Integrated
throughflow
mechanical
microfluidic sensors

Dennis Alveringh

INTEGRATED THROUGHFLOW MECHANICAL MICROFLUIDIC SENSORS

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The cover shows a resonance peak emerging from the noise¹ floor and dividing a droplet in two parts. Resonance plays an important role in all sensors described by this dissertation for measuring physical quantities of fluids.

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¹Is it noise?

INTEGRATED THROUGHFLOW MECHANICAL MICROFLUIDIC SENSORS

DISSERTATION

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the degree of doctor at the University of Twente,
on the authority of the rector magnificus,
prof. dr. T. T. M. Palstra,
on account of the decision of the graduation committee,
to be publicly defended
on Friday, 6 April 2018 at 14:45

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1

Introduction

The research in this dissertation¹ is motivated by the need for sensing of multiple physical quantities of fluids for medical and industrial applications. The described novel devices are limited to sensors that are fabricated using microtechnology, measure a mechanical fluid property using a mechanical transduction principle and can be integrated throughflow with other microfluidic devices on a single chip. The focus of the research lies on resolution limit analysis and improvement of Coriolis mass flow sensors and integration of flow and pressure sensors for density and viscosity sensing.

1

¹Many texts and figures of this dissertation have been published earlier in [1–13]. At the beginning of each chapter, the relevant papers are mentioned. This chapter is based on the publication:

- D. Alveringh, R. J. Wiegerink, and J. C. Lötters, “Towards system-level modeling and characterization of components for intravenous therapy,” in *Proceedings of the 2nd International Conference on MicroFluidic Handling Systems (MFHS 2014)*, Freiburg im Breisgau, Germany, 2014, pp. 106–109.

1.1 Background and motivation

As one of the most intelligent animals on earth, humans use tools to extend their capabilities [14]. Some tools enlarge the actuation impact of humans, like hammers and cars. Other tools provide a higher range and precision of sensing, like rulers and temperature sensors. Most tools are combinations of both, providing complex machines that may even be connected and communicate together. Only these complex machines allowed us to reach space [15], connect people via internet [16] and eliminate diseases [17].

Sensors can be seen as translators from the natural world to the world of machines. Many crucial substances in the natural world are fluids, e.g. oxygen and water. Animal bodies, for example, use fluids as main transport medium for energy, building material and even communication in the form of hormones. Hence, if humans want to interface to the world of machines, accurate sensing and control of fluids are essential. The control of medication delivery via intravenous therapy is an example of such a human-machine-interface. With this technology, liquid medication is directly injected in the blood of a patient. Control of the dose is of crucial importance for the health of the patient. For long-term and well-controlled intravenous therapy of medicines, e.g. antibiotics, pain killers, immunoglobulins or blood pressure medication, infusion pumps are regularly used. An infusion pump consists of an electric motor that pushes the plunger of a syringe filled with a liquid medicine. A simple infusion setup with a lumped element model is illustrated in Figure 1.1. The angular velocity of the electric motor is controlled and can be set by the medical staff. The output flow of the infusion pump is calibrated regularly. Nevertheless, the resistance and compliance of plungers, tubing and needles introduce a settling time of minutes before the flow at the patient's side is at the desired value [1, 18, 19] as plotted in Figure 1.2. Real-time feedback of the flow at the needle to the pump can form an improvement in these setups. It will act as a constant calibration of the infusion pump, enabling more accurate medicine delivery. Furthermore, with the right control loop, the settling time of the infusion could be reduced significantly by carefully increasing the pumping at the start.

The problem becomes worse when multiple infusion pumps with different medicines and different flow rates are combined and mixed. The flow rates of the composition of medicines after mixing is dependent on the set flow rates of the individual infusion pumps. When one pump is set at a higher flow rate, the patient will first receive the old composition that is left in the tubing at a higher flow rate before the new composition propagated through the system [20]. Therefore, in addition to measuring flow at the needle, measuring composition can be the next step in infusion improvement. An indirect method for measuring composition is by measuring fluid parameters like density, viscosity or relative permittivity as illustrated in Figure 1.3. When it is given which fluids are in the mixture and the fluid parameters of each individual fluid are known, assuming the mixing has a linear scaling effect on the parameters, the concentration of the fluids can be obtained.

Figure 1.1: Three graphical interpretations of a system for intravenous therapy [1], with (a) an edited photograph of the full system, (b) an illustration of the components and (c) a lumped element model. Due to the many components, the final flow through the needle will be subject to a significant settling time and other non-ideal effects. A feedback loop might improve this.

Figure 1.2: Measured flow at the end of an infusion pump with tubing [1]. Steps of 1 mL h^{-1} and 0 mL h^{-1} are set as indicated by the dashed line. Non-ideal effects of the setup are clearly visible from the measurement results. A close up of the measurements shows a settling time of minutes.

Figure 1.3: Different sensors measure the density, viscosity and relative permittivity of a mixture. When these parameters of the individual components of the mixture are known, the composition can be obtained. The effective mass flow of each component can be calculated. In principle, the number of components that can be distinguished in the mixture is one more than the amount of fluid parameters measured [21].

Besides medical applications, fluid sensing is essential in many other applications. For example in the chemical industry, where mixing the right amounts of liquids is of importance for the quality of the final product. Or in the semiconductor industry, where fast and accurate switching of gases in plasma reactors is used to fabricate integrated circuits.

The miniaturization of the sensors using microfabrication could result in advantages for most of these applications, e.g. better resolutions and lower unit prices [22]. Different fabrication methods are available in microtechnology and different materials can be used as channel material. The internal volumes of the channels in the sensors are also smaller: settling times are lower and faster control of flow and pressure is possible. Furthermore, integration of multiple sensors on one chip is possible without increasing the costs or complexity and makes the sensing of multiple quantities possible [23], which enables the measurement of e.g. the viscosity and density of the fluid.

1.2 Limits and aim of the research

Although sensors are usually specified for one physical quantity, they are sensitive to other physical quantities as well. A ruler, for example, is a common instrument to measure distance. However, as a result of thermal expansion, the ruler is also sensitive to temperature. A second sensor, that is specialized in measuring temperature, can be added to the setup. The results from the two sensors can be used to obtain the distance and the temperature and compensate for each other's measurement error. This can be seen as a form of synergy, since both sensors do not only measure both quantities, they also increase the resolution. Theoretically, the right sensor combination might seem to result in perfect resolution, but will be always limited by thermal noise. The synergy of microfluidic sensor combinations and the limitation due to thermal noise is the common thread in this dissertation.

At the MESA+ Institute for Nanotechnology at the University of Twente research has been performed on a universal technology for the fabrication of micro-sized channels for two decades. It started with buried silicon nitride channels in a silicon wafer [24, 25]. Later, a technology to fabricate suspended silicon nitride channels has been developed [26]. Latter technology has been the foundation of the micro Coriolis mass flow sensors realized by Haneveld et al. [27, 28] and further investigated and enhanced by Groenesteijn et al. [29–33].

The aim of the research described in this dissertation spans roughly two subjects:

- resolution limit analysis and improvement of microfabricated Coriolis mass flow sensors;
- integration of multiple sensors on a single chip for fluid parameter characterization.

The scientific review and progress described by the chapters of this dissertation are limited to sensors that are

- fabricated using microtechnology;
- measure a mechanical fluid quantity (flow, pressure, viscosity or density);
- use a mechanical transduction principle;
- can be integrated with other microfluidic devices on a single chip and
- can be placed throughflow (inline) with other fluidic devices.

1.3 Dissertation outline

Figure 1.4 illustrates the outline of this dissertation. The conceptual sensor in the illustration shows multiple fluid sensors integrated inline on a single chip: two pressure sensors, a mass flow sensor, density sensor and relative permittivity sensor. This conceptual sensor is described, first by reviewing what has been done, then by explaining the general fabrication and experimental methods and finally by going in detail about the theory and experiments of the individual sensing principles.

Chapter 2 acts as an introduction to microelectromechanical fluid sensors, i.e. pressure sensors, flow sensors, density sensors and viscosity sensors. It includes the basic physics behind each of the fluid sensing principles and briefly reviews earlier published work on the subject.

Chapter 3 describes the general methods used for the fabrication and readout of the sensors in this dissertation. The chapter starts with a detailed overview of the fabrication methods for microchannels. It continues with a section about actuation of mechanical resonators, since some of the fluid sensors in this dissertation need to be operated at their resonance frequency. Sensors have an output signal which needs to be interpreted; the next section therefore continues with two measurement methods for microfabricated sensors. The chapter ends with interfacing systems for these type of sensors.

Chapter 4 elaborates on the first individual sensor of the integrated sensor chip from Figure 1.4: the Coriolis mass flow sensor. This sensor, consisting of a suspended microchannel, is mechanically actuated at its resonance frequency. A mass flow changes the ratio between magnitudes of the mode shapes of the sensor, which is optically or capacitively detected. The first section of this chapter describes a method to improve the resolution of the integrated capacitive detection. Then, in the second section, the fundamental limits on the resolution due to thermomechanical noise are theoretically and experimentally analyzed. The chapter ends with an optical detection principle for mode analysis of white noise actuated microstructures.

Chapter 5 introduces pressure sensing mechanisms that are compatible with the fabrication technologies of Chapter 3. One of the sensors can be integrated in the Coriolis mass flow sensor and does therefore not require any extra chip space. The other sensor has a resistive readout and can be integrated with other resistive or capacitive sensors on the same chip with minimum risks on crosstalk.

Chapter 6 goes into detail about multi fluid parameter sensing using the sensors described in Chapters 4 and 5. The density can be measured directly from the Coriolis mass flow sensor, but needs to be calibrated. A model and experimental results are presented for both liquids and gases. For measuring viscosity, both the mass flow and pressure drop need to be sensed. Models for liquids and gases are explained and validated by measurements. The chapter ends with the introduction of a relative permittivity sensor. Although this is not a mechanical fluid sensor, its measured quantity is relevant for fluid composition measurements.

Figure 1.4: Illustration of the dissertation outline. Chapter 2 acts as an introduction to microelectromechanical fluid sensors, i.e. pressure sensors, flow sensors, density sensors and viscosity sensors. Chapter 3 describes the general methods used for the fabrication and readout of the sensors in this dissertation. Chapter 4 elaborates on the resolution limits and optimization of micro Coriolis mass flow sensors. Chapter 5 introduces pressure sensing mechanisms that are compatible with the fabrication technologies of Chapter 3. Chapter 6 goes into detail about multi fluid parameter sensing using the sensors described in Chapters 4 and 5.

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2

Theory and review

This chapter¹ provides an overview of earlier published mechanical microfluidic sensors, i.e. flow, pressure, density and viscosity sensors. Besides reviewing, this chapter also explains the basic physics concerning these types of microfluidic sensors.

In Section 2.2, multiple pressure sensors with a mechanical transduction principle are discussed. Most sensors are designed for pressure sensing outside the chip, i.e. the sensors cannot be integrated with other microfluidic devices on a single chip.

Section 2.3 discusses five types of mechanical flow transduction principles: drag-based, differential pressure, Coriolis, vortex and ultrasonic flow sensors. Especially the discussed Coriolis mass flow sensors operate in a throughflow configuration and there is potential for integrating these sensors with other microfluidic devices on a single chip.

Sections 2.4 and 2.5 contain brief reviews on density and viscosity sensing respectively. Only a few of the discussed sensors can be integrated throughflow with other microfluidic devices.

¹This chapter is based on the publications [1–3]:

- D. Alveringh, R. G. P. Sanders, R. J. Wiegerink, and J. C. Lötters, “Vortex generation and sensing in microfabricated surface channels,” in *Proceedings of the 29th IEEE International Conference on Micro Electro Mechanical Systems (MEMS 2016)*. Shanghai, China: IEEE, 2016, pp. 812–815;
- D. Alveringh, R. J. Wiegerink, J. Groenesteijn, R. G. P. Sanders, and J. C. Lötters, “Experimental analysis of thermomechanical noise in Coriolis mass flow sensors,” *Sensors and actuators A: Physical*, vol. 271, pp. 212–216, 2018;
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2.1 Introduction

Microfluidic sensors translate a physical fluidic quantity to an interpretable signal. Mechanical microfluidic sensors, e.g. pressure sensors and specific flow sensors, use a mechanical transduction principle to sense a fluidic quantity. The output mechanical quantity, usually displacement, can be detected optically or electrically. Figure 2.1 shows a schematic example of a mechanical microfluidic sensor.

Figure 2.1: Schematic example of an electromechanical pressure sensor. The pressure deforms a spring, the displacement of the spring changes the resistance of a potentiometer, and thus the output voltage; the output voltage is subsequently a measure for the pressure.

As mentioned in Chapter 1, the research in this dissertation, and thus the review in this chapter, is limited to mechanical microfluidic sensors that can be operated in a throughflow configuration and can be integrated with other microfluidic devices. Only sensors for mechanical fluid quantities are discussed:

- quantities that specify the state of the fluid, e.g. pressure and flow;
- properties of the fluid, e.g. density and viscosity.

2.2 Mechanical pressure transduction principles

Pressure is the effort variable in the fluid domain and is therefore equivalent to electrical voltage, mechanical force and torque. Physically speaking, pressure P can be seen as the total perpendicular force on a surface area:

$$P = \frac{F_A}{A}, \quad (2.1)$$

with F_A the total force caused by pressure on surface area A . The concept of pressure is generally used as a measure of the forces of fluids on the environment due to e.g. Brownian motion and gravity. In theory, the pressure is in this case only caused by the fluid at one side of the surface and is called absolute pressure. In practice however, this is only true when the other side of the surface is vacuum. Pressures are usually measured between two fluids applying a force at both sides of the surface, i.e. the differential pressure. When the pressure is measured compared to atmospheric pressure (which is approximately $1 \cdot 10^5$ Pa), it is called gauge pressure [4]. The gauge pressure is therefore approximately $1 \cdot 10^5$ Pa or 1 bar lower than the absolute pressure. All pressures in this dissertation are differential pressures or gauge pressures and are expressed in the unit bar, with $1 \text{ bar} = 1 \cdot 10^5$ Pa.

A straightforward method to measure force, caused by pressure, is by applying it to a spring as illustrated in Figure 2.1 a spring translates the force into a displacement x :

$$x = \frac{F_A}{c} = \frac{P \cdot A}{c}, \quad (2.2)$$

with c the stiffness of the spring. When it comes to microfabricated pressure sensors, most use this transduction principle. The springs in these structures are based on deforming membranes, as reviewed by Eaton et al. [5]

Since this review, improvements have been achieved, especially in the performance of the capacitive structures. The use and optimization of interdigitated electrodes instead of parallel plates can contribute to a higher linear response [6]. Another improvement in linearity and robustness can be achieved by making the capacitive plates touch each other in the center with an insulator in between [7, 8]; the contact area of the electrodes increases with increasing pressure. By decreasing the distance between the electrodes, the sensitivity can be increased [9]. Figure 2.2 shows illustrations of common pressure sensing structures.

Another step forward in pressure sensing is based on the compatibility with complementary metal oxide semiconductor (CMOS) fabrication processes. The CMOS process is the most common method for the fabrication of analog and digital integrated electronic circuits. Integration of the sensor with the electronics on a single-chip has advantages in noise reductions, packaging and low-cost mass fabrication [10, 11].

Membrane-based pressure sensor measures differentially, i.e. the sensing mem-

Figure 2.2: Illustrations of cross-sections of common implementations of microfabricated pressure sensors. With (a) a pressure sensor with capacitive readout, (b) a differential pressure sensor, (c) a touch mode pressure sensor and (d) a pressure sensor with piezoresistive readout.

brane has always two sides on which pressures act on. The pressure in the cavity can be seen as a reference pressure. By sealing the cavity hermetically under vacuum, the reference pressure is constant and controlled [12–14]. Besides, the sensor response is directly related to absolute pressure.

Above pressure sensors all use a silicon, ceramic or metal membrane for pressure sensing. Polymers have generally a lower Young's modulus and may be therefore an adequate membrane material. Disadvantages of polymers are hysteresis and creep. Polydimethylsiloxane (PDMS) is a widely used microfluidic device material and can be used for this purpose [15].

There are also other methods to measure pressure. Pirani gauges, for example, measure the heat flux between a heater and a heat sink, as a measure for the pressure [16]. Or, a spinning rotor gauge measures the pressure by finding the amount a spinning ball is slowed down due to the fluid around it [17]. Latter sensors are generally used for vacuum applications and have no implementations inside microchannels.

Although these sensors are designed to measure the pressure of the environment, some could be used in an inverted way, with the sensing fluid through a channel and the reference pressure outside the sensor. Still, throughflow pressure sensors that can be integrated with other microfluidic structures have not been presented to the best of the author's knowledge.

2.3 Mechanical flow transduction principles

In fluidic systems, the volumetric flow Q is the volume V passing per unit time t through the system. It is equivalent to current and velocity for electrical and mechanical systems respectively. The volumetric flow Q is equal to the flow profile $\vec{u}$ integrated over a surface area $\vec{A}$:

$$Q = \frac{dV}{dt} = \iint_A \vec{u} \cdot d\vec{A}. \quad (2.3)$$

For ideal linear time invariant systems with incompressible fluids, pressures and volumetric flows can be simply modeled using lumped element models. The sum of all volumetric flows needs to be zero from and to a system. However, for compressible fluids, i.e. a fluid with a variable density, this is typically not the case. The mass flow Φ is equal to the mass m passing per unit time t through the system:

$$\Phi = \frac{dm}{dt} = \rho \frac{dV}{dt} = \rho Q, \quad (2.4)$$

and obeys the law of conservation of mass. Thus, also for compressible fluids, the sum of the mass flows needs to be zero from and to a system. In this dissertation, liquids are considered incompressible and gases are considered compressible.

Microfabricated flow sensors have been developed for decades, started by van Putten et al. in 1974 with the first microfabricated thermal flow sensor [18]. This section only concerns mechanical flow sensors, i.e. drag-based, differential pressure, Coriolis, vortex and ultrasonic flow sensors.

2.3.1 Drag-based flow sensors

Drag-based flow sensors consist of one or more deformable obstacles (mostly cantilever beams or hair-like structures) in a fluid channel. The deformation of the beams can be measured in several ways: there are drag-based flow sensors with piezoelectric transducers, optical or capacitive read out. Since some of these readout principles are passive, these type of sensors have mostly lower power consumption than other flow sensing principles.

One of the first microfabricated drag-based flow sensor is proposed in [19] by Gass et al. The sensors obstacle is a cantilever beam (Figure 2.3a), formed by through-wafer etching. Piezoresistors were patterned and diffused in the chip for the electrical readout. Other microfabricated drag-based flow sensors can be integrated with CMOS on chip [20] or use a capacitive readout [21] or optical readout [22–24] (Figure 2.3b). These sensors are all placed perpendicular to the flow. There are also techniques to fabricate bended cantilevers on top of the chip to enable the placement of the chip parallel to the flow (Figure 2.3c) [25–27]. Polymer obstacles also exist [28, 29], like SU-8 [30] or polydimethylsiloxane (PDMS) [31].

Figure 2.3: Different concepts of drag-based flow sensors, with (a) a sensor with the chip perpendicular to the flow with piezoresistive readout, (b) optical readout (c) a sensor with the chip parallel to the flow and (d) an artificial hair flow sensor.

In contrast with drag force, the lift force is perpendicular to the flow direction. Svedin et al. proposed a lift based flow sensor [32]. This sensor consists of a cantilever beam with piezoresistors points parallel to the flow.

Artificial hair flow sensors (Figure 2.3d) are inspired on specific hair-like sensory systems of animals. The artificial hairs are most of the time relatively long (up to 1 mm) and it is common to integrate multiple hairs on one chip. One of the first artificial hair flow sensor is designed by Ozaki et al. using a piezoresistive readout. Their design is inspired on the work of Gnatzy et al. in 1980, who characterized mechanical properties of the sensory hairs of gryllus. Krijnen et al. developed a flow sensor with microfabricated SU-8 hairs with a capacitive readout [33–35]. The field of artificial hair sensors is a specific field of flow sensors, not only due to the complicated fabrication technologies but also because of the various measurement strategies. The review papers of Nawi [36] and Tao [37] give a more detailed overview of the origin, development and technology of this type of sensor.

No drag-based flow sensors that can be integrated in a microchannel have been reported. However, the sensors placed parallel to the flow, i.e. with structures perpendicular to the flow, have potential to be integrated in a microchannel. This could be achieved by bonding a wafer with a microchannel on top of the sensor.

2.3.2 Differential pressure flow sensors

Fundamentally, drag-based flow sensor and differential pressure flow sensors are not very different: there is a deforming structure as a result of flow. However, with differential pressure sensors, the channel in which the flow is present is defined (Figure 2.4). This means that the pressure on the deforming structure is measured and can be used as a measure for flow.

Figure 2.4: Differential pressure flow sensors consist generally of a channel with a defined fluidic resistance and two pressure sensors. The pressure drop over the channel is a measure for the flow. Each infinitesimal volume of the fluid in the parabolic flow profile undergoes pressure and viscous forces.

A generic model for a fluid flow through a circular channel as a function of pressure can be derived using the following force equilibrium, as indicated in Figure 2.4:

$$F_P - F_v = 0, \quad (2.5)$$

with F_P the force on the fluid as a result of a pressure difference ΔP on a surface area A_i and F_v a force caused by the viscous drag of the fluid in the other direction. The force acting on the fluid as a result of the pressure over a cylinder with radius r is:

$$F_P = A_i \Delta P = \pi r^2 \Delta P. \quad (2.6)$$

In the channel, the fluid has a flow profile as a function of the distance from the center. The fluid has a higher velocity in the center than at the edges, the fluid velocity u decreases in radial direction r . For viscous fluids, explained in Section 2.5, a force is needed to tear the layers of fluid apart. For circular channels, the surface area is equal to:

$$F_v = -A_c \eta \frac{du}{dr} = -2\pi r L_t \eta \frac{du}{dr}, \quad (2.7)$$

with A_c the surface area of the cylinder, equal to $2\pi r L_t$, L_t the length of the channel, η the dynamic viscosity, u the flow velocity and r the distance from the center of the channel in radial direction. Substitution in Equation 2.5 gives:

$$\pi r^2 \Delta P + 2\pi r L_t \eta \frac{du}{dr} = 0. \quad (2.8)$$

Or:

$$\frac{du}{dr} = -\frac{r\Delta P}{2L_t\eta}. \quad (2.9)$$

When a no-slip boundary condition is assumed, there is no fluid flow in the outer lamina at R :

$$u = 0|_{r=R}. \quad (2.10)$$

So, in integral form with above boundary condition:

$$\int_u^0 du = -\frac{\Delta P}{2L_t\eta} \int_r^R r dr, \quad (2.11)$$

$$u = \frac{\Delta P}{4L_t\eta}(R^2 - r^2). \quad (2.12)$$

Now, from equation 2.3, the volume flow Q is:

$$Q = \iint_A u dA = \int_R^0 \frac{\Delta P}{4L_t\eta}(R^2 - r^2)2\pi r dr. \quad (2.13)$$

Performing the integral leads to the Hagen-Poiseuille law and describes a linear relation between volume flow Q and pressure drop ΔP .

$$Q = \frac{\pi\Delta PR^4}{8\eta L_t}. \quad (2.14)$$

The ratio $(8\eta L_t)/(\pi R^4)$, consisting of channel and fluid parameters, could be interpreted as a fluidic resistance, parallel to electrical resistance or mechanical damping.

Differential pressure flow sensors can simply consist of a single differential pressure sensor in a channel. An example is presented by Cho et al. [38]. This sensor consists of a pressure sensor that measures the pressure differentially between the fluid outside the chip and in a microchannel inside the chip. The pressure sensor can also be integrated together with the microchannel in the form of an orifice [39] or as an obstacle in the center [40, 41]. A specific implementation of a differential pressure flow sensor with a single sensor is the Prandtl tube [42, 43], a variant on the Pitot tube. This flow sensor consists of a channel pointing towards the flow. At the end of the tube, the fluid flow stagnates which results in a pressure on the pressure sensor.

The differential pressure can also be measured using two absolute or gauge pressure sensors with a channel in between [44–46]. Also more than two pressure sensors can be integrated in a microfluidic channel to sense other fluid properties, e.g. relative permittivity [47].

2.3.3 Coriolis mass flow sensors

Measurement of mass flow has been heavily influenced by the development of Coriolis mass flow sensors [48, 49]. The operating principle of these sensors is straightforward: a mass flow through a vibrating channel induces distributed Coriolis forces. As a result, a second vibration mode is excited with its amplitude proportional to the mass flow. Therefore, Coriolis mass flow sensors are able to measure true mass flow and are independent of other fluidic parameters. A common implementation of a Coriolis mass flow sensor is shown in Figure 2.5. In this implementation, the channel vibrates in the twist mode and due to the Coriolis forces, the channel starts to vibrate in the swing mode as well. The ratio of amplitudes between these vibration modes is a measure for the mass flow.

Figure 2.5: Coriolis mass flow sensor based on a rectangular channel shape. By actuating the twist mode with force $F_A(t)$ resulting in a torque $\Omega_T(t)$, a mass flow Φ induces a Coriolis force $F_C(t)$ (or $\Omega_S(t)$) causing the channel to move in the swing mode as well.

Figure 2.6 shows an illustration of a particle moving with a constant velocity v through a channel. As mentioned, the channel of the Coriolis mass flow sensor is actuated in a vibration mode, e.g. in a rotating movement with angular velocity Ω . This will force the particle to move in a curved line, as the channel wall constrains the particle. The particle has simply traveled distance r as a function of time t as a

Figure 2.6: Moving particle through a rotating channel. After time t , the particle traveled distance r along the channel and distance l in vertical direction.

result of velocity v observed from the rotating channel:

$$r = v \cdot t. \quad (2.15)$$

As a result of the rotating channel, the angle θ became the product of the angular velocity and the time t , as observed from a fixed reference frame:

$$\theta = \Omega \cdot t. \quad (2.16)$$

However, if the particle is observed from a fixed reference frame, the particle has also moved vertical distance l . This distance is equal to the arc at radius r for small angles:

$$l = r \sin(\theta) \approx r\theta = r\Omega t. \quad (2.17)$$

The change in radius during time t is described by Equation 2.15 and can be substituted in Equation 2.17. The vertical displacement dl therefore becomes:

$$l = v\Omega t^2. \quad (2.18)$$

This motion can be described by a vertical acceleration; the second derivative of Equation 2.18. From the fixed reference frame, this specific form of the Coriolis acceleration makes the particle follow the rotation of the channel:

$$a = \frac{d^2 l}{dt^2} = 2\Omega v. \quad (2.19)$$

For vibrating channels with a moving fluid, it appears that the channel applies a force in opposite direction to keep the particle in the channel. When the particle has mass m , the Coriolis acceleration can be written as Coriolis force using Newton's second law of motion:

$$F_C = -ma = -2m\Omega v, \quad (2.20)$$

or in its general form:

$$\vec{F}_C = -2m\vec{\Omega} \times \vec{v}. \quad (2.21)$$

This force will influence the dynamics of the channel suspension and could therefore change the mode shape of the vibration. When a fluid flows with velocity u a distance dx with a mass dm in a channel, the mass flow Φ related to flow velocity u is in that case:

$$\Phi = \frac{dm}{dt} = \frac{dm}{dx} \frac{dx}{dt} = \frac{dm}{dx} u \rightarrow u = \frac{dx}{dm} \Phi. \quad (2.22)$$

For a channel with length W and constant density, the fluid velocity simply becomes $W/m\Phi$ and can be substituted in Equation 2.21:

$$\vec{F}_C(t) = -2W \left(\vec{\Omega}(t) \times \vec{\Phi} \right). \quad (2.23)$$

When the Coriolis mass flow sensor is harmonically actuated at frequency ω_T , the amplitude of the Coriolis force $\hat{F}_C$ can be related to the displacement amplitude $\hat{z}_T$ at

either end of the channel segment experiencing the Coriolis force:

$$F_C(t) = -2W\Phi\Omega_T(t) \quad (2.24)$$

$$= -2W\Phi \frac{d}{dt}\theta_T(t) \quad (2.25)$$

$$= -2W\Phi \frac{d}{dt}\hat{\theta}_T \sin(\omega_T t), \quad (2.26)$$

$$\hat{F}_C = -2W\Phi \hat{\theta}_T \omega_T \approx 4\omega_T \hat{z}_T \Phi, \quad (2.27)$$

with θ_T the time t dependent angle of the channel due to actuation and $\hat{\theta}_T$ its amplitude. The Coriolis force results in a torque $\hat{\tau}_C$ given by:

$$\hat{\tau}_C = \hat{F}_C L \approx 4L\omega_T \hat{z}_T \Phi, \quad (2.28)$$

with L the length as indicated in Figure 2.5.

Coriolis mass flow sensors have been used for flow measurements for decades and come in many different sizes and shapes [49]. The first microfabricated Coriolis mass flow sensor was published by Enoksson et al. [50, 51]. This sensor is fabricated by first etching two halves of the channel in two silicon wafers. Then, the wafers are bonded to form the channel structure. Finally, the channels are released using wet etching. The sensor is electrostatically actuated using an external electrode. The readout is performed optically using a laser and a two dimensional photo detector.

A few years later, another development of a microfabricated Coriolis mass flow sensor started [52–54]. The first steps of the fabrication process of this sensor is similar to the work of Enoksson et al. The channels are etched in a silicon wafer, the wafer is bonded to another silicon wafer. After this, the channels are released. This structure is then bonded to a glass wafer. The glass wafer has wafer-through etched inlets and metal electrodes and wiring. The sensor needs a vacuum environment to reduce air damping. This is achieved by sealing the sensor in a package with a getter material.

A third line of micro Coriolis mass flow sensors is based on surface channel technology, published in 2007 [55] and comprehensively described in Chapters 3. This technology is used by Haneveld et al. to fabricate a Coriolis mass flow sensor with external optical readout [56] and later on with integrated capacitive read out [57]. This sensor is also integrated with thermal flow sensors to increase the range and simplify calibration [58, 59]. Many improvements have been made in the years after that by Groenesteijn et al. They optimized the mechanics and actuation of the sensor by modeling [60], Lorentz actuation [61] and parametric excitation [62]. Also research has been done on micro bypasses for pressure drop reduction [63] and resolution improvements [64]. The fabrication method has also been developed further [65, 66], with support for proportional valves [67] and other microfluidic structures. An extensive overview can be found in [68].

Finally, a fourth line of micro Coriolis mass flow sensors is presented in 2017 by Monge et al. [69] This sensor is unique, since the channel is completely made of a polymer (SU-8). It has, due to its short and cost-effective fabrication process, potential to become a one-time use disposable sensor for medical applications.

