NANOIMPRINTED HIGH-FREQUENCY SURFACE ACOUSTIC WAVE DEVICES:

GENERATION, CHARACTERIZATION AND ACOUSTO-ELECTRIC TRANSPORT

Serkan Büyükköse

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tronik, Germany

Dr. B. Vratzov NT&D - Nanotechnology and Devices,
Germany

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Author: Serkan Büyükköse

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DISSERTATION

to obtain
the degree of doctor at the University of Twente,
on the authority of the rector magnificus,
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on account of the decision of the graduation committee,
to be publicly defended
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by

Serkan Büyükköse

born on 17 December, 1982
in İzmit, Turkey

This dissertation has been approved by:

Promotor:

Prof. dr. ir. W. G. van der Wiel

This thesis is dedicated to my family.

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Chapter 1

Introduction

This chapter starts with a brief introduction to surface acoustic waves (SAWs) with a review of breakthrough results, specifically on SAW-assisted charge carrier transport from the literature. In the second part of this chapter, background for the rest of the thesis is given by introducing a mathematical description of SAWs, their interaction with charge carriers, SAW-induced modulation mechanisms of the electronic structure of semiconductors, and finally their generation and detection methods. At the end of the chapter an outline of the thesis is presented.

1.1 Introduction to Surface Acoustic Waves

Surface acoustic waves (SAWs) are elastic deformations propagating along the surface of an elastic body. Since their amplitude decays rapidly into the bulk material, the acoustic energy which is carried by the SAW is highly confined to a depth of around one wavelength below the surface. In other words, the surface of the material naturally becomes a waveguide for SAWs. The existence of SAWs was predicted and described by Lord Rayleigh in 1885 [1]. In an isotropic medium, the particle motion is elliptical and in a plane which is normal to the surface and parallel to the propagation direction, referred to as the sagittal plane. The typical SAW velocity in a solid material is around a few km/s. This very low velocity compared to that of electromagnetic waves was the main motivation for the application of SAWs in electronics due to the fact that SAWs have a great advantage to build a resonator which is approx. 10^5 times smaller than an electromagnetic resonator that operates at the same frequency.

Until demonstration of electrical generation of SAWs via interdigital transducers (IDTs) on a piezoelectric medium [2], the main interest in SAWs was limited to seismology. Nowadays, SAW-based electronic signal processing devices, such as filters, resonators, and delay lines are extensively used in the mobile and wireless communication industry [3] with a production volume of several million of devices per day [4]. Another emerging commercial application of SAWs is sensor technology. Since the confinement of the acoustic field is in close vicinity of the surface, the amplitude and phase of the SAW is very sensitive to any perturbation on the sample surface. Therefore, SAW devices are applied as mass and chemical sensors [5]. In both areas there is a strong demand for higher operation frequencies, related to higher data transmission speeds, or higher sensitivity.

Even though SAW devices have been in commercial use for more than 60 years, in the field of semiconductor physics and technology there are still new and very exciting potential applications emerging as a consequence of recent advances in material growth and nanofabrication technology. Among them, acoustic charge transport (ACT) devices exploit the interaction between SAWs and charge carriers in low-dimensional systems. In piezoelectric semiconductors, SAWs can be used to modulate the optical and electronic properties by means of the associated lattice deformation and the respective piezoelectric field. Displacement and piezoelectric field carried by the

SAW interacts with the material by inducing a periodic modulation of the mechanical, optical and electronic properties of the semiconductor.

The SAW-induced charge transport device concept was demonstrated on silicon by Gaalema *et al.* and proposed to use SAW in charge coupled devices (CCDs) [6]. They used the fact that the piezoelectric field which accompanies the SAW extends beyond the surface and can interact with electrons in the adjacent semiconductor. In their work, SAWs were generated by an interdigital transducer on a LiNbO₃ substrate. A silicon wafer with SiO₂ layer was separated from a LiNbO₃ by a narrow air gap. They observed the synchronous drag of minority carriers by the electric field associated with the SAW. After demonstration of SAW-induced electron transport in this LiNbO₃/p-Si separated medium structure, Tsubouchi *et al.* showed minority charge transport in a monolithic Al/ZnO/SiO₂/p-Si structure [7]. Hoskins *et al.* focused on multilayer epitaxial GaAs structures to take advantage of the large optical absorption coefficient and high speed capabilities of GaAs relative to Si [8]. Their ACT device utilized the weakly piezoelectric nature of GaAs. SAWs were generated on a (100) GaAs substrate and electrons were transported by a SAW-induced piezoelectric field in the $\langle 110 \rangle$ direction in a buried channel formed by depleting an n-type epitaxial layer on a p-type or semi-insulating substrate. Tanski *et al.* used a new approach for the fabrication of a buried channel ACT device in which the electron charge packages are confined (in the direction normal to the substrate surface) in a GaAs potential well channel formed between two layers of (Al,Ga)As [9]. In this approach they eliminated the need of electrodes or back gating to form a charge transfer channel. SAWs have also been used to probe two dimensional electron gas (2DEG) systems because of their sensitivity to the conductivity in 2DEG. Wixforth *et al.* used SAW damping and velocity measurements as a sensitive probe of changes in the magneto-conductivity in the hall bar geometry [10, 11]. In 1995, Shilton *et al.* extended the 2DEG transport studies by measuring magnetic field dependence of acoustoelectric current instead of SAW damping or velocity changes [12, 13]. One year later, the first observation of SAW-induced current through a quantum point contact (QPC) was reported by Shilton *et al.* [14]. The QPC was electrostatically defined in a 2DEG by using split gate electrodes. They observed oscillatory acoustic current as a function of the gate voltage in the open channel regime. This was attributed to the interaction of the SAW and the slow-velocity electrons, which are mostly settled in the upper sub band of the QPC and close to the Fermi energy level. More interestingly, in the following works by the same group, they have also observed quantized current plateaus in units of

Figure 1.1: (a) The SAW-induced acoustoelectric current through a QPC obtained by changing split gate voltage for different values of SAW power. The SAW frequency is 2728.4 MHz. (b) Representation of the mechanism believed to be responsible for the plateau in acoustoelectric current. Dark regions show 2DEG reservoirs. Shaded regions show split gate metallization. Lines schematically show the distribution of the electrostatic potential. The electrostatic potential due to the SAW forms a succession of quantum dots in the channel, which move through with the SAW velocity v_{SAW} . (c) The electrons are captured from the 2DEG by the potential minima. Reproduced from Ref. [15].

$I = ef$ from the current measurements as a function of gate voltage beyond the pinch off voltage of QPC (see Fig. 1.1) [15, 16]. To explain their observation, they proposed a simple model in which electrons captured by the SAW reside in the minima of moving quantum wells which are created by the lateral confinement by the split gates in addition to the confinement in the propagation direction by the SAW. Split gates create a potential barrier along the QPC beyond the pinch off voltage which prohibits the current flow induced by the bias voltage. However, SAWs with sufficiently high amplitude can drag electrons through the barrier and transport them across the QPC by modulating the total electrostatic potential inside the channel. The number of electrons transported in one minimum of the SAW is determined by the Coulomb charging energy of the moving quantum wells. This physical system was proposed as a means of current standardization because of availability of measuring the current which is created by a certain number of electrons at a certain frequency. Since the frequency (time) can be determined very precisely, electron charge can be also calculated with a very high precision by dividing the measured current by frequency. In the following works by Cunningham *et al.* the increment of the precision of the current quantization was studied by eliminating noise sources and using counterpropagating SAW beams with a fine phase adjustment [17]. Additionally, a shallow etched QPC which allows precise control over the shape of the constriction was studied by the

Figure 1.2: (a) Photoluminescence spectra of an InGaAs/GaAs quantum well structure for different acoustic powers. The insets schematically depict the sample design with two IDTs and the storage of optically generated excitons in the potential of a SAW. (b) Ambipolar transport of trapped charges by a SAW. Rf pulse applied to IDT1 generates a SAW packet. Photogenerated electron-hole pairs are transported with sound velocity to a semi-transparent metal. Here the deliberate screening of the piezoelectric potential modulation occurs and radiative recombination of carriers is induced. Reproduced from Ref. [19].

same group [18].

In addition to the electron transport experiments mentioned above, SAWs have been intensively used to transport electron hole pairs in semiconductors. The first experimental demonstration of electron-hole pair transport in a GaAs heterostructure was performed by Rocke *et al.* in 1997 [19]. They used an IDT operating at 840 MHz on a GaAs (100) sample including a 10 nm thick undoped quantum well formed by InGaAs. Excitation of electron-hole pairs in the quantum well was achieved by laser illumination. As SAWs induce band edge modulation in semiconductors due to the accompanying piezoelectric field, optically excited electron-hole pairs were dissociated and spatially separated, increasing the life time of carriers. These photo-excited carriers were also confined in potential wells and carried by the SAW at sound velocity. Moreover, they showed that after transportation over macroscopic distances, a deliberate screening of the lateral piezoelectric SAW field leads to radiative recombination at a remote location from where the electron-hole pairs were generated (see Fig. 1.2). Santos *et al.* also investigated SAWs in detail. They demonstrated a contactless and spatially resolved method for mapping SAW-induced electric fields, and

the longitudinal and transverse components of the SAW particle displacement vector [20, 21]. They used microscopic photoluminescence (PL) measurements for electric field mapping. The high PL sensitivity to electric field enabled sampling of the field distribution in SAW semiconductor structures. SAW-induced reflection anisotropy was used to determine the longitudinal component of the displacement field, and the reflection modulation was used to collect information from both the longitudinal and transversal components. In later studies of the same group, they investigated both type I and type II modulation of band structures and electron-hole pair transport using spatially and time resolved PL measurements in semiconductors [22–25]. Type I modulation which is much weaker than type II modulation is the deformation potential modulation of the band caused by the local variations and symmetry of the crystal lattice induced by the strain field. This type of modulation induces a variation of the band gap between the minimum and the maximum in the regions of the maximum tension and compression, respectively. On the other hand, type II modulation is related to the piezoelectric potential created by the strain in crystals without a center of inversion. Dynamic dots (DDs) which consist of confined and mobile potentials by the interference of orthogonal SAW beams in GaAs QWs was also illustrated by Alsina *et al.* [26]. In a following study by Lima *et al.* [27], orthogonal SAW beams were used to control the propagation direction of the carriers at room temperature and they showed that approximately 70 % of the carriers were switched to a second SAW beam.

In addition to the SAW-induced carrier transport in two dimensional systems, one-dimensional isolated systems have also been investigated for electron transport [28, 29]. In these experiments, nanowires (NWs) or carbon nanotubes (CNTs) were placed on top of a LiNb_3 substrate. They used the fact that the SAW-induced piezoelectric field can extend approximately one wavelength above the surface. Kinzel *et al.* demonstrated SAW controlled modulation of the optical emission of single GaAs NWs [31]. In a following study, Hernandez-Minguez *et al.* demonstrated electron-hole pair transport and spatial control of their recombination in GaAs-based nanowires by using SAWs [30]. Their time-resolved PL measurements on GaAs NWs capped with an (In,Ga)As segment showed that their system can be used as a non-clasical photon source.

Except the charge carrier manipulation, SAWs have some recent applications such as micro object manipulation (micro particles, droplets, etc.) [32, 33]. Another

growing interest for SAW applications is phononic crystals (PCs) in which the density and the elastic constants are periodically changed. Therefore, it enables complete acoustic stop bands as an analogy to the photonic band gaps for electromagnetic waves in photonic crystals and may find promising applications, such as acoustic wave guiding or filtering [34, 35].

1.2 Surface Acoustic Waves in Semiconductors

Surface acoustic waves (Rayleigh waves) are a special form of acoustic waves that are guided by a material surface. These waves are polarized in the sagittal plane (x - y plane) with surface normal along z , and they propagate in the x direction (according to the reference system given in Fig. 1.3). Since the displacement and velocity components are in the x and y directions, they do not couple to the transverse waves which have a z component, perpendicular to the sagittal plane.

The mathematical description of surface acoustic waves is based on linear acoustic theory. To obtain the equation of motion for elastic deformations of a solid, we can consider an elemental volume of an elastic solid (see Fig. 1.3). Here, the stress applied in the x direction of each face is indicated. The force applied on each face is the product of the stress component and area (A). The summation of the forces acting in the x direction is thus

Figure 1.3: Stress tensor components (left) and x -directed forces (right) acting on an elemental volume of an elastic solid.

$$F_1 = [(T_{11} + \Delta T_{11})A_1 - T_{11}A_1] + [(T_{12} + \Delta T_{12})A_2 - T_{12}A_2] + [(T_{13} + \Delta T_{13})A_3 - T_{13}A_3]. \quad (1.1)$$

By using Newton's second law, one can obtain

$$\Delta T_{11}\Delta y\Delta x + T_{12}\Delta x\Delta z + T_{13}\Delta x\Delta y = \rho\Delta x\Delta y\Delta z \frac{\partial^2 u_1}{\partial t^2}. \quad (1.2)$$

Dividing Eq. (1.2) by the volume element $\Delta x\Delta y\Delta z$ and taking the limit at zero results in a partial differential equation which can be generalized to all three coordinates, one obtains

$$\sum_{j=1}^3 \frac{\partial T_{ij}}{\partial x_j} = \rho \frac{\partial^2 u_i}{\partial t^2}. \quad (1.3)$$

This is the equation of motion, relating the stress gradient to the displacement.

According to Hooke's law, for small deformations, the strain in a material is linearly proportional to the applied stress. This can be generalized to three dimensions, and a constitutive relation which relates stress to strain, can be obtained as

$$T_{ij} = \sum_{k,l=1}^3 c_{ijkl} S_{kl}. \quad (1.4)$$

Here c_{ijkl} is the elastic stiffness constant tensor and S_{kl} is the strain tensor, defined as

$$S_{kl} = \frac{1}{2} \left(\frac{\partial u_k}{\partial x_l} + \frac{\partial u_l}{\partial x_k} \right). \quad (1.5)$$

By using the equation of motion and the elastic constitutive equation, one can derive the wave equation for a plane acoustic wave propagating in a non-piezoelectric solid. Differentiation of Eq. (1.5) with respect to x_j with taking into account the symmetry of the strain matrix gives

$$\sum_{j=1}^3 \frac{\partial T_{ij}}{\partial x_j} = \sum_{j,k,l=1}^3 c_{ijkl} \frac{\partial^2 u_k}{\partial x_j \partial x_l}. \quad (1.6)$$

By equating the right hand side of Eq. (1.6) and Eq. (1.3) **the wave equation for non-piezoelectric elastic solids** can be obtained

$$\rho \frac{\partial^2 u_i}{\partial t^2} = \sum_{j,k,l=1}^3 c_{ijkl} \frac{\partial^2 u_k}{\partial x_j \partial x_l}. \quad (1.7)$$

In case of a piezoelectric crystal as the propagation medium, the equations describing the acoustic wave propagation are slightly modified. The coupling between the electric field and the strain induces a modification of the elastic and electromagnetic constitutive relations:

$$T_{ij} = c_{ijkl}^E S_{kl} - e_{kij} E_k, \quad (1.8)$$

$$D_i = \epsilon^S E_j + e_{ikl} S_{kl}. \quad (1.9)$$

Here $\mathbf{e}$ is the piezoelectric tensor, $\mathbf{E}$ the electric field, $\mathbf{D}$ is the electrical displacement field, and ϵ is the static dielectric tensor. These equations are known as the piezoelectric constitutive relations, which relate the stress, strain and electric field in a piezoelectric solid. An electric field wave now accompanies the elastic wave, and the wave velocity depends upon the elastic, piezoelectric, and dielectric properties.

The wave equation for piezoelectric media can be derived in a similar manner. The piezoelectric wave equation is typically written in terms of the displacement u and the electric potential. By taking into account that $E_k = -\partial\phi/\partial x_k$ one can write Eq. (1.8) as

$$T_{ij} = \sum_{k,l=1}^3 c_{ijkl} \frac{\partial u_l}{\partial x_k} + \sum_{k=1}^3 e_{ijk} \frac{\partial \phi}{\partial x_k}. \quad (1.10)$$

Substitution of equation of motion (1.3) into (1.8) yields

$$\sum_{j,k,l=1}^3 c_{ijkl} \frac{\partial^2 u_l}{\partial x_k \partial x_j} + \sum_{j,k=1}^3 e_{ijk} \frac{\partial^2 \phi}{\partial x_k \partial x_j} = \rho \frac{\partial^2 u_i}{\partial t^2}. \quad (1.11)$$

If equation (1.11) is compared with the wave equation in non-piezoelectric media (1.7), the presence of an additional term involving the electric potential ϕ is apparent. This term is responsible for the acoustic wave generation by applying a time varying electrical potential to the medium.

In general, there are three independent elastic modes for a given wave vector k with angular frequency $\omega_i = v_i k$ ($i = 1, 2, 3$) and phase velocity (v_i) specified by

the ratio $v_i = \sqrt{c_i/\rho}$, where c_i is a linear combination of the elastic and piezoelectric constants of the material. The medium behaves as if it were stiffer because of the higher elastic stiffness constant. This is known as *piezoelectric stiffening*.

