Acoustic wave chemical microsensors in GaAs

Stephen A. Casalnuovo, Edwin J. Heller, Vincent M. Hietala, Albert G. Baca, Richard Kottenstette, Susan L. Hietala, John L. Reno, and Gregory C. Frye-Mason

Sandia National Laboratories, Albuquerque, NM 87185-0603

ABSTRACT

High sensitivity acoustic wave chemical microsensors are being developed on GaAs substrates. These devices take advantage of the piezoelectric properties of GaAs as well as its mature microelectronics fabrication technology and nascent micromachining technology. The design, fabrication, and response of GaAs SAW chemical microsensors are reported. Functional integrated GaAs SAW oscillators, suitable for chemical sensing, have been produced. The integrated oscillator requires 20 mA at 3 V_{DC}, operates at frequencies up to 500 MHz, and occupies approximately 2 mm^2. Discrete GaAs sensor components, including IC amplifiers, SAW delay lines, and IC phase comparators have been fabricated and tested. A temperature compensation scheme has been developed that overcomes the large temperature dependence of GaAs acoustic wave devices. Packaging issues related to bonding miniature flow channels directly to the GaAs substrates have been resolved. Micromachining techniques for fabricating FPW and TSM microsensors on thin GaAs membranes are presented and GaAs FPW delay line performance is described. These devices have potentially higher sensitivity than existing GaAs and quartz SAW sensors.

Keywords: gallium arsenide, aluminum gallium arsenide, microsensor, surface acoustic wave, flexural plate wave, micromachining, integration

1. INTRODUCTION

GaAs is an intriguing material for microsensor and microactuator applications. Its range of physical transduction mechanisms coupled with the ability to grow chemically distinct, lattice-matched AlGaAs epitaxial layers makes possible a variety of micromachined devices[1]. In particular, GaAs is a rational choice for acoustic wave chemical microsensors[2] because of its inherent piezoelectricity. In earlier work[3] we have demonstrated that GaAs surface acoustic wave (SAW) devices have chemical sensitivities comparable to more widely studied quartz SAW devices. Other acoustic modes such as flexural plate waves (FPW) and thickness shear modes (TSM) have potentially higher chemical sensitivities but require thin piezoelectric membranes for generation. These devices can be produced in GaAs using micromachining techniques.

Monolithic integration of GaAs acoustic wave devices with GaAs microelectronics is important for a number of reasons, including those typically associated with integration: smaller size, increased reliability, lower power, and simplified packaging. In addition, improved performance is expected for these microsensors, which operate in the range from 100 MHz to 1 GHz, if integration permits all high frequency signals to be kept on a single chip. GaAs is particularly well suited for high frequency microelectronics integration, making complete DC-in, DC-out operation on a single chip a realizable goal.

Chemical microsensors pose packaging challenges not commonly found in other micromachine and microelectronics applications. Nanoliter to microliter volumes of liquid and gaseous analytes must be routed to the microsensor without interfering with other electronic components. This requires that fluid flow channels be attached directly to the microsensor die in a manner that does not compromise the acoustic or electronic function of the device.

In this report, we describe our efforts to produce integrated GaAs SAW chemical microsensors, high sensitivity micromachined GaAs FPW acoustic devices, and packaging technology for fluid transfer.

