A SMART LAMB-WAVE SENSOR SYSTEM
for the determination of fluid properties

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A SMART LAMB-WAVE SENSOR SYSTEM
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Progress is the realization of utopias
Oscar Wilde (1856-1900)

Voor Joke, aan mijn moeder
Ter nagedachtenis aan mijn vader
Leden van de promotiecommissie:

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Chapter 1

Introduction

1.1 Acoustic sensing in fluids

There is currently an increased demand for devices to determine various properties of fluids. Environmental pollution control is one of the reasons to develop sensors with which the quality of air and water can be monitored. In food processing, the monitoring and determination of such quantities as density, viscosity and particle concentration in a liquid are required. Medical interest in such devices is in the field of biochemical sensing in, for example, body fluids.

In 1947, Mason reported making use of ultrasonic devices to determine liquid properties [1]. It took until the late seventies before other groups developed activities in the field of sensing in fluids which are based on acoustic waves. Initially, research was focussed on chemical gas sensors. A few years later, there was an increase of interest in acoustic sensing in liquids. Applications of acoustic-wave devices for the determination of fluid properties (a fluid is defined as "any substance that can flow" and thus includes both gases and liquids) are presented in review papers [2,3,4].

Acoustic sensing is based on the modulation of acoustic waves which propagate along the surface of a solid medium. Both the phase velocity and the amplitude of the waves are altered by the modulation. The waves are generated and detected by using piezoelectric transducers. The separation of the fluid and the electrical signal domain by using the acoustical signal domain (tandem transducer concept) prevents parasitic and drift effects. By using the acoustic-
wave device as the frequency-determining element in an ultra-stable oscillator, the modulation of the waves is monitored very accurately.

Acoustic waves are attractive for the development of sensors for liquid properties such as density and viscosity because there are no moving parts, which are a drawback in many existing detectors. Acoustic waves can also be applied for use in (bio)chemical sensors. Small mass changes due to molecule adsorption at a (selective) chemically active thin film at the sensor surface can be monitored. The fact that solely mass adsorption is sufficient for the modulation of the acoustic wave is an advantage.

The sensitivity of acoustic-wave sensors can be increased by using wave types which show a high concentration of acoustic energy at the surface of the solid in which they propagate. Examples are Rayleigh waves or surface acoustic waves (SAW), Love waves and Lamb waves. For Rayleigh waves, most of the wave energy is located within the depth of one wavelength (typically between 10 and 80 μm in realized devices). Love waves are "trapped waves" in a thin guiding layer on top of the substrate. The thickness of the guiding layer is typically one tenth of the wavelength. Lamb waves can propagate in plates of which the thickness is smaller than the wavelength. By applying thin plates, a high concentration of acoustic energy at the surface is obtained. In bulk acoustic-wave (BAW) devices and acoustic plate-mode devices (such as the shear horizontally (SH) plate mode) the energy is distributed over the thickness of the substrate, which results in a lower sensitivity.

When considering sensing in liquids, shear-BAW and SH plate-mode devices show low acoustic radiation losses because the particle displacements at the surface are shear (in-plane). Of the three "sensitive" wave types mentioned, Rayleigh waves are not attractive for using in liquids because of the strong acoustic losses caused by the normal component of the particle displacement. Love waves are purely shear polarized waves and show little acoustic radiation into the liquid. Symmetric and asymmetric Lamb waves are associated with Rayleigh waves, and have both shear and normal displacement components. However, if the velocity of asymmetric Lamb waves is lower than the (compressional) sound speed in the liquid, the wave does not radiate energy into the liquid. Instead, a liquid layer is evanescently moved by the wave. Therefore, slow Lamb waves show a high sensitivity to fluid density in contrast to Love waves.
1.2 Silicon implementation

Because the silicon integrated circuit (IC) technology has developed to be the most reproducible, reliable and cost-effective fabrication technology in existence, activities to develop and make use of this technology for the fabrication of sensors has been growing fast since the seventies [5]. An additional feature of silicon implemented sensors is the possibility of integrating (part of) the electronic circuitry on the sensor chip thus giving a conditioned sensor output signal which can directly be connected to a standard electronic processor such as a microcontroller. A silicon sensor containing associated electronic circuitry is called a "smart sensor".

Acoustic waves can be generated and detected in silicon substrates by applying a piezoelectric zinc oxide thin film on top of the silicon, a planar technology compatible with the IC technology. The c-axis of the zinc oxide layers produced by standard zinc oxide deposition facilities is oriented perpendicular to the substrate surface. This allows the efficient generation and detection of Lamb waves and Rayleigh waves in ZnO-Si layered transducer. Using this technology, a monolithic Rayleigh wave oscillator for sensor applications was developed [6]. The velocity of zero-order asymmetric Lamb waves is co-determined by the thickness of the plate in which they propagate. The required slow waves are obtained if very thin plates are used (typically 2-10 μm). The use of silicon micromachining technology enables the fabrication of such thin plates.

1.3 Objective and organization of the thesis

This thesis describes the design and fabrication of a slow Lamb-wave sensor for the determination of physical and chemical properties of fluids. The acoustic and electronic parts are monolithically integrated which results in a multi-purpose smart sensor system on a single chip.
In chapter 2 the propagation of slow asymmetric Lamb waves in a thin plate is modeled by using the elementary theory for the propagation of flexural waves. A three-dimensional drawing of the ZnO-Al-SiO₂-Si layered plate is shown in Fig. 2.3. The sensitivity to such parameters as mass adsorption, density and viscosity are calculated.
The design considerations for the different parts of the sensor system are given in chapter 3. An oscillator setup was chosen because of its inherent high stability. This configuration yields a high resolution and a low detection limit. The combination of a silicon integrated acoustic-wave device with the bipolar amplifier circuitry imposes special design restrictions. In chapter 4 the sensor fabrication is described in detail, in particular the special processing for the fabrication of the Lamb-wave devices, which comprises the deposition and etching of piezoelectric zinc oxide layers and the micromachining of silicon for the fabrication of the thin plates in which slow Lamb waves are to be generated and detected. Special attention is given to the monolithic integration of the acoustic-wave device and electronic circuitry. The performance of the monolithically integrated sensor system is presented in chapter 5. In the final section of this chapter, the conclusions are presented together with the scope of future work.
Chapter 2  
Modeling of Lamb waves

2.1 Introduction

Lamb waves represent elastic disturbances propagating in a laterally infinite solid plate with free boundaries, for which displacements occur both in the direction of wave propagation and perpendicular to the plane of the plate boundaries. Two groups of waves can propagate through the plate independently of each other: symmetrical waves and asymmetrical waves. The plate deformations of both wave types are shown in Fig. 2.1 [7].

![Diagram of Lamb waves](image)

(a)  
(b)

Fig. 2.1  
(a) Symmetrical Lamb waves: on both sides of the middle plane, the longitudinal components are equal and the shear components have opposite signs.  
(b) Asymmetrical Lamb waves: on both sides of the meridian plane, the longitudinal component changes sign whereas the shear component does not.  
(After Dieulesaint).
In a plate of thickness $h$ at a frequency $\omega$ an infinite number of symmetrical and asymmetrical waves can exist, each with different phase and group velocities [8,9]. In Fig. 2.2 the phase velocities of the lowest three modes of symmetrical and asymmetrical waves are shown as a function of the normalized thickness $kh$, where $k=2\pi/\lambda$ is the wave number and $\lambda$ is the wavelength. The velocity of the asymmetric zero-order ($A_0$) Lamb wave decreases monotonously to zero for decreasing plate thickness. It is this property which makes the $A_0$ wave mode attractive for use in liquids, as discussed further on.

![Graph showing phase velocities of Lamb waves](image)

**Fig. 2.2** Phase velocities of the lowest three asymmetrical and symmetrical Lamb wave modes. $c$ is the phase velocity, $c_R$ the Rayleigh wave velocity (after Viktorov).

The curves shown in Fig. 2.2 are exact solutions of the Lamb-wave propagation problem. They are valid for a stress-free homogeneous isotropic laterally infinite flat plate [10]. When designing a Lamb-wave sensor, these conditions cannot all be achieved. Fig. 2.3 shows a three-dimensional drawing and a cross-section of a micromachined Lamb-wave sensor implemented in silicon. Instead of a free laterally infinite plate, this plate has restricted dimensions and is clamped on all sides by the silicon rim. The material is not homogeneous and isotropic but consists of a ZnO-Al-SiO$_2$-Si layered structure. And, due to the fabrication processes, the plate is stressed.

The propagation of waves in plates loaded by a fluid, governed by the exact equations of elasticity, is a complex problem and these equations are not attractive to use for the design of the sensors. The available software for the
The ZnO-Al-SiO$_2$-Si layered plate realized by silicon micromachining: three-dimensional drawing and cross-section (not to scale).

calculation of the velocity of Lamb waves provides no insight into the parameters that determine the sensitivity of the wave to fluid properties. In addition, prestress effects which occur in practical devices are not implemented in the available software. In this chapter simple equations are derived which approximate the exact solution for Lamb waves and which can be used as a design tool for silicon integrated Lamb-wave sensors for the determination of fluid properties. For this purpose, flexural motions in thin beams and plates are
considered in order to approximate the behavior of the $A_0$ Lamb-wave mode (in the simplest theory of flexural vibrations of beams and plates, it is assumed that the motion of each element of the beam or plate is purely one of translation in a direction perpendicular to the surface [11]).

As shown in Fig. 2.3, an acoustic delay line is used as the sensing element. It consists of two interdigital transducers placed in-line on a non-conductive piezoelectric substrate. The delay-line arrangement is reviewed in section 2.2. In section 2.3 equations are derived for the phase and group velocity of flexural waves in a thin beam and in a thin plate by using differential equations of motion. The equations approximate the Lamb-wave velocities and the validity of these approximations are evidenced by comparing them with the results of experiments on fabricated silicon devices.

In section 2.4, the effect of fluid loading of the plate on the phase velocity and attenuation is added to the equations given in 2.3. The influence of physical properties of liquids, such as density, viscosity and sound speed, on the wave propagation is discussed. Also, the sensitivity to mass adsorption at the plate surface in the gas phase and the liquid phase is considered.

### 2.2 Acoustic delay line as sensing element

The InterDigital Transducer (IDT) was invented in 1962 [12] and applied for the generation of bulk acoustic waves. This same structure proved to be highly useful for the generation of several other acoustic-wave modes, such as surface acoustic waves [13,14]. Also Lamb waves can be generated by using IDTs which increased the interest in the study of the generation and propagation of Lamb waves [15,16,17,18].

Fig. 2.4 shows a uniform single-electrode and a uniform split-electrode interdigital transducer, which consist of thin metal electrodes of equal width and spacing, and a constant overlap ($W$). The metal electrodes are placed on top of, or underneath a piezoelectric solid. By applying a radio-frequent electrical voltage to the transducer, the electrodes become alternately positively or negatively polarized, which creates an electric field distribution between the electrodes. The repetitive electric field produces a corresponding repetitive strain (stress) field in the piezoelectric substrate, resulting in acoustic waves propagating away from the transducer in both directions.
In the frequency domain, the interdigital transducer shows a pass-band behavior. The center frequency $f_c$ is determined by both the period $p$ and the phase velocity $c$ of the acoustic wave:

$$f_c = \frac{c}{p} \tag{1}$$

By placing a second transducer in line with the first, a delay line is formed. The generated wave travels through the propagation path to the receiving transducer where conversion of the acoustic signal into a voltage occurs. The delay-line configuration is a basic element for acoustic-wave based sensors. In the propagation path of the wave (the area covered by the IDTs and the free path between them) the interaction between the wave and the fluid takes place, resulting in the modulation of wave velocity and wave attenuation. A delay line with uniform IDTs is shown in Fig. 2.5.

Acoustic reflections within the transducers caused by the discontinuity in the acoustic-wave impedance can be suppressed by applying a split-electrode transducer as shown in Fig. 2.4. Reflections at the edges of the delay line can be dealt with by using an acoustic absorber or by using slanted edges, or a combination of both.

Many acoustic-wave devices have been realized in piezoelectric materials such as quartz, lithium niobate, and lithium tantalate. The use of (non-piezoelectric) silicon as the basis of an acoustic-wave device forces the deposition of a
piezoelectric layer on the silicon. The metal pattern for the IDT can be located either on top of this piezoelectric layer, or at the interface of the (oxidized) silicon substrate and the piezoelectric layer (see chapter 3). The described IDT is also used for the generation and detection of $A_0$ Lamb waves in thin silicon-based plates. In the work here reported, a ZnO-Al-SiO$_2$-Si layered structure as shown in Fig. 2.3 was used.

2.3 Flexural waves in unloaded thin plates

To obtain equations for the phase and group velocity of flexural waves in thin plates, we start by considering a differential element of an unloaded homogeneous isotropic thin beam undergoing a transverse motion (see Fig. 2.6), and assuming that plane cross-sections initially perpendicular to the neutral plane of the beam remain plane and perpendicular to the neutral plane (the $xz$ plane in the middle of the beam is called the neutral plane). The plane waves propagate in the $x$ direction. The element is acted on by a bending moment $M$ and a shear force $F$.

It should be noted that in the following theory, only the real positive roots of the equations are considered.
Fig. 2.6 (a) Cross-section of the beam, (b) thin beam undergoing transverse motion, (c) forces and moments on a differential element of the unloaded beam.

2.3.1 Elementary equations

The relationship between the bending moment $M$ and curvature of the beam (the displacements of the beam are assumed to be small) is given by [19]

$$M = -EI\frac{\partial^2 u_y}{\partial x^2}$$  \hspace{1cm} (2)

where $u_y$ the displacement in the $y$ direction, $E$ is Young's modulus and $I$ the second moment of area of the cross-section of the beam,
\[ I = \frac{W_b h^3}{12} \]  

where \( W_b \) is the width and \( h \) the thickness of the beam. The elementary equation of motion [19] for the element of a beam is

\[ -F + (F + \frac{\partial F}{\partial x} dx) = \rho A dx \frac{\partial^2 u_y}{\partial t^2} \]  

where \( A \) is the cross-sectional area of the beam and \( \rho \) is the mass density. This becomes

\[ \frac{\partial F}{\partial x} = \rho A \frac{\partial^2 u_y}{\partial t^2} \]  

If the rotary-inertia effects of the element are neglected, the moment equation becomes

\[ F = \frac{\partial M}{\partial x} \]  

Substitution gives

\[ \frac{\partial^2 M}{\partial x^2} = \rho A \frac{\partial^2 u_y}{\partial t^2} \]  

Finally,

\[ EI \frac{\partial^4 u_y}{\partial x^4} + \rho A \frac{\partial^2 u_y}{\partial t^2} = 0 \]  

is obtained. If time-harmonic (sinusoidal) free flexural plane waves propagate along the beam with phase velocity \( c \), the expression

\[ u_y = \hat{u}_y e^{i(\omega t - kx)} \]  

where \( \omega \) is the angular frequency, yields
\[ EI k^4 - \rho A \omega^2 = 0 \]  

(10)

By substitution of \( \omega = ck \), the following expression for the phase velocity \( c \) is found:

\[ c = k \sqrt{\frac{EI}{\rho A}} \]  

(11)

The group velocity \( c_{gr} \) is then given by

\[ c_{gr} = c + k \frac{\partial c}{\partial k} = 2c \]  

(12)

2.3.2 Presence of prestress

If the beam is prestressed due to an in-plane force \( N_b \) acting in the middle plane of the beam (in-plane stress, see Fig. 2.7), and if we assume that axial-inertia effects are negligible, the equation of vertical motion becomes [19]

\[ -F + (F + \frac{\partial F}{\partial x} dx) - N_b \theta + (N_b + \frac{\partial N_b}{\partial x} dx) (\theta + \frac{\partial \theta}{\partial x} dx) = \rho Adx \frac{\partial^2 u_y}{\partial t^2} \]  

(13)

where \( \theta \) is the angle between the direction of the prestress and the neutral plane in the considered element.

---

**Fig. 2.7** Forces and moments on a differential element of an unloaded homogeneous beam, including in-plane prestress.
The prestress is assumed constant, so that
\[ \frac{\partial N_b}{\partial x} = 0 \]  
(14)

which reduces the equation of vertical motion to
\[ \frac{\partial F}{\partial x} + N_b \frac{\partial \theta}{\partial x} = \rho A \frac{\partial^2 u_y}{\partial t^2} \]  
(15)

and with
\[ \theta = \frac{\partial u_y}{\partial x} \]  
(16)

we find
\[ \frac{\partial F}{\partial x} + N_b \frac{\partial^2 u_y}{\partial x^2} = \rho A \frac{\partial^2 u_y}{\partial t^2} \]  
(17)

And, by substitution of equations (2) and (6):
\[ EI \frac{\partial^4 u_y}{\partial x^4} + \rho A \frac{\partial^2 u_y}{\partial t^2} - N_b \frac{\partial^2 u_y}{\partial x^2} = 0 \]  
(18)

For time-harmonic free flexural waves this becomes
\[ EI k^4 - \rho A \omega^2 + N_b k^2 = 0 \]  
(19)

The corresponding phase velocity is given by
\[ c = k \sqrt{\frac{EI + \frac{N_b}{k^2}}{\rho A}} \]  
(20)

The corresponding group velocity is now
\[ c_{gr} = c + \frac{k^2 EI}{\rho A} \frac{1}{c} = c \left(1 + \frac{c_{stress \ free}^2}{c^2}\right) \]  
(21)
where $c_{stress\ free}$ is the phase velocity for a stress-free beam.

Equations (11) and (20) give the result that waves of infinitely short wavelength would travel with infinite velocity. This is physically incorrect, and the obtained relation is in fact only applicable to waves for which $\lambda >> h$. For smaller wavelengths, the motions of the element must again be considered. Two corrections to the elementary theory (ET) are discussed in the following sections: the rotary-inertia (RI) correction and the shear correction (SC).

### 2.3.3 Rotary-inertia correction

First, the assumption that the motion is purely one of translation in the $y$ direction is unjustified for short wavelengths (Fig. 2.8). An extra term concerning the rotary motion of the considered element must be added to the elementary equation of motion.

![Diagram showing forces and moments on a differential element of an unloaded homogeneous beam, including rotary inertia.](image)

*Fig. 2.8  Forces and moments on a differential element of an unloaded homogeneous beam, including rotary inertia.*

By taking the moments, the resultant couple must be equated to the product of the moment of inertia of the element and its angular acceleration.

If we assume zero in-plane stress,
(F - \frac{\partial M}{\partial x})dx = \rho I \frac{\partial^2 \psi}{\partial t^2} dx \tag{22}

where \( \psi \) is the angle through which the section rotated. For small deformations \( \psi \) will be given by

\[ \psi = \frac{\partial u_y}{\partial x} \tag{23} \]

so that

\[ F = \frac{\partial M}{\partial x} + \rho I \frac{\partial^4 u_y}{\partial x \partial t^2} \tag{24} \]

and

\[ \frac{\partial F}{\partial x} = \frac{\partial^2 M}{\partial x^2} + \rho I \frac{\partial^4 u_y}{\partial x^2 \partial t^2} \tag{25} \]

Substituting equation (2) and (6) yields

\[ \rho A \frac{\partial^2 u_y}{\partial t^2} = -EI \frac{\partial^4 u_y}{\partial x^4} + \rho I \frac{\partial^4 u_y}{\partial x^2 \partial t^2} \tag{26} \]

This equation contains the rotary inertia, and with the earlier used time-harmonic solution the following equation is obtained

\[ EIk^4 - \rho A \omega^2 - \rho I k^2 \omega^2 = 0 \tag{27} \]

For a stress-free beam the phase velocity \( c \) is now given by

\[ c = k \sqrt{\frac{EI}{\rho A \left(1 + \frac{(kh)^2}{12}\right)}} \tag{28} \]
or rewritten

\[ c = \frac{E(kh)^2}{\sqrt{\rho \left( 12 + (kh)^2 \right)}} \]  \hspace{1cm} (29)

If prestress is included, equation (27) becomes

\[ Elk^4 - \rho A\omega^2 + N_b k^2 - \rho I k^2 \omega^2 = 0 \]  \hspace{1cm} (30)

and the velocity in the prestressed beam is

\[ c = \sqrt{\frac{E + \frac{12N_b}{A(kh)^2}}{\rho \left( 12 + (kh)^2 \right)}} \]  \hspace{1cm} (31)

The correction term results in an asymptotic value for \( c \) at large values of \( kh \):

\[ c_{kh \rightarrow \infty} = \sqrt{\frac{E}{\rho}} = c_{long} \]  \hspace{1cm} (32)

where \( c_{long} \) is the longitudinal velocity. Thus, the incorporation of the rotary inertia yields a limit for the phase velocity. The limit found can be seen as the maximum velocity which can exist in an elastic solid.

