wafer is anodically bonded to the triple-wafer stack at overpressure in a CO₂-atmosphere to form hermetically sealed CO₂-chambers. Prior to anodic bonding, the wafers are separated from each other using electrostatic clamping to introduce the CO₂-gas inside the chamber (after vacuum ventilation), as illustrated in Fig. 8.7(a). Since anodic bonding is performed at an elevated temperature (~ 430 °C), the internal cavity pressure after cooling is approximately half the bonding pressure. Therefore, bonding was performed at overpressure up to 2 bar, encapsulating more gas for optimized absorption characteristics. Finally, the glass wafer is thinned down in HF to reduce glass absorption losses, and the bonded wafers are sawed into individual filters (or vice versa). The yield obtained for this process was higher than 95 %. Fig. 8.7(b) shows a top view and a cross-section of a fabricated CO₂-filter chip before the final glass etching step.

![Diagram](image_url)
Paper 5 demonstrated that a silicon dioxide thickness of 0.575 µm gave a maximum light transmission at the wavelength of CO₂ absorption (4.23 µm); the measured transmission through a silicon wafer coated on both sides with 0.575 oxide was 75 %. The influence of the pyrex glass thickness and the bonding pressure on the transmitted IR-beam were also investigated in paper 5. The results showed that the transmission through a 500 µm-thick pyrex glass wafer was only 40 %, while a 125 µm-thick glass wafer gave a transmission of 65 %. As expected, the bonding pressure had a strong effect on the absorption of the filter; at a pressure of 1 bar, the measured CO₂ absorption at 4.23 µm was 49 % while at a pressure of 2 bar, it reached almost 90 %.

For further optimization and understanding of the filters, numerical simulations based on a transfer matrix method were carried out in paper 6. These simulations indicate the degree of IR-light reflection at each interface and the absorption in each layer of the filter. The simulations and some measurement verifications were performed on two types of filters. The initial filter constituted the first filter type. It consists of a 500 µm-thick pyrex glass lid having no antireflective coating. The second type, the optimized filter, was fabricated with an oxide layer of 0.575 µm on the two silicon surfaces in the path of the IR-beam. These filters were bonded at 2 bar and had a cavity length of 1 mm. For the optimized filters, the glass lid was thinned down from an initial thickness of 500 µm to 125 µm to reduce the absorption through glass.

The simulation results are reported in Fig. 8.8. One can note the strong absorption of CO₂ for the reference beam (about 65 % of the initial beam for the optimized filter, and 31 % for the initial filter). One can also realize the benefit of having a thin glass lid. By reducing the glass lid thickness from 500 µm (initial filter) to 125 µm (optimized filter), the absorption in pyrex decreased from 61 % of the incoming light to 22 %. These simulations show the positive effect of the antireflective coating, which minimizes reflection losses for the reference beam. For the sample beam, higher reflection losses are introduced with the antireflective coating than without it. This is desirable since it is easier to compare the two beams if they are the same order of magnitude.
Fig. 8.8. Simulated optical transmission for the reference and the sample beams through the multilayers of the initial and optimized filters. These simulations are based on a transfer matrix method for multiple-reflections, multiple-media (paper 6). The reflection at each interface as well as the absorption in each layer are clearly seen.

To evaluate the fabricated filters, infrared transmission measurements were performed. The resulting optical transmission spectrum of the optimized CO$_2$-filter and the spectrum of the initial CO$_2$-chamber are shown in Fig. 8.9. Air is taken as reference (100 % transmission). These measurements confirm the strong absorption peak of CO$_2$ at 4.23 µm. As expected, optical losses have been minimized. The total transmission
increased from 3.0 % (initial filter) to 6.7 % (optimized filter). Our numerical simulations presented in Fig. 8.8 are in good agreement with the measurements (the total calculated transmissions for the reference beam are 2.3 % for the initial filter and 6.9 % for the optimized filter), which confirms the validity of the theoretical model.

Temperature cycling tests between 20 °C and 70 °C followed by new transmission measurements were performed and did not reveal any gas leakage, confirming the high quality of the hermetically sealed chambers.