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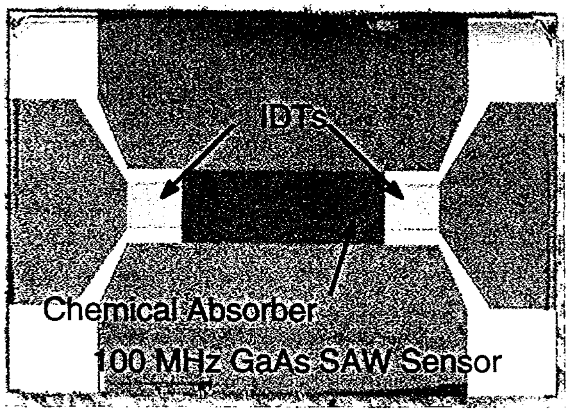
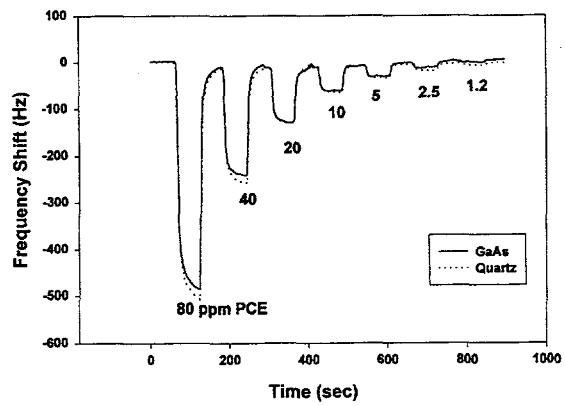
2. INTEGRATED GaAs SAW DELAY LINE OSCILLATOR FOR CHEMICAL SENSING

The application of GaAs SAW delay lines to chemical sensing has previously been reported[3]. These devices have chemical sensitivities almost identical to quartz SAW delay lines, which are the most commonly used acoustic wave sensor for gaseous chemicals. Figure 1, taken from Ref. 3, shows a picture of the GaAs SAW delay line sensor and a comparison of GaAs and quartz sensor response. The Au interdigital transducer (IDT) electrodes were produced using conventional microlithographic techniques compatible with microelectronics fabrication. For most efficient operation, the SAW must propagate along the [011] direction of (100) semi-insulating GaAs substrates[1].

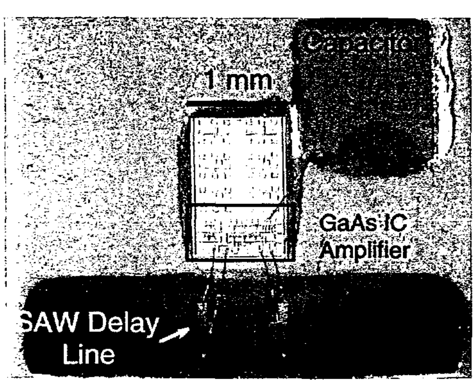


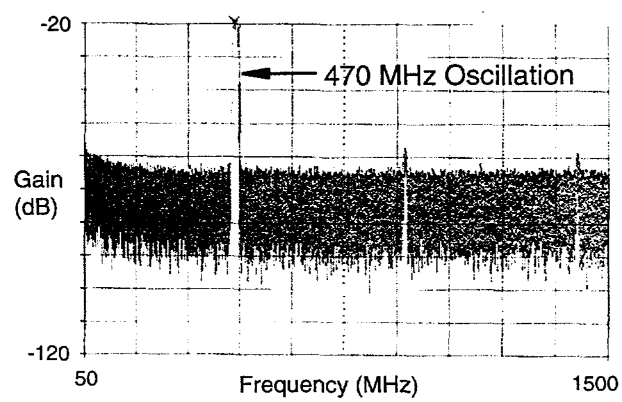


(a) (b)

Figure 1 GaAs SAW delay line chemical sensor. (a) Photograph showing IDT electrodes and selective chemical absorber. (b) Comparison of GaAs and quartz SAW sensor response to perchloroethylene (PCE). GaAs sensor response is essentially identical to conventional quartz sensor response. Both devices are coated with similar thicknesses of polyisobutylene which provides the sensitivity to PCE.

