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The Development of Integrated Chemical Microsensors in GaAs

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The Development of Integrated Chemical Microsensors in GaAs

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Abstract

Monolithic, integrated acoustic wave chemical microsensors are being developed on gallium arsenide (GaAs) substrates. With this approach, arrays of microsensors and the high frequency electronic components needed to operate them reside on a single substrate, increasing the range of detectable analytes, reducing overall system size, minimizing systematic errors, and simplifying assembly and packaging. GaAs is employed because it is both piezoelectric, a property required to produce the acoustic wave devices, and a semiconductor with a mature microelectronics fabrication technology. Many aspects of integrated GaAs chemical sensors have been investigated, including: surface acoustic wave (SAW) sensors; monolithic SAW delay line oscillators; GaAs application specific integrated circuits (ASIC) for sensor operation; a hybrid sensor array utilizing these ASICs; and the fully monolithic, integrated SAW array. Details of the design, fabrication, and performance of these devices are discussed. In addition, the ability to produce heteroepitaxial layers of GaAs and aluminum gallium arsenide (AlGaAs) makes possible micromachined membrane sensors with improved sensitivity compared to conventional SAW sensors. Micromachining techniques for fabricating flexural plate wave (FPW) and thickness shear mode (TSM) microsensors on thin GaAs membranes are presented and GaAs FPW delay line and TSM resonator performance is described.

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Introduction

Chemical sensing is an important technology in that it has a number of applications to Sandia's national security mission, including weapons nonproliferation, weapons treaty verification, chemical weapons detection on the battlefield, and public safety. Additionally, industrial process control, pollution prevention, and environmental remediation are significant commercial applications. In many of these applications, sensor size is a critical system constraint, where smaller size generally translates into lighter weight, smaller sample volumes, lower power consumption, and greater portability. Small sensors are also more readily arrayed into a system with a high degree of chemical discrimination, capable of operation in the complex chemical backgrounds found in real-world environments. However, a reduction in the size of the chemical transducer is not sufficient to reduce the overall sensor system size significantly, since system size is usually determined by the control electronics, electrical interconnections, and packaging. To bring about a significant reduction in system size, these other components must be considered as well. For chemical transducers that can be produced on semiconductor substrates, the monolithic integration of the transducer with its control electronics, through the use of integrated circuit (IC) microfabrication technology, can bring about the desired size reduction. Other benefits as well often accrue with monolithic integration, including enhanced manufacturability and reliability, minimized temperature dependence, simplified packaging and assembly, and reduced cost.

Gallium arsenide (GaAs) is an intriguing semiconductor material for the particular case of acoustic wave chemical sensors, when viewed in terms of integration. GaAs is intrinsically piezoelectric, which permits electrical generation of acoustic waves. A mature GaAs IC technology also exists, with commercial foundries able to provide application-specific integrated circuits (ASICs) from a customer's design. Coincidentally, this IC industry has driven two developments critical to integrated acoustic wave sensors: high purity, low defect density, crystalline GaAs substrates suitable for acoustic wave generation and high frequency analog ICs required to generate and detect the acoustic signals. Thus, the foundational technologies required for integrated GaAs acoustic wave chemical sensors are in place.

Acoustic wave chemical sensors operate at high frequencies, typically between 100 MHz and 500 MHz, because chemical sensitivity generally increases with acoustic frequency [1]. This frequency range underscores the importance of monolithic integration for this particular application. Because of a reduction in losses associated with transferring high frequency signals between discrete components, a monolithically integrated device that contains all high frequency electronic and acoustic components on a single substrate would have performance advantages in addition to the ones listed above. GaAs is particularly well suited for high frequency microelectronics integration, making complete direct current (DC) -in, DC-out operation on a single chip a realizable goal.

GaAs presents one final advantage for the development of acoustic wave chemical sensors. Extremely thin GaAs membranes with thickness on the order of 1 μm can be produced using micromachining techniques. These membranes can support acoustic modes with higher chemical sensitivity [1] than thicker substrates can. We have developed techniques for

producing these membranes and have studied the characteristics of the membrane acoustic modes.

