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Taste sensors utilizing high-frequency SH-SAW devices

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Abstract

Recent interest in the development of taste sensors has been motivated by their potential application in the food and beverage industries as well as environmental monitoring. This paper describes the development of novel high-frequency dual delay line and two-port resonator shear-horizontal surface acoustic wave (SH-SAW) microsensors for liquid analysis. The acoustic devices have been designed to function without the need for analyte-specific coatings and to operate at the wireless ISM frequency of 433 MHz; the sensors are small, robust and are built on a piezoelectric substrate (36° *YX* LiTaO₃). Liquid samples are introduced to the active sensing area via a microfluidic cell that has been fabricated using microstereolithography (MSL) and then attached to the SH-SAW sensor substrate. A low cost flow system has been developed and integrated with the sensor housing to create a fully automated measurement system. Tests have been performed on aqueous solutions with different tastants representing not only the four basic tastes of saltiness, sweetness, sourness, and bitterness but also of umami and metallic. Results show that good discrimination between the different taste samples is possible using both delay-line and resonator type piezoelectric devices. © 2006 Elsevier B.V. All rights reserved.

Keywords: Taste sensors; Micro-analytical system; Delay line; Resonator

1. Introduction

Human sensory tests are regularly employed in the food and beverages industries, environmental monitoring or other areas but results are based on subjective judgements and variations between panels can be up to 50% in terms of flavour units. Therefore, the development of 'objective' tools to detect taste is very much needed. Previous reports on artificial taste sensors often refer to them as so-called "electronic tongues" [1,2]. The most common types of sensors used are based on electrochemical techniques, such as potentiometry and voltammetry [3-5]. Potentiometric sensors employ zero-current and utilise either ion selective lipid membranes or ion selective electrodes and the potential at the working electrode caused by the ion exchange reaction is measured. Voltametric (amperometric) electronic tongues employ a potential to drive an electrode transfer reaction, and the resulting current, which depends on the concentration of the sample, is measured. Other sensing methods include optical and acoustic techniques. The 'optical electronic tongue' [6] mimics the mammalian tongue and has the ability to assay solution content for Ca²⁺, Ce³⁺, H⁺ and fructose using

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0925-4005/\$ - see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.snb.2006.04.040 colourimetric indicators. In the field of acoustic devices, quartz crystal microbalances (QCM) were the first ones used as sensors and they rely on the frequency changes due to mass changing on the device surface [7,8]. Using an array of such devices coated with hydrophilic mono- and dicarbon acids, organic and inorganic acids and amines in drinking water have been detected [9]. There have also been reports on quartz resonator devices coated with lipid/polymer membranes to measure different taste substances [10].

Here, we propose the use of small surface acoustic wave (SAW) sensors rather than the bulkier coated QCM devices. Operating a SAW device in the liquid phase requires a horizontally-polarized mode of vibration to reduce the acoustic loss in liquids; various devices using different piezoelectric crystals and different wave modes (Love waves, shear horizontal acoustic plate mode, flexural plate waves (FPW) or shear horizontal SAW) have been discussed in the literature [7,11-13]. However, liquid sensing with SAW devices usually employs devices with analytical coating films to enhance the chemical selectivity of the sensors. Also, when using electrochemical techniques ion selective electrodes/membranes are utilised to make them more analyte specific. The work reported here is based on devices with no bio-chemical layers, thus relying on a purely physical, rather than electrochemical, principle of operation. The adopted principle of operation makes the sensors more

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robust and durable since they are less susceptible to surface contamination than electrochemical sensors. The reduction in specificity is addressed through the use of dual designs and/or multi-parameter analysis.

This paper focuses on the design, fabrication and testing of two types of high-frequency SH-SAW sensors (delay line and resonator configurations), and the development of an automated measurement system for the rapid analysis of different liquid samples. One advantage of high-frequency acoustic devices is that of increased sensitivity-e.g. for quartz crystal microbalances the sensitivity increases with the square of the resonant frequency [8]. The majority of SAW resonators have been used mainly for gaseous phase measurements [14-17] and very few liquid applications with SAW resonators have been reported. Research on a one-port resonator [18] showed that the resonance frequency and the Q factor value are influenced by the viscosity of the liquid sample; two-port resonators [19] have been reported to respond to both the conductivity and the square root product of the density and viscosity of the liquid. Commercially available STW-resonators on LiTaO3 employing a SiO2 guiding layer have also been investigated for aqueous media [20].