The group velocity (stress-free) is now given by

\[ c_{gr} = 2c - c \frac{(kh)^2}{12 + (kh)^2} \]  \hspace{1cm} (33)

For small values of \( kh \) (\( kh \rightarrow 0 \)) we find \( c_{gr} \rightarrow 2c \) and for large values of \( kh \) (\( kh \rightarrow \infty \)) we find \( c_{gr} \rightarrow c \).

The correction for rotary inertia is known as the Rayleigh correction [20,11].

2.3.4 Shear correction

The second correction applied to the elementary theory (ET) is the shear correction (SC). If the wavelength is comparable to the thickness of the beam, longitudinal sections of elements of the beam do not remain rectangular during motion [21,11]. Shear forces will distort each element, as is shown in Fig. 2.9.
The slope of the axis of the beam, given by $\partial u_\gamma / \partial x$, now consists of a contribution $\psi$ caused by bending and a contribution $\gamma$ caused by shearing. The differential equation then becomes

$$\frac{\partial^2 u_y}{\partial t^2} + \frac{EI}{\rho A} \frac{\partial^4 u_y}{\partial x^4} - \frac{I}{A} \frac{1}{rG} \frac{\partial^4 u_y}{\partial x^2 \partial t^2} + \frac{\rho I}{rGA} \frac{\partial^4 u_y}{\partial t^4} = 0 \quad (34)$$

where the rigidity modulus $G = \frac{1}{2} E/(1+\nu)$, $\nu$ is the Poisson constant, and $r$ is an adjustment term which depends on the shape of the cross-section [11,19,22]. For rectangular cross-sections $r \approx 0.833$.

The time-harmonic wave solution then gives

$$-\omega^2 + \frac{EI}{\rho A} k^4 - \frac{I}{A} \frac{1}{rG} (1+\nu) k^2 \omega^2 + \frac{\rho I}{rGA} \omega^4 = 0 \quad (35)$$

which yields the following non-explicit expression for the phase velocity:

$$c = k \sqrt{\frac{EI}{\rho A \left(1 + \frac{(1+\nu) k^2 \omega^2}{rG} \right) \left(\frac{(kh)^2}{12}\right)}} \quad (36)$$
or, rewritten

\[ c = \sqrt{\frac{E(kh)^2}{\rho \left( 12 + \left( 1 + \frac{E - \rho c^2}{rG} \right)(kh)^2 \right)}} \]  

(37)

The asymptotic behavior of equation (35) is examined by letting \( kh \to \infty \) and substitution of \( \omega = ck \):

\[ c_{kh \to \infty}^4 - \left( \frac{rG+E}{\rho} \right) c_{kh \to \infty}^2 + \frac{ErG}{\rho^2} = 0 \]  

(38)

which yields two positive roots of interest:

\[ c_{kh \to \infty} = \sqrt{\frac{E}{\rho}} \quad \text{and} \quad c_{kh \to \infty} = \sqrt{\frac{rG}{\rho}} \]  

(39)

The first solution was given earlier when introducing the Rayleigh correction, the second root is a smaller upper-limit which is regarded as the physical limit. The shear correction is also known as the Timoshenko correction.

### 2.3.5 Plane waves in a clamped layered flat plate

As discussed in section 2.1, the silicon device under consideration is not homogeneous and isotropic but consists of a layered structure. In addition, the device is not a beam with free sides, but a four-sided clamped plate (see Fig. 2.3). These issues are addressed in this section after which the ultimate velocity expressions which can be used for the fabricated silicon implemented devices are given.

The zinc oxide layers are transversely isotropic because the c-axis is perpendicular to the substrate surface. In the <100>-cut silicon, a pure mode propagation direction is chosen ([110]). The oxide and aluminum layer are considered isotropic.
In order to adapt the equations derived for the velocities to the real situation, the silicon device is examined. If we consider normal stresses $\sigma_x$ and $\sigma_z$ on an element the strains are given by

$$\varepsilon_x = \frac{\sigma_x}{E} - \frac{\nu\sigma_z}{E}, \quad \varepsilon_z = \frac{\sigma_z}{E} - \frac{\nu\sigma_x}{E}$$  \hspace{1cm} (40)

When the beam is clamped in the $z$ direction, there is no strain in that direction: $\varepsilon_z = 0$. So,

$$\sigma_z = \nu\sigma_x, \quad \varepsilon_x = \frac{\sigma_x}{E}(1-\nu^2)$$  \hspace{1cm} (41)

yielding

$$\sigma_x = \frac{E\varepsilon_x}{(1-\nu^2)}$$  \hspace{1cm} (42)

Thus by replacing $E$ by $E^*=E/(1-\nu^2)$ the clamping of the sides is accounted for. Instead of the stiffness term $EI$ we can now use the bending stiffness $D$ of the plate [23]:

$$D = \frac{E_{\text{plate}}h^3}{12(1-\nu_{\text{plate}}^2)} = \frac{E^*_{\text{plate}}h^3}{12}$$  \hspace{1cm} (43)

and $\rho A$ can be replaced by $m$, $m$ being the weighted average plate mass per unit area.

When considering the material constants of the plate, $E_{\text{plate}}$, $\nu_{\text{plate}}$, and $\rho_{\text{plate}}$, it is clear that because of the layered structure, the values of these constants vary according to the thicknesses of the different layer materials. The constants are found by using the weighted values of the individual layer materials.

The phase velocities found for the elementary theory (ET), the corrected theories rotary inertia (RI) and shear correction (SC), and for in-plane stress (SC+N, $N=N_p/W_s$ is the in-plane prestress per unit width in N/m) are now given by
\[ c = k \sqrt{\frac{D}{m}} = \sqrt{\frac{E_{plate}^*(kh)^2}{12\rho_{plate}}} \quad ET \]  

\[ c = \sqrt{\frac{E_{plate}^*(kh)^2}{\rho_{plate}(12+(kh)^2)}} \quad RI \]  

\[ c = \sqrt{\frac{E_{plate}^*(kh)^2}{\rho_{plate} \left(12 + \left(1 + \frac{E_{plate}^* - \rho_{plate}c^2}{rG_{plate}}\right)(kh)^2\right)}} \quad SC \]  

\[ c = \sqrt{\frac{E_{plate}^* + \frac{12N}{h(kh)^2}}{\rho_{plate} \left(12 + \left(1 + \frac{E_{plate}^* - \rho_{plate}c^2}{rG_{plate}}\right)(kh)^2\right)}} \quad SC+N \] 

In Fig. 2.10 the plate wave velocities calculated by using the elementary theory (ET) and the corrected theories (RI and SC), are given as a function of the normalized silicon thickness \( h_{sl}/p \) for a ZnO-Al-SiO\(_2\)-Si layered plate. The normalized thicknesses (expressed in \( h/p \)) of the ZnO, Al, and SiO\(_2\) layer are 0.01625, 0.00375, and 0.00125, respectively. Weighted averages are used for finding the constants \( E_{plate}^* \) and \( v_{plate} \). In addition, the exact solution (ES) for A\(_0\) Lamb waves in a free plate is shown, calculated with the use of dedicated software [24] based on a matrix approach [25].

For \( h_{sl}/p > 0.1 \), the ET and RI curves give large errors. The elementary theory shows no limit of the velocity at increasing \( h_{sl}/p \) and the rotary-inertia correction yields the longitudinal velocity as an asymptotic value. The asymptotic velocity of the shear correction is only a few percent larger than the value found from
the exact solution for A₀ Lamb waves. The exact asymptotic value for $h_{si}/p \to \infty$ is the Rayleigh-wave velocity of the considered structure [22,26]. For $h_{si}/p < 0.1$ the results of the four methods lie close together.

Fig. 2.10 shows a detail of Fig. 2.10 for small values of $h_{si}/p$. These values correspond to the plate thickness of fabricated silicon plate wave sensors (the silicon layer thickness is 2 to 8 µm, the wavelength amounts to 80 µm, yielding normalized silicon thicknesses between 0.025 and 0.1). Note that at zero silicon thickness, the velocities given in Fig. 2.11 are related to the ZnO-Al-SiO₂ structure.

In the 0.025 to 0.1 region of the normalized silicon thickness, the minimum and maximum velocity deviation between the ET and the ES are 1 % (at $h_{si}/p=0.025$) and 15 % (at $h_{si}/p=0.1$). In addition, the results of the SC deviates only from 1 % to about 7 % from the ES, in the same range.

The influence of in-plane prestress is demonstrated in Fig. 2.12. A prestress of 500 MPa is assumed for the ET+N and SC+N curves (stresses of 500 MPa were found in practical devices). The used software for the calculation of the exact solution provides no prestress option. The stress increases the velocity, particularly if the plate becomes very thin.
Fig. 2.11  Wave velocities in a ZnO-Al-SiO$_2$-Si layered plate versus normalized silicon layer thickness calculated by using ET, RI, SC, and ES.

Fig. 2.12  Stress-free and prestressed (in-plane) wave velocities in a ZnO-Al-SiO$_2$-Si layered plate plotted for ET and SC (the in-plane stress is denoted by N).
Fig. 2.13  Experimental (error bars) and calculated (curves) wave velocities of a ZnO-Al-SiO$_2$-Si layered plate. Both stress-free and prestressed (in-plane) calculations are plotted for ET and SC.

In Fig. 2.13, experimental results (error bars) are shown in addition to the calculated curves$^a$. The horizontal error bars are caused by the uncertainty of the absolute thickness of the plates, the vertical error bars represent the measurement error of the operating frequency. The results correspond very well to the calculated curves and thereby confirm the validity of the beam and infinite-plate theory to describe the wave behavior.

$^a$ The devices used for these experiments were ZnO-Al-SiO$_2$-Si layered Lamb wave devices with layer thicknesses of 1.3\textmu m, 0.3\textmu m, 0.1\textmu m and 7\textmu m, respectively. Two different wavelengths were used: 80\textmu m and 96\textmu m. After measuring the operating frequency by using an impedance analyzer, the silicon layers were thinned (in steps) from the backside by plasma etching to a value of about 4.5\textmu m, and measured again.
2.4 Fluid loading of flexural waves

The phase and group velocity, and also the attenuation of flexural waves are altered if the plate is loaded by a fluid. There is a strict division of the nature of acoustic-wave fluid interaction between the purely inertial loading for subsonic velocities in plates, and resistive or damping-like loading for supersonic velocities. The lossless character of the subsonic waves makes them attractive for sensor applications.

In section 2.4.1, the equations given in section 2.3 are extended to account for the influence of fluid loading (semi-infinite) of the plate surface, by using an impedance method for the velocity of flexural waves. In section 2.4.2 the sensitivity of the wave velocity for several fluid properties is discussed, such as liquid density, viscosity and sound speed, and mass adsorption at the plate surface in a gaseous or liquid medium.

2.4.1 Loading by a semi-infinite fluid

If a plate and a fluid are subjected to a coupled vibration (no slip occurs), the normal displacement of plate particles and fluid particles are equal, which yields a boundary condition. The interaction is evaluated in terms of a wave impedance, an approximation only valid when the plate thickness is much smaller than the wavelength of the transverse wave in the solid [27]. The transverse wave impedance is defined as the ratio of applied pressure to resulting transverse velocity, at any point, under the forced time-harmonic wave motion generated by the pressure distribution of a hypothetical plane wave, which propagates parallel to the plate surface with the (variable) phase velocity $c$ [28].

The wave impedance is obtained by applying a distributed time-harmonic pressure $P(x)$:

$$P(x) = \hat{P} e^{j(\omega t - kx)}$$  \hspace{1cm} (48)

to the structure in the form of a time-harmonic (sinusoidal) traveling wave and deriving the plate response from the equation of motion [28,29]. The waves will propagate with a velocity at which a minimum of energy is required for their maintenance (after the pressure $P(x)$ has been "switched off"). The corresponding wave impedance of the plate-fluid system must be a minimum,
and when there is no loss in the system the impedance will be zero. Only the case of interaction is considered, where a laterally infinite plate is loaded on one side by a fluid of semi-infinite extent.

Fig. 2.14 Propagating plane wave in a fluid-loaded plate (one side).

Ideal fluids
Because of the absence of shear stresses in an ideal liquid, the stresses (pressure) existing in it always act perpendicular to any surface area in the liquid, and the force of the pressure applied to the fluid particles produces only translational motion (see Fig. 2.14).

By introducing the transverse velocity \( v_y \) of a particle at the surface of the plate moved by a wave propagating in the \( x \) direction

\[
v_y = \hat{v}_y e^{j(\omega t - kx)}
\]

in which

\[
v_y = \frac{\partial u_y}{\partial t} = j\omega u_y
\]

The differential equation of motion given in section 2.3 can be rewritten, now giving the pressure distribution for the generation of the wave [28]

\[
P(x) = j\omega mv_y + \frac{D}{j\omega} \frac{\partial^4 v_y}{\partial x^4}
\]
This yields a transverse wave impedance $Z_{plate}$ given by

$$Z_{plate} = \frac{P(x)}{v_y} = j\omega m + \frac{D\omega^3}{jc^4} \tag{52}$$

The impedance of an ideal (non-viscous) fluid loading the surface is found by using the wave equation for a velocity potential $\psi$ [30]

$$\nabla^2 \psi = \frac{1}{c_f^2} \frac{\partial^2 \psi}{\partial t^2} \tag{53}$$

where $c_f$ is the sound speed of the fluid. The pressure $P$ is now given by

$$P = -\rho_f \frac{\partial \psi}{\partial t} \tag{54}$$

where $\rho_f$ is the density of the fluid. The particle velocity in the $y$ direction is

$$v_y = \frac{\partial \psi}{\partial y} \tag{55}$$

When a semi-infinite fluid layer loads the plate, the wave impedance is calculated by applying a time-harmonic wave again. If there is a perfect coupling of fluid and plate (no slip occurs) the boundary condition is given by

$$\psi = \Psi e^{-jkx} e^{j\omega t} \tag{56}$$

at the surface $y=0$. The general solution

$$\psi(x,y,t) = \Psi e^{-jkx+ky} e^{j\omega t} \tag{57}$$

has to satisfy the boundary condition. The propagation constant $k_y$ is found by
substitution of equation (57) in the wave equation

\[ k_y = \sqrt{\frac{\omega^2 - k^2}{c_f^2}} = \frac{\omega}{c} \sqrt{\frac{c^2}{c_f^2} - 1} \]  

(58)

The following expressions for the pressure, particle velocity and fluid impedance are found:

\[ P = -j\omega \rho_f \psi, \quad v_y = -jk_y \psi, \quad Z_f = \omega \frac{\rho_f}{k_y} \]  

(59)

Thus, the impedance of the fluid \( Z_f \) is

\[ Z_f = \frac{\rho_f c}{\sqrt{\frac{c^2}{c_f^2} - 1}} \]  

(60)

Two regions are observed: a high-velocity region \( c > c_f \) and a low-velocity region \( c < c_f \). In the high-velocity region, the impedance \( Z_f \) is real and radiation is directed into the fluid resulting in acoustic losses in the plate [28,31]. In the low-velocity region \( Z_f \) becomes imaginary, and the load is a mass reactance and causes no radiation losses. When the wave velocity equals the fluid velocity, wave propagation is not possible because of the infinite loads.

The lossless wave mode, existing if \( c < c_f \), is very attractive for sensor applications, not only because of the absence of propagation losses in an ideal fluid, but also because of the low frequency of operation and the high sensitivity, as discussed further on.

We continue to examine this special case for flexural waves.

Realizing that for the coupled plate-fluid system the transverse velocities of both fluid and plate must be equal at the plate surface, the resulting impedance \( Z \) is found by placing \( Z_{\text{plate}} \) and \( Z_f \) in series,

\[ Z = Z_{\text{plate}} + Z_f \]  

(61)

For \( c < c_f \) the impedance of the fluid is imaginary and can be written as
\[ Z_f = j \frac{\rho_f c}{\sqrt{1 - \frac{c^2}{c_f^2}}} \]  
(62)

which yields

\[ Z = j \left( \omega m - \omega^3 \frac{D}{c^4} + \frac{\rho_f c}{\sqrt{1 - \frac{c^2}{c_f^2}}} \right) \]  
(63)

The impedance is purely imaginary and the required velocity of propagation of free flexural waves is found by setting \( Z = 0 \), yielding

\[ c = k \sqrt{\frac{D}{m + \rho_f \delta_f}} \]  
(64)

Thus, the liquid loading of the plate can be seen as a mass reactance, not only the plate mass but also the mass of an evanescent-moved fluid layer determines the wave velocity. This fluid layer thickness \( \delta_f \) is represented by

\[ \delta_f = \frac{1}{k \sqrt{1 - \frac{c^2}{c_f^2}}} \]  
(65)

**Viscous fluids**

In viscous fluids, shear stresses will occur if a transverse wave propagates through the plate, which results in a (small) alteration of the velocity caused by viscous mass-loading. The viscous mass-loading \( m_{\text{visc}} \) is given by [32,27]

\[ m_{\text{visc}} = \frac{\rho_f \eta}{\sqrt{2\omega}} \]  
(66)

in which the viscosity \( \eta \) is represented.

Only Newtonian liquids are considered, where the viscosity is independent of the
wave frequency\(^b\) [33,34]. The wave velocity (\(ET\)) including the viscous mass-loading and prestress now becomes

\[
c = k \sqrt{\frac{D + \frac{N}{k^2}}{m + \rho_f \delta_f^+ \sqrt{\frac{\rho_f \eta}{2\omega}}}}
\]  
\(67\)

Also for the velocity expressions found for the rotary-inertia and shear corrections, the mass reactance and the viscous mass-loading can be added to the plate mass \(m\).

Due to the viscous loading, the wave propagation is no longer lossless and an attenuation factor \(\alpha\) can be derived [35,36]

\[
\alpha = \left(\frac{\omega}{2}\right)^{3/2} \frac{\delta_f^2 k \sqrt{\rho_f \eta}}{2Dk^2 + N + \frac{\omega^2 \rho_f \delta_f}{k^2}}
\]  
\(68\)

where the corrections for rotary inertia and shear in the plate are neglected. \(\alpha\) gives the loss in Neper/m (1 Neper = 8.686 dB).

\[2.4.2\] Sensitivity

The fact that the phase velocity of the \(A_0\) Lamb wave depends on the plate mass and the fluid properties adjacent to the plate surface opens up the way to several sensor applications.

Because wave amplitude and wave velocity are affected differently by the fluid parameters, simultaneous monitoring of several properties is possible. For

\(^b\) A liquid is considered to be Newtonian if \(\omega \tau < 1\), where \(\omega\) is the angular frequency and \(\tau\) the liquid shear relaxation time given by \(\tau = \eta/G_w\). \(G_w\) is the high frequency rigidity modulus of the liquid and has a typical value of 1 GPa, \(\eta\) is the shear viscosity. The viscosity at which the liquid is considered Newtonian depends on the wave frequency: a frequency of 12 MHz results in \(\eta < < 13\) Pa.s.
example, in low-viscous liquids the density strongly influences the velocity while the viscosity has a negligible effect. The propagation losses, however, are greatly dependent on the viscosity as can be seen in equation (68).

In this section, the sensitivity to mass adsorption from a gaseous or a liquid medium (chemosensor) and the sensitivity to liquid density, viscosity and sound speed is discussed.

For the determination of the sensitivity, the simple equations derived from the elementary theory (ET) are used, because they provide a good insight into the design parameters for optimization of the sensitivity. The plate is assumed free from in-plane prestress and the fluid medium is assumed to be semi-infinite on one side of the plate.

The sensitivity $S_\omega^X$ is defined as

$$S_\omega^X = \frac{1}{\omega} \frac{\partial \omega}{\partial X} \quad \text{or} \quad S_f^X = \frac{1}{f} \frac{\partial f}{\partial X} \quad (69)$$

where $X$ is the quantity of interest.

In the calculations, the following plate parameters are assumed: plate mass $m=2.08 \times 10^2$ kg/m$^2$, total plate thickness $h=7$ μm ($h_{\text{zno}} = 1.3$ μm, $h_{\text{z}} = 0.3$ μm, $h_{\text{zioz}} = 0.1$ μm, $h_{\text{i}} = 5.3$ μm), IDT period $p=80$ μm, Young’s modulus $E^* = 157$ GPa, which yields a plate bending-stiffness $D=4.48 \times 10^6$ Nm and an operating frequency in air of 14.41 MHz (no in-plane stress).

Mass adsorption from a gaseous medium

In a gaseous medium, the angular frequency of a flexural wave is

$$\omega = k^2 \sqrt{\frac{D}{m}} \quad (70)$$

The sensitivity to mass adsorption per unit area at the sensor surface is given by

$$S_\omega^m = \frac{1}{\omega} \frac{\partial \omega}{\partial m} = -\frac{1}{2m} \quad (71)$$

Thinning the plate results in a larger sensitivity to mass adsorption.