In this report, we describe the development of integrated GaAs acoustic wave chemical sensors from the initial studies of GaAs surface acoustic wave (SAW) delay line sensors through the first integrated SAW oscillators and ultimately to the monolithically integrated, DC-in, DC-out SAW sensor array. We also describe our studies of acoustic modes in thin GaAs membranes.

Discrete GaAs SAW Delay Line Sensor

As shown in Figure 1, SAW delay line sensors consist of a piezoelectric substrate and two interdigitated transducers (IDTs) formed by photolithographic patterning of a thin metal layer. Application of an alternating voltage to the input transducer generates an alternating strain field that launches a surface acoustic wave. The acoustic wave travels along the substrate surface before being converted back into an electrical signal by the output transducer. The time delay resulting from the transit of the acoustic wave between the IDTs gives rise to name of these devices.

Figure 1: Schematic of a surface acoustic wave sensor.

The velocity and attenuation of the propagating wave are very sensitive to properties, such as mass, temperature, and viscoelasticity, of thin films formed on the device surface. For example, increases in surface mass loading cause decreases in wave velocity that can be used to detect picogram mass changes [1]. By coating the acoustic path with a material that sorbs a chemical analyte of interest, this sensitivity can also be used to develop chemical sensors [1-3].

When configured in an oscillator circuit, as shown in Figure 2, changes in the delay line acoustic velocity, ΔV_R , can be observed as a frequency shift, Δf , which is equivalent to a phase change, $\Delta\theta$. This oscillator approach converts a very small velocity change into a measurable frequency increment, which can be determined with parts per billion resolution. In the limit of small perturbation, the response to these surface changes can be expressed as:

$$\frac{\Delta f}{f_o} = \frac{\Delta\theta}{\theta_o} = \frac{\Delta V_R}{V_{Ro}} = -k_m \frac{\Delta m}{m_o} - k_T \frac{\Delta T}{T_o}, \quad (1)$$

in which k_m is the mass sensitivity and k_T is the temperature sensitivity. Figure 3 shows the frequency shifts experienced by GaAs and quartz delay line oscillators due to surface mass loading.

Figure 2: Block diagram of a traditional single-channel (2-port) SAW sensor system.

Figure 3: Frequency responses of poly(isobutylene)-coated 100 MHz GaAs and quartz SAW devices to part per million (ppm) level step challenges of perchloroethylene.

Comparisons of GaAs and quartz delay line oscillators show that GaAs devices have similar chemical sensitivities (Figure 3) with slightly higher insertion loss and good phase linearity [4]. Typically, ST-quartz is used as a substrate for SAW delay line chemical sensors because of its chemical stability and low thermal drift. GaAs does have higher thermal drift than quartz but this can be compensated for in sensor design (see discussion below). These results demonstrate that GaAs can be used for acoustic wave chemical sensors without accepting a significant penalty in sensor performance.

GaAs SAW delay lines are fabricated on (100) semi-insulating substrates with the delay lines oriented in the [011] direction. These substrates are standard for microelectronics applications and the [011] direction maximizes the coupling between electromagnetic and acoustic waves. These delay lines operate between 100 MHz and 500 MHz, where the pitch of the IDT finger pairs determines the frequency of the device. Typically, each IDT contains 50 finger pairs. The input and output IDTs are separated by 190 acoustic wavelengths and are 30

wavelengths wide. IDTs consist of a 500 Å to 1000 Å thick gold layer (depending on device frequency) on top of a 200 Å thick titanium adhesion layer, deposited by thermal evaporation. These devices are fabricated in Sandia's Compound Semiconductor Research Laboratory (CSRL). A photograph of a 100 MHz GaAs SAW delay line is shown in Figure 4. The entire device is approximately 1 cm long.

Figure 4: Photograph of GaAs SAW delay line chemical sensor. The delay line operates at 100 MHz. The sensor substrate is approximately 1 cm long.