In this work, we present custom designed LiTaO₃ delay lines and two-port resonators that have been successfully used to identify the basic tastes: saltiness, sweetness, sourness, and bitterness, as well as umami and metallic tastes without the need of any guiding layer or selective coating.

2. Principle of operation

The sensing principle of the proposed SH-SAW sensors is based on the shear horizontal acoustic wave–liquid interaction that results in perturbations of the propagation characteristics of the wave. These perturbations are translated to the electrical domain and can be monitored via signal attenuation, phase or frequency shifts.

An acoustic wave propagating on a free surface of a piezoelectric substrate will have an associated electric field that can extend into the liquid above, allowing for electrical perturbations of the wave properties. Metallizing the propagating surface, the piezoelectric potential is zeroed and thus the acoustic wave will respond to mechanical properties of the sensing liquid. Defining the boundary conditions of the sensing areas allows for some flexibility in choosing the functionality/sensitivity of a specific sensor configuration. For example, a dual delay line sensor can be made sensitive to both mechanical (e.g. density, viscosity) and electrical (e.g. conductivity, permittivity) properties of the liquid under test if two sensing areas with different boundary conditions are used: one metallized and one free. If differential measurements are taken between the two delay lines, this configuration will respond mainly to acousto-electrical interactions eliminating the mechanical ones.

Based on the perturbation theory proposed by Auld [21], the analytical formulations of the SH-SAW–liquid interactions have been derived by Kondoh and applied to the case of dual/triple delay line devices [12,22].

Mechanical interactions can be expressed in terms of viscous coupling and mass liquid loading. In the derivation of the mechanical interaction effects, metallized sensing surfaces are used, as this allow the elimination of the acousto-electric effects. When a Newtonian liquid with a viscosity of η and density ρ_1 is loaded on the sensor, attenuation and phase shift due to the viscous coupling can be expressed as [12]:

$$\frac{\Delta v}{v} = -\frac{v v_{\rm pc}^2}{4\omega P} \left(\sqrt{\frac{\omega \eta' \rho_{\rm l'}}{2}} - \sqrt{\frac{\omega \eta \rho_{\rm l}}{2}} \right) \tag{1}$$

$$\frac{\Delta\alpha}{k} = \frac{vv_{\rm pc}^2}{4\omega P} \left(\sqrt{\frac{\omega\eta'\rho_{\rm l'}}{2}} + \sqrt{\frac{\omega\eta\rho_{\rm l}}{2}} \right) \tag{2}$$

where v is the phase velocity, v_{pc} the particle velocity component of the shear horizontal mode, ω the angular frequency, P the power flow per unit width and k is the wave number; also ' indicates a perturbed quantity. The mass loading effect can be derived allowing for an isotropic thin liquid film of thickness h and density ρ to load uniformly the metallized surface of the sensor. Considering that the liquid properties do not change before and after perturbation, the velocity shift and attenuation change can be obtained as

$$\frac{\Delta v}{v} = -\frac{\mu h v_{\rm pc}^2}{4P} \left(\rho' - \frac{\mu'}{v^2} \right) \tag{3}$$

$$\frac{\Delta\alpha}{k} = 0 \tag{4}$$

where μ' is the Lamè constant of the film [12].