For a sensor with a plate mass $m$ of $2.08 \times 10^2$ kg/m$^2$, a sensitivity of $-24$ m$^2$/kg or $-240$ cm$^2$/g is found. For the device operating at 14.41 MHz, an adsorption of a mass of 1 ng/cm$^2$ results in a frequency shift of -3.5 Hz.
Mass adsorption from a liquid medium
When one side of the plate is immersed in a semi-infinite viscous liquid, the angular frequency is

$$\omega = k^2 \frac{D}{\sqrt{m + \rho_f \delta_f + \rho_l \eta / 2\omega}} \quad (72)$$

The sensitivity to mass adsorption in a viscous liquid medium is approximated by

$$S_\omega = \frac{1}{\omega} \frac{\partial \omega}{\partial m} = -1 \left( \frac{m + \rho_f \delta_f + \rho_l \eta / 2\omega}{2} \right)^{-1} \quad (73)$$

Both $\delta_f$ and the viscous mass-loading term $m_{\text{visc}} = (\rho_l \eta / 2\omega)^{1/2}$ are assumed to be constant here. In low-viscous liquids ($\eta < 10^2$ Pa.s), the viscous mass-loading term can be neglected (at sufficiently high frequencies). It is remarkable that the absolute density and viscosity of the liquid have a dramatic effect on the sensitivity. The liquid parameters in which the sensor is operated must be known to determine the sensitivity.

The sensitivity to mass adsorption from a watery medium ($\rho_f = 1000$ kg/m$^3$, $\eta = 0.001$ Pa.s, $c_f = 1500$ m/s) at a silicon plate with given parameters (operating at a frequency of 10.89 MHz in water, Lamb-wave velocity 870 m/s) is about -13.7 m$^2$/kg or -137 cm$^2$/g. The mass sensitivity, compared to the sensitivity if operated in a gaseous medium, is almost halved. An adsorption of 1 ng/cm$^2$ results in a frequency shift of 1.5 Hz.

Both the approximated (upper curve) and the numerically obtained mass sensitivity (lower curve) in water are shown in Fig. 2.15. The deviation from the approximated value due to the alteration of $\delta_f$ and the viscous mass-loading term appears to be non-negligible: for the layered plate described earlier (in the figure marked by the vertical broken line, layer thicknesses given by 1.3 $\mu$m ZnO, 0.3 $\mu$m Al, 0.1 $\mu$m SiO$_2$ and 5.3 $\mu$m Si), the actual sensitivity is 12.4 m$^2$/kg.
Fig. 2.15 Approximated (upper curve) and numerically obtained (lower curve) mass sensitivity of a thin-plate Lamb wave operating in water.

**Density**

The sensitivity to liquid density is approximated by

$$S^p_\omega = \frac{1}{\omega} \frac{\partial \omega}{\partial \rho} \approx -\frac{\delta_f^* \sqrt{\frac{\eta}{8k \rho_f}}}{2 \left( m + \rho_f \delta_f^* + \frac{\rho_f \eta}{2k} \right)}$$  \hspace{1cm} (74)

Again, $\delta_f$ and the viscous mass-loading term are assumed constant. In water, and for the given plate parameters, a sensitivity of almost $2.2 \times 10^4$ m$^2$/kg is found, or 2400 Hz/(kg/m$^3$) at an operating frequency of 10.89 MHz. The extremely high sensitivity becomes clear in the following example: if the density of the water changes by 1%, a frequency change of 24000 Hz will be found.

The sensitivity to density can also be expressed in a relative form:

$$R^p_\omega = \frac{\rho_f}{\omega} \frac{\partial \omega}{\partial \rho_f}$$  \hspace{1cm} (75)

yielding a sensitivity of -0.22 %/\% (1 % density increase causes -0.22 %
frequency change). Also here, the real sensitivity has to be calculated numerically. In Fig. 2.16 the numerically found sensitivity (lower curve) and the approximated sensitivity (upper curve) are given as a function of the normalized silicon thickness, for a watery medium. For our plate the actual sensitivity amounts to 0.19 %/%. 

![Graph showing sensitivity value](image)

Fig. 2.16 Approximated (upper) and numerically found (lower) curves of the density sensitivity of a thin-plate Lamb wave sensor operating in water.

**Viscosity**

The approximate sensitivity to liquid viscosity is

\[
S^o_\omega = \frac{1}{\omega} \frac{\partial \omega}{\partial \eta} = \frac{- \sqrt{\frac{\rho_f}{2\omega \eta}}}{4 \left(m + \rho_f \delta_f + \frac{\rho \eta}{2\omega}\right)}
\]

(76)

Also in this case, \(\delta_f\) and the viscous mass-loading term are assumed constant. In water, and with the earlier given plate parameters, the approximate sensitivity amounts to about -0.59/Pa.s or -6.4 MHz/Pa.s for a device operating at 10.89 MHz. The relative sensitivity is \(-5.9 \times 10^4 \%/%\) which implies that a 1 % change in the water viscosity results in a 5.9 ppm change in frequency (for the
device described here this means a shift of 64 Hz).
The exact and approximated sensitivity to viscosity in a watery medium obtained from numerical calculations is given in Fig. 2.17. The two curves do not differ very much. Compared to the sensitivity to density, the viscosity sensitivity is very low.

\[ S_{\omega}^m = \frac{1}{\omega} \frac{\partial \omega}{\partial c_f} = \frac{+\rho_f c_f^2 c_f^2 \delta_f^3 k^2}{2 m + \rho_f \delta_f^+ \sqrt{\frac{\rho_f n}{2\omega}}} \]  
(77)

The viscous mass-loading term is assumed constant. For the 7 \( \mu \)m thick layered plate, a sensitivity of \(+7.1 \times 10^{-5}\) s/m is found in water, corresponding to a relative sensitivity of +0.11 %/\% (a 1 % increase of sound speed yields 0.11 % increase in frequency). In contradiction to the sensitivity to mass, density and viscosity,
the sensitivity to the sound speed has a positive sign. In addition, thicker plates show a higher sound speed sensitivity. This is also shown in Fig. 2.18, where the approximated (lower curve) and numerical (upper curve) results are given.

**Fig. 2.18** Approximated (upper curve) and numerically obtained (lower curve) sensitivity for sound speed of a thin-plate Lamb wave sensor operating in water.

**Acoustic losses caused by viscous loading**
The equation for the attenuation factor has been given in the previous section. If the sensor is operated at a velocity much smaller than the sound speed of the fluid, the attenuation factor is proportional to the root of the product of viscosity and density. For the sensor parameters used in this section, the attenuation is given by

\[ \alpha = 57 \sqrt{\rho_f \eta} \]  

(78)

In water, this yields a loss of 57 Neper/m, or 5 dB/cm. If the length of the propagation path amounts to 5 mm, the extra loss caused by viscous loading will
be 2.5 dB. The viscosity sensitivity of the attenuation is approximated by

\[ S_\alpha^\eta = \frac{1}{\alpha} \frac{\partial \alpha}{\partial \eta} = \frac{1}{2\eta} \]  

(79)

yielding 500 /Pa.s or 2.9×10^4 Neper/(Pa.s.m) or 2.5×10^3 dB/(Pa.s.cm) in water. For a 5 mm long propagation path this becomes 1240 dB/(Pa.s). A 10% increase in the viscosity of the water causes an extra propagation loss of 0.12 dB.

The approximated density sensitivity of the attenuation is

\[ S_\alpha^{\rho_f} = \frac{1}{\alpha} \frac{\partial \alpha}{\partial \rho_f} = \frac{1}{2\rho_f} \]  

(80)

In water, this yields 5×10^-4 (kg/m^3)^{-1} or 2.9×10^{-2} (Neper/m)/(kg/m^3). For the 5 mm long wave path the loss is 1.2×10^3 dB/(kg/m^3). The relative density sensitivity is equal to the viscosity sensitivity, a 10% density increase of the water results in an increase of the propagation loss of 0.12 dB. Note, that for many liquids the relative viscosity changes occurring can be much higher than the relative density changes.

**Discussion of sensitivity**

Designing a sensor for a specific application immediately implies making the sensor insensitive to the other parameters. This has to be investigated for each application. For example, the large sensitivity to the sound speed could easily be a disturbing factor when measuring density. The sensitivity to sound speed is suppressed by operating at \( c \ll c_f \), making \( c_f \) less dominant. Thinning the plate increases the sensitivity to mass adsorption and density, but decreases the sensitivity to the sound speed (as can be seen in Fig. 2.18). The viscosity sensitivity is very low for low-viscous liquids.

To show the influence of the absolute values of liquid parameters on the sensitivity, the different numerically obtained sensitivities for water, ethanol and a silicone oil (PS040) are given in Table 2.1. The silicone oil has a relatively high viscosity and a low sound speed, ethanol has a low density and sound speed compared to water (again the sensor is considered for which the total plate thickness \( d=7\, \mu m \), the plate mass \( m=2.08\times10^{-2}\, \text{kg/m}^2 \), and the wavelength \( \rho=80\, \mu m \).

The sensitivities obtained in ethanol are comparable with water, except for the sensitivity to the sound speed. The lower sound speed of ethanol (1170 m/s, for
water 1500 m/s) increases the dominance of $\delta \rho$. The sound speed of the silicone oil is even lower which results in the highest sensitivity to sound speed alterations. The viscosity sensitivity in silicone oil is about 20 times higher than in water or ethanol, but still small compared to the density sensitivity. The influence of viscosity can in most cases be neglected.

Table 2.1  Numerically calculated sensitivities for mass adsorption, density, viscosity and sound speed of a ZnO-Al-SiO$_2$-Si layered Lamb-wave sensor immersed in different liquids: water, ethanol, and silicone oil PS040 (20 °C).

<table>
<thead>
<tr>
<th></th>
<th>water</th>
<th>ethanol</th>
<th>si-oil (PS040)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho_f$ [kg/m$^3$]</td>
<td>1000</td>
<td>790</td>
<td>958</td>
</tr>
<tr>
<td>$\eta$ [Pa.s]</td>
<td>0.001</td>
<td>0.0012</td>
<td>0.048</td>
</tr>
<tr>
<td>$c_f$ [m/s]</td>
<td>1500</td>
<td>1170</td>
<td>1000</td>
</tr>
<tr>
<td>$c$ [m/s]</td>
<td>870</td>
<td>874</td>
<td>808</td>
</tr>
<tr>
<td>$S_{\omega m}$ [kg/m$^2$]^{-1}</td>
<td>-12.4</td>
<td>-11.0</td>
<td>-8.1</td>
</tr>
<tr>
<td>$R_{\omega \nu}$ [%/%)</td>
<td>-0.193</td>
<td>-0.166</td>
<td>-0.170</td>
</tr>
<tr>
<td>$R_{\omega \eta}$ [%/%)</td>
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<td>-4.55x10^{-4}</td>
<td>-2.42x10^{-3}</td>
</tr>
<tr>
<td>$R_{\omega \gamma}$ [%/%)</td>
<td>+0.096</td>
<td>+0.206</td>
<td>+0.314</td>
</tr>
</tbody>
</table>
Chapter 3  

Sensor system design

3.1 Introduction

In chapter 2 it was concluded that the phase velocity of an $A_0$ Lamb wave propagating in a plate is not only determined by the properties of the plate, but also by the fluid which contacts the plate. The influence of fluid loading and mass adsorption at the plate on both wave velocity and wave amplitude was described, and it was concluded that the acoustic losses are low if the phase velocity is lower than the compressional (longitudinal) velocity of sound in the surrounding fluid. The slow $A_0$ Lamb wave is, therefore, supremely suitable to use in sensors for the determination and monitoring of properties of liquids such as density, viscosity and sound velocity. Further, by covering the plate with a thin selective (bio)chemical layer, the adsorption of a (bio)chemical compound can be detected in both gaseous and liquid environment.

A sensor system is obtained when the sensing element (consisting of a Lamb-wave delay line) is used as the frequency-determining element in an oscillator. Small frequency changes caused by perturbations at the surface of the sensing element can be measured very accurately. By applying an (automatic) level control, the gain of the amplifier is a measure for the wave attenuation in the acoustic device. This feature allows the development of multi-purpose sensor systems. An example is the simultaneous determination of the density and viscosity of a liquid. The principle operation of the acoustic-wave based oscillator is described in section 3.2.
The design of the oscillator is discussed in section 3.3. The slow $A_0$ Lamb wave requires a low value of plate thickness-to-wavelength ratio ($h/p < 0.1$). The silicon micromachining technology makes the fabrication of very thin plates (a few microns thick) possible. The required wavelength and therewith the required physical dimensions of the acoustic device are small, and a silicon microsensor is obtained. The non-piezoelectric silicon forces the use of a piezoelectric overlay. The performance of the thin-plate Lamb-wave delay line depends strongly on the choice of the layered structure, the thickness of the layers and the design of the transducers and delay line.

In the design of the oscillator-electronics, the delay line is taken as a starting point. Both a transimpedance and a voltage amplifier are considered.

The ultimate design of the monolithic oscillator comprises slanted delay lines in a ZnO-Al-SiO$_2$-Si layered plate structure of $2.8\times8$ mm$^2$, and a voltage-to-voltage gain-controlled amplifier setup. The layout of the realized monolithic sensor system on a $10\times10$ mm$^2$ chip, consisting of two independent oscillators of $5\times10$ mm$^2$, is described in section 3.4.

### 3.2 Acoustic-wave based oscillator

#### 3.2.1 Oscillator principle

Acoustic sensors are based on the detection of changes in wave velocity and attenuation, caused by modulation at the surface of the material in which the wave propagates. If an acoustic-wave delay line is placed in an oscillator loop as the frequency-determining element, the velocity shift causes a shift of the oscillation frequency. If the attenuation alters, an adjustment of the amplification is required.

The basic scheme of a delay-line oscillator is given in Fig. 3.1. The amplifier output is fed back by a delay line [37]. $|A(\omega)|$ and $|B(\omega)|$ are the amplifier gain and the delay-line losses, respectively. The oscillation conditions are a loop gain of unity and a loop phase of $2\pi m_i$ ($m_i$ is an integer):

$$|A(\omega)| \cdot |B(\omega)| = 1$$  \hspace{1cm} (81)

$$\phi_{\text{loop}} = \phi_{\text{dl}} + \phi_{\text{amp}} + \phi_{\text{t1}} + \phi_{\text{t2}} = -2\pi m_i$$  \hspace{1cm} (82)
Fig. 3.1  Basic scheme of a delay-line oscillator.

where $\phi_{dl}$ is the phase shift of the delay line, $\phi_{amp}$ the phase shift of the amplifier and $\phi_{t1}$ and $\phi_{t2}$ are the phase shifts of each of the two interdigital transducers which generate and detect the acoustic waves. The loop phase is mainly determined by the (acoustic) contribution of the delay line: $\phi_{dl} = -\omega \tau$; where $\omega$ is the angular frequency and $\tau$ is the acoustic delay time. The delay time depends on the length of the propagation path (or wave path) and is defined as the center-to-center distance between the two transducers: $\tau = L_{path}/c$, where $c$ is the phase velocity of the wave. If $\phi_{dl} \gg \phi_{amp} + \phi_{t1} + \phi_{t2}$, the phase condition yields $\omega = 2\pi m/\tau$ and the frequency difference between two phase nulls (modes) in the oscillator is given by $1/\tau$.

The actual oscillation frequency mode depends on the oscillator type. If the level is not controlled, the oscillation condition is reached by saturation. Since the transfer characteristics of the acoustic delay line show several phase nulls which satisfy the oscillation conditions of equations (81) and (82) the use of a saturation amplifier would not guarantee a fixed oscillation frequency. The mode at which oscillation starts depends on the noise in the loop at the moment of switching on [38].

If a level control (gain control, GC) is applied, oscillation will always occur at the designed mode (the mode with the smallest amplitude loss). After switching-on, the amplitude of this mode will increase until the oscillation condition is reached. Therefore, the gain-controlled amplifier setup is preferred.
3.2.2 Instability

Short term
The short-term instability of the oscillator, defined as the instability in a time interval of one second or less, determines the sensor resolution and lowest detection limit.

A definition for the short-term instability of oscillators $s_{short}$ is given by:

$$ s_{short} = \frac{1}{\omega_0} \sqrt{\frac{\omega_0^2}{(\Delta\omega)^2}} \quad (83) $$

The frequency deviations, $\Delta\omega$, measured in a time interval are averaged. Noise is the main cause for the frequency deviations in this short interval and the short-term instability is directly related to the spectral band width $B$ of the oscillator [39]:

$$ s_{short} = \frac{1}{\omega_0} \sqrt{\frac{\pi B}{T_i}} \quad (84) $$

where $T_i$ represents the sampling time. If an automatic level control is applied which keeps the output at a constant (A.C.) level $u_0(t)$ the bandwidth is given as [40]:

$$ B = \frac{A_{amp}^2 S_n}{u_0^2(t) \frac{2\pi}{\omega_0^2} \tau^2} \quad (85) $$

where $S_n$ is the amplifier noise density spectrum, and $A_{amp}$ is the amplifier gain. The noise contribution of the acoustic device is neglected in this consideration.

Combination of equations (84) and (85) yields:

$$ s_{short} = \frac{A_{amp}}{\omega_0 \tau} \sqrt{\frac{S_n}{2 T_i u_0^2(t)}} \quad (86) $$

From equation (86) we find that the oscillator short-term instability is improved if the required amplifier gain is low (which can be realized by using low-loss acoustic delay lines), by decreasing the noise density spectrum (by using a low-
noise amplifier), by increasing the output voltage level of the amplifier and by increasing the delay time (by increasing the acoustic path length of the delay line or by decreasing the wave velocity). In practice, low-noise electronic circuitry can be realized for the frequency range considered in this work (up to 20 MHz).

Theoretical considerations of the noise contribution of layered acoustic-wave devices are not available. It has been shown, though, that wave reflections in the transducer and at the edges of the delay line, and (crystal) imperfections in the propagation path affect the noise behavior [41].

Medium term
The medium-term instability of an acoustic oscillator (instability over a time period of a few minutes to a few hours) is mainly dominated by drift caused by temperature variations. The temperature affects both amplifier and delay line.

The influence of phase fluctuations in the amplifier on the oscillation frequency is given by

$$\frac{df}{d\phi_{amp}} = \frac{1}{2\pi \tau}$$

(87)

According to equation (82), the loop phase is mainly determined by the delay line if a long path length is applied. The influence of amplifier phase fluctuations can be suppressed in this case.

Aging
Aging is measured over a time period of one month to several years. In layered acoustic devices in which prestress is present, aging might occur as a result of stress relaxation. This effect can be minimized by controlling the stress of the layers during fabrication. Also, the metals used in the device can diffuse into other layers. The elastic and electrical properties of the layers are altered slowly, which causes frequency shifts.

3.2.3 Signal transfer in the delay-line oscillator

In the oscillator setup the delay line is connected to the electronic amplifier. The input and output impedance of the amplifier influence the power transfer through the delay line.
The equivalent transmitting and receiving networks are shown in Fig. 3.2 [42]. The radiation conductance of the IDTs, $G_{a1}(\omega)$ and $G_{a2}(\omega)$ have a maximum at $\omega = \omega_0$, which are denoted as $\hat{G}_{a1}$ and $\hat{G}_{a2}$. $C_{t1}$ and $C_{t2}$ are the transmitting and receiving IDT capacitances, respectively. The transmitting IDT is connected to a voltage generator. At the receiver, the incoming acoustic power is represented as a dependent current source $I_R$, and the receiving IDT is loaded with the amplifier input $Y_L = G_L + j\omega C_L$. For the sake of simplicity, the electrode resistances and the parasitic capacitances and inductances are ignored.

![Equivalent circuits for both transmitting (IDT1) and receiving transducer (IDT2).](image)

**Fig. 3.2** Equivalent circuits for both transmitting (IDT1) and receiving transducer (IDT2).

Half of the acoustic power radiated from the conductance ($\hat{G}_{a1}$) is transmitted in the wrong direction, away from the receiving IDT, and is considered as loss. The power $P_T$ directed to the receiving IDT is

$$P_T = \frac{G_0^2 \hat{G}_{a1}}{2 \left(G_0 + \hat{G}_{a1} + j\omega_0 C_{t1}\right)^2} U_0^2 \quad (88)$$

where $U_0$ is the amplifier effective output voltage and $G_0$ the amplifier output conductance.

The effective current $I_R$ delivered by the receiver can be determined by assuming a matched load:

$$Y_L = \hat{G}_{a2} - j\omega_0 C_{t2}$$
$$G_L = \hat{G}_{a2} = G_{a2}(\omega_0) \quad (89)$$

The IDT capacitance $C_{t2}$ is thought to be compensated by an inductance in $Y_L$, the receiver circuit shown in Fig. 3.2 can now be simplified as shown in Fig. 3.3.