Integrated SAW Delay Line Oscillator

To demonstrate the concept of monolithic integration of microelectronics and acoustic wave sensors, we have designed and fabricated a single-chip SAW delay line oscillator [5] in GaAs. Oscillator fabrication employs metal-semiconductor field effect transistor (MESFET) technology (see Figure 5), chosen because of its fabrication simplicity and wide commercial applicability. An epitaxial channel design was used in this work for convenience; ion implanted MESFETs are also suitable. The only significant modification to a conventional MESFET process is the addition of one processing step to deposit a metal film for IDT formation.

The GaAs-based oscillator consists of a four-stage MESFET amplifier combined in a feedback loop with a SAW delay line, as shown in Figure 6 for a 470 MHz design. This oscillator is a simple demonstration of technology that can be used for chemical sensing. Passive elements included capacitors and resistors. No inductors were used because of their large size at this frequency. The amplifier was designed for 40 dB of gain to overcome the unmatched insertion loss of the SAW delay line, as discussed in more detail below. On-chip bypass capacitors (not shown in the schematic) of 2 pF and 4 pF were included for the gate and drain supplies, respectively. A picture of a fabricated device is shown in Figure 7. The oscillator size is approximately 1.9 x 1.2 mm².

The oscillator was tested on wafer with a spectrum analyzer with V_d approximately 3 V and V_g approximately 0.3 V. The oscillator functioned at 471.86 MHz with narrow line operation, as shown in Figure 8. The power consumption was 50-60 mW and could be reduced significantly if the SAW delay line was properly impedance matched and by simple optimization

of the RF amplifier design. The 200 MHz and 350 MHz oscillators with a similar design were also operational. The chip size for these designs are $3.5 \times 1.6 \text{ mm}^2$, and $2.1 \times 1.3 \text{ mm}^2$, respectively. The SAW delay line represents a larger fraction of the chip size for the lower frequencies. However, electronic support functions are likely to take up more space in more complex monolithic sensors.

Figure 5: A schematic representation of a self-aligned GaAs MESFET process for monolithic integration with acoustic wave devices.

The insertion loss of the 470 MHz oscillator was measured to be 30-35 dB, which is about 3 dB higher than for ST-quartz in a similar configuration. A lower insertion loss would be desirable so that a lower amplifier gain can be used. In this work, a large part of the insertion loss comes from reflections due to the lack of impedance matching. For applications requiring noise performance such as RF filters, matching with an L-C network is essential; for sensor applications, it is arguable that the extra space and complexity of adding inductors is not justified. Phase differences between reference and sensor delay lines are easily measured even if the oscillation frequency is not predictable. At these frequencies, an extra gain stage with MESFETs, capacitors, and resistors is more space conserving as compared to the inductors required for a proper impedance match, and for this initial demonstration offered less risk. As oscillator frequencies become higher, electronic gain becomes more difficult and inductor-based matching networks become smaller and more desirable.

Figure 6: Schematic of a 470 MHz SAW oscillator circuit.

Figure 7: A microphotograph of a 470 MHz GaAs SAW oscillator. The die is 1.9 mm by 1.2 mm

Figure 8: The frequency spectrum of a GaAs SAW oscillator operating at 471.86 MHz.

SAW Sensor Array

Because of the limited selectivity of the sorbing coatings applied to SAW chemical sensors, a single SAW sensor is insufficient to identify analytes in a realistic background of interferants. In practice, an array of sensors, each with a different selective coating, is used to make reliable chemical identification. An array of sensors also allows redundancy and error-checking, important advantages in the overall system. In this section, we discuss 3 array configurations: a 2-element array, to present the electronics design approach and demonstrate temperature compensation; a hybrid 4-element array with chemical sensitivity data; and the monolithically integrated, 4-element GaAs array.

Temperature Compensated Dual Sensor System

Dual sensor approaches are commonly used to compensate for undesired sensor responses. In a classic dual sensor system, two sensors are maintained in identical environments except one is introduced to the desired stimulus. The resulting responses (frequencies) can then be subtracted to allow for removal of the undesired background responses due to temperature or other surface changes.

