

## **ELASTIC WAVES FOR MINIATURIZED PIEZOELECTRIC SENSORS: APPLICATIONS TO PHYSICAL QUANTITY MEASUREMENTS AND CHEMICAL DETECTION**

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The frequency is the quantity measured with the best accuracy and time and length standards are defined with it. In the domain of the metrology closer of manufacturing applications (process control or instrumentation), sensors based on the frequency variation of oscillators, are potentially interesting because several reasons: high resolution, good sensitivity and digital signal by accounting.

This paper is dedicaced to an overview of oscillators used as sensor in which general principles and definitions are presented and illustrated with different devices built in SAW technologies (temperature, pressure, acceleration).

Different technological ways can be used to build the sensitive SAW sensor and chemical detector applications enable to describe the different possibilities as quartz or  $\text{LiNbO}_3$  substrates or silicon substrate using Rayleigh waves or Lamb modes.

### **1. Introduction**

Elastic nonlinearities in piezoelectric materials are responsible for the sensitivity of acoustic devices to external perturbations such as temperature, pressure, force, and electric field. The perturbations change the elastic properties of the medium and hence the phase velocity of the wave. If the elastic wave device is connected to a feedback amplifier to form a sensitive oscillator, a sensor results, when the frequency is the readout quantity.

Temperature, pressure, force and gas sensors that incorporate miniaturized BAW resonators and SAW devices are described. The technologies used, like photolithography and chemical etchnig processes, enable to mass-produce sensors with excellent reproducibility of the specifications.

## 2. Principles of elastic wave sensors

Bulk and surface elastic wave devices such as resonators and delay lines are sensitive to temperature, pressure, force, mass loading, and electric field. These physical quantities act on the device either directly (as in the case of temperature) or indirectly by means of a test body that transforms the mechanical quantity into quasistatic stress modifying the elastic properties of the substrate. The predeformation of the medium is superimposed on the particle displacement associated with the vibration. If the vibration does not influence the perturbation, the nonlinear phenomenon can be linearized so that the problem is the classical one but with modified elastic constants [1], [2], [3].

In an oscillator sensor the variation of the resonance properties of the devices caused by a physical quantity is translated into a frequency shift of the oscillator. Advantages of this type of sensor are the good short-term stability  $\sigma$  and hence its good intrinsic resolution. A typical curve of short-term stability of a SAW oscillator is presented in Fig. 1.

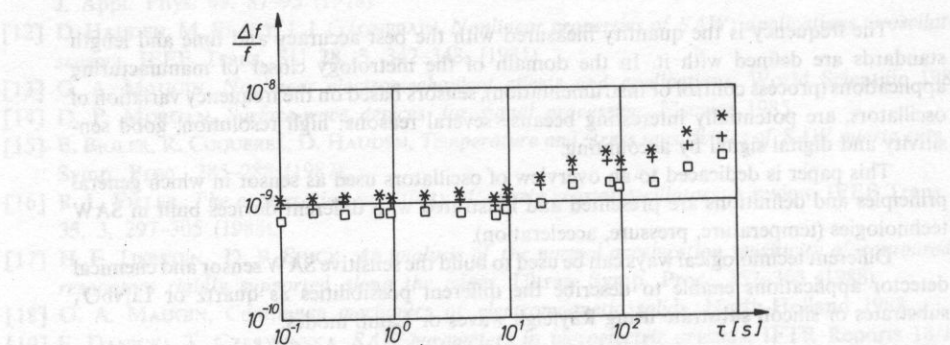


FIG. 1. Short-term stability of SAW delay line at 105 MHz used in force sensor

The sensitivity  $S$  to the external quantity determines the scaling factor of the sensor. The limiting resolution  $r$  is affected by the short-term stability over the time during which the frequency is measured and by the scaling factor.

$$r = \sigma \cdot f_0 / S$$

where  $f_0$  is the oscillator frequency before applying the perturbation.

Though departure from linearity may no longer be a problem for sensors because of the digital electronics used with them, limitations are the frequency repeatability and the aging that are sometimes too large to permit reaching ultrahigh performances. Repeatability and aging have an influence on the unperturbed frequency and often on

the scaling factor. Moreover, it is still difficult to model their influences accurately.

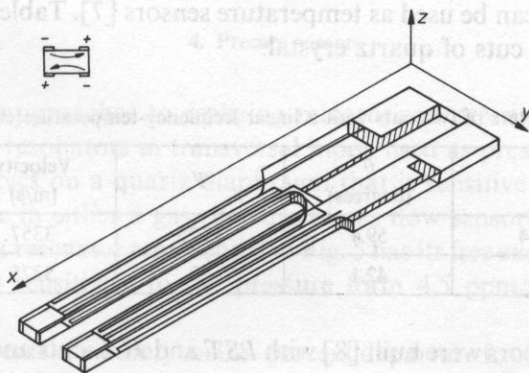
Typical values of short-term stability and aging are summarized in Table 1 for miniaturized BAW (MBAW) and SAW oscillators.