The maximum available average power $P_L$ dissipated in $G_L$ by the effective
current \( I_L = \frac{1}{2} I_R \) is given by

\[
P_L = \frac{I_R^2}{4G_L} = \frac{I_R^2}{4\hat{G}_{a2}}
\]

(90)

\( P_R \) is the received power. The maximum efficiency of the power conversion in the receiving IDT is 50\%, hence

\[
P_L = \frac{1}{2} P_R
\]

(91)

and

\[
I_R^2 = 2\hat{G}_{a2} P_R
\]

(92)

If propagation losses are neglected, \( P_R = P_T \), and combination of equations (88) and (92) gives

\[
I_R^2 = \frac{\hat{G}_{a2} G_0^2 \hat{G}_{ai}}{(G_0 + \hat{G}_{ai} + j\omega_0 C_{ll})^2} U_0^2
\]

(93)

Now that the current delivered by the current generator in the receiving IDT is known, the current through an arbitrary real part \( G_L \) of the input impedance of the amplifier load (Fig. 3.2) can be determined as
\[ I_{G_L} = \frac{G_L}{(G_L + \hat{G}_{a2} + j\omega_0 (C_{i2} + C_L))} \ I_R \]  

(94)

yielding

\[ \frac{I_{G_L}^2}{U_0^2} = \frac{G_L^2}{(G_L + \hat{G}_{a2} + j\omega_0 (C_{i2} + C_L))^2} \frac{\hat{G}_{a2} \hat{G}_0 \hat{G}_{a1}}{(G_0 + \hat{G}_{a1} + j\omega_0 C_{i1})^2} \]  

(95)

and because

\[ U_{G_L} = \frac{I_{G_L}}{G_L} \]  

(96)

the voltage transfer can be written as

\[ \frac{U_{G_L}^2}{U_0^2} = \frac{1}{(G_L + \hat{G}_{a2} + j\omega_0 (C_{i2} + C_L))^2} \frac{\hat{G}_{a2} \hat{G}_0 \hat{G}_{a1}}{(G_0 + \hat{G}_{a1} + j\omega_0 C_{i1})^2} \]  

(97)

If \( G_0 \gg |\hat{G}_{a1} + j\omega_0 C_{i1}| \) and \( C_{i2} \gg C_L \), then

\[ \frac{I_{G_L}^2}{U_0^2} = \frac{G_L^2 \hat{G}_{a1} \hat{G}_{a2}}{(G_L + \hat{G}_{a2} + j\omega_0 C_{i2})^2} \]  

(98)

and

\[ \frac{U_{G_L}^2}{U_0^2} = \frac{\hat{G}_{a1} \hat{G}_{a2}}{(G_L + \hat{G}_{a2} + j\omega_0 C_{i2})^2} \]  

(99)

If current sensing is applied (transimpedance concept), the amplifier input conductance should be high, \( G_L \gg |\hat{G}_{a2} + j\omega_0 C_{i2}| \) and the transfer is given by
\[ \frac{I_{G_1}^2}{U_0^2} = \hat{G}_{a1} \hat{G}_{a2} \]  

(100)

If voltage-sensing is applied then \( G_L \ll |\hat{G}_{a2} + j\omega_0 C_{in}| \) and the transfer is given by

\[ \frac{U_{G_1}^2}{U_0^2} = \frac{\hat{G}_{a1} \hat{G}_{a2}}{(\hat{G}_{a2} + j\omega_0 C_{in})^2} \]  

(101)

For both cases, only a small amount of electrical power is dissipated in the load. For the transimpedance configuration, this implies that all the power is transmitted, while for the voltage-to-voltage transfer, the ratio between reflected and transmitted waves is determined by the receiver [43]. For the monolithic Lamb-wave oscillator, a voltage-to-voltage amplifier is preferred, as will be discussed in section 3.3.3.

### 3.3 Design

#### 3.3.1 Silicon implementation

Slow Lamb-wave devices which are suitable for use in liquids are obtained if the plate thickness-to-wavelength ratio is small. In practice, a plate thickness of less than 10 \( \mu \)m is required, which is very difficult to realize in homogeneous piezoelectric substrates such as quartz or lithium niobate. In addition, the handling of such thin plates is extremely difficult. In silicon, however, micromachining techniques allow the fabrication of thin plates in the middle of a chip. The edges of the chip keep their original thickness and support the plate. Although several materials can be employed for the fabrication of the thin plate, e.g. aluminum or silicon nitride, silicon is the most attractive material when aiming at a monolithic integration of electronic circuitry and the acoustic device. If using aluminum or silicon nitride, extra process steps would be required during the IC fabrication processing which should not take place in order to preserve the quality of the IC processes.

By applying electrochemically controlled etching (ECE), the bulk silicon is etched from the back leaving the n-epitaxial layer (which is grown on the silicon
substrate) unharmed. As mentioned, the realization of acoustic devices in silicon requires a piezoelectric overlay for the generation and detection of acoustic waves. Piezoelectric zinc oxide is one of the attractive overlay materials because it has a high piezoelectric coupling factor and it can be fabricated relatively easily through sputtering, a technique compatible with IC-technology.

For acoustic devices consisting of a piezoelectric overlay on a non-piezoelectric substrate (in this case a SiO₂-Si layered structure), four layered transducer configurations are considered [44,45]. They are shown in Fig. 3.4.

![Diagram of transducer configurations](image)

Fig. 3.4 Four layered transducer configurations: A, B, C and D.

A configuration is chosen according to the demands made on the acoustic device. These demands include the capacitance, conductance and conversion losses of the transducer, the velocity, frequency, delay time and propagation losses of the waves, and the sensitivity to the measurand [46,47,48,49,50,51].

For the Lamb-wave sensor we used configuration D, as explained in the following section. A three-dimensional drawing of the resulting structure of the Lamb-wave device is shown in Fig. 2.3 in chapter 2. A two-dimensional cross-section is given in Fig. 3.5.

This structure can be realized by making use of integrated circuit (IC) compatible processing: deposition and patterning of zinc oxide and aluminum. The combination of the bipolar IC technology and the acoustic-wave device technology is discussed in chapter 4.
3.3.2 Lamb-wave delay-line design considerations

Transducer
The design of the acoustic delay line begins with the examination of the transducer properties. The conductance of a single-electrode transducer of equal electrode width and electrode distance is given by

$$\hat{G}_a = 8 \kappa^2 N_f f_0 C_t$$  \hspace{1cm} (102)

$$C_t = N_t W_t C_{\text{norm}}$$  \hspace{1cm} (103)

where $\kappa^2$ is the piezoelectric coupling coefficient, $N_f$ is the number of finger pairs of the transducer, $C_t$ is the transducer capacitance, $W_t$ is the transducer aperture and $C_{\text{norm}}$ the normalized capacitance of a finger pair in F/m.

It was shown in section 3.2.3 that for the transimpedance concept, a low-loss acoustic device is obtained if the transducer conductances are both high. For the voltage-to-voltage amplifier concept, a large conductance of the transmitting transducer combined with a small conductance of the receiver would give an optimal transfer. However, if the output impedance of the delay line becomes higher, the noise behavior of the oscillator will be affected. In addition, if the conductance of the transmitting transducer is increased by increasing the number of finger pairs $N_f$, the bandwidth decreases. If the bandwidth becomes smaller than the mode distance, the oscillator could oscillate at frequencies of which the insertion loss is more than 3 dB higher than the peak value, as discussed in the
following section. To obviate possible disturbances of this kind, the number of finger pairs of the transmitter and receiver was chosen to be equal. In order to optimize the conductance of the transducers the individual terms of equation (102) are now investigated.

The wave length is determined by the finger spacing (period $p$) in the transducer, the wave velocity mainly by the plate material(s) and the total layer thicknesses of the plate. A low velocity is required in order to suppress acoustic radiation into a loading liquid. Therefore, the frequency will be restricted to relatively low values.

![Graph](image)

**Fig. 3.6** Piezoelectric coupling coefficient $\kappa^2$ versus the normalized silicon layer thickness $h_{si}/p$ for the four layered configurations.

To obtain a high transducer conductance, a high piezoelectric coupling coefficient is also required. The piezoelectric coupling coefficient and the phase velocity of layered plate structures were calculated by using dedicated software [52]. The configuration of the layered structure and the layer thicknesses have a substantial influence on the piezoelectric coupling. Fig. 3.6 gives the coupling coefficient of the $A_0$ Lamb wave as a function of the normalized silicon layer thickness ($h_{si}/p$) for the four different layered structures and Fig. 3.7 as a function of the normalized zinc oxide layer thickness ($h_{zno}/p$). In Fig. 3.8 the phase velocity for these structures is shown (assuming a stress-free plate). It
Fig. 3.7 Piezoelectric coupling coefficient $\kappa^2$ versus the normalized zinc oxide layer thickness $h_{\text{zno}}/p$ for the four layered configurations.

must be noted that experimentally found coupling factors confirm the shape of the curves of Fig. 3.6 and Fig. 3.7, but show lower absolute values. The requirement of a low phase velocity (in practice <1500 m/s) combined with a high piezoelectric coupling coefficient makes configurations A and C useless since the piezoelectric coupling factor is very low at thin zinc oxide films. The coupling coefficients of configurations B and D show a peak for thin zinc oxide films. Because the fabrication of zinc oxide on a homogeneous surface shows a higher quality than zinc oxide grown on a non-homogeneous surface (e.g. silicon-dioxide on which the aluminum patterns are located), configuration D is preferred for the fabrication of the Lamb-wave device. Another advantage of this structure is the suppression of electromagnetic feedthrough (crosstalk) from the transmitting transducer to the receiving transducer. The semiconducting properties of silicon are a main cause of crosstalk in silicon-based delay lines [53,54]. By decoupling the aluminum plate which is located between the silicon and the zinc oxide, EM feedthrough is suppressed dramatically. The aluminum transducer electrodes are patterned on top of the zinc oxide.

The proper thicknesses of the silicon and zinc oxide layer are found from Fig. 3.6 and Fig. 3.7, respectively. The normalized silicon layer thickness should have a value of at least 3 % in order to avoid the area where the piezoelectric
coefficient lowers rapidly. At larger silicon thicknesses the coupling coefficient lowers slowly, at 8% the coupling factor is about 80% of its peak value. If we assume a silicon plate thickness between 4 and 7 μm, the optimum transducer period is 80 to 130 μm. For the zinc oxide layer, the optimum coupling factor is obtained at a value between 2% and 5%. The presence of mechanical compressive prestress in the zinc oxide can cause the performance of the device to deteriorate. Therefore, this layer should be kept as thin as possible, around 2%. The $A_0$ Lamb-wave phase velocity in this layered structure in air is in the range of 800 to 1400 m/s (if the plate is free of in-plane prestress).

**Number of finger pairs and path length**

Now that the transducer configuration, layer thicknesses and period are known, the last transducer design step concerns the metal electrodes (the number of finger pairs $N_f$ and the finger overlap (aperture) $W_f$). In order to minimize the conversion loss, the product $N_f^2W_f$ should be large, a low capacitance calls for a low product $N_fW_f$. A large number of finger pairs combined with a very small aperture satisfies both demands. However, if the number of finger pairs chosen is too large, the transducer bandwidth will become very small. Further, the space on the chip is restricted because of yield and costs. In our case, a practical maximum plate size is 3×8 mm².
Fig. 3.9  Phase and frequency transfer characteristics of a Lamb-wave delay line where the -3 dB pass-band equals the mode distance.

When using a gain-controlled amplifier, the oscillation condition of equation (82) implies that the phase null at which the oscillator operates is not necessarily at the peak of the delay-line transfer. Due to phase shifts in the IDTs and the amplifier, the phase null is shifted. The number of phase nulls (modes) in the pass-band should not be too small in order to prevent oscillation far from the Lamb-wave frequency with the lowest attenuation. In Fig. 3.9 it is shown that when the -3 dB pass-band $B$ is larger or equal to the mode distance $\Delta f_{\text{mode}}$, the maximum extra loss caused by a shift from the peak frequency amounts to 3 dB, which is an acceptable value. The mode distance is determined by the number of wavelengths $n$ in the wave path of the delay line $n = L_{\text{path}}/\lambda$: $\Delta f_{\text{mode}} = f_0/n$. The bandwidth is determined by the IDTs; $B = 0.88f_0/N_r$. The number of modes covered by the pass-band is $0.88n/N_r$. The requirement to have at least one mode in the pass-band yields $n \geq 1.14 N_r$.

If the two transducers lie close together ($L_{\text{path}}/\lambda < 4N_r$) the mode distance and bandwidth can deviate considerably from the calculated values. This is caused by the fact that the distance covered by the waves launched by the electrodes closest to the receiving IDT suffer less reflections within the IDT, and smaller
propagation losses. These waves are more dominant than the waves launched by
the electrodes at the outer side of the delay line, and therefore there is an
effective delay time that is shorter than the calculated delay time: \( \tau_{\text{eff}} < \tau \).
Similarly, the effective path length is shorter, thus providing a larger effective
mode distance. In addition, the bandwidth will increase because of this effect.
However, because both \( N_{\text{eff}} \) and \( n_{\text{eff}} \) become smaller, the number of modes within
the pass-band will not change significantly.
The typical dimensions of the Lamb-wave delay lines are collected in Table 3.1.

\textbf{Table 3.1  Typical dimensions of slow Lamb-wave sensors.}

<table>
<thead>
<tr>
<th>layer thicknesses [\text{\mu m}]</th>
<th>4.0-7.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>silicon</td>
<td>0.2-0.7</td>
</tr>
<tr>
<td>aluminum</td>
<td>0.1-0.2</td>
</tr>
<tr>
<td>oxide</td>
<td>0.7-2.0</td>
</tr>
<tr>
<td>zinc oxide</td>
<td>5.0-9.9</td>
</tr>
<tr>
<td>total</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>wave length [\text{\mu m}]</th>
<th>80-120</th>
</tr>
</thead>
<tbody>
<tr>
<td>( A_0 ) wave velocity in air [\text{m/s}]</td>
<td>700-1400</td>
</tr>
<tr>
<td>number of finger pairs ( N_f )</td>
<td>25-45</td>
</tr>
<tr>
<td>aperture ( W_t ) [\text{mm}]</td>
<td>2.0-2.8</td>
</tr>
<tr>
<td>path length (center-to-center)</td>
<td></td>
</tr>
<tr>
<td>in mm</td>
<td>2.4-5.0</td>
</tr>
<tr>
<td>in number of wavelengths</td>
<td>30-60</td>
</tr>
<tr>
<td>plate width [\text{mm}]</td>
<td>2.8</td>
</tr>
<tr>
<td>plate length [\text{mm}]</td>
<td>8</td>
</tr>
<tr>
<td>sensing area [\text{mm}^2]</td>
<td>8-20</td>
</tr>
</tbody>
</table>

\textbf{Suppression of reflections}
Distortions of the transfer characteristics occur because of the interference of
reflected waves with the main signal. Reflections are caused by mechanical or
electrical loading of the surface in the propagation path (transducer reflections,
edge reflections) and regeneration of waves by the electro-acoustic conversion
process in the transducers. Reflections at the IDT can be suppressed by applying a split-electrode structure [55].

The edge reflections can be severe and precautions have to be taken in order to eliminate them. In Rayleigh wave devices, acoustic absorbers have shown to be effective [56,57]. They consist of a soft material (e.g. polyimide) located on the surface at the edges of the device. For silicon-implemented Lamb-wave devices, the use of an acoustic absorber is not possible. First, the absorber must have an effective length equal to several wavelength in order to act efficiently, which is unattractive because of the extra space required. Second, such an acoustic absorber should have a thickness equal to or larger than the wavelength (80 μm) which is not compatible with standard IC processing. In addition, the thick layer would introduce stress in the plate thus affecting the performance.

The use of slanted edges, which are located at some distance from the transducers and reflect the waves totally away from the wave path, would also cost too much space, the plate area would have to be doubled in order to realize this.

A method to eliminate reflections which can be applied to Lamb-wave devices is the use of slightly slanted edges, close to the IDT, which satisfy

\[ \tan \alpha_r = i \frac{P}{2W_i} \quad (i \in \mathbb{N}, \quad i \geq 1) \quad (104) \]

where \( \alpha_r \) is the slanting angle. Assuming \( i=1 \), each reflection in the left half of the wave beam has a 180° phase difference with a reflection in the right half of the wave beam, so that they cancel each other out (Fig. 3.10). In practice, the cancellation will be somewhat more accurate at larger \( i \)'s. Only little extra space is taken by this method of slightly slanted edges.

**Optimization of the sensitivity**

The sensitivity of the Lamb-wave oscillator for the different fluid parameters was discussed in chapter 2. For density sensing, and for (bio)chemical applications, the sensitivity is increased by decreasing the plate mass, i.e. thinning the plate. The sensitivity for the sound speed of the liquid is decreased by decreasing plate thickness. The limiting factor in optimization of the sensitivity is the strength of the thin plate. Control of residual stress in the layers of the plate is crucial to ensure the robustness of the sensor system.
3.3.3 Amplifier design considerations

Starting conditions
The Lamb-wave oscillator consists of three main parts: the frequency-determining element (the acoustic delay line), the amplifier, and the amplitude-determining element (level control). The combination of acoustic device and electronic circuitry imposes special demands on each part. For the design of the two electronic parts, the physical and electrical properties of the delay line are taken as a starting point.

Not only the oscillation frequency, but also the amplitude attenuation in the propagation path is of interest, because it gives information about the properties of the loading fluid, as shown in chapter 2. By applying a level-controlled oscillator, the gain can be monitored. Another advantage of the level-controlled oscillator was discussed in section 3.2.1 and concerns the prevention of oscillation at different modes.

The acoustic losses in the delay line are typically 25 to 30 dB in air, 30 to 40 dB in a low-viscous liquid and 35 to 45 dB if loaded by a high-viscous
liquid. The gain range of the gain-controlled amplifier should cover at least 20 dB.

An amplifier bandwidth of 15 MHz is sufficient for the $A_0$ Lamb-wave oscillator. A larger bandwidth would increase the risk of oscillations at other peaks than the $A_0$ peak (for example the $S_0$ peak).

When coupling the delay line and the amplifier, the signal transfer is determined by the amplifier impedances and by the IDT impedances, as shown in section 3.2.2. Considering the ZnO-Al-SiO$_2$-Si layered structure, some statements can be made with respect to the electrical properties of the IDT that also affect the signal transfer.

D.C. biasing aspects
First, the electric voltage over the zinc oxide must not exceed the breakdown voltage of the zinc oxide used, which is about 4 V/μm (see chapter 4).

In the bipolar IC-process used (DIMES-01), the PNP transistors have low cut-off frequencies (20 MHz) and are not used in the oscillation loop. The use of only NPNs complicates the control over the absolute values of the D.C.-biasing voltages. This results in non-zero D.C.-bias levels at the input and output, and in a voltage difference between the in- and output of the amplifier.

In order to prevent electrical breakdown of the thin zinc oxide layer, the electrodes and the plate within an IDT should carry the same D.C. voltage. Since the two IDTs of the delay line will carry different D.C. voltages, the aluminum plate underneath the zinc oxide must be split into separate plates for each transducer. In order to suppress acoustic reflections that could occur at the edges of these plates in the propagation path, they are provided with a reflection-battlements structure (see Fig. 3.11). The length of the battlements corresponds to a quarter wavelength so that reflected waves are cancelled out. In practice, the amplifier input has a D.C.-voltage level of about 3 V, while the output is set to about 9 V.

Second, since zinc oxide is not a perfect isolator, a leakage resistance is found between the electrodes located at the top of the zinc oxide and also between each of the electrodes to the conductive plate underneath the zinc oxide. These last two leakage resistances can have different values due to local imperfections in the zinc oxide.

The elimination of the D.C. voltage between electrodes and plate prevents current leakage caused by the leakage resistance of the zinc oxide. The D.C.
behavior of the zinc oxide thus becomes irrelevant. Note, that for the A.C. behavior the leakage resistance is still of influence.

Third, there is a leakage resistance between the two transducers. In the loop, not only A.C. signals are amplified but also D.C. signals. The D.C.-voltage difference between the two transducers amounts to about 6 V. Even at a high leakage resistance of 1 MΩ, the leakage current amounts to 6 µA. If the leakage resistances of the two electrodes of the receiver are not exactly equal, the leakage current generates a small voltage difference between the two electrodes of the receiver, which will be amplified. This might result in clipping of the amplifier (if the difference amounts to 10 kΩ (only 1 %), the voltage difference due to 6 µA leakage current is 6 mV. A 50 dB amplification yields a D.C. difference voltage of almost 2 V which exceeds the dynamic range (± 1 V). The amplifier would clip under this condition).

In order to prevent this, the zinc oxide layer can also be split into two separate layers by etching it from the propagation path. Again, battlement edges are used to suppress reflections.