Shown in Figure 9 is a block diagram of a novel dual-sensor approach that allows for integration and can be scaled to larger array size. In this system, phase is compared (subtracted) rather than frequency. One SAW device (Ch R) is used as a reference channel and is assumed isolated from the surface stimulus of interest. The reference oscillator drives a second acoustic channel (termed test channel, Ch T) on the same substrate. Note the unique use of the acoustic wave propagating in either direction from the center IDT to provided the necessary power split

of the reference and test channels. Multiplying the test signal with the reference signal and observing the DC component of the result performs the phase difference [6]. A parasitic second harmonic is present at the phase output and is ignored (filtered off). It should be noted that this approach has converted the velocity change into a voltage change. This allows for easy measurement with low-cost electronics, but also undoubtedly loses resolution compared to a time measurement.

Figure 9: Block diagram of a dual-channel (3-port) SAW sensor system.

This design compensates for changes in the sensor temperature. Since the two acoustic paths are on the same substrate, they can be assumed to be at exactly the same temperature. Therefore, referring back to Equation (1), assuming that only the test channel only “sees” the mass uptake; the temperature effects will exactly cancel and only the mass uptake of the test channel will be present at the output. This compensation is almost perfect except for the slight change in mass sensitivity as the reference channel center frequency changes over temperature. Additionally, this approach is tolerant to “mode-hopping” (changes in the longitudinal mode number) due to the subtractive nature of the technique. In practice, the necessary application of the chemically selective thin film to the test channel will change the temperature coefficient of the test channel and compromise the complete elimination of the temperature terms in the subtraction. This unfortunate effect will be an issue for any measurement approach and substrate material.

Figure 10 shows a comparison of the temperature dependence of a single GaAs delay line oscillator and the dual delay line approach. The oscillator output has been converted from a frequency shift to a phase shift to aid in the comparison. The oscillator exhibits the relatively large (compared to ST-quartz) temperature coefficient of approximately 50 ppm/°C typical of GaAs. The phase difference between the two channels of the dual delay line is almost independent of temperature. The residual temperature dependence in this device is probably due to thermal effects in the amplifier.

(a)

(b)

Figure 10: (a) Photograph of the reference/sensor dual delay line device. The central IDT launches SAWs both to the left and right where they are detected by the IDTs at either end. This dual delay line device is approximately 2 mm long. (b) Comparison of the temperature induced phase shift of the dual delay line device to a single delay line oscillator.

Electronics Design for the Four-Element SAW Array

The dual-channel temperature compensated SAW sensor approach can be easily extended to large arrays of acoustic sensor channels. A schematic of a four-element SAW delay line array configuration is shown in Figure 11. A large bi-directional central transmitting IDT spans each of four smaller receiving transducers. Appropriate chemically sorbent coatings are applied to three of the regions between the transmitting IDT and the receiving IDTs, with the fourth left uncoated to provide a reference phase for the array. As in the dual sensor system, an oscillator circuit drives the transmitter, launching an acoustic wave to the receivers. Phase comparator circuitry measures the relative phase of each of the receivers with respect to the reference and provides a DC output voltage proportional to the relative phase. The block diagram of a four-channel system, including electronic circuitry, is shown in Figure 12. Extension to n -channels simply requires splitting the reference signal $2n-1$ ways (n inputs for each acoustic channel and mixer drive for $n-1$ channels).

Figure 11: Schematic of an array of four surface acoustic wave devices on a single substrate.

Figure 12: Block diagram of a four-channel (5-port) SAW sensor system.

The electronics diagramed in Figure 12 incorporate several novel design features. All signals represented by single lines in this figure are implemented differentially in the actual design. The amplifier blocks shown as triangles are specially designed limiting differential amplifiers providing small phase errors at large over-drive conditions. The amplifiers also provide a log output signal, which allows for an accurate power measurement of the signals from each SAW output port. This permits measurement of the insertion loss of each SAW channel. The mixers are implemented as Gilbert cell mixers, which homodyne the test signal to DC. The input signals to the mixers are hard limited (square waves) by the amplifiers so in effect the mixer operates digitally, performing the exclusive-or function. This provides linear phase detection and provides two-quadrant phase detection as shown in Figure 13. This detection response provides a unique output if the two signals' phase difference remains within the appropriate two quadrants of phase space (1 and 2 or 3 and 4).