On the other hand, for the derivation of the acousto-electric interactions, mechanical effects are eliminated. This can be practically achieved by taking differential measurements between free and metallized sensing surfaces. Thus, the relationships for changes in velocity and attenuation of the SH-wave due to the electrical properties of the liquid are given by

$$\frac{\Delta v}{v} \approx -\frac{K_{\rm s}^2}{2} \frac{(\sigma'/\omega)^2 + (\varepsilon_{\rm r}'\varepsilon_0 - \varepsilon_{\rm r}\varepsilon_0)(\varepsilon_{\rm r}'\varepsilon_0 + \varepsilon_{\rm P}^{\rm T})}{(\sigma'/\omega)^2 + (\varepsilon_{\rm r}'\varepsilon_0 + \varepsilon_{\rm P}^{\rm T})^2}$$
(5)

$$\frac{\Delta\alpha}{k} \approx \frac{K_{\rm s}^2}{2} \frac{(\sigma'/\omega)(\varepsilon_{\rm r}\varepsilon_0 + \varepsilon_{\rm P}^{\rm T})}{(\sigma'/\omega)^2 + (\varepsilon_{\rm r}'\varepsilon_0 + \varepsilon_{\rm P}^{\rm T})^2} \tag{6}$$

where K_s^2 is the electromechanical coupling coefficient when a reference liquid (e.g. distilled water) is loaded on the free surface, ε_p^T the effective permittivity of the SAW crystal, ε_r the relative permittivity of the reference liquid (distilled water), ε_r' and σ' are the relative permittivity and conductivity of the tested liquid sample. From Eqs. (5) and (6) the circle formulae in (7) and (8) can be obtained and used to generate a permittivityconductivity chart [12,23]:

$$\begin{bmatrix} \frac{\Delta v}{v} + \frac{K_{s}^{2}}{4} \frac{(2\varepsilon_{r}'\varepsilon_{0} - \varepsilon_{r}'\varepsilon_{0}) + \varepsilon_{P}^{T}}{\varepsilon_{r}'\varepsilon_{0} + \varepsilon_{P}^{T}} \end{bmatrix}^{2} + \left[\frac{\Delta \alpha}{k}\right]^{2}$$
$$= \left[\frac{K_{s}^{2}}{4} \frac{\varepsilon_{r}\varepsilon_{0} + \varepsilon_{P}^{T}}{\varepsilon_{r}'\varepsilon_{0} + \varepsilon_{P}^{T}}\right]^{2}$$
(7)

$$\left[\frac{\Delta v}{v} + \frac{K_{\rm s}^2}{2}\right]^2 + \left[\frac{\Delta \alpha}{k} - \frac{K_{\rm s}^2}{4}\frac{\varepsilon_{\rm r}\varepsilon_0 + \varepsilon_{\rm P}^{\rm T}}{\sigma'/\omega}\right]^2 = \left[\frac{K_{\rm s}^2}{4}\frac{\varepsilon_{\rm r}\varepsilon_0 + \varepsilon_{\rm P}^{\rm T}}{\sigma'/\omega}\right]^2 \tag{8}$$

The chart can then be used to determine electrical properties of the liquid based on the experimental measurements of phase and attenuation.

The liquid–SAW interactions for the resonator case can be treated in a similar manner to those identified for the delay line. The resonator proposed in this paper has an electrically free cavity, thus the sensor output will be a complex combination of both mechanical and acousto-electrical effects given by Eqs. (1)–(6). This response to the multitude of physical parameters (viscosity, density, conductivity, and permittivity) involved in SAW–liquid interactions can enhance the sensors ability to differentiate between various samples. However, such a response can make the analysis and the extraction of specific liquid properties more difficult. Generally, the resonator amplitude and phase response can be expressed as a complex function of the liquid physical properties, e.g. viscosity— η , density— ρ , electrical conductivity— σ , electric permittivity— ε and temperature—T as

$$A, \Phi = f(\eta, \rho, \sigma, \varepsilon, T) \tag{9}$$

3. Experimental

Previous results obtained by the research group at Warwick University for SH-SAW devices operating at lower frequencies (61.2 MHz) were encouraging [24] and new sensors have been fabricated for higher frequencies (400-500 MHz). This results in smaller, lower cost devices operating at free wireless ISM frequencies. Two sensor configurations have been analyzed, designed and fabricated: a dual delay line and a two-port resonator. A photograph of the dual delay line sensor is given in Fig. 1a. The inter-digitated transducers (IDTs) are made of 25 thin, solid finger pairs, have an acoustic aperture of 800 µm and each finger has a width of 2.4 µm. The opening on the 'free' sensing area has a width of 480 µm and overall die dimensions are 5100 µm × 2700 µm.