**Table 1.** Typical values of short-term stability  $\sigma$ , aging rate of SAW and miniaturized BAW oscillators used as sensors

Sensor Frequency	Short-Term Stability $\sigma$	Aging Rate
100 MHz–1 GHz (SAW)	$2.10 - 10 - 10^{-9}$	$\text{few} \times 10^{-6}/\text{month}$
20 kHz–2 MHz (MBAW)	$8.10 - 11 - 10^{-9}$	$\text{few} \times 10^{-6}/\text{year}$

### 3. Temperature sensors

Different ultrasonic temperature sensors have been studied; the most popular is the HP-28 MHz temperature sensor [4]. Two ways have been used to miniaturize quartz temperature sensors: tuning-fork resonators have been used at low frequencies [5], [6] and SAW quartz oscillators employing doubly rotated cuts have operated at high frequencies. In both cases, the photolithographic technology used to realize the elastic devices enables one to improve the device reproducibility and the production yield.



**FIG. 2.** Tuning-fork temperature sensor (after [3])

Figure 2 shows the **tuning-fork resonator** used as a temperature sensor. Its resonance frequency of 262 kHz varies linearly as a function of temperature, with a sensitivity of  $34.6 \cdot 10^{-6}$  K and a linearity defect of  $2.10^{-4}$  over 100 K due to the quadratic term (Fig. 3).

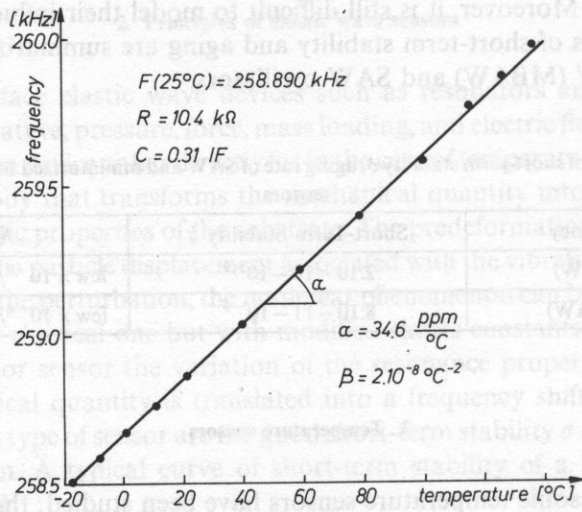


FIG. 3. Frequency shift versus temperature of tuning-fork resonator (after W. ZINGG)

However, an accuracy of  $\pm 0.5^{\circ}\text{C}$  can be reached after a preliminary calibration and a digital adjustment during operation. The complete oscillating sensor made in Switzerland is commercially available under the trade name “Thermopack”.

In the case of **surface acoustic waves**, doubly rotated cuts with linear characteristics and high sensitivities can be used as temperature sensors [7]. Table 2 gives theoretical characteristics of two cuts of quartz crystal.

Table 2. Parameters of two cuts with a linear frequency-temperature characteristic

Quartz cuts	$\Phi$	$\theta$ (degrees)	$\Psi$	Velocity (m/s)	TCF ( $10^{-6}/\text{K}$ )
LST	11.4	59.4	35.25	3357	33
JCL	0	42.1	39	3275	22

Temperature sensors were built [8] with *LST* and *JCL*-cuts and their sensitivities, resolutions and time constants measured. Fig. 4 represents the measured frequency-temperature (*F-T*) characteristics of both cuts. The *JCL* sensor had a sensitivity of 2.2 kHz/K, and the *LST*-probe a sensitivity of 3.4 kHz/K an oscillator-frequency of 100 MHz. Short-term stability of the SAW temperature oscillator is  $10^{-8}$  over 1–3 sec. Thus the intrinsic resolution is about 0.1 mC°.

Time constants were measured under several types of heating: thermal diffusion form a contact point, convection and conduction. Table 3, summarizing the values of the time constants measured at 90 percent of the final value, enables comparison with Hewlett-Packard’s temperature sensor under the same conditions.