Figure 13: Diagram of the normalized DC component from the Gilbert cell mixer (XOR).

To achieve the desired full four-quadrant phase detector, the phase "dither" circuits as shown in the block diagram were added. The phase dither circuits allow for a given test channel to be selectively advanced or retarded in phase by a fixed predetermined amount. Inputs are

provided to cycle through three phase states to determine the phase uniquely over the complete 2π phase space. Operation of the dither sequence can be illustrated by an example shown by the three labeled points in Figure 13. Assume the actual phase is at the point A. From a single DC output measurement, one cannot determine if the actual phase is in quadrant #2 or quadrant #3 (normalized output is the same at points A and B). Using the phase dither, the response curve can be "shifted" to determine the "slope" of the output and hence the correct quadrant. Assuming that the dither shifts the output from point A to point C, the negative slope indicates the actual point is in quadrant #2. Three dither states are provided to handle the event where the measured and dithered points straddle a quadrant boundary (as with points A and B in Figure 13). In general all three dither states must be considered to guarantee unique phase detection over all phase space.

Hybrid Four-Element SAW Array

An array of SAW sensors and the associated microelectronics was first produced using a hybrid packaging approach rather than through monolithic integration [7]. Although it requires more assembly than the monolithic approach, the hybrid device prototypes can be produced more rapidly and can be used to test the sensor design and circuit functionality. It also represents a significant size reduction compared to conventional SAW sensor systems.

The SAW array is fabricated on a ST-quartz substrate. In this case, quartz was selected because the requirement for monolithic integration was removed and quartz has slightly better acoustic performance than GaAs. The oscillator amplifier and phase comparator circuitry are custom GaAs ASICs attached directly to the quartz substrate. Circuit interconnections are made by patterning metal paths directly onto the quartz die. Wire bonding is used to connect the ICs to the metal paths on the quartz. Although not monolithically integrated, this device incorporates all high frequency components on the quartz substrate so that the packaged part operates in the desired DC in/DC out mode. A photograph of the multi-chip SAW array configuration is shown in Figure 14. The hybrid device requires 90 mA at 2.5 V to operate the GaAs ASICs, which can readily be provided by batteries. The SAW oscillator operates at 510 MHz and the phase comparators have a sensitivity of 1 V per 180 degrees of phase.

These devices have been tested as chemical sensors. The response of the SAW array to dimethyl methyl phosphonate (DMMP) is shown in Figure 15. The varied response of the 3 different coatings indicates their relative sensitivity to DMMP and demonstrates how the array can be used to discriminate among a number of analytes and interferants.

Figure 14: Hybrid version of SAW microsensor array. The ST-quartz die size is 6.9 mm by 8.6 mm. Each of the GaAs ICs is approximately 1 mm x 2 mm. The device operates in a DC in/DC out mode.

Figure 15: Hybrid SAW array response to 15 parts per million of DMMP. The three microsensor coatings were BSP3 Hydrogen-bond acid [8], Ethyl cellulose, and OV-275 (a cyano modified polysiloxane).

Monolithic SAW Sensor Array

We have completed fabrication of the first fully monolithic version of the SAW sensor array described above. This device is based on a GaAs SAW array of the same configuration shown in Figures 11 and 12 and uses the same GaAs microelectronic circuitry as the die shown in Figure 14, but puts all these components onto a single GaAs substrate. Figure 16 (a) shows one of the four-element arrays with the four delay lines arranged diagonally across the center of the die and the amplifier and phase comparator circuitry placed in the corners. The die measures 4.6 mm by 4.6 mm. The Sandia-designed microelectronics are fabricated on the monolithic devices at a commercial GaAs IC foundry. The partially processed wafers are transferred to the CSRL for IDT fabrication. Prior to IDT fabrication, a plasma etch is used to remove several microns of dielectric material to expose the GaAs surface. The IDTs are then patterned on the GaAs surface using a metal lift-off process. Figure 16(b) shows the frequency spectrum of the delay line oscillator portion of the device, which operates at approximately 692.5 MHz and draws 28.5 mA at 3 V_{dc}. Oscillator function demonstrates that the post-processing of the IDTs is compatible with the microelectronics fabrication process. The temperature dependence of the phase comparator output has been tested and this is insensitive to temperature, as expected. These devices are now being packaged for further electronic and chemical testing.