The two-port resonator sensor is shown in Fig. 1b. Resonators have linear phase response and lower insertion loss (IL) when compared to the delay line (the IL values for the delay line sensor loaded with various liquids were around 14–18 dB, while for the resonator sensor IL were between 7.6 and 8.4 dB). The resonator

has been designed here such that both port IDTs have the same parameters as the IDTs of the delay line sensor described above. The resonating cavity is 192 μ m (20 λ) wide and the reflectors on each side of the cavity consist of 400 strips (positive and negative reflectors) of 2.4 μ m pitch. Overall die dimensions are 4600 μ m \times 3000 μ m.

The 36° YX LiTaO₃ (lithium tantalate) piezoelectric crystal has been chosen as the substrate because of its high coupling coefficient and support of the SH mode. Both types of device, the delay line configuration and the resonator configuration, have been built on the same wafer. IDTs and reflectors have been patterned using a standard UV photolithographic process at Georgia Institute of Technology. A 130 nm thick layer of aluminium has been sputtered on the top of a pre-cleaned wafer. To minimize the surface charges associated with the pyroelectric effect of LiTaO₃ substrate, a thin layer of Al has been deposited conformally on the backside of the wafer. Then a negative resist (Futurrex NR7-1000P) was spun on top of the Al layer, baked and then exposed using a MA6 mask aligner. A reactive ion etch step was used to remove the unwanted Al and so the desired pattern has been transferred to the wafer. Backside Al has been removed and a RR4 resist remover has been used to strip the photoresist from the metal pattern.

After fabrication the sensors were mounted and ultrasonically wire bonded onto custom PCB boards with SMA connectors to interface with the measuring equipment. Miniature MSL (microstereolithography) liquid cells $(15.5 \text{ mm} \times 9.0 \text{ mm})$ have been designed and fabricated using an Envisiontec Prefactory rapid prototyping system. The cell has a flow channel of 800 µm width and a 500 µm height. A photograph of the miniature MSL liquid cell is shown in Fig. 2. After the final curing step involved in the MSL fabrication, the liquid cell has been coated with 2 µm of Parylene C, using a PDS 2010 Labcoater from the Speciality Coating Systems. The Parylene C coating step was necessary to improve the flow in the liquid cell and to ensure chemical inertness. Inlet and outlet tubes were attached to the liquid cell and then it was mounted on top of sensing area. A picture showing attachment of the liquid cell onto the dual delay line senor is shown in the Fig. 3.

A custom build liquid flow system was developed for the SH-SAW sensors measurements. A block diagram of the liquid flow system is shown in Fig. 4. This design of the flow system was based on the selection between two sample liquids and a cleaning/reference liquid. The components in flow system were chosen based on the chemical inertness of the materials



Fig. 1. (a) Dual delay line configuration: free line at top showing window for sensing liquid above; (b) two-port resonator configuration.



Fig. 2. Photograph of the miniature MSL liquid cell.



Fig. 3. Delay line sensor mounted on a custom PCB with a MSL cell on top.



Fig. 4. Diagram of liquid flow system and the automated measurement set-up.



Fig. 5. Photograph of the prototype micro analytical taste system.

they are made from. The sample liquids are held in custom designed chemically inert stainless steel vessels with a capacity of approximately 25 ml and the cleaning reference liquid is held in a 500 ml container (polypropylene). The liquids are pumped into the liquid cell and over the sensing area of the devices using a micro diaphragm liquid pump supplied by KNF Neuberger UK Ltd. (model No. NF KT 10 dc 24V). The pump can deliver liquids at a maximum flow rate of 0.1 l/min (controlled using the electronics and a manual throttle valve) and has high chemical resistance with a PTFE diaphragm. The selection between the sample liquids and that of the reference liquid is made using micro valves controlled using the software. Micro inert valves (MIV) supplied by Lee Products Ltd. were used (part number: LFRX050000B dc 12V). The flow rate of the liquids in the system was monitored using a flow meter supplied by Farnell UK Ltd. (S8011R flow transducer part number: 178-923) and the temperature was also monitored. Fig. 5 shows a photograph of prototype flow system (polypropylene cleaning liquid container not shown due to large size).