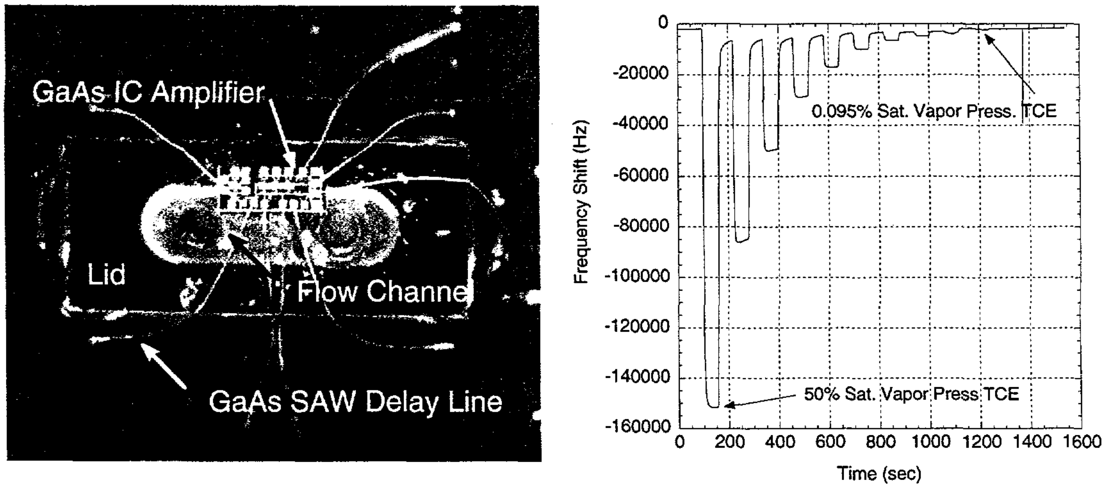
As the next step in pursuit of the fully integrated microsensor, GaAs IC amplifiers were designed and fabricated to drive the delay lines. Figure 2 shows the amplifier and the frequency response of the oscillator produced by wiring a GaAs SAW delay line in the feedback path of the amplifier. These two components were then packaged with a miniature gas flow channel (see discussion of packaging in Section 3) and tested as a chemical sensor as shown in Fig. 3. Chemical sensitivity to the gaseous analyte is conferred by coating the delay line with a selective absorbing material[2]. As the analyte is absorbed by this film, changes in the film's mass and viscoelastic properties cause a shift in the oscillator's frequency.





(a) (b)

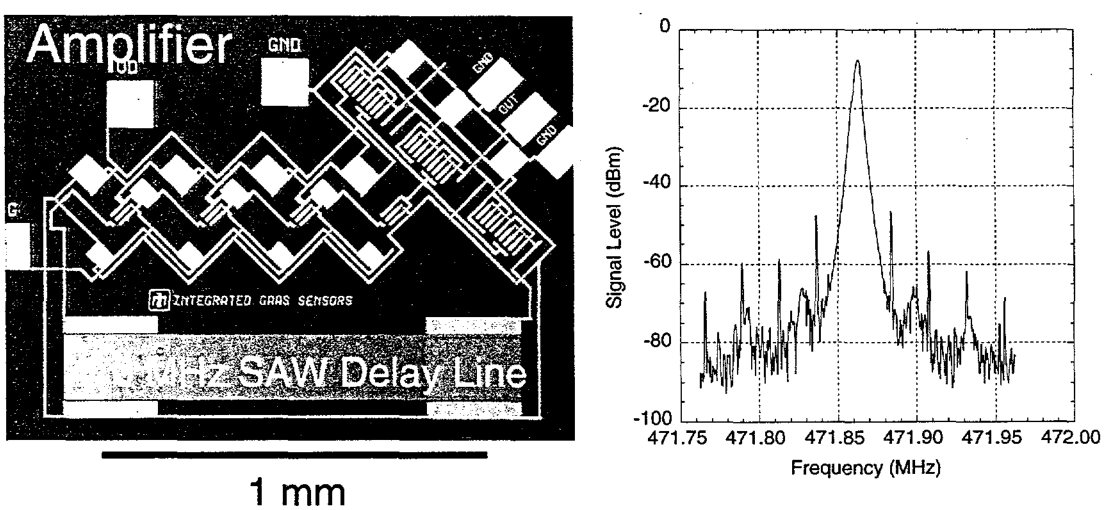
Figure 2 GaAs SAW delay line oscillator. (a) Photograph showing discrete GaAs IC amplifier wire-bonded to SAW delay line. (b) Frequency response demonstrating 470 MHz resonance of hybrid oscillator.