A.C. aspects

A large A.C. signal delivered by the amplifier to the transmitting IDT improves the signal-to-noise ratio and increases the oscillator stability as discussed in section 3.2.2. The signal is limited by the breakdown voltage of the zinc oxide, and the maximum current that can be delivered by the amplifier. An output signal of 400 mV_{pp} is low enough to prevent breakdown, and the required
current of 2.5 mA_peak at 15 MHz can be delivered by the amplifier (the bias current in the output stage is about 4 mA per side).
The output stage behaves as a symmetrical voltage source. The low output impedance has the advantage that reflected waves that reach the transmitting transducer cannot generate voltages over the transducer which prevents regeneration of waves. Triple transit reflections are suppressed [43]. Both amplifier input and output are symmetrical which suppresses the influence of common-mode signals such as capacitive and resistive crosstalk in the device. A differential common-emitter (CE) amplifier can be used as a symmetrical voltage-sensing input with a high common-mode rejection (CMR).
A high input impedance is easily realized for a CE input stage, which is an advantage because small (integrated) decoupling capacitors (10 pF) can now be used between amplifier and IDT. The decoupling causes the amplifier to be insensitive to large differences in D.C. impedances in the IDT.
Current sensing is also possible in the acoustic oscillator [58] and can be realized by using a common-base (CB) or a shunt-feedback input stage. It has the advantage that also at the receiving transducer, regeneration of waves is suppressed because of the low input impedance. However, it must be noted that for both configurations, it is much more complex to realize a high CMR. The CE input stage is preferred and, therefore, the final device is a voltage-to-voltage amplifier.

**Amplifier setup**
The amplifier is built up of four blocks, an input stage (voltage-to-current convertor), a current amplifier, a variable current attenuator, and an output stage consisting of a current-to-voltage convertor (Fig. 3.12).

![Amplifier diagram](image)

*Fig. 3.12 The amplifier consisting of four stages.*

The first block is the differential CE stage discussed in the previous section, the gain amounts to 10 mA/V. The input impedance of the differential amplifier is formed by 7 kΩ resistance parallel to a capacitance of 0.5 pF. The second stage is a current amplifier (30x) designed such that its noise contribution is negligible. The third block is an amplifier with a variable current gain of the
Gilbert type [59] (gain variable between 0.02$x$ and 0.9$x$). This amplifier block shows poor noise behavior, but because amplification has taken place in the two preceding blocks, the contribution to the total noise is also negligible. The output block is based on a symmetrical common-collector (CC) stage which provides the current and voltage required ($\pm$ 2.5 mA$\text{p}$ at $\pm$ 200 mV$\text{p}$). The output impedance is low (about 30 $\Omega$) and the gain is 2000 V/A. The four stages together yield a dynamic range of more than 32 dB, a maximum gain of 54 dB and a bandwidth of about 15 MHz.

![Diagram](image)

*Fig. 3.13* Current-gain-controlled stage consisting of a voltage-controlled Gilbert pair.

The amplifier gain-control takes place in the third stage. The current delivered by the Gilbert pair is determined by a control voltage $U_{GC}$ (Fig. 3.13). In order to control the oscillator amplitude, the amplitude of the output signal is converted into a D.C. signal which is compared to a reference voltage. The voltage difference is, after amplification, used to adjust the amplification of the third stage. When the attenuation increases, for example when there is liquid loading, the gain is increased so that the output is sustained at the same level. The steering voltage of the gain-controlled amplifier is also an output of the sensor system, which makes it possible to monitor the losses occurring in the acoustic device.

Acoustic delay lines with different acoustic losses and different operating frequencies can all be operated with the same amplifier because of the wide control range. Through buffers, the frequency and gain monitor are obtained. The oscillator is shown schematically in Fig. 3.14.
3.4 The monolithic oscillator

Before the electronics and the acoustic device were integrated on one single chip, they were fabricated in separate chips and tested in a hybrid setup. In the separate designs, the future monolithic integration was taken into consideration. In the acoustic device, the aluminum and zinc oxide layers were removed from the rim area in which the electronics were projected, and the electronic circuitry was designed to be long and narrow, so that it would fit into the rim. If thus designed, the adding of the electronics to the acoustic device does not require any extra space. By using the side-rims, parallel to the wave path, the acoustic waves do not touch the electronic circuitry, obviating any possible acoustic interference.

The realized monolithic sensor system contains two complete oscillators and the chip measures 10×10 mm². The dimensions of a single oscillator are 5×10 mm².
The realized monolithic oscillators were designed from the design specifications given in section 3.3. The electronic circuitry takes up an area of 1.2×4.2 mm² per oscillator, the ZnO-Al-SiO₂-Si layered plate area covers 2.8×8 mm² in which a slanted delay line is placed (angle with the rim of 80/2200). The period (wavelength) is 80 μm, the aperture is 2.2 mm and the IDTs have 38 or 42 finger pairs. Both uniform and split-electrode transducers were fabricated. The typical design thicknesses are 1.3 to 1.7 μm zinc oxide, 0.65 μm aluminum, 0.1 μm oxide and 7.5 μm silicon (optionally, the silicon layer was 'thinned' by plasma etching the back down to 4 μm). The aluminum (and optionally also the zinc oxide) was etched from the propagation path. The aluminum plate of each IDT was directly connected to the electronic circuitry. In order to be able to test the electronic circuitry and the acoustic device independently, the IDT electrodes were not connected to the electronic circuitry on-chip. Connections were realized by wire bonding to the housing. Coupling of the two independent oscillators on the chip was prevented by keeping the electronic circuits of each oscillator apart, and by placing them at opposite edges. The temperature of the chip can be elevated by using an aluminum resistive heater located at the middle rim of the chip.

A photograph of the realized dual oscillator is shown in Fig. 3.15.

The sensor system requires a D.C. power supply of 12 V (40 mA). The dual oscillator system has five outputs: the two single frequencies (up to 20 MHz, 100 mV pp, the difference frequency (up to 1 MHz, 100 mV pp) and the two gain level outputs (-1 V to +1 V). A frequency mixer is integrated on the chip. One oscillator is directly coupled to the mixer, the connection of the second oscillator to the mixer is realized through bonding wires to the package.

The fabrication of the monolithic oscillator system is more complicated than the fabrication of the hybrid oscillator. The processes for the fabrication of the acoustic part must be made compatible to the bipolar IC-process for the fabrication of the electronics. The technology necessary for the realization of the monolithic oscillator system is addressed in chapter 4.

The performance of these oscillators with respect to operating frequency, delay-line losses, reflection suppression, short-term instability, temperature behavior, and fluid loading is described in chapter 5.
Fig. 3.15  Photograph of the 10×10 mm² monolithic dual oscillator system.
Chapter 4  

Device fabrication

4.1 Introduction

For the fabrication of silicon sensors, standard IC-processes can be used. Usually, some extra process steps are required to attain the sensor-function, or to enhance the sensitivity and the stability. However, the integration of these steps in an IC fabrication process is hampered by the restrictions imposed by the process. The process steps and material properties are subjected to tight control in order to maintain the standards necessary to produce functioning electronic devices. Therefore, a general fabrication strategy was developed in which the sensor process steps are placed in "process modules" [60]. These modules are added to the well-established 2 μm bipolar IC-process of the Delft University of Technology, denoted as the DIMES-01 process.

The combination of Lamb-wave devices and electronic circuitry requires the bulk micromachining of silicon for the fabrication of the thin plates (2-8 μm), and deposition of piezoelectric zinc oxide layers for the generation and detection of acoustic waves. These processes are placed in separate modules: the bulk micromachining module (BMM1-module) and the zinc-oxide module (ZNO-module).

The (monolithic) Lamb-wave oscillator fabrication sequence is described in the following section. The relevant parameters of the DIMES-01 bipolar IC-process are given in section 4.3. The process steps of the silicon micromachining module are reviewed in section 4.4. In the last section, the fabrication and further
processing of piezoelectric zinc oxide are discussed. Extra attention was given to the determination and prevention of the occurrence of stress in zinc oxide during the growth process.

4.2 Lamb-wave sensor fabrication sequence

The complete fabrication sequence of the Lamb-wave sensor is described with the aid of Fig. 4.2. Details of the technology are given in sections 4.3, 4.4 and 4.5.

The starting material are double-side polished p-type \textless 100\textgreater -cut silicon wafers, diameter 100 mm and thickness 525 \textmu m. A top-view photograph of the dual Lamb-wave sensor system is shown in Fig. 3.15. The electronics are located at the side of the acoustic delay line. A photograph of a 100 mm diameter silicon wafer containing 36 dual-sensor systems is shown in Fig. 4.1.

![Fig. 4.1 Photograph of a 100 mm diameter wafer containing 36 dual sensor systems.](image-url)
The wafers undergo the standard IC-process, including the interconnect metallization (0.6 μm) and a PECVD oxide passivation (0.8 μm). The oxide is removed from the propagation path.

Back deposition and patterning (plasma etched) of PECVD nitride (360 nm), used as masking layer during the bulk-etch process.

A piezoelectric zinc oxide layer is deposited on the front of the wafer.

Deposition and patterning (wet-etch) of 250 nm aluminum (100% Al, vacuum evaporation).

The zinc oxide above the electronic circuitry is removed (wet-etch).

KOH bulk etching of silicon (plate fabrication). The standard silicon n-epilayer thickness of the DIMES-01 process is 4 μm. To increase the plate thickness, a BN implantation is used. The silicon is etched using an electrochemical etch-stop at the BN-substrate junction, leaving a 7.5 μm thick plate.

If required, the plate can be thinned by plasma etching of the back.
4.3 Bipolar IC-process

The electronic circuitry was fabricated in the DIMES-01 process. A detailed description of this process which was especially developed for smart sensor research can be found in [61].

A short description of the process used for the fabrication of the electronic functions in the monolithic Lamb-wave device is given in Table 4.1. The device parameters of the NPN and PNP transistors used are given in Table 4.2.

Table 4.1  DIMES-01 process flows for transistors.

<table>
<thead>
<tr>
<th>mask name</th>
<th>process step</th>
<th>WP-PNP transistor</th>
<th>BW-NPN transistor</th>
</tr>
</thead>
<tbody>
<tr>
<td>BN</td>
<td>buried n-layer</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>--</td>
<td>n-epitaxy</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>DP</td>
<td>p⁺ diffusion</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>DN</td>
<td>n⁺ diffusion</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>--</td>
<td>oxide stripping</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>--</td>
<td>oxide growth 330 nm</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>WP</td>
<td>B⁺ implantation</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>BW</td>
<td>B⁺ implantation</td>
<td>-</td>
<td>•</td>
</tr>
<tr>
<td>WN</td>
<td>As⁺ implantation</td>
<td>•</td>
<td>•</td>
</tr>
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<td>--</td>
<td>anneal 1000 °C</td>
<td>•</td>
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<td>CO</td>
<td>contact window</td>
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<td>•</td>
</tr>
<tr>
<td>IC</td>
<td>metallization</td>
<td>•</td>
<td>•</td>
</tr>
<tr>
<td>--</td>
<td>PECVD oxide 0.8 μm</td>
<td>o</td>
<td>o</td>
</tr>
<tr>
<td>CT</td>
<td>local PECVD oxide strip</td>
<td>o</td>
<td>o</td>
</tr>
<tr>
<td>--</td>
<td>alloying</td>
<td>•</td>
<td>•</td>
</tr>
</tbody>
</table>

* = step used for the fabrication of the transistor
○ = optional
- = step not used for the transistor
Table 4.2  Device parameters of the NPN and PNP transistors.

<table>
<thead>
<tr>
<th>parameter</th>
<th>WP-PNP</th>
<th>BW-NPN</th>
</tr>
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<tbody>
<tr>
<td>on-mask emitter area [μm²]</td>
<td>6×6</td>
<td>2×4</td>
</tr>
<tr>
<td>on-mask base length [μm]</td>
<td>3</td>
<td>-</td>
</tr>
<tr>
<td>current gain β</td>
<td>60</td>
<td>100</td>
</tr>
<tr>
<td>forward Early voltage [V]</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>base-emitter breakdown voltage [V]</td>
<td>32</td>
<td>5.9</td>
</tr>
<tr>
<td>base-collector breakdown voltage [V]</td>
<td>29</td>
<td>28</td>
</tr>
<tr>
<td>emitter-collector breakdown voltage [V]</td>
<td>15</td>
<td>12</td>
</tr>
<tr>
<td>maximum collector current [μA]</td>
<td>180</td>
<td>3000</td>
</tr>
<tr>
<td>minimum $V_{be}$ with $β=100$ [V]</td>
<td>-</td>
<td>0.3</td>
</tr>
<tr>
<td>minimum $V_{be}$ with $β=60$ [V]</td>
<td>0.3</td>
<td>-</td>
</tr>
<tr>
<td>cut-off frequency ($V_c=3$ V) [GHz]</td>
<td>0.02</td>
<td>5</td>
</tr>
</tbody>
</table>

4.4 Silicon bulk micromachining module

4.4.1 Fabrication of thin plates in silicon

In chapter 2 we discussed that the zero-order asymmetric ($A_0$) Lamb wave shows low acoustic losses when loaded by a liquid, if the phase velocity is lower than the compressional sound velocity of the liquid. The phase velocity of the slow Lamb wave depends on the thickness of the plate in which it propagates: the velocity decreases when the plate is thinned. Water has a sound speed of about 1500 m/s at room temperature. In general, the sound speed of liquids is between 1000 and 1600 m/s. In chapter 3 it was shown that for a ZnO-Al-SiO₂-Si layered plate, Lamb waves with phase velocities lower than 1500 m/s are feasible if the total plate thickness is between 5 and 10 μm. The required silicon layer thickness is then between 2 and 8 μm.

Micromachining technology is used for the fabrication of thin plates in silicon. This technology has been highly developed over the last decades for the batch fabrication of silicon sensors containing microstructures such as membranes, cantilever beams, and bridges [62,63,64,65,66].
A silicon bulk micromachining module has been developed which is fully IC-process compatible. There are no high-temperature steps which could affect the performance of the integrated electronics, the highest process temperature applied is 300 °C.

The module contains the following process steps.

After the last IC-process step (the etching of the aluminum leads) a silicon nitride masking layer is deposited at the back of the wafer using plasma-enhanced chemical vapor deposition (PECVD). The nitride is patterned by dry-etching. The next step consists of the actual etching of the silicon. In the electrochemically-controlled etching (ECE) technique the n-type silicon epitaxial layer (n-epi) is supplied with a bias voltage to prevent it from being etched. The etching, which is started from the back of the silicon wafer, stops automatically at the reverse-biased pn-junction and the remaining plate shows a high uniformity in thickness.

The thickness of the epilayer is determined by the IC-process, each process has its fixed epilayer thickness. Changing the epilayer thickness is not possible without affecting the transistor performance. In the DlMES-01 process that is used for the fabrication of the sensor, the epilayer thickness is 4 μm. In order to be able to fabricate thicker plates, a buried n-type (BN) diffusion was used. The BN penetrates approximately 3.5 μm into the silicon substrate, thereby shifting the pn-junction at which the electrochemical etching stops, leaving a plate of 7.5 μm thick. In Fig. 4.3, a SEM photograph of a silicon plate fabricated using ECE is shown. The epilayer was partly provided with a BN implantation, thus obtaining a thicker remaining plate. The thin part of the plate (left) consists of the 4 μm thick silicon n-epi, the thicker part (right) measures about 7.5 μm.

The processes are described in the following sections.

### 4.4.2 Electrochemically-controlled etching of silicon

**Etchant**

The etchant used for the bulk etching of silicon must fulfill requirements concerning the dependence of the etch rate on the crystal orientation and on an externally applied electric potential. In addition, the silicon etch rate should be high enough to etch the silicon wafer within a reasonable amount of time, while the etch rate of the masking material should be low.

KOH meets all the mentioned requirements. It selectively etches the [110] and the [100] orientation while the [111] orientation is etched much more slowly. In <100>-cut silicon wafers KOH etching produces side-walls set at an angle of
SEM of a silicon plate (cross-view) fabricated by ECE. The left part consist of only the epilayer (4 μm), the right side is provided with a BN implantation, leaving a thicker plate (7.5 μm).

54.7° with respect to the surface.
KOH shows a strong drop in the etch rate at a passivation potential of larger than about 0.6 V, a property which is used for introducing an automatic etch-stop at a pn-junction. The silicon wafers have a thickness of around 525 μm. A practical maximum for the etch period is about 6 hours, yielding a required etch rate of 85 μm/hr. This etch rate is obtained by applying an etch solution of 33 weight% KOH in water at 85 °C (Fig. 4.4) [66].

The wafer is clamped in a specially designed etch holder which is provided with an anode contact to the epilayer of the wafer. A platinum cathode is located in the solution. Only the back of the wafer contacts the etch solution and the unmasked areas are etched. The etching stops at the junction if a positive voltage larger than +0.6 V is applied. The etched wafer is removed from the holder and, optionally, the silicon plate is further thinned by using a plasma etch.

Plate uniformity and surface roughness
The thickness uniformity and surface roughness of the remaining plates are crucial for the performance of the slow Lamb-wave device. A non-uniform thickness causes phase velocity dispersion while surface roughness increases the acoustic losses due to acoustic reflections.
The uniformity in the plate and the surface roughness were determined by using
an alpha step 200 (Tencor Instruments). Typical values are ± 0.1% uniformity in a 7.5 μm silicon plate of 2.8×8 mm². The roughness was less than 1 nm.

4.4.3 PECVD silicon nitride films

Masking layer
At low etching temperatures, silicon dioxide can be used as a masking layer for the bulk etching of silicon in KOH. However, the etch rate of SiO₂ in KOH increases fast when the temperature increases. The etch rate required in order to etch the complete thickness of the silicon wafer (525 μm) forces the use of a temperature of 85 °C during etching. At this temperature, the etch rate of standard thermally grown SiO₂ layer is higher than 1 μm/hr which makes it unsuitable for masking purposes.

Silicon nitride displays a much lower etch rate in high-temperature KOH. Low-pressure chemical-vapor-deposited (LPCVD) silicon nitride layers show a
stoichiometry close to Si$_3$N$_4$ and, in general, a high density and a low degree of impurities. The etch rate of LPCVD nitride in KOH is very low, the selectivity towards silicon is better than 5000:1. However, LPCVD silicon nitride has the disadvantage that it must be deposited before the deposition of the aluminum, because of its process temperature of about 850 °C. Also, both back and front of the wafer are covered by nitride, so that it has to be removed again from the front.

Therefore, the production of high-quality plasma-enhanced chemical-vapor-deposited (PECVD) nitride has gained interest. The deposition temperature is low (typically 300 °C), so that deposition can take place after completion of the IC processing, including metallization. In addition, during the PECVD process only one side of the wafer is covered by nitride, the electronic circuitry located on the front is not affected by the PECVD process.

The PECVD-produced silicon nitride layers have a lower density and more impurities than LPCVD-produced nitride films and, in the past, the quality of PECVD nitride produced in R&D deposition systems was too low to use as reliable masking during high-temperature KOH etching. However, the quality and reproducibility of modern deposition systems has been greatly improved, not in the least because of the possibility of fully computer-controlled processing.

**Deposition**

The silicon nitride films are grown in a fully automated PECVD reactor chamber (STS 310 deposition system).

Amorphous PECVD silicon nitride films can be fabricated by a silane-nitrogen and/or silane-ammonia reaction. In plasma deposition chambers, ammonia is preferred to nitrogen because of its lower ionization energy. The dissociation of ammonia produces NH$_2$ and NH, the dissociation of N$_2$ gives N, and the dissociation of silane produces SiH$_3$, SiH$_2$, and SiH. The resulting silicon nitride can best be given by Si$_x$N$_y$, where x and y can deviate from the stoichiometric values. The deposition parameters such as power level, pressure, temperature, gas composition, and flow rate all have influence on the stoichiometry of the silicon nitride film. [67,68].

The influence of the gas composition during nitride growth on the quality was investigated in terms of etch rate in KOH and pin-hole density. The amount of pin-holes after KOH etching is an important indicator of the quality of the layer. A few pin-holes do not affect the etch process because of the direction-dependent etch rate. Every hole results in a pyramid-shaped pit, its depth depending on the width of the pin-hole. However, if there are too many pin-
holes the rims of the chip can be etched, which lowers the chip strength. The clustering of silicon during growth of the silicon nitride layer can also cause holes during KOH etching, because the silicon clusters are attacked by KOH. Silicon clustering can occur when the SiH$_4$/NH$_3$ ratio becomes so high that a part of the free silicon is not bonded with nitrogen but condensates at the substrate surface, possibly bonded with hydrogen [67].

The optimum deposition conditions found are presented in Table 4.3. Layers fabricated using this set of conditions showed a low etch rate (40 nm/hr) and low pin-hole density (0-10 on a 4-inch wafer) after etching in 33 % KOH at 85 °C. The uniformity of the layers is ± 5 % over a 100 mm wafer.

The nitride should withstand at least 6 hours of immersion, yielding a required thickness of 240 nm. In practice, layers of 360 nm were used.