Coriolis mass flow sensors are inherently throughflow devices and have therefore potential for integration with other microfluidic devices.

2.3.4 Vortex flow sensors

Vortex flow sensors consist of a channel with a bluff body and a pressure sensing element, as illustrated in Figure 2.7. The bluff body changes the laminar flow to a turbulent flow. The vortices of the turbulent flow cause an alternating pressure at the position of the pressure sensing element. The frequency of this pressure is dependent on the volume flow in the channel.

Figure 2.7: A bluff body in a channel may induce vortices in the flow. The frequency, detected by a sensing element, is a measure for the flow.

Whether or not turbulence occurs in the channel can be estimated by the dimensionless Reynolds number Re , defined by:

$$Re = \frac{u L_c \rho}{\eta}, \quad (2.29)$$

with u the flow velocity, L_c the characteristic length, ρ the density and η the dynamic viscosity of the fluid. As a rule of thumb, flow profiles with Reynolds numbers lower than 2300 are laminar and higher than 2300 are turbulent [70]. However, vortices may occur in specific cases with lower Reynolds numbers. Turbulence in microchannels is not common, since the characteristic lengths are small. Pedersen et al. presented in 2003 the first semi-MEMS vortex flow sensor, i.e. a microfabricated sensor in a conventional channel [71]. The pressure sensing element in the vortex flow sensor consists of a microfabricated piezoresistive membrane. The housing of the membrane has two ports at both sides to measure the alternating pressure. In 2009, Kim et al. proposed a sensor that has a flow dependent frequency readout [72]. Their sensor consists of a cantilever beam with piezoelectric material. Since the cantilever beam acts as a bluff body, turbulence behind the beam will occur and this makes the beam vibrate. The flow also causes a drag force on the beam, which increases the beam's

stiffness and therefore its resonance frequency. In 2010, Zylka et al. proposed a vortex flow sensor based on a silicon cantilever beam [73]. This beam is placed in the center of a pipe, mounted on a trapezoidal holder that also acts as the bluff body. A piezoresistive strain gauge is attached to the cantilever beam, which converts the fluidic vortices to an alternating voltage. Ju et al. used turbulence induced vibration for flow sensing in a different way in 2011 [74]. They fabricated an optical fiber inside a microchannel. The fiber vibrates due to the vortices and acts as optical readout. The channels are made in a glass wafer. A glass cover is fusion bonded on the channel wafer, then, the fiber is inserted in the channel.

In 2016, Alveringh et al. worked on a vortex flow sensor integrated in a microchannel. The structure consists of a silicon nitride supply channel, two injectors and a vortex channel. Figure 2.8 shows a SEM image of the device. The characteristic length is decreased gradually in the injector and increased abruptly from injector to vortex channel. The Reynolds number Re inside the injectors is therefore estimated between 200 and 420, which can be enough for vortex shedding [75]. Furthermore, the flows from both injectors interfere with each other. A rough approximation for the vortex shedding frequency can be found using the Strouhal number St , which is approximately 0.2 for Reynolds numbers in the order of $1 \cdot 10^2$ [75]:

$$St = \frac{f L_c}{U} = 0.2 \rightarrow f \approx 10^4 \dots 10^5 \text{ Hz},$$

with f the vortex shedding frequency, L_c the characteristic length of the vortex channel.

Figure 2.8: SEM picture of the vortex flow sensor of Alveringh et al. The fluid path causes vortices in the channel, which deform the membrane on top of the channel. These deformations can be measured optically.

The measurement results, obtained with laser Doppler vibrometry, are plotted in Figure 2.9 for a flow range from approximately 0.8 g h^{-1} to 1.3 g h^{-1} . Flows outside this range did not result in vibrations of the channel ceiling.

Figure 2.9: Measurement results of the vortex flow sensor from Alveringh et al for different pressures.

Vortex flow sensors can operate throughflow and can be integrated with other microfluidic devices on a single chip. However, a minimum flow is needed to start the vortex shedding. Besides, a better understanding and modeling of this type of sensors is needed.

2.3.5 Ultrasonic flow sensors

Ultrasonic flow sensors induce and measure acoustic vibrations in a fluid to measure flow velocity, as illustrated in Figure 2.10. They can therefore be seen as mechanical flow sensors. A simple ultrasonic flow sensor consists of two ultrasonic transducers: one sends the acoustic waves into the channel and the other receives them. The speed of the traveling acoustic wave through the medium is dependent on the fluid flow when observed from a fixed reference frame; the time between sending and receiving the wave is a measure for the flow. For turbulent, multi-phase flows or suspensions, Doppler shifts occur and can also be used for flow measurements.

Realizing such a device using microtechnology might be challenging, since the distance the acoustic waves travel are short and so travel times will be small. Besides, the ultrasonic transducers need to be integrated in the microfluidic fabrication process and size, leading to little power and high resonance frequencies. Currently, there have not been any microfabricated ultrasonic flow sensors reported. However,

Figure 2.10: Ultrasonic transducers induce and measure acoustic vibrations in the channel. The time of flight of the acoustic waves is a measure for the flow.

the ultrasonic mixer from Jagannathan et al. is a microchannel with microfabricated ultrasonic transducers [76]. It consists of a glass substrate bonded to a PDMS cover with the channel. Zinc oxide is deposited between metal electrodes to form the ultrasonic transducer. The active mixer from Yang et al. works similar [77], but has a glass microchannel and a PZT-on-silicon transducer.

The first steps in integrating ultrasonic transducers in microchannels are taken, but a functional microfabricated ultrasonic flow sensor has not been presented yet. Figure 2.11 shows two structures that consist of silicon nitride channels with PZT and interdigital transducers on top. The rectangular ultrasonic transducers in Figure 2.11a could be used to induce acoustic waves in the fluid in the microchannel. A second transducer, or multiple transducers, could be used to measure the time of flight by phase detection. Figure 2.11b shows an implementation with circular transducers. The devices have not been characterized yet.

Figure 2.11: Two structures with piezoelectric transducers on top of a microfluidic channel. The structures might be useful for ultrasonic flow sensing. The channel is not visible in the SEM images but runs underneath the structures from the upper-left to lower-right corner.

2.4 Mechanical density sensing

The density ρ of a fluid, or the reciprocal of the specific volume v_{sp} , can be defined by:

$$\rho = \frac{1}{v_{sp}} = \frac{dm}{dV}, \quad (2.30)$$

with infinitesimal mass dm in an infinitesimal volume dV . However, this only holds at macroscopic level. When the volume dV is only a few molecules in size, the density is not a fixed quantity anymore, since molecules will enter and leave the volume constantly [78].

For a known volume, the density can be easily determined by measuring the mass. For microfabricated structures, mechanical resonators are often used [79]. A mechanical resonator has often the shape of a cantilever beam. The resonance frequency ω_0 is dependent on the stiffness c of the beam and the mass m :

$$\omega_0 \propto \sqrt{\frac{c}{m}}. \quad (2.31)$$

The mass of a substance could, for example, be measured by applying it to the cantilever beam, measure the resonance frequency and compensate for the mass of the beam (Figure 2.12). This approach is commonly used in sensing the mass of biological samples, like cells or biomolecules [79]. Many structures in the review of Johnson et al. need to be placed in a test solution or samples need to be attached to the sensing structure; there is no microchannel integrated in the resonator itself. Burg et al. presented a device in 2007 [80] that integrated a microchannel in a cantilever beam. This is called a suspended microchannel resonator. This resonator is electrostatically actuated. Other suspended microchannel resonator shapes are also presented, like the plate resonator of [81].

Figure 2.12: The resonance frequency of the cantilever beam structure (a) is dependent on the density of the fluid around it. A microchannel can be integrated in the resonator throughflow density sensing (b).

Since a Coriolis mass flow sensor is also a suspended microchannel resonator, it can be used for density measurements too [68, 82].

2.5 Mechanical viscosity sensing

The dynamic viscosity η is a measure for the resistance of a Newtonian fluid to shearing flows. A velocity gradient of the fluid du/dz results in a shear stress σ [4], proportional to the dynamic viscosity:

$$\sigma = \eta \frac{du}{dz}. \quad (2.32)$$

An example is shown in Figure 2.13, when two plates are moving relative to each other with a fluid in between, the force F_v on the plate will be:

$$F_v = -\eta A \frac{v}{H}, \quad (2.33)$$

with A the surface area of the plate, v the velocity difference between the plates and H the distance between the plates. The kinematic viscosity ν is by definition equal to the ratio of dynamic viscosity η and density ρ :

$$\nu = \frac{\eta}{\rho}. \quad (2.34)$$

Figure 2.13: The dynamic viscosity is a measure for the resistance of a fluid to shearing flows. When the upper plate moves with velocity v , the fluid's viscosity causes a force F_v in opposite direction.

A common way to measure viscosity is by measuring the viscous drag of the fluid on a mechanical resonator. One implementation is presented by Andrews et al. in 1995 [83]. The authors microfabricated a silicon spring suspended plate that is electrostatically actuated. By superimposing a high frequency signal upon the actuation signal, the movement can be capacitively detected. Also Lorentz actuation [84] and piezoelectric actuation is reported. The devices with latter actuation method vary from externally actuated and optically characterized cantilevers [85] to fully integrated sensor chips [86]. Combined density and viscosity measurements using a piezoelectric actuated cantilever is also reported. Wilson et al. use a mechanical model to obtain viscosity and density from the resonance frequency and damping of the cantilever beam [87]. The resonator does not always need to have the shape of a cantilever. A comb-drive that is electrostatically actuated for viscosity measurements of gases has also been presented [88]. The pull-in time of this structure is dependent

on the viscosity of the gas in between.

Although most literature describes mechanical resonators for sensing viscosity, there are different sensing principles. Jakoby et al. worked on a sensor that consists of an interdigital transducer on a piezoelectric substrate [89]. The structure generates a love wave that propagates at the surface of the substrate. The decay of this love wave is dependent on the viscosity of the fluid around the device, which is detected with a second interdigital transducer. Van Baar et al. used a resistor array inside a channel for sensing viscosity [90]. The resistor array can be used for thermal flow sensing, but also separates the flow. The flow profile needs to develop again after separation. The resistor array measures the entrance length, which is a measure for the viscosity.

Another different sensing method is based on a differential pressure flow sensor [91]. The pressure sensors in this mass flow sensors consist of flexible channels that expands under pressure. The expansion causes an increase of volume and so a change in relative permittivity. This change is capacitively detected. The authors measure the time it takes to fill the sensor and use a model based on Hagen-Poiseuille law to obtain the viscosity.

2.6 Concluding remarks

- Microfabricated pressure sensors belong to the oldest and most developed microfabricated fluid sensors. Nevertheless, not many throughflow sensors or sensors that can be integrated with other microfluidic devices have been reported.
- Five types of mechanical microfabricated flow sensors have been discussed in this chapter. Especially Coriolis mass flow sensors operate throughflow and there is potential for integrating these sensors with other microfluidic devices on a single chip. Drag-based flow sensors and differential pressure flow sensors are usually not integrated in a microchannel, but need to be placed in a larger conventional channel. Not much research has been done on microfabricated vortex and ultrasonic flow sensors.
- Mechanical density and viscosity sensors generally consist of resonating cantilever beams. The density and viscosity can be obtained from the resonance frequency and the damping respectively. Some sensors report on an integrated microchannel in the beam, enabling throughflow density sensing.
- Table 2.1 shows an overview of the technologies on throughflow operation, integratability, readiness and robustness. Based on the review, Coriolis mass flow sensors need a comprehensive microfluidic fabrication process. It is possible to design pressure sensors in the same technology as will be described in Chapter 5. It also enables density and viscosity sensing using these devices by using a physical fluid model as is described in Chapter 6.

Table 2.1: Qualitative comparison of microfabricated pressure sensing, flow sensing, density sensing and viscosity sensing technologies on throughflow operation, integratability and readiness. Integratability is estimated by how universal the fabrication technology is for integrating different microfluidic devices on the same chip. Readiness is estimated by the age and the amount of research done on the technology.

	Microfabricated							
	Flow sensors							
	Pressure sensors	Drag-based	Differential pressure	Coriolis	Vortex	Ultrasonic	Density sensors	Viscosity sensors
Throughflow	–	–	–	++	+	+	+	o
Integratability	+	o	+	++	+	+	+	o
Readiness	++	o	+	o	–	–	o	–
Robustness	++	–	++	+	o	++	+	+

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3

Fabrication and characterization methods

This chapter¹ describes all methods that are used in the rest of this dissertation for experiments. First, in section 3.2 multiple fabrication methods for microchannels are discussed. These microchannels can be released and have metal electrodes and wiring on top for actuation and readout. Therefore, these microchannels can be used for micro Coriolis mass flow sensors reviewed in Subsection 2.3.3.

After fabrication, active structures (e.g. mechanical resonators) need to be actuated when characterized. Section 3.3 explains different methods to actuate resonators at their resonance frequency.

If the fabricated microstructure is a sensor, the output needs to be read out. One method is optically using laser Doppler vibrometry, explained in Section 3.4. Another method is using capacitive sensing structures, explained in Section 3.5.

In Section 3.6, practical implementations for the electric and fluidic interfacing to the chip are discussed.

3

¹This chapter is based on the publications [1–4]:

- D. Alveringh, R. G. P. Sanders, R. J. Wiegerink, and J. C. Lötters, “Phase relation recovery for scanning laser Doppler vibrometry,” *Measurement Science and Technology*, vol. 28, no. 2, p. 025208, 2017;
- D. Alveringh, T. V. P. Schut, R. J. Wiegerink, and J. C. Lötters, “Coriolis mass flow and density sensor actuation using a phase-locked loop,” in *Proceedings of the 3rd Conference on MicroFluidic Handling Systems (MFHS 2017)*, 2017, pp. 102–105;
- D. Alveringh, R. G. P. Sanders, J. Groenesteijn, T. S. J. Lammerink, R. J. Wiegerink, and J. C. Lötters, “Universal modular fluidic and electronic interfacing platform for microfluidic devices,” in *Proceedings of the 3rd Conference on MicroFluidic Handling Systems (MFHS 2017)*, Enschede, the Netherlands, 2017, pp. 106–109,
- D. Alveringh, R. J. Wiegerink, and J. C. Lötters, “Inline relative permittivity sensing using silicon electrodes realized in surface channel technology,” in *Proceedings of the 31th IEEE International Conference on Micro Electro Mechanical Systems (MEMS 2018)*. Belfast, United Kingdom: IEEE, 2018, pp. 840–843.

3.1 Introduction

The realization of microfluidic sensors is roughly divided in two stages:

- fabrication of the chip using microtechnology;
- fluidic and electric interfacing of the chip to characterization equipment.

The publications reviewed in Chapter 2 described almost as many different sensors as fabrication processes. For microfluidic sensors, it is very common to develop a specific fabrication process for each sensor. Besides, custom fluidic and electric interfacing methods including readout electronics are usually designed for the specific sensor. Thus, one cannot fall back on conventional fabrication and interfacing methods used for integrated circuits.

All microfluidic sensors presented in this dissertation, use the same fabrication and interfacing methods described in this chapter.

3.2 Fabrication of microchannels

Surface channel technology is the title of a family of microfabrication technologies for the realization of semi-circular microfabricated silicon rich silicon nitride channels [5–7]. The channels are realized inside a silicon wafer with, contrary to what the name suggests, bulk micromachining. Nevertheless, the channels are directly connected to the surface of the wafer, in contrast with buried channel technology [8, 9]. Surface channel technology supports channels with diameters of approximately $10\text{ }\mu\text{m}$ to $100\text{ }\mu\text{m}$. Besides, wide shallow channels, up to $500\text{ }\mu\text{m}$, can also be fabricated. There are multiple versions of surface channel technology; the most recent version is explained first. Then, the conventional surface channel technology and future variants are presented. Figure 3.1 illustrates how a mask design translates to fabricated suspended microchannels with metal wiring.

Figure 3.1: Microchannel fabrication using surface channel technology, with (a) a mask design of a suspended channel with metal wiring, (b) isometric view of the structure and (c) SEM image of the fabricated structure.

3.2.1 Silicon-on-insulator-based surface channel technology

The silicon-on-insulator-based surface channel technology process starts with the realization of inlets and outlets in the backside of a silicon-on-insulator wafer [6, 7]. Then, channels are made in the device layer of the wafer. After this, metal structures are deposited and patterned for electronic readout. The process ends with releasing the channels from the silicon. Figure 3.2 shows a summarizing overview of the fabrication steps. Figure 3.3 shows three examples of structures that can be realized using this technology. The full fabrication process with details of every step, including lithography, is described Appendix A.

Figure 3.2: Illustration of the fabrication steps of silicon-on-insulator-based surface channel technology in isometric view. The fabrication starts by applying a silicon nitride layer (a-b) and anisotropically etching inlets and outlets in the handle layer (c). By etching isotropically in silicon through slits in a silicon nitride layer, microchannels are fabricated (d-f). The channel walls are realized by applying an extra silicon nitride layer (g). Metal is deposited for wiring (i-j). An isotropic silicon etch releases the channel from the silicon and enables the fabrication of isolated silicon electrodes(k-l).

Figure 3.3: Examples of structures that can be realized using silicon-on-insulator-based surface channel technology, with (a) a suspended channel with metal wiring, (b) isolated silicon electrodes at both sides of the channel and (c) crossing metal conductors.

Substrate selection and hard mask deposition

The fabrication is done in a silicon-on-insulator wafer with thermal silicon oxide of approximately $5\text{ }\mu\text{m}$ (Figure 3.2a). The thermal silicon oxide forms as well the buried oxide layer as the bottom layer. Any residual stress in the thermal oxide is therefore at both sides of the handle layer and so bending of the wafer is limited. The handle layer is $400\text{ }\mu\text{m}$ and the device layer is $50\text{ }\mu\text{m}$ in thickness. Both are made of highly doped silicon to increase electrical conductance. Full details of the used substrates are described in Steps 1 and 2 in Appendix A.

With low pressure chemical vapor deposition, a silicon rich silicon nitride (SiRN) layer of $1\text{ }\mu\text{m}$ is deposited (Figure 3.2b). The deposition is based on the chemical reaction between ammonia (NH_3) and dichlorosilane (SiH_2Cl_2) [10]. The ratio of the gases are tuned to form a low-stress silicon rich silicon nitride layer of $< 50\text{ MPa}$. Details of the recipe are listed in Step 5 in Appendix A.

This layer will be used as hard mask during the process, but also forms partly the ceiling of the channels.

Inlets and outlets etching

Inlets and outlets are realized at the bottom of the wafer (Figure 3.2c). First by reactive ion etching (RIE) through the silicon nitride layer and the silicon oxide layer. The recipe consists of a mixture of argon (Ar) and fluoroform (CHF_3) with a high capacitively coupled plasma power. This partly physical etching recipe therefore results in a low selectivity and high directionality. Because of its low selectivity, a

thick layer of photoresist is used. This anisotropic unselective dry etching recipe is used later in the process too. Details of this etching recipe are given in Step 7 in Appendix A. A specific recipe for silicon oxide etching could be used when thinner resist is preferred, the recipe in Step 8 in Appendix A has a higher selectivity.

Then, inlets are formed through the silicon handle layer until the buried oxide layer. The used deep reactive ion etching recipe is based on the work of Jansen et al. [11, 12] and allows for high aspect ratio anisotropic etching through silicon with high selectivity. It is a Bosch process with an etching step using sulfur hexafluoride (SF_6) alternated with a deposition step using octafluorocyclobutane (C_4F_8). Because of its high selectivity, the etch stops at the buried oxide layer. Details of this deep reactive ion etch are given in Step 9 in Appendix A.

Channel etching and wall deposition

The channels are formed in the device layer of the wafer. This is done by isotropic plasma etching through slits in the silicon nitride. The slit pattern, with features of $5\text{ }\mu\text{m}$ by $1.5\text{ }\mu\text{m}$, needs to be transferred in the silicon nitride hard mask (Figure 3.2d) using the anisotropic unselective dry etching recipe described above. Because of the unselective nature of the etch, an extra hard mask of chromium is sputtered on the wafer.

After the slits are formed in the silicon nitride, channel molds are formed under the slit pattern (Figure 3.2e). This reactive ion etch (Step 14 in Appendix A) uses a recipe without capacitively coupled plasma with SF_6 and is therefore completely isotropic. The slit size, slit density and etching time defines the channel size.

After the channels are formed, the silicon oxide layer between the channels and the inlets can be removed. This can be done using 50 % of hydrogen fluoride (HF) in water (Figure 3.2f). The buried oxide layer can be used as extra layer of channels. This extra layer of channels is for example used for the valve in the integrated mass flow controller presented in [13]. All channel molds and connections to inlets are formed in this stadium of the fabrication process. A second deposition step of silicon nitride (approximately $1.5\text{ }\mu\text{m}$) is performed to form the channel walls and seal the slit pattern (Figure 3.2g).

Metal electrode deposition and etching

Before deposition of the metal layer on the wafer, pits need to be realized to allow for connections to the silicon device layer. The anisotropic unselective dry etching recipe is used for this (Figure 3.2h).

The metal wiring and electrodes are formed by sputtering first 15 nm of chromium (Cr) and then 200 nm of gold (Au) on top of the silicon nitride (Figure 3.2i). Patterning is done using reactive ion beam etching (RIBE) with Ar (Figure 3.2j), specified in Step 27 in Appendix A. Only wires are patterned in this etch, the comb-shaped electrodes are defined in the release etch.

Release etch

The final steps concern the releasement of the channels and the definition of the comb shape electrodes. First, an anisotropic unselective dry etching recipe (Step 30 in Appendix A) is used to etch through the metal layer and nitride. Then, a similar isotropic reactive ion etch recipe as for the channel formation is used to etch around the channels (Figure 3.2k). This etching recipe is performed in steps at low temperature to allow the released structures to cool down. Etching is done until the buried oxide layer is reached. Then, a vapor phase HF etch removes the buried oxide layer and another isotropic reactive ion etch is performed to provide more space for movement (Figure 3.2l) around the suspended channels.

Figure 3.4 shows four SEM images of structures fabricated with surface channel technology.

Figure 3.4: Scanning electron microscopy images of structures fabricated with surface channel technology, with (a) a suspended channel, (b) suspended comb-shaped electrodes, (c) channels in silicon and (d) cut channel.

3.2.2 Conventional surface channel technology

Chronologically, the conventional surface channel technology has been developed before the silicon-on-insulator-based surface channel technology [5], but after the buried channel technology [8, 9].

The structures are realized in a highly boron doped silicon wafer (Figure 3.5a). A deposition step of silicon nitride (approximately 500 nm) using LPCVD is performed (Figure 3.5b). Then, slits (5 μm by 2 μm) are etched using reactive ion etching in the silicon nitride (Figure 3.5c). With isotropic plasma etching, the channel structures are realized in the silicon (Figure 3.5d).

Figure 3.5: Illustration of the fabrication steps of conventional surface channel technology in isometric view. The fabrication starts by applying a silicon nitride layer (a-b). By etching isotropically in silicon through slits in a silicon nitride layer, microchannels are fabricated (c-d). Inlets and outlets are anisotropically etched from the bottom of the wafer (e). The channel walls are realized by applying an extra silicon nitride layer (f). Metal is deposited for wiring (g-h). An isotropic silicon etch releases the channel from the silicon and enables the fabrication of isolated silicon electrodes (i).

In contrast to the silicon-on-insulator-based surface channel technology, the inlets and outlets are etched with deep reactive ion etching after realizing the channels (Figure 3.5e). After this, a second LPCVD step (approximately 1 μm) to form the silicon nitride channels is performed (Figure 3.5f). The metal wires and electrodes are sputtered (15 nm of Cr and 200 nm of Au, Figure 3.5g) and patterned with reactive ion beam etching (Figure 3.5h). Then, a single isotropic etching step in the silicon releases the channels (Figure 3.5i).

3.2.3 Piezoelectric integration

Concepts for the integration of a piezoelectric material with surface channel technology are described in the dissertation of Groenesteijn [14]. Zeng et al. [15] presented the first device with this technology, a Coriolis mass flow sensor, in 2018. The steps for the integration of the piezoelectric material are directly after sealing the channels with silicon nitride, i.e. after the step in Figure 3.5f.

First, a seed layer (LaNiO_3) is deposited using pulsed laser deposition (Figure 3.7b). Then, also using pulsed laser deposition, the piezoelectric material lead zirconate titanate (PZT) is deposited (Figure 3.7c).

As electrode material on top of the PZT layer, platinum with titanium as adhesion layer are sputtered (Figures 3.7d–3.7e). The metal layer is patterned using reactive ion beam etching (Figure 3.7f). The PZT layer is patterned using reactive ion etching (Figure 3.7g).

Then, the process continues with the deposition and patterning of extra metal electrodes (Figure 3.7i) and release etching (Figure 3.7h). Figure 3.6 shows SEM images of the piezoelectric actuation structures on a Coriolis mass flow sensor.

Figure 3.6: SEM images of Coriolis mass flow sensor actuation structures, consisting of piezoelectric films on the suspended channels.

Figure 3.7: Extra steps needed for piezoelectric integration in surface channel technology. First, a seed layer is deposited (a-b). Then, piezoelectric material is deposited (c). As electrode material, platinum with titanium are deposited and patterned (d-f). Then, the piezoelectric material is etched (g). Extra metal electrodes are deposited and patterned (h) and the structure is released (i).

3.2.4 Multi level channel technology

Concepts for combining the buried channel technology from Tjerkstra et al. [8] and de Boer et al. [9] with the surface channel technology of Dijkstra et al. [5] are proposed in the dissertation of Groenesteijn et al. [14]. This multi level channel technology has not been realized yet, but the technical fabrication steps are implemented.

First, silicon nitride (500 nm) is deposited using LPCVD on a silicon wafer (Figure 3.9b). This silicon nitride layer functions as hard mask and as channel ceiling, similar to the other surface channel technologies. Then, an extra hard mask of silicon oxide (1 μm) is deposited using LPCVD with tetraethylorthosilicaat (TEOS). This layer acts

only as hard mask (Figure 3.9c).

The slit pattern, for the surface channels, is patterned in the silicon oxide layer, but not in the silicon nitride layer underneath (Figure 3.9d). Trenches, for the buried channels, are patterned in both layers (Figures 3.9e and 3.9f). With deep reactive ion etching, trenches are etched in the silicon wafer with high aspect ratio, of approximately $2\text{ }\mu\text{m}$ in width and $50\text{ }\mu\text{m}$ in depth (Figure 3.9g).

Then, using thermal oxidation, silicon oxide is grown on the sidewalls of the trenches (Figure 3.9h). This layer acts as a protection layer during the channel etch. Using reactive ion etching, the silicon oxide bottom of the trenches and the silicon nitride under the slits is etched (Figure 3.9i). At this moment, the silicon where the channels will be formed is not protected by any hard mask.

Using isotropic reactive ion etching, the buried channels and the surface channels are formed in one step (Figure 3.9j). Then, all silicon oxide is stripped using hydrogen fluoride (Figure 3.9k).

An extra thermal oxidation step is achieved, this narrows the silicon trenches (Figure 3.9l). The trenches need to be filled with silicon nitride during the ceiling step before the slits are sealed, otherwise there will be a leakage between buried and surface channels. Then, using a second LPCVD step, silicon nitride seals all buried and surface channels (Figure 3.9m). A release etch could be performed to release the structure from the silicon (Figures 3.9n and 3.9o).

Multiple levels of channels that can cross each other with or without being connected increase microfluidic design possibilities. A simple example of a mask design for which multiple layers of channels are essential is shown in Figure 3.8. This mixer separates the fluid at one side of the channel and merges it at the other side of the channel.

Figure 3.8: Example of a mask design for a mixer in multi level channel technology. The circular structures are high density slits and trenches, the surface and buried channels will connect here.

Figure 3.9: Illustration of the fabrication steps of multi level channel technology. The fabrication is based on surface and buried channel technology as explained in the text.

3.3 Actuation of microchannel resonators

Mechanical resonators, e.g. a Coriolis mass flow sensor, need to be externally actuated to move. A microchannel fabricated with a piezoelectric film (Subsection 3.2.3), can be actuated by simply applying an alternating voltage to the piezoelectric actuator elements. However, this technology has not been used for the devices in this dissertation. Another method is Lorentz actuation, for which only a metal track on the suspended channel and a magnetic field is needed.

3.3.1 Feed-forward Lorentz actuation

For Lorentz actuation, the channel is placed in a magnetic field. When an alternating current is applied through a metal track on the resonator, a Lorentz force acts on the channel segments that are perpendicularly placed in the magnetic field as indicated in Figure 3.10. The magnetic component of the Lorentz force $\vec{F}$ is defined by:

$$\vec{F} = q\vec{v} \times \vec{B}, \quad (3.1)$$

with q the charge, $\vec{v}$ the velocity of the charge and $\vec{B}$ the magnetic field. All charges q have a velocity v due to the current i in channel segment L , this means that Equation 3.1 can be rewritten to:

$$\vec{F} = i\vec{L} \times \vec{B}. \quad (3.2)$$

Or, when only considering the magnitudes of the vectors:

$$F = iLB, \quad (3.3)$$

By feeding a harmonic signal $\hat{u} \sin(\omega_0 t)$ to the metal track with resistance R , the force on the channel segments is:

$$F(t) = \frac{\hat{u} \sin(\omega_0 t) LB}{R}, \quad (3.4)$$

Figure 3.10: Illustration of a Coriolis mass flow sensor with Lorentz actuation. An alternating current through a metal track on the Coriolis mass flow sensor in a uniform magnetic field causes the suspended channel to vibrate in twist mode.

The actuated mode (twist mode) of the Coriolis mass flow sensor can be modeled as a second order system (Subsection 4.2.1). This means that it has a resonance frequency dependent on the modal stiffness and mass of the channel structure, including the fluid. In the first generation of actuation electronics, the frequency is manually tuned to this resonance frequency.

3.3.2 Actuation control using analog amplification

Since the frequency changes with the density of the fluid inside the channels, a feedback system is realized for the resonator. The resonator has an extra track on the channel, as illustrated in Figure 3.11. When moving in the magnetic field, this track generates an induction voltage. This induction voltage is amplified by two differential operational amplifiers and fed back to the actuation track. In this way, an electromechanical oscillator is realized, automatically driving at its mechanical resonance frequency. A gain control circuit is integrated at the final stage to control the output voltage. This makes this implementation for actuation control less dependent on the resistance of the tracks.