In isotropic materials, elastic vibrations can be classified according to particle displacement as longitudinal (LA) and transverse (TA) acoustic modes. Specific solutions of the wave equation satisfying the mechanical and electrical boundary conditions give rise to surface modes. These boundary conditions are imposed by the continuity of the strain and electric displacement vectors across the interfaces if a surface acoustic wave propagates in a medium composed of layers parallel to the surface. If the coordinate system is defined by the unit vectors x , y and z (where x is parallel to the propagation direction of the acoustic wave and z is perpendicular to the surface, and the medium supporting the acoustic wave is assumed to fill the half-space $z < z_{\text{surf}}$), the boundary conditions enforcing the continuity of the stress and electric displacement at the interface $z = z_{\text{int}}$ are given by

$$\begin{aligned} T_{iz}(z_{\text{int}}^+) &= T_{iz}(z_{\text{int}}^-), \quad i = x, y, z \\ D_z(z_{\text{int}}^+) &= D_z(z_{\text{int}}^-). \end{aligned} \quad (1.12)$$

Figure 1.4: (a) Displacement of the lattice points under the SAW field (arrows indicate the rotation direction). (b) transverse (u_z) and longitudinal (u_x) components of the displacement field $\mathbf{u}$ in the depth direction of the medium (inset: 2D image of vertical displacement field).

In case the upper layer corresponds to the vacuum, $T_{iz}(z_{\text{int}}^-)$ becomes zero.

Solutions of the wave equation which decay into the material and fulfill the boundary conditions are localized near the surface. These solutions are generally a combination of two or three bulk modes. The Rayleigh mode consists of a superposition of longitudinal (u_x) and transverse (u_z) polarized displacement components with a displacement field $\mathbf{u} = (u_x, 0, u_z)$. Displacement of the lattice points and the depth dependence of the different components of the Rayleigh mode can be seen in Fig. 1.4.

1.2.1 Interaction of Surface Acoustic Waves with Charge Carriers

Mobile carriers in a conducting layer can strongly interact with the SAW piezoelectric field. This interaction causes a dynamical screening of the electric field associated with the SAW. This screening can be utilized to quantitatively determine the electromechanical coupling coefficient K^2 of a certain material. The ratio between the SAW velocities measured in the presence and absence of a very thin metallic film on the surface gives the energy conversion efficiency in the system. The electromechanical coupling coefficient is given by

$$K^2 = 2(v' - v)/v = 2\Delta v/v. \quad (1.13)$$

Here, v' and v are the SAW velocities in case of a perfectly screened surface, by means of a thin metal layer, and a free surface, respectively. However, here, the situation is described for a highly conductive metal layer which is deposited on top of the surface and the screening of the SAW piezoelectric field is supposed to be perfect.

In case of intermediate conductivity, the interaction of mobile carriers with SAWs was investigated by Ingebrigtsen [36]. In this work, a system composed of a semiconductor layer on top of an insulating, piezoelectric substrate was used. Assuming a semiconductor film thickness of a , which is small compared to the acoustic wavelength, a relation which gives the attenuation per radian was obtained as

$$\frac{\alpha}{k} \simeq -\frac{1}{2}K^2\epsilon_1 \frac{\eta\omega_c a/v}{(\epsilon_1 + \epsilon_2)^2\eta^2 + (\omega_c a/v)^2}. \quad (1.14)$$

Here, α is the attenuation constant, k the wave number, a the finite film thickness,

$\omega_c = \sigma/\epsilon_s$ (dielectric relaxation frequency), $v_0 = \mu E_0$ (carrier drift velocity), $\eta = 1 - v_0/v$, $\epsilon_1 = \epsilon_p/\epsilon_s$, $\epsilon_2 = \epsilon_0/\epsilon_s$. ϵ_0 , ϵ_s and ϵ_p are the permittivity of the free space, the semiconductor and the piezoelectric layer, respectively. According to this relation, when the semiconductor is thin compared to the acoustic wavelength, the maximum attenuation per radian occurs when

$$\omega_c a/v \simeq \eta(\epsilon_1 + \epsilon_2). \quad (1.15)$$

This relation indicates that for thin films ($ka \ll 1$), the optimum conductivity is independent of frequency.

Wixforth *et al.* used this result to investigate a system in which the SAW is propagating on a semi-insulating piezoelectric semiconductor such as GaAs, having near its surface a quasi-two-dimensional electron system [11]. The formulation describing the attenuation per length and the relative change in velocity for the interaction of SAWs with a two-dimensional electron system was given by

$$\Gamma = k \frac{K_{eff}^2}{2} \frac{\sigma_{\square}/\sigma_M}{1 + (\sigma_{\square}/\sigma_M)^2}, \quad (1.16)$$

$$\frac{\Delta v}{v_0} = \frac{K_{eff}^2}{2} \frac{1}{1 + (\sigma_{\square}/\sigma_M)^2}. \quad (1.17)$$

Here, the sheet conductivity is $\sigma_{\square} = \omega_c(\epsilon_1 + \epsilon_2/k)$. For frequencies $\omega \ll 1/\tau_t$, where τ_t

Figure 1.5: Attenuation coefficient Γ per unit wave vector k (left), change in SAW velocity $\Delta v/v_0$ in units of K_{eff}^2 as a function of the sheet conductivity $\sigma_{\square}$ of the 2DES (right). Taken from [11].

is the transport relaxation time in the 2DES, $\sigma_{\square}$ can be regarded as frequency independent. For conductivities $\sigma_{\square} \ll \sigma_M$ the attenuation per unit length is proportional to the conductivity, for $\sigma_{\square} \gg \sigma_M$ it is proportional to $1/\sigma_{\square}$. Also, the velocity shows a steplike change in the vicinity of $\sigma_{\square} = \sigma_M$ (see Fig. 1.5).

1.2.2 Surface Acoustic Wave induced Band Modulation

As previously mentioned, SAWs periodically modify the band edges of the underlying semiconductor medium. There are two main mechanisms that modulate the band structure. The first mechanism is the deformation potential modulation of the band gap (E_g), which is based on the variation in volume and the symmetry of the crystal lattice induced by the SAW strain field. This modulation induces $E_g^{\min}$ and $E_g^{\max}$ points in the band structure, corresponding to maximum tension

Figure 1.6: (a) Strain field and (b) modulation of the conduction band and valence band edges of a semiconductor crystal by a SAW. The piezoelectric potential associated with the strain induces a type II modulation of the band edges. The region of the compression and tension induced by the strain superimposes a small modulation of the band gap (E_g) with maximum and minimum values given by $E_g^{\max}$ and $E_g^{\min}$, respectively. The diagram in (b) also illustrates the spatial separation of photo-generated carriers by the piezoelectric field. Taken from [37].

and compression, respectively (see Fig. 1.6). The second mechanism is based on the piezoelectric potential (Φ_{SAW}) created by the SAW-induced strain field, which induces a type II modulation, as shown in Fig. 1.6.

Type II modulation plays a key role in acousto-electric transport. In case of optical excitation electrons and holes are spatially separated and trapped in the maximum and minimum points of corresponding band edges. This spacial separation reduces the overlap between the wave functions of electrons and holes, giving a rise to the carrier life time, thus increases the carrier transport distance. More elaborate discussion on the band structure modulation by SAW can be found in Ref. [37].

1.2.3 Generation and Detection of Surface Acoustic Waves

Generation of SAWs requires energy transduction from one type of energy to acoustic energy. This can be achieved by using many different methods of transduction; such as mechanical, piezoelectric, optical and thermal transduction. SAWs are most commonly generated by electrical means using the inverse piezoelectric effect. The electrical generation of SAWs can only be applied for piezoelectric materials, and uses comb-shaped interlocking electrode arrays deposited on the surface. Such a configuration of electrodes is called an *interdigital transducer* (IDT). IDTs comprise two sets of periodically arranged metallic electrodes in a plane. The spatial period of these metallic electrodes defines the SAW wavelength (λ_{SAW}). In the simplest type of IDT (the single-finger type), each period contains two fingers with both width and separation equal to $\lambda_{\text{SAW}}/4$ (see Fig. 1.7(a)).

When a radio frequency (RF) signal is applied to such an IDT, an oscillating elastic strain will be produced underneath each pair of electrodes. If the applied voltage has a frequency of $f_{\text{SAW}} = v_{\text{SAW}}/\lambda_{\text{SAW}}$, where v_{SAW} is the SAW velocity of the substrate, and λ_{SAW} is the SAW wavelength, all oscillations underneath each pair will have the same phase and constructively interfere, resulting in a coherent beam of SAWs. Indeed, any stress produced at time t by a pair of fingers travels $\lambda_{\text{SAW}}/2$ during the half-period $T/2$ at speed v_{SAW} . At $t + T/2$, the stress arrives under the neighboring pair of electrodes, where the voltage produces a stress with the same phase. The stress due to the second pair of fingers adds constructively to the first. The frequency which produces a coherent SAW beam is called the *resonance frequency*. If the frequency shifts away from this value, the interference between the elastic

Figure 1.7: (a) Single-finger (SF) IDT design, (b) double-finger (DF) IDT design, (c) floating-electrode unidirectional IDT (FEUDT) design.

vibrations generated by the various pairs of electrodes is not totally constructive, and the resulting signal is reduced. Therefore, increasing the number of electrodes leads to a narrower bandwidth of the IDT frequency response.

For materials with weak piezoelectricity, the generation of strong SAWs require long IDTs (typically of a few hundred λ_{SAW}) with a large number of fingers. However, variation in electrical and mechanical impedance at the surface, due to, e.g., metallic electrodes, gives rise to reflection and scattering into bulk modes whenever an elastic wave encounters a transducer. These losses increase with frequency, as the acoustic field concentrates more closely to the surface. The internal reflections can be minimized using the split-finger configuration shown in Fig. 1.7(b). In this configuration, each IDT finger electrode is split into two, and one period consists of four fingers with a width and separation of $\lambda_{\text{SAW}}/8$. In contrast to the single-finger configuration, waves reflected from two neighboring electrodes have a phase difference of π and the interference is destructive (see Fig. 1.8). While the double finger configuration reduces the acoustic reflections, it requires the electrodes to be half the width to operate the IDT at the same frequency as an IDT with a single-finger configuration.

In addition to the single-finger and double-finger IDT designs, there are some other special electrode configurations to increase the SAW device performance. One of these configurations is the *floating-electrode unidirectional transducer* (FEUDT) (see Fig. 1.7(c)), which enhances the acoustic power flow in the forward direction and enables higher SAW amplitudes [38].

Electrical detection of SAWs can be performed by transforming the SAW into an electrical signal. This can be achieved using a delay line (DL) configuration, which consists of two IDTs facing each other: a generator and receiver. Opposite to SAW generation, a SAW arriving at the receiver IDT induces an electric potential

Figure 1.8: Waves reflected from corresponding edges of two neighboring fingers of width $\lambda/4$, separated by a spacing equal to $\lambda/4$, add constructively because the path difference causes a phase shift of 2π (a). These reflections are suppressed when the number of fingers in a period are doubled, corresponding a finger width and spacing equal to $\lambda/8$ (b). In this case, the reflected waves has a phase shift of π .

on the finger electrodes. Although one can detect the SAW with this method, it is more suitable to get the frequency response of the IDT or characterize acoustic and piezoelectric properties of the medium, such as S-parameters, SAW velocity, attenuation or electromechanical coupling coefficient. There are other techniques to detect the acoustic and the electric components of the SAW field, such as optical reflection and transmission [39, 40], interferometry [41–43], and photoluminescence [44]. These techniques have a lateral resolution down to less than $1\mu\text{m}$. In addition, acoustic displacements can be resolved with very high lateral resolution using scanning acoustic force microscope (SAFM) techniques [45].

1.3 Outline of the Thesis

This thesis focuses on the generation of high frequency SAWs for acousto-electric transport in semiconductors using electrical excitation method.

Chapter 2 gives an introduction to the experimental methods that have been used in the course of this research. Nanoimprint lithography, reactive ion etching and metal film formation are described in the device fabrication techniques. SAW device analysis and transport measurements are described in the characterization methods.

In Chapter 3, a method to fabricate sub-100 nm structures by using a novel planarization technique and appropriate material configuration is introduced. This chapter specifically addresses the lift-off issue and critical dimension control at nanometer scale.

Chapter 4 focuses on excitation of high frequency SAWs on a ZnO/SiO₂/Si multilayer structure. This layered structure has an advantage to excite the higher order Rayleigh modes in addition to the fundamental mode, thus enabling higher resonance frequencies. Confirmation of the experimental observations was performed by using finite element analysis method.

Since the interaction of charge carriers with SAWs causes an attenuation of SAW-induced piezoelectric field, substrate properties becomes more important for acousto-electric transport. Additionally, integration of the real devices to the recent electronics requires more careful material design. Chapter 5 addresses these issues and discusses a Si-based multilayer structure with a high resistive substrate.

Chapter 6 demonstrates high frequency acousto-electric transport in GaAs-based nanowires. SAW-induced piezoelectric field created in LiNbO₃ substrate penetrates above the surface and allows the trapping of photogenerated electrons and holes at the spatially separated minima and maxima of the conduction and the valance bands of the nanowire placed on LiNbO₃ surface. Small SAW wavelength enables studying in a regime at which SAW wavelength is smaller than the transport distance in contrast to the previous reports in the literature. Carrier transport is verified by using GaAs/AlGaAs core/shell nanowires which have a segment containing (In,Ga)As, which enables intentional recombination of the acoustically transported carriers at a remote position.

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Chapter 2

Experimental Methods

In this chapter, the experimental methods that have been used in the course of this research are introduced. The first section focuses on the fabrication methods and describes the nanoimprint lithography process. An adequate description of the etching and film formation techniques are also given. In the second half of the chapter, the characterization techniques are introduced, such as network analysis and the optical setup for the electron-hole pair transport measurements.

2.1 Device Fabrication Techniques

Increasing the lithography resolution is a straightforward strategy to fabricate high-frequency SAW devices, because the spatial periodicity of the finger electrodes determines the IDT frequency. However, resolution of conventional optical lithography is limited by diffraction, which is inherent to the wave nature of light. Therefore, nanoimprint lithography (NIL) is a good candidate for SAW device fabrication because of its high resolution and low cost capability. Additionally, precise pattern definition by NIL is the main advantage of the process. NIL has been introduced by Chou *at al.* in 1996 as an alternative to optical lithography and to new generation lithography techniques such as electron beam lithography, X-ray lithography and scanning proximal probe lithography techniques [1]. The working principle of NIL is based on the mechanical deformation of a polymer-coating material on a substrate for replication of the desired pattern. Although different alternatives of the method have appeared after its invention, there are mainly two important NIL methods available: *Hot embossing nanoimprint lithography* and *UV-based nanoimprint lithography*. While both methods use a template (also called “mold”) which is already patterned at nano-scale (or/and micro-scale) by using any lithography method, they differ from each other in terms of operation conditions and materials used, as explained below.

Although NIL enables the definition of patterns at sub-micron scale, further processing is required to get the final device structure. One of the most critical steps is reactive ion etching (RIE). RIE is generally necessary to remove the residual layer which remains on the surface after the imprint. It can also be used to remove other resist or metallic layers underneath the NIL resist according to the material composition used in the device fabrication process. Another technique which was used for our device fabrication is metallization: evaporation of a metal target and condensation of the evaporated atoms of the target material on top of a relatively cold wafer surface.

Below these fabrication techniques will be discussed in a detailed and specific way for our device fabrication process.

2.1.1 Hot Embossing Nanoimprint Lithography

Hot Embossing NIL or thermal NIL (T-NIL) is the earlier NIL technique. As its name suggests, the process includes a thermal treatment step. A thermoplastic polymer material (NIL resist) is spin coated on a substrate to form a thin film layer. Subsequently, a previously patterned template is brought in contact with the substrate under a certain pressure. While the pressure is applied, the system is gradually heated up. When the temperature reaches above the glass transition temperature of the NIL resist, the resist starts melting and fills the cavities in the template. Then, the system is cooled down and the template is removed by leaving a replication of the pattern. After removing the template, a residual layer exists on the surface which is generally removed by using a further etching process such as RIE.

2.1.2 UV-based Nanoimprint Lithography

UV-based NIL (UV-NIL) uses a UV-curable polymer material as a resist instead of a thermoplastic material [2]. The template is made of a transparent material. After

Figure 2.1: (Types of nanoimprint lithography: thermal (hot embossing) nanoimprint (left) and UV-based nanoimprint (right)).

the template and the resist-coated substrate are pressed together and the cavities are completely filled by the resist, the resist is cured by UV light and then the resist becomes solid. After removing the template, a replication of the pattern and a very thin residual layer remains on the surface (see Fig. 2.1).

The main difference between UV-NIL and T-NIL is the resist material, which is low in viscosity (liquid) at room temperature in case of UV-NIL. The liquid nature of the resist in UV-NIL is the main advantage, because it can be shaped easily by applying a low pressure without need of heating, thus enabling room temperature operation.

The UV-NIL process can be divided into two main categories. The first one is a single-step process in which one imprinting process is performed at a time by using a substrate-size or smaller template. In the second type, a chip-sized template is used in a step and repeat process. This method has been introduced by Willson's group and called step-and-flash nanoimprint lithography (SFIL) [3]. The method chosen for the IDT fabrication in this work is the SFIL technique. As mentioned before, the residual layer on the surface needs to be removed for further pattern transfer down to substrate. As the IDT fabrication process requires metallization and lift off steps, an appropriate side wall profile (under-cut) of the patterned resist is also essential. However, it is a challenging task to get an appropriate under-cut profile in case of a single layer, or a double layer resist with similar RIE etch rates. Another concern regarding the final device structure, is the critical dimension (CD) control. When the residual layer is removed in plasma etching, in addition to the vertical etching, there will be lateral etching resulting shrinkage of the defined features. To overcome these obstacles, we have used a process which involves some extra steps in addition to the standard SFIL process [4, 5].