<table>
<thead>
<tr>
<th>Table 4.3 Typical deposition conditions for PECVD silicon nitride.</th>
</tr>
</thead>
<tbody>
<tr>
<td>gas flows</td>
</tr>
<tr>
<td>pressure</td>
</tr>
<tr>
<td>power</td>
</tr>
<tr>
<td>temperature</td>
</tr>
<tr>
<td>deposition rate</td>
</tr>
</tbody>
</table>

4.4.4 Plasma etching of silicon

A plasma etch (SF$_6$/CHF$_3$ mixture) was used for further thinning of the silicon plates after KOH etching. The etch rate amounts to 0.25 μm/min for flat silicon wafers. The rate for thinning the plates appeared lower, about 0.12 μm/min. This is probably because the plates are located in a 0.5 mm deep pit. The etch conditions are given in Table 4.4.
Table 4.4  Process conditions for plasma etching of silicon.

| gas flows      | SF₆, 20 SCCM  
|               | CHF₃, 7.5 SCCM |
| pressure       | 40 mTorr      |
| power          | 100 W         |
| temperature    | 40 °C         |
| etch rate      | 250 nm/min (flat silicon wafer)  
|                | 120 nm/min (silicon in a 0.5 mm deep pit) |
| selectivity towards resist | 1:7 |

4.5 Zinc oxide module

Piezoelectric zinc oxide thin-films are widely used in transducers. Applications can be found in mechanical sensors, bulk acoustic-wave (BAW) devices, surface acoustic-wave (SAW) devices, Lamb-wave devices, acoustic microscopy and acousto-optic devices. Advantages are the relatively low deposition temperature, the high piezoelectric coupling coefficient and the excellent bonding on various substrate materials, in particular silicon, silicon dioxide, silicon nitride, and metals such as gold and aluminum. The combination of the silicon substrate and zinc oxide provides the possibility of monolithic integration of acoustic elements with electronic circuitry. Examples of monolithic acousto-electronic devices are SAW convolvers [69,70], SAW (programmable) filters [71,72,73, 74], barrier-modulated tap (BMT) SAW detectors [75], integrated resonant sensors [76] and acoustic-wave based sensors [77,78].

Zinc oxide also has some disadvantages. It is amphoteric and chemically very reactive. When used in devices, it has to be protected against the environment by gas-tight packaging (housing filled with an inert gas). Furthermore, the fabrication of reproducible high-quality zinc oxide requires an extremely well-controlled deposition process. Also, zinc oxide films can show high stress due to the growth process. Knowledge of the influence of the deposition parameters and crystal orientation of the surface of the substrate material on the residual stress is of crucial importance. Finally, zinc can affect the performance of the
bipolar electronic circuitry and it has to be handled with care in an IC-facility. For these reasons, the zinc oxide processes are placed in a separate IC-compatible process module: the zinc oxide module. This module starts after the IC fabrication process has been completed.

The zinc oxide module not only incorporates the deposition and patterning of piezoelectric zinc oxide films, but also the deposition and patterning of aluminum on top of zinc oxide. Some special cleaning fluids and wet etchants which do not attack zinc oxide are used.

The first step of the module consists of the deposition of the zinc oxide on the fully processed silicon wafer. The typical zinc oxide layer thickness for Lamb-wave applications is between 0.7 and 2 μm. On top of the zinc oxide, an aluminum film is deposited (250 nm thick) in which the interdigital patterns are etched. The last step consists of the removal of the zinc oxide from the electronic circuitry.

The etching of windows in the zinc oxide for the realization of electrical feedthroughs from the IDTs (on top of the zinc oxide) to the electronic circuitry is optional. The process steps required for the feedthroughs can be omitted by realizing these few contacts by wire bonding to the housing.

In the following sections the process steps are described.

### 4.5.1 Deposition of ZnO

High-quality piezoelectric zinc oxide is deposited mainly by sputtering techniques, e.g. triode sputtering, D.C. and R.F. magnetron sputtering. The process temperature is between 150 and 450 °C and sputter rates of up to 20 μm/hr are feasible. Sputtered zinc oxide layers are dense, highly oriented, and show a high degree of surface flatness. Zinc oxide has a hexagonal structure, class 6-mm (Fig. 4.5). The crystals of the poly-crystalline layer show a preferential growth of their c-axis perpendicular to the substrate surface. Highly oriented films show a high piezoelectric coupling factor. High-quality zinc oxide layers of thicknesses between 0.1 and 48 μm have been reported [79,80, 81,82,83,84,85,86,87,88,89,90,91].

The zinc oxide layers reported in this work were fabricated in a Varian 3119 R&D D.C. sputter-coater. Several tests were performed on zinc oxide layers made under different conditions and on different substrates. A set of typical sputtering conditions is given in Table 4.5. Unless mentioned otherwise, all layers reported here were fabricated under these conditions.
Fig. 4.5  The structure of ZnO.

Table 4.5  Typical ZnO deposition conditions.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>target material</td>
<td>Zn (99.999 %)</td>
</tr>
<tr>
<td>sputter gas</td>
<td>O₂</td>
</tr>
<tr>
<td>substrate temperature</td>
<td>375 °C</td>
</tr>
<tr>
<td>target-substrate spacing</td>
<td>11 cm</td>
</tr>
<tr>
<td>oxygen pressure</td>
<td>0.9 kPa</td>
</tr>
<tr>
<td>D.C. sputter power</td>
<td>1.5 kW (500 V, 3 A)</td>
</tr>
<tr>
<td>sputter rate</td>
<td>100 nm/min</td>
</tr>
</tbody>
</table>
4.5.2 Film characterization

The quality of piezoelectric zinc oxide films has been a subject in literature for over 25 years. Sputter deposition of zinc oxide shows superior properties over other deposition methods, but the growth and quality remains a subject of concern, as recent publications bear witness [92,93].

In chapter 3, the preferred layered structure for silicon-integrated slow Lamb-wave devices was described: ZnO-Al-SiO₂-Si. The growth processes optimized for the fabrication of thin high-quality zinc oxide layers on aluminum were discussed. For the characterization of the piezoelectric zinc oxide films, the following methods were used: scanning electron microscopy (SEM), X-ray diffraction (2θ-scan, rocking curve, pole figure), and surface profiling (uniformity and surface roughness). In addition, the breakdown voltage of the layers was determined.

Scanning electron microscope (SEM)

The morphology of the zinc oxide films is visualized by using scanning electron microscopy (SEM). SEM gives information about crystal orientation, grain size, and porosity.

![Cross-view SEM picture of a zinc oxide layer fabricated by using the standard growth conditions.](image)

The SEM-picture in Fig. 4.6 shows a layer fabricated by using the typical growth conditions. The zinc oxide layer is poly-crystalline with the c-axis
oriented perpendicular to the substrate surface. The thickness of the layer shown
is about 10 μm, a clear difference between the morphology of the first microns
of zinc oxide and the zinc oxide above the first microns can be seen. In the first
microns, the zinc oxide is denser and the poly-crystalline structure cannot be
seen clearly. The difference in structure affects the etch rate of the zinc oxide,
as discussed below.

**X-ray diffraction**

Zinc oxide films grown on Al-SiO₂-Si and Al-Si layered structures were
examined by X-ray diffraction using a Siemens D500 diffractometer with
diffracted-beam graphite monochromator (Cu-Kα radiation). Highly oriented zinc
oxide layers show a strong reflection intensity from the (002) planes (and,
therefore, also strong (004) and (006) peak intensities), and low intensities from
other zinc oxide planes (such as (102), (103), (104), (105)). Fig. 4.7 shows a
typical 2θ,scan of a 1.2 μm thick zinc oxide layer grown using the typical
growth parameters, on an Al-SiO₂-Si layered substrate. The peak intensity is
given in counts per second (cps). The (002), (004), and (006) peaks are clearly
present, and of the other possible peaks only the (105) shows some intensity.
The aluminum shows a strong (111) orientation, no other than the (111) and
(222) peak were found.

In Fig. 4.8 a similar scan is obtained for a ZnO-Al-SiO₂ layered structure. The
aluminum was grown directly on the silicon substrate, and shows some intensity
for the Al(200) peak. Comparison of this figure with Fig. 4.9 shows the
influence of the orientation of the aluminum layer on the orientation of the
subsequently grown zinc oxide layer. The aluminum in Fig. 4.9 presents not
only the (111) and (200) peaks, but also ((311), (331), (420), and (422). The
zinc oxide shows in addition to the (002), (004), and the (006) peaks, intensities
for the (102), (103), (104), and (105) peaks. The (piezoelectric) quality of this
zinc oxide layer is low.

It is of crucial importance, therefore, that highly (111) textured aluminum is
used. A precise description of the growth mechanisms is not available, but it is
assumed that growing zinc oxide on substrates of (closely) matching crystal
symmetry improves its crystallinity [87,88,90,91].
Fig. 4.7 2θi-scan of a 1.2 μm thick ZnO layer on an Al-SiO2-Si layered structure (the aluminum has a strong (111) orientation).

Fig. 4.8 2θi-scan of a 1.2 μm thick ZnO layer on an Al-Si layered structure (the aluminum has a (111) orientation, but the (200) peak is also present).
Fig. 4.9 2θ₁-scan of a 1.2 μm thick ZnO layer on an Al-Si layered structure (both the aluminum and the zinc oxide show several peaks).

By measuring X-ray rocking curves, the distribution of the c-axis orientation is determined [94]. In Table 4.6 the degree of crystal orientation for ZnO layers grown on different substrate materials but under equal conditions is given. The substrate material has a clear influence on the c-axis distribution.

Table 4.6  c-axis distribution of zinc oxide films grown on different substrates.

<table>
<thead>
<tr>
<th>Substrate Material</th>
<th>C-axis Distribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>±15°</td>
</tr>
<tr>
<td>Al₃(111)-SiO₂-Si</td>
<td>±6°</td>
</tr>
<tr>
<td>Al₃(111)-Si</td>
<td>±3°</td>
</tr>
<tr>
<td>Al₃(low (111) orientation)-Si</td>
<td>±8°</td>
</tr>
</tbody>
</table>

In Fig. 4.10 a typical three-dimensional pole figure of a 1.3 μm thick ZnO layer (grown on silicon) is given (obtained by using a Siemens D5000 diffractometer).
It shows the random orientation of the ZnO crystals in the plane of the substrate, typical for a poly-crystalline material.

Fig. 4.10 Typical pole figure of a 1.3 μm thick ZnO layer grown on silicon.

**Uniformity and surface roughness of ZnO layers**

A high uniformity of the thickness of the plate in which the slow Lamb waves are generated is required in order to prevent velocity dispersion. The relation between the plate thickness and the phase velocity of the slow Lamb wave was described in chapter 2.

A rough plate-surface causes wave reflections and hence propagation losses. Therefore, a smooth zinc oxide surface is required.

The uniformity and surface roughness measurements were performed using an alpha step 200 (Tencor Instruments). The uniformity is measured over a 100 mm diameter wafer (substrate material: p-type <100>-cut silicon, standard deposition parameters, zinc oxide layer thickness 1.5 μm). The non-uniformity within a 10 mm diameter circle amounts to ±1 %. The surface roughness of a 1.5 μm thick piezoelectric zinc oxide layers was about 4 nm.
Breakdown voltage
The zinc oxide layers used are relatively thin (1-2 μm). The breakdown voltage was determined in order to establish design rules with respect to the maximum permissible voltages over the layer. This is of importance because of the monolithic integration of the acoustic device with electronic circuitry. The use of decoupling capacitors must be obviated so that D.C. voltage levels will be directly coupled to the acoustic device, creating an electric field between the IDT pattern and the aluminum layer located underneath the zinc oxide. The breakdown voltage was measured on several devices and values between 4 and 10 V/μm were found for 1 to 2 μm thick layers.

4.5.3 Processing of zinc oxide

ZnO is a very reactive material and it is attacked by standard etch solutions such as aluminum etch. Also standard cleaning procedures can damage the ZnO layer, and ZnO could cause contamination of the process chambers. Therefore, different process steps have to be used once the ZnO layer has been deposited on the wafer [95]. The processes are discussed in the following sections.

Cleaning
After the deposition of the zinc oxide the wafers have to be cleaned before further processing. The standard cleaning fluids (nitric acid fuming 100 %, water) damage the ZnO layer and cannot be used. Organic solvents, on the other hand, do not affect the zinc oxide. A cleaning cycle containing acetone, toluene and dimethylsulfoxide (DMSO) was used (see Table 4.7). During cleaning in these fluids, ultrasonic agitation was applied.
Table 4.7  Three-step cleaning cycle for zinc oxide.

<table>
<thead>
<tr>
<th></th>
<th>acetone, 5 min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DI water rinse, 1 min.</td>
</tr>
<tr>
<td></td>
<td>dry</td>
</tr>
<tr>
<td>2</td>
<td>toluene, 5 min.</td>
</tr>
<tr>
<td></td>
<td>DI water rinse, 1 min.</td>
</tr>
<tr>
<td></td>
<td>dry</td>
</tr>
<tr>
<td>3</td>
<td>DMSO (dimethylsulfoxide), 5 min.</td>
</tr>
<tr>
<td></td>
<td>DI water rinse, 1 min.</td>
</tr>
<tr>
<td></td>
<td>dry</td>
</tr>
</tbody>
</table>

Etching
Zinc oxide is attacked by all common acids and bases. Because of the crystalline structure of zinc oxide, the etch rate of many etchants is direction-dependent. Grain size and porosity of the zinc oxide layer also affect the etch rate [81]. It should be noted that the structure of the zinc oxide at the start of growth,

Table 4.8  Etchants for etching of zinc oxide films.

<table>
<thead>
<tr>
<th>etchant</th>
<th>etch rate series 1 [μm/min]</th>
<th>etch rate series 2 [μm/min]</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>H$_2$PO$_4$, 10 ml HAc, 10 ml H$_2$O, 100 ml</td>
<td>1.5</td>
</tr>
<tr>
<td>b</td>
<td>H$_2$PO$_4$, 1 ml HAc, 100 ml H$_2$O, 100 ml</td>
<td>0.8</td>
</tr>
</tbody>
</table>

approximately the first few microns, is different from the structure above the first microns (as is shown in Fig. 4.6). For slow Lamb-wave devices, only thin zinc oxide films are required, typically 0.7-2 μm thick. The etchants used to pattern the zinc oxide are given in Table 4.8. The etch temperature was 20 °C, the etchants were stirred. HPR204 photoresist was used as a masking layer.
The test samples of series 1 were oxidized silicon substrates covered with 1 µm thick zinc oxide. The samples of series 2 were oxidized silicon covered by a 600 nm thick sputtered aluminum layer (containing 1% silicon), the zinc oxide layers were between 0.8 and 1.7 µm thick. The above etch rates all relate to etching parallel to the c-axis of the zinc oxide crystal.

The etchants show a lower etch rate for zinc oxide grown on aluminum, which indicates that the quality of zinc oxide grown on aluminum is higher than that grown on silicon dioxide. For the fabrication processes reported here, etchant #a was used for the fast etching of thick ZnO layers, etchant #b was used for slower etching. The etch rate of these mixtures varies approximately by 30% at a temperature change of 5 °C. The shelf lives of solutions #a and #b are at least one week.

The undercutting of a 1 µm thick zinc oxide layer is about 3 µm for etchant #a, and 5 µm for etchant #b. The lateral etching is faster than the axial etching, but in this application the undercutting of the zinc oxide is not critical. By using a dry-etching process, undercutting can be minimized [96].

![Fig. 4.11](image-url) **Photograph showing the undercutting of a 1 µm thick zinc oxide film after wet etching. The dark areas represent zinc oxide, in the light areas the zinc oxide is etched.**

In Fig. 4.11, the undercutting of a 1 µm thick zinc oxide using etchant #b is shown. The dark areas represent the zinc oxide, in the light areas the zinc oxide is etched. In the test blocks, the gaps and lines were designed with equal width (the values are given in µm in the photograph). The photograph shows that in
the 10 μm test block the lines are almost totally etched, indicating 5 μm undercutting.

Fabrication of aluminum patterns on top of zinc oxide layers
For the fabrication of aluminum patterns on top of zinc oxide, an aluminum layer on top of the zinc oxide is vacuum evaporated. This layer should be kept as thin as possible, since thick aluminum patterns induce more reflections of the acoustic waves, thus affecting the device performance. A minimum thickness of 200 nm is required to ensure good electrical conductivity.

For the etching of the aluminum, a solution of KOH and K₃Fe(CN)₆ in water can be used (see Table 4.9). For the thin aluminum layers the slow etch solution was used. The etch temperature was 20 °C, the etchants were stirred. HPR204 photoresist was used as a masking layer.

The undercutting of the aluminum depends only on its thickness, the axial and lateral etch rates are equal.

![Table 4.9](image)

<table>
<thead>
<tr>
<th>etchant</th>
<th>etch rate [μm/min]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 g KOH, 10 g K₃Fe(CN)₆, 100 ml H₂O</td>
<td>0.5</td>
</tr>
<tr>
<td>1 g KOH, 10 g K₃Fe(CN)₆, 600 ml H₂O</td>
<td>0.2</td>
</tr>
</tbody>
</table>

4.5.4 Aspects of mechanical prestress in the sensor plate

The main contributor to stress in the layered plate is the zinc oxide layer. The slow Lamb-wave phase velocity increases significantly when in-plane stress (or prestress) occurs, as shown in chapter 2. An excess of stress could increase the velocity such that it exceeds the sound velocity of the loading liquid, thus causing dramatic propagation losses. In addition, stressed zinc oxide will show relaxation behavior in time and cause aging effects which change the specifications. When zinc oxide films are used in a thin layered structure such as the slow Lamb-wave device, the stress causes buckling, which makes the plate very fragile and unusable for sensor purposes.

For these reasons, the influence of the growth parameters on the residual stress
in zinc oxide was investigated. X-ray diffraction analysis was used to determine the amount of stress occurring in the layered structure. In addition, the usefulness of a stress-relaxation process to decrease the zinc oxide stress after deposition was examined.

**Stress in zinc oxide films**

Stress in deposited thin-films usually contains a thermal component and an intrinsic component. The thermal component is caused by the difference between film deposition temperature and the device’s operating temperature. The thermal expansion coefficient of zinc oxide (a-axis: $4 \times 10^{-6} \text{ K}^{-1}$) is larger than that of silicon ($2.33 \times 10^{-6} \text{ K}^{-1}$), and thus there is a tensile in-plane stress in the zinc oxide due to the cooling after deposition.

The intrinsic stress is caused by imperfections which occur in the crystals during film growth. The intrinsic stress in zinc oxide is compressive and its magnitude can be much larger than the (tensile) thermal component, thus giving rise to a residual compressive stress and strain. It has been demonstrated that the growth parameters such as temperature, pressure, power, gas mixture, and sputter setup can have influence on the intrinsic stress [92,97,98,99,100,101].

The intrinsic stress in films with a high melting point but which have been deposited at a low temperature is large. At low deposition temperatures, the adsorbing atoms have too little energy to arrange themselves in their lowest energy state and intrinsic stresses are built up. However, if the deposition temperature is too high, other negative effects can take place such as large grain growth.

At high gas pressure, the particles arriving at the substrate have lower energy due to the greater number of collisions before deposition, and arrangement becomes more difficult. At very low pressure, the energy of the adsorbed atoms can become too high which causes a bombardment of the substrate. This hampers the formation of the film and causes intrinsic stress.

A higher deposition rate (because of a higher sputter power) results in higher particle energy at the substrate, thus yielding a smoother arranging of the adsorbed atoms. If the rate is too high, the adsorbed atoms might be encapsulated by succeeding atoms before their having found the lowest state of energy, which again results in higher intrinsic stress.

Several zinc oxide layers were grown on Si and Al-Si wafers (the aluminum contained 1% silicon and had a thickness of 0.6 μm). The stress in the zinc oxide layer was determined by measuring the (006) peak shift using X-ray diffraction [94]. The zinc oxide layers grown directly on silicon showed a much
larger peak shift than the zinc oxide layers grown on aluminum-covered silicon. Apparently, the number of defects in zinc oxide grown on aluminum is less than in zinc oxide grown on silicon. The difference in in-plane lattice constants might be an explanation; $a_{ZnO} = 3.18 \, \text{Å}$, $a_{Si} = 5.43 \, \text{Å}$, $a_{Al} = 4.04 \, \text{Å}$. The lattice constant of ZnO matches better with aluminum than with silicon.

The practical influence of pressure, temperature and sputter rate (power) on the stress in the zinc oxide was investigated for ZnO-Al-Si layered substrates. The results are shown in Fig. 4.12, Fig. 4.13 and Fig. 4.14, respectively.