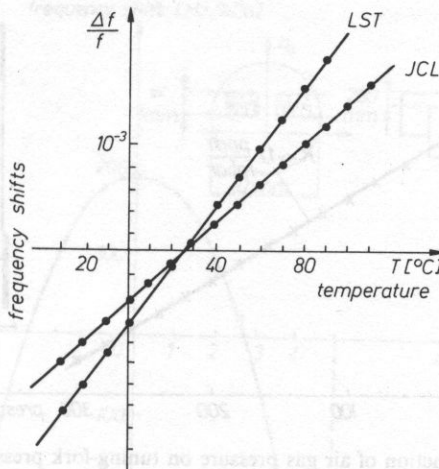


FIG. 4. Frequency shift versus temperature for *LST*-cut and *JCL*-cut quartz

Table 3. Time constants of BAW and SAW temperature sensors

Probes	Diffusion	Convection	Conduction
Bulk waves (H.P.)	1000 s	91 s	6 s
Surface waves	550 s	75 s	0,3 s

#### 4. Pressure sensors

Two different approaches to realizing minaturized pressure sensors have been studied: bulk-wave resonators in transversal mode used as pressure gas sensors, and surface acoustic waves on a quartz diaphragm that is sensitive to the bending force induced by pressure in either a gas pressure or gas flow sensor.

The **tuning-fork resonator** represented in Fig. 5 has its free ends grooved with holes to increase natural sensitivity to the pressure from 4.5 ppm/kPa to 11 ppm/kPa in air [6].

The measurements show only an 0.8-percent departure from linearity between 10 and 300 kPa and an excellent reproducibility. Frequency shifts of a sensor operating at 25 kHz as a function of air pressure are plotted on the same figure. Counting the frequency for 2.5 sec permits reaching a resolution of 1 Pa. One application is an altimeter because altitude can be measured with an accuracy of 1 m at 1000 m high, if temperature is well controlled.

Models of pressure sensitivity have been developed for optimizing SAW pressure sensors. The simplest device consists in directly applying pressure on a rectangular crystal plate fixed by epoxy on a rigid plane. When a *ST*-cut quartz SAW oscillator at 105 MHz is subjected to hydrostatic pressure from 0 to 300 kPa, the sensitivity is about

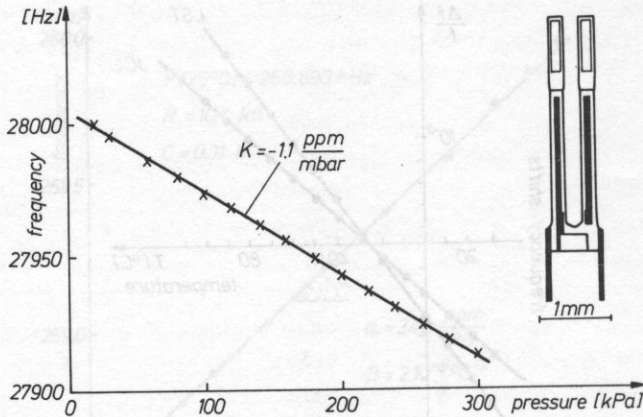


FIG. 5. Frequency shift as function of air gas pressure on tuning-fork pressure sensor (after W. ZINGG)

3 Hz/kPa and is rather low for sensor applications [3]. If instead a thin anisotropic plate is clamped at its edges and subjected to a pressure, it will be bent strongly [9], [10], [11]. The sensitivity can be increased further by using a thin circular diaphragm (Fig. 6).

Figure 7 gives the pressure sensitivity for a Y-cut quartz SAW sensor as a function of the mean position of the transducers along the  $a_3$ -axis. The diaphragm thickness is 250  $\mu\text{m}$  and its diameter is 10 mm. The nominal oscillator frequency is 105 MHz. For