Figure 3.11: Schematic of feedback-based actuation for Coriolis mass flow sensors. A metal track on the Coriolis mass flow sensor in a magnetic field provides an induction voltage to an amplifier circuit. The amplifier circuit drives the actuation track on the Coriolis mass flow sensor. This forms an electromechanical oscillator, which actuates the Coriolis mass flow sensor at its mechanical resonance frequency.

3.3.3 Actuation control using a phase-locked loop

In spite of the convenience and performance improvements with the actuation control using analog amplifiers, there are still multiple drawbacks on this actuation method:

- a mechanical disturbance, e.g. vibration, of the Coriolis mass flow sensor will influence the actuation directly;
- a fluidic disturbance, e.g. air bubble, instantly changes the actuation frequency for a short time;
- the analog circuit amplifies the signal rather than synthesizes it.

Synthesizing the actuation signal in a controlled way can help overcome these drawbacks. This is done for high frequencies for telecommunication applications with a phase-locked loop [16]. Phase-locked loops are also used for controlled actuation of servo motors [17].

Theory

A basic phase-locked loop consists of three components as is illustrated in Figure 3.12.

Figure 3.12: Basic phase-locked loop consisting of a phase detector, a low-pass filter and a voltage controlled oscillator. The phase detector measures the phase difference between the output and input signal. The voltage controlled oscillator is directly tuned by this phase mismatch and synthesizes a harmonic signal that is synchronous with the input signal.

First, a phase detector finds the phase difference between the output signal $u_o(t)$ and the input signal $u_i(t)$. The input signal can be modeled as a harmonic signal with ω_i the frequency and ϕ_i the phase:

$$u_i(t) = \hat{u}_i \sin(\omega_i t + \phi_i). \quad (3.5)$$

The detected phase difference is then:

$$\phi_e = \phi_i - \phi_o, \quad (3.6)$$

which can be seen as the error that the phase-locked loop needs to solve. The second component is a low-pass filter. It filters the ripple at the output of the phase detector and high frequent disturbances from the input signal. The last component is a

voltage controlled oscillator. This oscillator synthesizes a periodic signal with a frequency dependent on the input. The voltage controlled oscillator always provides an output signal at a frequency, also when no input is given. This is called the quiescent frequency. The output frequency ω_o of the signal is:

$$\omega_o = \omega_0 + K \cdot \phi_e, \quad (3.7)$$

with ω_0 the quiescent frequency and K the sensitivity of the voltage controlled oscillator. The output signal is therefore equal to:

$$u_o(t) = \hat{u}_o \cdot \sin((\omega_0 + K \cdot \phi_e)t + \phi_o). \quad (3.8)$$

Note that the frequency of the output signal is corrected based on the phase difference between output and input signal. This means that not only the output frequency will approach the input frequency, but the phases will be synchronized as well.

Design

The phase-locked loop is realized using a Cypress Semiconductor PSoc 5 development kit and is based on the work of De Lima Fernandes [18]. This development kit has a programmable system on chip with digital and analog electronic components. Figure 3.13 shows an overview of the phase-locked loop implementation in the programmable system on chip.

Figure 3.13: Implementation of the phase-locked loop with the connections to the Coriolis mass flow sensor in a programmable system on chip.

The input signal is sampled by the programmable system on chip. An embedded analog comparator converts the signal to a square wave to make it compatible with digital electronics. An XOR-gate compares the square wave with the synthesized output of the phase-locked loop. This stage performs the phase detection: the XOR-

gate provides an high output at every sample when the square wave from the induction track is not equal to the synthesized actuation voltage. The duty cycle of the output signal is therefore dependent on the phase mismatch. The output of the XOR-gate is connected to a low-pass filter, which converts the pulses to an analog voltage. This first order low-pass filter is implemented using an embedded operational amplifier with an external capacitor and resistor, tuned for a cut off frequency of approximately 1 Hz.

The input of the voltage controlled oscillator is connected to a bias voltage generator to set the quiescent frequency. The bias voltage generator is realized using an embedded operational amplifier. The voltage controlled oscillator with output voltage $u_{vco,o}$ is implemented as an embedded current source i_{vco} that charges an external capacitor C :

$$u_{vco,o} = \frac{1}{C} \int i_{vco} dt = \frac{i_{vco} t}{C}. \quad (3.9)$$

An embedded comparator connects the capacitor to ground when the threshold, equal to the input of the voltage controlled oscillator $u_{vco,i}$, is reached:

$$u_{vco,o} = u_{vco,i}, \quad (3.10)$$

and so:

$$u_{vco,i} = \frac{i_{vco} t}{C} \rightarrow \frac{\omega_0}{2\pi} = \frac{1}{t} = \frac{u_{vco,i}}{C i_{vco}}. \quad (3.11)$$

An embedded flip-flop is used to force the duty cycle of the output signal to be 50 %. An embedded signal synthesizer is used to synthesize a sine wave for the actuation voltage of the Coriolis mass flow sensor.

Measurements

To test the phase-locked loop for Coriolis mass flow sensor actuation, multiple fluids are applied to the sensor and the frequencies are recorded using a Keysight 34461A multimeter. The experiments are conducted with nitrogen, water, propan-2-ol and a mix of propan-2-ol and water (equal volume). The results are plotted in Figure 3.14 and show that the actuation circuit adjusts the frequency to the resonance frequency of the Coriolis mass flow sensor.

The stability is investigated by finding the mean and standard deviation of 5001 measurements in approximately 20 ks for nitrogen. The mean is 2672.55 Hz and the standard deviation is 0.15 Hz, calculated from the data in Figure 3.15.

Figure 3.14: Measured resonance frequencies for four different substances.

Figure 3.15: Stability measurement (left) of 5001 samples (20 ks) for nitrogen with constant pressure and mass flow. The Fourier transform (magnitude only) is shown right, the result consists of $1/f$ noise and white noise.

3.4 Laser Doppler vibrometry

As indicated in previous sections, the structures in this dissertation are micro-sized and have resonance frequencies in the kilohertz range. The movement amplitudes are too small and the frequencies are too high to characterize with a regular optical microscope. Laser Doppler vibrometry is a method to measure the velocity at a small point defined by the spot size of the laser beam ($\sim 10\mu\text{m}$ for a Polytec MSA-400 [19]). It is based on the Doppler shift of a coherent light beam (laser) as a result of an out-of-plane moving target. By sampling the velocities in time, periodic movements can be made easily visible. Figure 3.16 shows an illustration of a basic laser Doppler vibrometry setup.

Figure 3.16: Illustration of a basic laser Doppler vibrometry setup. A laser is split into a measurement and a reference beam. A moving surface modulates the measurement beam, as a result of the Doppler shift. The reflecting beams are subtracted and fed into a detector.

Laser Doppler vibrometry is a non-invasive optical method to measure velocity profiles [20]. The technique has a wide variety of applications, ranging from modal testing of large structures [21] to microsystem analysis [22] and artwork diagnostics [23].

In a laser Doppler vibrometer, a laser beam is split in two using a beam splitter. One beam is pointed at the moving surface to be measured (measurement point), the other beam acts as a reference beam (reference point). The moving surface reflects a frequency modulated beam as a result of the Doppler shift.

$$\Delta f_{m|r} = 2 \frac{v_{m|r}}{\lambda}, \quad (3.12)$$

with $\Delta f_{m|r}$ the frequency shift of the measurement or reference beam, λ the wavelength of the laser and $v_{m|r}$ the velocity of the measurement point or the reference point. Both beams are optically combined and interfere with each other. The resulting beam is fed to an optical detector and electrically demodulated to a signal that represents the periodic velocity v and is equal to the difference between the velocities

of the measurement point v_m and the reference point v_r [24].

$$v = v_m - v_r. \quad (3.13)$$

For mode shape analysis, multipoint measurements are essential. There are roughly three methods for multipoint laser Doppler vibrometry: synchronous measurements with multiple beams, continuous single beam scanning and discrete single beam scanning [20]. Scanning laser Doppler vibrometers are able to automatically position the laser spot. In this way, a surface can be scanned to obtain the velocity information of multiple points on the surface. This enables the characterization of mode shapes of all kinds of mechanical structures, but only if the phase information between the points is known as well. This is usually done by triggering on the actuation signal.

For many microstructures in this dissertation, laser Doppler vibrometry has been used for for diagnostic and characterization purposes.

3.5 Readout of capacitive sensing structures

In mechanical microsensors, the measured quantity is converted into a displacement with a mechanical structure. Since almost all signal processing is done using electronics, this mechanical signal needs to be converted to an electrical signal. Besides piezoelectric, piezoresistive, resistive or inductive methods, capacitive detection might be the simplest method to implement in a sensor. It only needs a fixed electrode and an electrode at the moving structure, the capacitance decreases with increasing distance between the electrodes. Although implementation of capacitive readout structures might be simple, accurate capacitance measurements needs more comprehensive signal processing and shielding than e.g. resistive readout.

3.5.1 Charge amplification

There are multiple ways to measure capacitances [25]. For example, the capacitance can be made part of an electronic oscillator; the output frequency will be a measure for the capacitance. Another straightforward method is by connecting the capacitance to a current integrator or, generally called, charge amplifier. Charge amplification is used for all capacitive readouts in this dissertation.

Figure 3.17: Schematic of a charge amplifier. The output voltage of this circuit is dependent on the input capacitance.

The to be measured capacitance C_s is at one side connected to an input voltage u_i and at the other side connected to a simple implementation of a charge amplifier using an operational amplifier, as illustrated in Figure 3.17. As mentioned, this is a current integrator, the current i_{fb} through the feedback capacitor C_{fb} is therefore equal to:

$$i_{fb} = C_{fb} \frac{du_{fb}}{dt}. \quad (3.14)$$

For an ideal operational amplifier, the input voltages of both terminals is equal to zero in this case. This makes $u_{fb} = -u_o$ and $u_s = u_i$. The input terminals have infinite input resistance and thus the input current is also zero, this means that $i_s = i_{fb}$, therefore:

$$i_{fb} = C_s \frac{du_i}{dt} = -C_{fb} \frac{du_o}{dt} \rightarrow C_s du_i = -C_{fb} du_o, \quad (3.15)$$

or, when there is no initial charge:

$$u_o = -\frac{C_s}{C_{fb}} u_i. \quad (3.16)$$

It appears that voltage u_o is a measure for the capacitance C_s . This only works when an alternating input voltage u_i is applied, since capacitors filter direct currents.

Since there is a voltage applied to the to be measured capacitance C_s , a parasitic capacitance to ground at the left-hand side of capacitor C_s has no influence on the output signal. A parasitic capacitance to ground at the right-hand side of capacitor C_s has in theory no influence, since the negative input of the operational amplifier is kept at ground level by the circuit. In practice, due to the limited open-loop gain of the operational amplifier, the voltage at the negative input varies. Nevertheless, the current through this parasitic capacitance is still orders of magnitude lower than the current through capacitor C_s . However, this parasitic capacitance does result in amplification of amplifier noise.

3.5.2 Lock-in amplification

For capacitive microsensors, the capacitance changes are generally in the attofarad to femtofarad range. Many external factors can influence the capacitance or voltages in the charge amplifier. Since the input voltage of the capacitance with the charge amplifier has a known frequency, filtering the output signal with a very narrow band-pass filter helps to reduce all unwanted signal components at other frequencies. A lock-in amplifier acts as a very narrow band-pass filter and is also able to detect magnitude and phase information from the input signal compared to a reference signal. A basic lock-in amplifier setup is illustrated in Figure 3.18.

Figure 3.18: Schematic of a lock-in amplifier. The lock-in amplifier is only sensitive for input signals that have the same frequency as the reference signal.

The function of a lock-in amplifier can be clarified with a mathematical derivation. Consider a harmonic input signal $u_i(t)$ with amplitude $\hat{u}_i$, frequency ω_0 and phase ϕ_i :

$$u_i(t) = \hat{u}_i \sin(\omega_0 t + \phi_i), \quad (3.17)$$

and reference signal $u_{\text{ref}}(t)$ with amplitude $\hat{u}_{\text{ref}}$:

$$u_{\text{ref}}(t) = \hat{u}_{\text{ref}} \sin(\omega_0 t). \quad (3.18)$$

The mixer multiplies both signals, i.e. using the following goniometric identity:

$$\sin(\alpha) \cdot \sin(\beta) = \frac{1}{2}(\cos(\alpha - \beta) - \cos(\alpha + \beta)), \quad (3.19)$$

the output signal of the mixer $u_{\text{m}}(t)$ is:

$$u_{\text{m}}(t) = \hat{u}_{\text{i}} \hat{u}_{\text{ref}} \sin(\omega_0 t + \phi_{\text{i}}) \cdot \sin(\omega_0 t) = \frac{1}{2} \hat{u}_{\text{i}} \hat{u}_{\text{ref}} \underbrace{(\cos(\phi_{\text{i}}))}_{\text{DC}} - \underbrace{\cos(2\omega_0 t + \phi_{\text{i}}))}_{\text{AC at } 2\omega_0}, \quad (3.20)$$

or in words: the output signal of the mixer contains a DC-component dependent on the amplitude and phase of the original signal and a component with double frequency compared to the original signal. The low pass filter filters out the double frequency component:

$$u_{\text{o}} = \frac{1}{2} \hat{u}_{\text{i}} \hat{u}_{\text{ref}} \cos(\phi_{\text{i}}). \quad (3.21)$$

Generally, a commercially available lock-in amplifier has a second detector, consisting of a mixer and filter. The reference signal fed to this mixer is 90° shifted. The output signal of the second detector is therefore:

$$u_{\text{o}90} = \frac{1}{2} \hat{u}_{\text{i}} \hat{u}_{\text{ref}} \sin(\phi_{\text{i}}). \quad (3.22)$$

and can be used to distinguish the amplitude and phase from the input signal.

3.5.3 Static capacitance readout

The charge amplifier and lock-in amplifier can be combined. For static capacitance measurements, a carrier frequency is fed to the capacitance to be measured C_{s} . A charge amplifier circuit is used to convert this capacitance to a voltage. Then, a lock-in amplifier demodulates this signal at the carrier frequency and outputs a digital magnitude and phase. Figure 3.19 shows the electronic circuit of this readout method.

3.5.4 Synchronous capacitance readout

For synchronous capacitance changes, i.e. a capacitance that changes in time synchronously with an externally applied signal, an extra demodulation step is integrated in the circuit. After charge amplification, demodulation at the carrier frequency is achieved using a mixer filter. Then, the second lock-in amplifier stage locks in on the modulation frequency of the capacitance. Figure 3.20 shows the electronic circuit of

Figure 3.19: Circuit schematic for a static capacitance readout. The charge amplifier converts the input capacitance to a voltage. The lock-in amplifier is used to demodulate the carrier signal. It also makes the readout only sensitive for the frequency of the carrier signal.

this readout method

Figure 3.20: Circuit schematic for a synchronous capacitance measurement. Compared to the static capacitance measurement, an extra lock-in amplification stage is added to the signal path. This lock-in amplifier synchronizes with the modulated capacitance as a result of mechanical actuation.

3.6 Microfluidic chip assembly and interfacing

After fabrication, the sensor needs to be interfaced to characterize it. Both fluidic and electronic connections need to be made for microfluidic sensors. This section describes multiple chip assembly methods, how fluidic interfacing can be achieved and how the electronics from Section 3.5 can be integrated in the setup.

3.6.1 Specialized interfacing method

The Coriolis mass flow sensor from Haneveld et al. [26] for example, has a fluid path and two capacitive readout structures with capacitance changes in the femtofarad range. A specific printed circuit board is designed for this chip, as is illustrated in Figure 3.21. Magnets for the Lorentz force actuation are adhesively mounted in trenches at both sides (Figure 3.21b). The chip is adhesively mounted on the copper surface in the center (Figure 3.21c). Then, the chip is wirebonded to the printed circuit board (Figure 3.21d) and pin headers are soldered for electrical connections to the measurement equipment (Figure 3.21e). Finally, fluidic connectors (Swagelok® 1/16") are adhesively mounted on the backside of the printed circuit board or a 3D printed fluidic connector.

The assembled chip can now be electrically and fluidically interfaced. However, this method has some drawbacks: the assembly is very specific, labor-intensive and riskful work. Furthermore, other microfluidic devices might have different dimensions or need more electric or fluidic connections, i.e. the method is not universal.

Figure 3.21: Assembly of the chip for the specialized interfacing method. Magnets are glued in the PCB (b). Then, the chip is glued on the PCB (c) and wirebonded (d). After this, pinheaders are soldered (e) and a fluidic connector is glued (g and h) at the backside.

3.6.2 Universal modular interfacing method

The complexity of the electronic and fluidic interface is even higher with chips that contain multiple sensors and/or actuators [13, 27–29]. To gain efficiency, robustness and simplicity, a universal interfacing platform has been designed and built.

The assembly for this interfacing method consists of adhesively mounting the chip on the chip holder board (Figure 3.22b) and wirebonding (Figure 3.22c). There is no need for the assembly of magnets or soldering the pin headers, since this is implemented in the main board design.

Figure 3.22: Assembly of the chip for the universal modular interfacing method. The chip is glued on the pcb (b) and wirebonded (c).

Interfacing platform

Figure 3.23 shows an overview of the platform. The chip holder board (illustrated in Figure 3.22a) has been inspired by the conventional packaging method, but has multiple improvements. It features 8 fluid connections, 72 electric connections and 72 grounding connections for shielding.

The chip holder board can be clamped on the main board with four screws. The electrical connections are realized with pogo pin connectors. For each signal pin, there is a ground pin diagonally alternated in the 2×10 and 2×4 connectors to improve shielding. The main board has 16 junction gate field-effect transistors (JFET) directly connected to 16 of the 72 signal pins. These transistors can be used as close-to-the-chip amplifiers for capacitive measurements.

Connections to the main board, from e.g. modules that will be explained later, can be made using micro-miniature coaxial (MMCX) connectors. Figure 3.24b shows

Figure 3.23: Illustration of all components of the interfacing platform. The chip is mounted on a chip holder board. This board is fluidically connected with a 3D printed fluid block to tubing with flat bottom connectors. The chip holder board is connected electrically via pogo pins to the main board. Coax cables connect the main board to electronic interfacing modules.

Figure 3.24: Backside (a) of the main board with the fluid block and the connectors to the modules and frontside (b) of the main board with a chip holder board being mounted.

the frontside with the chip holder board being placed on the main board.

Figure 3.24a shows the fluidic connector on the main board. This polymer 3D-printed fluidic connector allows for up to 8 fluidic connections to the chip holder board. The fluidic contact is made using o-rings placed in grooves in the fluidic connector.

The power board has 8 Peripheral Component Interconnect Express (PCIe) connectors to hold and supply power to the modules. The pinout is not consistent with the PCIe standard; the connectors are just used because of their capability to clamp and connect to a PCB directly. Multiple voltages can be applied to the modules via the PCIe connectors, but the default supplied voltage is 10 V. Every module has its own voltage regulator to provide a reliable power source for the electronics. Multiple pins of the PCIe connectors are interconnected to provide a possible bus implementation in the future. A photographical impression of the complete setup is shown in Figure 3.25.

Figure 3.25: Photograph of the main board with multiple modules.

Electronic interfacing modules

These modules can be connected using coaxial cables via MMCX connectors to the main board. The set consists of all modules needed to actuate and characterize a capacitive Coriolis mass flow sensor.

- A high frequency oscillator which provides two different frequencies and has besides the default 5 V square wave output a tuneable amplitude output and an inverted output. The oscillators can be used to provide a carrier signal for e.g. capacitive readout structures as explained in Subsections 3.5.3 and 3.5.4.
- A charge amplifiers module with demodulation circuits, able to do static and synchronous capacitive measurements as described in Subsections 3.5.3 and 3.5.4.
- A mechanical resonator actuator that is able to inductively detect the resonance frequency, amplify this signal and actuate the resonator at its resonance frequency as described in Subsection 3.3.2.

3.6.3 Performance

A Coriolis mass flow sensor has been completely interfaced with the relevant modules. The performance of the platform has been tested by measuring the phase shift output of a Coriolis mass flow sensor without flow. The standard deviation has been calculated from the results as a measure for the measurement error. This is done for both the novel platform and the conventional electronics [14]. The phase detection is done using two Stanford Research Systems SR830 lock-in amplifiers. The integration time of the phase detection is varied between 10 ms and 10 s. In Figure 3.26, it can be seen that the novel platform has a noise level of approximately 2.5 times lower.

Figure 3.26: Standard deviations measured as a function of lock-in integration times of 10 ms to 10 s for both the conventional and the novel electronics.

3.7 Concluding remarks

- Surface channel technology provides a universal way to fabricate microfluidic devices. Suspended microchannels with metal wiring on top can be fabricated with this technology. This enables the realization of throughflow pressure sensors, flow sensors and other mechanical microfluidic sensors. Novel developments in surface channel technology may allow piezoelectric actuation and multi level channels.
- Three resonator actuation methods have been explained. Due to the influence of the fluid on the resonance frequency, it is essential to actuate microchannel resonators with a feedback-based actuation method.
- Measurement methods for quasi-static and synchronous capacitance changes have been discussed. These electronics are used throughout this dissertation for the readout of e.g. capacitive pressure and flow sensors.
- A novel universal interfacing platform has been realized and provides a time-efficient way to assemble, interface and characterize microfluidic devices. The high number of electric and fluidic connections combined with the modularity of the electronics enables the characterization of many different types of sensors and/or actuators.

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4

Resolution limits of micro Coriolis mass flow sensors

This chapter¹ starts with an analysis of the fundamental resolution limit due to thermomechanical noise of Coriolis mass flow sensors. The analysis is based on the equipartition theorem. In an experimental setup, the displacement of the channel due to thermomechanical noise is measured using a laser Doppler vibrometer for temperatures between 300 K and 700 K. The results show RMS vibration amplitudes of 38 pm to 57 pm over a bandwidth of 13 Hz centered around the resonance frequency, in good agreement with the theoretical prediction. This corresponds to a noise equivalent mass flow of 0.3 ng s^{-1} .

The next section explains a readout method that increases the phase shift and may help reaching the thermomechanical noise floor without improving the readout electronics. By adding two additional read out electrodes, the actuation mode signal is partially canceled, allowing for higher sensitivity to the Coriolis mode, and thus larger phase shifts for the same mass flows. A factor three increase of sensitivity was observed.

The thermomechanical noise is only measured for one point on the surface of the sensor. For mode analysis, multiple points need to be measured and the phase relation between the points needs to be known. Therefore, a method for laser Doppler vibrometry to find the phase relation between points without triggering is presented in the last section. This method consists of performing the surface scan in two stages: one scan with the reference beam at a fixed point and one scan with the reference beam on a moving point. The algorithm calculates the phase and reconstructs the velocity of each point. This method is experimentally verified with two different micro structures.

¹This chapter is based on the publications [1–3]:

- D. Alveringh, J. Groenesteijn, R. J. Wiegerink, and J. C. Lötters, “Improved capacitive detection method for Coriolis mass flow sensors enabling range/sensitivity tuning,” *Microelectronic engineering*, vol. 159, pp. 1–5, 2016;
- D. Alveringh, R. G. P. Sanders, R. J. Wiegerink, and J. C. Lötters, “Phase relation recovery for scanning laser Doppler vibrometry,” *Measurement Science and Technology*, vol. 28, no. 2, p. 025208, 2017;
- D. Alveringh, R. J. Wiegerink, J. Groenesteijn, R. G. P. Sanders, and J. C. Lötters, “Experimental analysis of thermomechanical noise in Coriolis mass flow sensors,” *Sensors and actuators A: Physical*, vol. 271, pp. 212–216, 2018.

4.1 Introduction

The difference between the minimum and maximum value of the quantity a sensor can measure is called range. The minimum change a sensor can detect is called resolution in this dissertation. The performance of a sensor is often indicated by the dynamic range, which is the ratio between range and resolution. Figure 4.1 gives an illustration of a sensor with indicated range, resolution and dynamic range.

Figure 4.1: A distance sensor (ruler) with specified range and resolution. The range is equal to the length of the ruler, the resolution is in this case dependent on the limitation of the human eye to distinguish the interval lines.

A sensor usually translates a physical quantity into an electrical signal. Figure 4.2 illustrates this translation with a linear graph. The slope of the curve is called sensitivity.

Figure 4.2: Calibration graph of a sensor with specified resolution and sensitivity.

In practice, the resolution limitations of mechanical sensors can be categorized by three different phenomena:

- noise from external sources and dependence on other physical quantities;
- resolution limitations of the readout electronics;
- the intrinsic thermomechanical noise.

The noise from external sources can be reduced by mechanical decoupling of the sensor from the surroundings, e.g. putting the sensor in vacuum and assemble it to a large mass. Other external influences may be compensated for as described in

Chapter 5. The resolution of the sensor can be increased by improvements in the electronic design itself, e.g. shielding, using better components or allowing larger power consumption (Chapter 3). Another option is to reduce the influence of the readout electronics by increasing the sensitivity of the sensor (Section 4.3), i.e. the same true value leads to a higher measured value. Only the thermomechanical noise is, for temperatures higher than 0 K, impossible to reduce to zero and defines therefore a fundamental limit for the resolution.

4.2 Thermomechanical noise limits

With downscaling Coriolis mass flow sensors by using microfabricated channels, the resolution has been improved significantly [4]. However, due to the smaller channel mass, also thermomechanical noise comes into play. Therefore, the fundamental limit to the resolution of microfabricated Coriolis mass flow sensors is given by thermomechanical noise. These fundamental limits have been studied for micro devices like accelerometers, [5–7] atomic force microscopy probes [8–10], resonators [11] and beams [12]. However, the resolution limits of Coriolis mass flow sensors have never been studied. A similar approach as for accelerometers is used in this section to derive the fundamental noise limit of Coriolis mass flow sensors, validate the result by measurements and derive a model for the noise equivalent mass flow.

4.2.1 Theory

When there is no external force or torque applied to a mechanical mass-spring-damper system, there still is thermomechanical noise. Since temperature is simply a measure for the energy of the molecular motion in the system, the total energy E (kinetic E_{kin} and potential E_{pot}) must be equal to the thermal energy and obey the equipartition theorem [5]. An expression for the equipartition theorem for a rotational mechanical system is therefore:

$$E = E_{\text{pot}} + E_{\text{kin}} = \frac{1}{2}K\langle\theta_n^2\rangle + \frac{1}{2}J\langle\Omega_n^2\rangle = k_B T, \quad (4.1)$$

with K the rotational stiffness, $\langle\theta_n^2\rangle$ the time average of the noise angle spectral density squared J the mass moment of inertia, $\langle\Omega_n^2\rangle$ the time average of the noise angular velocity spectral density squared, k_B the Boltzmann constant and T the temperature. The thermomechanical noise is modeled by first deriving equations for the time domain and frequency response of the Coriolis mass flow sensor mechanics. From this, the noise torque spectral density τ_n and the noise angle spectral density $\hat{\theta}_n$ are found using the equipartition theorem.

Time domain response

The twist and swing mode, as illustrated by Figure 4.3, can both be modeled as a second order mechanical system [13–15] in the rotation domain.

Following to Newton's second law of motion for rotations, the sum of all torques $\tau_i(t)$ on a mass moment of inertia must be equal to the product of the angular acceleration $d\Omega(t)/dt$ and the mass moment of inertia J :

$$\tau_j(t) = \sum \tau_i(t) = J \frac{d\Omega(t)}{dt}, \quad (4.2)$$

Figure 4.3: Coriolis mass flow sensor based on a rectangular tube shape. By actuating the twist mode, a fluid flow induces a Coriolis force causing the channel to move in the swing mode as well.

with $\tau_J(t)$ the torque on the mass moment of inertia, $\Omega(t)$ the angular velocity and t the time. The second order system consists of three components: the mass moment of inertia J , the rotational damping R and the rotational stiffness K . Therefore, the sum of all torques on the mass moment of inertia is:

$$\sum \tau_J(t) = \tau_R(t) + \tau_K(t) + \tau_{\text{ext}}(t), \quad (4.3)$$

with $\tau_R(t)$ the torque of the rotational damping, $\tau_K(t)$ the torque of the rotational stiffness and $\tau_{\text{ext}}(t)$ an externally applied torque.