In our work, we have used an ImprioTM 55 tool from Molecular Imprints, Inc.. The IDT fabrication process starts with coating the substrate with a transfer layer (a thin organic resist or bottom antireflective coating), which will be removed at the end of the process in a lift-off solution. The NIL resist droplets are dispensed on top of the transfer layer and the transparent imprint mold is pressed on top of the surface with a low force (a few newton). Because of the applied force and low viscosity, the NIL resist completely fills the trenches in the mold. Then the NIL resist is hardened by exposure with UV light through the transparent mold. When the mold is removed, the replication of the mold structures is completed. This pattern definition process cycle is

Figure 2.2: Step and flash nanoimprint lithography cycle. The photograph in the center belongs to a 4 inch wafer with 24 imprinted areas using the NIL template designed for our SAW devices.

repeated multiple times on a standard substrate. For the transfer of patterns through the resist layers and appropriate metallization, an intermediate step between pattern definition (SFIL) and pattern transfer (reactive ion etching) is applied. An inorganic polymer layer (hydrogen silsesquioxane (HSQ)) is used to planarize the pre-patterned surface. HSQ offers a high degree of planarization and a very high etching selectivity with the underlying organic layers (imprint resist and transfer layer). After this, a fluorine-based RIE process is used to etch the HSQ layer until exposing the elevated patterns. Then, the NIL resist and the transfer layer are etched down to the substrate by using O_2 plasma etching. The high etch selectivity between the planarization and underlying (transfer and imprint resist) layers enables an appropriate undercut profile, which is essential for the metal liftoff process. Figure 2.2 shows an imprint cycle and an imprinted and HSQ coated 4 inch wafer in the middle.

2.1.3 Reactive Ion Etching

RIE is the most common dry etching method used in the field of micro and nano fabrication. In this method, not only a physical removing mechanism takes place, but also a chemical reaction is used to remove a material from the surface which enables a material selective etching. Chemically reactive species are created by using a gas flow discharge to dissociate and ionize the molecules which are chosen for a specific reaction

according to the material that needs to be removed from the surface. RIE comprises the following processes: generation, DC bias formation, transport of plasma-generated reactive intermediates, adsorption, reaction, desorption and pumpout of volatile reaction products [6]. In the *generation*, the glow discharge mechanism is used to create ions from a suitable feed gas and a gas phase etching environment is created. In this etching environment, neutrals, electrons, photons, radicals, positive and negative ions exist. *DC bias formation* occurs due to the higher mobility of the electrons under the RF electric field compared to the much more massive ions. These electrons cause a negative charge formation on the capacitively coupled electrode and build up a dc self bias voltage. *Transport of plasma-generated reactive intermediates* is the transport of the intermediates to the substrate surface by diffusion and the forcing of the positive ions from the plasma to the substrate surface by means of the dc self-bias, respectively. The *absorption* process describes the absorption of the reactive radicals on the substrate surface. The *reaction* process refers to the chemical reaction between the absorbed species and the material on the substrate surface. *Desorption* is the desorption of the reaction products from the surface. After the desorption takes place, the species diffuse into the plasma and have to be pumped out to prevent a possible plasma induced dissociation of the product molecules and redeposition. This step is called *pumpout of volatile reaction products*.

In Fig. 2.3, the basic elements of a typical RIE tool can be seen. Here a capacitively coupled plasma (CCP) is used to generate the plasma. In addition to the CCP, the inductively coupled plasma (ICP) method can be used in a RIE tool. In this case, an RF voltage is supplied to a coil which is wrapped around the plasma discharge region. A time-varying magnetic field induced by the coil causes the plasma

Figure 2.3: Schematic diagram of the apparatus used for RIE. Redrawn from [7].

to circulate in the plane parallel to the CCP electrode. Relatively fast electrons cause more ion formation by collisions. The combination of CCP and ICP enables the control of the number of ions (via ICP) and the energy of the ions (via CCP) without significant perturbation of each other.

The main process parameters in RIE are ICP power, CCP power, gas flow rates, chamber pressure, distance between the substrate and the plasma source and temperature. All these parameters affect the etching rate and the profile of the etched patterns. In this study, both types of RIE apparatus were used: an electrotech PF 340 and an Adixen AMS100DE apparatus. The etching parameters which were used to etch different materials are given in Appendix B.

2.1.4 Metallization

Metal film formation was performed by using electron beam evaporation (EBE). This technique is commonly used for an appropriate lift-off because of the directional nature of the deposition. Another advantage which makes the EBE a preferred vacuum evaporation technique is the elimination of contamination sources by crucibles, heaters and support materials, because electron beam heats very small region on the evaporation material in contrast to thermal evaporation technique. In EBE, electrons are thermoionically emitted from a filament and accelerated by an electric field. The accelerated electron beam is deflected by using a magnetic field to focus the beam to the evaporation material. The kinetic energy of the electrons is transferred to the evaporation material by heating up the material. The heated material starts to melt and evaporate resulting in vapor formation. Under high vacuum, this vapor causes a film formation by recondensation onto a cooler substrate. In this study, the metallic films were evaporated in a Balzers evaporator (BAK600).

2.2 Characterization Methods

In this section, the electrical and optical techniques used to characterize the devices and to perform experiments will be introduced.

Figure 2.4: Schematic illustration of the signal flow in a two-port network. a_1 and a_2 represents the applied voltages at port 1 and 2, and the output terms b_1 and b_2 represents the voltage signals reflected from port 1 and 2, respectively.

2.2.1 Interdigital Transducer Measurements

The performance of IDTs can be characterized considering a delay line as a network which has two ports: Input (generation IDT) and output (receiver IDT) ports (see Fig. 2.4). This kind of network can be evaluated by using scattering parameters (S-parameters). S-parameters are frequency dependent complex numbers and they relates the traveling waves in a transmission line.

The mathematical relation between the S-parameters and the input/output signals for a two-port network is given by a 2×2 matrix, called the scattering matrix,

$$\begin{pmatrix} b_1 \\ b_2 \end{pmatrix} = \begin{pmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{pmatrix} \begin{pmatrix} a_1 \\ a_2 \end{pmatrix}. \quad (2.1)$$

Here, the input terms (a_1 and a_2) represent the applied voltages at port 1 and 2, and the output terms (b_1 and b_2) represent the voltage signals reflected from port 1 and 2, respectively. The S-parameters are expressed as $S_{<out><in>}$, where $<out>$ and $<in>$ denote the output (port 2) and input (port 1) port numbers of the DL. If the output and input numbers are the same ($i = j$), S_{ii} represents the reflection coefficient, and similarly if the output and input numbers are different, S_{ij} represents the transmission coefficient.

Figure 2.5 illustrates a typical reflection and transmission measurement for IDT devices in a DL. Commonly S-parameters are represented in dB or dBm unit, which are the power ratio of the measured power referenced to a reference power or to one miliwatt (mW), respectively. S_{11} is the input reflection coefficient and S_{21} is the forward transmission coefficient. The power sent into the generator IDT is significantly reflected. However, for the resonance frequency, at which electrical power is converted

into acoustic power, some of the applied power should be transmitted resulting in a reduction of the reflected power. Therefore, the S_{11} curve shows a dip at the resonance frequency, while the S_{21} curve shows a peak at the same frequency, indicating an increase in the transmitted power to the receiver IDT. The electro-acoustic conversion efficiency for an IDT can be measured from the difference between the reflection coefficient at resonance ($S_{11}^{(\text{res})}$) and at non-resonance ($S_{11}^{(\text{no-res})}$) frequencies, and can be written as $\Delta S_{11} = S_{11}^{(\text{no-res})} - S_{11}^{(\text{res})}$. Since the dBm unit is logarithmic, this formula can not be evaluated directly. Conversion from dBm to mW can be done via $P(\text{dBm}) = 10 \log[P(\text{mW})]$. Therefore, ΔS_{11} in mW is given by

$$\Delta S_{11} = 10^{S_{11}^{(\text{no-res})}/10} - 10^{S_{11}^{(\text{res})}/10}. \quad (2.2)$$

In this study, two different network analyzers were used: a “R&S ZVB2”(Rohde-Schwarz) with a frequency range from 10 MHz to 20 GHz, and a “N5244A PNA-X” (Agilent) with a frequency range from 10 MHz to 43.5 GHz and time domain measurement option. Time domain measurements were performed to suppress the direct electromagnetic coupling between the generator and receiver IDTs. Figure 2.6 shows an example of a frequency and time domain measurement of a device fabricated on a LiNbO_3 substrate. More detailed explanation of the time domain measurement can be found in chapter 4. Two-port network analyzer measurements have been performed by using microwave probes (Model 40A from GGB industries Inc.) with ground-signal (G-S) and signal-ground (S-G) configuration. To minimize measurement errors, first a

Figure 2.5: An example of S_{11} (reflection) and S_{21} (transmission) measurements for an IDT delay line with a wavelength of 400 nm fabricated on a LiNbO_3 substrate.

Figure 2.6: a) Frequency domain transmission spectrum, (b) time domain reflection spectrum, (c) layout of delay line, and (d) reflection spectrum obtained after eliminating (shorting) the electromagnetic signal.

calibration procedure was performed before the network measurements on the actual devices. A calibration substrate (CS-8 from GGB industries Inc.) was used to apply a “SOLT” (short, open, load, through) calibration technique.

2.2.2 Photoluminescence Measurements

The electronic dispersion relation in semiconductors breaks into bands, forming a forbidden energy gap between the bands (the band gap) as a result of the periodic potential subjected to the electrons. Therefore, the optical properties of semiconductors are determined by the transitions between energy bands rather than between discrete energy levels. Figure 2.7 shows an inter-band transition.

As illustrated in Fig. 2.7(a), in the absorption process, an electron is moved from the valance band to the conduction band by means of the photon energy leaving behind a hole in the valance band. This transition can take place for eneries larger than the band gap. Therefore, the absorption process has a threshold energy given

by the band gap.

Indeed, interband luminescence is more complicated than the illustration in Fig. 2.7. For the emission to take place, there must be an electron in the conduction band and a hole (an unoccupied state) in the valence band. Injection of the electrons and holes can be achieved by a few different methods, electrically as in a light-emitting diode (LED), or by optical excitation. In case of optical excitation, the method is called *photoluminescence*. Because the excited electrons and holes relax very rapidly (thermalize) to their respective band edges at which they recombine, the radiative transition energy is generally very close to the band gap energy E_g . In this respect, photoluminescence can be described as a process which has three steps: excitation, thermalization and recombination.

In some semiconductors (such as GaAs, GaN), the conduction band minimum and valance band maximum are located at the same k value in the band diagram (direct band gap). For these semiconductors, radiative recombination of electron-holes pairs can occur without requiring an interaction with a phonon. However, in some semiconductors (such as Si, Ge), the band edges are not located at the same k value (indirect band gap), and the carriers can not recombine without interacting with a phonon to conserve momentum.

Figure 2.8 illustrates the PL measurement setup. For these experiments, the sample was mounted in a liquid helium flow cryostat that has an optical window to let the laser beam into the chamber. A laser source with a wavelength of 756 nm was modulated with an optical chopper. The laser beam was focused onto the sample by

Figure 2.7: Inter-band transitions in a semiconductor: (a) absorption and (b) emission. E_g is the band gap energy. Redrawn from [8].

Figure 2.8: Illustration of the optical measurement setup: The sample was mounted in a microscope cryostat for spatially resolved PL measurements at 20 K. A laser beam with a wavelength of 756 nm was used as an excitation source. The laser beam was focused onto the sample by using a 50 $\times$ objective. The emitted PL was collected by the same objective, imaged on the entrance slit of a spectrometer, and detected with both spatial and spectroscopic resolution by a cooled charge-coupled device (CCD) camera.

using a 50 $\times$ objective. In some cases, to be able to get a smaller beam size, a 100 μm pinhole was placed in front of the objective. The Emitted PL was collected by the same objective and imaged by means of a spectrometer and a cooled charge-coupled device (CCD) camera. During the acousto-electric transport measurements a laser beam was applied and the PL emission was collected under an RF signal, which was applied to the IDT to excite the SAWs by using the RF coaxial connections.

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Chapter 3

Developing a Fabrication Process for Sub-100 nm IDT Finger Electrodes

This chapter presents a novel global contact planarization technique based on the spin-on-glass material HSQ and demonstrates its excellent performance on patterns of 70 nm up to several microns generated by UV-NIL. The HSQ layer (~165 nm) is spin coated on the imprinted organic layer and planarized by pressing it with a flat wafer at room temperature. Before retracting the planarization wafer, the HSQ is hardened by baking at 120 or 70 °C, depending on the underlying material. Fluorine-based RIE is used to etch the HSQ (etch-back) down to the top of the features in the organic imprint layer. Subsequently, oxygen-based RIE is used to etch the organic imprint layer in the exposed regions, thereby transferring the imprinted pattern down to the substrate. The etch selectivity between the HSQ and the underlying layers is found to be more than 1:100, enabling very accurate pattern transfer with excellent critical dimension control and well-defined undercut profile suitable for further metal lift-off processes. The dependence of the contact planarization quality on the HSQ spinning speed and pressure is investigated, achieving a global planarization degree as good as 93 %, an improvement of 45 % compared to standard spin coating planarization.

3.1 Introduction

In microfabrication and nanofabrication, sometimes global (wafer-scale) planarization is required, while in other cases only partial or local planarization is sufficient. Spin-on-glass (SOG) is a widely used planarization material in semiconductors. SOG materials are siloxanes or silicates mixed in alcohol-based solvents, and exhibit a good planarization capability [1]. However, the degree of planarization (DOP) decreases with increasing spacing between features [2], and subsequent pattern transfer by RIE is also limited by the design and spacing between features. In some cases, such as in multi-step fabrication processes, high-quality planarization is also crucial and simple application of a SOG gives insufficient global planarization. Although there exist some improved SOG-based planarization techniques, they generally require more than one SOG layer or additional layers of different materials [3], or additional etch processes [4].

In this work, high-quality global contact planarization with only one layer of SOG was realized for parallel reproduction of patterns with dimensions in the micrometer and nanometer ranges. Contact planarization [5] was obtained by applying force at room temperature onto the liquid planarization material via an optically flat template in a manner similar to hot embossing lithography. The planarization material is hardened via a thermal curing process and leaves a flat surface on the wafer after retraction of the planarization wafer. Importantly, this method is capable of achieving both excellent local and global planarization. HSQ was chosen as SOG material because of its room-temperature deformation capability. Although HSQ is ordinarily used as a negative-tone electron-beam lithography (EBL) resist, experimental investigations showed that it can also be applied as a nanoimprint resist at room temperature [6, 7]. Excellent etch resistance of HSQ under oxygen-based reactive ion etching is another advantage for pattern transfer. Critical dimension loss during etching of the organic residual layer that remains after UV-based nanoimprint lithography can be prevented by using HSQ. As an etch mask, the thickness of the HSQ layer all over the substrate is critical. The specific aim of the planarization in this work was to reduce thickness variations in the HSQ layer on patterned and non-patterned regions over the entire substrate.

In the literature there are some examples of contact planarization of patterned surfaces [8, 9]. Sun et al. used polymethylmethacrylate (PMMA) and novolak resin

materials to planarize a surface, where in contrast to our HSQ planarization method; they used relatively high temperature (175 °C) and pressure (up to 50 bar) to force the planarization materials to flow over the substrate [8]. A different method offered planarization at room temperature of pre-patterned surfaces using a UV-based imprint process (SFIL) and flat templates [9]. In the latter study, the planarization on wafer scale is achieved in a step-and-repeat mode, where in every step a single imprint field is planarized. This die-by-die planarization method offers good results, however on a wafer scale it is very slow and critical for contaminations. In our case, HSQ enables a fast room-temperature planarization process, and its high etch selectivity with respect to the underlying organic resists makes it a favorable material for planarization and dimension control.

Even though in this chapter features are defined by UV-based nanoimprint lithography, it should be noted that this planarization method is very generic and can be also applied to any other structures defined by other lithography techniques or etching processes.

3.2 Experimental Details

Pattern fabrication on Si wafers was carried out using step-and-flash imprint lithography (S-FILTM) on an ImprioTM 55 tool from Molecular Imprints, Inc. To obtain high-aspect-ratio features, 100 mm double-sided polished (DSP) Si wafers were first spin coated with a transfer layer and hot baked (see Fig.3.1(a)). DUV-30J from Brewer Science, Inc., normally applied as organic bottom antireflective coating (BARC), or PMMA was used as transfer layer because of their low etching resistance compared to HSQ under oxygen plasma, and solubility of the PMMA in standard solvents (e.g. acetone) for potential lift-off applications. A 2wt % PMMA (Mw 350 kD) in toluene was prepared for our experiments. The PMMA itself was purchased from Sigma Aldrich, Inc. DUV-30J and PMMA were spin coated at 3000 rpm and baked on a hot template at 150 °C and 120 °C, respectively. The features were imprinted on top of the transfer layer into a low-viscosity, organic, UV-curable material at room temperature and under low force (2-3 N). As an imprint material MonomatTM (from Molecular Imprints, Inc.) was used. This imprint material was deposited by field-to-field dispensing and cured through the transparent imprint template using broad-band UV-light (exposure dose 80 mJ/cm², 230-360 nm wave length) (see Fig.3.1(b) and (c)).