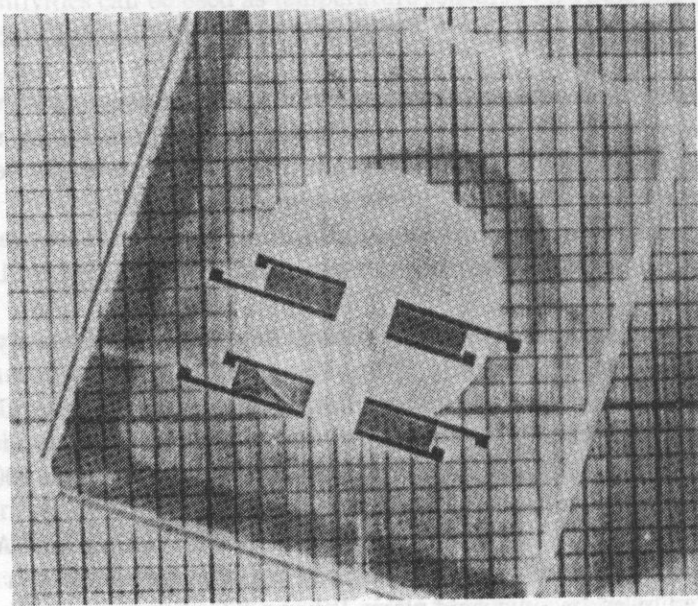


FIG. 6. Monolithic thin diaphragm for SAW pressure sensor

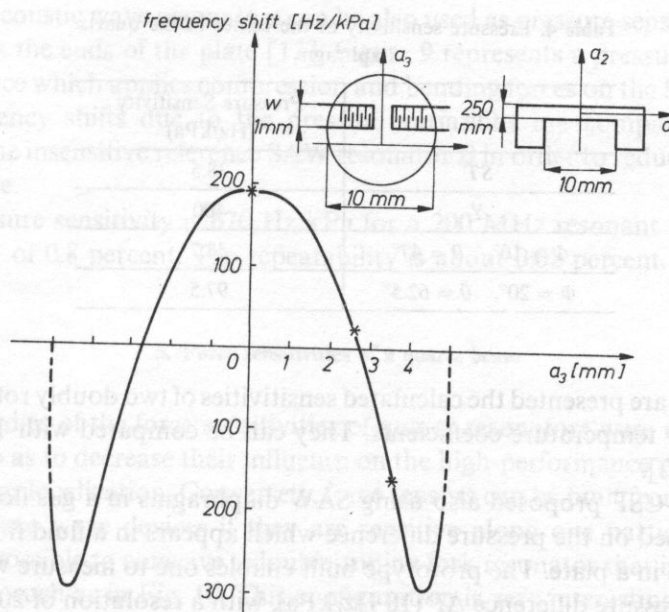


FIG. 7. SAW pressure sensor sensitivity as function of transducer position

a beamwidth  $W = 1$  mm, three experimental points have been obtained. At the center of the diaphragm ( $a_3 = 0$ ), the sensitivity is about 200 Hz/kPa and it is almost opposite that at  $a_3 = 4$  mm. Using this result, a dual-channel sensor with a sensitivity twice as large has been built (400 Hz/kPa). The differential device also permits a large degree of temperature compensation. The measured frequency-temperature shifts are within 1 kHz over a temperature range from  $-40^\circ\text{C}$  to  $80^\circ\text{C}$ . This corresponds to about 2 kPa over the total temperature range (Fig. 8).

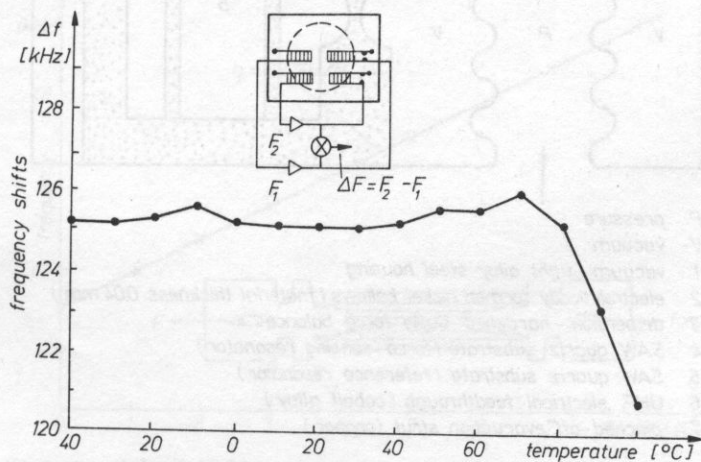


FIG. 8. Temperature compensation of SAW pressure sensor

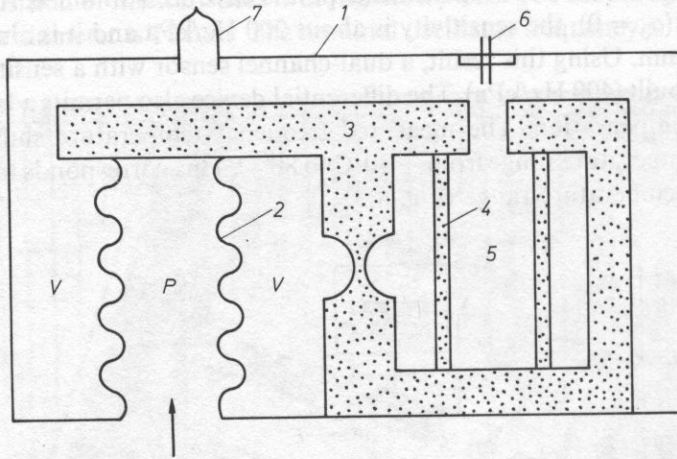
**Table 4.** Pressure sensitivity at the center of the quartz diaphragm

Cut	Pressure Sensitivity (Hz/kPa)
<i>ST</i>	38.5
<i>Y</i>	400
$\Phi = 10^\circ, \theta = 47^\circ$	48.7
$\Phi = 20^\circ, \theta = 62.5^\circ$	97.5

In Table 4 are presented the calculated sensitivities of two doubly rotated cuts with zero first-order temperature coefficients. They can be compared with *Y*- and *ST*-cut sensitivities [12].