The mass moment of inertia can be found using the general equation:

$$J = \iiint_V \rho r^2 dV, \quad (4.4)$$

with ρ the density, r the distance in radial direction from the rotation axis and dV an infinitesimal volume. The equation can be rewritten as a function of mass of an infinitesimal volume dm :

$$J = \int_m r^2 dm. \quad (4.5)$$

Segment I is modeled as a rod with length W rotating around its center as indicated in Figure 4.3. The total mass of the structure is m . So, with given aspect ratio $W/L = 8/5$, the mass of segment I is equal to $8/13m$. The infinitesimal volume dm becomes therefore:

$$dm = 8/13 \frac{dr}{W} m. \quad (4.6)$$

Segment II is modeled as a rod at distance $W/2$ from the rotation axis. In this case, it could be simplified to a point mass of $5/13m$. The total mass moment of inertia is

therefore:

$$J = \underbrace{\int_{-\frac{W}{2}}^{\frac{W}{2}} \frac{8}{13} \frac{m}{W} r^2 dr}_{\text{segment I}} + \underbrace{\frac{5}{13} m \left(\frac{W}{2} \right)^2}_{\text{segment II}}. \quad (4.7)$$

Performing the integral leads to:

$$J = r^3 \frac{8}{39} \frac{m}{W} \Big|_{-\frac{W}{2}}^{\frac{W}{2}} + \frac{5}{13} m \left(\frac{W}{2} \right)^2 = \frac{23}{156} m W^2 \approx \frac{1}{7} m W^2. \quad (4.8)$$

For the swing mode, a similar method could be used to find the mass moment of inertia J_S . For this case, the part of segment I close the the rotation axis is neglected, the other part of segment I is modeled as a rod at distance L from the rotation axis. Segment II is modeled as a rod with length L rotating around its end.

$$J_S = \underbrace{\int_0^L \frac{5}{13} \frac{m}{L} r^2 dr}_{\text{segment II}} + \underbrace{\frac{4}{13} m L^2}_{\text{segment I}} = \frac{17}{39} m L^2 \approx \frac{1}{2} m L^2. \quad (4.9)$$

A torsion spring integrates the angular velocity $\Omega(t)$ and results in a torque $\tau_K(t)$:

$$\tau_K(t) = -K \int \Omega(t) dt. \quad (4.10)$$

The torque $\tau_R(t)$ caused by damping is assumed to be proportional to the angular velocity:

$$\tau_R(t) = -R \Omega(t). \quad (4.11)$$

With the relations for the rotational damping and rotational stiffness, Equation 4.2 becomes:

$$J \frac{d\Omega(t)}{dt} = -R \Omega(t) - K \int \Omega(t) dt + \tau_{\text{ext}}(t), \quad (4.12)$$

or:

$$\tau_{\text{ext}}(t) = J \frac{d\Omega(t)}{dt} + R \Omega(t) + K \int \Omega(t) dt. \quad (4.13)$$

The equation as a function from the angle $\theta(t)$ rather than angular velocity can be obtained by integration of $\Omega(t)$:

$$\theta(t) = \int \Omega(t) dt, \quad (4.14)$$

and so Equation 4.13 becomes:

$$\tau_{\text{ext}}(t) = J \frac{d^2 \theta(t)}{dt^2} + R \frac{d\theta(t)}{dt} + K \theta(t). \quad (4.15)$$

Frequency domain response

The frequency domain response from the time domain derivation can be obtained using following Fourier transform pair [16]:

$$\mathcal{F}\left(\frac{d^n}{dt^n} f(t)\right) = (j\omega)^n f(\omega). \quad (4.16)$$

Equation 4.15 becomes therefore in the frequency domain:

$$J(j\omega)^2 \theta(\omega) + Rj\omega \theta(\omega) + K \theta(\omega) = \tau_{\text{ext}}(\omega), \quad (4.17)$$

with j the imaginary unit and ω the frequency. Rewritten:

$$\theta(\omega) = \frac{\tau_{\text{ext}}(\omega)}{-J\omega^2 + Rj\omega + K} = \frac{\tau_{\text{ext}}(\omega)}{K\left(1 - \frac{J}{K}\omega^2 + j\frac{R}{K}\omega\right)}, \quad (4.18)$$

and with these, an expression for the angular magnitude $\hat{\theta}(\omega)$ can be obtained [17]:

$$\hat{\theta}(\omega) = \left| \frac{\tau_{\text{ext}}(\omega)}{K\left(1 - \frac{J}{K}\omega^2 + j\frac{R}{K}\omega\right)} \right| \quad (4.19)$$

$$= \frac{\hat{\tau}_{\text{ext}}(\omega)}{K\sqrt{\left(1 - \frac{J}{K}\omega^2\right)^2 + \left(\frac{R}{K}\omega\right)^2}}. \quad (4.20)$$

The damping, mass moment of inertia and rotational stiffness can be rewritten as the dimensionless damping ratio ζ and the resonance frequency ω_0 :

$$\zeta = \frac{R}{2\sqrt{KJ}} \quad \text{and} \quad \omega_0 = \sqrt{\frac{K}{J}}. \quad (4.21)$$

And thus, Equation 4.20 as a function of the damping ratio ζ and the resonance frequency ω_0 :

$$\hat{\theta}(\omega) = \frac{\hat{\tau}_{\text{ext}}}{K\sqrt{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + 4\zeta^2\left(\frac{\omega}{\omega_0}\right)^2}}. \quad (4.22)$$

The quality factor Q is a dimensionless measure for how underdamped the system is, i.e. the system has a sharper and higher resonance peak when the quality factor is

higher. For high quality factors, the following relation holds:

$$Q = \frac{\omega_0}{\Delta\omega} = \frac{1}{2\zeta}, \quad (4.23)$$

with $\Delta\omega$ the bandwidth at half the magnitude of ω_0 . The quality factor can be substituted in Equation 4.22:

$$\hat{\theta}(\omega) = \frac{\hat{\tau}_{\text{ext}}}{K \sqrt{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}}. \quad (4.24)$$

Thermomechanical noise

Equation 4.24 defines the angular magnitude for the system as a function of an externally applied torque. This equation can be simply rewritten to a noise angle spectral density when a noise torque spectral density τ_n is applied. Note that the units of these quantities are $\text{rad}/(\sqrt{\text{rad/s}})$ and $\text{Nm}/(\sqrt{\text{rad/s}})$. This expression can be used with the equipartition theorem as expressed in Equation 4.1. Since half of the energy is potential energy in this situation, a simpler relation between angle and temperature can be found.

$$K \langle \theta_n^2 \rangle = k_B T. \quad (4.25)$$

The time average of the displacement squared $\langle \theta_n^2 \rangle$ can be calculated by integrating over the full spectrum:

$$\langle \theta_n^2 \rangle = \frac{1}{2\pi} \int_0^\infty \hat{\theta}_n(\omega)^2 d\omega. \quad (4.26)$$

Using Equation 4.24, the integral becomes:

$$\langle \theta_n^2 \rangle = \frac{1}{2\pi} \int_0^\infty \left(\frac{\tau_n}{K \sqrt{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}} \right)^2 d\omega \quad (4.27)$$

$$= \frac{\tau_n^2}{2\pi K^2} \int_0^\infty \frac{1}{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2} d\omega, \quad (4.28)$$

or simplified, with $\xi = \left(\frac{\omega}{\omega_0}\right)$:

$$\langle \theta_n^2 \rangle = \frac{\tau_n^2 \omega_0}{2\pi K^2} \int_0^\infty \frac{1}{(1 - \xi^2)^2 + \frac{1}{Q^2} \xi^2} d\xi \quad (4.29)$$

$$= \frac{\tau_n^2}{2\pi K^2} \int_0^\infty \frac{1}{(\xi^2)^2 + \left(\frac{1}{Q^2} - 2\right)(\xi^2) + 1} d\xi. \quad (4.30)$$

The roots of the denominator can be found:

$$\xi_{root,1|2}^2 = \frac{2Q^2 \pm \sqrt{1 - 4Q^2} - 1}{2Q^2}. \quad (4.31)$$

From relation 3.264 from [18], the following relation holds:

$$\int_0^\infty \frac{1}{(\xi^2 - \xi_{root,1}^2)(\xi^2 - \xi_{root,2}^2)} d\xi = \frac{\pi}{2} \frac{\left(-\xi_{root,2}^2\right)^{-\frac{1}{2}} - \left(-\xi_{root,1}^2\right)^{-\frac{1}{2}}}{\xi_{root,1}^2 - \xi_{root,2}^2} = \frac{Q\pi}{2}. \quad (4.32)$$

And thus, the outcome of the integral in Equation 4.27 is:

$$\langle \theta_n^2 \rangle = \frac{\tau_n^2 \omega_0 Q}{4K^2}. \quad (4.33)$$

Now, Equation 4.25 can be solved.

$$\frac{K}{2\pi} \int_0^\infty \hat{\theta}_n(\omega)^2 d\omega = \frac{\tau_n^2 \omega_0 Q}{4K} = k_B T. \quad (4.34)$$

The solution provides a simple expression for the noise torque spectral density τ_n :

$$\tau_n = \sqrt{4k_B T R}, \quad (4.35)$$

and can be interpreted as a mechanical expression of Johnson-Nyquist noise. The damping constant R is the only mechanical parameter in Equation 4.35. This is in line with the equipartition theory, since in thermal equilibrium the energy lost by damping must be compensated for by τ_n . The damping constant R can also be written as a function of the quality factor Q using the relations in Equation 4.21:

$$\tau_n = \sqrt{4k_B T \frac{\omega_0 J}{Q}}. \quad (4.36)$$

Equations 4.21 and 4.36 can be substituted in Equation 4.24 when the noise angle spectral density has to be found for a specific frequency.

$$\hat{\theta}_n(\omega) = \frac{\sqrt{4k_B T \frac{\omega_0 J}{Q}}}{J\omega_0^2 \sqrt{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}}. \quad (4.37)$$

4.2.2 Measurement setup

The measured device is fabricated using silicon-on-insulator-based surface channel technology described in Subsection 3.2.1. The noise displacement spectral density

$\hat{z}_n(\omega)$ is measured at the channel corners are labeled 1 and 2 in Figure 4.3. Substituting Equation 4.35 in Equation 4.24 and multiplying by $W/2$ gives an expression for this noise displacement spectral density as a function of frequency:

$$\hat{z}_n(\omega) \approx \frac{W}{2} \hat{\theta}_n(\omega) = \frac{W}{2} \frac{\sqrt{4k_B T \frac{\omega_0 J}{Q}}}{J \omega_0^2 \sqrt{\left(1 - \left(\frac{\omega}{\omega_0}\right)^2\right)^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}}. \quad (4.38)$$

The noise displacement spectral density is obtained by measuring the velocity of the channel using laser Doppler vibrometry at different temperatures, as schematically illustrated in Figure 4.4.

Figure 4.4: Experimental setup. A microfabricated Coriolis mass flow sensor is placed in a vacuum chamber on a vibration-free table. The temperature of the sensor between 300 K to 700 K using a power supply and a thermocouple. A laser Doppler vibrometer is used to measure the velocity of the channel.

The sensor chip is based on a design from [19]. The sensor chip is placed on an electrical heater. A thermocouple is attached to the silicon substrate of the chip using high temperature glue. This setup is placed in a vacuum chamber ($\sim 1 \cdot 10^{-6}$ mbar) to decrease air damping and increase the quality factor, making the noise displacement of the sensor detectable. The channel of the sensor is also connected to the vacuum. The laser Doppler vibrometer (Polytec MSA-400) was used to measure the discrete velocity spectrum in a bandwidth of 13 Hz in steps of 156 mHz around the resonance frequency. The velocity is converted to a displacement spectral density [20]. One measurement is done every 120 s and a temperature step is made every hour ranging from 300 K to 700 K. The measurements are done for both increasing and decreasing temperatures. Two complete temperature cycles were conducted.

4.2.3 Measurement results

Equation 4.38 was fitted to each of 1987 noise spectra that were measured, using fit parameters ω_0 , Q and T . Figure 4.5 shows four measurements together with the fitted equation as example. The resulting fit parameters have realistic values and are plotted in Figure 4.6. The mass moment of inertia J is approximated at $4 \cdot 10^{-14} \text{ kg m}^2$ based on the geometry of the channel, the width W is 4 mm.

Figure 4.5: Fits of the measured displacements for four different temperatures using Equation 4.38.

It appears that the resonance frequency decreases with temperature. This can be explained by a decrease in stiffness due to a decrease in Young's modulus by approximately 2 % for the full temperature range, which is in agreement with results from literature [21]. The quality factor also decreases with increasing temperature by the same order of magnitude as reported by Kim et al. [22] for silicon resonators. The fitted temperature corresponds well with the measured temperature, which is plotted in the same figure.

During the measurements, the displacement is sampled and a discrete Fourier transform (DFT) is applied, i.e. every DFT line in the spectrum defines the root mean square (RMS) amplitude of the noise in the band of that DFT line. The RMS displacement amplitude $\hat{z}_{n,\text{RMS}}$ over a larger bandwidth can be obtained from the following summation:

$$\hat{z}_{n,\text{RMS}} = \sqrt{\sum_{i=i_{\text{start}}}^{i_{\text{end}}} \hat{z}_n(i)^2}, \quad (4.39)$$

with i_{start} the first and i_{end} the last DFT line. The summation was performed for all 1987 DFT spectra, resulting in the RMS displacement amplitudes over a 13 Hz bandwidth around the corresponding resonance frequency, as shown in Figure 4.7.

Figure 4.6: Fitted frequency, quality factor and temperature for temperature cycle I.

The same data is plotted in Figure 4.8, but now as function of temperature, excluding the data points obtained during large gradients in temperature. The same figure also shows the theoretical RMS displacement amplitudes calculated using Equation 4.38 and Equation 4.39, with $\omega_0 = 2640\text{ Hz}$ and $Q = 8000$. A second model is also shown that includes the fact that the quality factor changes from 20000 to 1700 for temperatures ranging from 0 K to 700 K. For both temperature cycles, as well as for increasing and decreasing temperature, the measured RMS displacement amplitudes correspond to the models.

Figure 4.7: Full data set for temperature cycle I of the temperature and the RMS amplitudes of the displacement plotted in time.

Figure 4.8: Measured RMS noise and theoretical RMS noise plotted against temperature.

4.2.4 Signal to noise ratio

The sensor with currently the best resolution was presented by Groenesteijn et al. [23]. This sensor was microfabricated with a silicon nitride channel with a diameter of 31 μm and a resolution of approximately 14 ng s^{-1} was reported. Yet, the paper does not conclude if the dominant noise is caused by the sensor itself or the detection electronics.

In this implementation, the channel is actuated in the twist mode and due to the Coriolis forces, the channel also vibrates in the swing mode. The ratio of amplitudes of both modes is a measure for the mass flow. Note that it is also possible to operate the sensor by actuating the swing mode so that Coriolis forces will induce a twist mode. As found in Equation 2.24, the Coriolis force as a function of twist displacement amplitude $\hat{z}_T$ and mass flow Φ is:

$$\hat{F}_C = -2W\Phi \hat{\theta}_T \omega_T \approx 4\omega_T \hat{z}_T \Phi. \quad (4.40)$$

The Coriolis force results in a torque $\hat{\tau}_C$ given by:

$$\hat{\tau}_C = \hat{F}_C L \approx 4L\omega_T \hat{z}_T \Phi. \quad (4.41)$$

This torque can be interpreted as the desirable signal, since it is directly dependent on mass flow. The undesirable signal is the torque due to noise. The SNR signal to noise ratio (SNR) for a Coriolis mass flow sensor can be estimated, which is the ratio between Equation 4.41 and Equation 4.35 multiplied by the square root of the bandwidth $\sqrt{\Delta f}$:

$$\text{SNR} = \frac{\hat{\tau}_C}{\tau_{n,S} \sqrt{\Delta f}}. \quad (4.42)$$

It appears that the signal to noise ratio is independent of the system transfer.

The actuated twist mode has its own resonance frequency; the Coriolis force induces the swing mode at the same frequency. The swing mode has also its own resonance frequency. Therefore, the noise of two different modes at two different frequencies might be relevant:

- the displacement noise of the twist mode at the resonance frequency of the swing mode, however, this is not in the band of the readout electronics;
- the displacement noise of the twist mode on the resonance frequency of the twist mode, however, this only gives a non-significant increase in actuation;
- the displacement noise of the swing mode at the resonance frequency of the swing mode is not interesting because of both of above mentioned reasons;
- the displacement noise of the swing mode at the resonance frequency of the twist mode.

For a Coriolis mass flow sensor as indicated in Figure 4.3, using the twist mode for actuation and the swing mode for detection, the noise torque spectral density is given by:

$$\tau_{n,S} = \sqrt{4k_B T \frac{\omega_S J_S}{Q_S}}, \quad (4.43)$$

with mass moment of inertia for the swing mode J_S as specified by Equation 4.9. Then Equation 4.42 can be rewritten as:

$$\text{SNR} = \frac{\omega_T \hat{z}_T \Phi \sqrt{8Q_S}}{\sqrt{k_B T \omega_S m \Delta f}}. \quad (4.44)$$

The parameters ω_T , ω_S , $\hat{z}_T$, m and Q_S are coupled, e.g. the twist mode resonance frequency ω_T is smaller when mass m is larger. Nevertheless, the equation can be used for a SNR estimation. Using Equation 4.44, noise equivalent mass flow Φ_n (corresponding to $\text{SNR} = 1$) can be defined:

$$\Phi_n = \frac{\sqrt{k_B T \omega_S m \Delta f}}{\omega_T \hat{z}_T \sqrt{8Q_S}}. \quad (4.45)$$

Filling in the numbers (Table 4.1) for the sensor reported by Groenesteijn et al. [23], a value of 0.3 ng s^{-1} is found. This limit is almost in the same order of magnitude as the resolution for thermal flow sensors (0.02 ng s^{-1}) [24]. Hence, this sensor is still limited by noise in the readout circuitry or by other disturbances and an improvement by a factor of 50 is possible.

Table 4.1: Approximated numbers of the sensor from Groenesteijn et al. [23] for atmospheric pressure.

Boltzmann constant	k_B	1.3806 J K^{-1}
Room temperature	T	300 K
Mass of the channel	m	$17.5 \text{ } \mu\text{g}$
Bandwidth	Δf	1 Hz
Twist mode resonance frequency	ω_T	$16587 \text{ rad s}^{-1} \triangleq 2640 \text{ Hz}$
Twist mode displacement amplitude	$\hat{z}_T$	$10 \text{ } \mu\text{m}$
Swing mode resonance frequency	ω_S	$9425 \text{ rad s}^{-1} \triangleq 1500 \text{ Hz}$
Swing mode quality factor	Q_S	40

4.3 Sensitivity improvement

Since the resolution of the Coriolis mass flow sensor is not yet limited by thermomechanical noise, an improved resolution can be achieved by improving the readout. One way is by improving the resolution of the capacitance and phase detection electronics. Another is by optimizing the placement and geometry of the readout electrodes at the Coriolis mass flow sensor. The latter has been done by placing the capacitive electrodes closer to the twist axis at both sides of the channel [23]. This makes the readout less sensitive to the twist mode and causes therefore a higher phase shift for the same Coriolis force as is illustrated in Figure 4.9.

Figure 4.9: A conventional capacitive read out (left) provides a phase shift between the two output signals, however due to the small amplitude of the Coriolis motion the phase shift is small. Positioning the electrodes closer to the center makes the phase shift $\mathcal{A}$ times more sensitive to the swing mode.

Another way to reduce the twist mode component from the output signals is by cancellation. For this, two extra capacitive electrodes are needed.

4.3.1 Design

The operating principle of a capacitive readout with actuation mode cancellation is illustrated in Figure 4.10. Another pair of electrodes is added further away from the rotation axis, where the actuation mode amplitude is much larger. By scaling the size of these electrodes and connecting them in parallel to the readout electrode at

the other side of the rotation axis it is possible to cancel most of the actuation mode while keeping the same (or even slightly increased) sensitivity for the detection mode. Therefore, a much larger phase shift is obtained at the same mass flow.

Figure 4.10: A conventional capacitive read out (left) provides a phase shift between the two output signals, however due to the small amplitude of the Coriolis motion the phase shift is small. The improved read out (right) uses two additional electrodes to cancel part of the twist mode; therefore, the phase shift is larger allowing for much higher sensitivity.

Figure 4.11 shows the photomask design of the sensor. The channel design is similar to the design reviewed in Section 2.3.3.

Due to residual stress, the long silicon nitride channels bend upward multiple micrometers at the position of the electrodes. As a result, the capacitance varies with the distance between the electrodes and thus with the motion of the channel for small amplitudes. The large electrodes have a width W_{large} of approximately $950\mu\text{m}$ and the centers are located $475\mu\text{m}$ from the rotation axis (half of the width). The small electrodes have a width W_{small} of approximately $200\mu\text{m}$ and are located $575\mu\text{m}$ from the rotation axis. The width of the small electrodes W_{small} can be increased to $280\mu\text{m}$ with a distance of $615\mu\text{m}$ to the rotation axis by connecting extra comb fingers in parallel. The actuation signal due to the twist mode is proportional to the width and the location of the electrodes, since the channel acts as a lever. The Coriolis signal due to the swing mode is only proportional to the width of the electrodes. This phenomenon makes discrimination between the two modes, and thus cancellation of

Figure 4.11: Photomask design of the new sensor, showing the new capacitive readout with large electrodes with width W_{large} and additional small electrode structures. The small electrodes have width W_{small} .

the actuation mode signal, possible.

Each of the two output signals C_1 and C_2 consists of an actuation mode component and a Coriolis mode component. The actuation mode component C_{act} can be expressed as:

$$C_{\text{act}} = \hat{C}_{\text{act}} \cdot \sin(\omega t), \quad (4.46)$$

with $\hat{C}_{\text{act}}$ the amplitude, ω the frequency and t the time. As mentioned above, the Coriolis component has a 90° phase shift. Thus, it can be expressed as:

$$C_{\text{cor}} = \hat{C}_{\text{cor}} \cdot \cos(\omega t), \quad (4.47)$$

with $\hat{C}_{\text{cor}}$ the Coriolis mode component. The two capacitive output signals can now be written as:

$$C_1 = C_{\text{cor}} + C_{\text{act}}, \quad (4.48)$$

$$C_2 = C_{\text{cor}} - C_{\text{act}}, \quad (4.49)$$

The sum $C_{\text{act}} + C_{\text{cor}}$ is:

$$\hat{C}_{\text{act}} \cdot \sin(\omega t) + \hat{C}_{\text{cor}} \cdot \cos(\omega t) = \hat{C}_{\text{cmb}} \cdot \sin(\omega t + \phi), \quad (4.50)$$

with $\hat{C}_{\text{cmb}}$ the amplitude of the combined signal and $\sin(\omega t + \phi)$ the combined signal

with phase shift ϕ . This equation can be rewritten to:

$$\sin(\omega t) + \frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}} \cdot \cos(\omega t) = \frac{\hat{C}_{\text{cmb}}}{\hat{C}_{\text{act}}} \cdot \sin(\omega t + \phi). \quad (4.51)$$

To find an expression for the phase shift, the following trigonometric identity

$$\cos(\phi) \cdot \sin(\omega t) + \sin(\phi) \cdot \cos(\omega t) = \sin(\omega t + \phi), \quad (4.52)$$

is rewritten to:

$$\sin(\omega t) + \tan(\phi) \cdot \cos(\omega t) = \frac{1}{\cos(\phi)} \cdot \sin(\omega t + \phi). \quad (4.53)$$

By combining Equation 4.51 and Equation 4.53 it can be concluded that:

$$\tan(\phi) = \frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}} \rightarrow \phi = \arctan\left(\frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}}\right), \quad (4.54)$$

and

$$\frac{1}{\cos(\phi)} = \frac{\hat{C}_{\text{cmb}}}{\hat{C}_{\text{act}}} \rightarrow \hat{C}_{\text{cmb}} = \frac{\hat{C}_{\text{act}}}{\cos(\phi)} = \frac{\hat{C}_{\text{act}}}{\cos\left(\arctan\left(\frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}}\right)\right)}. \quad (4.55)$$

And since

$$\cos(\arctan(\beta)) = \frac{1}{\sqrt{1 + \beta^2}}, \quad (4.56)$$

Equation 4.55 can be simplified to:

$$\hat{C}_{\text{cmb}} = \hat{C}_{\text{act}} \sqrt{1 + \left(\frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}}\right)^2} = \sqrt{\hat{C}_{\text{act}}^2 + \hat{C}_{\text{cor}}^2}. \quad (4.57)$$

Thus, Equations 4.46 and 4.47 become:

$$C_1 = \hat{C}_{\text{cmb}} \cdot \sin(\omega t + \phi), \quad (4.58)$$

$$C_2 = -\hat{C}_{\text{cmb}} \cdot \sin(\omega t - \phi). \quad (4.59)$$

The phase difference $\Delta\phi$ between C_1 and C_2 is:

$$\Delta\phi = \phi - (-\phi) = 2\phi = 2\arctan\left(\frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}}\right). \quad (4.60)$$

For small phase shifts, $\tan(\Delta\phi) \approx \Delta\phi$; the phase shift is proportional to the ratio of the Coriolis mode amplitude and actuation mode amplitude. From this, it follows that the phase shift can be increased (factor $\mathcal{A}$) by reducing the actuation signal. This

can be achieved by subtracting the signal of the small electrodes from the signal of the large electrodes.

$$\mathcal{A} \cdot \Delta\phi = 2 \frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{act}}} = 2 \frac{\hat{C}_{\text{cor}}}{\hat{C}_{\text{large}} - \hat{C}_{\text{small}}}. \quad (4.61)$$

Note that the Coriolis mode component C_{cor} will slightly increase when the small electrodes are connected. However, the small electrodes are much more sensitive to the twist mode than for the swing mode due to their position. The cancellation factor $\mathcal{A}$ is equal to the ratio between the conventional readout and the readout with cancellation:

$$\mathcal{A} = \frac{\hat{C}_{\text{large}}}{\hat{C}_{\text{large}} - \hat{C}_{\text{small}}}, \quad (4.62)$$

and can be estimated using the geometric definitions in Figure 4.11. As mentioned before, the amplitude of the large electrodes $\hat{C}_{\text{large}}$ is proportional to the product of the width of the electrode (W_{large}) and the distance between the center of the electrode and the rotation axis ($W_{\text{large}}/2$):

$$\hat{C}_{\text{large}} \propto W_{\text{large}} \cdot \frac{W_{\text{large}}}{2}. \quad (4.63)$$

The amplitude of the small electrodes $\hat{C}_{\text{small}}$ is in a similar way proportional to the geometry:

$$\hat{C}_{\text{small}} \propto W_{\text{small}} \cdot \left(W_{\text{large}} + \frac{W_{\text{small}}}{2} \right). \quad (4.64)$$

The cancellation factor $\mathcal{A}$ is based on Equations 4.62, 4.63 and 4.64 therefore:

$$\mathcal{A} = \frac{W_{\text{large}}^2}{W_{\text{large}}^2 - W_{\text{small}}^2 - W_{\text{large}} W_{\text{small}}}. \quad (4.65)$$

With the approximated values $W_{\text{large}} = 950\mu\text{m}$ and $W_{\text{small}} = 200\mu\text{m} \vee 280\mu\text{m}$, the following cancellation factors are estimated:

- $W_{\text{small}} = 0\mu\text{m}$: $\mathcal{A} = 1$ (no cancellation);
- $W_{\text{small}} = 200\mu\text{m}$: $\mathcal{A} = 1.9$;
- $W_{\text{small}} = 280\mu\text{m}$: $\mathcal{A} = 3.1$.

The amplitudes of the signals fed to the readout electronics decrease with higher cancellation as is described by Equation 4.57. Increasing the actuation amplitude of the channel will increase the amplitudes of the signals. However, the improvement of the output resolution with this method is limited, since the capacitance of the electrodes do not change linearly with the displacement. The derivation in this section assumes the relation to be linear. The smaller signal amplitude does allow to increase the amplitude of the carrier frequency.

4.3.2 Experimental setup

The measured device is fabricated using silicon-on-insulator-based surface channel technology described in Subsection 3.2.1. A scanning electron microscopy (SEM) close-up is shown in Figure 4.12. As described by the specialized interfacing method in Subsection 3.6.1, the chip is glued on a printed circuit board. The inlet and outlet are at the bottom of the chip and directly connected to holes in the PCB. A 3D-printed fluid connector is glued at the other side of the printed circuit board.

Figure 4.12: SEM close up of the channel with the large and small electrode structures at one side of the channel.

The fabricated devices were characterized by applying a flow and measuring the phase shift between the two output signals for three situations: no actuation mode cancellation, actuation mode cancellation with $\mathcal{A} = 1.9$ and actuation mode cancellation with $\mathcal{A} = 3.1$.

A well-defined mass flow is applied using a commercially available mass flow controller (Bronkhorst® M12p) in combination with a pressurized tank filled with deionized and filtered water. The mass flow controller is placed after the device under test to prevent it from contaminating the micro channels.

Figure 4.13 shows a block schematic of the interface electronics used for capacitive readout. A 1 MHz carrier signal is applied to the electrode structures on the moving channel. The fixed electrode structures are kept at virtual ground by using two charge amplifiers. The resulting amplitude modulated signals are demodulated using analog multipliers and low-pass filters, giving analog voltages proportional to the capacitances. Two lock-in amplifiers are used to detect the phase shift between these signals. Details about synchronous capacitance measurements are written in Subsection 3.5.4.

Figure 4.13: Block schematic of the electronic measurement setup.

4.3.3 Characterization

Figure 4.14 shows measurement results for mass flow of water from 0 g h^{-1} up to 10 g h^{-1} , without and with actuation mode cancellation. Arctan-fits are applied through the measurements. The theoretical cancellation $\mathcal{A}$ of 1.9 and 3.1 result in approximately the same measured increases in phase shifts, as is described in Equation 4.61.

Figure 4.14: Conventional readout and improved readout measurement results using actuation mode cancellation. The phase shift increases a factor 1.9 or 3.1 dependent on the amount of cancellation.

4.3.4 Dynamic sensitivity tuning

Besides manual sensitivity tuning by changing the size of the cancellation electrodes, electronic sensitivity tuning may be possible by changing the carrier amplitude of the cancellation electrodes. A controller circuit could be applied between the output and the cancellation carrier amplitude. This would make the cancellation carrier amplitude dependent on the output phase shift of the Coriolis mass flow sensor. The controller could hold the phase shift to a fixed phase shift output. The cancellation carrier amplitude would become a measure for the flow. Figure 4.15 shows an experimental setup with a proportional control loop.

One advantage would be that the phase detector can always work in its most sensitive range. Furthermore, the relation between mass flow and cancellation carrier amplitude would not be an arctangent-curve, as it is the case for the direct relation between mass flow and phase shift. With an optimized design, cancellation carrier amplitude control could lead to an improvement in dynamic range, making it more accurate for lower flows and more linear for higher flows. A drawback would be that the cancellation carrier amplitude around zero flow would increase to infinity; the controller would be still trying to achieve the fixed phase shift. A hard-coded fixed minimum flow threshold would solve this.

Figure 4.15: The output phase shift from the lock-in amplifiers is fed back to the carrier generators for the small electrodes. A higher flow results in a higher phase shift, but due to the feedback loop, the cancellation is increased; the phase shift goes to its setpoint and the cancellation voltage becomes a measure for the mass flow.

4.3.5 Design improvements

Extra electrodes provide extra data which could be used for cancellation. The extra electrodes could also be used for pressure measurements as is described in Chapter 5. The addition of electrodes could also help in mode analysis. Besides a slight increase in air damping and mass, the addition of electrodes does not have significant drawbacks. Therefore, the Coriolis mass flow sensors shown in Figure 4.16 could be interesting samples for research purposes on the subject of actuation mode cancellation, pressure compensation and mode analysis.

Figure 4.16: SEM images of two possible electrode improvements, with (a) modular electrode configuration on top of the channel and (b) electrodes at multiple locations of the channel for mode analysis.

4.4 Mode analysis of noise actuated structures

As used in the first section of this chapter and described in Chapter 3, laser Doppler vibrometers are able to measure the velocity of a single point compared to a reference point by analyzing the Doppler shift of the laser beams. Usually, vibrometers can only measure one point simultaneously. In many commercially available laser Doppler vibrometers, the laser point can be scanned to obtain an out-of-plane velocity profile of a surface. It is essential in this case that the phase information of the velocities between points is measured as well to be able to fully reproduce the velocity profile of the surface. If the surface is actuated by an unknown actuation signal, triggering can be done on a signal provided by an external sensor, i.e. a force sensor that is mounted on the sample. But this is very difficult for small structures (e.g. microsystems) or vulnerable items (e.g. artwork). A second vibrometer may solve this by measuring one moving point and making the first vibrometer trigger on that signal. However, this makes the setup twice as large and twice as expensive. This section describes a measurement method with post-processing algorithm to recover the phase information by measuring the surface in two stages: one scan with the reference beam at a fixed point and one scan with the reference beam on a moving point.