Figure 16: Monolithic GaAs SAW sensor array. (a) Photograph of die showing IDTs in the center and microelectronics in the corners. Die size is 4.6 mm x 4.6 mm. (b) Frequency spectrum of integrated oscillator, demonstrating successful delay line integration.

Micromachined GaAs Membranes

As a sensor material, GaAs has advantages beyond the integration of acoustic wave transducers and microelectronics. With the application of micromachining processes analogous to those found in Si micro-electro-mechanical systems (MEMS) technology, it is possible to produce freely suspended GaAs membranes that retain all the crystalline properties of bulk GaAs [9]. We have used this approach to fabricate acoustic wave devices on piezoelectric GaAs membranes as thin as 0.5 μm [10]. There are two advantages to this approach [1, 11]. The first is that it is possible to launch acoustic modes in these membranes, specifically, flexural plate

waves (FPW) and thickness shear modes (TSM), that will propagate when the sensor is submerged in a liquid. The SAW mode is highly damped in a liquid and does not propagate along the sensor substrate. The second advantage is that the sensitivity to added mass for each of these modes increases with decreasing membrane thickness. Potentially, FPW and TSM acoustic wave chemical sensors can be made much more sensitive than technically realizable SAW sensors.

Flexural Plate Wave Delay Lines

FPW delay lines are similar in construction to SAW delay lines in that they comprise two sets of IDT electrodes separated by some distance. There is one significant difference, however. FPW devices are formed on piezoelectric membranes for which the thickness of the membrane is small compared to the acoustic wavelength. SAW devices exist in the opposite limit for which the acoustic wavelength is small compared to the substrate thickness. FPW devices have a particular advantage relative to chemical sensing. In the FPW limit, the sensitivity of the device increases as the reciprocal of the membrane thickness [1] while the SAW device sensitivity scales with the acoustic wavelength. SAW sensitivity is therefore limited by the resolution of the microlithography process which sets a lower limit on the intra-IDT spacing and, hence, the SAW wavelength. FPW sensitivity is limited by the fracture strength of the membrane and is potentially many times more sensitive than a SAW device.

FPW membranes are formed from GaAs epitaxial layers using a process similar to surface micromachining of Si wafers. First, a layer of AlGaAs (typically 70% Al) is epitaxially grown on a GaAs substrate. This serves as the sacrificial layer in the surface micromachining process (see Figure 17). A GaAs membrane layer is then grown on top of the AlGaAs layer. Each layer is on the order of 1 μm thick. The FPW IDTs are fabricated on the top GaAs layer using conventional microlithographic techniques. As with the SAW devices, the FPW IDTs must be oriented so that the FPW propagates along the [011] direction of the (100) wafer. Small openings are then photolithographically patterned and etched through the GaAs layer down to the AlGaAs layer using any one of a number well known GaAs etches [12]. The device is then immersed in a liquid selective etchant that preferentially removes the AlGaAs layer under the FPW GaAs membrane. HF acid solutions are employed because of their high etch selectivity of AlGaAs relative to GaAs [12, 13]. The devices are then rinsed and dried. Figure 18 shows an example of a fabricated GaAs FPW delay line.

Figure 17: Schematic cross-section of epitaxial layers used in GaAs membrane fabrication. (a) Starting material. Epitaxial layers are typically 1 μm thick. (b) After sacrificial AlGaAs etch, showing suspended GaAs membrane.

Figure 18: (a) Photomicrograph of two FPW delay lines (top view). The openings in the top GaAs layer can be seen above and below the IDTs. The delay lines are approximately 1.5 mm long and 0.3 mm wide. (b) Schematic cross section of the FPW delay line. The openings in the GaAs epitaxial layer are not shown.