Figure 3.1: UV-based nanoimprint lithography: (a) first wafers were coated with an organic transfer layer (e.g. PMMA), (b) a UV-curable, organic imprint material was dispensed on the surface locally adjusting the volume, (c) force was applied with a transparent template, and the imprint material was exposed to UV light, (d) after removing the template, imprinted features were obtained on the surface.

After S-FIL, the residual imprint material and transfer layer remained on the surface (see Fig.3.1(d)), and for further pattern transfer they need to be etched. A schematic overview of the process is given in Fig. 3.2.

The imprint templates (molds) used for the experiments were fabricated by standard EBL and RIE techniques with an 25 mm x 25 mm active area (imprint area), consisting of different test features such as line and spaces, dots, or holes with dimensions from 60 nm up to 500 nm and pitch sizes varying from 1:1 up to 1:5.

HSQ, XR-1541 6 % with carrier solvent methhyllisobutylketone, from Dow Corning, Inc., was deposited on top of the imprinted layer by spin-coating (see Fig.3.2(b)). In contrast to our work, if a thermoplastic planarization material is used, the planarization process has to be performed at higher temperature than the glass transition temperature (T_g) to make the material less viscous. However, in our case, heating does not make HSQ softer, but instead makes it harder to be deformed under pressure. Therefore, planarization was performed at room temperature (RT) and HSQ was not thermally treated after spin coating. For the contact planarization, a blank 100 mm DSP Si wafer was used as flat template (see Fig.3.2(c) and (d)). Prior to planarization, the flat Si template was treated with an anti-sticking release layer to facilitate an easy separation of the template from the HSQ layer. 1H,1H,2H,2H- perfluorodecyltrichlo silane was deposited on the flat Si template via gas-phase evaporation method under vacuum [10]. A hot embossing NIL tool (from Obducat) was used to press the flat

Figure 3.2: Planarization process steps for imprinted features as explained in the text: (a) features defined by UV-assisted nanoimprint lithography, (b) HSQ spin coating, (c) room-temperature contact planarization by a flat stamp, (d) HSQ heating at constant pressure, (e) contact planarized surface, (f) etching-back HSQ overlayer. After step (f) two different process routes can be followed, depending on the fabrication requirements: (g)-1 pattern transfer with an undercut resist profile via oxygen-based RIE, (h)-1 metallization, (f)-1 transfer layer removal (e.g. by acetone in case of PMMA), (g)-2 straight resist edge profile, (h)-2 further etching into substrate, (f)-2 transfer layer removal.

Si template onto the HSQ coated wafers at room temperature, in this tool pressure is applied by means of compressed air, ensuring pressure uniformity over the entire imprint area (Soft Press[®] technology).

After getting a planarized surface (see Fig.3.2(e)), the etch-back of the over-layer of HSQ (see Fig.3.2(f)) and pattern transferring through the residual imprint material and transfer layer that remained after S-FIL (see Fig.3.2(g)-1), were performed under CHF_3 plasma and O_2 plasma, respectively, in an Electrotech PF 340 apparatus. Cross-sections of samples were examined by a LEO 1550 high-resolution scanning electron microscope (HR-SEM) and topographical data were obtained using a DI 3100a Veeco atomic force microscope (AFM).

3.3 Results and Discussion

Several imprinted line-shaped features with different widths from sub-100 nm to 500 nm and separation from 100 nm to 2.5 μm were prepared for planarization experiments. The height of the features was around 100-150 nm. The transfer layer thickness was 60 nm for DUV-30J and 120 nm for PMMA. A homogenous residual layer of organic imprint material was also found around 60 nm after imprinting. In order to perform good planarization, the planarization layer needs to be as thick as possible. On the other hand, in order to have a well-controlled back etch step with

Figure 3.3: HSQ layer thickness versus spin rotation speed.

Figure 3.4: HR-SEM pictures of HSQ spin-coated samples with (a) 100 nm line width and 100 nm period, and (b) 500 nm line width and 500 nm period.

an appropriate stop at the top of the imprinted features, we aimed at an as thin as possible over-layer of HSQ. Therefore, as a first step we have investigated the spin coating dynamics of HSQ on blank Si wafers for different spin rotation speeds. Figure 3.3 shows the HSQ thickness versus rotational speed during spin-coating, varying from 500 rpm up to 3500 rpm in 500 rpm steps. For 1500 and 2000 rpm, the thicknesses are around 135 ± 3 and 115 ± 2 nm, respectively, and show good uniformity over the substrate. For 500 rpm, the HSQ film lost its thickness uniformity on the wafer. It seems that these thicknesses are sufficient to fill the trenches. However, the over-layer thickness (the thickness of HSQ on top of the features) is also critical for contact planarization. Especially in the case of largely separated features, there has to be sufficient material accumulated on the elevated features to be transferred towards the gaps between the features during the application of pressure. For 1000 rpm, the coating thickness was found to be 166 ± 3 nm, while keeping the coating uniformity. We therefore used 1000 rpm as rotational speed during spin coating.

As mentioned above, with only spin-coating, HSQ is able to give suitable results for features with low height (40 nm) and high feature density (65 %) [11], where the feature density is defined as $[\text{line width}/(\text{line width} + \text{spacing})]$. Nevertheless, when the feature height increases or the feature density decreases, additional steps are required to obtain adequate planarization. In our case, the feature height is around 100-150 nm and feature densities vary from 50 % to 17 %.

Figure 3.4 shows a sample, only spin coated with HSQ and baked at 120 °C,

Figure 3.5: HR-SEM pictures of contact-planarized samples (HSQ imprint material and DUV-30J transfer layer) with (a)-(b) 40 bar, (c) 20 bar, and (d) 10 bar pressure applied to the flat stamp.

without any additional steps. It can be seen that planarization degrades when the feature size and separation increase, giving a maximum of ~ 50 nm height difference. Even for high-density features there is still no complete local planarization.

To study the pressure dependence, a number of contact planarization experiments were performed with HSQ spin coated samples at room temperature under a pressure of 10 bar, 20 bar and 40 bar. Afterwards, the temperature was increased to 120 °C for 2 min. in order to increase the HSQ hardness without removing the template. Figure 3.5 shows the dependence of the HSQ planarization on the applied pressure. There is no significant difference between the results for different pressures, and HSQ redistribution from over-layer to cavity between features is still possible even at 10 bar.

A quantitative measurement of the step height reduction was performed using AFM. Planarization was performed under 40 bar on spin coated samples without pre-baking. To demonstrate the quality of planarization, a parameter known as the planarization factor can be defined as $\beta = 1 - t_f/t_i$, where t_i and t_f are the initial and final step heights, respectively (see Fig. 3.6(a) inset). The “degree of planarization” (DOP) is given by $100 \times \beta$. Figure 3.6(a) shows the surface profiles of 100 nm wide lines and 200 nm spacing at different stages; after imprinting, spin coating and pla-

narization, where, β_p is the planarization factor after contact planarization, and β_s is the planarization factor after spin coating, but before contact planarization. The initial HSQ layer thickness was around 165 nm. The DOP increased from 88 % to 95 % by contact planarizing the sample with the flat template. Figure 3.6(b) also shows the planarization degree for features with 100 nm and 500 nm line widths for different spacing values (varying from 100 nm to 2.5 μm). A DOP of around 95 % can be accomplished for all feature sizes and spacing. It should be noted that the surface profiles were taken in the close vicinity of the featured area; in other words, Fig. 3.6(a) and Fig. 3.6(b) give information about local planarization on the surface.

Figure 3.6: (a) Surface profiles of imprinted, HSQ spin coated and contact-planarized samples and definition of t_i and t_f (inset), (b) planarization factors for different pattern sizes before (β_s) and after (β_p) contact planarization.

Figure 3.7: HR-SEM pictures of patterned and unpatterned regions on the wafer. (a) and (b) shows patterned and unpatterned regions after spin coating, and (c) and (d) show patterned and unpatterned regions after contact planarization. Mold feature period is 337.5 nm (line width:spacing = 1:1.25) for (a), and 320 nm (line width:spacing = 1:1.8) for (c).

To demonstrate global planarization, high-resolution SEM pictures from patterned and non-patterned regions, 2-3 mm away from each other, were taken for both contact planarized and non-planarized samples. Further distances, more than 2-3 mm, have already same value because of the spin coating uniformity. Figures 3.7(a) and (b) show nearly 64 % DOP after HSQ spin coating. However, after contact planarization, the global DOP increased to 93 % (see Fig. 3.7(c) and (d)). This DOP value means uniform HSQ thickness over the sample after the etch-back process and that certain regions on the wafer can be prevented from being exposed by ionic plasma. In other words, HSQ is not only an etch mask for featured areas but also an etch mask for non-featured areas.

As indicated in Fig. 3.2, for pattern transferring it is also possible to get perpendicularly etched side walls by optimizing etching parameters in addition to undercut side walls. Although in our work it is specifically aimed to get undercut profiles after pattern transferring for metallization and lift-off purposes, an example of perpendicular pattern transfer also given to demonstrate the capability of this general-purpose method (see Fig. 3.8).

For further pattern transferring for lift-off, the contact planarized HSQ over-layer

Figure 3.8: Perpendicular pattern transfer into the residual and DUV-30J transfer layer according to Fig. 2(h). Imprinted features have 150 nm, 100 nm and 50 nm line width from left to right (all 125 nm spacing).

was etched back for 3 min. under CHF_3 plasma with 40 W RF power, 25 sccm gas flow and 10 mTorr chamber pressure. The organic imprint layer etching and transfer layer etching were performed under O_2 plasma with 20 W RF power, 20 sccm gas flow and 10 mTorr chamber pressure. Figures 3.9 and 3.10 show HR-SEM pictures of etched samples with different feature sizes. While the DUV-30J layer coated samples were cured at 120 °C under pressure after the planarization, the PMMA coated samples were cured at 70 °C to prevent possible damage to the PMMA layer because of its T_g

Figure 3.9: HSQ/imprint material (~ 60 nm)/DUV-30J (~ 60 nm) layer coated samples etched under CHF_3 plasma for 4.2 min, followed by O_2 plasma for 2 min. After CHF_3 plasma etching, the HSQ thickness is ~ 40 nm (a) mold feature period 1125 nm (line width: spacing = 1:1.25), (b) mold feature period 320 nm (line width : spacing = 1:1).

Figure 3.10: HSQ/ImprintMat (~ 60 nm)/PMMA (~ 100 nm) layer coated samples etched under CHF_3 plasma for 3 min and following O_2 plasma for 2 min. After CHF_3 plasma etching HSQ thickness is ~ 75 nm (a) mold feature size is 195 nm period grating (65 nm 1:2 lines and spacing), (b) mold feature size is 200 nm period grating (100 nm 1:1 lines and spacing).

between 85 and 165 °C. The suitability of HSQ as a hard etching mask, due to the etch selectivity between HSQ and underlying layers under O_2 plasma, is clearly observed in both Fig. 3.9 and 3.10. Very accurate transfer of 100 nm lines into the PMMA with 30 nm under-etched profile on each side clearly indicates critical dimension control capability of our method. For the structures in Fig. 3.11, an SFIL template with a 240 nm period grating (1:1 line and spacing) was used for pattern definition. After planarization and pattern transfer with resist undercut, metallization and lift-off were performed. As a result 30 nm thick gold lines, with a 5 nm Cr underneath, were obtained with a very high CD control.

3.4 Conclusion

It has been shown that global contact planarization with HSQ can be achieved at room temperature with a flat and blank template. This technique has shown an

Figure 3.11: 30 nm thick gold lines, with 5 nm Cr layer underneath, evaporated on a Si substrate after getting an undercut resist profile as illustrated in Fig. 2(g). Imprint mold feature period is 240 nm (line width:spacing =1:1).

improvement of 45 % compared to standard spin coating planarization. Because of the inorganic nature of HSQ, HSQ shows excellent etch selectivity with respect to organic underlying layers. This was demonstrated by pattern generation with UV-based NIL and RIE processes. Very precise control of the critical dimensions and well-defined undercut resist profiles were obtained, suitable for metal deposition followed by lift-off. HSQ contact planarization is a generic method and can therefore also be applied for other lithography techniques and multilayer applications because of its simplicity, fastness, low cost and efficiency.

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Interdigital Transducers on a ZnO/SiO₂/Si Multi-layer

In this chapter ultrahigh frequency surface acoustic wave devices were fabricated on ZnO/SiO₂/Si substrate using step-and-flash nanoimprint lithography combined with HSQ planarization. Excellent critical dimension control was demonstrated for interdigital transducers with finger electrode widths from 125 nm down to 65 nm. Fundamental and higher-order Rayleigh modes

up to 16.1 GHz were excited and detected, which is the highest frequency for ZnO-based transducers on silicon reported so far. Surface acoustic modes were confirmed with numerical simulations. Simulation results showed good agreement with the experimental data.

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4.1 Introduction

As explained in chapter 1, SAWs are widely applied in both research and industry. Their unique properties make them suitable not only for electrical signal processing, but also for other applications such as sensing [1], acousto-optical modulation as well as the control of excitons, electrons, and spins [2, 5] in semiconductors. For almost all SAW applications, there is a strong demand for higher frequencies, for example to enhance processing speed or to reach the quantum regime.

There are two main factors determining the IDT resonant frequencies: the acoustic properties of the (piezoelectric) substrate, and the IDT period, constrained by lithography resolution. Single-crystal piezoelectric substrates such as quartz, lithium niobate (LiNbO_3), and lithium tantalate (LiTaO_3) are often used because of their high electromechanical coupling coefficients, advantageous for converting the energy of the RF excitation into mechanical energy of the SAW. However, their SAW velocities are relatively low, limiting the operating frequency [6]. Substrates with overlayers of different materials with sharply varying elastic properties offer an alternative to get higher frequencies, as they enable the excitation of higher-order SAW modes with higher velocity v_{SAW} . In multi-layer systems, it is interesting to combine high-velocity, but non-piezoelectric substrates, such as Si, SiC, sapphire and diamond [7]-[10] with a piezoelectric thin film. Zinc oxide (ZnO) is a very popular piezoelectric thin-film material because of its high electromechanical coupling constant and ease of fabrication. In this work, a $\text{ZnO}(230 \text{ nm})/\text{SiO}_2(100 \text{ nm})/\text{Si}$ multilayer substrate was used. We have deliberately chosen a rather standard (low-resistive) Si substrate to demonstrate that our technology can result in extremely high-frequency SAW devices, which can be monolithically integrated with common electronic circuitry. The SiO_2 layer was included, as it was shown that an insulating buffer layer between the piezoelectric film and the substrate also increases the electromechanical coupling [11, 12].

The resolution of standard photolithography (minimum finger width of approximately $0.5 \mu\text{m}$) limits the operation frequency to around a few GHz, even for high-velocity substrates [13]. EBL is a good alternative for high-frequency IDTs because of its sub-100 nm resolution. However, the EBL parameters are generally strongly dependent on the substrate properties. This makes it difficult to design a standard lithography procedure for a variety of substrates, apart from reproducibility issues, low throughput and costliness [14, 15].

As explained above, nanoimprint lithography is a good candidate for fabricating IDT devices. IDTs on LiNbO_3 with center frequencies of 1.488 GHz (170 nm IDT finger width) and 2.38 GHz (130 nm IDT finger width) have been reported with hot embossing NIL and UV-NIL, respectively [16, 17]. A multilayer lift-off process using UV-NIL was also developed to fabricate 140 nm IDT fingers on LiNbO_3 [18]. Recently a UV-NIL process with a high-aspect-ratio stamp has been reported to fabricate 100 nm IDT fingers on LiNbO_3 with 8.6 GHz center frequency [19]. However, further improvement of the finger resolution of IDTs and reproducibility of the process is limited by the ability to get an appropriate undercut profile, which is essential for metal lift-off.

In this study, an alternative lithography technique, “step-and-flash nanoimprint lithography” (SFIL), with a novel material system for lift-off was used to reach very high resolution and higher reproducibility for the fabrication of IDTs. This fabrication method is based on the previous chapter [20]. While this method has the resolution of EBL, it is basically substrate independent with higher throughput compared to standard EBL.

4.2 Experimental Details

Sputtering is the most common deposition method for making polycrystalline ZnO thin films in acoustic applications. In this work, ZnO thin films were deposited on a double side polished (DSP) p-type Si (100) substrate (5-10 Ωcm) with 100 nm thick thermal oxide using an RF magnetron sputtering system with a ZnO target using argon gas (flow 10 sccm) and 50 W RF power at a process pressure of 3 mTorr. During the deposition, the substrate was at room temperature and rotated at 60 rpm to get uniform film thickness. The ZnO film thickness was measured to be 230 nm by a surface profilometer. An atomic force microscope (AFM) was used to measure the surface roughness. The crystalline structure and grain size of the ZnO film were confirmed by x-ray diffraction (XRD) measurements.