![Graph showing transmission vs. wavelength for different filter types](image)

**Fig. 8.9.** Measured transmission for the reference beam versus wavelength for both the initial and optimized filters (reference is air).

### 8.4 IR-source and CO₂-filter system for CO₂-measurements

CO₂-measurements were performed using a silicon IR-source chip with a cavity depth of 220 µm and a 1 mm CO₂-chamber filter bonded at 2 bar with oxide as antireflective coating (0.575 µm) and with a 125 µm-thick glass. The IR-source chip and the CO₂-filter chip were mounted and wire-bonded in a standard ceramic holder, see paper 6. The detector unit consisted of a conventional IR-bandpass filter (BW=80 nm) and a standard PbSe IR-detector. The test channel used for the measurements was 9 mm long. The IR-sources were supplied with 15 ms-long pulses.

The amplitudes of the sample and reference signals at different CO₂ concentrations are reported in Fig. 8.10(a). As expected, the sensitivity for CO₂ concentration is higher for the sample than for the reference beam. This is due to the preabsorption of the reference beam in the CO₂-chamber. The ratio between the sample and the reference signals is plotted in Fig. 8.10(b), which shows a typical calibration curve for the whole IR-sensor.
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system. The measured values of Fig. 8.10(b) were neither electrically filtered nor averaged which explains the deviation of these points from the fitted calibration curve. In a real time CO₂-measurement, the CO₂-concentration measured values are filtered with a special algorithm to minimize noise and averaged over 4 to 8 breath-to-breath cycles, leading to very accurate measurements. These measurements show high CO₂-sensitivity in the range of interest (between 0 % and 10 % of CO₂) which indicates that the desired performance of the micromachined IR-sources and CO₂-chambers can be achieved.

![Graphs](image)

Fig. 8.10. Measured (a) detector reference and sample signals and (b) sample to reference signal ratio, as a function of the CO₂-concentration in a test channel. Note that the light source intensities of the sample and reference IR-sources were different for these measurements.
9 Summary of appended papers
The appended papers describe the design, fabrication, simulations and measurements on different low-pressure sealed and gas-filled micromachined devices. Paper 1 presents an investigation of squeeze-film damping on the $Q$-factor and resonance frequency for resonant test structures. Paper 2 presents a low-pressure encapsulation technique with hermetically-sealed electrical feedthrough conductors for electrode integration. Paper 3 presents a method to realize deep etching in Pyrex glass. Paper 4 presents a dynamic simulation model for the resonant double-loop test structures. Paper 5 and 6 present the IR-source and CO$_2$-filter system for measurement of CO$_2$-concentration. Paper 7 presents the “burst” technology which eliminates electrical feedthrough conductors and crosstalk between excitation and detection. Paper 8 presents the fully low-pressure encapsulated and closed-loop operated density sensor using the “burst” technology.

Paper 1
A simplified silicon resonant structure derived from a liquid density and mass flow sensor design was used to investigate the effect of air damping on its $Q$-factor and resonance frequency. The structure was anodically bonded to glass lids having different recess depths. A theory for the squeeze-film damping and its relation to recess depth and air pressure was presented. $Q$-factor measurements were made for the different vibration modes. Low-pressure encapsulation with a resulting cavity pressure of 1 mbar was realized. The interior pressure was deduced by comparing $Q$-values of similar structures varying only in the fact that they had a leak channel which made it possible to vary the interior pressure.

Paper 2
Low-pressure encapsulated resonators with hermetically sealed lateral electrical feedthrough metal conductors were fabricated. The encapsulated device consists of a triple-stack sandwich with the resonator in the middle and two glass lids with metal electrodes forming a hermetically sealed low-pressure cavity. The internal cavity pressure was the same as that obtained for encapsulated structures without integrated electrodes, i.e. 1 mbar. The integrated electrodes were used for both electrostatic excitation and capacitive detection. An electronic circuit was developed for capacitive detection. The special electrode design made it possible to have electrodes on both glass lids using only standard batch-processing steps at the wafer level.
**Paper 3**

An investigation of deep etching in Pyrex glass is presented. The method utilizes an anodically bonded silicon substrate as a mask. Depths of more than 500 µm can be achieved very easily and the structured glass wafer is still anodically bondable after the silicon mask removal followed by an appropriate surface treatment. Lateral underetching of 1.5 times the etched depth was measured, which is about 5 times less than when a chromium and gold mask is used. This technique was used to increase the quality factor of a resonator from 1000 to 5000 using its bonded frame as a mask. The presented technique makes vertical electrical contacting through glass possible.