For the delay line measurements a signal generator (HP 8648C) and a vector voltmeter (HP 8508A) were used to excite and record the SAW parameters (attenuation and phase change). The operating frequency of the delay line device was found to be 460.84 MHz. The liquid cell pipes were connected to the liquid flow system controlled by a computer via a LabViewTM interface.

The set-up for the two-port resonator sensor was similar to that used for the dual delay line sensor. Measurements have been performed with a network analyser (Agilent 8753ES) and amplitude and phase shift information have been recorded. The resonance frequency of this device in air was found to be 433.7 MHz. Due to the larger number of taste samples tested with the resonator configuration, a manual injection of the test samples and the cleaning liquid has been performed.

4. Results and discussion

Aqueous solutions representing different medium level tastes have been prepared as liquid test samples. Four samples rep-



Fig. 6. Delay line measurements of the four basic tastes solutions.

resenting the basic tastes, saltiness, sweetness, sourness, and bitterness, have been chosen for the initial tests on the delay line configuration: 0.1 mol saline, sucrose and acetic acid solutions and 0.0012 mol quinine sulfate solutions. Quinine sulfate has a very low solubility in water that dictated the lower concentration of this sample. During the testing the samples have been randomised and de-ionised (DI) water has been employed as a cleaning fluid in between two test samples. Amplitude and phase information have been recorded for each delay line and their relative difference in terms of attenuation and phase difference is computed and scattered plotted in Fig. 6.

The resonator configuration has been tested with six taste samples in which umami and metallic taste samples have been introduced. The four basic tastes (sweet, salty, sour and bitterness) have been prepared using sucrose, sodium chloride, acetic acid and quinine hydrochloride, respectively. The fifth umani taste solution has been prepared using monosodium glutamate (MSG) and the solution simulating the metallic taste/sensation has been made with iron sulfate. As quinine hydrochloride has a much higher limit of solubility in water than quinines sulfate, all the solutions have been prepared with a 0.1 mol concentration. Again the samples have been randomised and DI water has been employed as a cleaning fluid in between two test samples. The results obtained are presented as a scattered plot in Fig. 7 that shows 100% separation and clustering of the six taste samples. From the graph it is clear that both amplitude and phase signals carry the information necessary for sample discrimination. Conductivity of the taste samples has been measured at room temperature (approximately 24 °C) with a Jenway 4310 meter and is given in Table 1. In the positive phase of the plot, the

 Table 1

 Details and properties of the taste solutions at room temperature

Sucrose	Sodium chloride	Acetic acid	Quinine hydrochloride	MSG ^a	Iron sulfate ^b	Caffeine
Fisher	BDH	Fisher	Sigma	Fluka	Aldrich	Sigma
C ₁₂ H ₂₂ O ₁₁	NaCl	$C_2H_4O_2$	$C_{20}H_{24}N_2O_2\cdot HCl\cdot 2H_2O$	C5H9NO4·Na	FeSO ₄ ·7H ₂ O	$C_8H_{10}N_4O_2$
342.29	58.43	60.04	396.91	187.14	278.01	194.19
3.11µS	2.62 MS	140 µS	1690 µS	1540 µS	2.52 MS	9.08 µS
	Sucrose Fisher C ₁₂ H ₂₂ O ₁₁ 342.29 3.11µS	Sucrose Sodium chloride Fisher BDH C ₁₂ H ₂₂ O ₁₁ NaCl 342.29 58.43 3.11 µS 2.62 MS	Sucrose Sodium chloride Acetic acid Fisher BDH Fisher $C_{12}H_{22}O_{11}$ NaCl $C_{2}H_{4}O_{2}$ 342.29 58.43 60.04 3.11 μ S 2.62 MS 140 μ S	SucroseSodium chlorideAcetic acidQuinine hydrochlorideFisherBDHFisherSigma $C_{12}H_{22}O_{11}$ NaCl $C_2H_4O_2$ $C_{20}H_{24}N_2O_2 \cdot HCl \cdot 2H_2O$ 342.2958.4360.04396.91 $3.11 \mu S$ 2.62 MS140 μS 1690 μS	$\begin{array}{llllllllllllllllllllllllllllllllllll$	SucroseSodium chlorideAcetic acidQuinine hydrochlorideMSG ^a Iron sulfate ^b FisherBDHFisherSigmaFlukaAldrich $C_{12}H_{22}O_{11}$ NaCl $C_2H_4O_2$ $C_{20}H_{24}N_2O_2 \cdot HCl \cdot 2H_2O$ $C_5H_9NO_4 \cdot Na$ FeSO ₄ · 7H ₂ O342.2958.4360.04396.91187.14278.01 $3.11 \mu S$ 2.62 MS140 μS 1690 μS 1540 μS 2.52 MS