(a) (b)

Figure 3 (a) Photograph of the hybrid GaAs SAW oscillator packaged as a chemical sensor, showing lid with flow channel, delay line, and IC amplifier. (b) Sensor response to varying levels of trichloroethylene (TCE) vapor.

To achieve the DC-in operation desired in the fully integrated sensor, a monolithically integrated GaAs SAW delay line oscillator stage has been developed. This device incorporates both the delay line and the amplifier on a single GaAs substrate. Figure 4 shows this integrated device and its frequency response. The integrated oscillator requires 20 mA at 3 V_{DC}, operates at frequencies up to 500 MHz, and occupies approximately 2 mm^2. It is currently being packaged for testing as a chemical sensor.

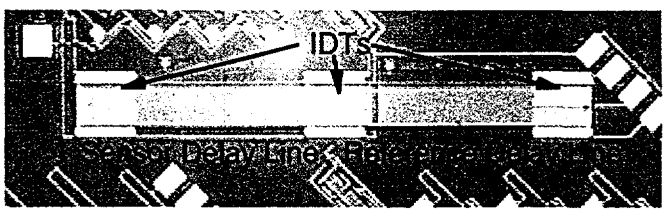


(a) (b)

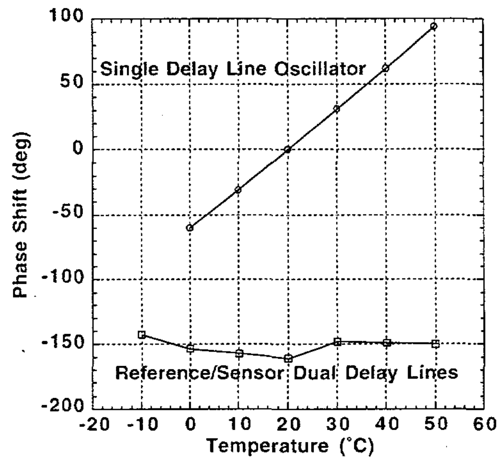
Figure 4 (a) Photograph of the monolithically integrated GaAs SAW oscillator. This device requires 20 mA at 3 V_{DC} to operate. (b) Oscillator frequency response demonstrating sharp resonance at approximately 472 MHz.

To achieve the desired DC-out operation, a high frequency phase comparator stage has been developed. This component monitors the phase difference between a delay line sensor and a reference delay line and outputs a DC voltage proportional to this phase difference. The phase delay of the sensor channel varies with the mass of the analyte absorbed by the chemically selective layer on the delay line, in a manner similar to that of the delay line oscillator sensor described above. The phase comparator outputs a DC voltage that changes at 1 V_{DC} per π radians of phase difference between the sensor and reference channels or approximately 5.5 mV_{DC} per degree of phase difference. It requires 6 mA at 3 V_{DC}, operates up to 500MHz, and occupies approximately 1 mm^2.

An added benefit to incorporating a reference delay line and phase comparator is that phase shifts due to temperature changes are automatically cancelled. One of the principal disadvantages of GaAs acoustic wave sensors, when compared to quartz devices, is the relatively large temperature-induced phase (or frequency) drift of 50 parts per million per degree Celsius (ppm/°C). However, by fabricating the sensor and reference delay lines on the same substrate, both devices experience the same drift with temperature and the common drift is cancelled by the phase comparator. This dual delay line structure is shown in Fig. 5 along with a comparison of the temperature drift a single delay line oscillator (Figs. 1 and 3) sensor to the drift of the reference/sensor dual delay line device. In this configuration, the reference delay line is in the feedback path of the integrated amplifier and together they form a reference oscillator. The sensor delay line is also driven by the same amplifier but the sensor signal does not feed back into the amplifier. A vector voltmeter was used to measure the phase difference between the two delay lines.