Nanoimprint lithography and a subsequent lift-off process were applied for the high-frequency IDT fabrication. The imprint template used in this work was produced by IMS Chips (Germany), and consisted of IDT devices with finger widths of 125 nm, 100 nm, 80 nm and 65 nm, corresponding to SAW wavelengths of 500 nm, 400 nm, 320 nm, and 260 nm, respectively. Figure 4.1 shows the process steps: first the

substrate was spin coated with a 80 nm thick PMMA transfer layer (495A 2 % from MicroChemicals) at 3000 rpm and baked on a hot plate at 120 °C for 2 min (see Fig. 4.1(a)). PMMA was used because of its low etching resistance compared to HSQ under oxygen plasma as well as its solubility in standard solvents (e.g. acetone) for easy lift-off. Pattern definition on the ZnO/SiO₂/Si substrate was carried out using S-FILTM on an ImprioTM 55 tool from Molecular Imprints, Inc. As imprint material a low-viscosity, organic, and UV-curable resist was used (MonomatTM from Molecular Imprints, Inc.) by field-to-field dispensing deposition (see Fig. 4.1(b)). The patterns were replicated by pressing the template into the fluidic resist at room temperature and under low force of 2-3 N (see Fig. 4.1(c)). After curing the resist through the

Figure 4.1: Fabrication process flow: (a) coating of the wafer with an organic transfer layer, (b) dispensing of UV-curable, organic imprint material on the surface with local volume adjustment, (c) imprint with a transparent template, (d) UV-light curing of the resist, (e) IDT structures defined in the imprint resist after removal of the template, (f) spin coating of HSQ planarization layer, (g) CHF₃ plasma etching of HSQ layer to expose elevated structures in organic layer, (h) O₂ plasma etching to transfer pattern down to the substrate, (i) metal deposition by e-beam evaporation, (j) lift-off in acetone.

transparent imprint template using broadband UV light with an exposure dose of 80 mJ/cm^2 at 230-360 nm wavelength (see Fig. 4.1(d)), the template was detached leaving its negative replication into the hardened resist (see Fig. 4.1(e)). For the next step, a planarization layer of 160 nm HSQ, XR-1541 6 % (from Dow Corning, Inc.) was deposited by spin coating on top of the imprinted features (see Fig. 4.1(f)), giving an excellent etch selectivity of more than 1:100 to the imprint and transfer materials. After getting a planarized surface, the etch-back of the overlayer of HSQ (see Fig. 4.1(g)), and pattern transferring through the residual imprint material and transfer layer that remained after S-FIL (see Fig. 4.1(h)), were performed under CHF_3 plasma and O_2 plasma, respectively, in a reactive ion etching apparatus (Adixen AMS100DE). For the metallization, 25 nm Au with an adhesion layer of Cr (2-3 nm) was grown by using an e-beam evaporation tool (see Fig. 4.1(i)). Finally lift-off was performed by dipping the sample into acetone followed by ultrasonication (see Fig. 4.1(j)).

The frequency spectrum characterization of the IDTs was carried out using an Agilent N5244A PNA-X network analyzer. Two-port network analyzer measurements have been performed by using microwave probes (Model 40A from GGB industries Inc.) with Ground-Signal (G-S) and Signal-Ground (S-G) configuration. The network analyzer was calibrated using a SOLT (short, open, load, through) calibration technique with a calibration substrate (CS-8 from GGB industries Inc.).

4.3 Results and Discussion

The thermodynamically stable crystal structure phase of ZnO has wurtzite symmetry with a hexagonal unit cell [21] and shows a preferential growth of c-axis (002) perpendicular to the substrate surface, as the ZnO (002) surface has the smallest surface free energy [22]. For SAW applications, highly c-axis oriented ZnO films are required because of their higher piezoelectric activity [23]. Figure 4.2 shows the XRD spectrum of the ZnO film indicating a polycrystalline structure. The (002) peak of ZnO corresponds to the c-axis perpendicular to the surface. The additional ZnO (100) peak might be attributed to the granular structure of the film, and it is generally observed at lower growth temperatures and higher Ar: O_2 gas mixture ratio [24, 25]. By using the Scherer formula, we calculated a grain size of around 15 nm. The AFM image shows that the root mean square (rms) roughness of the thin film is 2.5 nm for a $2 \mu\text{m} \times 2 \mu\text{m}$ area (inset).

Figure 4.2: XRD pattern and rocking curve of the ZnO (002) peak (upper inset, FWHM 0.540) showing the accurate c-axis orientation of the ZnO layer deposited by rf sputtering on SiO_2/Si substrate and AFM image of $2\text{ }\mu\text{m} \times 2\text{ }\mu\text{m}$ ZnO surface indicating a rms surface roughness of approximately 2.5 nm (lower inset).

Figure 4.3 shows SEM images of the IDT device with 65 nm finger width and spacing after nanoimprint lithography. The SFIL process is capable of replicating structures very precisely and reproducibly. From 125 nm down to the 65 nm lines and spaces, the dimensions are successfully transferred into the imprint resist on PMMA.

For further pattern transferring with lift-off, the spin coated HSQ overlayer was etched back under CHF_3 plasma. The organic imprint resist layer and transfer layer (PMMA) etching were performed under O_2 plasma. As mentioned in the previous section, HSQ plays a very important role for precise pattern transfer through the transfer layer as a planarization and excellent etch mask. It should be noted that uniform HSQ thickness over the sample is required to prevent certain regions on the sample from being exposed by ionic plasma. In other words, HSQ is not only an etch mask for featured areas, but also an etch mask for non-featured areas during O_2 plasma etching. In case of large-scale (a few hundred of microns) continuous shape of contact pads, HSQ thickness will be same on the contact pads and on the non-featured areas (areas away from IDTs) after the spin coating. Such non-uniform thickness of HSQ results in metallized region around the IDT devices at the end of the fabrication

Figure 4.3: (a) SEM image of an imprinted IDT device with line width and spacing of 65 nm, (b) zoom in image of fingers.

process. Another alternative method, “HSQ contact planarization” discussed in the previous chapter [20], will not be sufficient to remove HSQ over such long distances. In order to have sufficient planarization (uniform HSQ thickness) without additional planarization steps to the spin coating, we have used comb shape contact pads for the IDT devices.

Figure 4.4 shows the optical and SEM images taken after HSQ and transfer layer etching. HSQ provides sufficient planarization for the pattern transfer down to the substrate, preventing non featured areas (areas away from IDTs) from plasma etching (see Fig. 4.1(h)). For successful lift-off, an under-etched side profile is essential. In our previous work, we have already shown that very high etch selectivity between HSQ and underlying organic layers (1:100) provides sufficient under-etched profile for lift-off [20] .

In very high frequency applications, a well-defined periodicity and shape of the

Figure 4.4: Optical (a, b) and SEM (c, d) images of samples after oxygen plasma etching. Light and dark areas in the SEM image correspond to the substrate and the HSQ coated region, respectively.

electrodes have critical importance and determine the device performance. Figure 4.5 shows SEM images of 125 nm, 100 nm, 80 nm and 65 nm interdigital finger electrodes after metallization and lift-off, demonstrating high regularity and smooth edges of the electrodes, hence very high critical dimension (CD) control of the fabrication process.

In the measured transmission spectrum, the direct electromagnetic coupling (cross talk) between the generator and receiver IDTs suppresses the acoustoelectric response of the delay line. Moreover, in case of low-resistivity substrates ($< 3 \text{ k}\Omega\text{m}$), it becomes more difficult to study the transmission spectrum because of the considerable attenuation of the acoustic transmission due to free charges in silicon substrate [26]. Since the electromagnetic signals travel at the speed of light, i.e. much faster than the SAW velocity, it is possible to separate them from the acoustic ones in a Fourier-transformed spectrum (time domain). The gating technique can be used in the time domain to filter out the crosstalk signal [27]. In this study, the gating technique has been applied for the represented transmission data below.

Figure 4.6 highlights the capability of our devices to be operated at ultrahigh frequencies. Full design parameters of the IDT delay lines (DLs) are listed in table 4.1. For each DL, we have observed three resonances in both the reflection and transmis-

Figure 4.5: SEM images of IDT devices with 25 nm thick Au finger electrodes at the end of the fabrication process: (a) and (b): 125 nm, (c) and (d): 100 nm, (e) and (f): 80 nm, (g) and (h): 65 nm line width and spacing.

sion spectrum. Figures 4.6(a) and 4.6(b) show the reflection and transmission spectra of IDTs with 500 nm and 400 nm wavelengths, which hereafter will be referred to as DL1 (delay line 1) and DL2 (delay line 2), respectively. In the frequency spectra, the resonance peak labeled R1 corresponds to the Rayleigh mode with the lowest natural frequency, called the fundamental or first-order Rayleigh mode. Rayleigh surface acoustic modes result from the superposition of a longitudinal and a transverse

Figure 4.6: IDT reflection and transmission spectra for (a) DL1 ($\lambda = 500$ nm), (b) DL2 ($\lambda = 400$ nm), (c) DL3 ($\lambda = 320$ nm) and (d) DL4 ($\lambda = 260$ nm). The peaks labeled R1, R2, R3 and R4 correspond to the fundamental Rayleigh mode, second-order Rayleigh mode, third-order Rayleigh mode and fourth-order Rayleigh mode, respectively. Two different transmission curves were measured from IDTs with different separations. Blue (upper) curves: measured from ~ 405 μm separated IDTs, Red (lower) curves: measured from ~ 1081 μm separated IDTs.

acoustic mode which is polarized perpendicular to the surface, leading to a displacement field $\mathbf{u} = (u_x, 0, u_z)$ [3]. The modes labeled R2, R3 and R4 are associated with the second, third and fourth order Rayleigh modes, respectively. These higher-order Rayleigh modes normally appear when the acoustic velocity in the overlayers is lower than in the substrate and are determined by the relative thickness of the film ($d_{\text{ZnO}}/\lambda_{\text{SAW}}$) [7, 11]. The frequency of these modes depends on the ratio ($d_{\text{ZnO}}/\lambda_{\text{SAW}}$) between the overlayer thickness and the acoustic wavelength. The assignment of the observed resonances to Rayleigh modes is substantiated by numerical simulations, discussed below. In our multilayer system, the SAW velocity of the Si substrate is larger than that of both the ZnO and SiO₂ layers [28, 29]. Because of the fact that

Table 4.1: Design parameters of the IDT delay lines.

	λ (nm)	Number of fingers	Aperture (μm)	Distance between IDTs (μm)
DL1	500	1399	30	405.87 & 1081.87
DL2	400	1749	30	405.73 & 1081.87
DL3	320	2187	30	405.45 & 1081.87
DL4	260	2691	30	405.60 & 1081.87

the displacement field of SAWs is confined to a region of around one wavelength from the surface, the layer thicknesses play a crucial role in the response of the IDTs. In the present material system, the ZnO and SiO₂ thicknesses are around 230 nm and 100 nm, respectively. One can expect that the reflection of the SAW from the substrate should increase with the further reduction of the acoustic wavelength, which makes excitation of higher Rayleigh modes more likely.

For DL1, the relative thickness ($d_{\text{ZnO}}/\lambda_{\text{SAW}}$) is 0.46, where d_{ZnO} is the thickness of the ZnO film. The first-order Rayleigh mode appears at 4.697 GHz, corresponding with a velocity of 2348 m/s. Higher-order Rayleigh modes have higher velocities and therefore higher resonance frequencies. Second and third-order modes are observed at 8.002 GHz and 9.495 GHz with corresponding velocities of 4001 m/s and 4747 m/s, respectively. In the transmission spectrum, the resonance peak of the second-order mode has the highest amplitude, which indicates that the electromechanical coupling constant (k^2) of this mode is higher than that of the first and third modes (see table 4.2). The standard method described in the IEEE standards, has been used to calculate k^2 values from the admittance curves, which were extracted from the reflection data [30].

For DL2, the relative thickness is 0.56 and the first-order Rayleigh mode (R1) appears at 5.737 GHz, corresponding with a velocity of 2295 m/s. The second (R2) and third-order (R3) modes have resonances at 9.137 GHz and 11.03 GHz, with velocities of 2655 m/s and 4412 m/s, respectively. The transmission spectrum reveals that the dominant mode is also the second mode for a relative thickness of 0.56.

Figures 4.6(c) and 4.6(d) show both reflection and transmission spectra of the delay lines with 320 nm (DL3) and 260 nm (DL4) wavelength, which is comparable with the distance to the Si substrate from the upper surface of ZnO ($d_{\text{Si}} = 330$ nm, $d_{\text{Si}}/\lambda_{\text{SAW-DL3}} = 1.03$ and $d_{\text{Si}}/\lambda_{\text{SAW-DL4}} = 1.27$). The resonances observed at 10.541 GHz, 12.959 GHz and 14.260 GHz for DL3 are mediated by the second-order (R2), third

Table 4.2: Experimental results of resonance frequency, velocity and electromechanical coupling coefficient for different excitation modes of DL1 ($\lambda = 500$ nm), DL2 ($\lambda = 400$ nm), DL3 ($\lambda = 320$ nm) and DL4 ($\lambda = 260$ nm) devices. R1, R2, R3 and R4 are first-order (fundamental), second-order, third-order and fourth-order Rayleigh modes, respectively.

DL1	R1			R2			R3			R4		
	f (GHz)	v (m/s)	k^2	f (GHz)	v (m/s)	k^2	f (GHz)	v (m/s)	k^2	f (GHz)	v (m/s)	k^2
1	4.697	2348	0.007	8.002	4001	0.030	9.495	4747	0.010	-	-	-
2	5.737	2295	0.012	9.137	3655	0.036	11.03	4412	0.014	-	-	-
3	-	-	-	10.54	3373	0.032	12.96	4147	0.013	14.26	4563	0.025
4	-	-	-	12.17	3164	0.033	15.14	3937	0.031	16.13	4194	0.028

order (R3) and fourth-order (R4) Rayleigh modes, respectively. For DL4, the resonance peaks were observed at 12.170 GHz (R2), 14.144 GHz (R3) and 16.132 GHz (R4). The absence of the fundamental Rayleigh modes for DL3 and DL4 might be explained by the low value of the electromechanical coupling coefficient of the fundamental Rayleigh modes for these wavelengths.

For the device operation, especially at very high frequencies, not only the velocity and electromechanical coupling coefficient, but also the acoustic propagation loss has importance. SAW scattering at the grain boundaries of the ZnO film is one of the main reasons of propagation loss. Propagation loss depends on the ratio of the wavelength to the grain size, and decreases with decreasing grain size [31]. At very high frequencies, propagation loss is expected to be too high due to the exponential dependence on

Figure 4.7: Propagation losses for Rayleigh wave modes in delay line devices as a function of frequency.

Table 4.3: Material constants used for FEM analysis.

Material		Au	ZnO	SiO ₂	Si
Density (kg/m ³)	ρ	19300	5665	2350	2330
Elastic constants (Pa $\times 10^{11}$)	C_{11}	2.202	2.0900	0.785	1.944
	C_{12}	1.604	1.2035	0.161	0.352
	C_{13}	1.604	1.0460	0.161	0.639
	C_{33}	2.202	1.6570	0.785	1.657
	C_{44}	0.299	0.4230	0.312	0.796
	C_{66}	0.299	0.4440	0.312	0.509
Piezoelectric constants (C/m ²)	e_{15}	—	-0.480	—	—
	e_{31}	—	-0.573	—	—
	e_{33}	—	1.321	—	—
Relative permittivity	ϵ_{11}	—	7.38	2.37	11.68
	ϵ_{33}	—	7.38	2.37	11.68

frequency. To study propagation loss of the SAW modes in our devices, we have used delay lines with different separations between input and output IDTs. Insertion losses (transmission spectra) were measured for each device with two different separations (see Fig. 4.6). By using the dB difference between resonance peak values ($|\alpha_2| - |\alpha_1|$) and propagation distances ($405.87 \mu\text{m}$ and $1081.87 \mu\text{m}$), we calculated propagation losses for different acoustic wavelengths and modes. Figure 4.7 shows the propagation losses with unit of dB/mm. For the first mode (R1) of DL2, we are not able to measure a clear difference between the resonance peaks because of the residual cross talk signal at this frequency. Figure 4.7 reveals that the R3 mode of DL1 has a minimum loss of $\sim 2.6 \text{ dB/mm}$ ($13 \times 10^{-4} \text{ dB}/\lambda$). Maximum propagation loss was calculated for the R4 mode of DL4, which is $\sim 22 \text{ dB/mm}$ ($58 \times 10^{-4} \text{ dB}/\lambda$). As illustrated in Fig. 4.7, the propagation losses for the same type of modes in different delay lines tend to increase with the higher frequencies. It is found that for R2 and R3, the propagation losses are weakly frequency dependent until around 9 GHz and 11 GHz, respectively.

The assignment of the SAW modes was confirmed by finite element method (FEM) calculations, using COMSOL[®] Multiphysics software. Eigenfrequency analysis was used to determine the resonance frequencies and the modes of deformation in the multilayer system. The material parameters used in the calculations, are given in Table 4.3. As IDTs have a large number of fingers, and the length of these fingers is a few hundred times longer than their width, edge effects can be neglected. Therefore, the finger pairs can be regarded as an infinitely extended periodic structure and the

Figure 4.8: FEM calculation results for DL1 ($\lambda = 500$ nm). Upper row shows vertical component of the displacement field for R1, R2 and R3 modes (left to right). Lower row shows electric potential distribution in the ZnO(230 nm)/SiO₂(100 nm)/Si multilayer for R1, R2 and R3 modes (left to right). The numbers on the colored legend have arbitrary unit.

model geometry can be reduced to one wavelength [32]. The geometries used in the model are given in Figs. 4.8 and 4.9 with the results. SAW propagation direction was chosen in the x -axis corresponding to $[110]$ crystal direction of the silicon substrate. The vertical component of the displacement field and the corresponding electric potential distribution were used to define the piezoelectrically active surface acoustic modes. Certain calculated modes, which yield a symmetric displacement (u_z) along the x -axis and a reversely polarized electric potential on the fingers, were assigned as piezoelectrically active. This confirms that these modes can be excited by using reversely polarized finger pairs (as in an IDT) via the inverse piezoelectric effect.