**Paper 4**

A dynamic simulation model for a vibrating fluid density sensor is presented. The model includes electrostatic excitation, four mechanical vibration modes and the influence of gas-film damping. The model is implemented and simulated with the circuit simulation program APLAC. It consists of several electrical equivalent blocks, each representing one vibration mode. The total block diagram of the model consists of an actuation part, a resonator and a displacement measurement part. Simulated and measured $Q$-values and frequency responses at various pressures are in good agreement.

**Paper 5**

New CO$_2$ optical gas filters were fabricated using silicon micromachining techniques. They consist of chambers filled with CO$_2$ formed by an anodic bonding procedure performed at overpressure. The fusion and anodic bonding techniques used in the fabrication led to very good air-tight gas chambers. To increase the transmission, the silicon wafer lid was coated on both sides with an appropriate antireflective coating material and the glass wafer was thinned down. These optical gas filters were specially designed for integration into a gas sensor system to provide a reference signal for measurements of respiratory gases present in patient airways during anesthesia or intensive care.

**Paper 6**

The design, fabrication and characterization of an optimized optical IR-source and CO$_2$-filter system for the measurement of CO$_2$ concentration are presented. This optical gas sensor system is specially designed for the measurement of respiratory gases present in patient airways during anesthesia or intensive care. The IR-sensor assembly consists of an IR-source chip with two pairs of diagonally arranged IR-sources for the generation of sample and reference beams. The reference beam is needed for long term stability and to compensate for cuvette window contamination. Test measurements of the optical sensor
system show high CO₂-sensitivity which indicates desired performance of the micromachined IR-sources and CO₂-chambers.

**Paper 7**

A novel method for excitation and detection of resonant silicon sensors based on discontinuous, “burst” excitation is presented. The solution eliminates the crosstalk between excitation and detection by separating them in time, and eliminates the need for feedthrough conductors for encapsulated resonators. The method was successfully tested with outside glass lid electrodes on closed-loop operated silicon resonant density and pressure sensors. High excitation voltages alternating between 0 V and 150 V could be applied to the resonators with measured detection electronics sensitivities of 0.4 fF. Signal-to-noise ratios as high as 100 (density sensor) and 360 (pressure sensor) were obtained. A number of only 2 excitation periods with a “burst” cycle frequency of 115 Hz and a “burst” duty cycle of 1% was sufficient to select and lock the resonance frequency (28 042 Hz) for the tested pressure sensor. The same electrodes could be used for both excitation and detection. A novel solution is also presented which eliminates the charging effect of dielectric surfaces which otherwise can be a problem for capacitive detection.

**Paper 8**

A fully low-pressure encapsulated and closed-loop operated resonant fluid density sensor is presented. The encapsulation is performed at wafer level in vacuum by anodically bonding the silicon densitometer between two glass lids with metals electrodes for electrostatic excitation and capacitive detection. The density measurements were performed using the “burst” technology presented in paper 7. The electrodes could be placed on top of the glass lids without using electrical feedthroughs, and a cavity gap of 100 µm could be formed between the recessed glass lid surface and the silicon tube to reduce squeeze-film damping. The closed-loop “burst” technology enabled continuous measurements of fluid densities. The sensor showed high density sensitivities in the order of –200 ppm (kg/m³)⁻¹, a high mechanical $Q$-factor of 3400 for air in the tube, and low temperature sensitivities of –29 ppm °C⁻¹ in the range 20-100 °C.
10 Conclusions

