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Influence of electromagnetic interferences on the mass sensitivity of Love mode surface acoustic wave sensors

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10 Abstract

3

Surface acoustic waveguides have found an application for (bio)chemical detection. The mass modification due to surface adsorption leads 11 to measurable changes in the propagation properties of the waveguide. Among a wide variety of waveguides, the Love mode device has 12 been investigated because of its high mass sensitivity. The acoustic signal launched and detected in the waveguide by electrical transducers 13 is accompanied by an electromagnetic wave; the interaction of the two signals, easily enhanced by the open structure of the sensor, creates 14 interference patterns in the transfer function of the sensor. The interference peaks are used to determine the sensitivity of the acoustic device. 15 We show that electromagnetic interferences generate a distortion in the experimental value of the sensitivity. This distortion is not identical 16 for the two classical instrumentation of the sensor that are the open and the closed loop configurations. Our theoretical approach is completed 17 by the experimentation of an actual Love mode sensor operated under liquid conditions and in an open loop configuration. The experiment 18 indicates that the interaction depends on frequency and mass modifications. 19 20 © 2004 Published by Elsevier B.V.

21 Keywords: Surface acoustic wave; Electromagnetic wave; Love mode; Interferences; Gravimetric sensitivity; Biosensor

22

23 1. Introduction

Acoustic waves guided by the surface of solid structures 24 form waveguides used as delay lines and filters in telecom-25 munications [1]. Waveguides support different modes with 26 specific strain and stress fields [2]. The acoustic velocity of 27 each mode depends on different intrinsic and extrinsic pa-28 rameters such as the mechanical properties of the materials, 29 the temperature or the applied pressure. Waveguides are used 30 as sensors when the velocity change is linked to environmen-31 tal changes. For gravimetric sensors, the outer surface of the 32 waveguide is exposed to mass changes. Due to the confine-33 ment of the acoustic wave energy close to the surface, these 34 35 sensors are well suited for (bio)chemical sensors operating in gas or liquid media. Among a wide variety of waveguides

used for that purpose, Love mode sensors have attracted an 36 increasing interest during the last decade [3,4]. A Love mode 37 is guided by a solid overlayer deposited on top of a sub-38 strate material. The usual substrates are piezoelectric ma-39 terials like quartz, lithium tantalate and lithium niobate [5]. 40 Associated to specific crystal cut of these substrates, the Love 41 mode presents a shear-horizontal polarization that makes it 42 suitable for sensing in liquid media. 43

Current research in Love mode sensors concerns the guid-44 ing materials in order to optimize the sensitivity, that is the 45 variation of the acoustic signal under surface modifications. 46 Typical materials under investigations are dielectrics like sili-47 con dioxide and polymers, and more recently semiconductors 48 with piezoelectric properties like zinc oxide [6–8]. Although 49 the dispersion relation for Love mode is well set and the de-50 pendence of the sensitivity of the liquid loaded sensor to the 51 overlayer thickness has been thoroughly investigated [9-11], 52 little has been devoted to study the role played by the structure 53 of the sensor and their transducers. 54

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In this paper, we investigate the role played by the structure 55 of the sensor and by the interferences between the acoustic 56 and the electromagnetic waves on the sensitivity. In the first 57 part, we present a general model of the transfer function in-58 cluding the influence of electromagnetic interferences. In the 59 second part, we show how these interferences modify the sen-60 sitivity in open and closed loop configurations of the sensor. 61 Finally, these effects are illustrated experimentally on a Love 62 mode sensor. 63

64 2. Modeling

Waveguide sensors consist of a transducing part and a 65 sensing part. The transducing part includes the generation and 66 the reception of acoustic signals and their interfacing to an 67 electrical instrumentation. The most common transducers are 68 the widespread interdigital transducers (IDTs) on piezoelec-69 tric substrates introduced by White and Voltmer in 1965 [12]. 70 Although the transducing part can be involved in the sensing 71 part, practical sensing is confined to the spacing between the 72 transducers. This confinement takes especially place when 73 liquids are involved since these produce large and unwanted 74 capacitive coupling between input and output electrical trans-75 ducers. This coupling dramatically deteriorates the transfer 76 function and is an important issue for the instrumentation and 77 the packaging of the sensors. 78

The sensor itself is configured as a delay line formed by 79 two transducers separated by a certain distance. The sensor 80 can also be configured as a resonator but we will restrict our 81 approach to the delay line configuration because the operation 82 principle in these two configurations is not similar. The Love 83 mode sensor is sketched in Fig. 1. Transducers with a con-84 stant apodization are identified to their midpoint; the distance 85 between the midpoints is L and the interdigitated electrodes 86 have a periodicity λ_{T} . The sensing part is located between 87 the transducers and covers a total length D so that $D \leq L$. 88 The guided mode propagates with a phase velocity $V = \omega/k$, 89 where $\omega = 2\pi f$ is the angular frequency and $k = 2\pi/\lambda$ is the 90 wavenumber. The waveguide is dispersive when the group 91 velocity ($V_{\rm g} = d\omega/dk$) differs from the phase velocity. 92