Thomson-CSF proposed also using SAW diaphragms in a gas flow sensor. The principle is based on the pressure difference which appears in a fluid flowing through a hole grooved in a plate. The prototype built enables one to measure with very good linearity the pressure difference  $\Delta P$  (18 Hz/kPa), with a resolution of 20 Pa. Response time is less than 1 ms.

Figure 7 gives the pressure sensitivity for a *Y*-cut quartz SAW sensor as a function of the pressure difference  $\Delta P$  and the temperature  $T$ . The sensitivity is about 18 Hz/kPa at 20°C and 100°C. The sensitivity is about 18 Hz/kPa at 20°C and 100°C.



- P* pressure
- V* vacuum
- 1 vacuum-tight alloy steel housing
- 2 electrolytically formed nickel bellows (material thickness 0,04 mm)
- 3 dispersion-hardened CuBe force balance
- 4 SAW quartz substrate (force-sensing resonator)
- 5 SAW quartz substrate (reference resonator)
- 6 UHF electrical feedthrough (cobalt alloy)
- 7 pinched-off evacuation strut (copper)

**FIG. 9.** Schematic of the pressure sensor (after M. R. RISCH)



Surface acoustic wave resonators can be also used as pressure sensors if the forces are applied on the ends of the plate [13]. Figure 9 represents a pressure sensor using a balanced force which applies compression and bending forces on the SAW resonator *A*. The frequency shifts due to the pressure variations are compared with those obtained on the insensitive reference SAW resonator *B* in order to reduce the influence of temperature.

The pressure sensitivity is 670 Hz/kPa for a 200 MHz resonant frequency with a nonlinearity of 0.8 percent. The repeatability is about 0.02 percent.

### 5. Force sensitivities of a quartz beam

Many studies of the force sensitivities of quartz resonators were made to model these effects so as to decrease their influence on the high-performance resonators used in navigation or localization. Conversely, force sensors can be built from miniaturized bulk and surface wave devices if they are sensitive along one particular axis. For instance, it is possible to conceive a double-tuning fork resonator that is very sensitive to axial forces, such as in Fig. 10. This configuration is very interesting because of its excellent linearity, negligible hysteresis, good thermal stability, and low aging. Moreover, its sensitivity is very high:  $10^{-3}$ /N between 0–10 N and a repeatability better than  $6 \cdot 10^{-4}$  over the force range [6].

The force sensitivities of a cantilever quartz beam on which surface acoustic waves are propagating have also been studied [14]. Forces are induced by acceleration acting on an inertial mass. The device consists of a thin rectangular plate of quartz clamped at an edge and subjected to compression and flexure forces at the opposite edge by means of an inertial mass *M*. Sizes of the device are given on Fig. 11 and the surface acoustic wave is propagating along the main axis of the plate ( $a_1'$ ).

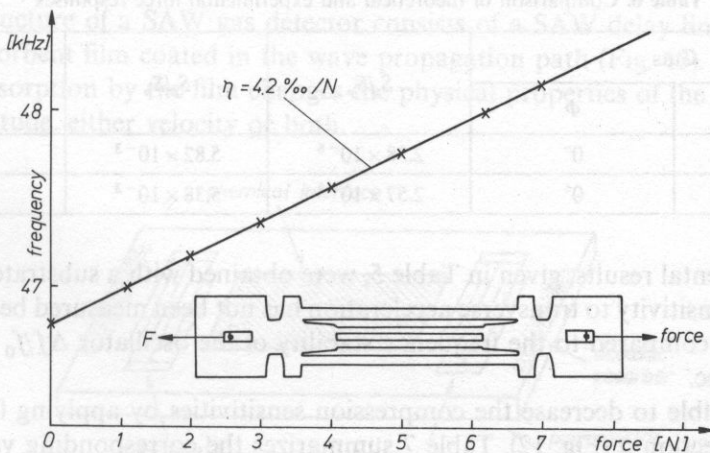


FIG. 10. Frequency shift of double tuning-fork resonator under axial forces (after W. ZINGG)

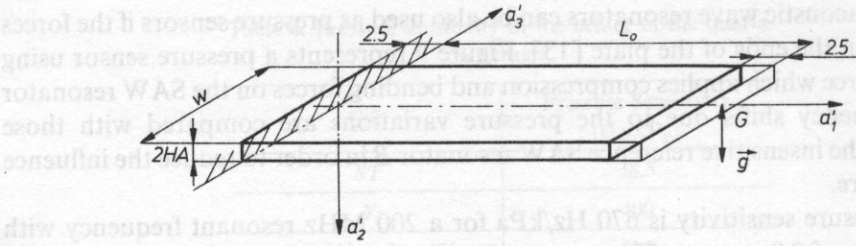


FIG. 11. Schematic of quartz cantilever beam ( $L_0 = 200$  mm,  $2HA = 0.5$  mm,  $W = 10$  mm,  $M = 2.6$  g)

Three different forces can be applied on the SAW substrate as the inertial mass is accelerated: main bending force  $F_2$  along the  $a'_2$  axis, transverse bending force  $F_3$  ( $a'_3$ ), and compression force  $F_1$  ( $a'_1$ ).