(a)



(b)

Figure 5 (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. The residual temperature dependence in the dual delay line device is probably due to thermal effects in the amplifier.

The fully integrated sensor will incorporate a reference SAW delay line oscillator, a delay line sensor, and a phase comparator monolithically integrated on a single GaAs substrate for DC-in, DC-out operation. This device is currently being fabricated. Integrated sensor arrays with multiple delay line sensor elements are also being fabricated.

3. GaAs SAW DELAY LINE SENSOR PACKAGING

A crucial aspect of the integrated microsensor is the fluidic package that exposes the SAW delay line sensor to microliter volumes of the analyte gas but must not interfere with the acoustic or electronic function of the device. The package must include miniature flow channels, inlet and outlet ports, and seal directly to the GaAs substrate, all with a minimum dead volume. This has been accomplished with machined glass lids. The glass substrate, initially 1 mm thick and 3 inches in diameter, has 100 µm deep channels ground into it. The channels are approximately 2 mm long and 500 µm wide, just large enough to enclose the SAW delay line. At either end of the channel, holes are ground through the glass substrate to accept microcapillary tubes as inlet and outlet ports. The glass substrates are then cut into individual lids with a dicing saw. The microcapillary tubes are inserted into the lids and fixed in place with epoxy.

The lids are assembled on the GaAs substrates over the SAW delay lines and clamped temporarily in place. A small drop of low viscosity Epoxy Technology OG113 UV-curing epoxy is then placed along the edge of the lid where it contacts the substrate. Capillary forces cause the epoxy to wick everywhere under the lid where it is in contact with the substrate. The epoxy flow terminates when it reaches the ground channel in the lid, leaving the delay line free of epoxy. The epoxy is then cured with UV light and the clamp is removed. Because this is a low temperature assembly process, neither the polymer selective coatings on the delay lines nor the microelectronics is adversely affected. An example of this packaging process is shown in Fig. 3

4. MICROMACHINED GaAs FPW 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[2] 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 Fig. 6). 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 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[4]. 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[4,5]. The devices are then rinsed and dried.

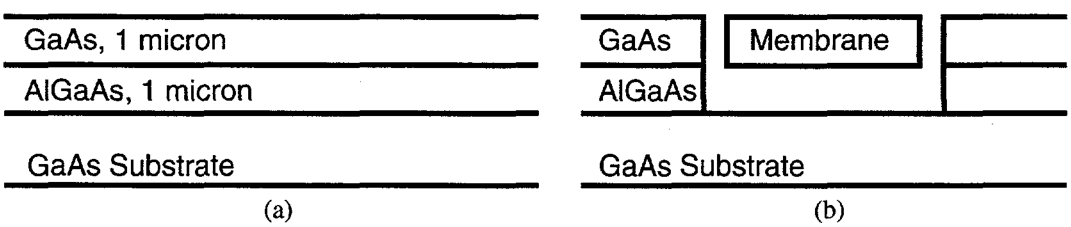


Figure 6 Schematic cross-section of epitaxial layers used in GaAs membrane fabrication. (a) Starting material. Epitaxial layers are typically 1 μm thick. (b) After sacrificial AlGaAs etch, showing suspended GaAs membrane.

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[6]. 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 N_2 atmosphere, freezing the cyclohexane. The cyclohexane sublimes over a period of a few minutes, leaving the devices dry and unstuck. Examples of these membranes after drying are shown in Fig. 7. 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.

FPW delay lines on 3 μm thick GaAs membranes have been fabricated and tested (see Fig. 8). The sacrificial AlGaAs layer (70% Al) was 2 μm thick. Membranes as large as 3 mm by 500 μm have been produced which are sufficiently large for the delay lines. Membranes can be arbitrarily long but the width is limited by the ability of the selective etchant to diffuse under the membrane.