4.4.1 Theory

In Figure 4.17a, the measurement beam of a laser Doppler vibrometer scans a simple seesaw-like structure in five points (points $z_0 \dots z_4$). The reference is simply pointed at a fixed surface. This conventional setup (fixed reference scan) provides the amplitudes $\hat{v}_{z_i}$ of the out-of-plane velocities v_{z_i} along the surface of the seesaw, which will be a V-shape in this example, since the sides will have higher velocities than the center. The velocity v_{z_i} at one frequency ω can be described by:

$$v_{z_i} = \hat{v}_{z_i} \sin(\omega t + \phi_{z_i}), \quad (4.66)$$

where the phase ϕ_{z_i} is unknown, since it is unknown when in time above velocity is acquired.

For a second scan (differential scan), the reference beam is pointed at a moving point z_0 . The amplitude $\hat{v}_{z_0-z_0}$ of z_0 will be zero now and will increase to twice the amplitude at z_4 : $\hat{v}_{z_4-z_0}$ as is illustrated in the graph in Figure 4.17b. The velocities $v_{z_i-z_0}$ can be described by:

$$v_{z_i-z_0} = \hat{v}_{z_i-z_0} \sin(\omega t + \phi_{z_i-z_0}). \quad (4.67)$$

In the fixed reference scan, the amplitude $\hat{v}_{z_0}$ of z_0 with respect to the fixed reference is also measured. For every other point z_i , the amplitude $\hat{v}_{z_i}$ with respect to the fixed reference and the amplitude $\hat{v}_{z_i-z_0}$ with respect to the moving reference are

Figure 4.17: Illustration of the two stage measurement principle: (a) a scan is done with a fixed reference, (b) a differential scan is done with a moving reference and (c) using an algorithm in post-processing, the phase relation between the points can be recovered.

known. The following equation holds:

$$v_{z_i-z_0} = v_{z_i} - v_{z_0}. \quad (4.68)$$

Above velocities are defined in Equations 4.66 and 4.67. These are substituted in following equation. The underbraces show how the relevant variables are defined, i.e. by the fixed reference measurement ('fixed'), the differential measurement ('diff') or if it needs to be recovered ('rcvr').

$$\underbrace{\hat{v}_{z_i-z_0}}_{\text{diff}} \underbrace{\sin(\omega t + \phi_{z_i-z_0})}_{\text{rcvr}} = \underbrace{\hat{v}_{z_i}}_{\text{fixed}} \underbrace{\sin(\omega t + \phi_{z_i})}_{\text{rcvr}} - \underbrace{\hat{v}_{z_0}}_{\text{fixed}} \underbrace{\sin(\omega t + \phi_{z_0})}_{\text{rcvr}}, \quad (4.69)$$

for above trigonometric identity, the following relation between amplitudes and phases hold [25]:

$$\hat{v}_{z_i-z_0}^2 = \hat{v}_{z_i}^2 - \hat{v}_{z_0}^2 + 2\hat{v}_{z_i}\hat{v}_{z_0}\cos(\phi_{z_i} - \phi_{z_0}), \quad (4.70)$$

and

$$\phi_{z_i - z_0} = \arctan \left(\frac{\hat{v}_{z_i} \sin(\phi_{z_i}) + \hat{v}_{z_0} \sin(\phi_{z_0})}{\hat{v}_{z_i} \cos(\phi_{z_i}) + \hat{v}_{z_0} \cos(\phi_{z_0})} \right), \quad (4.71)$$

with the measured amplitudes ($\hat{v}_{z_0}$, $\hat{v}_{z_i}$ and $\hat{v}_{z_i - z_0}$), the phase $\phi_{z_i} - \phi_{z_0}$ can be recovered (Figure 4.17c) by rewriting Equation 4.70:

$$\phi_{z_i} - \phi_{z_0} = \arccos \left(\frac{\hat{v}_{z_i - z_0}^2 - \hat{v}_{z_i}^2 + \hat{v}_{z_0}^2}{2\hat{v}_{z_i} \hat{v}_{z_0}} \right). \quad (4.72)$$

The complete velocity profile for one frequency of the moving surface can be reconstructed now, since the amplitude and the phase are known.

4.4.2 Measurement setup

The used Polytec MSA-400 is equipped with a Polytec OFV-552 differential fiber-optic interferometer. It has an automated discrete scanning function to scan the surface with the measurement beam. The reference beam has to be set manually.

The two stage phase relation recovery measurement and postprocessing is experimentally validated using three different microstructures: a Coriolis mass flow sensor [13], a cantilever beam and the vortex flow sensor described in Subsection 2.3.4 [26].

The Coriolis mass flow sensor is actuated by Lorentz actuation, driven by a waveform generator that sweeps from 0 kHz to 20 kHz. 25 points are defined on the twisting channel (Figure 4.18) and resembles the seesaw example in Figure 4.17. The fixed reference is placed at an anchor and the moving reference is placed at a high-amplitude spot on the channel.

Figure 4.18: Scanning electron microscopy (edited) image of the top of a micro Coriolis mass flow sensor with an impression of the scan points (cyan), fixed reference (red) and moving reference (green). In the actual measurement, there were 25 scanpoints.

The cantilever beam is actuated by a piezo actuator driven by white noise (Agilent 33220A). A 2D-array of measurement points is placed on the surface (Figure 4.19).

The fixed reference is placed at the anchor of the beam and the moving reference is placed at the tip, where the amplitude is the largest.

Figure 4.19: Scanning electron microscopy (edited) image of the top of a cantilever beam with an impression of the scan points (cyan), fixed reference (red) and moving reference (green).

The amplitudes of the relevant frequencies (e.g. the swing mode of the cantilever beam) of all points of both measurement stages are exported to files and processed using numerical computing software. Equation 4.72 is used to recover the phase and equation 4.66 is used to reconstruct the velocity function of each point. The algorithm consists of only a few trigonometric operations, multiplications and summations per point, which is not intensive in computing power.

4.4.3 Measurement results

In Figure 4.20, the reconstructed phase for each point for the Coriolis mass flow sensor is plotted. The phase is 180° shifted between the left and right side of the center, similar to the illustration of Figure 4.17. From these phases, the velocities of one period for all points are reconstructed (Figure 4.21). The reconstruction is done using the amplitudes at the sensor's resonance frequency for the twist mode at 2.5 kHz, hence, the plotted period corresponds to $400 \mu\text{s}$.

Figure 4.20: Phase profile of the Coriolis mass flow sensor (Figure 4.18) actuated by a sweep of frequencies in twist mode. There is a clear shift of 180° between the left and right sides of the center.

Figure 4.21: Velocity profile of multiple moments in the period of a Coriolis mass flow sensor (Figure 4.18) actuated by a sweep of frequencies in twist mode.

In Figure 4.22, the reconstructed velocities for the cantilever beam, in swing mode at 24 kHz, are plotted. Figure 4.23 shows the same structure, but in twist mode at 60 kHz. Both measurement results are obtained from the same two scans.

Figure 4.22: Velocity profile of multiple different moments in the period of a beam (Figure 4.19) actuated by noise in swing mode.

Figure 4.23: Velocity profile of multiple different moments in the period of a beam (Figure 4.19) actuated by noise in twist mode.

Figure 4.24 shows the velocities of the membrane of the vortex channel at 75 kHz. A wave shape can be observed, starting at the vortex source at the left of the channel where the branches of the heart meet. Different phases along the channel are visible, e.g.: when the velocity at the source decreases, the velocity further in the channel is at its maximum.

Figure 4.24: Rendered images of the membrane velocities of the vortex channel after phase relation recovery, with yellow/white a positive velocity and red/black a negative velocity.

4

4.4.4 Discussion

The recovery algorithm is tested for a single frequency with high amplitude for different structures. The recovery of a spectrum of frequencies for the phase is expected to be possible, but is not validated. Recovering the phases for a spectrum of frequencies enables curve fitting and averaging on the amplitudes and may result in more accurate phase recovery.

The recovery of all structures is done using one moving reference. It is expected that the recovery will be more accurate when multiple scans are done with more reference positions. Furthermore, this will be more robust when extreme positive and negative phase shifts need to be detected.

An extensive quantitative analysis must be conducted to find the accuracy of this method compared to alternatives using triggering.

4.5 Concluding remarks

- The mechanical motion caused by thermomechanical noise in a micro Coriolis mass flow sensor is modeled and validated by measurements. For the current Coriolis mass flow sensor with the best resolution, a noise equivalent mass flow of 0.3 ng s^{-1} is derived. This shows that the resolution of this sensor is still not limited by thermomechanical noise, but by the readout circuitry or external influences. Coriolis mass flow sensors may become a serious alternative to thermal flow sensors for measuring extremely low flows.
- The actuation signal component in the output signal of Coriolis mass flow sensors can be reduced using a second pair of electrodes. It is shown that this actuation mode cancellation causes higher phase shifts at the cost of a lower output signal amplitude. Nevertheless, it improves the resolution of the mass flow sensor when the phase detector has limitations in phase-resolution. An increase of sensitivity by a factor of 3 is experimentally demonstrated.
- An algorithm to recover the phase relation between different measured points in a laser Doppler vibrometer setup for a non-triggerable signal is proposed and validated. The algorithm requires a two stage measurement, i.e. the surface has to be scanned with a fixed reference and differentially with a moving reference. The method can be implemented in existing laser Doppler vibrometers and enables mode analysis of noise actuated structures.

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5

Surface channel technology compatible pressure sensors

This chapter¹ describes two types of pressure sensing mechanisms compatible with surface channel technology. Both mechanisms can be integrated with the Coriolis mass flow sensor described in Chapters 2 and 4, as will be discussed in Chapter 6. This chapter focuses on the design and characterization of the mechanisms.

The first sensing mechanism consists of a membrane that deforms when a pressure is applied. Metal structures are deposited on top of the membrane. These structures change in resistance as a result of the membrane deformation and are connected in a Wheatstone bridge, so no complex interfacing electronics are needed. Characterization of the sensor shows a linear sensitivity of $4 \cdot 10^{-5} \text{ bar}^{-1}$ for a gauge pressure range from 0 bar to 1 bar.

The second sensing mechanism is based on a suspended U-shaped channel. Due to the asymmetric cross-section of the channel, a pressure causes out-of-plane bending of the tip. Electrodes at the tip change in capacitance due to the deformation. This structure is easily scalable by changing its length. The most sensitive structure has a sensitivity of 1 fF bar^{-1} for measured gauge pressures from 0 bar to 1 bar and the response fits well to analytical and finite element models.

The structure of a Coriolis mass flow sensor can be used in the same way as the second sensing mechanism for pressure measurements. This chapter therefore ends with analysis and characterization of this fully integrated mass flow and pressure sensing mechanism.

¹This chapter is based on the publications [1–3]:

- D. Alveringh, J. Groenesteijn, R. J. Wiegerink, and J. C. Lötters, “Inline pressure sensing mechanisms enabling scalable range and sensitivity,” in *Proceedings of the 18th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS 2015)*. Anchorage, United States of America: IEEE, 2015, pp. 1187–1190;
- D. Alveringh, R. J. Wiegerink, and J. C. Lötters, “Integrated pressure sensing using capacitive Coriolis mass flow sensors,” *Journal of Microelectromechanical Systems*, vol. 26, no. 3, pp. 653–661, 2017;
- D. Alveringh, T. V. P. Schut, R. J. Wiegerink, W. Sparreboom, and J. C. Lötters, “Resistive pressure sensors integrated with a Coriolis mass flow sensor,” in *Proceedings of the 19th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS 2017)*. Taipei, Taiwan: IEEE, 2017, pp. 1167–1170.

5.1 Introduction

As mentioned in Chapter 1, miniaturization of sensors using microfabrication could result in advantages for many applications, e.g. better resolutions and lower unit prices [4, 5]. Besides, different fabrication methods are available in microtechnology and different materials are available as channel material compared to conventional fabrication methods.

Disadvantages are also involved with microfabricated channels. Many fabrication methods for microchannels, made of e.g. PDMS [6], SU-8 [7], silicon [8] or silicon nitride [9, 10] are not circular as is illustrated in Figure 5.1. Non ideal effects when using the channels are self-evident, like: dead volumes, complicated flow patterns and pressure dependent deformation of the structures. The non-circular shapes as described in the references are often inherent to the fabrication process and not easily solved without radical changes.

Figure 5.1: Artist's impression of multiple non-circularly shaped microchannels, with (a) PDMS or SU-8 channels bonded on silicon [6, 7], (b) etched channels in silicon bonded on silicon [8], (c) channels of silicon nitride on or in silicon [9–11] and (d) close-up of the cross-section of a (suspended) surface channel.

Surface channel technology has the same non-ideal effect. A cross-section of the channel is illustrated in Figure 5.1d [12]. Yet, with an adequate channel design and when calibrated well, the pressure dependent deformation of the channel can be used for pressure sensing.

5.2 Cross-sectional deformation pressure sensing

The cross-sectional deformation pressure sensor (CSDPS) consists of a channel with a flat ceiling that deforms due to pressure. The change in displacement can capacitively be detected as described in [1] and [11]. This capacitive sensor is based on a partially released channel with comb shaped electrodes on both sides. At one side, the channel has long fingers ($200\text{ }\mu\text{m}$) and the stator has short fingers ($50\text{ }\mu\text{m}$). The long fingers are slightly bent downwards due to material stress. Increase in pressure will deform the ceiling of the channel upwards, tilting the fingers downwards and increasing the distance between the fingers (Figure 5.2). The fingers at the other side of the channel behave contrariwise, resulting in decreasing distance. This asymmetry allows for a differential readout, which reduces environmental effects, like temperature.

Figure 5.2: Operating principle of a cross-sectional deformation pressure sensor with capacitive readout. The sensor consists of a channel with comb-shaped electrodes at the tip. A pressure deforms the ceiling of the channel and displaces the comb fingers at both sides. asymmetry of the comb finger lengths makes differential measurement possible.

A resistive readout is more straightforward to interface and is less susceptible to crosstalk when other devices on the same chip are interfaced. The design of the capacitive cross-sectional deformation pressure sensor has therefore been altered to support a resistive readout. In this design, the channel is not released, but is fixed in the silicon. Thin-film gold resistors are deposited on top of the channel. The resistors have a meandering shape to increase resistance change. They are connected in a Wheatstone bridge as indicated in Figure 5.3. Two resistors of the bridge are placed over the center of the channel and will elongate due to the deformation; the other two resistors are placed at the sides and will be compressed. The pressure sensors operate inline and do not introduce extra volume or pressure drop. Chapter 6 describes how these sensors can be integrated with other surface channel technology compatible fluid sensors.

Figure 5.3: Illustration of the resistive pressure sensor. The non-circularly shaped channel deforms due to pressure and elongates/compresses the thin-film electrodes on the ceiling.

5.2.1 Finite element model

The membrane of the channel is modeled as a clamped-clamped beam of silicon nitride [13] using a finite element model in COMSOL Multiphysics® 4.4 with the dimensions given in Table 5.1. By integrating the strain on top of the membrane, an approximation of the elongation of the resistors is found. The obtained elongations are also shown in Table 5.1. The resistance for all four resistors is proportional to the elongation:

$$R_i \propto W_R + \Delta W_{R_i}, \quad (5.1)$$

with R_i resistor i , W_R the length of one resistor segment and ΔW_{R_i} the elongation of resistor i . The output voltage u_{out} of the Wheatstone bridge is defined as follows:

$$u_{\text{out}} = \left(\frac{R_2}{R_1 + R_2} - \frac{R_4}{R_3 + R_4} \right) u_{\text{in}}, \quad (5.2)$$

with u_{in} the input voltage of the Wheatstone bridge. Substitution of the numbers from Table 5.1 in Equations 5.1 and 5.2 results in a theoretical sensitivity of $2 \cdot 10^{-5} \text{ bar}^{-1}$.

Table 5.1: Dimensions and results of the finite element model.

Pressure	P	1 bar
Membrane thickness	H_m	$3.5\text{ }\mu\text{m}$
Membrane width	W_m	$72\text{ }\mu\text{m}$
Resistor segment width	W_R	$40\text{ }\mu\text{m}$
Young's modulus	E	295 GPa
R_1 elongation	ΔW_{R_1}	-0.47 nm
R_2 elongation	ΔW_{R_2}	0.94 nm
R_3 elongation	ΔW_{R_3}	0.94 nm
R_4 elongation	ΔW_{R_4}	-0.47 nm

The figure shows a cross-sectional view of a membrane sensor. The membrane is fixed at both ends, indicated by 'fixed' labels and curved arrows. A 'SiRN' layer is shown on the left side of the membrane. A 'pressure' is applied to the top surface of the membrane, indicated by a downward arrow. A color scale on the right indicates strain, ranging from -6.5×10^{-5} (dark red) to 6.5×10^{-5} (dark blue), with intermediate values at -40, -20, 0, 20, 40, and 60. A coordinate system with 'x' and 'z' axes is shown at the bottom left.

Figure 5.4: Illustration of the finite element simulation with boundary conditions of the membrane of a cross-sectional deformation pressure sensors. The color represents the displacement.

5.2.2 Experimental setup

The device is fabricated using conventional surface channel technology described in Subsection 3.2.2. A scanning electron microscopy image of the device is shown in Figure 5.5. The chip is mounted and wire bonded on a printed circuit board using the specialized interfacing method described in Subsection 3.6.1. The inlet is connected to a nitrogen gas supply. The outlet is connected to a mass flow controller.

Figure 5.6 shows the electronic interfacing. The Wheatstone bridges are fed by a harmonic signal. The resistances of the tracks are in the order of $100\text{ }\Omega$. A higher amplitude leads to a higher output voltage and therefore a higher signal-to-noise ratio. However, it also leads to a higher current and thus a higher temperature of the resistors. Modeling the temperature behavior of the complex resistor structures on top of the silicon nitride membrane is challenging and beyond the scope of this dissertation. An amplitude of 100 mV results in less than $100\text{ }\mu\text{W}$ and is assumed to be small enough to not cause significant heating. With a modeled sensitivity of $2 \cdot 10^{-5}\text{ bar}^{-1}$, the sensitivity of the output voltage is $2\text{ }\mu\text{V bar}^{-1}$.

Figure 5.5: Scanning electron microscopy image of the pressure sensor. In this case, the sensor contains three parallel channels to reduce the pressure drop. This allowed for the integration of three pressure sensors.

Figure 5.6: The electronic interfacing of both sensors. A 100 mV signal with a frequency of ~ 1 kHz is fed to the Wheatstone bridge. The signal from the readout terminals is demodulated and filtered by a lock-in amplifier.

A relatively low carrier frequency for lock-in amplification of ~ 1 kHz is chosen. This frequency is high enough for lock-in amplification, but is not in the range that capacitive coupling and electromagnetic interference become issues. The output terminals are connected to the differential input of a lock-in amplifier (Stanford Research Systems SR830). The lock-in amplifier also supplies the carrier frequency.

5.2.3 Characterization

For the pressure sensor calibration, gauge pressures are applied using a pressure controller (Bronkhorst® EL-PRESS) from 0 bar to 1 bar in steps of 0.1 bar. The results in Figure 5.7 show no hysteresis and a linear response with a sensitivity of $4 \mu\text{V bar}^{-1}$, i.e. $4 \cdot 10^{-5} \text{ bar}^{-1}$, which is in the same order of magnitude as the model describes.. An offset of approximately 1 mV is observed and digitally subtracted from the measurement results. Robustness tests show that the sensor can handle at least 10 bar.

Figure 5.7: Measurement results for increasing and decreasing gauge pressures ranging from 0 bar to 1 bar. The measurement results are corrected for offset.

The influence of temperature on the sensor has not been investigated; the sensor has been characterized at room temperature. Reducing the offset of the Wheatstone bridge should be considered in a future design, by an improved resistor design or by an analog offset reduction circuit. The sensitivity ($4 \cdot 10^{-5} \text{ bar}^{-1}$) of the sensor is two orders of magnitude lower than other Wheatstone bridge-based pressure sensors ($6 \cdot 10^{-3} \text{ bar}^{-1}$) [14]. The resistors are made of gold and thus have a lower dependence on strain than piezoresistive materials. However, the device is therefore completely compatible with surface channel technology. Besides, this sensor operates throughflow with relatively narrow membranes ($72 \mu\text{m}$ compared to 1 cm [14]).

5.3 Longitudinal channel deformation pressure sensing

Pressures in a semi-circular channel have more consequences than the deformation of the ceiling. The asymmetry of the channel causes a difference in stiffness between the upper and lower part of the suspended channel. The tip will therefore bend upwards when a positive pressure is applied inside. The longitudinal channel deformation pressure sensor (LCDPS) is based on this effect. The pressure in the channel causes a force on the walls. Transversal forces cancel each other around the channel. However, the pressure results in a net longitudinal force at the tip. The asymmetry causes the channel to bend upwards, this can be detected by a change in capacitance as is illustrated in Figure 5.8.

Figure 5.8: The asymmetry of the channel's cross-section (Figure 5.1d) causes a variation in stiffness along z -direction, forcing it to deform under pressure. The electrodes at the tip enable capacitive readout.

5.3.1 Analytical model

Modeling of the channel in Figure 5.8 is done by applying two simplifications.

- Only half (one member of the pair of channels) of the U-shaped channel is modeled as a straight channel, since the full U-shaped channel has twice the stiffness but also twice the force at the tip, which results in the same displacement.
- The complicated channel section is divided in three parts: the ceiling, the channel walls and the floor. The three parts are simplified to an asymmetric I-beam, as is illustrated in Figure 5.9.

The asymmetric I-beam is analyzed by first defining the geometry, including the absolute centers and the neutral axes. Then, a net torque is derived from the longitudinal force on the asymmetric I-beam. Finally, the second moments of area is derived and used in combination with the net torque to find the displacement at the tip.

Figure 5.9: Cross-section of the channel and its equivalent I-beam model with the definition of the dimensions.

The three solid surfaces, A_1 , A_2 and A_3 , have the following area:

$$A_i = W_i H_i, \quad (5.3)$$

with W_i and H_i the width and height of surface area i respectively. These surfaces have absolute centers $z_{c,i}$:

$$z_{c,1} = H_3 + H_2 + \left(\frac{H_1}{2}\right), \quad (5.4)$$

$$z_{c,2} = H_3 + \left(\frac{H_2}{2}\right), \quad (5.5)$$

$$z_{c,3} = \left(\frac{H_3}{2}\right). \quad (5.6)$$

The neutral axis z_n is defined as the axis through the I-beam where the stresses and strains are zero. Therefore, it passes through the centroid. The z -coordinate z_n can be found by the above defined absolute centers and the surface areas of the parts [15]:

$$z_n = \frac{z_{c,1}A_1 + z_{c,2}A_2 + z_{c,3}A_3}{A_1 + A_2 + A_3}. \quad (5.7)$$

The channel deforms as a result of the torque τ caused by the net force F due to the pressure P and the distance z_m between the neutral axis z_n and the point on which the force acts (in the centroid of surface A_f , approximated by $z_{c,2}$):

$$\tau(P) = F(P) z_m = P A_F z_m = P A_F (z_n - z_{c,2}), \quad (5.8)$$

with A_F the surface area on which the pressure acts. From the Huygens-Steiner theorem [15], the second moments of area I_i can be derived for the three parts.

$$I_1 = \frac{1}{12} W_1 H_1^3 + A_1 (z_1 - z_n)^2, \quad (5.9)$$

$$I_2 = \frac{1}{12} W_2 H_2^3 + A_2 (z_n - z_2)^2, \quad (5.10)$$

$$I_3 = \frac{1}{12} W_3 H_3^3 + A_3 (z_n - z_3)^2. \quad (5.11)$$

The sum

$$I = \sum I_i = I_1 + I_2 + I_3 \quad (5.12)$$

is the second moment of area of the I-beam. A derivative of the beam theory of Euler-Bernoulli states that torque τ , the Young's modulus E , the second moment of area I and the displacement $\Delta z(x)$ are related [16]:

$$\tau(P) = -EI \frac{d^2 \Delta z(x)}{dx^2}, \quad (5.13)$$

or, rewritten to find the displacement at the tip:

$$\Delta z(P) = \int_0^{L_0} \int \frac{\tau(P)}{EI} dx dx = \frac{\tau(P) L_0^2}{2EI}, \quad (5.14)$$

with L_0 the length of the I-beam. The dimensions and constants can be found in Table 5.2. Inner surface area A_F is derived from a microscope image of a section of the channel, the Young's modulus E for nitride is taken from literature [13].

Table 5.2: Dimensions and constants for the model.

Top flap width	W_1	60 μm
Side wall width	W_2	2 · 0.5 μm
Bottom width	W_3	9 μm
Top flap height	H_1	3.6 μm
Side wall height	H_2	37 μm
Bottom height	H_3	0.5 μm
Default vertical offset at the tip of the channel	z_0	3.5 μm
Lengths of the tested channels	L_0	1000 μm
		1750 μm
		2500 μm
Inner surface area of the channel	A_F	1400 μm^2
Young's modulus	E	295 GPa

5.3.2 Finite element models

The mechanics of the structure are also simulated using COMSOL Multiphysics® 4.4 with the ‘solid mechanics’ physics. A longitudinal channel deformation pressure sensor is drawn using the dimensions in Table 5.2. A custom material with the specified Young’s modulus is used to model the silicon nitride. To reduce simulation time and memory usage, only half of the structure is simulated using a ‘mirror’ boundary. A force, corresponding to a pressure of 1 bar at the surface, is applied at the end of the channel. The length of the channel is a variable parameter and swept from 600 μm to 3000 μm . Figure 5.10 shows an illustration of the 3D-structure with the boundary conditions.

Figure 5.10: Illustration of the finite element simulation with boundary conditions of the longitudinal channel deformation pressure sensors of 1000 μm . The color represents the displacement.

To verify the I-beam approximation of the analytical model, a similar finite element simulation is done. In this simulation, an I-beam with the dimensions from Table 5.2 is drawn with the same material and same boundary conditions, but, since this structure needs less mesh elements, the mirror condition was not needed. Figure 5.11 shows an illustration of the structure with the boundary conditions.

The analytical model and both finite element models are combined with a capacitance model and are compared with measurement results below.

Figure 5.11: Illustration of the finite element simulation with boundary conditions of the I-beam approximation with a length of 1000 μm . The color represents the displacement.

5.3.3 Capacitance model

The readout capacitor consists of two electrodes: one is attached to the tip of the structure and the other one is fixed. The capacitance is therefore a function of the displacement $\Delta z(P)$, and thus also a function of pressure.

The electrodes of the capacitor are comb-shaped and are simulated with COM-SOL Multiphysics® 4.4. With physics ‘electrostatics’, the capacitances for multiple electrode gaps, varying from $-30 \mu\text{m}$ to $30 \mu\text{m}$, are simulated (Figure 5.12).

Figure 5.12: Illustration of the finite element simulations of the electrostatic behavior of the electrodes. A summary of the boundary conditions, geometry and an impression of the result is shown. The resulting capacitances for different gaps $\Delta z(P)$ are plotted in Figure 5.13.

The simulation results are shown in Figure 5.13. The results are fitted and form the capacitive model $C_{\text{model}}(\Delta z(P))$:

$$C_{\text{model}}(\Delta z(P)) = -1.903 \cdot 10^{-4} \Delta z(P)^2 - 6.364 \cdot 10^{-12} \Delta z(P) + 5.622 \cdot 10^{-14}. \quad (5.15)$$

Figure 5.13: Simulation results (points) of the capacitance as a result of displacement. The polynomial fit for $|\Delta z| < 6 \mu\text{m}$ is used as model $C_{\text{model}}(\Delta z(P))$.

5.3.4 Model comparison

An initial displacement z_0 will be there due to internal stress from the fabrication process. This displacement is added as offset to the three mechanical models. The resulting deformations as a function of pressure are printed in Figure 5.14.

By combining the deformations of the mechanical models with the capacitance model, the capacitances for a pressure of 1 bar are calculated as a function of the length of the channel and are plotted in Figure 5.15.

The I-beam model is a reasonable approximation, since the results of both finite element simulations and the analytical model are very similar.

Figure 5.14: Results of the analytical and both finite element models. The deformation, with correction for offset, for channel sizes of 1000 μm, 1750 μm and 2500 μm are plotted for pressures from 0 bar to 2.5 bar. The approximated capacitance is based on the capacitance model as described by Equation 5.15.

Figure 5.15: Analytical result and finite element simulations of the scalability of the longitudinal channel deformation pressure sensors.

5.3.5 Experimental setup

The experiment is done with the three longitudinal channel deformation pressure sensors in Figure 5.16: channels with a length of 1000 μm , 1750 μm and 2500 μm . The devices are fabricated using silicon-on-insulator-based surface channel technology described in Subsection 3.2.1. A scanning electron microscopy image of the sensors is shown in Figure 5.17.

Three sensors are characterized by applying a gauge pressure with nitrogen between 0 bar and 1 bar using a pressure controller (Bronkhorst® EL-PRESS). The electrical readout is done using a charge amplifier to convert the capacitance to a voltage as described in Subsection 3.5.3. A sine wave ($\sim 1 \cdot 10^4$ Hz, 1 V) is fed to the combs. A lock-in amplifier (Stanford Research Systems SR830) is used to lock in on the frequency and reduce environmental noise. A schematic diagram of the setup, including the electronic readout, is shown in Figure 5.18.

Figure 5.16: Scanning electron microscopy image collage of the three different longitudinal channel deformation pressure sensors used in the experiments.

Figure 5.17: Scanning electron microscopy image of the fabricated longitudinal channel deformation pressure sensor.

Figure 5.18: Measurement setup for the characterization of the longitudinal channel deformation pressure sensor. A N_2 pressure is applied at the sensor using a pressure controller. The capacitance measurements of the sensor is done using a carrier frequency fed to the capacitive readout structure of the sensor. A charge amplifier converted the capacitance to a voltage, which is demodulated using a lock-in amplifier.

5.3.6 Characterization

The measurement results are shown in Figure 5.19. Longer channels result in higher sensitivities: 0.2 fF bar^{-1} for the $1000 \mu\text{m}$ channel, 0.4 fF bar^{-1} for the $1750 \mu\text{m}$ channel and 1 fF bar^{-1} for the $2500 \mu\text{m}$ channel. The calculated results from the analytical model are also plotted and correspond very well to the measurements.

Figure 5.19: Capacitive measurement results for gauge pressures from 0 bar to 1 bar for the different structures with lengths of $1000 \mu\text{m}$, $1750 \mu\text{m}$ and $2500 \mu\text{m}$. The results of the analytical model are also plotted.