Conductivity for 0.01 M quinine hydrochloride solution is $234 \,\mu\text{S}$.

^a L-Glutamic acid monosodium salt monohydrate.

^b Iron(II) sulfate heptahydrate.



Fig. 7. Resonator measurements of the six different taste solutions.



Fig. 8. Bitter samples tested with the resonator sensor.

samples having the same order of magnitude for the conductivity cluster around the same phase range.

Initial experiments have also been conducted using the resonator configuration to separate the samples within the same taste family. Three different bitter samples (caffeine and two quinine samples with 0.1 and 0.01 mol concentrations) were randomised and tested. Again Fig. 8 shows a clear clustering of different bitter samples. These preliminary tests suggest that is possible to discriminate between samples with various concentrations. The concentrations of the synthetic taste samples used are fairly high when compared to the threshold limits reported for human sensing. Further dilution tests need to be performed to determine the SH-SAW sensors limit of detection. Previous experiments on lower frequency delay line (61.2 MHz) indicated that the diluted solutions converge towards water and the limit of detection is around 0.1% or 1 part in 10^3 [24].

The plots show that both devices were capable to discriminate 100% between the different taste solutions. The resonator sensor seemed to be more stable than the delay line, taste samples clustered better and a better separation between the clusters has been achieved.

5. Conclusions

This paper presents novel SH-SAW taste sensors employing high-frequency dual delay line and resonator sensor configurations that operate at a wireless ISM frequency of 433 MHz. The devices are small, robust, low power and can discriminate between six basic synthetic taste samples without the need of any selective or bio-chemical membrane. The sensing principle is based on physical interaction mechanisms rather than chemical ones and taste classification is achieved, indirectly, on the basis of the physical parameters of each sample.

In addition to the sensing system, an automated low cost liquid flow system has also been developed. Initial results are encouraging and applications are envisaged for the rapid, lowcost (and, possibly, wireless) screening of liquid samples in the food and beverage industries as well as in biomedical and environmental monitoring. Initial results on bitter samples are also signalling possible applications in the pharmaceutical industry. However, the sensitivity of these devices has to be further investigated, especially with regard to the limitations of the previous generation of our devices [24], where the limit of detection was estimated at 0.1%. Furthermore, the physical operating principle of these piezoelectric devices, i.e. the lack of any analyte-specific coating, will limit their range of applications. For example, such devices are unlikely to be able to detect trace levels of a specific taste within a complex mixture. Nevertheless, we believe that this type of device can offer robust, low-cost screening of certain bioliquids and thus commercial application.

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Biographies

Irina Leonte graduated with a BSc in Electrical Engineering and an MSc in Environment Monitoring Computer Science Systems from "Gh. Asachi" Technical University of Iasi (Romania) in 2000 and 2001 respectively. In 2002 she received an MSc in Advanced Electronic Engineering, with distinction, from University of Warwick. Since 2003 she has been with the Sensors Research Laboratory, School of Engineering, University of Warwick where she is currently completing work on her PhD project. Her research interests include high frequency surface acoustic wave sensors, biosensors, micro-fluidics, MSL packaging and small antennas for wireless microsystems.

Gurmukh Sehra BSc, PhD, MIEE, MIEEE, received his BEng degree in Electronic Engineering and PhD in Engineering from University of Warwick in 2005. He is currently working as a postdoctoral research associate in the Sensors Research Laboratory, School of Engineering, University of Warwick. His main research interests are SAW sensing, bio-sensing, optical sensing, actuators, microsystems and ultrasonic sensing.

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