Drying these thin membranes poses the same problems faced by Si micromachining. As the rinse liquid wicks away from underneath a membrane, surface tension at the liquid/air meniscus is sufficient to pull the membrane down to the substrate where it sticks permanently. This problem is avoided by sublimation drying. After etching, the devices are rinsed first in isopropyl alcohol and then in cyclohexane. While still wet, the devices are cooled to 1°C on a thermoelectric cooler in a N₂ atmosphere, freezing the cyclohexane. The cyclohexane sublimates over a period of a few minutes, leaving the devices dry and unstuck. Examples of these membranes after drying are shown in Figure 19. Note that no bowing is observed in the cantilever structures, evidence of the absence of residual stress in the GaAs membrane. This is the result of lattice-matched epitaxial growth in the GaAs/AlGaAs material system.

Since FPW IDTs are fabricated before the membrane is formed, it is possible to test both SAW and FPW propagation with the same device. The results of one such measurement is shown in Figure 20. Prior to the release etch that forms the membrane, the delay line operates in the SAW mode with a peak in its frequency response at 320 MHz. After the release etch, the peak in the frequency response shifts to 220 MHz. This shift to lower frequency is one characteristic of FPW modes and results from the reduced acoustic phase velocity for FPW modes relative to SAW modes.

FPW phase velocity scales with t/λ , where t is the membrane thickness and λ is the FPW wavelength. FPW velocity goes to zero for small t/λ and approaches the SAW value in the limit of large t/λ . For a given material, FPW velocity falls on a single curve for all values of t and λ , when plotted against t/λ . This dependence on t/λ is a clear indication of FPW mode propagation. A comparison of FPW phase velocity data to the theoretical prediction is shown in Figure 21 for several membrane thicknesses. Phase velocity is determined by multiplying the frequency corresponding to the maximum response (as shown in Figure 20) by the wavelength (determined by the IDT spacing). This is just a restatement of the relationship $V = f\lambda$. The theoretical curve is completely determined by GaAs materials properties. As such, there are no adjustable parameters to fit to the data. As seen in the Figure 21, the agreement between theory and experiment is excellent. FPW delay line chemical sensors are now being fabricated.

Figure 19: Scanning electron micrographs of GaAs membranes for FPW fabrication. The lower micrograph shows a 3 mm x 0.5 mm membrane attached to the substrate only at its corners. The upper right micrograph shows a 200 μm x 40 μm cantilever attached to the substrate only along its base. The cantilever exhibits no bowing along its length, evidence of the lack of stress in the epitaxial layer. The upper left micrograph enlarges the edge of the membranes, showing the gap between the membrane and the substrate where the AlGaAs layer has been removed.

Figure 20: Comparison of SAW and FPW frequency response. Measurements are taken on the same device before (SAW) and after (FPW) the membrane release etch. The shift to lower frequency is characteristic of the FPW mode, the result of a reduced acoustic phase velocity.

Figure 21: Comparison between measured FPW phase velocity and theoretical prediction for three membrane thicknesses (0.5, 1.0, and 2.8 μm) and three acoustic wavelengths. No adjustable parameters are used to fit the data.

Thickness Shear Mode Resonators

TSM resonators share three important characteristics with FPW delay lines that make them attractive for sensor applications. First, the sensitivity to added surface mass increases as the thickness of the substrate (or membrane) decreases. Second, this acoustic mode can be excited by electrodes placed on a single surface, simplifying fabrication. And, finally, TSM resonators can operate in both liquids and gases. There are two significant differences, as well. TSM resonators can function as single-port devices, simplifying the circuitry required for operation. TSM resonant frequency also increases as the membrane thickness decreases, pushing the operating frequency above 1 GHz (see Eq. 2 below) for the membranes considered in this project. These characteristics of TSM resonators make them attractive candidates for integrated chemical microsensors.

Using the same micromachining techniques described for FPW devices above, it is possible to create TSM resonators in GaAs membranes, where the two surfaces of the membrane act as acoustic “mirrors” to trap acoustic energy within the membrane. For GaAs TSM resonators, the resonant frequency is given by

$$f = 1.67/t \quad (2)$$

where the resonant frequency, f , is in gigahertz, the membrane thickness, t , is measured in microns, and the prefactor, 1.67, is determined by the acoustic shear velocity in GaAs [11]. Our measurements of the resonant frequency are in good agreement with the predicted result for the 0.5 μm thick GaAs membranes. Figure 22 shows the results for two membranes with different electrode patterns. The slight shift in frequency is likely due to different mass loading produced by the two electrodes. Measurements are made with a high frequency network analyzer.