![Graph showing stress in the ZnO film versus the oxygen pressure](image1)

**Fig. 4.12** Stress in the ZnO film versus the oxygen pressure (sputter temperature 375 °C, sputter current 2.8 A).

![Graph showing stress in the ZnO film versus sputter temperature](image2)

**Fig. 4.13** Stress in the ZnO film versus sputter temperature (pressure 6 mTorr, sputter current 2.8 A).
In the range of 4 to 8 mTorr, a pressure of 6 mTorr (100% O₂, temperature 375 °C, sputter current 2.8 A) gave the lowest (compressional) stress (Fig. 4.12). Increasing the temperature from 225 to 375 °C (6 mTorr, 2.8 A) lowers the residual stress, from -300 to -100 MPa (Fig. 4.13). Increasing the sputter current from 2.4 to 3.1 A (6 mTorr, 375 °C) also shows a decreasing compressional stress, at about 3.0 A the stress inverses from compressional to tensile (Fig. 4.14). The compressive intrinsic stress is now in the same order of magnitude as the tensile thermal stress, resulting in a zero residual stress.

Nonetheless, the zinc oxide films deposited on the actual process wafers exhibited higher (compressive) stress levels. The fact that these wafers are only locally covered with aluminum strongly influences the stress.

Stress relaxation

Post-deposition relaxation of stress in zinc oxide is possible through annealing. Through the heat treatment boundary defects are eliminated and the c-axis orientation of the poly-crystalline zinc oxide is improved [99,102,103, 104,105,106].

Both ZnO-Si and ZnO-Al-Si layered structures were annealed at temperatures between 280 °C and 400 °C, for periods up to 16 hours. Annealing was performed in vacuum, the samples were cooled quickly by placing them on a cold-block (in air). In Fig. 4.15, the results of the annealing is shown for ZnO-Si and ZnO-Al-Si layered samples. The anneal time of each measurement was 960 minutes.
Fig. 4.15  Zinc oxide stress before and after annealing treatment (anneal time of 960 min. per measurement). The white and gray dots are from a ZnO-Si layered structure, the black dots are from a ZnO-Al-Si structure.

The level of stress was substantially decreased for ZnO-Si layered samples at temperatures above 260 °C. For the ZnO-Al-Si layered samples the initial stress was much lower and only at high annealing temperatures (300-350 °C) some relaxation was observed. However, the acoustic performance of Lamb-wave devices annealed at 350 °C decreases dramatically, possibly due to aluminum diffusion into the zinc oxide.
Chapter 5

The experimental results are presented in this chapter. The packaging and measurement setup of the devices are described in section 5.1. The performance of the delay line and the amplifier are discussed in sections 5.2 and 5.3, respectively. The monolithic sensor system is presented, and examined in terms of stability and temperature behavior with and without liquid loading (section 5.4). The system was employed for different applications which is described in section 5.5. First, the sensitivity for mass adsorption in air was investigated (by determination of the influence of a deposited elastic thin layer). A preliminary measurement in which particles were adsorbed from a liquid was performed to investigate the capability for biosensor applications. Measurements in liquids were conducted for density and viscosity sensing, and for the determination of the weight percentage of liquid mixtures.

This chapter is ended with conclusions and the outlook given in section 5.6.

5.1 Packaging and measurement setup

The sensor chips were mounted in a ceramic pin-grid array (PGA, type cavity-down) provided with a square-shaped laser-drilled hole. The sensor chip was placed "flush" to the surface of the PGA after which they were glued together using epoxy. Aluminum bonding wires were used to realize electrical connections to the PGA. The front side of the chip was protected by covering it with a cap (Fig. 5.1). The 'sensing' side and the 'electronic' side of the chip are perfectly separated.

The two sides of the chip are shown in the photographs reproduced in Fig. 5.2.
The PGA is simply plugged into a zero-force connector, which provides the input and output connections to the power supply and PC-controlled measurement instruments such as a network analyzer (NWA), frequency counter, and multimeter. The delay line and the electronics were integrated on one chip but were designed such that they could be tested separately.

The fluid loading experiments were conducted in three different setups. The first is an open cuvette in which an aluminum block is sealed to the PGA by means of a viton O-ring. The cuvette has a volume of 50 cm$^2$ and can be filled with any liquid (Fig. 5.3).
The second setup is a closed cell consisting of an aluminum block provided with a fluid inlet and outlet, and an O-ring sealing (Fig. 5.4). The volume above the sensor is about 150 μl. This cell can be used as a flow cell. A photograph of this setup can be seen in Fig. 5.5.

The third setup was formed by mounting a glass plate directly on the silicon chip. The stream area of each sensing element was about 1.5 mm², the volume of the flow chamber was 12 μl. The glass plates were provided with fluid inlets and outlets, as shown in Fig. 5.6. Both steady and flow experiments are possible.
Fig. 5.5  Photograph of the closed cell setup. The aluminum top-block has been removed from the sensor.

in this setup. Each of the setups has its specific advantage; the open cuvette is an accessible setup, it can be filled and emptied very easily; the flow setup provides a proper heat sink to the sensor, stabilizing the temperature; and the glass-cover setup requires only a small volume of fluid.

Fig. 5.6  Micro flow cell consisting of a glass plate mounted on the silicon chip.
5.2 Acoustic performance

5.2.1 Transfer characteristics

The transfer characteristics of the complete Lamb-wave delay lines were measured by using an HP 8753 network analyzer (NWA), equipped with an S-parameter test set. The input and output signals of the Lamb-wave delay line are balanced by using low-loss transformers. As discussed in chapter 2, in addition to asymmetric Lamb modes, symmetric Lamb modes can also exist in a thin plate.

![Graph showing insertion loss (IL) as a function of frequency in air (thin curve) and in water (thick curve) of a ZnO-Al-SiO₂-Si layered monolithically integrated Lamb-wave delay line.](image)

*Fig. 5.7 Insertion loss (IL) as a function of frequency in air (thin curve) and in water (thick curve) of a ZnO-Al-SiO₂-Si layered monolithically integrated Lamb-wave delay line.*

In Fig. 5.7 a typical frequency transfer (S-parameter S₂₁, 50 Ω termination) of a ZnO-Al-SiO₂-Si layered plate is given (thin curve), showing the A₀ mode and the S₀ mode at 19 and 94 MHz, respectively. At 120 MHz another peak is found which is the third harmonic of the A₀ mode, for which the wavelength $\lambda=p/3$
(the device under test has a period $p=80$ μm). The background of the transfer lies at a level of about 80 dB at 20 MHz, which shows the high suppression of electromagnetic feedthrough. The thick curve gives the transfer if the plate is loaded by water, demonstrating the strong frequency shift of the $A_0$ mode and some extra attenuation due to viscous losses. In contrast to the $A_0$ mode, the $S_0$ mode shows almost no frequency shift due to the water loading because its motion is mostly shear. Viscous losses also lower the $S_0$ peak. The third harmonic of the $A_0$ mode completely vanishes if loaded by water. This is explained by the velocity of this harmonic wave which is about 3200 m/s, much higher than the sound speed of sound in the water (1500 m/s). The radiation of acoustic energy into the water causes high propagation losses.

![Frequency transfer of an $A_0$ Lamb-wave in a ZnO-Al-SiO$_2$-Si layered plate (in air).](image)

Fig. 5.8 shows the typical $A_0$ frequency transfer of one of the delay lines which was used in the monolithic oscillator. The slanted delay line has a total thickness of about 8.25 μm (1.5 μm ZnO, 0.65 μm Al, 0.1 μm SiO$_2$ and 6.0 μm Si). The slanted delay line has one uniform single-electrode and one uniform split-
Fig. 5.9 Frequency transfer of a non-slanted delay line.

electrode transducer, each with 38 finger pairs. The period is 80 μm and the aperture 2.2 mm. The center-to-center distance of the transducers is 60 wavelengths (4.8 mm).

The experimentally found center frequency is 18.8 MHz and the insertion loss is 28 dB ($S_{21}$, 50 Ω).

From the phase, a mode distance of about 660 kHz is found, which is considerably more than the calculated 313 kHz distance (the mode distance is calculated by $f_c \alpha/L_{path}$, $f_c = 18.8$ MHz and $L_{path} = 60 \lambda$). The effective delay time $\tau$ is shorter because of the small edge-to-edge distance of the two transducers, which is only $20\lambda$, while the center-to-center distance is $60\lambda$. The electrodes of the sending and receiving IDT that are close together have more influence on the delay time than the electrodes of each IDT that are near the edges of the device. The waves launched from the last electrodes have to cover a much longer path than the waves from the first electrodes, and suffer from attenuation and reflections occurring within the transducer. For long IDT distances this effect is less significant because in this case a wave launched by the first electrode covers
almost the same distance as the wave from the last electrode. The mode distance experimentally found (660 kHz), yields an effective path length of $L_{\text{eff}} = 28\lambda$.

Also, the frequency distance between the first two amplitude nulls (defined as $2f_r/N_r$, where $N_r$ is the number of finger pairs of the longest IDT) is influenced by the short edge-to-edge distance of the transducers. Instead of the calculated 990 kHz, a width of 1900 kHz is found, yielding an effective number of finger pairs $N_{r,\text{eff}} = 20$. However, the number of modes within the -3 dB pass-band is not changed because both the effective path length and the effective number of finger pairs decrease equally.

### 5.2.2 Suppression of reflections

Fig. 5.9 shows the frequency transfer of a non-slanted delay line, with a transducer structure similar to the slanted delay line. The edge reflections interfere with the main signal and deteriorate the transfer. Comparing this figure to Fig. 5.8 shows the dramatic suppression of edge reflections achieved by applying the slightly slanted delay lines.

### 5.2.3 Prestress

The experimentally found center frequencies of the devices were somewhat higher than designed. This is due to residual stress, mostly in the zinc oxide layer. The residual stress or prestress in the device described above amounts to about 600 MPa, increasing the center frequency from 17.9 to 18.8 MHz. Also in the other devices compressive stresses of between 200 and 700 MPa were found. The zinc oxide deposition conditions for zero residual stress (see chapter 4) were found for silicon wafers completely covered with aluminum. The wafers in which the Lamb-wave delay-line oscillators are fabricated are only partly covered with aluminum during zinc oxide deposition. This influences the growth and the result is a deviating level of stress in the zinc oxide layer.
Fig. 5.10  Frequency transfer of an $A_0$ Lamb wave loaded by air.

Fig. 5.11  Frequency transfer of an $A_0$ Lamb wave loaded by water.
5.2.4 Liquid loading

The influence of liquid loading on the insertion loss of the $A_0$ wave is seen by comparing Fig. 5.10 (which is a blow-up of the transfer given in Fig. 5.8) and Fig. 5.11. The typical frequency transfer of one device is given in air and in water. The water shifts the center frequency from 18.8 MHz to 14.5 MHz and the insertion loss increases from 28 to 34 dB. A striking improvement of the smoothness of the pass-band behavior and phase is observed. The reflected waves that were not eliminated by the slanted edges and the split-finger electrode have a longer path to cover than the main signal which propagates from one IDT directly to the other IDT. Consequently, the viscous damping has a larger effect on the interfering reflections, thereby improving the shape of the transfer characteristics.

The time-domain plot (obtained by using the Fast Fourier Transform facility of the NWA) is shown in Fig. 5.12, for air and water loading. At $t=0$ a pulse is applied to the sending IDT. Due to the influence of the water loading, the $A_0$

![Graph showing the time-domain response (FFT) in air and in water of an $A_0$ Lamb wave.](image)

*Fig. 5.12 Time domain response (FFT) in air and in water of an $A_0$ Lamb wave.*
Fig. 5.13  Amplifier transfer characteristics.

Lamb-wave velocity decreases and the delay time (the time in which the wave travels from one IDT to the other) increases. The insertion loss is increased due to the water, and the reflections which are seen in air are suppressed dramatically. The EM-feedthrough propagates with the speed of light, and is seen as a peak at t=0 seconds.

5.3 Amplifier performance

The amplifier transfer characteristics were obtained by using a network analyzer. The measured amplifier gain and phase are shown in Fig. 5.13. The -3 dB bandwidth found from the figure amounts to 15 MHz.
Fig. 5.14 Amplifier gain-control voltage versus the gain.

Fig. 5.15 Phase changes in the amplifier versus the gain.
The measured gain control voltage $U_{GC}$ versus the gain is plotted in Fig. 5.14 and shows a small frequency dependency in the range of 12 to 18 MHz due to the fall-off of the amplifier gain. For low-loss devices (25-30 dB), the sensitivity for changes in wave attenuation is smaller than for high-loss devices (40-45 dB). The energy consumption of the amplifier is 500 mW. The operating temperature in air reaches a value of about 50 °C, when loaded by a liquid the temperature lowers to 40 °C.

The gain level could affect the phase of the amplifier and deteriorate the performance. In Fig. 5.15 it is shown that the influence of the gain level is small. In the range of 25 to 35 dB gain, the maximum deviation is 0.25°, corresponding to about 450 Hz.

5.4 Oscillator performance

The monolithic oscillator systems realized consist of two identical oscillators fabricated in one 10×10 mm² chip. The dual configuration serves for suppression of unwanted common signals such as fluctuations in temperature, pressure or fluid flow. If used as a (bio)chemical sensor, the influence of fluid density and viscosity is suppressed by the dual configuration. Both oscillator outputs are mixed and filtered, leaving the frequency difference as the output signal.

5.4.1 Oscillator instability

The frequency instability of the sensor system depends strongly on the measurement setup used, especially when loaded by a liquid. If the sensor is temperature stabilized, the short-term instability in air amounts to about 30 ppb (0.5 Hz at an operating frequency of 18 MHz, standard deviation of 100 measurements at 50 ms gate, HP 5335A frequency counter).

If the open cuvette is used for liquid loading, a substantial increase of the instability is found (for water loading the instability is between 1 to 50 ppm which corresponds to 10 to 700 Hz at 14 MHz).

If the flow cell is used, the water-loaded instability of the oscillator varies from 0.1 ppm in a non-flow situation to 0.5 ppm at low flows (< 1 ml/min) and to 10-30 ppm at high flows (> 3 ml/min).

The lowest instability was obtained with the use of the glass-cover: 50 to 100 ppb (0.7-1.5 Hz at 14 MHz) in a steady situation (no flow). An instability measurement is shown in Fig. 5.16.
Fig. 5.16 Oscillator frequency instability of a water-loaded oscillator using the glass-cover setup (standard deviation, 100 measurements, gate 50 ms).

The high instability in the open cuvette is attributed to the open boundary of the liquid column to the air. Acoustic vibrations from other instruments placed in the vicinity of the measurement setup allow the liquid to move and vibrate above the sensor, which increases the instability. If the liquid column is closed, without entrapping any air, the stability is improved because free movement of the water is no longer possible. This explains the higher stability found if the flow cell or the glass-cover were used. These results demonstrate the importance of careful packaging.

Experiments on the noise of the acoustic delay line verified that water loading increases the phase noise of the Lamb-wave delay line, which explains the increase of the short term instability, even when packaged adequately. The phenomena causing the noise increase have not been investigated, but considering the evanescent moved liquid layer as a part of the waveguide makes a noise contribution from the liquid plausible.

The hybrid system, where electronics and acoustic delay line are fabricated in separate chips showed comparable instability behavior [107].
5.4.2 Temperature behavior

The temperature behavior of the delay line and the amplifier were examined separately.
A monolithic dual oscillator was provided with two temperature sensors. One was glued at the middle rim of the dual delay line, the other at a side rim. The aluminum resistive heater located at the middle rim was used to heat the Lamb-wave sensor. The temperatures of the middle rim and the side rim were measured and averaged to obtain the temperature of the plate, located between the two temperature sensors. In Fig. 5.17, the center frequency of the $A_0$ wave versus the average temperature is given. The temperature coefficient of the delay line found from the figure is $-1.2 \text{kHz/°C}$.

![Graph showing the relationship between $f_c$ and $T$](image)

Fig. 5.17 $A_0$ Lamb-wave center frequency versus the delay line temperature.

The temperature sensitivity of the amplifier phase amounts to $-0.07 \text{°C}$. For a mode distance of 660 kHz, the frequency shift due to a temperature change amounts to $-130 \text{Hz/°C}$. Combining the results for delay line and amplifier yields a temperature sensitivity of the complete oscillator of less than $-1.4 \text{kHz/°C}$ in the range of 20 to 70 °C. A temperature test on a complete oscillator gave a temperature dependence of $-1.1 \text{kHz/°C}$ in the range from 25 to 50 °C.
5.5 Sensor applications

The $A_0$ Lamb-wave sensor can be used for many different applications. Examples are chemical sensing in gases or liquids, curing behavior of polymers, paints, or epoxies, and the determination of physical properties of liquids. We hereby briefly discuss the possibilities for gas sensing and biosensing. In addition, frequency and gain level response to liquid loading are examined for the determination of density and viscosity of liquids. Finally, the accurate determination of the composition of liquid-mixtures is discussed.

5.5.1 Mass sensitivity

For the determination of the mass-loading sensitivity, the center frequency of a Lamb-wave oscillator was measured before and after the deposition of a 0.2 $\mu$m thick polymethyl methacrylate (PMMA) layer (approximately $2\times10^{-4}$ kg/m$^2$) on top of the thin plate. The frequency shift found for a ZnO-Al-SiO$_2$-Si layered sensor with plate mass $m = 2.4\times10^{-2}$ kg/m$^2$ and operating at 18.8 MHz amounts to -74 kHz (or -3.7 Hz/ng/cm$^2$), which corresponds well to the theoretical value of -3.9 Hz/ng/cm$^2$ (measurements in air).

Gas sensing, the detection of adsorption of particles at the sensor surface, is an application of the device. Compared to Rayleigh-wave gas sensors the sensitivity is between 4 and 10 times higher. For the (selective) adsorption of the gas molecules, the plate area has to be provided with a (selective) thin-film chemical interface which is capable of adsorbing the particles. An advantage of acoustic gas sensors is that the adsorption of particles by itself is the mechanism which creates the sensor response, no extra mechanism is required (such as, for example, an optical or electrical effect for optical or resistive gas sensors, respectively).

The gas sensor application is here not further discussed.

5.5.2 Biochemical measurements

Biochemical applications in liquids also require a (bio)chemical interface for the selective adsorption of particles. A preliminary experiment was performed where a biochemical interface (human serum albumin, HSA) was deposited at the
Fig. 5.18 Sensor response to HSA adsorption compared to the HSA-to-silicon adsorption isotherm. $C_b$ is the HSA concentration in the solution, $C_s$ is the adsorbed HSA at the sensor surface.

sensor surface. The sensor (a hybrid oscillator system) was exposed to different concentrations of HSA solved in phosphate buffered saline (PBS) and the deposition of mass was monitored [107]. In Fig. 5.18, the experimental results and the adsorption isotherm obtained from radio-active labeled adsorption at silicon test samples are shown. The calculated mass sensitivity of the test device in a watery environment amounts to 16 m$^2$/kg (or 2.2 Hz/ng/cm$^2$). However, the experimentally found sensitivity is about three times 3 lower: 0.8 Hz/ng/cm$^2$. The further development of a Lamb-wave biosensor is part of future research, and is, therefore, not discussed here.

5.5.3 Frequency response to liquid loading

The oscillator frequency response to different liquids was determined using the glass-cover setup (steady situation). The sensor was loaded by a liquid and after a warm-up period of 1 minute the oscillation frequency and gain control voltage were measured. Between measuring two liquids, the sensor was cleaned by using water and ethanol. The experimental and calculated frequencies are given in
Table 5.1. The calculation program is fitted to the measurement in water after which the other responses are calculated. The sensor response can be predicted very well for the tested low-viscous liquids. The response to the high-viscous glycerol deviates a few percent from the calculated value.

<table>
<thead>
<tr>
<th>fluid</th>
<th>( f_{\text{calc}} ) [MHz]</th>
<th>( f_{\text{exp}} ) [MHz]</th>
<th>deviation [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>air</td>
<td>18.030</td>
<td>18.045</td>
<td>+0.1</td>
</tr>
<tr>
<td>water</td>
<td>13.920</td>
<td>13.924</td>
<td>+0.0</td>
</tr>
<tr>
<td>methanol</td>
<td>12.800</td>
<td>12.761</td>
<td>-0.3</td>
</tr>
<tr>
<td>ethanol</td>
<td>12.720</td>
<td>12.855</td>
<td>+1.1</td>
</tr>
<tr>
<td>glycerol</td>
<td>13.350</td>
<td>13.770</td>
<td>+3.1</td>
</tr>
</tbody>
</table>

5.5.4 Gain level response to liquid loading

To demonstrate viscosity sensing, glycerol-water solutions (0-100 %) were used. The viscosity of this mixture changes a few decades from 0 % to 100 % glycerol.