In Fig. 4.8, simulation results for an IDT with 500 nm wavelength (DL1) are presented. R1, R2 and R3 modes appear at 4.656 GHz, 7.941 GHz and 10.361 GHz,

Figure 4.9: FEM calculation results for DL4 ($\lambda = 260$ nm). Upper row shows vertical component of the displacement field for R2, R3 and R4 modes (left to right). Lower row shows electric potential distribution in the ZnO(230 nm)/SiO₂(100 nm)/Si multilayer for R2, R3 and R4 modes (left to right). The numbers on the colored legend have arbitrary unit.

respectively. The displacement field of the fundamental Rayleigh mode (R1) is mostly concentrated in the 230 nm thick ZnO layer, and it completely vanishes in a distance of one wavelength (see Fig. 4.8(a)). The electric potential profile for the same mode exhibits a symmetric distribution along the x-axis (see Fig. 4.8(b)). The potential around the ZnO/SiO₂ interface is higher than on the electrodes, which implies that the induced potential in this region will be higher than the applied potential on the finger electrodes during IDT operation. Higher-order Rayleigh modes are characterized by the number of displacement nodes (minimal amplitude points) in the z-component of the displacement field along the vertical direction (z-axis). The displacement field of the second mode (R2) penetrates more into the substrate than the first mode (see Fig. 4.8(c)). The node along the z-axis which appears in the ZnO layer denotes the second-order Rayleigh mode (R2). The symmetric potential distribution of the R2

Figure 4.10: Comparison of the frequencies obtained experimentally and numerically for all IDT devices with their difference in percent. R1, R2, R3 and R4 labels represent the fundamental, first-order, second-order, third-order and fourth order Rayleigh modes, respectively.

mode has also a node in the ZnO layer and has its maximum value on the finger electrodes (see Fig. 4.8(d)). The penetration depth of the displacement field reaches a maximum for the third mode (R3), exhibiting two nodes in the ZnO layer (see Fig. 4.8(e)) and the maximum value of the electric potential appears in the ZnO layer (see Fig. 4.8(f)).

The simulation results for an IDT with 260 nm wavelength (DL4) can be seen in Fig. 4.9. One can derive that, in contrast to DL1, the lowest excited resonance frequency for DL4 corresponds to the second-order Rayleigh mode (R2), and it appears at 11.915 GHz (Fig. 4.9(a) and 4.9(b)). The other calculated resonances, at 14.964 GHz and 18.228 GHz, correspond to the R2 mode (Fig. 4.9(c) and 4.9(d)) and R3 mode (Fig. 4.9(e) and 4.9(f)), respectively. The calculated displacement field and electric potential profiles for all devices confirm that the modes are all piezoelectrically active. In other words, the corresponding type of deformations can be excited by IDTs.

A comparison of the measured and calculated modes for all IDT devices is given in Fig. 4.10 with the relative difference between them in percent. The calculated frequencies are in very good agreement with the measured values, especially for the fundamental (R1) and second-order (R2) modes. The deviation between experiment

and simulation for the R3 and R4 modes can possibly be attributed to the uncertainties in the ZnO properties.

4.4 Conclusion

In conclusion, ultrahigh frequency IDTs on a ZnO/SiO₂/Si multilayer system were fabricated, using nanoimprint lithography and electrical characterization was performed. Crucial to our novel approach is the application of a hydrogen silsesquioxane (HSQ) layer that provides excellent planarization combined with very high etching selectivity, critical dimension control and easy lift-off. The highest resonance frequency, reported so far for ZnO-based transducers on silicon (16.1 GHz) was measured on an IDT with 65 nm finger width. Our experimental results are in very good agreement with numerical simulations of the specific multilayer system. The IDT performance is expected to improve after optimizing the ZnO growth parameters and lowering the Si substrate conductivity. Our technology has great potential to reach even higher frequencies in case of higher velocity substrates, such as SiC, sapphire or diamond.

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Chapter 5

Ultrahigh-Frequency Surface Acoustic Wave Generation for Acoustic Charge Transport in Silicon

This chapter demonstrates electrical generation of ultrahigh-frequency surface acoustic waves on silicon substrates, using high-resolution UV-based nanoimprint lithography, hydrogen silsequioxane planarization and metal lift-off. Interdigital transducers were fabricated on a ZnO layer sandwiched between two SiO₂ layers on top of a Si substrate. Excited modes up to 23.5 GHz were observed. Depth profile calculations of the piezoelectric field show this multilayer structure to be suitable for acoustic charge transport in silicon at extremely high frequencies with moderate carrier mobility requirements.

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5.1 Introduction

For acoustoelectric devices, most studies have been done on piezoelectric semiconductors, such as GaAs-based systems where the energy band diagram is modulated by the SAW-induced piezoelectric and strain fields [1–7]. For weakly piezoelectric semiconductors, enhancement of the electromechanical coupling is normally achieved by introducing a piezoelectric thin film underneath the IDTs with a higher electromechanical coupling constant than the substrate. To provide piezoelectric potential modulation of the band structure in case of non-piezoelectric materials, this piezoelectric thin film needs to cover the whole surface, or at least the acoustic path. ZnO is a well-known material to enhance the electromechanical coupling of weakly piezoelectric materials or to excite SAWs even in non-piezoelectric substrates (such as silicon) because of its relatively large coupling coefficient [8]. As additional advantage, the presence of a thin ZnO film on top of a high-velocity substrate allows the excitation of overtones of the fundamental SAW mode with higher velocity, and hence a higher frequency owing to the “slow on fast” configuration [1]. However, being a reactive material, unprotected ZnO is CMOS incompatible, making it difficult to realize ZnO-based acoustoelectric devices on silicon chips. Efficient acoustoelectric charge transport by means of a SAW-induced potential modulation in Si requires a very high-frequency IDT because of the very low electron and hole mobility in Si compared to GaAs. In the case that efficient acoustoelectric charge transport in silicon is accomplished, acoustic transport devices similar to those developed in GaAs-based systems are likely to be integrated into silicon circuits.

In this chapter, we have fabricated very high-frequency IDT devices on a layered structure of $\text{SiO}_2/\text{ZnO}/\text{SiO}_2/\text{Si}$ and investigated the excited SAW modes both experimentally and theoretically. IDTs were fabricated by UV-based nanoimprint lithography and lift-off processes [9] with a SAW operation frequency as high as 23.5 GHz, using a ZnO film grown on a thermally oxidized Si substrate, covered with a 20 nm thick SiO_2 capping layer. This frequency is even higher than our recently reported highest frequency on Si using a ZnO film [9]. The present study is intended to investigate the potential for acoustoelectric applications in silicon-based structures.

5.2 Experimental Details

As a substrate a highly resistive Si ($>10 \text{ k}\Omega$) wafer was used. A high-quality thermal oxide layer with a thickness of 105 nm was grown on Si in an oven at 900°C . ZnO films with thicknesses of 40 nm and 80 nm were grown on top of the SiO_2 layer by rf magnetron sputtering using an Ar/ O_2 gas mixture. The deposition conditions were optimized in order to get the films hexagonal with the c-axis oriented (along the growth direction). This orientation is required for large piezoelectric coupling, and was verified by x-ray diffraction (XRD) measurements, shown in Fig. 5.1. The subsequent 20 nm SiO_2 layer, which was deposited in the same sputtering system, protected the ZnO film from being attacked by the lift-off solvent during the subsequent fabrication steps. For IDT fabrication, the samples were first spin coated with an 80 nm thick wet developable layer of WiDE-C 80 (Brewer Science) and baked at 150°C . A 10 nm thick adhesion promoter layer of TranSpinTM (Molecular Imprints, Inc.) was spin coated on top of the WiDE-C material to provide better adhesion of the NIL resist.

Figure 5.1: XRD pattern and zoom in of the ZnO (002) peaks taken from $\text{SiO}_2/\text{ZnO}/\text{SiO}_2/\text{Si}$ structures (inset). Results are showing the accurate c-axis orientation of the ZnO layers of 40 nm and 80 nm thickness with 0.530 and 0.480 of full width at half maximum (FWHM), corresponding to 16 nm and 17 nm grain sizes, respectively.

UV-based NIL was performed on an Imprio 55 tool by using the S-FILTM process and MonoMatTM from Molecular Imprints, Inc. as organic imprint resist. After nanoimprint, the samples were planarized with a 160 nm thick HSQ layer by spin coating. For pattern transfer, RIE was used. First, the HSQ layer was back-etched down to the top of the elevated imprint structures in a CHF₃-based plasma. Second, these elevated structures were etched down to the substrate in O₂-based RIE using the HSQ as an etch mask. The high etch resistance of HSQ functioned hereby as an etch mask providing an appropriate undercut profile for the final lift-off step. In the last step, 50 nm thick Al electrodes were deposited in an e-beam evaporation system followed by lift-off in 96 % HNO₃. The process scheme and further details are described in previous chapters [9, 10].

To study the SAW characteristics, two-port IDTs with single-finger (SF) and double-finger (DF) configurations were fabricated. The widths of the IDT finger electrodes were 125 nm, 100 nm, 80 nm and 65 nm with metallization ratio 0.5, corresponding to acoustic wavelengths of four times and eight times the finger width for SF and DF IDTs, respectively. The IDT aperture was kept 30 μm for all devices and the input and output IDTs were separated by 306 μm and 405 μm for DF-IDTs and SF-IDTs, respectively.

5.3 Results and Discussion

In layered structures, the ratio between the SAW wavelength and the piezoelectric layer thickness determines the number of overtones. Emergence of overtones of the fundamental mode is more likely when the contrast in velocity between the overlayer and the substrate increases. In the present multilayer system, the SAW velocity of the Si substrate is larger than that of the ZnO film. The ZnO/SiO₂ films form, therefore, an acoustic waveguide, supporting overtones. The overtones are referred to as higher-order Rayleigh modes, with nodes in the depth direction. In contrast to a homogenous solid medium, SAW modes become dispersive in such a layered system.

Figure 5.2 shows the frequency response of two IDTs having acoustic wavelengths $\lambda_{\text{SAW}} = 1 \mu\text{m}$ and 640 nm. The resonance labeled R1 is associated with the first-order Rayleigh mode, which is the fundamental surface mode in piezoelectric materials. The other modes labeled R2 and R3 are associated with the second and third order Rayleigh modes, respectively. These modes have higher SAW velocities and thus

Figure 5.2: Reflection (S_{11}) and transmission (S_{21}) spectra of DF-IDT devices with (a) 1 μm and (b) 640 nm wavelength. R1, R2 and R3 correspond to the first, second and third order Rayleigh modes, respectively. H3 and H5 indicate the third and fifth harmonics. Reflection curves are vertically offset for clarity.

resonances at higher frequencies. The peaks additionally labeled H3 and H5 show the third and fifth harmonics of related modes; for example, R2H3 indicates the third harmonic of the second mode. In order to correctly assign the SAW modes, the acoustic field distribution for each mode was obtained from numerical simulations based on a linear elastic model [1].

Figure 5.3 (a) compares the calculated and measured frequencies for all IDT devices. The calculations show very good agreement with the measured resonant

Figure 5.3: Measured (circles) and calculated (squares) frequency (a) and velocity (b) of SAW modes. SF and DF refer to single- and double-finger IDTs, respectively. R1, R2 and R3 are first, second and third order Rayleigh modes.

frequencies. Figure 5.3(b) shows the calculated and measured acoustic velocities of the excited modes. In a layered system, the SAW velocity is expected to take a value between the bulk velocities of the low- and high-velocity material. For larger $\lambda_{\text{SAW}}/d_{\text{ZnO}}$ ratio, the SAW velocity tends to increase. The acoustic field penetrates more into the higher-velocity Si substrate, and the fraction of the acoustic field in Si increases, while that in the ZnO layer decreases, leading to a higher SAW velocity. The results show that the R1 and R2 SAW modes for DF-IDTs give a higher velocity

than the same order modes for SF-IDTs at equal $\lambda_{\text{SAW}}/d_{\text{ZnO}}$ ratios. This is the result of the thickness ratio of the ZnO and thermally grown SiO₂ layer ($d_{\text{ZnO}}/d_{\text{SiO}_2}$), which is 0.38 and 0.76 for SF and DF-IDTs, respectively. The fraction of the acoustic field penetrating into the Si will be less for the SF-IDTs compared to the DF-IDTs for the same $\lambda_{\text{SAW}}/d_{\text{ZnO}}$, resulting in a lower acoustic velocity. The resonance frequency of the R3 mode for the SF IDT with 400 nm wavelength showed the highest measured velocity, 5,833 m/s, which is very close to the predicted transverse bulk velocity of Si with polarization parallel to the c-axis ($v_{\text{T}} = (c_{44}/\rho)^{1/2} = 5,845$ m/s). The two horizontal dashed lines indicate the transverse velocity (v_{T}) for Si and ZnO, respectively. A SAW mode with a velocity higher than v_{T} of the substrate is expected to couple with bulk modes and to show a leaky SAW nature, as the acoustic power flows into the bulk substrate resulting in very large insertion loss. Hence, 5,845 m/s can be considered as an upper limit for non-leaky SAW modes in the present material system. As all of the measured velocities remain below this upper limit, the resonance modes measured in this study have non-leaky SAW nature.

For the acoustoelectric application of SAWs, the carrier drift velocity has great importance. The piezoelectric potential generated by the SAWs modulates the energy band structure of the material within around one wavelength from the surface and creates mobile potential modulations moving along the acoustic path at sound velocity. These potential modulations can capture electrons (holes) in the minima (maxima) of the conduction (valance) band. Efficient transport requires the transport of carriers in the same wave cycle (or the same minima/maxima) along the transport path without loss. To satisfy this condition, the carriers should be able to follow the SAW-induced mobile potential modulations. Therefore, the minimum required mobility is $\mu_{\text{min}} = v_{\text{SAW}}/E_{\text{x}}$, where v_{SAW} is the SAW velocity and E_{x} the piezoelectric field induced in the propagation direction. Electric field and potential distributions are determined by the acoustic linear power density P_{l} , which is defined as the total acoustic power flow per unit length across the acoustic beam ($P_{\text{l}} = \text{acoustic power}/\text{IDT aperture}$).

In order to understand how efficiently IDT devices on SiO₂/ZnO/SiO₂/Si structure can be implemented for the acoustic charge transport in the high-frequency regime, calculations for the piezoelectric field (E_{x}) and potential distribution (Φ_{SAW}) were carried out with a nominal RF power $P_{\text{rf}} = 25$ dBm for different vibration modes. The power which is converted into the acoustic mode propagating in one direction was calculated from the amplitude of the dip in reflection (S_{11}) spectra. Calculations

Figure 5.4: Depth profile of piezoelectric field (E_x) and piezoelectric potential (Φ_{SAW}) for R1 and R2 modes in an IDT device with $\lambda_{\text{SAW}} = 1 \mu\text{m}$. The dashed and solid lines indicate a shorted (metallized) and open (non-metallized) surface condition.

were carried out for both open (metal free) and short (metallized) surface conditions to reveal the effect of metal structures on the surface.

Figure 5.4 shows the depth dependence of E_x and Φ_{SAW} of the R1 and R2 modes, calculated for the IDT with $1 \mu\text{m}$ wavelength. A thin metal film deposited on the sputtered SiO_2 changes the surface condition and short circuits the piezoelectric potential, thus changing the depth profile underneath. In the present material system, most of the acoustic transport in Si is expected at the SiO_2/Si interface. For the R1 mode, the open surface condition gives a higher field value at the interface compared to the shorted surface. However, the R2 mode exhibits opposite characteristics for the short circuited surfaces. For the higher-order modes, the short circuited surface condition gives a much higher piezoelectric field value at the interface than that of the free surface condition. The minimum required mobility values calculated from E_x for the SiO_2/Si interface are given in Fig. 5.5. These values are less than both electron and hole mobilities at room temperature in intrinsic silicon [11]. In a recent experimental work on ambipolar acoustic transport in silicon by Barros et al. [12], the minimum mobility for efficient transport was reported to be $730 \text{ cm}^2/\text{Vs}$ for an IDT on $\text{ZnO}/\text{SiO}_2/\text{Si}$ operating at 750 MHz with a linear acoustic power density of 76 W/m .

Figure 5.5: The minimum required mobility $\mu_{\min}$ for efficient acoustic charge transport at the SiO₂/Si interface for different SAW modes. The fundamental mode exhibits higher required mobility for the shorted surface condition. For higher-order modes, a metallized surface modifies the depth profile, giving a lower required mobility value. (Inset: The layer sequence and thicknesses).

In their experiments, this relatively high value was a limiting factor for ambipolar transport because of the low hole mobility [12]. According to our results, ultrahigh frequency SAWs can overcome this limitation problem for efficient hole transport.

5.4 Conclusion

In conclusion, we have fabricated ultrahigh frequency SAW devices up to 23.5 GHz on SiO₂/ZnO/SiO₂/Si by using a CMOS-compatible fabrication process. Excited modes are well verified by numerical calculations. Piezoelectric field distribution calculations showed that the SAW-induced field is strong enough for efficient charge (both electron and hole) transport at the Si/SiO₂ interface. Our results show that the higher-order modes are advantageous to get higher acoustoelectric current owing to their higher frequency without losing the required mobility. Additionally, metal structures on the SiO₂ surface modulating the piezoelectric field, might be used as an acoustic charge guide for certain vibration modes to enhance the transport.