Figure 22: TSM resonances for two 0.5 μm thick GaAs membranes. Predicted resonant frequency is 3.34 GHz.

Multiple resonances were observed in several 1.0 μm membranes, making identification of the fundamental mode difficult (see Figure 23). From Eq. 2, the expected resonance is at 1.67 GHz. In these devices as well, variations in surface mass loading due to differences in the IDT patterns are expected to shift the resonant frequency slightly.

We did not observe a resonance in the 3.0 μm membranes near the predicted resonant frequency of 557 MHz. We suspect that coupling efficiency between the electromagnetic signal and the acoustic wave decreases as the membrane thickness increases, perhaps due to the electrode geometry. Modeling studies are underway to evaluate the electrode design.

Figure 23: TSM resonances in 3 different 1.0 μm thick GaAs membranes. Multiple resonances are observed in individual devices. The predicted resonant frequency is 1.67 GHz.

Summary

We have described the development of microfabricated GaAs SAW chemical sensors, culminating in a fully monolithic device that integrates all high frequency components onto a single GaAs substrate. We have also demonstrated novel FPW delay lines and TSM resonators on thin GaAs membranes, fabricated with surface micromachining techniques. These devices can operate in liquids and gases and are potentially more sensitive chemical detectors than SAW devices.

References

- [1] D. S. Ballantine, Jr., et al., Acoustic Wave Sensors: Theory, Design, and Physico-Chemical Applications, Academic Press, New York, 1997.
- [2] G. C. Frye, et al., J. Inst. of Env. Sci. 34, 30, 1996.
- [3] J. W. Grate, et al., Anal. Chem. 65, 1868, 1993.
- [4] G. Frye, R. Kottenstette, E. Heller, J. Brinker, S. Casalnuovo, A. Sellinger, N. Raman, and Y. Lu, "Optimizing Surface Acoustic Wave Sensors for Trace Chemical Detection", *Proc. Transducers '97*, 2, p. 1322, IEEE, Piscataway, NJ, 1997.
- [5] A. G. Baca, E. J. Heller, V. M. Hietala, S. A. Casalnuovo, G. C. Frye-Mason, J. F. Klem, and T. J. Drummond, "Development of a GaAs-Based Monolithic Surface Acoustic Wave Integrated Chemical Microsensor," IEEE J. of Solid State Circuits, 34(9), 1254-1258, 1999.
- [6] Recall that: $\sin(\omega t + \theta_1)\sin(\omega t + \theta_2) = \frac{1}{2}(\cos(\theta_1 - \theta_2) - \cos(2\omega t + \theta_1 + \theta_2))$.
- [7] E. J. Heller et al., Proceedings of ECS, October, 1999.
- [8] BPS3 coatings provided by Jay Grate, Pacific Northwest National Laboratories.
- [9] K. Hjort, "Gallium Arsenide Micromechanics: A Comparison to Silicon and Quartz", Alta Frequenza-Rivista di Elettronica, vol. 6, pp. 48-54, 1994.
- [10] S.A. Casalnuovo et al., "Acoustic Wave Chemical Microsensors in GaAs", in Proceedings of the SPIE Conference on Micromachined Devices and Components IV, vol. 3514, 1998, pp. 103-110.
- [11] J. Soderkvist and K. Hjort, "The Piezoelectric Effect of GaAs Used for Resonators and Resonant Structures", Journal of Micromechanics and Microengineering, vol. 4, 1994, pp. 28-34.
- [12] C. I. H. Ashby, "Etching", *Properties of Gallium Arsenide, Third Edition*, M. R. Brozel, G. E. Stillman, eds., Chapter 18, INSPEC, London, 1996.
- [13] K. Hjort, "Sacrificial etching of III-V compounds for micromechanical devices", *J. Micromech. Microeng.*, 6, pp. 370-375, 1996.