To demonstrate the FPW mode behavior, the frequency response of the delay lines was measured prior to the sacrificial etch, when the SAW mode propagates on the thick (425 μm) substrate, and after etch, when the FPW mode propagates on the membrane. A characteristic of the FPW mode is that it propagates at a lower acoustic velocity than the SAW mode[2,7]. Therefore, the FPW delay line will exhibit a peak in its frequency response at a lower frequency than the SAW delay line, given equal wavelengths (as determined by the IDT finger spacing). The frequency response of two delay lines before and after the membrane release is shown in Fig. 9. Acoustic wavelengths for the two devices are 9 μm and 14 μm, sufficiently large to reach the FPW limit in 3 μm thick membranes. Frequency response is measured using a network analyzer. As shown in Fig. 9, the frequency response of both devices exhibits the shift to lower frequency expected for FPW modes. The magnitude of the frequency shift for both devices is in good agreement with the theoretical predictions. The oscillations observed in the SAW resonance peak for the 14 μm delay line (Fig. 9a) are probably due to coupling between the SAW mode and the electromagnetic radiation mode at the test station probe tips.

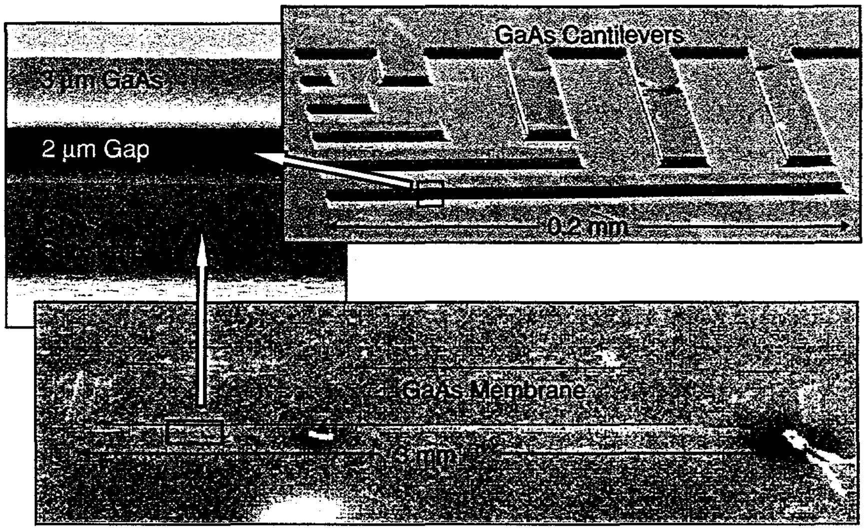
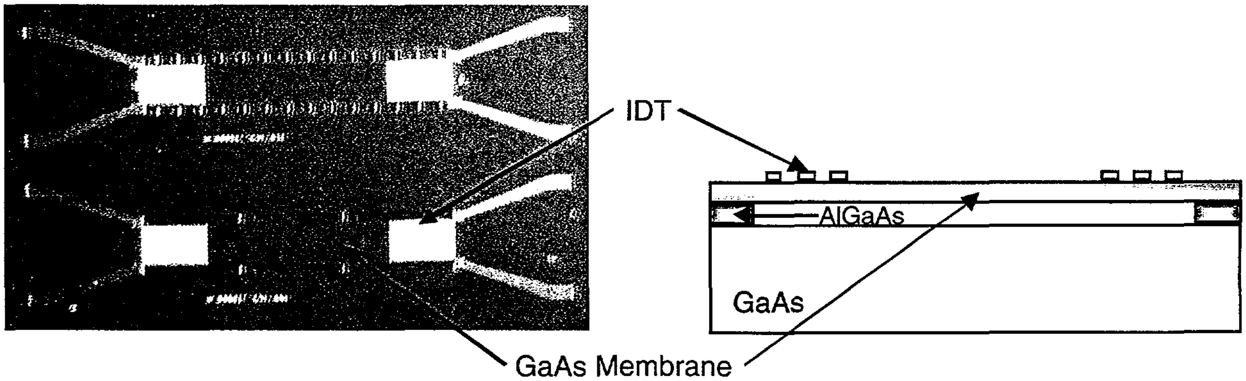
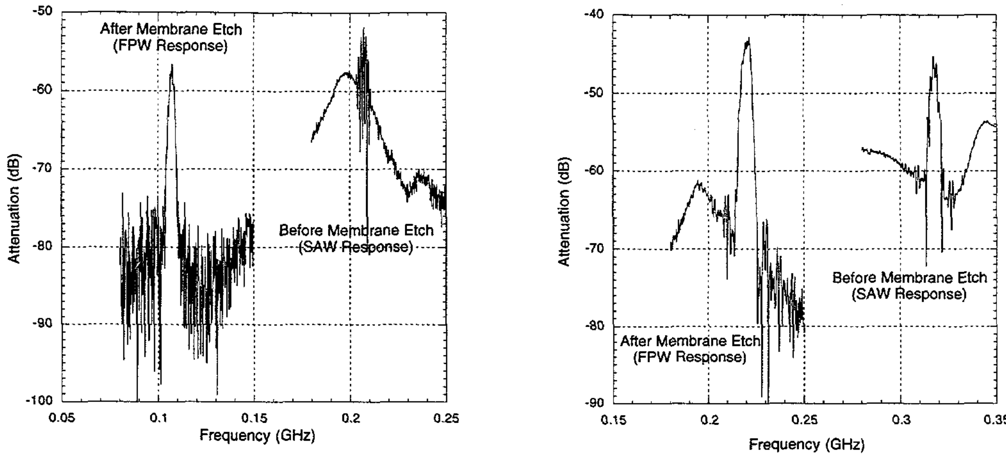