The sensitivity is much lower than other capacitive pressure sensors in recently published articles [17, 18], which are 15 pF bar^{-1} and $0.66 \text{ pF log}^{-1}(\text{bar})$ respectively. However, this sensor is made of a material with a relatively high Young's modulus (silicon nitride) and operates throughflow with small internal volumes, deformation due to pressure and electrode sizes are therefore smaller. An improvement in performance can be achieved by including more capacitive readout structures at the channel, increasing the capacitance and enabling more accurate detection of the vibration modes.

The temperature dependence of the pressure sensing mechanism has not been investigated. It is expected that temperature will influence the residual stress and initial bending of the channel. The influence may be reduced by adding a reference sensor without fluid.

5.4 Coriolis mass flow sensor structure pressure sensing

As described in Section 5.3, out of plane bending of U-shaped longitudinal channel deformation pressure sensors can be read out statically to obtain pressure information. The structure of a Coriolis mass flow sensor is very similar to the longitudinal channel deformation pressure sensor. The deformation of a Coriolis mass flow sensor with a length of $2500\text{ }\mu\text{m}$, as indicated in Figure 5.16, is investigated using white light interferometry [19]. The deformation, as plotted in Figure 5.20, is comparable to the model described in Section 5.3 for U-shaped longitudinal channel deformation pressure sensors.

Figure 5.20: Deformation of the suspended channel of a Coriolis mass flow sensor measured by white light interferometry [19]. The model is based on the analytical model in Section 5.3.

By actuating the suspended channels, flow and density measurement are possible while the static displacement, caused by pressure, can still be determined. The ratio between the two modes can be measured using two capacitive readout structures which produce the signals $C_1(t, P)$ and $C_2(t, P)$ as illustrated in Figure 5.22. The pressure can be found by measuring the offset (Figure 5.21c) of $C_1(t, P)$ and $C_2(t, P)$.

Figure 5.21: Movement of a Coriolis mass flow sensor, with (a) the twist mode due to actuation, (b) the swing mode due to the Coriolis force and (c) the static offset due to the pressure.

5.4.1 Analytical model

When one side of the readout structures is considered, the following equation describes the capacitance $C_{1|2}(t, P)$ of the moving comb.

$$C_{1|2}(t, P) = C_{\text{model}}(z_{1|2}(t, P)), \quad (5.16)$$

where $C_{\text{model}}(z_{1|2}(t, P))$ is the capacitance model as a function of position as described in Equation 5.15, and the position $z_{1|2}(t, P)$ is given by:

$$z_{1|2}(t, P) = \Delta z(P) + z_0 + \beta \sin\left(\omega t + \frac{\phi}{2}\right), \quad (5.17)$$

with β a constant factor dependent on the Lorentz actuation and actuation current and z_0 a static displacement due to residual stress in the channel material. The

Figure 5.22: The output signals of the Coriolis mass flow sensor: the phase shift is dependent on the mass flow, the frequency is dependent on the density and the ratio between the harmonics is dependent on the pressure.

capacitance increases with decreasing distance and decreases with increasing distance. A maximum in capacitance is reached when the combs cross each other. This causes the introduction of higher harmonics in the signal, illustrated in Figure 5.22 and described in [11]. A larger offset $\Delta z(P)$, as a result of a higher pressure, leads to smaller higher harmonics. The amplitudes of the first two harmonics are found by discretization of the time-domain signal $C_{1|2}(t,P)$ and applying a fast Fourier transform (FFT). Figure 5.23 shows the magnitudes of the first two harmonics for multiple absolute displacements $\Delta z(P) + z_0$.

The Coriolis mass flow sensor has two combs for phase detection. Both provide a set of harmonics which are dependent on the pressure. The pressure at both comb positions are different when there is a flow, since the channel between the combs has a pressure drop. This effect may be used as a differential pressure flow sensor, like has been done in [20].

Figure 5.23: The normalized amplitudes of the first two harmonics of the Coriolis mass flow sensor as a result of the displacement. Calculated from the analytical model.

5.4.2 Experimental setup

The measurement setup for the experiment is printed in Figure 5.24. The gauge pressure at the outlet of the sensor is controlled between 0 bar and 1 bar in steps of 0.2 bar using a pressure controller and a valve. For each pressure step, the mass flow is varied from 0 mg h^{-1} to 35 mg h^{-1} using a flow controller (Bronkhorst® EL-FLOW) at the inlet of the sensor. A reference pressure sensor is also in line with the flow at the inlet of the sensor.

The device under test is actuated by an actuation controller with feedback, which drives the sensor structure always exactly at the resonance frequency as explained in Subsection 3.3.2. The capacitive measurement is done using a custom built circuit consisting of a charge amplifier and an analog lock-in amplifier (mixer/filter combination) as explained in Subsection 3.5.4. Two commercially available lock-in amplifiers (Stanford Research Systems SR830) are used to lock-in on the actuation frequency and provide the amplitudes, phases and frequencies to a computer. For every unique flow and pressure combination, the amplitudes, phases and frequencies of both the first and second harmonic are obtained.

Figure 5.24: Measurement setup for dynamic characterization. A N_2 flow is controlled at the inlet of the sensor and a pressure is controlled at the outlet of the sensor. A pressure sensor measures also the pressure at the inlet. The sensor is actuated using an actuation controller at its resonance frequency. The capacitive sensing is done similar to the static measurement. But in this situation, custom built electronics were used to do the demodulation and lock-in amplifiers (on the actuation frequency) were used to measure the magnitudes, phases and frequencies of the two readout signals corresponding to the capacitance values.

5.4.3 Characterization

For increasing pressure, the first harmonic gains in amplitude while the second harmonic loses amplitude (Figure 5.25), as is predicted by the model. The pressure in the device under test is both dependent on the controlled pressure at the outlet and the controlled flow at the inlet, clearly visible in the results as increasing groups of points with the same color.

Figure 5.25: Harmonics measurement for gauge pressures from 0 bar to 1.4 bar (measured by the inline pressure sensor at the inlet) for the Coriolis mass flow sensor. The different colors represent the different pressure setpoints of the pressure controller.

The phase shift as a result of the flow is also measured and is shown in Figure 5.26. The resulted phase shift is also dependent on pressure.

Figure 5.26: Flow measurement for gauge pressures from 0 bar to 1 bar (measured by the pressure controller) for the Coriolis mass flow sensor.

In Figure 5.27, the difference of the second harmonics between the two combs as a function of the flow is shown. This difference is proportional to the pressure drop between the two combs and is a measure for the flow, similar to how differential flow sensors operate.

Figure 5.27: Normalized amplitude of the difference of the second harmonic between the two combs for gauge pressures from 0 bar to 1 bar (measured by the pressure controller) plotted against mass flow.

The flow measurements of Figure 5.26 are corrected for the pressure dependence, using the information from the pressure measurement of Figure 5.25. The results are presented in Figure 5.28 and show how the proposed pressure sensing mechanism can significantly improve flow measurements by removing pressure dependence.

Figure 5.28: Flow measurement based on the results of Figure 5.26 with pressure correction using the results of Figure 5.25. Corrected for offset.

The concept of pressure sensing using the static deformation of the Coriolis mass flow sensor is validated and two examples of practical uses (i.e. differential pressure flow sensing and pressure dependence compensation for flow sensors) are demonstrated. An in-depth quantitative analysis of the static deformation and mode shapes of the Coriolis mass flow sensor and the influence on the harmonics of the output signal is needed for reliable flow/pressure sensing.

An implementation of a micro Coriolis mass flow sensor with multiple readout structures is shown in Figure 4.16. This enables improved static deformation and mode analysis. Also better results may be obtained when smart algorithms for sensor fusion are implemented.

5.5 Concluding remarks

- A design for a cross-sectional channel deformation pressure sensor is presented and validated. A hysteresis-free transfer of the resistive readout with a sensitivity of $4 \cdot 10^{-5} \text{ bar}^{-1}$ for gauge pressures ranging from 0 bar to 1 bar is shown. This sensor operates throughflow and can therefore be easily integrated in the fixed channels of a microfluidic system fabricated using surface channel technology. Furthermore, the resistive readout is straightforward to interface and is not susceptible to crosstalk when other devices with a resistive or capacitive readout on the same chip are interfaced.
- Multiple longitudinal channel deformation pressure sensors are designed, fabricated and characterized. The most sensitive design has a validated range of 1 bar with a sensitivity of 1 fF bar^{-1} . This throughflow pressure sensor is easily scalable by changing its length and can be integrated with other microfluidic devices fabricated using surface channel technology. Although the structure might not be the preferred choice for flow/pressure sensor integration because of its capacitive readout with low sensitivity, it gives good insight of the pressure dependent deformation of suspended surface channels.
- The longitudinal channel deformation pressure sensor can be completely integrated in a Coriolis mass flow sensor structure. This enables the sensor to measure pressure in addition to mass flow and density without the need for any modifications to the structure. Measurement of the pressure in a micro Coriolis flow sensor can also be used to compensate for pressure dependence of the flow sensor or for differential pressure flow sensing. In-depth quantitative analyses of the static deformation and mode shapes of the Coriolis mass flow sensor are needed for reliable flow/pressure sensing.

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Fluid parameter sensing

This chapter¹ describes the use of fluid sensors fabricated using surface channel technology for obtaining fluid properties. Real-time measurement of different fluid parameters enables fluid characterization and composition measurements.

The first and second section explain how the resistive cross-sectional deformation pressure sensor can be integrated with a Coriolis mass flow sensor for viscosity measurements of liquids and gases respectively. For liquids, kinematic viscosity derivation using a model based on the Hagen-Poiseuille law is validated with propan-2-ol in water mixtures. For gases (argon and nitrogen), a comprehensive model of the pressure drop related to mass flow is experimentally verified.

The third section covers theory and experiments of density sensing using a Coriolis mass flow sensor. The resonance frequency of this sensor is used as a measure for the density. The addition of resistive cross-sectional deformation pressure sensors on the same chip helps with compensating for the pressure dependence. The sensor is calibrated for liquid propan-2-ol in water mixtures and gases (nitrogen and argon).

Besides density and viscosity, other parameters are interesting for fluid characterization too. The relative permittivity varies significantly between fluids that are chemically related. The relative permittivity sensor, described in the last section, enables non-contact composition measurements of chemicals and is fully compatible with surface channel technology.

¹This chapter is based on the publications [1–4]:

- D. Alveringh, T. V. P. Schut, R. J. Wiegerink, W. Sparreboom, and J. C. Lötters, “Resistive pressure sensors integrated with a Coriolis mass flow sensor,” in *Proceedings of the 19th International Conference on Solid-State Sensors, Actuators and Microsystems (TRANSDUCERS 2017)*. Taipei, Taiwan: IEEE, 2017, pp. 1167–1170;
- J. Groenesteijn, D. Alveringh, T. V. P. Schut, R. J. Wiegerink, W. Sparreboom, and J. C. Lötters, “Micro Coriolis mass flow sensor with integrated resistive pressure sensors,” in *Proceedings of the 3rd Conference on MicroFluidic Handling Systems (MFHS 2017)*, Enschede, the Netherlands, 2017, pp. 16–19;
- T. V. P. Schut, D. Alveringh, W. Sparreboom, J. Groenesteijn, R. J. Wiegerink, and J. C. Lötters, “Fully integrated mass flow, pressure, density and viscosity sensor for both liquids and gases,” in *Proceedings of the 31th IEEE International Conference on Micro Electro Mechanical Systems (MEMS 2018)*. Belfast, United Kingdom: IEEE, 2018, pp. 218–221;
- D. Alveringh, R. J. Wiegerink, and J. C. Lötters, “Inline relative permittivity sensing using silicon electrodes realized in surface channel technology,” in *Proceedings of the 31th IEEE International Conference on Micro Electro Mechanical Systems (MEMS 2018)*. Belfast, United Kingdom: IEEE, 2018, pp. 840–843.

6.1 Introduction

Combining sensors has synergistic potential in sensing fluids. For example, the combination of a thermal flow sensor and a Coriolis mass flow sensor does not only increase the range significantly [5], but also enables the measurement of heat capacity [6]. Or, the combination of a flow sensor and a pressure sensor makes viscosity measurements possible [6]. Besides, the pressure dependence of the Coriolis mass flow sensor can be compensated using pressure sensors. Despite the proven possibility of measuring multiple fluid parameters on a single chip, simultaneous measurements with such a chip were not presented yet.

This chapter describes fluid parameter sensing using the Coriolis mass flow sensor from Chapters 3 and 4, the resistive cross-sectional deformation pressure sensor from Chapter 5 and a relative permittivity sensor that will be introduced. Since all sensors are compatible with surface channel technology, these can all be integrated on a single chip. An artist's impression of such a device is shown in Figure 6.1. This figure shows two resistive cross-sectional deformation pressure sensors integrated both upstream and downstream of a Coriolis mass flow sensor. The Coriolis mass flow sensor provides mass flow measurements, the pressure sensors provide pressure drop measurements. Combination of these measured variables enables the estimation of the kinematic viscosity. The resonance frequency of the Coriolis mass flow sensor also provides a measure for the density of the fluid, estimation of the dynamic viscosity is therefore also possible. Integration of a relative permittivity sensor adds the measurement possibility of another fluid parameter for fluid characterization.

Figure 6.1: Illustration of the integration of two resistive pressure sensors with a capacitive Coriolis mass flow sensor and a relative permittivity sensor on a single chip. The multiple sensors allow for the measurement of flow and pressure drop, and therefore enable the estimation of viscosity.

6.2 Viscosity sensing of liquids

To measure viscosity, the concept illustrated by Figure 6.1, consisting a Coriolis mass flow sensor with two resistive pressures sensors, is realized. Figure 6.2 shows SEM images of the fabricated device.

Figure 6.2: Scanning electron microscopy overview (a) of the chip with the Coriolis mass flow sensor and resistive pressure sensors at both sides. And (b) a close up of the resistive pressure sensor.

6.2.1 Fluid mechanical model

For incompressible Newtonian fluids at low Reynolds numbers, the volume flow Q through the Coriolis mass flow sensor will obey the Hagen-Poiseuille law as is derived in Section 2.3.2:

$$Q = \frac{\pi \Delta P R_{\text{eff}}^4}{8 \eta L_t}, \quad (6.1)$$

with ΔP the pressure drop, R_{eff} the effective channel radius, η the dynamic viscosity and L_t the channel length. The Coriolis mass flow sensor detects mass flow, so rewriting Equation 6.1 for mass flow ($\Phi = \rho Q$, as stated in Section 2.4), the equation becomes:

$$\Phi = \frac{\pi \Delta P \rho R_{\text{eff}}^4}{8 \eta L_t}. \quad (6.2)$$

The kinematic viscosity ν is defined by Equation 2.34:

$$\nu = \frac{\eta}{\rho}, \quad (6.3)$$

and can be substituted in Equation 6.2:

$$\Phi = \frac{\pi \Delta P R_{\text{eff}}^4}{8 \nu L_t}. \quad (6.4)$$

Or, rewritten:

$$\nu = \frac{\pi \Delta P R_{\text{eff}}^4}{8 \Phi L_t}. \quad (6.5)$$

The values used for the total channel length L_t and effective channel radius R_{eff} are 13.41 mm and 31 μm respectively.

6.2.2 Measurement setup

The chip is mounted and wire bonded on a printed circuit board using the specialized interfacing method described in Subsection 3.6.1. Validation of the model is done by applying different fluids at multiple pressures to the sensor. Liquid solutions of propan-2-ol in water of 0 %, 25 %, 50 %, 75 % and 100 % (volume percentages) are applied with gauge pressures of 3 bar, 4 bar, 5 bar and 6 bar at room temperature. The liquids are applied using a reservoir tank, pressurized by nitrogen. Mass flows are varied between 0 g h^{-1} and approximately 12 g h^{-1} .

Figure 6.3 shows the electronic interfacing of both the pressure sensor and the Coriolis mass flow sensor. The Wheatstone bridges are fed by sine wave with an amplitude of 100 mV and a frequency of $\sim 1 \text{ kHz}$. The output terminals are connected to the differential input of a lock-in amplifier as described in Subsection 5.2.2. The readout of the capacitive structures of the Coriolis mass flow sensor is achieved using the synchronous capacitance measurement as specified in Subsection 3.5.4.

Figure 6.3: The electronic interfacing of both sensors. A 100 mV signal with a frequency of ~ 1 kHz is fed to the Wheatstone bridge. The signal from the readout terminals is demodulated and filtered by a lock-in amplifier. The Coriolis mass flow sensor is interfaced using custom demodulation electronics and a lock-in amplifier for frequency and phase detection. Only half of the setup is shown.

6.2.3 Measurement results

Figure 6.4 shows the output signal (phase shift) of the Coriolis mass flow sensor against mass flow. The results are only shown for 3 bar and 4 bar, to reduce the complexity of the plot. It appears that the phase shift of this sensor is almost only dependent on mass flow and independent of pressure or density.

The pressure drop, plotted in Figure 6.5, is proportional to the mass flow. Again,

the results are only shown for 3 bar and 4 bar, to reduce the complexity of the plot. The slope appears to be defined by the fluid parameters, since the pressure drop for propan-2-ol is three times higher than for water.

Figure 6.4: Phase shift as a result of mass flow through the Coriolis mass flow sensor for water and propan-2-ol for gauge pressures of 3 bar (filled symbol) and 4 bar (open symbol) with linear fits. The mass flows were varied between 0 g h⁻¹ and approximately 12 g h⁻¹.

Figure 6.5: The pressure drop over the Coriolis mass flow sensor for water and propan-2-ol for pressures of 3 bar (filled symbol) and 4 bar (open symbol) with linear fits. The mass flows were varied between 0 g h⁻¹ and approximately 12 g h⁻¹.

The measured mass flows and pressure drops are used in the Hagen-Poiseuille-based model (Equation 6.5). The obtained kinematic viscosities are plotted in Figure 6.6 and compared with values from literature [7]. Despite outliers, it appears that the measurement results match with the kinematic viscosities from literature. The outliers could be explained by inaccurate manual dosing and temperature variations. Future characterization of the sensor should be performed in a temperature controlled environment.

Figure 6.6: Results of the viscosity sensor characterization for mixes of propan-2-ol solutions in water for four different pressures averaged for different mass flows.

6.3 Viscosity sensing of gases

The same device can be used to find the viscosity of gases. However, more advanced fluid modeling is needed, since Hagen-Poiseuille law is only valid for incompressible fluids. Gases are compressible and so the density cannot be assumed constant along the channel. Therefore, also volume flow cannot be assumed constant.

6.3.1 Fluid mechanical model

A mathematical relation between mass flow and pressure drop can be derived from the change in energy [8, 9]. In a simple case, when the fluid is incompressible, the potential energy due to pressure will decrease along the fluid path, but the dissipated energy will increase; the total energy will stay constant. Therefore, the change in energy must be zero. For gases in the channels of the sensor, the following equation is used to find the pressure drop and mass flow relation:

$$dE_p + dE_k + dE_f + dE_a = 0 \quad (6.6)$$

with dE_p the change in potential energy of pressure, dE_k the change in kinetic energy due to fluid motion, dE_f the change of dissipated energy due to friction and dE_a the change in energy due to additional losses. Other changes in energy, e.g. due to gravity, are assumed insignificant. Figure 6.7 shows an illustration of the different changes of variables of a fluid moving through a channel.

Figure 6.7: Illustration of the changes in fluid position x , velocity u , specific volume v_{sp} and pressure P . The mass flow Φ is constant for every position in the channel. These variables influence the changes in potential, kinetic and dissipated energy, the total energy E is constant.

Change in potential energy due to pressure difference

The change in potential energy dE_p due to pressure difference dP over an infinitesimal volume dV is equal to:

$$dE_p = dV dP, \quad (6.7)$$

This can be written as a function of infinitesimal mass dm as follows:

$$dE_P = \frac{dm}{\rho(x)} dP. \quad (6.8)$$

with $\rho(x)$ the density at position x . The reciprocal of density is the specific volume ($v_{sp} = 1/\rho$). With specific volume v_{sp} at position x , equation 6.8 becomes:

$$dE_P = dm v_{sp}(x) dP. \quad (6.9)$$

Change in kinetic energy due to change in fluid motion

When a fluid is moving with flow velocity u at position x , the infinitesimal kinetic energy for mass dm is equal to

$$dE_k = dm \frac{u(x)}{\alpha} du. \quad (6.10)$$

with α a correction factor, which is 1/2 for fully developed laminar flow [8]. This correction factor compensates for the non-uniform velocity profile of the flow. The flow velocity $u(x)$ can differ along the channel. The mass flow Φ , however, is equal for each position in the channel. Equation 6.10 can be rewritten as a function of mass flow Φ and specific volume $v_{sp}(x)$ using:

$$u(x) = \frac{\Phi v_{sp}(x)}{A_i}, \quad (6.11)$$

with A_i the surface area of the cross-section of the channel. Therefore:

$$dE_k = dm \frac{\Phi v_{sp}(x)}{\alpha A_i} d\left(\frac{\Phi v_{sp}(x)}{A_i}\right) = dm v_{sp}(x) \frac{\Phi^2}{\alpha A_i^2} dv_{sp}. \quad (6.12)$$

Change of energy dissipation due to friction

As a result of shear friction force F_f , work dE_f is done by the fluid by moving over distance dx :

$$dE_f = F_f dx. \quad (6.13)$$

This shear force is the resultant force caused by the internal viscous forces in the flow. The derivation of the Hagen-Poiseuille law in Section 2.3.2 is based on these viscous forces. This derivation results in a function for the pressure drop ΔP_f and is, by rewriting Equation 2.14, equal to:

$$\Delta P_f = \frac{8\gamma\eta L_t Q}{\pi R_{eff}^4}, \quad (6.14)$$

with η the dynamic viscosity, L_t the total length of the channel, Q the volume flow and R_{eff} the effective channel radius. A correction factor γ is added to correct for any non-ideal effects, like the roughness and non-circularity of the channel. For an infinitesimal distance dx of the channel, the pressure drop due to friction dP_f becomes:

$$dP_f = \frac{8\gamma\eta Q}{\pi R^4} dx. \quad (6.15)$$

When this pressure is applied to the inner surface area A_i of the channel, the total force F_f on this surface area is:

$$F_f = dP_f A_i = \frac{8\gamma A_i \eta Q}{\pi R^4} dx, \quad (6.16)$$

or, rewritten as a function of mass flow Φ :

$$F_f = v_{\text{sp}}(x) \frac{8\gamma A_i \eta \Phi}{\pi R^4} dx. \quad (6.17)$$

The distance dx can be rewritten using following equation:

$$dx = \frac{1}{A_i} dV = \frac{v_{\text{sp}}(x)}{A_i} dm, \quad (6.18)$$

and thus, Equation 6.17 can be rewritten as a function of infinitesimal mass dm :

$$F_f = dm v_{\text{sp}}(x)^2 \frac{8\gamma\eta\Phi}{\pi R^4}. \quad (6.19)$$

For simplification, the effective radius can be written as inner surface area ($A_i = \pi R_{\text{eff}}^2$). Equation 6.19 substituted in Equation 6.13 leads to:

$$dE_f = dm v_{\text{sp}}(x)^2 \frac{8\gamma\eta\pi\Phi}{A_i^2} dx. \quad (6.20)$$

Change of energy dissipation due to additional losses

Bends in the channel cause additional losses, since the flow needs to redevelop after each bend. This effect is modeled in literature [10] as a pressure drop ΔP_a . The change in energy becomes in that case:

$$dE_a = \Delta P_a A_i dx, \quad (6.21)$$

with:

$$\Delta P_a = \kappa \frac{u(x)^2}{v_{\text{sp}}(x)} = \kappa \frac{\Phi^2 v_{\text{sp}}(x)}{2A_i^2}. \quad (6.22)$$

with κ the additional losses factor. This pressure drop is the full pressure drop over the full length of the channel with κ dependent on the number and the shape of the bends. For an infinitesimal pressure drop dP_a over a channel length dx , it is expected to scale linearly:

$$dP_a = \Delta P_a \frac{dx}{L_t} = \kappa \frac{\Phi^2 v_{sp}(x)}{2A_i^2} \frac{dx}{L_t}, \quad (6.23)$$

with L_t the total length of the channel. Infinitesimal distance dx can be written as an infinitesimal mass dm using Equation 6.18:

$$dP_a = dm v_{sp}(x)^2 \frac{\kappa \Phi^2}{2A_i^3 L_t}. \quad (6.24)$$

The change in energy as a result of additional losses becomes:

$$dE_a = dm v_{sp}(x)^2 \frac{\kappa \Phi^2}{2A_i^3 L_t} A_i dx = dm v_{sp}(x)^2 \frac{\kappa \Phi^2}{2A_i^2 L_t} dx. \quad (6.25)$$

Energy function

The terms in the energy function (Equation 6.6) can now be substituted by the individual energy definitions in Equations 6.9, 6.12, 6.20 and 6.25.

$$\begin{aligned} 0 = & dm v_{sp}(x) dP \\ & + dm v_{sp}(x) \frac{\Phi^2}{\alpha A_i^2} dv_{sp} \\ & + dm v_{sp}(x)^2 \frac{8\gamma\eta\pi\Phi}{A_i^2} dx \\ & + dm v_{sp}(x)^2 \frac{\kappa \Phi^2}{2A_i^2 L_t} dx. \end{aligned} \quad (6.26)$$

Dividing by dm and $v_{sp}(x)^2$ leads to:

$$\frac{1}{v_{sp}(x)} dP + \frac{\Phi^2}{\alpha v_{sp}(x) A_i^2} dv_{sp} + \frac{8\gamma\eta\pi\Phi}{A_i^2} dx + \frac{\kappa \Phi^2}{2A_i^2 L_t} dx = 0. \quad (6.27)$$

Now, the integration along the channel can be performed. Note that at $x = 0$, the pressure is equal to P_1 and the specific volume is equal to $v_{sp,1}$. And at $x = L$, the pressure is equal to P_2 and the specific volume is equal to $v_{sp,2}$.

$$\int_{P_1}^{P_2} \frac{1}{v_{sp}(x)} dP + \int_{v_{sp,1}}^{v_{sp,2}} \frac{\Phi^2}{\alpha v_{sp}(x) A_i^2} dv_{sp} + \int_0^{L_t} \frac{8\gamma\eta\pi\Phi}{A_i^2} dx + \int_0^{L_t} \frac{\kappa \Phi^2}{2A_i^2 L_t} dx = 0. \quad (6.28)$$

The gas is assumed to be ideal. This means that the ideal gas law could be used to relate the specific volume $v_{sp}(x)$ to a pressure $P(x)$ at position x :

$$P(x)v_{sp}(x) = R_{sp}T, \quad (6.29)$$

with R_{sp} the specific gas constant. In this case, the integral becomes:

$$\int_{P_1}^{P_2} \frac{P(x)}{R_{sp}T} dP + \int_{v_{sp,1}}^{v_{sp,2}} 2 \frac{\Phi^2}{\alpha v_{sp}(x) A_i^2} dv_{sp} + \int_0^{L_t} \frac{8\gamma\eta\pi\Phi}{A_i^2} dx + \int_0^{L_t} \frac{\kappa\Phi^2}{2A_i^2 L_t} dx = 0. \quad (6.30)$$

Integration leads to:

$$\frac{P_2^2 - P_1^2}{2R_{sp}T} + \frac{1}{\alpha} \ln\left(\frac{v_{sp,2}}{v_{sp,1}}\right) \frac{\Phi^2}{A_i^2} + \frac{8\gamma\eta\pi L_t \Phi}{A_i^2} + \frac{\kappa\Phi^2}{2A_i^2} = 0, \quad (6.31)$$

or:

$$\frac{P_2^2 - P_1^2}{2R_{sp}T} + \frac{\Phi^2}{A_i^2} \left(\frac{1}{\alpha} \ln\left(\frac{v_{sp,2}}{v_{sp,1}}\right) + \frac{\kappa}{2} \right) + \frac{8\gamma\eta\pi L_t \Phi}{A_i^2} = 0. \quad (6.32)$$

Again, the specific volumes in above equation can be rewritten as a function of pressure:

$$\frac{P_2^2 - P_1^2}{2R_{sp}T} + \frac{\Phi^2}{A_i^2} \left(\frac{1}{\alpha} \ln\left(\frac{P_1}{P_2}\right) + \frac{\kappa}{2} \right) + \frac{8\gamma\eta\pi L_t \Phi}{A_i^2} = 0. \quad (6.33)$$

Equation 6.33 holds for laminar compressible flows in a circular channel without gravity. In literature, it is usually written as a function of a friction factor with multiple variables combined [8] or even with substituted numbers. Since the relation of the dynamic viscosity η with mass flow Φ and pressures P_1 and P_2 is interesting for viscosity sensing, this equation is not further simplified. However, Equation 6.33 is a quadratic equation, it can therefore simply be solved for mass flow Φ as a function of pressures P_1 and P_2 using the quadratic formula:

$$\Phi = \frac{\alpha \left(\sqrt{\frac{2A_i^2}{RT} \left(\frac{\kappa}{2} + \frac{1}{\alpha} \ln\left(\frac{P_1}{P_2}\right) \right) (P_1^2 - P_2^2) + 64\gamma^2 L_t^2 \pi^2 \eta^2 - 8\gamma\pi L_t \eta} \right)}{2 \ln\left(\frac{P_1}{P_2}\right) + \alpha \kappa}. \quad (6.34)$$

Viscosity

The dynamic viscosity η can be derived from Equation 6.33:

$$\eta = \frac{A_i^2}{8\pi L_t \Phi} \left(\frac{P_1^2 - P_2^2}{2R_{sp}T} - \frac{\Phi^2}{A_i^2} \left(\frac{1}{\alpha} \ln\left(\frac{P_1}{P_2}\right) + \frac{\kappa}{2} \right) \right). \quad (6.35)$$

6.3.2 Experimental results

Relations 6.34 and 6.35 are fitted using experimental results. The same setup as for liquids (Figure 6.3) is used. Nitrogen and argon flows are applied at 5 bar, 6 bar, and 7 bar with flows ranging from 0 g h^{-1} to 6 g h^{-1} . The phase shift of the Coriolis mass flow sensor as a result of mass flow is plotted in Figure 6.8. The pressure drop is plotted in Figure 6.9. The lines in the latter figure represent the model from Equation 6.34. The used constants found by manually fitting are shown in Table 6.1.

Table 6.1: Constants for the model for the pressure drop of gas flows.

Total channel length	L_t	13.41 mm
Effective channel radius	R_{eff}	$34.5\text{ }\mu\text{m}$
Specific gas constant for nitrogen	$R_{\text{sp},\text{N}}$	$296.8\text{ J kg}^{-1}\text{ K}^{-1}$
Specific gas constant for argon	$R_{\text{sp},\text{Ar}}$	$208\text{ J kg}^{-1}\text{ K}^{-1}$
Temperature	T	293 K
Flow correction factor	α	0.5
Correction factor	γ	1.9
Additional losses factor	κ	6

Figure 6.8: Measured phase shift from the Coriolis mass flow sensor for nitrogen and argon for two different pressures.