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Chapter 6

Photoexcited Carrier Transport in GaAs Nanowires by Surface Acoustic Waves

The oscillating piezoelectric fields accompanying surface acoustic waves are able to transport charge carriers in semiconductor heterostructures. In this chapter, we demonstrate high-frequency (above 1 GHz) acoustic charge transport in GaAs-based nanowires deposited on a piezoelectric substrate. The short wavelength of the acoustic modulation, smaller than the length of the

nanowire, allows the trapping of photogenerated electrons and holes at the spatially separated energy minima and maxima of conduction and valence bands, respectively, and their transport along the nanowire with a well-defined acoustic velocity towards indium-doped recombination centers.

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6.1 Introduction

The unique properties of semiconductor nanowires (NWs) allow for the easy exploitation of quantum effects in one-dimensional systems [1], thus opening new interesting perspectives in the field of nanostructured semiconductor devices. These new possibilities are mainly based on the typically small cross-section of NWs, which favours mesoscopic size effects, and also on the removal of the constraints associated with the epitaxial growth in case of combination of dissimilar materials. Moreover, the NW geometry allows for easy interaction with light, making them good candidates for new concepts in opto-electronic devices [2–4]. Although there are several challenges to create functional semiconductor devices for specific applications, the successful synthesis of NWs in a controllable manner, regarding both geometry and chemical composition, gives improved functionalities. For instance, radial growth for core/shell configuration [5] and axial growth for heterostructures [6] enable us to reduce unintentional surface states and to control the band structure along two directions.

In order to take advantage of their special electro-optical properties, an efficient carrier transport mechanism which eliminates the electrical contact issue on nanostructures [7] is important. In the last years, surface acoustic waves (SAWs) have proved to be a promising candidate for carrier control in two-dimensional (2D) semiconductor structures allowing both spatial and temporal manipulation: the modulated dynamic piezoelectric potential accompanying the strain field of SAWs allows spatial modulation of carriers confined in a semiconductor structure when its distance to the surface is less than the SAW penetration length. The acoustically confined carriers are then transported along the semiconductor structure with the well-defined SAW velocity. This mechanism has been used for carrier as well as for spin transport over distances of several tens of micrometres in GaAs-based quantum wells [8–10].

This acoustic mechanism for carrier manipulation has recently been extended to semiconductor nanowires deposited on top of a strong piezoelectric substrate, normally LiNbO_3 . The piezoelectric potential induced by SAW can be written as $\Phi_{\text{SAW}}(x, t) = \Phi_{\text{SAW}_0} \sin[2\pi(x/\lambda_{\text{SAW}}) - (t/T_{\text{SAW}})]$, ($0 < x < l_d$). Here, Φ_{SAW_0} is the potential amplitude, T_{SAW} and λ_{SAW} are the period and wavelength of the SAW, respectively, and l_d is the carrier transport distance. In this way, acoustically induced electric currents in GaN NWs have been demonstrated [11]. Furthermore, in GaAs NWs the

Figure 6.1: (a) Experimental set-up. The GaAs nanowire with a (In,Ga)As region is placed on the LiNbO₃ substrate parallel to the SAW path, with the (In,Ga)As region (violet NW section) opposite to the IDT that generates the SAWs. The carriers are generated by a laser beam focused at the edge of the GaAs region (green NW section). (b) Acoustic transport mechanism: The photo-generated electrons and holes are trapped at the spatially separated and piezoelectrically induced energy minima and maxima at the conduction and valence band edges, respectively. These trapped carriers are then transported by the SAW with acoustic velocity to the (In,Ga)As region, where they recombine at quantum-dot-like centers emitting photons with energies well below those the emitted by the GaAs.

acoustically induced quenching of the photoluminescence (PL) [12], as well as SAW-induced ambipolar carrier transport and antibunched photon emission [13] have been reported. In all these cases, however, the carrier transport distance, l_d , was much shorter than the SAW wavelength, λ_{SAW} . Under these conditions, the piezoelectric field essentially induces a seesaw-like oscillating voltage gradient along l_d [13]. Due to this voltage gradient, carriers of opposite sign drift along opposite directions with a velocity determined by the mobility of each carrier type. The simultaneous transport of electrons and holes along the SAW propagation direction with the well-defined acoustic velocity, however, requires the opposite condition, that is, $\lambda_{\text{SAW}} < l_d$. For example, in case of $l_d = 5 \mu\text{m}$ and $v_{\text{SAW}} = 3980 \text{ m/s}$ (SAW velocity for LiNbO₃), SAWs with a frequency $f_{\text{SAW}} = v_{\text{SAW}}/l_d \geq 0.8 \text{ GHz}$ must be excited to fulfill this condition.

In this chapter, we demonstrate the acoustic transport of charge carriers along GaAs NWs at a frequency well above 1 GHz and over distances overcoming the acoustic wavelength (cf. Fig. 6.1(a)), thus fulfilling the conditions for simultaneous transport of electrons and holes in the same direction. The SAW, generated by an interdigital transducer (IDT) fabricated on the piezoelectric substrate by nanoimprint lithography, traps the optically generated electrons and holes at different positions

along the acoustic wavelength and transports them with a well-defined velocity from the generation point towards the opposite end of the NW, where they are trapped by quantum-dot-like recombination centers and recombine radiatively, cf. Fig. 6.1(b).

6.2 Experimental Details

The delay line for SAW generation and transport consists of two identical IDTs fabricated on a 128° Y-cut LiNbO_3 wafer. LiNbO_3 is a strong piezoelectric material providing an intense piezoelectric field for the manipulation of carriers in semiconductor structures placed on its surface [11–13]. In our experiments, instead of a conventional IDT design, a FEUDT design was used, which enhances the acoustic power flow in forward direction, thus enabling higher SAW amplitudes [17]. To fabricate the IDTs, we used UV-NIL combined with HSQ planarization and RIE [18, 19]. In this process, first the LiNbO_3 substrate was coated with an 80 nm thick WiDE-C 80 material (Brewer Science) acting as a transfer layer and a 10 nm thick layer of TranSpinTM (Molecular Imprints, Inc.) used as adhesion promoter for the following nanoimprint step. The substrate was then patterned by step and flash UV-NIL on an Impro 55 tool (from Molecular Imprints, Inc.) and subsequently planarized with a 160 nm thick spin-coated HSQ layer. In the following two-step RIE process, first the planarization layer was etched back in a fluorine based plasma until the top of the imprinted features was opened. Then using oxygen plasma, the underlying residual imprint material and transfer layer were etched down to the substrate, providing a negative side wall profile for the lift-off process. In the last step, a ~ 40 nm thick Al layer, sandwiched between ~ 4 nm thick Ti layers, was deposited and lifted off in nitric acid.

The GaAs NWs were grown by molecular beam epitaxy (MBE) using a self-assisted vapor-liquid-solid growth method [14, 15] on a Si(111) substrate. At the end of the growth process, the Ga shutter was closed and the growing surfaces were exposed to In and As fluxes for a short time. In most cases, this procedure leads to the formation of a segment of approximately 300 nm length containing (In,Ga)As at the top of the GaAs section, as indicated on Fig. 6.1. In other cases (as, for instance, for the nanowire shown in Fig. 6.2), the top region of the NW emits at the GaAs wavelength, thus indicating that (In,Ga)As is incorporated on the GaAs surface around the middle of the NW axis. Finally, an $\text{Al}_x\text{Ga}_{1-x}\text{As}$ shell with x

nominally equal to 0.3 was incorporated to prevent non-radiative recombination at the free surfaces [16]. The resulting NWs had a narrow size distribution with an average length of $7\text{ }\mu\text{m}$ and diameter of 150 nm. They were then transferred from the Si substrate to the LiNbO_3 surface by gently pressing and rubbing the Si across the LiNbO_3 surface, along a direction parallel to the IDT delay line axis. In this way, we obtain a preferred alignment of the NWs parallel to the SAW propagation direction (cf. Fig. 6.1).

The experiments on acoustic transport were performed at a nominal temperature of 20 K in a cold-finger cryostat equipped with an optical access window and coaxial connections for the application of radiofrequency (RF) signals to the IDTs. Electrons and holes are optically injected into the NW by a tightly focused ($50\times$ objective, spot diameter of $1.5\text{ }\mu\text{m}$) laser beam of wavelength 756 nm placed at the edge of the NW closest to the IDT that generates the SAW. The laser pulse repetition rate was synchronized with the SAW. The NW luminescence is collected by the same objective that focuses the laser beam and sent to the input of a single grating monochromator connected to a charge-coupled device (CCD) camera for time-integrated spectroscopic measurements. The image of the SAW propagation path is parallel to the input slit of the monochromator, so that the resulting 2D image at the CCD camera shows the PL emitted by the NW with both spatial (along the NW axis) and spectroscopic resolution.

6.3 Results and Discussion

Figure 6.2(a) shows the room-temperature rf power reflection (S_{11}) and transmission (S_{11}) coefficients for the IDT used in our experiments. The S_{11} spectrum shows clear minima at frequencies fulfilling the condition $f_{\text{SAW}} = n \times v_{\text{SAW}}/\lambda_0$, n being an integer, v_{SAW} the acoustic velocity, and λ_0 the IDT fundamental wavelength. The latter is defined by the periodicity of the IDT finger grating, which in this case is $5\text{ }\mu\text{m}$. The measurements reported below were obtained by using the second harmonic of the IDT, whose frequency at the temperature of the experiment is $f_{\text{SAW}} \approx 1.57\text{ GHz}$. This corresponds to an acoustic period of $T_{\text{SAW}} = 1/f_{\text{SAW}} \approx 0.65\text{ ns}$ and an acoustic wavelength $\lambda_{\text{SAW}} = v_{\text{SAW}}/f_{\text{SAW}} \approx 2.57\text{ }\mu\text{m}$. As will be seen below, this wavelength fulfills the requirement $\lambda_{\text{SAW}} < l_d$ mentioned above for simultaneous acoustic transport of electrons and holes in the same direction with the well-defined SAW velocity.

Figure 6.2: (a) Reflection (S_{11}) and transmission (S_{21}) coefficients as a function of the frequency of the RF signal applied to the IDT of the acoustic delay line where the NWs are deposited. (b) Photoluminescence (PL) image of NW1 under homogeneous laser illumination. The plot shows the spectroscopically resolved PL (horizontal axis) as a function of the position along the nanowire (vertical axis). $x = 0$ corresponds to the edge of the NW closest to the IDT. The (In,Ga)As recombination centers are located at $x \approx 3 \mu\text{m}$ (grey dash-dotted line). We use a logarithmic color scale.

Several nanowires, grown on the same wafer and deposited on the same delay line, were studied in our experiments. Here, we show experimental results for two of them, denoted as NW1 and NW2. Figure 6.2(b) shows the PL emitted by NW1, in the absence of a SAW, as a function of the emission wavelength (horizontal scale) and position along the NW axis (vertical scale). The excitation was provided by a defocused laser beam covering the full NW. The PL image shows strong emission centers around 828 nm in the region $0 < x < 3 \mu\text{m}$ ($x = 0$ is defined as the lower end of the NW, the closest to the IDT generating the SAW), corresponding to the section containing the pure GaAs core. Additional, less intense, peaks at $x_r \approx 3 \mu\text{m}$ (dash-dotted line in Fig. 6.2(b)) with wavelengths above 850 nm correspond to recombination centers at the region where In is incorporated. The different emission wavelengths correspond

Figure 6.3: Spectrally (horizontal axis) and spatially (vertical axis) resolved PL images of NW1 under overall laser illumination at an RF frequency applied to the IDT of (a) 1.540 GHz and (b) 1.573 GHz. The color scale is the same in both images. (c) rf-frequency dependence of the PL emitted by the GaAs segment of NW1 (—■—) (integrated in the region indicated by the dashed rectangle in (a)) and the reflection curve (S_{11}) of the IDT around the frequency of the second harmonic at room temperature (—△—). All PL measurements were made at a nominal temperature of 20 K.

to centers with different sizes and In concentrations. Above the In-doped region, PL emission at the typical GaAs wavelength is recovered. Similar behaviour was observed for all NWs studied, with emission wavelengths for In-doped recombination centers extending to values even above 900 nm, as in the case of NW2, where the distance between the lower end and the position of the In-doped centers is $l_d \approx 5 \mu\text{m}$.

To determine the acoustic modulation efficiency at low temperatures, we recorded the PL emitted by the NWs as a function of the RF frequency applied to the IDT, when the NW is entirely illuminated by a defocused laser beam. Figures 6.3(a) and (b) show the detected PL emission in the GaAs segment of the NW1 for a constant RF power (P_{rf}) of 5 dBm and two different RF frequencies applied to the IDT. The

Figure 6.4: (a), (b) Spectral and spatially resolved images of two (In,Ga)As recombination centers at $x_r = 5 \mu\text{m}$ in NW2 under remote optical excitation by a tightly focused laser at $x = 0$, and for two powers: (a) $P_{rf} = -37$ dBm and (b) $P_{rf} = +3$ dBm. (c) Spectrally resolved PL emitted by the NW2 at x_r as a function of P_{rf} . The curves are vertically shifted for clarity.

RF frequency in Fig. 6.3(a) is out of the resonance of the IDT, while the one applied in Fig. 6.3(b) corresponds to the second harmonic of the FEUDT (cf. Fig. 6.2(a)). The lower PL intensity in Fig. 6.3(b) is attributed to the spatial separation of photo-excited carriers in the GaAs segment of the NW due to the type II modulation of the electronic band structure created by the SAWs, as illustrated in Fig. 6.1(b). Figure 6.3(c) shows the spatially and spectroscopically integrated PL spectra, from the emission region marked as a dashed rectangle in Fig. 6.3(a), as a function of the RF frequency. When the frequency of the RF signal is at IDT resonance, the generated SAWs suppress about 40% of the PL emitted when the RF frequency is out of resonance.

Figure 6.4 shows the spatially and spectrally resolved PL emitted by two (In,Ga)As

recombination centers of NW2 situated at $x_r = 5 \mu\text{m}$, when carriers are optically injected by a tightly focused laser beam at $x = 0$ and acoustically transported towards x_r . For very low SAW power (cf. Fig. 6.4(a)), the recombination center emitting at $\lambda_\alpha = 915 \text{ nm}$ (marked as *center α* on the image) is populated by carrier diffusion along the NW. When acoustic transport is activated (cf. Fig. 6.4(b)), the additional carriers transported by the acoustic wave also populate a second recombination center (marked as *center β*) with emission energy about 2.5 meV higher than the previous one (emission wavelength $\lambda_\beta = 913.4 \text{ nm}$). We probed the possible correlations between the two lines by measuring their intensity, in absence of SAWs, as a function of the laser power used to excite the centers: we observed a non-related behaviour, indicating that they are generated by independent emission centers with slightly different size and/or In content.

Figure 6.4(c) shows the PL evolution of these two centers as a function of P_{rf} . The onset of acoustic carrier transport, determined as the RF power at which *center β* begins to emit light, is observed at $P_{\text{rf}} = -7 \text{ dBm}$. Above this value, the intensity of *center β* continually increases until it reaches a maximum at $P_{\text{rf}} = 3 \text{ dBm}$, and then it decreases at higher P_{rf} . PL suppression at high acoustic powers is attributed to the fact that high piezoelectric fields are able to transport carriers towards the In-doped centers, but also to detrapp them, thereby preventing carrier recombination.

In order to unambiguously demonstrate that the PL modulation of Fig. 6.4 is induced by acoustic charge transport, we performed experiments where both the laser pulses and the RF signal are amplitude modulated (AM). By applying the same AM reference signal ($f_{\text{AM}} = 227 \text{ Hz}$) to both devices, and controlling the relative phase delay between them, we can choose between the simultaneous application of laser and SAWs (*in phase*), or the introduction of a π phase delay, so that optical excitation occurs during the half period time intervals in which SAW generation is suppressed (*out of phase*). Since the heat thermalization rate in our setup is expected to be smaller than the AM frequency, PL changes in the *out of phase* configuration are only related to the average temperature change induced on the sample by RF absorption. Measurements *in phase*, in contrast, also contain the effects induced by the acoustic transport of carriers.

Figure 6.5 shows the RF power dependence of the PL emitted by centers α and β for the *out of phase* (Fig. 6.5(a)) and *in phase* (Fig. 6.5(b)) configurations. In the *out of phase* case, an almost P_{rf} independent PL intensity curve is obtained. Even at

Figure 6.5: Spectrally integrated PL emitted by the recombination centers α and β of NW2 as a function of RF power for (a) *out of phase* synchronization of laser pulses and SAWs and (b) *in phase* synchronization. The lines are guide to the eye.

high P_{rf} values, RF-induced heating is only observed as a small reduction of the PL intensity of *center α*. In the *in phase* situation, both centers show the same behaviour as Fig. 6.4: a constant value for low acoustic powers, followed by a continuous increase of the PL intensity above a critical power of -7 dBm. Once the highest intensity is reached at $P_{max} = +3$ dBm, it decreases monotonically until it is totally quenched at +19 dBm. The intensity variation is especially pronounced in *center β*, where the acoustic transport of carriers at P_{max} enhances the PL by more than 400% with respect to the low power range.