Figure 7 Scanning electron micrographs of GaAs membranes for FPW fabrication. The lower micrograph shows a 3 mm x 0.5 mm membrane attached to the substrate only at its corners. The upper right micrograph shows a 200 μm x 40 μm cantilever attached to the substrate only along one edge. The cantilever exhibits 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.



(a) (b)

Figure 8 (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.



(a) (b)

Figure 9 Comparison of GaAs acoustic delay line frequency response before sacrificial AlGaAs etch (SAW mode) and after etch (FPW mode). GaAs membrane is 3 μm thick. Frequency shift of minimum attenuation peak on going from SAW mode to FPW mode is in good agreement with theoretical predictions. (a) Acoustic wavelength = 14 μm. Oscillations in SAW peak are probably due to interference between the acoustic mode and the radiative mode at the test probes. (b) Acoustic wavelength = 9 μm.

FPW fabrication processes are compatible with GaAs microelectronics so integration and packaging of FPW chemical sensors are possible in the same manner as described above in Sections 2 and 3 for SAW sensors. Other acoustic configurations, for example, FPW resonators that use the edges of the membrane to define the resonant cavity, are smaller and potentially more sensitive. Other acoustic modes can also be fabricated on the GaAs membranes. In particular, TSM resonators are exciting candidates for extremely small, high sensitivity liquid and gas chemical sensors.

5. SUMMARY

The design and fabrication of acoustic wave chemical sensors in GaAs have been discussed. SAW delay line sensors are the furthest developed. Discrete delay lines, IC amplifiers to drive the delay lines, and phase comparators for output signal processing have been demonstrated. Chemical detection using these components has also been demonstrated. A monolithically integrated GaAs oscillator incorporating a delay line and an amplifier has been designed, fabricated, and tested. This device requires only a DC voltage input to operate. The integration of the phase comparator and a reference delay line for complete DC-in, DC-out, single chip operation is underway. An added advantage to this approach is that signal drift caused by temperature variation is eliminated.

GaAs FPW delay lines have been fabricated and tested. These devices are built on thin GaAs membranes produced by surface micromachining techniques. Measured FPW results agree well with theoretical predictions. The development of integrated chemical sensors based on these devices is underway. FPW and TSM resonators are also being investigated.

6. ACKNOWLEDGEMENTS

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

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