Sensitivities are calculated for several cuts of quartz crystal which have a small temperature sensitivity.

Tables 5 and 6 give sensitivities  $S_p$  to the main bending force  $F_2$ , the ratio of the transverse bending force sensitivity to the main sensitivity  $S_t/S_p$ , and the ratio of the compression force sensitivity to the main sensitivity  $S_c/S_p$  for *ST*, *X* cut and *AT*, *X* cut.

Table 5. Acceleration sensitivities of quartz crystal cuts

<i>ST</i> -cut ( $\Psi = 0$ )	Theoretical Values (Hz/g)	Experimental Values (Hz/g)
Main bending force	1387	1318
Transverse bending force	$3.3 \times 10^{-3}$	not measurable
Compression force	8	10

Table 6. Comparison of theoretical and experimental force responses

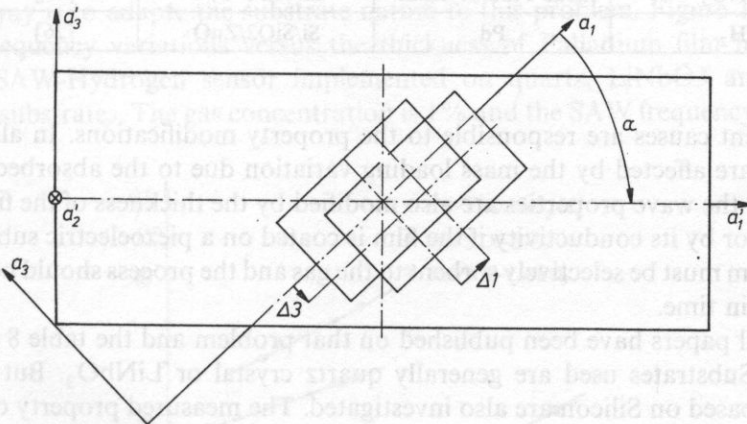
Cuts		$S_t/S_p$	$S_c/S_p$	$S_p$ (Hz/g)
$\theta$	$\phi$			
a) $42.75^\circ$	$0^\circ$	$2.38 \times 10^{-6}$	$5.82 \times 10^{-3}$	1387
b) $35.25^\circ$	$0^\circ$	$2.57 \times 10^{-6}$	$5.38 \times 10^{-3}$	1352

Experimental results, given in Table 5, were obtained with a substrate of *ST*-cut quartz. The sensitivity to transverse acceleration has not been measured because it has a small value compared to the frequency stability of the oscillator  $\Delta f/f_0 = 2 \times 10^{-9}$  over 0.1–10 sec.

It is possible to decrease the compression sensitivities by applying forces in an azimuthal direction  $a$  (Fig. 12). Table 7 summarizes the corresponding values of  $S_p$ ,  $S_c/S_p$ ,  $S_t/S_p$  of *ST*-, *AT*-cuts when the azimuthal angle  $a$  is equal to a critical angle  $a_c$ .

**Table 7.** Force sensitivities at critical azimuthal angle  $a_c$ 

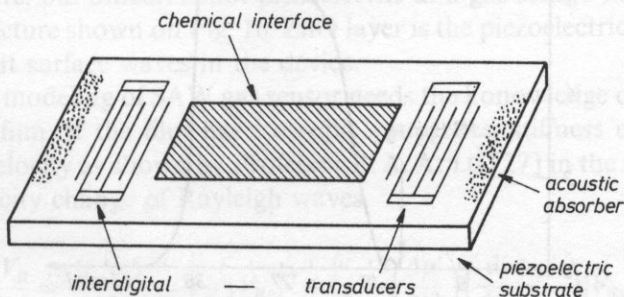
Cuts	$ST, X$	$AT, X$
$a_c$	$62.5^\circ$	$53.5^\circ$
$S_p$ (Hz/g)	90	85
$S_c/S_p$	$< 5 \times 10^{-6}$	$< 5 \times 10^{-6}$
$S_t/S_p$	$1.7 \times 10^{-3}$	$1.2 \times 10^{-2}$

**FIG. 12.** Rotation  $\alpha$  and translations  $\Delta 1, \Delta 3$  of transducers

### 6. Gas sensors

The structure of a SAW gas detector consists of a SAW delay line with a thin selectively sorbent film coated in the wave propagation path (Fig. 13).