Finally, we calculated the relative efficiency of the acoustic transport mechanism,

defined as

$$r_t = \frac{I_{\text{PL}}(\text{R})}{I_{\text{PL}}(\text{G}) + I_{\text{PL}}(\text{R})}. \quad (6.1)$$

Here, $I_{\text{PL}}(\text{G})$ and $I_{\text{PL}}(\text{R})$ are the PL intensities collected from the generation (G) and recombination (R) regions, that is, GaAs and (In,Ga)As segments, respectively, under SAW excitation. Figure 6.6(a) shows the spectrally integrated PL emission from both segments of NW1 as a function of P_{rf} , while Fig. 6.6(b) shows the values of r_t estimated from Fig. 6.6(a). The small r_t for $P_{\text{rf}} < -15$ dBm is due to carrier diffusion. Between -15 and -3 dBm, the acoustically induced quenching of the PL at G due to carrier ionization, together with the enhancement of the carrier density transported towards the recombination centers at R, contribute to an increase in r_t from barely 10% to a maximum acoustic transport efficiency of about 60% in this NW. Similar results are also observed for NW2. This is very similar to the one obtained using SAWs with lower frequencies in [13, 20], indicating that the high-frequency mechanism does not diminish the efficiency of the acoustic transport process.

Figure 6.6: (a) Acoustic power dependence of the spectrally integrated PL emitted by NW1 at the GaAs carrier generation region (G) and the recombination centers in the (In,Ga)As segment (R). (b) Relative efficiency of acoustic transport, r_t , as defined at Eq. 6.1, for the centers in panel (a).

6.4 Conclusion

In conclusion, we have demonstrated carrier transport along micrometric distances in GaAs nanowires by using a high-frequency SAW ($f_{\text{SAW}} = 1.57$ GHz) that fulfills $\lambda_{\text{SAW}} < l_d$. Under these conditions, the optically generated electrons and holes are trapped at the minima and maxima of SAW-modulated conduction and valence band, respectively, and are simultaneously transported in the same direction with the well-defined acoustic wave velocity. The relative transport efficiency of this acoustic transport mechanism is about 60%, of the same order as the values obtained at lower SAW frequencies.

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Appendices

Appendix A: Nanoimprint Template Design for Interdigital Transducer Devices

NIL templates were designed in education version of AutoCAD[®] software (from Autodesk, Inc.). Quartz NIL templates were manufactured by IMS chips (Germany). Drawings of the designs for Mask_1 (SF and FEUDT designs) and Mask_2 (DF design) are given in Fig. A.1 and Fig. A.2, respectively. Structures in the design appears as cavities with a structure depth 100 nm+/-10 nm. The dimension of the template is 65 mm $\times$ 65 mm $\times$ 6.35 mm with a mesa at the center. Height of the mesa which includes structures is 15 μ m. Design parameters of IDT devices can be seen in table A.1.

Table A.1: Design parameters of the IDT delay lines.

λ (nm)	Number of fingers	Aperture (μ m)	Distance between IDTs (μ m)
1000	1400	30	306.37 & 1081.77
800	1752	30	305.53 & 1081.77
640	2184	30	307.41 & 1081.77
520	2692	30	306.34 & 1081.77
500	1399	30	405.87 & 1081.87
400	1749	30	405.73 & 1081.87
320	2187	30	405.45 & 1081.87
260	2691	30	405.60 & 1081.87

Figure A.1: Drawing of NIL template used to fabricate SF-IDT and FEUDT devices.

Figure A.2: Drawing of NIL template used to fabricate DF-IDT devices.

Appendix B: Etching Recipe

Etching parameters used in Adixen AMS100DE apparatus for RIE is given in table B.1. These parameters were used at $-10\text{ }^{\circ}\text{C}$ with a sample holder position of 120 mm from the source.

Table B.1: Etching parameters for the materials.

Material	Gas flow (sccm)				Time (s)	CCP-ICP Power (W)	Pressure (mbarr)
	CHF_3	He	O_2	Ar			
HSQ	60	150	10	-	130	200-100	8×10^{-3}
WiDE-C + MonoMat	-	-	8	40	45	100-400	8×10^{-3}

Summary

This thesis deals with both the excitation and characterization of high-frequency SAWs, as well as with SAW-induced charge transport by using an electrical excitation method. SAWs are widely used and very important in both research and industry. In piezoelectric materials, they can be excited through the inverse piezoelectric effect by using interdigital transducers (IDTs). Because of the piezoelectricity of the material, a piezoelectric potential wave accompanies the mechanical wave. The unique SAW properties make them suitable not only for signal processing, but also for sensing applications, photonics, and charge transport. The SAW frequency is determined by the periodicity of the IDT electrodes and the acoustic velocity of the material. Almost for all applications, there is a strong demand for higher frequencies, for example to enhance the processing speed or to reach the single-charge manipulation regime.

Chapter 1 gives a brief introduction to SAWs with a review of breakthrough results, specifically on SAW-assisted charge carrier transport from the literature. In the second part of this chapter, some background for the rest of the thesis is given by introducing a mathematical description of SAWs, their interaction with charge carriers, SAW-induced modulation mechanisms of the electronic structure of semiconductors, and finally their generation and detection methods.

In Chapter 2, the experimental methods that have been used in the course of this research are introduced. The first section focuses on the fabrication methods and describes the nanoimprint lithography (NIL) process. A description of the etching and film formation techniques is also given. In the second half of the chapter, the characterization techniques are introduced, such as network analysis and the optical setup for the electron-hole pair transport measurements.

In Chapter 3, a method to fabricate sub-100 nm structures by using a planarization technique and an appropriate material configuration is introduced. This chapter specifically addresses the lift-off issues and critical-dimension control at the nanometer scale. It has been shown that global contact planarization with HSQ can be achieved at room temperature with a flat and blank template. This technique has given an improvement of 45 % compared to standard spin coating planarization. Because of the inorganic nature of HSQ, HSQ shows excellent etch selectivity with respect to or-

ganic underlying layers. This was demonstrated by pattern generation with UV-based NIL and reactive-ion etching (RIE) processes. Very precise control of the critical dimensions and well-defined undercut resist profiles were obtained, suitable for metal deposition followed by lift-off. HSQ contact planarization is a generic method and can therefore also be applied for other lithography techniques and multilayer applications because of its simplicity, fastness, low cost and efficiency.

In Chapter 4, ultrahigh frequency IDTs on a ZnO/SiO₂/Si multilayer system were fabricated, using NIL and electrical characterization was performed. Crucial to our novel approach is the application of the HSQ planarization layer mentioned above that provides excellent planarization combined with very high etching selectivity, critical dimension control and easy lift-off. The highest resonance frequency reported so far for ZnO-based transducers on silicon (16.1 GHz), was measured on an IDT with 65 nm finger width. Our experimental results are in very good agreement with numerical simulations of the specific multilayer system. The IDT performance is expected to improve after optimizing the ZnO growth parameters and lowering the Si substrate conductivity. Our technology has great potential to reach even higher frequencies in case of higher velocity substrates, such as SiC, sapphire or diamond.

In Chapter 5, ultrahigh frequency SAW devices up to 23.5 GHz were fabricated on SiO₂/ZnO/SiO₂/Si by using a CMOS-compatible fabrication process. Excited modes are well verified by numerical calculations. Piezoelectric field distribution calculations showed that the SAW-induced field is strong enough for efficient charge (both electron and hole) transport at the Si/SiO₂ interface. Our results show that the higher-order modes are advantageous to get higher acoustoelectric current owing to their higher frequency without losing the required mobility. Additionally, metal structures on the SiO₂ surface modulating the piezoelectric field, might be used as an acoustic charge guide for certain vibration modes to enhance the transport.

Finally, in Chapter 6, the oscillating piezoelectric fields accompanying SAWs were used to demonstrate carrier transport along micrometer distances in GaAs nanowires by using a high-frequency SAW ($f_{\text{SAW}} = 1.57$ GHz). The short wavelength of the acoustic modulation, smaller than the length of the nanowire, allows the trapping of photogenerated electrons and holes at the spatially separated energy minima and maxima of conduction and valence bands respectively, and their transport along the nanowire with a well-defined acoustic velocity towards indium-doped recombination centers. The relative transport efficiency of this acoustic transport mechanism

was found to be about 60%.

To conclude, the developed NIL fabrication technique can be implemented to generate ultrahigh frequency SAWs on top of different substrates. This method enables very high critical-dimension control on the devices. Integration of SAW-technology into silicon-based technology is of special interest for the realization of low-cost, integrated devices. Additionally, the importance of contactless manipulation of charge carriers has been pointed out with different proposals, such as the precise measurement of electron charge or single-photon generation. In this respect, the SAW frequency is of crucial importance for the device performance. We believe that this study provides an important contribution to future SAW applications.

Samenvatting

Dit proefschrift behandelt zowel de opwekking en karakterisatie van hoogfrequente akoestische oppervlaktegolven (Engels: surface acoustic waves, SAWs), als SAW-geïnduceerd ladingstransport door gebruik te maken van elektrische excitatie. SAWs worden veel gebruikt en zijn zeer belangrijk in onderzoek en in de industrie. In piëzo-elektrische materialen kunnen ze opgewekt worden door het inverse piëzo-elektrische effect door gebruik te maken van interdigitale transducenten (Engels: interdigital transducers, IDTs). Door de piëzo-elektrische eigenschap van het materiaal gaat de mechanische golf gepaard met een piëzo-elektrische potentiaal golf. De unieke eigenschappen van SAWs maken ze niet alleen geschikt voor signaalverwerking, maar ook voor sensortoepassingen, fotonica en ladingstransport. De SAW-frequentie wordt bepaald door de periodiciteit van de IDT elektrodes en de akoestische snelheid in het materiaal. Voor bijna alle toepassingen is er een sterke vraag naar hogere frequenties, bijvoorbeeld voor het verhogen van de snelheid of om het enkele ladingsmanipulatie regime te bereiken.

Hoofdstuk 1 geeft een korte introductie over SAWs met een overzicht van de belangrijkste resultaten uit de literatuur, in het bijzonder aangaande SAW-geassisteerd ladingstransport. In het tweede gedeelte van dit hoofdstuk, wordt er de nodige achtergrond gegeven voor de rest van het proefschrift in de vorm van een introductie van de wiskundige beschrijving van SAWs, hun interactie met ladingsdragers, SAW-geïnduceerde modulatiemechanismen van de elektronische structuur van halfgeleiders en tot slot hun opwekkings- en detectiemethodes.

In Hoofdstuk 2 worden de experimentele methodes geïntroduceerd die gebruikt zijn in de loop van dit onderzoek. Het eerste gedeelte focust op de fabricagemethodes en beschrijft het nano-imprintlithografie (Engels: nanoimprint lithography, NIL) proces. Ook wordt er een beschrijving van de ets- en filmformatietechnieken gegeven. In het tweede gedeelte van het hoofdstuk worden de karakteriseringsmethodes geïntroduceerd, zoals de netwerkanalyse en de optische meetopstellingen voor de elektron-gat-paar-transportmetingen.

In Hoofdstuk 3 wordt er een methode geïntroduceerd om sub-100 nm structuren te fabriceren door middel van een vereffeningstechniek (Engels: planarization)

en een geschikte materiaalconfiguratie. Dit hoofdstuk gaat met name in op de lift-off problemen en de controle van de kritische dimensies op de nanometerschaal. Er wordt aangetoond dat globale contact planarization met HSQ gerealiseerd kan worden bij kamertemperatuur met een vlakke, lege wafer. Deze techniek heeft tot een 45 % verbetering geleid vergeleken met standaard spin-coating planarization. Door het anorganische karakter van HSQ geeft HSQ uitstekende etsselectiviteit ten opzichte van de onderliggende organische lagen. Dit werd aangetoond door het genereren van patronen met UV-gebaseerde NIL- en reactieve-ionenets (RIE) processen. Zeer precieze controle van de kritische dimensies en goed gedefinieerde overhangprofielen van de resist werden verkregen, geschikt voor metaaldepositie gevolgd door lift-off. HSQ contact planarization is een generieke methode en kan daarom ook toegepast worden voor andere lithografietechnieken en multilaag-toepassingen dankzij de eenvoud, snelheid, lage kosten en efficiëntie.

In Hoofdstuk 4 werden IDTs met ultrahoge frequentie gefabriceerd op een ZnO/SiO₂/Si multilaag-systeem door middel van NIL. De elektrische karakterisering wordt beschreven. Cruciaal voor onze nieuwe aanpak is de toepassing van de hierboven beschreven HSQ planarization laag die voor een uitstekende planarization zorgt in combinatie met een hoge etsselectiviteit, kritieke dimensiecontrole en een gemakkelijke lift-off. De hoogste resonantiefrequentie tot dan toe gerapporteerd voor ZnO-gebaseerde systemen op silicium (16.1 GHz) werd gemeten op een IDT met 65 nm vingerbreedte. Onze experimentele resultaten zijn in goede overeenstemming met numerieke simulaties van dit specifieke multilaag-systeem. We verwachten dat de prestaties van de IDT zullen verbeteren na optimalisatie van de ZnO groeiparameters en het verlagen van de geleiding van het siliciumsubstraat. Onze technologie heeft grote potentie om zelfs hogere frequenties te bereiken in het geval van substraten met een hogere akoestische golfsnelheid, zoals SiC, saffier of diamant.

In Hoofdstuk 5 werden er ultrahoge frequentie SAW devices tot 23.5 GHz gefabriceerd op SiO₂/ZnO/SiO₂/Si door gebruik te maken van een CMOS-compatibel fabricageproces. Geëxciteerde modes worden goed beschreven door numerieke berekeningen. Berekeningen van de distributie van het piezo-elektrische veld laten zien dat het SAW-geïnduceerde veld sterk genoeg is voor efficiënt ladingstransport van zowel elektronen en gaten aan het Si/SiO₂ interface. Onze resultaten laten zien dat de hogere-orde modes gunstig zijn om een hogere akoesto-elektrische stroom te verkrijgen dankzij hun hogere frequentie zonder de vereiste mobiliteit te verliezen.

Daarnaast kunnen metalen structuren op het SiO_2 oppervlak die het piëzo-elektrisch veld moduleren, gebruikt worden als een geleider voor akoestische lading voor bepaalde vibratiemodes om het transport te verbeteren. Als laatste werden in Hoofdstuk 6 de oscillerende piëzo-elektrische velden behorende bij de SAWs gebruikt om ladingstransport te realiseren over micrometer afstanden in GaAs nanodraden door gebruik te maken van hoog-frequente SAWs ($f_{\text{SAW}} = 1.57 \text{ GHz}$). De korte golflengte van de akoestische modulatie, kleiner dan de lengte van de nanodraad, maakt het mogelijk om optisch gegenereerde elektronen en gaten op te sluiten in de ruimtelijk gescheiden energieminima en maxima van respectievelijk de geleidings- en valentieband. Tevens maakt de korte golflengte hun transport door de nanodraad met een goed-gedefinieerde akoestische snelheid richting de indium gedoteerde recombinatiecentra mogelijk. De relatieve transportefficiëntie van dit akoestische transport werd vastgesteld op ongeveer 60 %.

In conclusie, de ontwikkelde NIL fabricagetechniek kan gebruikt worden op verschillende substraten om ultrahoog frequente SAWs op te wekken. Deze methode maakt een hoge controle van de kritische dimensies mogelijk. Integratie van de SAW-technologie in silicium gebaseerde technologie is van bijzonder belang voor het realiseren van geïntegreerde devices tegen lage kosten. Daarnaast is er gewezen op het belang van contactloze manipulatie van ladingsdragers in verschillende voorstellen, zoals de precieze meting van de elektronlading of enkele-foton productie. In dit opzicht is de SAW-frequentie van cruciaal belang voor de deviceprestatie. Wij geloven dat deze studie een belangrijke bijdrage levert aan toekomstige SAW-toepassingen.

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Author Bibliography

Serkan Büyükköse was born in İzmit, Turkey, on December 17, 1982. He obtained his B.Sc. degree from Dokuz Eylül University, İzmir, Turkey, in 2005. Following, he obtained his M.Sc. degree from Department of Physics in İzmir Institute of Technology (IZTECH), Turkey, in 2009. During his M.Sc. study, he worked as teaching and research assistant at IZTECH. He carried out his M.Sc. work on “Electrical surface modification and characterization of metallic thin films using scanning probe microscope (SPM) nanolithography method” under supervision of Prof. Dr. Salih Okur. He joined NanoElectronics Group of the University of Twente in October 2009 as a PhD candidate and worked on the subject “Nanoimprinted High-Frequency Surface Acoustic Wave Devices: Generation, Characterization and Acousto-Electric Transport” under supervision of Prof. Dr. Wilfred G. van der Wiel. His doctoral research is described in this thesis.

List of Publications

1. **S. Büyükköse**, A. Hernández-Mínguez, B. Vratzov, C. Somaschini, L. Geelhaar, H. Riechert, W. G. van der Wiel and P. V. Santos *High-frequency acoustic charge transport in GaAs nanowires* Nanotechnology (2014)
(in press)
2. **S. Büyükköse**, B. Vratzov, J. van der Veen, P. V. Santos and W. G. van der Wiel *Ultrahigh-frequency surface acoustic wave generation for acoustic charge transport in silicon* Appl. Phys. Lett. **102**, 2 (2013)
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3. **S. Büyükköse**, B. Vratzov, D. Ataç, J. van der Veen, P. V. Santos and W. G. van der Wiel *Ultrahigh-frequency surface acoustic wave transducers on ZnO/SiO₂/Si layered structure realized using nanoimprint lithography* Nanotechnology **23**, 315303 (2012)
(<http://dx.doi.org/10.1088/0957-4484/23/31/315303>)
4. **S. Büyükköse**, B. Vratzov, W. G. van der Wiel *High-quality global hydrogen silsequioxane (HSQ) contact planarization for nanoimprint lithography* J. Vac. Sci. Technol. B **29**, 2 (2011)
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