Using Equation 6.35, the viscosity of the gas from the data of the sensor can be obtained. Results are plotted in Figure 6.10.

Figure 6.9: Measured pressure drop from the resistive pressure sensors for nitrogen and argon for two different pressures.

Figure 6.10: Viscosity for argon and nitrogen using the fitted model and data from Figures 6.8 and 6.9.

A first attempt to sense viscosity of gases using the integrated mass flow and pressure sensor is demonstrated. However, fit parameters are needed to match the model to the measurement results. The model consists of many constants (e.g. effective channel radius), all are estimated and have an uncertainty. Furthermore, the channel structure is complex, since it consists of multiple bends and a rough surface. Thorough and structural experimental analyses of the pressure drops of surface channels are needed to improve the model. For example, measuring the pressure drop of straight surface channels with varying diameters could validate the model without bends.

6.4 Density sensing of fluids

The micro Coriolis mass flow sensor can mechanically be seen as a second order rotational mass spring damper system. Inherent to this, the structure has a resonance frequency dependent on its mechanical parameters, and also on the fluid inside the channel as reviewed in Section 2.4. Multiple electronic actuation circuits to detect and drive the sensor at its resonance frequency are proposed in Section 3.3. When calibrated properly, the resonance frequency can be used as a measure for the density of the fluid.

6.4.1 Fluid mechanical model

The resonance frequency of a second order mechanical system in the rotation domain is dependent on the rotational stiffness K and the mass moment of inertia J . This expression is derived from Equation 4.15 in Section 4.2.1.

$$\omega_0 = \sqrt{\frac{K}{J}}, \quad (6.36)$$

Due to the pressure inside the channel, the channel deforms as discussed in Chapter 5. This is expected to affect the rotational stiffness. When this effect is assumed linear, it can be modeled as follows:

$$K = K_0(1 + \beta P), \quad (6.37)$$

with K_0 the initial stiffness of the channel without pressure. The mass moment of inertia J is equal to the sum of the mass moment of inertia of the channel J_{ch} and the mass moment of inertia J_{f} of the fluid inside the channel:

$$J = J_{\text{ch}} + J_{\text{f}}. \quad (6.38)$$

The mass moment of inertia J_{ch} of the channel for the twist mode is derived in Section 4.2.1:

$$J_{\text{ch}} = \frac{1}{7} m_{\text{ch}} W^2, \quad (6.39)$$

with m_{ch} the mass of the channel and W the length of the channel. The mass moment of inertia J_{f} of the fluid as a function of the density ρ_{f} is:

$$J_{\text{f}} = \frac{1}{7} \rho_{\text{f}} V_{\text{L}} W^2, \quad (6.40)$$

with V_{L} the volume inside the channel. Due to pressure deformations, the volume increases. It is assumed that this is an insignificant effect.

Substitution of Equations 6.39 and 6.40 in 6.36 leads to a model for the resonance frequency of the Coriolis sensor as a function of the density of the fluid in the channel

ρ_f and pressure P :

$$\omega_0 = \sqrt{\frac{7K_0(1 + \beta P)}{W^2(m_{ch} + \rho_f V_f)}}. \quad (6.41)$$

The used dimensions of the sensor are in Table 6.2. By fitting parameters K_0 and β , Equation 6.41 provides a calibration equation for density sensing using a Coriolis mass flow sensor and can be rewritten as:

$$\rho_f = \frac{7K_0(1 + \beta P)}{\omega_0^2 W^2 V_{ch}} - \frac{m_{ch}}{V_{ch}}. \quad (6.42)$$

Table 6.2: Dimensions of the Coriolis mass flow for density sensing.

Channel segment width	W	4 mm
Total channel length	L_t	13.41 mm
Effective channel radius	R_{eff}	34.5 μm
Channel volume	$V_{ch} = \pi R_{eff}^2 L_t$	$5 \cdot 10^{-11} \text{ m}^3$
Channel mass	m_{ch}	14 μg

6.4.2 Measurement setup

Figure 6.11 shows the relevant part of the measurement setup for density sensing. The density measurements are done simultaneously with the viscosity measurements explained in Sections 6.2 and 6.3 using the measurement setup shown in Figure 6.3.

6.4.3 Measurement results

Figure 6.12 shows the measured resonance frequency of the Coriolis mass flow sensor with the fitted model from Equation 6.41 for liquids. Figure 6.13 shows the results for gas measurements.

Figure 6.11: Relevant density sensing setup based on the full setup of Figure 6.11. Pressures with nitrogen and argon are applied using a valve controlled by hand. Liquids can be applied with this pre-pressure on a liquid reservoir. The resonance frequency is detected using the synchronous capacitive readout of the Coriolis mass flow sensor.

Figure 6.12: The resonance frequency of the Coriolis mass flow sensor for water and propan-2-ol for pressures between 3 bar and 6 bar. The lines represent the fitted models.

Figure 6.13: The resonance frequency of the Coriolis mass flow sensor for nitrogen and argon for pressures between 5 bar and 8 bar. The lines represent the fitted models.

The values for fitting variables K_0 and β are shown in Table 6.3. The fitted rotational stiffness and pressure dependence have standard deviations of $1 \cdot 10^{-7} \text{ Nm rad}^{-1}$ and $3 \cdot 10^{-9} \text{ bar}^{-1}$ respectively for the different liquid mixtures, it is therefore assumed that there is no significant dependence on the concentration. As expected, the frequency is very dependent on density and slightly dependent on pressure.

The model from Equation 6.42 is used with the fitting variables for water from Table 6.3 to obtain the density. The results are plotted in Figure 6.14. Also reference density values from literature are shown in the same figure [7].

It appears that the resulted measured densities became pressure independent with the presented model and that accurate density sensing within a maximum error of 1.5 % is feasible for liquids. For gases, the densities are approximately three orders of magnitude lower and are able to be measured as proven by measurements. Yet, a thorough calibration of the sensor is needed, the presented model with fixed constants is not sufficient to distinguish gases by density. For compressible fluids, i.e. gases, and large flows, the density is variable along the channel.

Table 6.3: Fitting variables for the density model with the rotational stiffness K_0 and the pressure dependence β .

Propan-2-ol in water	Rot. stiffness K_0 (N m rad ⁻¹)	Pressure dep. β (bar ⁻¹)
0 %	$9.667 \cdot 10^{-6}$	$9.6 \cdot 10^{-8}$
25 %	$9.480 \cdot 10^{-6}$	$10 \cdot 10^{-8}$
50 %	$9.471 \cdot 10^{-6}$	$9.3 \cdot 10^{-8}$
75 %	$9.435 \cdot 10^{-6}$	$9.7 \cdot 10^{-8}$
100 %	$9.597 \cdot 10^{-6}$	$10 \cdot 10^{-8}$
Nitrogen	$9.470 \cdot 10^{-6}$	$7.7 \cdot 10^{-8}$
Argon	$9.490 \cdot 10^{-6}$	$6.5 \cdot 10^{-8}$

Figure 6.14: Results of the density sensor characterization for mixes of propan-2-ol solutions in water for four different pressures averaged for different mass flows.

6.5 Relative permittivity sensing of liquids

The relative permittivity can roughly be seen as the resistance of a material against an electric field. Although the relative permittivity might seem to be a very specific quantity that is only interesting for specific electronic purposes, it has interesting properties in a more indirect way. The value varies a lot between fluids. E.g. methanol has a relative permittivity of approximately 30 % higher than ethanol [11], while the transparency and density are quite similar. It has therefore great potential for fluid characterization and composition measurements. Latter method has applications in flow chemistry for the production of chemicals and accurate medication delivery using intravenous therapy.

Not much has been published on relative permittivity sensing of fluids. Some US patents [12–16] claim different measurement principles, but are all not microfabricated and cannot be integrated with other sensors. The only microfabricated inline relative permittivity sensor is embedded in the system of Lötters et al. [17]. While the paper shows the significance of relative permittivity sensing for fluid characterization and composition measurement, the sensor has a relatively large electrode distance of 50 μm and it has not been modeled in detail. Therefore, dependence on other fluid parameters and parasitic effects are unknown and cannot be compensated for.

For linear, homogeneous, isotropic and non-dispersive materials as dielectric, the capacitance for a parallel plate capacitor is [18]:

$$C_s(\epsilon_r) = \epsilon_0 \epsilon_r \frac{A}{d} = \beta \epsilon_r, \quad (6.43)$$

with ϵ_0 the constant vacuum permittivity, ϵ_r the relative permittivity, A the surface area of the electrodes and d the distance between the electrodes, all combined in β . As follows directly from this equation, a high sensitivity can be achieved by a high A/d -ratio, i.e. a large electrode area and/or small distance between the electrodes.

6.5.1 Design

Figures 6.15a and 6.15b show an illustration of the proposed sensor. A microfluidic channel, with a channel wall of $< 1 \mu\text{m}$, is realized in the device layer of a silicon-on-insulator wafer and extends sideways into the buried oxide layer. Electrodes are realized at both sides of the channel forming capacitances to the handle layer through the thin channel of fluid. The relative permittivity is found by measuring the impedance between the silicon electrodes and the silicon handle layer. The impedance is estimated by measuring the transfer from a voltage v_i at one electrode to the current i_o at the other electrode, see Figure 6.15c:

$$i_o = \frac{v_b}{Z_s || Z_p}, \quad (6.44)$$

Figure 6.15: Illustration of the relative permittivity sensor in isometric (a) and cross-sectional view (b). The relative permittivity of the fluid in the microchannel can be obtained by measuring the impedance between the two isolated silicon electrodes, modeled as an electrical circuit in (c).

with $\parallel$ the parallel operator, i.e. $Z_a \parallel Z_b = (Z_a^{-1} + Z_b^{-1})^{-1}$, and impedance $Z_i = 1/(j\omega C_n)$. The handle layer voltage v_b is:

$$v_b = i_i (Z_b \parallel Z_s \parallel Z_p), \quad (6.45)$$

and the total current is:

$$i_i = \frac{v_i}{Z_s \parallel Z_p + Z_b \parallel Z_s \parallel Z_p}. \quad (6.46)$$

Substitution leads to an expression for the impedance from v_i to i_o :

$$Z_m = \frac{v_i}{i_o} = \frac{2(C_s(\epsilon_r) + C_p) + C_b}{j\omega(C_s(\epsilon_r) + C_p)^2} = \frac{1}{j\omega C_m}, \quad (6.47)$$

or:

$$C_m = \frac{(C_s(\epsilon_r) + C_p)^2}{2(C_s(\epsilon_r) + C_p) + C_b} = \frac{(\beta\epsilon_r + C_p)^2}{2(\beta\epsilon_r + C_p) + C_b}. \quad (6.48)$$

The fabrication is done using the technology described in subsection 3.2.1. Impor-

tant is that the buried oxide layer etch is carefully timed as illustrated in Figure 6.16, as this forms the sensing channels.

Figure 6.16: Slightly altered fabrication process based on the silicon-on-insulator-based surface channel technology explained in Subsection 3.2.1. Etching more of the buried oxide layer for the fabrication of micro channels between device and handle layer.

Two devices are fabricated:

- a structure to test the conduction between the metal layer to the silicon layer and the isolation of the silicon electrodes (Figure 6.17a);
- the relative permittivity sensor as described above (Figure 6.17b).

The test structure consists of an isolated silicon island. A metal horizontal wire crosses the island on top of the silicon nitride. A vertical wire consists of a metal track that is connected to the silicon island underneath using the etched pit in the silicon nitride. The vertical and horizontal wires cross without contact. The photomask design is shown in Figure 6.17a.

The photomask design of the sensor itself is based on the illustration in Figure 6.15. The channel splits in two for on-chip mixing purposes, but this feature is not used in the experiments presented in this dissertation. Figure 6.17b shows the mask design. Figure 6.18 shows a scanning electron microscopy image of the fabricated test structure. Figure 6.19 shows SEM images of the fabricated sensor and a photograph of the sensor assembled on a printed circuit board.

Figure 6.17: Photomask designs with (a) the structure for testing the isolation of the silicon electrode, and the conduction from metal layer to silicon. There is a wire between bondpads A and C and between B and D. The wires cross, but should not be connected to each other. And (b) the structure of the relative permittivity sensor.

Figure 6.18: SEM image of the fabricated test structure.

Figure 6.19: SEM image of the fabricated device and a photograph of the device adhesively mounted to a printed circuit board.

6.5.2 Characterization

Preliminary to the sensor characterization, the isolation of the silicon electrode is checked using the test structure. Besides, the conductivity of the metal to silicon interfaces is tested using the same structure. The resistance between bondpads A and C in Figure 6.17a was $35\ \Omega$. The resistance between bondpads B and D was similar, $38\ \Omega$. The resistance between bondpads C and D was $> 100\ \text{M}\Omega$. It can be concluded that the island is well isolated and is addressable by connecting it to an external bondpad.

The sensor is characterized at room temperature using the universal modular interfacing method described in Subsection 3.6.2 and an HP4194A impedance analyzer as illustrated in Figure 6.20. Nitrogen, hexane, trichloromethane, propan-2-ol, ethanol and methanol with known relative permittivities [11, 19] are applied using a syringe.

Figure 6.20: Electric and fluidic interfacing. The chip is mounted to a chip holder board. The chip holder board is electrically connected to a main board and fluidically connected to a 3D printed fluid block. An impedance analyzer is connected to the main board in a four-terminal setup. The fluid block is connected to a syringe.

The magnitudes and phases of the impedances are obtained for all fluids in the frequency range from 1 MHz to 10 MHz with a logarithmic sweep in 401 steps. These magnitudes are plotted in Figure 6.21. The measured capacitances C_m (using Equation 6.47) are averaged for all 401 frequency steps. The resulting capacitance of each fluid is plotted in Figure 6.22 as a function of relative permittivity. The figure also shows the fitted theoretical response according to Equation 6.48, with $\beta = 0.82\ \text{pF}$, $C_p = 8\ \text{pF}$ and $C_b = 7.5\ \text{nF}$. These values are in the same order of magnitude as estimated by Equation 6.43 using the measured dimensions from Figure 6.19.

In future work, the measurement setup will be improved with accurate temperature control, since the relative permittivity of many substances is temperature

Figure 6.21: Measured magnitudes of the impedances for different fluids from 1 MHz to 10 MHz.

Figure 6.22: Sensor characterization results against relative permittivity values from literature with model fit.

dependent. By improving the resolution, relative permittivity sensing for gases might be possible. Parasitics (C_p) and drift can be reduced by differential measurements using a second on-chip empty reference sensor.

6.6 Concluding remarks

- A micro Coriolis mass flow sensor has been successfully integrated with two resistive cross-sectional deformation pressure sensors. The device has been characterized and it was shown that the viscosity for liquids can be determined from the mass flow and the pressure drop using a Hagen-Poiseuille-based model. Combining a mass flow sensor with pressure sensors thus has a synergistic advantage. Although the obtained kinematic viscosities of propan-2-ol in water mixtures corresponds to values from literature, future characterization of the sensor should be performed in a temperature controlled environment to investigate its resolution.
- The same sensor chip has been used for gas viscosity measurements. Because of the compressible character of gases, a comprehensive model is derived for the mass flow as a function of the pressure drop. Fit parameters are needed to match the model to the measurement results. The dynamic viscosities of nitrogen and argon are obtained from experiments using this model and correspond to literature. Due to the complexity of the sensing structures and the fluid physics of gases, thorough and structural experimental analyses are needed to fully understand the sensor for measuring gas viscosities.
- The Coriolis mass flow sensor can be used for density sensing of fluids, since its resonance frequency is dependent on the density of the fluid inside the vibrating channel. A universal model for both liquids and gases has been derived and its parameters are found from measurements. The model incorporates compensation for the pressure dependence of the sensor. Accurate density sensing of liquids is experimentally verified with a maximum error of 1.5 % compared to reference values from literature.
- A relative permittivity sensor is designed, fabricated and characterized for different fluids. The sensor is compatible with silicon-on-insulator-based surface channel technology. The response of the sensor shows good correspondence to values from literature. The device enables therefore accurate throughflow, real-time and non-invasive relative permittivity measurements with low internal volumes.

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7

Conclusion and outlook

This chapter summarizes the conclusions of all previous chapters and provides an outlook on the research topic. The conclusion is divided in two parts: the fundamental resolution limits of Coriolis mass flow sensors and the synergy of flow/pressure sensor integration. The outlook consists of recommendations for future research on resolution improvement of Coriolis mass flow sensors, surface channel technology, flow/pressure sensor integration and fluid physics modeling.

7.1 Conclusion

There is a need for sensing different physical quantities of fluids for medical and industrial applications. Many microfabricated pressure, flow, density and viscosity sensors have been developed over the last decades. It has been a trend that each sensor has its own fabrication process and that integration on a single chip is uncommon. Besides, most sensors are not designed to operate in a throughflow configuration. Compared to other flow sensors, microfabricated Coriolis mass flow sensors need an extensive microfluidic fabrication process that also allows fabrication of many other types of devices. Furthermore, Coriolis mass flow sensors are by definition throughflow devices. It therefore enables the integration of other microfluidic devices on the same chip.

The research described in this dissertation is twofold: (1) resolution limit analysis and improvement of Coriolis mass flow sensors and (2) integration of flow and pressure sensors for density and viscosity sensing.

7.1.1 Fundamental resolution limits of Coriolis mass flow sensors

The resolution limitations of Coriolis mass flow sensors can roughly be categorized by the noise from external sources and dependence on other physical quantities, resolution limitations of the readout electronics and the intrinsic thermomechanical noise. First steps are made to analyze the latter phenomenon by modeling the influence of thermomechanical noise on the mechanics of a micro Coriolis mass flow sensor. In an experimental laser Doppler vibrometry setup, the displacement of the channel due to thermomechanical noise is measured for temperatures between 300 K and 700 K. The results show RMS vibration amplitudes of 38 pm to 57 pm over a bandwidth of 13 Hz centered around the resonance frequency, which is in good agreement with the theoretical model. The model has been used to derive a noise equivalent mass flow of 0.3 ng s^{-1} for the Coriolis mass flow sensor with currently the best resolution. Coriolis mass flow sensors may therefore become a serious alternative to thermal flow sensors for measuring extremely low flows, with resolutions presented of 0.02 ng s^{-1} . However, it can be concluded that the Coriolis mass flow sensor with the best resolution, i.e. 14 ng s^{-1} , is still limited by noise in the readout circuitry.

This experiment has been conducted by measuring only a single point at the thermomechanical noise actuated channel. For mode analyses, the movement of multiple points need to be measured. In addition, the phase information between each point needs to be known. For thermomechanical actuated structures, triggering is impossible. An algorithm to recover the phase relation between multiple measured points in a laser Doppler vibrometer setup for a non-triggerable signal is proposed and validated. The algorithm requires a two stage measurement, i.e. the surface has to be scanned with a fixed reference and differentially with a moving reference. The method can be implemented in existing laser Doppler vibrometers and enables mode

analysis of noise actuated structures.

An improved resolution can be achieved by optimizing the placement and geometry of the readout electrodes of the Coriolis mass flow sensor. This makes the sensor output less dependent on the actuated mode and more dependent on the Coriolis force induced mode. A further step can be taken by adding a second pair of electrodes which partly cancel the actuation signal component in the output signal of a Coriolis mass flow sensor. It is shown that this actuation mode cancellation results in higher phase shifts and a lower output signal amplitude. Nevertheless, it improves the resolution of the sensor when the phase detector has limitations in phase-resolution. An increase of sensitivity by a factor of 3 is experimentally demonstrated.

7.1.2 Synergy of flow and pressure sensor integration

Surface channel technology provides a universal way to fabricate microfluidic devices. Suspended microchannels with metal wiring on top can be fabricated with this technology. This enables the realization of throughflow pressure sensors, flow sensors and other mechanical microfluidic sensors.

The first presented design for a surface channel technology compatible pressure sensor in this dissertation can be easily integrated in the fixed channels of a micro Coriolis mass flow sensor. It has a resistive readout based on a Wheatstone bridge and shows a hysteresis-free transfer with a sensitivity of $4 \cdot 10^{-5} \text{ bar}^{-1}$ for gauge pressures ranging from 0 bar to 1 bar. Furthermore, the resistive readout is straightforward to interface and is not susceptible to crosstalk when other devices with a resistive or capacitive readout on the same chip are interfaced.

A second surface channel technology compatible pressure sensor has been presented. This sensor is based on the out-of-plane bending of a suspended microchannel. The deformation is detected capacitively, with an experimentally verified sensitivity of 1 fF bar^{-1} . Although the structure might not be the preferred choice for flow/pressure sensor integration because of potential crosstalk with the capacitive readout of the Coriolis mass flow sensor, it gives good insight in the pressure dependent deformation of suspended surface channels. Furthermore, the micro Coriolis mass flow sensor itself is a suspended microchannel, and can therefore be used as a pressure sensor. By distinguishing the amplitudes of the different harmonics from the Coriolis mass flow sensor readout, pressure information can be detected in addition to mass flow. This can be used to compensate for a pressure dependence of the flow sensor, differential pressure flow sensing or viscosity measurements.

The micro Coriolis mass flow sensor can also be integrated with the resistive cross-sectional deformation pressure sensor. Both sensors on a single chip have been characterized simultaneously. The viscosity for liquids can be determined from the mass flow and the pressure drop using a Hagen-Poiseuille-based model. Combining a mass flow sensor with pressure sensors thus has a synergistic advantage. The obtained kinematic viscosities of propan-2-ol in water mixtures corresponds with values from

literature. The same sensor chip has been used for gas viscosity measurements. Because of the compressible nature of gases, an extensive model is derived for the mass flow as a function of the pressure drop. Fit parameters are needed to match the model to the measurement results. After calibration, the sensor shows good correspondence to values from literature.

The Coriolis mass flow sensor can be used for density sensing of fluids, since its resonance frequency is dependent on the density of the fluid inside the vibrating channel. A universal model for both liquids and gases is derived and its parameters are found from measurements. The model incorporates compensation for the pressure dependence of the sensor. Accurate density sensing of liquids is experimentally verified with a maximum error of 1.5 % compared to reference values from literature. Multiple electronic resonator actuation methods are explained for this purpose.

A relative permittivity sensor is designed, fabricated and characterized for different fluids. The sensor is compatible with silicon-on-insulator-based surface channel technology. The response of the sensor shows good correspondence to values from literature. The device enables therefore accurate throughflow, real-time and non-invasive relative permittivity measurements with low internal volumes.

Due to an increase in complexity of the sensor chips, e.g. the need for simultaneous measurement of capacitive and resistive readout structures, a novel electric and fluidic interfacing platform has been realized. This platform provides a time-efficient way to assemble, interface and characterize microfluidic devices. The high number of electric and fluidic connections combined with the modularity of the electronics enables the characterization of many different types of sensors and/or actuators.

7.2 Outlook

To fully understand the resolution limitations of micro Coriolis mass flow sensors, the full multiphysical path from mass flow to electrical output signal has to be investigated. Next to the sensor itself, each component of the readout electronics needs to be analyzed for its noise behavior. From the conclusions of this research, improvements to the resolution can be achieved by improving the phase detection electronics or increasing the sensitivity by optimization of the capacitive readout electrodes. Another approach would be to use a (piezo)resistive readout structures to perform the mode shape analysis.

On the subject of fabrication, an improvement would be to incorporate piezoelectric material deposition and patterning in surface channel technology. This enables on-chip actuation of Coriolis mass flow sensors and other moving structures. It could also be used for different readout methods for the presented sensors.

The resistive cross-sectional deformation pressure sensors have shown good potential for single chip flow/pressure sensor integration. These sensors should be thoroughly calibrated under different controlled circumstances, e.g. different temperatures and humidities. Furthermore, multiple samples should be characterized, since differences can certainly occur due to non-ideal effects in the fabrication process.

The fluid physics of liquids and gases in surface channels need to be investigated further to improve the modeling of the pressure drop as a result of mass flow. This enables more accurate viscosity sensing and helps in the optimization of sensor designs in surface channel technology. A systematic method could consist of measuring the pressure drop of different channel designs with varying diameter, length and bends.

A

Fabrication details

This appendix contains a summary of the fabrication steps for the silicon-on-insulator based surface channel technology. All used chemicals with relevant color coding are listed in Section B.2.

A

A.1 Silicon nitride deposition

A

1 - Substrate selection: device wafers

- Wafer type: silicon-on-insulator.
- Device layer: $50\mu\text{m} \pm 1\mu\text{m}$.
- Buried oxide layer: $5\mu\text{m} \pm 250\text{nm}$.
- Handle layer: $400\mu\text{m} \pm 25\mu\text{m}$.
- Oxide layer on bottom: $5\mu\text{m}$.
- Device and handle layer Orientation: $\langle 100 \rangle$.
- Device and handle layer dopant: p-type, boron.
- Device and handle layer resistivity: $< 0.05\Omega\text{cm}$.

2 - Substrate selection: dummy wafers

- Wafer type: monocrystalline silicon.
- Orientation: $\langle 100 \rangle$.
- Diameter: 100 mm.
- Thickness: $525\mu\text{m} \pm 25\mu\text{m}$.
- Polished: double side.
- Dopant: p-type, boron.
- Resistivity: $5\Omega\text{cm} \cdots 10\Omega\text{cm}$.

3 - Curvature measurement

- Machine: Veeco Dektak 8.
- Scan length: 80 mm.
- Stylus force: 5 mg.
- Duration: 60 s.
- Profile: hills and valleys.

4 - Pre-furnace cleaning

- Clean organic traces: 10 min in 99 % HNO_3 .
- Quick dump rinse.
- Clean metallic traces: 10 min in 69 percent HNO_3 at 95°C .
- Quick dump rinse.
- Etch native oxide: 1 min with 1 % HF .
- Quick dump rinse.
- Dry.

5 - LPCVD of SiRN

- Machine: Tempress furnace.
- Aimed thickness: $1\text{ }\mu\text{m}$.
- Deposition rate: 4 nm min^{-1} .
- SiH_2Cl_2 flow: 77.5 sccm.
- NH_3 flow: 20 sccm.
- Temperature: $850\text{ }^\circ\text{C}$.
- Pressure: 150 mTorr.
- N_2 flow: 250 sccm.
- Check the thickness of the layer using ellipsometry.
- Check for particles using a cold light source.

A.2 Inlets and outlets

6 - Lithography of inlets and outlets on backside

- Check the backside mask.
- Dehydration bake: 5 min at 120 °C.
- Spin coat: HMDS 30 s at 2500 rpm on backside.
- Spin coat: OIR 908-35 30 s at 2500 rpm for 4.5 μm on backside.
- Leave 10 h in low-UV area to outgas resist.
- Expose: 12 s at 12 mW cm^{-2} with soft contact on backside.
- Post-exposure bake: 1 min at 120 °C.
- Develop: 60 s with OPD 4262.
- Quick dump rinse.
- Dry.
- Hardbake: 120 min at 120 °C.
- Check the lithography.

7 - Reactive ion etch of SiRN

- Machine: Adixen AMS 100.
- Time: 5 min at 300 nm s^{-1} .
- Substrate position: 200 mm.
- Substrate temperature: 20 °C.
- He coolant pressure: 10 mbar.
- Vacuum valve: 100 %.
- Inductively coupled plasma: 1200 W.
- Capacitively coupled plasma: 150 W.
- CHF_3 flow: 100 sccm.
- Ar flow: 100 sccm.

8 - Reactive ion etch of SiO_2

- Machine: Adixen AMS 100.
- Time: 10 min at $0.5 \mu\text{m s}^{-1}$.
- Substrate position: 120 mm.
- Substrate temperature: -10°C .
- He coolant flow: 150 sccm.
- Pressure: $8.5 \cdot 10^{-3}$ mbar.
- Inductively coupled plasma: 2800 W.
- Capacitively coupled plasma: 350 W.
- C_4F_8 flow: 20 sccm.
- CH_4 flow: 15 sccm.

9 - Deep reactive ion etch of Si

- Machine: Adixen AMS 100.
- Time: 36 min at $11 \mu\text{m s}^{-1}$.
- Substrate position: 110 mm.
- Substrate temperature: -40°C .
- He coolant pressure: 10 mbar.
- Vacuum valve: 16.5 %.
- Inductively coupled plasma: 2500 W.
- Capacitively coupled plasma: pulsed, 10 ms at 60 W and 90 ms at 0 W.
- Process: Bosch, so alternating SF_6 and C_4F_8 flow.
- SF_6 flow: 7 s at 500 sccm.
- C_4F_8 flow: 1.5 s at 100 sccm.
- Check if the BOX layer has been reached.

10 - Resist and fluorocarbon strip

- Machine: PVA TePla GIGAbatch 360.
- Step 1: 10 min at 600 sccm Ar, 0.6 mbar, 1000 W.
- Step 2: 10 min at 250 sccm O_2 , 0.5 mbar, 800 W.
- Step 3: 1 min at 237 sccm O_2 , 13 sccm CF_4 , 0.5 mbar, 800 W.
- Step 4: 1 min at 250 sccm O_2 , 0.8 mbar, 800 W.

A.3 Channels

11 - Sputtering of Cr

- Machine: T'COthy.
- Time: 60 s (pre-sputter) + 200 s (deposition).
- Aimed thickness: 50 nm.
- Target: Cr.
- Pressure: $6.6 \cdot 10^{-3}$ mbar with Ar.
- Power: 200 W.
- Deposition rate: 15 nm min^{-1} .

12 - Lithography of channels

- Check the channel mask.
- Dehydration bake: 5 min at 120°C .
- Spin coat: HMDS 30 s at 4000 rpm.
- Spin coat: OIR 907-17 30 s at 4000 rpm for $1.7 \mu\text{m}$.
- Prebake: 90 s at 95°C .
- Clean the mask.
- Expose: 3.5 s at 12 mW cm^{-2} with vacuum contact and preexposure delay of 2 min.
- Post-exposure bake: 1 min at 120°C .
- Develop: 60 s with OPD 4262.
- Quick dump rinse.
- Dry.
- Check the lithography.

13 - Reactive ion etch of Cr and SiRN

- Machine: Adixen AMS 100.
- Time: 6 min to etch 50 nm Cr and $1 \mu\text{m}$ SiRN.
- Substrate position: 110 mm.
- Substrate temperature: 20°C .
- He coolant pressure: 10 mbar.
- Vacuum valve: 100 %.
- Inductively coupled plasma: 1200 W.
- Capacitively coupled plasma: 150 W.
- CHF_3 flow: 100 sccm.
- Ar flow: 100 sccm.