Gas absorption by the film changes the physical properties of the surface wave either amplitude, either velocity or both.

**FIG. 13.** Schematic of a SAW gas sensor

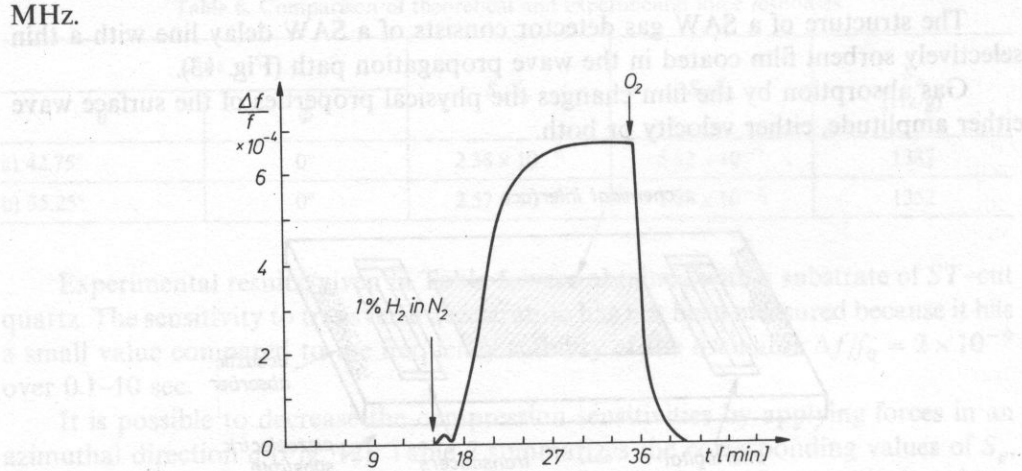
**Table 8.** Some gas sensor examples

Gas	Chemical film	Propagation Substrate and cut	Reference
H <sub>2</sub>	Pd	Quartz-ST, X	[22]
NH <sub>3</sub>	Pt	Quartz-ST, X	[23]
NO <sub>2</sub>	Phtalocyanine	Quartz-ST, X	[21]
H <sub>2</sub> S	WO <sub>3</sub>	LiNbO <sub>3</sub> -Y, Z	[24]
SO <sub>2</sub>	Triethanolamine	LiNbO <sub>3</sub> -Y, Z	[24], [18]
NO <sub>2</sub>	Lead Phtalocyanine	LiNbO <sub>3</sub> -Y, Z	[25]
H <sub>2</sub>	Pd	Si/SiO <sub>2</sub> /ZnO	[26]

Different causes are responsible to the property modifications. In all cases, the properties are affected by the mass loading variation due to the absorbed gas mass. Sometimes, the wave properties are also modified by the thickness of the film (dispersion effect) or by its conductivity if the film is coated on a piezoelectric substrate. The chemical film must be selectively sorbent to the gas and the process should be reversible and stable in time.

Several papers have been published on that problem and the table 8 gives some examples. Substrates used are generally quartz crystal or LiNbO<sub>3</sub>. But composite structures based on Silicon are also investigated. The measured property of the SAW has been the velocity shift, then the frequency variation of an associated oscillator.

For example, describing the features of an hydrogen gaz sensor built by the italian team of Roma (E. VERONA et al.). An ST-cut of quartz crystal delay line is coated with wave propagation on path with a Palladium film, which is selectively sensitive to hydrogen. The film thickness is about 2000 Å and the wave frequency is close to 100 MHz.

**FIG. 14.** Time response of a SAW H<sub>2</sub> sensor upon gas absorption and desorption (after E. VERONA)



Upon gas exposure, the phase velocity reaches in a few minutes a new steady state value (Fig. 14).

When the gaz exposure is switched off, desorption takes place and the velocity comes back to the initial value.

The different time constants (rise time, fall time) are depending on the temperature and the concentration of the gas.

Generally, if the gas concentration increases, velocity variations and the fall time increase and the rise time is decreasing.

To improve the resolution in relative velocity variation (or frequency variation), the film thickness could be thicker; but that also increases the rise time and the fall time. Another way is to adapte the substrate nature to this problem. Figure 15 shows the relative frequency variations versus the thickness of Palladium film measured on different SAW-Hydrogen sensor implemented on quartz, LiNbO<sub>3</sub> and composite-Silicon substrates. The gas concentration is 1% and the SAW frequency is 80 MHz.