Low-pressure encapsulated resonators with hermetically sealed lateral electrical feedthrough metal conductors were successfully fabricated. The final bond was performed at $10^{-5}$ mbar without using any getter material or gas evacuation procedure after the anodic bonding procedure. The final internal cavity pressure obtained was 1 mbar, determined indirectly by measurement of the quality factor. The entire fabrication of the encapsulated structures with integrated electrodes was performed at the wafer level using only standard batch-fabrication processes. The lateral metal conductors were deposited on both glass lids for electrostatic excitation and capacitive detection. It was shown that a lateral metal conductor thickness of 44 nm together with an oxide thickness of 430 nm result in a hermetic seal. A long-term stability test of the low-pressure encapsulated structures showed that after storage for two years, no leakage has been observed indicating that the pressure inside the cavity is preserved.

The effect of squeeze-film damping due to the oscillation close to the lid walls was investigated and a theoretical model for damping was presented. The measurements and theory showed that the resonator vibration damping is dominated by squeeze-film damping for small recess depths (20 µm or less) and that a pressure below 1 mbar is needed to achieve $Q$-factors of more than 3000. It was also shown that the structures without glass lids have higher support losses than the structures bonded to glass lids. At 0.1 mbar, $Q$-values of 20 000 were measured for the non-encapsulated structures while $Q$-values of 30 000 were obtained for the structures encapsulated between glass lids. Using a bonded silicon substrate as mask for deep glass etching, the gap between the glass wall and the resonator could be increased on one side, yielding a $Q$-value of 5000 at 1 mbar.

A novel method for closed-loop excitation and detection of resonant silicon sensors based on discontinuous, “burst” excitation was presented. The method eliminates electrical feedthrough conductors and suppresses the crosstalk between excitation and detection. The “burst” technique was successfully tested with outside glass lid electrodes on closed-loop operated silicon resonant density sensors. The sensors were encapsulated in a cavity by low-pressure anodic bonding, and a large cavity gap of 100 µm between the resonator and the lid wall could be formed to reduce squeeze-film damping. Quality factors of 3400 and signal-to-noise ratios of 100 were measured. The liquid density could be measured continuously with the feedback control electronics. The sensor showed high-density sensitivities in the order of $-200$ ppm (kg/m$^3$)$^{-1}$ and low temperature sensitivities of $-29$ ppm °C$^{-1}$ in the range 20-100 °C. It was demonstrated that the same electrodes could be used for both excitation and detection. An electronic sensitivity of 0.4 fF was
measured and could be combined with high excitation voltages alternating between 0 V and 150 V. A number of only two excitation periods with a “burst” cycle frequency of 115 Hz and a “burst” duty cycle (i.e. the excitation time relative to the free oscillation time) of 1 % was sufficient to select and lock the resonance frequency.

A CO\textsubscript{2} optical gas filter was presented. It consists of 1 mm-deep micromachined cavity chambers filled with CO\textsubscript{2} during an anodic bonding procedure performed at overpressure (2 bar) in a CO\textsubscript{2} atmosphere. The final chamber pressure obtained was 1 bar. The filter was optimized with a 0.575 µm thick silicon dioxide as an antireflective coating to improve the transmission through silicon and with a glass lid thinned down to 125 µm to reduce the absorption through glass. Numerical simulations to study the filter optical characteristics were performed and were in good agreement with the measurements. The transmission through the optimized filter was 6.7 %, with a strong and desired absorption of 65 % inside the chamber. Temperature cycling tests followed by new transmission measurements showed that the fusion and anodic bonding gave high quality hermetically sealed chambers. This optical gas filter was integrated in a gas sensor system to provide a reference signal for measurement of the CO\textsubscript{2} concentration present in patient airways during anesthesia or intensive care.

As a part of the gas sensor system, IR-sources were fabricated and evaluated. They consist of incandescent polysilicon filaments coated with silicon nitride across a 220 µm deep cavity. They were electrically modulated with a relatively fast modulation of less than 10 ms and a relatively low power consumption of approximately 1 W. Test measurements of the optical sensor system showed high CO\textsubscript{2}-sensitivity in the range of interest (between 0 % and 10 % CO\textsubscript{2}).
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12 References

Vacuum-sealed and gas-filled micromachined devices