14 - Reactive ion etch of Si

- Machine: SPTS Pegasus.
- Time: 15 min for channels of 40 μm .
- Substrate temperature: -19°C .
- He coolant pressure: 1.2 mbar.
- Pressure: 0.1 mbar.
- Inductively coupled plasma: 3000 W.
- Capacitively coupled plasma: 0 W.
- SF_6 flow: 600 sccm.
- Check channel diameter and if the BOX layer has been reached for relevant structures.

15 - Wet etch of SiO_2

- 50 % HF with $1 \mu\text{m min}^{-1}$ until test structures are released.
- Quick dump rinse.
- Soak 120 min in DI water.
- Quick dump rinse.
- Dry.

16 - Vapor HF etch of SiO_2

- Machine: Idonus HF vapor phase etcher.
- Time: dependent on structures, determine with dummies.
- Temperature: 37°C .
- Dry for 24 h in nitrogen vapor.

17 - Wet strip of resist and Cr

- Strip resist: 15 min at 95 °C in piranha solution ($3 \text{ H}_2\text{SO}_4 + \text{H}_2\text{O}_2$).
- Etch Cr: 15 min in chromium etchant.
- Quick dump rinse.
- Soak 120 min in DI water.
- Quick dump rinse.
- Remove metal residues: 15 min of RCA-2 ($\text{HCl} + \text{H}_2\text{O}_2 + 5 \text{ H}_2\text{O}$).
- Quick dump rinse.
- Soak 120 min in DI water.
- Quick dump rinse.
- Dry.
- Measure slit width and use this value to calculate the SiRN deposition layer thickness.

18 - Pre-furnace cleaning

- Clean organic traces: 10 min in 99 % HNO_3 .
- Quick dump rinse.
- Clean metallic traces: 10 min in 69 percent HNO_3 at 95 °C.
- Quick dump rinse.
- Etch native oxide: 1 min with 1 % HF.
- Quick dump rinse.
- Remove residues: 120 min soak in DI water.
- Quick dump rinse.
- Dry.

19 - LPCVD of SiRN

- Machine: Tempress furnace.
- Aimed thickness: slit width $\cdot 0.65 \text{ }\mu\text{m}$.
- Deposition rate: 4 nm min^{-1} .
- SiH_2Cl_2 flow: 77.5 sccm.
- NH_3 flow: 20 sccm.
- Temperature: 850 °C.
- Pressure: 150 mTorr.
- N_2 flow: 250 sccm.
- Check the thickness of the layer using ellipsometry.
- Check for particles using a cold light source.

A.4 Electrodes

20 - Lithography of silicon bondpads

- Check the silicon bondpads mask.
- Dehydration bake: 5 min at 120 °C.
- Spin coat: HMDS 30 s at 4000 rpm.
- Spin coat: OIR 907-17 30 s at 4000 rpm for 1.7 μm .
- Prebake: 90 s at 95 °C.
- Clean the mask.
- Expose: 4 s at 12 mW cm^{-2} with hard contact.
- Post-exposure bake: 1 min at 120 °C.
- Apply dicing foil on the backside to prevent liquids entering the inlets.
- Develop: 60 s with OPD 4262.
- Quick dump rinse.
- Dry.
- Remove dicing foil.
- Hardbake: 10 min at 120 °C.
- Check the lithography.

21 - Reactive ion etch of SiRN

- Machine: Adixen AMS 100.
- Time: 5 min at 300 nm s^{-1} .
- Substrate position: 200 mm.
- Substrate temperature: 20 °C.
- He coolant pressure: 10 mbar.
- Vacuum valve: 100 %.
- Inductively coupled plasma: 1200 W.
- Capacitively coupled plasma: 150 W.
- CHF_3 flow: 100 sccm.
- Ar flow: 100 sccm.

22 - Resist strip

- Machine: PVA TePla GIGAbatch 360.
- Time: 10 min.
- Step 1: 10 min at 600 sccm Ar, 0.6 mbar, 1000 W.
- Step 2: 10 min at 360 sccm O_2 , 160 sccm Ar, 0.6 mbar, 800 W.

23 - Removal of native SiO₂

- Machine: Idonus HF vapor phase etcher.
- Time: 5 min.
- Temperature: 37 °C.
- Continue with sputtering directly after removal of native oxide.

24 - Sputtering of Cr

- Machine: T'COthy.
- Time: 60 s (pre-sputter) + 60 s (deposition).
- Aimed thickness: 15 nm.
- Target: Cr.
- Pressure: $6.6 \cdot 10^{-3}$ mbar with Ar.
- Power: 200 W.
- Deposition rate: 15 nm min^{-1} .

25 - Sputtering of Au

- Machine: T'COthy.
- Time: 60 s (pre-sputter) + 270 s (deposition).
- Aimed thickness: 200 nm.
- Target: Au.
- Pressure: $6.6 \cdot 10^{-3}$ mbar with Ar.
- Power: 200 W.
- Deposition rate: 15 nm min^{-1} .

26 - Lithography of electrodes

- Check the metal mask.
- Dehydration bake: 5 min at 120 °C.
- Spin coat: HMDS 30 s at 4000 rpm.
- Spin coat: OIR 907-17 30 s at 4000 rpm for 1.7 μm .
- Prebake: 90 s at 95 °C.
- Clean the mask.
- Expose: 4 s at 12 mW cm^{-2} with hard contact.
- Post-exposure bake: 1 min at 120 °C.
- Apply dicing foil on the backside to prevent liquids entering the inlets.
- Develop: 60 s with OPD 4262.
- Quick dump rinse.
- Dry.
- Remove dicing foil.
- Hardbake: 1 min at 120 °C.
- Check the lithography.

27 - Reactive ion beam etch of Cr/Au

- Machine: Oxford Ionfab 300.
- Neutralizer current: 100 mA.
- RF generator power: 300 W.
- Beam current: 50 mA.
- Beam voltage: 300 V.
- Beam accelerator: 300 V.
- Cool gas: 5 Torr.
- Platen temperature: 15 °C.
- Platen drive: 5 rpm.
- Platen position: 0°...–20°.
- Ar flow for neutralizer: 5 sccm.
- Ar flow for beam: 5 sccm.
- Check the electrode shapes and sizes.

28 - Resist strip on metal

- Machine: PVA TePla GIGAbatch 360.
- Time: 10 min.
- O₂ flow: 250 sccm.
- H₂ flow: 250 sccm.
- Pressure: 0.7 mbar.
- Power: 800 W.

A.5 Release

29 - Lithography of release structures

- Check the release mask.
- Dehydration bake: 10 min at 120 °C.
- Spin coat: SU-8 30 s at 3000 rpm for 5.2 μm .
- Prebake: 1 min at 50 °C, 1 min at 65 °C, 3 min at 95 °C, slowly cooldown to 20 °C.
- Clean the mask.
- Expose: 10 s at 10 mW cm^{-2} with hard contact.
- Post-exposure bake: 1 min at 50 °C, 1 min at 65 °C, 2 min at 80 °C, slowly cooldown to 20 °C.
- Apply dicing foil on the backside to prevent liquids entering the inlets.
- Develop: 6 $\times$ 30 s with RER600.
- Rinse with propan-2-ol.
- Dry.
- Remove dicing foil.
- Hardbake: 120 min at 120 °C.
- Check the lithography.

30 - Reactive ion etch of Cr and Au

- Machine: TEtske.
- Time: 10 min.
- Substrate temperature: 10 °C.
- Vacuum valve: 100 %.
- Pressure: 10 mTorr.
- Power: 60 W.
- DC voltage: -500 V $\cdots$ -540 V.
- CHF_3 flow: 20 sccm.
- O_2 flow: 0 sccm.

31 - Reactive ion etch of Si

- Machine: SPTS Pegasus.
- Time: 3×5 min etch + 2 min cooldown.
- Substrate temperature: -19°C .
- He coolant pressure: 1.2 mbar.
- Pressure: 0.1 mbar.
- Inductively coupled plasma: 1000 W and 600 W when the channels are released.
- Capacitively coupled plasma: 0 W.
- SF_6 flow: 600 sccm.
- Check if the BOX layer has been reached.

32 - Vapor HF etch of SiO_2

- Machine: Idonus HF vapor phase etcher.
- Time: 25 min.
- Temperature: 37°C .

33 - Reactive ion etch of Si

- Machine: SPTS Pegasus.
- Time: $3 \times (5 \text{ min (etch)} + 2 \text{ min (cooldown)})$.
- Substrate temperature: -19°C .
- He coolant pressure: 1.2 mbar.
- Pressure: 0.1 mbar.
- Inductively coupled plasma: 1400 W.
- Capacitively coupled plasma: 0 W.
- SF_6 flow: 600 sccm.
- Check the depth of the release etch.

34 - Resist and fluorocarbon strip on metal

- Machine: PVA TePla GIGAbatch 360.
- Time: 60 min.
- O_2 flow: 250 sccm.
- H_2 flow: 250 sccm.
- Pressure: 0.7 mbar.
- Power: 800 W.

B

Nomenclature

This appendix specifies the symbols used in this dissertation for physical quantities, chemicals and measurement setups.

B

B.1 Physical quantities

B.1.1 General

Table B.1: Symbols and units of general physical quantities.

Symbol	Description	Unit	SI Unit
t	Time	s	s
f	Frequency	Hz	s^{-1}
ω	Angular frequency	rad s^{-1}	s^{-1}
E	Energy	J	$\text{kg m}^2 \text{s}^{-2}$
P	Power	W	$\text{kg m}^2 \text{s}^{-2} \text{s}^{-1}$

Table B.2: Notations and mathematical definitions.

Natural logarithm	$\ln(x) = \log_e(x)$
Logarithm to base b	$\log_b(x) = \frac{\ln(x)}{\ln(b)}$
Logarithm to base 10	$\log(x) = \log_{10}(x)$
Vector	$\vec{a} = \begin{bmatrix} a_x \\ a_y \\ a_z \end{bmatrix}$
Absolute value of vector	$\hat{a} = \vec{a} = \sqrt{a_x^2 + a_y^2 + a_z^2}$
Magnitude	$\hat{v}(\omega) = v(j\omega) = \sqrt{\Re(v(j\omega))^2 + \Im(v(j\omega))^2}$
Phase	$\arg(v(j\omega)) = \arctan(v(j\omega))$
Harmonic signal	$v(t) = \hat{v} \sin(\omega t)$
Parallel operator	$Z_a Z_b = (Z_a^{-1} + Z_b^{-1})^{-1}$
Fourier transform	$f(j\omega) = \mathcal{F}(f(t)) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} f(t) e^{-j\omega t} dt$

Table B.3: Constants.

Symbol	Description	Value	Unit	SI Unit
e	Euler's number	2.7182		
π	Peripherea	3.1415		
j	Imaginary unit	$\sqrt{-1}$		
ϵ_0	Vacuum permittivity	8.8542	F m^{-1}	$\text{A}^2 \text{s}^4 \text{kg}^{-1} \text{m}^{-3}$
k_B	Boltzmann constant	1.3806	J K^{-1}	$\text{kg m}^2 \text{s}^{-2} \text{K}^{-1}$

B.1.2 Mechanical**Table B.4:** Symbols and units of mechanical quantities.

Symbol	Description	Unit	SI Unit
L	Length (in x-direction)	m	m
W	Width (in y-direction)	m	m
H	Height (in z-direction)	m	m
R	Radius	m	m
A	Area	m ²	m ²
V	Volume	m ³	m ³
x	Position in x-direction	m	m
y	Position in y-direction	m	m
z	Position in z-direction	m	m
z_n	Noise displacement spectral density	m Hz ^{-$\frac{1}{2}$}	m s ^{$\frac{1}{2}$}
r	Position in radial direction	m	m
l	Arc	m	m
v	Velocity	m s ⁻¹	m s ⁻¹
a	Acceleration	m s ⁻²	m s ⁻²
F	Force	N	kg m s ⁻²
D	Damping	N s m ⁻¹	kg s ⁻¹
ζ	Damping ratio		
m	Mass	kg	kg
c	Stiffness	N m ⁻¹	kg s ⁻²
θ	Angle	rad	
θ_n	Noise angle spectral density	rad Hz ^{-$\frac{1}{2}$}	rad s ^{$\frac{1}{2}$}
Ω	Angular velocity	rad s ⁻¹	s ⁻¹
α	Angular acceleration	rad s ⁻²	s ⁻²
τ	Torque	N m	kg m ² s ⁻²
τ_n	Noise torque spectral density	N m Hz ^{-$\frac{1}{2}$}	kg m ² s ^{-$\frac{3}{2}$}
R	Rotational damping	N m s rad ⁻¹	kg m ² s ⁻¹
J	Mass moment of inertia	kg m ²	kg m ²
K	Rotational stiffness	N m rad ⁻¹	kg m ² s ⁻²
I	Second moment of area	m ⁴	m ⁴
σ	Stress	Pa	kg m s ⁻²
ϵ	Strain		
ρ	Density	kg m ⁻³	kg m ⁻³

B.1.3 Electrical

Table B.5: Symbols and units of electrical quantities.

Symbol	Description	Unit	SI Unit
Q	Charge	C	A s
i	Current	A	A
u	Voltage	V	$\text{kg m}^2 \text{A}^{-1} \text{s}^{-3}$
R	Resistance	Ω	$\text{kg m}^2 \text{A}^{-2} \text{s}^{-3}$
L	Inductance	H	$\text{kg m}^2 \text{A}^{-2} \text{s}^{-2}$
C	Capacitance	F	$\text{A}^2 \text{s}^4 \text{kg}^{-1} \text{m}^{-2}$
Z	Impedance	Ω	$\text{kg m}^2 \text{A}^{-2} \text{s}^{-3}$
Y	Admittance	S	$\text{A}^2 \text{s}^3 \text{kg}^{-1} \text{m}^{-2}$
ϵ_r	Relative permittivity		
SNR	Signal to noise ratio		
$\mathcal{A}$	Cancellation factor		

B.1.4 Fluid and thermal

Table B.6: Symbols and units of fluid and thermal quantities.

Symbol	Description	Unit	SI Unit
u	Flow velocity	m s^{-1}	m s^{-1}
V	Volume	m^3	m^3
Q	Volume flow	$\text{m}^3 \text{s}^{-1}$	$\text{m}^3 \text{s}^{-1}$
M	Mass	kg	kg
Φ	Mass flow	kg s^{-1}	kg s^{-1}
P	Pressure	10^{-5} bar	$\text{kg s}^{-2} \text{m}^{-1}$
ρ	Density	kg m^{-3}	kg m^{-3}
v_{sp}	Specific volume	$\text{m}^3 \text{kg}^{-1}$	$\text{m}^3 \text{kg}^{-1}$
η	Dynamic viscosity	Pa s	$\text{kg s}^{-1} \text{m}^{-1}$
ν	Kinematic viscosity	$\text{m}^2 \text{s}^{-1}$	$\text{m}^2 \text{s}^{-1}$
R_{sp}	Specific gas constant	$\text{J kg}^{-1} \text{K}^{-1}$	$\text{m}^2 \text{s}^{-2} \text{K}^{-1}$
T	Temperature	K	K
Re	Reynolds number		
St	Strouhal number		
Kn	Knudsen number		

B.2 Chemicals

Table B.7: Chemicals with alternative names, formula and phase by room temperature.

Name	Formula	Phase
Propan-2-ol	C_3H_7OH	Liquid
Isopropanol		
IPA		
Ethanol	C_2H_5OH	Liquid
Ethyl alcohol		
Propanone	$(CH_3)_2CO$	Liquid
Acetone		Liquid
Hexane	C_6H_{14}	Liquid
Trichloromethane	$ChCl_3$	Liquid
Chloroform		
Methanol	CH_3OH	Liquid
Methyl alcohol		
Water	H_2O	Liquid
Oxidane		
Dihydrogen oxide		
Nitric acid	HNO_3	Liquid
Hydrogen nitrate		
Sulfuric acid	H_2SO_4	Liquid
Vitriol		
Hydrogen peroxide	H_2O_2	Liquid
Dihydrogen dioxide		
Hydrochloric acid	HCl	Liquid
Hydrogen fluoride	HF	Liquid
Fluorocarbon	C_xF_y	
Tetrafluoromethane	CF_4	Gas
Octafluorocyclobutane	C_4F_8	Gas
Hydrogen	H_2	Gas
Nitrogen	N_2	Gas
Helium	He	Gas
Argon	Ar	Gas
Oxygen	O_2	Gas
Methane	CH_4	Gas
Azane	NH_3	Gas
Ammonia		
Dichlorosilane	SiH_2Cl_2	Gas
Sulfur hexafluoride	SF_6	Gas
Fluoroform	CHF_3	Gas

Name	Formula	Phase	
Hexamethyldisilazane HMDS	$C_6H_{19}NSi_2$	Liquid	
Polydimethylsiloxane PDMS	$CH_3[Si(CH_3)_2O]_nSi(CH_3)_3$	Solid	
Silicon	Si	Solid
Silicon dioxide	SiO_2	Solid
Silicon nitride	Si_3N_4	Solid
Silicon rich nitride	SiRN	Solid
Chromium	Cr	Solid
Gold	Au	Solid
Titanium	Ti	Solid
Platinum	Pt	Solid
Lanthanum nickel trioxide	$LaNiO_3$	Solid
Lead zirconate titanate PZT	$Pb[Zr_xTi_{1-x}]O_3$	Solid
Photoresist		Solid
Piranha solution	$3 H_2SO_4 + H_2O_2$	Liquid	
RCA-2 solution	$HCl + H_2O_2 + 5 H_2O$	Liquid	

B.3 Symbols

B.3.1 Electronic

DC voltage source		Voltage meter
DC current source		Current meter
AC voltage source		Amplifier
Capacitor		Differential amplifier
Resistor		Comparator
Inductor		Low pass filter
Ground		XOR gate

B.3.2 Fluidic

Pressure source		Pressure meter
Flow source		Mass flow meter
Filter		Roughing pump
Reservoir		Turbomolecular pump
Atmospheric pressure		Valve

Summary

Measurement of flow is essential for continuous dosing of fluids. Accurate dosing is important for medical applications, e.g. drug delivery using intravenous therapy, of which the settling time of the flow as a result of tubing and needles is significant. In industrial applications, e.g. controlling gas concentrations in reaction chambers for the production of semiconductors, measurement of flow is also crucial. By measuring flow, pressure and fluid properties, such as density and viscosity, the composition of mixtures can be measured, provided that the ingredients are known. Miniaturization of these sensors using microtechnology offers advantages in terms of resolution, mass production, channel wall material and internal volumes. This dissertation describes novel designs, fabrication methods and experiments of microfluidic sensors that use a mechanical transduction principle and that can be integrated throughflow with other sensors on a single chip.

Especially microfabricated Coriolis mass flow sensors are suitable for this purpose. A Coriolis mass flow meter consists of a suspended channel that is actuated at one of its resonance frequencies. A fluid flow through the channel causes a secondary vibration mode at the same frequency. A family of methods to fabricate such microfluidic sensors has been described and is called surface channel technology. These methods enable the fabrication of suspended silicon nitride channels of 10 μm to 100 μm . A metal layer can be deposited and patterned to realize wiring and electrodes. A number of improvements have been presented, enabling the fabrication of silicon electrodes at the sides of the channels, piezoelectric material on top of the channels and multiple layers of channels. For characterization, sensors need fluidic and electrical connections. Therefore, a universal and modular platform has been developed. The assembly process of the sensor for characterization with this platform requires only two steps: the sensor has to be glued on a printed circuit board and wirebonded. The printed circuit board can then be clamped into the main setup, which leads directly to 8 fluid connections and 72 electrical connections. Electronic modules can be connected to the main setup for actuation and reading.

Since Coriolis mass flow sensors use a mechanical transduction principle, the resolution is fundamentally limited by thermomechanical noise. A thermomechanical noise model has therefore been developed. The Coriolis mass flow sensor is modeled as a second order system. The excitation by noise is derived from the equipartition

principle. RMS amplitudes are measured with laser Doppler vibrometry and correspond to theory. A noise equivalent mass flow of 0.3 ng s^{-1} has been derived for currently most accurate Coriolis mass flow sensor. The resolution of the latter is not yet limited by thermomechanical noise and can be improved by at least a factor of 10. One way to improve the resolution is by increasing the sensitivity to mass flow. This can be achieved by decreasing the influence of the actuation mode on the output signal and thus increasing the sensitivity of the Coriolis induced mode. This can be realized for capacitive Coriolis mass flow sensors by the addition of two readout electrodes that are crosswise connected to the original electrodes. The thermomechanical noise has only been measured for a single point of the channel of a Coriolis mass flow sensor. For vibration mode analysis, the magnitudes and phases of several points need to be known. Laser Doppler vibrometers can measure the magnitudes of the velocities of different points by scanning, the phase information is then obtained by triggering on the actuation signal. The actuation signal must therefore be known. The phase information can still be retrieved from unknown signals using a two stage measurement and the presented post processing.

Surface channel technology is also suitable for the fabrication of throughflow pressure sensors. Two designs have been presented. One design consists of a channel with a deforming ceiling dependent on pressure. Resistive readout structures in a Wheatstone bridge detect this deformation with a sensitivity of $4 \cdot 10^{-5} \text{ bar}^{-1}$. The other pressure gauge consists of a suspended U-shaped channel. Due to the non-circular cross section of the channel, it deforms in its entirety. This displacement is capacitively detected with a sensitivity of 1 fF bar^{-1} . Coriolis mass flow sensors share this phenomenon: it has been validated that Coriolis mass flow meters can also measure pressure simultaneously with mass flow. This can be achieved by measuring the static deflection of the capacitive readout structures in addition to phase shift. If pressure and mass flow are known, the viscosity can be derived using a fluid modeling. The Hagen-Poiseuille law is sufficient for liquids. A more complex model has been derived for gases, since gases are compressible. Density can also be measured, since the resonance frequency of a Coriolis mass flow sensor is dependent on the density of the liquid in the channels. This resonance frequency is also dependent on the pressure, but this can be compensated for with the pressure sensors. Surface channel technology has also been used to realize a relative permittivity sensor. This sensor consists of two silicon electrodes at both sides of the channel and measures the capacity through the fluid.

Samenvatting

Het meten van debiet is essentieel voor het continu doseren van vloeistoffen en gassen. Nauwkeurig doseren is onder andere belangrijk voor medische toepassingen, zoals bij medicijntoediening via een infuus, waarbij de insteltijd van het debiet als gevolg van slangen en naalden significant is. Ook bij industriële toepassingen, zoals het regelen van gassamenstellingen in reactiekamers bij de productie van halfgeleiders, is het meten van debiet cruciaal. Door naast debiet ook druk en vloeistofeigenschappen, zoals dichtheid en viscositeit, te meten is het mogelijk de samenstelling van mengsels te achterhalen, mits bekend is uit welke vloeistoffen of gassen het mengsel bestaat. Het miniaturiseren van deze sensoren door middel van microtechnologie biedt voordelen op het gebied van resolutie, massafabricage, kanaalwandmateriaal en interne volumes. Dit proefschrift beschrijft nieuwe ontwerpen, fabricagemethodes en experimenten van microfluidische sensoren die een mechanisch transductieprincipe hebben en serieel geïntegreerd kunnen worden met andere sensoren op een enkele chip.

Met name microgefabriceerde Coriolis-massadebietmeters zijn hiervoor geschikt. Een Coriolis-massadebietmeter bestaat uit een vrijhangend kanaal dat in trilling wordt gebracht op één van zijn resonantiefrequenties. Een vloeistofstroom door de buis veroorzaakt een secundaire trillingsvorm op die frequentie. Een familie van methodes om dergelijke microfluidische sensoren te fabriceren is beschreven en wordt surface channel technology genoemd. Deze methodes maken het mogelijk om vrijhangende silicium nitride kanalen te fabriceren van ongeveer 10 μm tot 100 μm . Een metalen laag kan worden aangebracht en gestructureerd voor de realisatie van bedrading en elektrodes. Een aantal verbeteringen is gepresenteerd, waarbij silicium elektrodes aan de zijkanten van de kanalen kunnen worden geplaatst, piëzo-elektrisch materiaal kan worden aangebracht en kruisende kanalen kunnen worden gefabriceerd. Voor het karakteriseren van een gefabriceerde sensor moet fluidisch en elektrisch contact worden gemaakt. Een universele en modulair platform is hiervoor ontwikkeld. Het montageproces van de sensor voor karakterisatie met dit platform vergt slechts twee stappen: de sensor moet worden gelijmd op een printplaat en worden bedraad door middel van wirebonding. Deze printplaat kan dan in de basisopstelling worden geklemd; dit leidt direct tot verbinding met 8 vloeistofaansluitingen en 72 elektrische aansluitingen. Elektronische modules kunnen worden aangesloten op de basisopstelling voor actuatie en uitlezing.

Aangezien Coriolis-massadebietmeters een mechanisch transductieprincipe hebben, is de resolutie fundamenteel begrensd door thermomechanische ruis. Er is daarom een thermomechanischeruis-model opgesteld. Hiervoor is de Coriolis-massadebietmeter gemodelleerd als een tweede-ordessysteem. De excitatie door ruis is afgeleid van het equipartitiebeginsel. RMS amplitudes zijn gemeten met laser-Dopplervibrometrie en sluiten aan bij de theorie. Een ruis-equivalent massadebiet van 0.3 ng s^{-1} is afgeleid voor de momenteel nauwkeurigste Coriolis-massadebietmeter. De resolutie van laatstgenoemde is nog niet beperkt door thermomechanische ruis en kan nog met tenminste een factor 10 verbeterd worden. Een mogelijke manier om de resolutie te verbeteren is door de gevoeligheid voor massadebiet te verhogen. Dit kan worden bewerkstelligd door de invloed van de actuatiemode op het uitgangssignaal te verlagen en daarmee de gevoeligheid voor de Coriolis-krachtgeïnduceerde mode te verhogen. Door twee extra uitleeselektrodes kruislings met de originele elektrodes te verbinden kan dit gerealiseerd worden voor capacitieve Coriolis-massadebietmeters. Een factor 3 in gevoeligheid is experimenteel bewezen. De thermomechanische ruis is alleen gemeten voor een enkel punt van het kanaal van een Coriolis-massadebietmeter. Voor trillingsmode-analyse moeten de magnitudes en fases van meerdere punten bekend zijn. Laser-Dopplervibrometers kunnen de magnitudes van de snelheden van verschillende punten meten door te scannen, de faseinformatie wordt verkregen door te triggeren op het actuatiesignaal. Hiervoor moet het actuatiesignaal bekend zijn. Door middel van een tweetrapsmeting en de gepresenteerde nabewerking kan toch de faseinformatie worden achterhaald van onbekende signalen.

Surface channel technology is ook geschikt om serieel plaatsbare drukmeters te fabriceren. Twee ontwerpen zijn gepresenteerd. Eén ontwerp bestaat uit een kanaal waarvan het plafond vervormt door druk. Resistieve uitleesstructuren in een brug van Wheatstone detecteren deze vervorming met een gevoeligheid van $4 \cdot 10^{-5} \text{ bar}^{-1}$. De andere drukmeter bestaat uit een vrijhangend U-vormig kanaal en vervormt in zijn geheel omhoog dankzij zijn niet-cirkelvormige kanaaldoornede. Deze verplaatsing wordt capacitief gedecteerd met een gevoeligheid van 1 fF bar^{-1} . Coriolis-massadebietmeters delen deze eigenschap: gevalideerd is dat Coriolis-massadebietmeters tegelijk met massadebiet ook druk kunnen meten. Dit kan door naast de faseverschuiving ook de statische uitwijking van de capacitieve uitleesstructuren op te nemen. Als druk en massadebiet bekend zijn kan de viscositeit worden afgeleid met de juiste modelvorming. Voor vloeistoffen voldoet de wet van Hagen-Poiseuille. Wegens de samendrukbaarheid is er voor gassen een complexer model afgeleid. Dichtheid kan ook worden gemeten, aangezien de resonantiefrequentie van Coriolis-massadebietmeters afhankelijk is van de dichtheid van de vloeistof in de kanalen. Deze resonantiefrequentie is ook afhankelijk van de druk, maar dit kan worden gecompenseerd met de drukmeters. Surface channel technology is ook gebruikt om een relatievepermittiviteitsmeter te realiseren. Deze sensor bestaat uit twee silicium elektrodes aan weerszijden van het kanaal en meet de capaciteit door de vloeistof.

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Nawoord

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¹Aangenomen dat de gemiddelde diameter van zo’n knolselderij 10 cm is [1] en iedere knolselderij niet in diameter afneemt door rotting.

je wilt, in ieder geval wil ik kwijt dat we afgelopen jaren samen zoveel van elkaar geleerd hebben! De tijd dat we samen zijn, en dus ook tijdens mijn promotie, was je met jouw liefde en interesse mijn belangrijkste steun, teamgenoot, sparringpartner en vriendin ♡. Daarom heb je in dit proefschrift een nog veel groter aandeel dan je denkt. En wegens je sensitiviteit zul je altijd mijn favoriete sensor blijven!

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DENNIS ALVERINGH

ENSCHDEDE, FEBRUARI 2018

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About the author

Dennis Alveringh was born on December 3, 1988 in Dronten, the Netherlands. While attending primary and secondary education, he got a passion for technology, physics and all other random things the universe has to offer. He therefore started his study Electrical Engineering at the University of Twente, where he was besides studying active in Vereniging Campus Kabel, E.T.S.V. Scintilla, Green Vibrations and Twente Academy. During his master, he joined VTT Technical Research Centre of Finland for a three month internship. After that, he finished his studies in 2013 on the subject of a microfabricated multi-axis capacitive force/torque sensor. Then he joined the MESA+ Institute for Nanotechnology at the same university for his PhD research. Details of his research are described in this dissertation.

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Humans are only able to see electromagnetic radiation from 400 THz to 800 THz...

Humans can only hear acoustic waves from 20 Hz to 20 kHz...

And, we still do not understand all physical basics, of the universe, like gravity and spacetime...

Thus, we are more blind than sighted, our perception is fully subjective.

Integrated *throughflow* mechanical *microfluidic* sensors

Dennis Alveringh

For many medical and industrial applications, measurement of flow is essential for continuous dosing of fluids. When other physical quantities of fluids are sensed next to flow, e.g. density and viscosity, the composition of the fluid can be obtained. Miniaturization of these sensors using microtechnology offers advantages in terms of resolution, mass production, channel wall material and internal volume.

The research described in this dissertation spans roughly two subjects:

- resolution limit analysis and improvement of microfabricated Coriolis mass flow sensors;
- integration of flow, pressure, density, viscosity and relative permittivity sensors on a single chip.

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