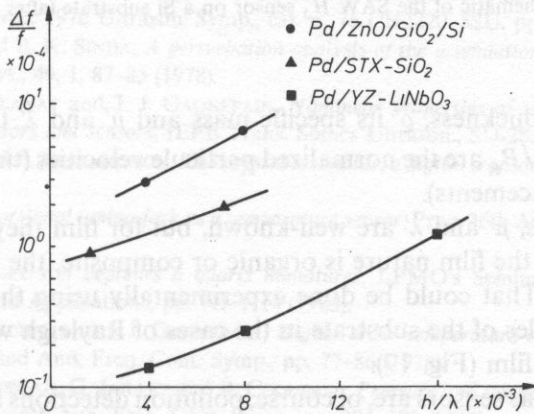


FIG. 15. Relative frequency shifts vs. Pd layer thickness for different SAW substrates (after E. VERONA)

If quartz and LiNbO<sub>3</sub> substrates are piezoelectric, then sensors have simple regular structure, but Silicon is not piezoelectric and gas sensor needs to implement composite structure shown on Fig. 16. ZnO layer is the piezoelectric film deposited on Si/SiO<sub>2</sub> to excite surface waves in the device.

Complete modeling of SAW gas sensor needs the knowledge of elastic constants of the coated film. If the film has isotropic properties, stiffness constants leads to calculate the velocity as shown by Professor B. A. AULD [27] in the relation giving the fractional velocity change of Rayleigh waves

$$\frac{\Delta V_R}{V_R} = -\frac{1}{hV_R h P_R} \left[ \rho' |V_{Ry}|^2 + \left\{ \rho' - \left( \frac{4\mu'}{V_R^2} \right) \left( \frac{\lambda' + \mu'}{\lambda' + 2\mu'} \right) |V_{Rz}|^2 \right\} \right]$$

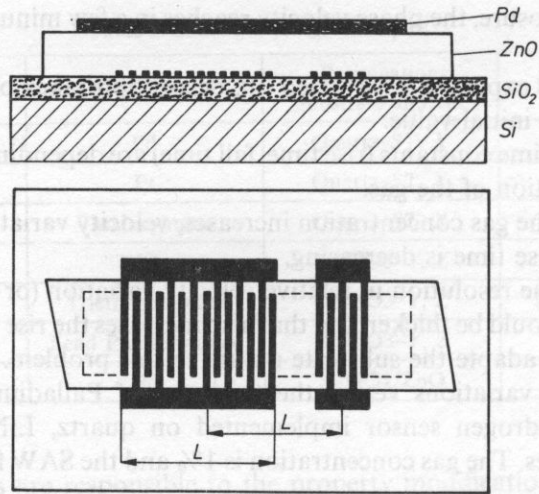


FIG. 16. Schematic of the SAW  $H_2$  sensor on a Si substrate (after E. VERONA)

where  $h$  is the film thickness,  $q'$  its specific mass and  $\mu'$  and  $\lambda'$  the Lamé constants.  $|V_{Rz}|/\sqrt{P_R}$  and  $|V_R|/\sqrt{P_R}$  are the normalized particle velocities (time derivatives of the instantaneous displacements).

For bulk metals,  $\mu'$  and  $\lambda'$  are well-known, but for film they are often undetermined. Moreover, if the film nature is organic or composite, the unknown constants must be measured. That could be done experimentally using the velocity variation value versus cut angles of the substrate in the cases of Rayleigh waves or bulk waves interacting with the film (Fig. 17).

Application of gas sensors are, of course, pollution detections in air, but also in the case of gas weapon detection, which is became a serious potential problem today.

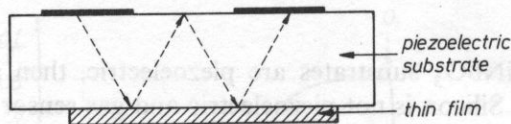


FIG. 17. Lamb wave device for measurement of film constants and for viscosity measurements

## 7. Conclusion

Surface acoustic devices or miniaturized bulk wave resonators are potentially efficient for sensor applications with different physical or chemical measurements. Some of them have been described in this paper.

Other applications studied in different laboratories include high-voltage probes, magnetic sensors, and deposited mass sensors. In the first application,  $\text{LiNbO}_3$  is often used because of its strong piezoelectric coupling [15], [16], or because of the coupling with conduction electrons [17].

All of these sensors have frequency as output quantity, and so give digital information after frequency counting. In metrology and instrumentation, as in industrial activities, digital electronics is playing an increasing role in process and control, and new sensors will be necessary to use with new standards. With these sensors one obtains a direct value of the quantity from the frequency, or by determining frequency shift relative to a reference device, or measurement after frequency multiplication and mixing to increase sensitivity and resolution.

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