In Fig. 5.19, the gain level of the oscillator is plotted versus the weight percentage glycerol in water. Fig. 5.20 shows the insertion loss \( IL \) versus the square root of the liquid viscosity-density product. Both the oscillator response calculated from the oscillator and the insertion loss determined by use of a network analyzer are plotted. Due to dissipation in the circuitry (240 mW in each oscillator) the operating temperature of the liquid-loaded oscillator is 40 °C. At this elevated temperature, the viscosity of the glycerol-water mixtures is considerably lower. The NWA measurements are obtained at 20 °C. The two curves correspond very well, and curves obtained from different devices showed a high reproducibility. Nonetheless, the curves deviate notably from the calculated insertion loss. At 100 % glycerol (20 °C) the experimental loss is 42 dB while the calculated loss (using the equation given in section 2.4.1)
Fig. 5.19  Gain level of the Lamb-wave sensor versus the weight percentage of glycerol in water.

Fig. 5.20  Insertion loss IL of the Lamb-wave sensor versus the square root of the viscosity-density product.
amounts to about 120 dB for a path length of 4.8 mm. This is (partly) due to the shorter effective path length as discussed in section 5.2. The appearance of slip at the solid-liquid interface [108,109] or relaxation effects [110,111] might also contribute to the decreased loss, but because of the low frequencies used these effects are expected to be small.

5.5.5 Composition of liquids

Mixtures of liquids have a specific course of density, viscosity and sound speed which makes the determination of the composition of the liquid by measuring these physical properties possible. In particular, the high sensitivity for density allows the accurate determination of the weight percentages of a specific compound of a mixture.

![Graph showing the relationship between oscillator frequency and weight percentage of ethanol in water.]

Fig. 5.21 Calculated (dashed curve) and experimental oscillator frequency versus the weight percentage ethanol in water.

For validation of this, the weight percentage of water in water-ethanol mixtures was determined. In Fig. 5.21, the calculated and experimental frequency response to water-ethanol solutions are shown, the operating temperature of the
sensor is 40 °C. The high correspondence of the two curves demonstrates the possibilities for accurate mixture measurements. The curves show a maximum frequency at about 25 % ethanol in water. This is explained as follows: When increasing the percentage of ethanol in water, the density of the mixture lowers, thus increasing the \( A_0 \) Lamb-wave velocity and thus the oscillator frequency. However, the sound speed of ethanol is much lower than the sound speed of water, and since the sound speed of the mixture lowers rapidly when ethanol is added, the evanescent moved liquid layer becomes thicker, increasing the liquid loading of the sensor and therewith decreasing the frequency. At 100 % ethanol, the sound speed (about 1200 m/s) is very close to the \( A_0 \) wave velocity of about 1040 m/s. The influence of the liquid loading has become dominant, and small changes in the percentage of ethanol in water have a considerable influence on the oscillator frequency.

In the region of 90 to 100 % ethanol, the sensitivity of the sensor is 44 kHz/% water, yielding a resolution of better than 10\(^4\) % water in ethanol.

5.6 Conclusions and outlook

5.6.1 Conclusions

Modeling of asymmetric zero-mode Lamb waves
The flexural-wave approximation of asymmetric zero-mode (\( A_0 \)) Lamb waves described in chapter 2 is a sufficiently accurate method to predict the wave velocities in micromachined ZnO-Al-SiO\(_2\)-Si layered thin plates loaded by different fluids. The equations derived are a useful tool for the design of sensitive sensors.

Technology for the monolithic Lamb-wave sensor system
The technologies of bipolar IC processing, bulk micromachining of silicon, and piezoelectric zinc oxide layers have been successfully combined for the realization of small smart Lamb-wave oscillators for sensor applications. The high fabrication yield already attained (75 %) provides the prospect of efficient production of low-priced sensor systems. Decrease of the residual stress introduced by the zinc oxide layer would further improve the performance of the system.
Oscillator setup
The designed monolithic Lamb-wave oscillators show stable oscillation and low acoustic losses when loaded by a fluid (except for high-viscous liquids). The dual output of the system gives both the Lamb-wave velocity and amplitude. This can be used to simultaneously determine different properties of a fluid such as density and viscosity.

Packaging
The packaging and measurement setup appeared to be of crucial importance for the stability of the sensor. Free movement of the liquid that loads the sensor must be prevented in order to attain high oscillator stability. This can be accomplished by closing off the liquid from the environment by gas-free packaging.

Applications
The Lamb-wave oscillator is a generic sensor system for many applications. In this work, the system has been demonstrated for the determination of physical properties of liquids: density and viscosity. In addition, the accurate determination of the composition of liquid mixtures using the system has been shown, and the prospects for (bio)chemical sensing discussed. The frequency response of the sensor system corresponds very well to the calculations. The insertion loss found experimentally is lower than expected but is very reproducible.

5.6.2 Outlook
Further development of the sensor system depends on the selected application. The current system has already been prepared for use with a microcontroller which allows for automatic process control. By adding more sensors to the system, an array setup can be realized for the simultaneous determination of physical and (bio)chemical properties of fluids. The packaging problem of the sensor requires special attention for each practical application.

The temperature sensitivity of the system can be compensated by applying one oscillator of the dual system as a reference. By loading it with a fixed reference liquid, the oscillation frequency of this reference oscillator is determined only by the temperature.
In this work, the $S_0$ Lamb mode has not been considered for sensing. Yet, the velocity and amplitude modulation of the $S_0$ mode caused by liquid loading differs from the $A_0$ mode. The $S_0$ mode reacts to mass adsorption but is insensitive to the liquid density. A Lamb-wave sensor which makes use of both modes might be very useful for determination of physical properties (using $A_0$) and (bio)chemical properties (using $S_0$) of a liquid simultaneously. Except for the pass-bands of both $A_0$ and $S_0$ modes, the delay line behaves as a filter with a high stop-band rejection. The two modes are not harmonically coupled and their center frequencies are far apart. This makes the use of one Lamb-wave delay line as the frequency-determining element for two oscillators possible, one operating at the $A_0$ frequency and the other at the $S_0$ frequency. Further investigation of this double-mode Lamb-wave sensor is required.
References

Chapter 1


Chapter 2

Modeling of Lamb waves


Chapter 3

Sensor system design


Chapter 4

Device fabrication


[92] A.D. Sathe and E.S. Kim, "Techniques to control residual stress in ZnO films," Proc.of the 7th Int. Conf. on Solid-State Sensors and Actuators, Transducers '93, Yokohama, Japan, pp. 158-161.


Chapter 5

Experiments


List of symbols

\( \alpha \) \hspace{1cm} \text{attenuation factor [Neper/m]}
\( \alpha_r \) \hspace{1cm} \text{slanting angle [°]}
\( \gamma \) \hspace{1cm} \text{angle of rotation of an element of a beam due to a shear force [°]}
\( \delta_r \) \hspace{1cm} \text{decay length of the evanescent-moved fluid layer [m]}
\( \varepsilon \) \hspace{1cm} \text{strain}
\( \eta \) \hspace{1cm} \text{shear viscosity [Pa.s]}
\( \theta \) \hspace{1cm} \text{angle between the direction of prestress and the neutral plane [°]}
\( \theta_i \) \hspace{1cm} \text{incident angle of an X-ray beam [°]}
\( \kappa^2 \) \hspace{1cm} \text{piezoelectric coupling coefficient}
\( \lambda \) \hspace{1cm} \text{wavelength [m]}
\( \nu \) \hspace{1cm} \text{Poisson's ratio}
\( \rho \) \hspace{1cm} \text{density [kg/m\textsuperscript{3}]} \n( \rho_f \) \hspace{1cm} \text{fluid density [kg/m\textsuperscript{3}]} \n( \sigma \) \hspace{1cm} \text{stress [N/m\textsuperscript{2}]} \n( \tau \) \hspace{1cm} \text{acoustic delay time [s]}
\( \phi_{\text{amp}} \) \hspace{1cm} \text{phase shift caused by the amplifier [°]}
\( \phi_{\text{dl}} \) \hspace{1cm} \text{phase shift caused by the delay line [°]}
\( \phi_{\text{loop}} \) \hspace{1cm} \text{loop phase [°]}
\( \psi \) \hspace{1cm} \text{angle of rotation of an element of a beam due to rotary inertia [°]}
\( \omega \) \hspace{1cm} \text{angular frequency [rad/s]}
\( \omega_0 \) \hspace{1cm} \text{angular oscillation frequency [rad/s]}
\( A \) \hspace{1cm} \text{cross-sectional area [m\textsuperscript{2}]} \n( A(\omega) \) \hspace{1cm} \text{amplifier gain}
( A_0 \) \hspace{1cm} \text{asymmetric zero-order}
( A_{\text{amp}} \) \hspace{1cm} \text{amplifier gain}
( B \) \hspace{1cm} \text{spectral bandwidth [Hz]}
B(ω) delay line loss

\( c \) phase velocity [m/s]

\( C_b \) concentration in the solution [kg/m³]

\( c_f \) fluid sound speed [m/s]

\( c_{gr} \) group velocity [m/s]

\( C_L \) amplifier input capacitance [F]

\( c_{long} \) longitudinal wave-velocity [m/s]

\( C_{norm} \) normalized capacitance of a finger pair [F/m]

\( C_s \) adsorbed mass at sensor surface [kg/m²]

\( c_{stress\_free} \) phase velocity in a stress-free plate or beam [m/s]

\( C_t \) transducer capacitance [F]

\( D \) bending stiffness [Nm]

\( E \) Young’s modulus [N/m²]

\( F \) shear force [N]

\( f_0 \) oscillation frequency [Hz]

\( f_c \) center frequency [Hz]

\( G \) rigidity modulus [N/m²]

\( G_0 \) power-supply output-conductance [S]

\( G_a \) maximum transducer radiation conductance [S]

\( G_a(\omega) \) transducer radiation conductance [S]

\( G_L \) amplifier input conductance [S]

\( h \) plate thickness [m]

\( I \) second moment of area [m⁴]

\( IL \) delay-line insertion loss [dB]

\( I_R \) current delivered by the receiving transducer [A]

\( k \) wave number [m⁻¹]

\( k_y \) propagation constant [m⁻¹]

\( L_{eff} \) effective center-to-center distance between two IDT’s in a delay line [m]

\( L_{path} \) center-to-center distance between two IDT’s in a delay line [m]

\( m \) plate mass per unit area [kg/m²]

\( M \) bending moment [Nm]

\( m_i \) integer

\( m_{visc} \) viscous mass loading term [kg/m²]

\( n \) number of wavelength in the propagation path

\( N \) prestress per unit width [N/m]

\( N_b \) in-plane force [N]

\( N_t \) number of finger pairs of the transducer

\( N_{t,eff} \) effective number of fingerpairs

\( p \) IDT period [m]

\( P \) pressure [Pa]

\( P(x) \) pressure distribution [Pa]
$P_L$  power dissipated in $G_L$ [W]
$P_R$  received power [W]
$P_T$  transmitted power [W]
$r$  adjustment term
$R$  relative sensitivity
$S$  sensitivity
$S_0$  symmetric zero-order
$S_n$  noise density spectrum [W/Hz]
$s_{short}$  short term instability
$t$  time [s]
$T$  temperature [°C]
$T_1$  sampling time interval [s]
$u$  particle displacement [m]
$U_0$  amplifier output voltage [V]
$u_d(t)$  output voltage level of the amplifier [V]
$U_{GC}$  gain control voltage [V]
$v$  particle velocity [m/s]
$W_b$  beam width [m]
$W_r$  transducer aperture [m]
$Y_L$  amplifier input admittance [S]
$Z$  transverse wave impedance of the coupled plate-fluid system [Ns/m³]
$Z_f$  transverse wave impedance of the fluid [Ns/m³]
$Z_{plate}$  transverse wave impedance of the plate [Ns/m³]
$\Delta f_{mode}$  frequency distance between two adjacent phase nulls [Hz]
List of constants

The following constants were used in this thesis:

$E_{\text{Si}[110]}$ 169 [GPa]
$E_{\text{SiO}_2}$ 70 [GPa]
$E_{\text{Al}}$ 71 [GPa]
$E_{\text{ZnO}}$ 131 [GPa]

$\nu_{\text{Si}[100]}$ 0.06
$\nu_{\text{SiO}_2}$ 0.2
$\nu_{\text{Al}}$ 0.34
$\nu_{\text{ZnO}}$ 0.42

$\rho_{\text{Si}}$ 2330 [kg/m$^3$]
$\rho_{\text{SiO}_2}$ 2200 [kg/m$^3$]
$\rho_{\text{Al}}$ 2700 [kg/m$^3$]
$\rho_{\text{ZnO}}$ 5720 [kg/m$^3$]
Summary

The design and technology of a smart Lamb-wave sensor system for the
determination of physical and (bio)chemical properties of fluids are described in
this thesis.

Acoustic wave types of which the acoustic energy is confined to the surface of
the solid in which they propagate are very sensitive to modulations which occur
at the surface. Lamb waves propagating a very thin plate show such a high
density of energy at the plate-surface. Asymmetric zero-order Lamb-wave
devices can be used in a gaseous environment, but also in liquids if the wave
velocity is lower than the sound speed of the liquid. In this case, no acoustic
energy is radiated into the loading liquid. The surface modulation caused by the
presence of a fluid or by adsorption of molecules at the surface of the plate
results in an alteration of the wave velocity and attenuation and this makes
Lamb waves attractive for fluid-sensor applications. The perfect separation of the
front of the sensor system where the waves are generated and detected and the
sensing back which contacts the fluid, is also an attractive feature of the Lamb-
wave sensor.

Lamb waves are generated and detected by using interdigital piezoelectric
transducers. By placing two transducers in-line on a substrate, an acoustic delay
line is obtained which is a basic element of acoustic sensors.

The velocities of slow Lamb waves are approximated by employing the
elementary theory for the propagation of flexural waves in thin plates. For plate
thicknesses small compared to the wavelength, simple expressions are obtained
which are used as a design tool for the realization of sensitive fluid sensors. The theoretical sensitivity to mass adsorption, and to changes in density and viscosity of an adjacent fluid has been determined.

By employing the Lamb-wave delay line as the frequency-determining element in an ultra-stable level-controlled oscillator, a small and accurate instrument is obtained. The oscillator frequency and the gain level are independently affected by the loading fluid, so that different properties of the fluid are monitored simultaneously. This feature allows the development of multi-purpose sensor systems. An example is the simultaneous determination of the density and viscosity of a liquid.

For slow Lamb waves a plate thickness-to-wavelength ratio typically of between 0.01 and 0.2 is required. The silicon micromachining technology makes the fabrication of very thin plates (a few microns thick) possible. For the generation and detection of the acoustic waves, a piezoelectric zinc oxide film is deposited on top of the silicon. The transducer configuration used determines the efficiency of the conversion of electrical to acoustical energy. The final design of the monolithic oscillator comprised slanted delay lines in a ZnO-Al-SiO$_2$-Si layered plate structure of 2.8×8 mm$^2$.

The fabrication of Lamb-wave devices in silicon enables integration of electronic circuitry and the sensor on one single silicon chip. Advantages of this silicon implementation of the device are in high performance and reliability, small size and low production costs.

For the design of the amplifier, the delay line was taken as a starting point. The insertion losses in the delay lines are typically 25 to 30 dB in air and 30 to 45 dB if loaded by a liquid. The voltage amplifier developed covers this whole range. The operating frequency of the asymmetric Lamb-wave oscillator ranges from 10 to 20 MHz.

The fabricated monolithic oscillator shows small acoustic losses and stable oscillation. The experiments have demonstrated the usefulness of the system for several applications such as the determination of density and viscosity of liquids, the determination of the composition of liquid mixtures, and (bio)chemical sensing.
Samenvatting

In dit proefschrift worden de werking en realisatie van een smart Lamb-golf sensorsysteem voor de bepaling van vloeistof- en gaseigenschappen beschreven.

Akoestische golftypen waarvan de akoestische energie geconcentreerd is aan het oppervlak van het materiaal waarin ze zich voortplanten, zijn erg gevoelig voor veranderingen die zich aan het oppervlak voordoen. Lamb golven die zich voortplanten in een zeer dunne plaat vertonen een zeer hoge energiedichtheid aan het plaatoppervlak. Asymmetrische nulde-orde Lamb-golf elementen kunnen zowel in een gas als in een vloeistof gebruikt worden. De oppervlakte-modulering die wordt veroorzaakt door de aanwezigheid van een gas of een vloeistof, of door de adsorptie van moleculen aan het oppervlak van de plaat, beïnvloedt de snelheid en de amplitude (demping) van de Lamb golf. Dit maakt de Lamb golven zeer geschikt voor sensortoepassingen. De perfecte scheiding tussen de voorkant van de sensor waar de golven opgewekt en gedetecteerd worden, en de metende achterkant waar het contact tussen de sensor en het gas of de vloeistof plaatsvindt, is ook een aantrekkelijke eigenschap van de Lamb golf sensor.

Met behulp van de elementaire theorie voor de propagatie van buigingsgolven in dunne platen zijn vergelijkingen afgeleid waarmee de snelheden van langzame Lamb golven onder verschillende gas- en vloeistofbelastingen berekend zijn. Deze vergelijkingen geven een goed inzicht in de verschillende sensorparameters die de mate van gevoeligheid voor gas- en vloeistofeigenschappen bepalen. Voorbeelden van deze parameters zijn de dikte en de materiaaleigenschappen van de plaat. De theoretische gevoeligheid is bepaald voor de adsorptie van moleculen aan het plaatoppervlak en voor veranderingen in de dichtheid en de viscositeit van een vloeistof die in contact staat met de sensor.
De Lamb golven worden opgewekt en gedetecteerd door interdigitale piezo-elektrische transduceren. Door twee van deze transduceren op lijn op een substraat te plaatsen wordt een akoestische vertragingslijn verkregen, een basiselement voor akoestische sensoren.

De Lamb golf vertragingslijn is als frequentiebepalend element toegepast in een zeer stabiele spanningsgeregeld oscillator waarmee een klein en nauwkeurig meetinstrument is verkregen. De oscillatiefrequentie en het versterkingsniveau worden onafhankelijk beïnvloed door verschillende eigenschappen van het gas of de vloeistof waardoor multi-functionele sensorsystemen ontwikkeld kunnen worden. Een voorbeeld is het gelijktijdig bepalen van de dichtheid en de viscositeit van een vloeistof.

De vertragingslijn is als startpunt genomen voor het ontwerp van de versterker. De verliezen in de vertragingslijnen zijn typisch 25 tot 30 dB in lucht en 30 tot 45 dB in vloeistoffen. De ontwikkelde spanningsversterker bestrijkt dit gehele bereik. De werkfrequentie van de asymmetrische nulde-orde Lamb-golf oscillator ligt tussen 10 en 20 MHz.

Voor langzame Lamb golven is een plaatdikte/golflengte verhouding tussen 0.01 en 0.2 nodig. Met silicium microbewerkingstechnieken zijn platen van slechts enkele micrometers dik gerealiseerd. Voor het opwekken en detecteren van de Lamb golven is een piezo-elektrisch laagje zinc oxide op het silicium aangebracht. De configuratie van de transducer bepaalt de efficiëntie van de omzetting van elektrische naar akoestische energie. Het uiteindelijke ontwerp van de in silicium geïntegreerde Lamb golf oscillator bevat een ZnO-Al-SiO₂-Si gelaagde plaatstructuur met een totale dikte van circa 8 micrometer en een oppervlak van 2.8×8 mm². De golflengte bedraagt 80 micrometer.

De fabricage van Lamb golf elementen in silicium heeft het mogelijk gemaakt om elektronische schakelingen met de sensor op één silicium chip te integreren. De voordelen van deze monolithische silicium-implementatie zijn een hoge betrouwbaarheid, kleine afmetingen en een goede prijs/prestatie verhouding.

Het gefabriceerde monolithische sensoreenststel vertoont lage akoestische verliezen en een stabiele oscillatie. De experimenten tonen de bruikbaarheid van het systeem voor verschillende toepassingen aan, zoals voor de bepaling van de dichtheid en viscositeit van vloeistoffen, voor de bepaling van de samenstelling van vloeistofmengsels en voor (bio)chemische metingen.
Acknowledgement

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List of publications


About the author

Michael J. Vellekoop was born in Amsterdam, on June 3, 1960. In 1978 he received the VWO diploma from College Leeuwenhorst in Noordwijkerhout. In the same year he started a study fysische techniek (physics) at the HTS Dordrecht. He received the B.Sc. degree in physics in 1982. From 1982 to 1984 he worked as a reserve officer at the Electrical Materials Laboratory of the Royal Netherlands Naval College. Since 1984 he has been with the Electrical Engineering Department of the Delft University of Technology where he is engaged in micro-acoustic devices research at the Electronic Instrumentation Laboratory. He is author and co-author of twenty-five scientific papers. In addition, he is co-founder and managing director of Xensor Integration bv. The company, founded in 1988, is involved with development and production of silicon sensors and actuators. In 1989 he started on a research project sponsored by the Dutch Technology Foundation (STW) in the field of monolithic Lamb-wave oscillators for sensor applications. The results of this research are described in this thesis.