The velocity is a function of the frequency and of the 93 surface density $\sigma = M/A$ for a rigidly bound and non viscous 94 mass M per surface area A. For an uniformly distributed mass, 95 the surface density is rewritten in terms of material density ρ 96 and thickness d by $\sigma = \rho d$. The phase velocity for an initial 97 and constant mass σ_0 is denoted V_0 , and the group velocity 98 V_{g0} . In the sensing part, the phase velocity is V and the group 99 velocity $V_{\rm g}$. According to this model, the transit time τ on the 100 delay line is given by 101

102
$$\tau = \frac{D}{V} + \frac{L - D}{V_0}.$$
 (1)

Electromagnetic interferences are due to the cross-talk between the IDTs [13]. The electromagnetic wave (EM) emitted by the input transducer travels much faster than the acoustic





Fig. 1. Structure of the acoustic device.

wave and therefore is detected at the output transducer with-106 out noticeable delay. At the output transducer, the two kinds 107 of waves interact with an amplitude ratio, denoted by α , that 108 creates interference patterns in the transfer function $H(\omega)$ of 109 the delay line. The transfer function itself is given by the ratio 110 of the output to the input voltages. The transfer function with 111 electromagnetic interferences is modeled by the following 112 equation: 113

$$H(\omega) = \underbrace{H_{\mathrm{T}}(\omega) \exp(-\mathrm{i}\omega\tau)}_{\text{delay line}} + \underbrace{\alpha H_{\mathrm{T}}(\omega)}_{\mathrm{EM coupling}} . \tag{2}$$
 114

The transfer function $H_{\rm T}(\omega)$ is associated to the design of the transducers. The total transfer function can be rewritten as $H(\omega) = ||H(\omega)|| \exp(i\phi)$ where expressions for the amplitude $||H(\omega)||$ and the phase ϕ are obtained with help of complex algebra:

$$\|H(\omega)\| = \|H_{\rm T}(\omega)\| \|\sqrt{1 + 2\alpha \cos(\omega\tau) + \alpha^2}\|; \qquad (3) \quad {}_{120}$$

$$\phi = \phi_0 - \arctan\left(\frac{\sin(\omega\tau)}{\alpha + \cos(\omega\tau)}\right). \tag{4}$$

The phase ϕ_0 corresponds to the packaging of the sensor 122 and is due to different aspects linked to the instrumentation. 123 It will be assumed independent of the frequency and of the 124 sensing event. The synchronous frequency $\omega_{\rm T} = 2\pi f_{\rm T}$ is de-125 termined by the design of the IDTs and is generally equal to 126 the maximum amplitude of $||H_{T}(\omega)||$ when the wavelength 127 of the acoustic wave λ_0 matches the transducers periodicity 128 λт. 129

The relations (3) and (4) are the sources of ripples in the transfer function at the ripple frequency $\Delta \omega \simeq 2\pi/\tau$, its exact expression depends also of the dispersion on the line. Interference peaks corresponding to the maximum effect are observed at quantified frequencies f_n when $\cos(2\pi f_n \tau) = -1$, that is for frequencies such that

$$f_n = \frac{2n+1}{2\tau} \tag{5}$$

where $n \in \mathbb{N}$ is the interference mode number. A direct relation to the velocity in the sensing area is obtained from this latter equation as seen by replacing the transit time τ by its definition:

¹⁴¹
$$V = \frac{2DV_0 f_n}{(2n+1)V_0 + 2(D-L)f_n}.$$
 (6)

The interference mode numbers are determined by considering the uncovered delay line; in such case $V = V_0$ and D = L, and *n* for the interference peak located below the synchronous frequency (i.e. for $f_n \leq f_T$) is given by

$$n = \left\lfloor \frac{L}{\lambda_{\rm T}} - \frac{1}{2} \right\rfloor \tag{7}$$

while the other peaks are labeled subsequently to their posi-tion with respect to the peak referenced by Eq. (7).

The relative amplitude peak to peak of the perturbation on the amplitude has a maximum effect (in dB) equals to $40 \log[(1 + \alpha)/(1 - \alpha)]$. The amplitude (in dB and normalized to have $||H_T(\omega)|| = 1$) and the phase (in radians) as a function of the frequency are simulated in Figs. 2–5 for different values of α .

¹⁵⁵ Under the influence of the interferences, the phase has ¹⁵⁶ different behaviors function of α :

- (1) when $\alpha = 0$ (no interferences), the phase is linear with the frequency and has a periodicity equal to 2π (Fig. 2);
- (2) when $\alpha < 1$, the phase is deformed but has still a periodicity equal to 2π (Fig. 3);
- 161 (3) when $\alpha = 1$, the phase has a periodicity equal to π 162 (Fig. 4);
- (4) when $\alpha > 1$, the periodicity is lower than π (Fig. 5);
- (5) when $\alpha \to \infty$, the phase is not periodic anymore and its value tends to ϕ_0 .

This specific behavior of the phase under the influence of
the electromagnetic interferences has to be considered while
evaluating the sensitivity.



Fig. 2. Relative insertion loss (top) and phase (bottom) of the transfer function for $\alpha = 0$.



Fig. 3. Relative insertion loss (top) and phase (bottom) of the transfer function for $\alpha = 1/2$.



Fig. 4. Relative insertion loss (top) and phase (bottom) of the transfer function for $\alpha = 1$.



Fig. 5. Relative insertion loss (top) and phase (bottom) of the transfer function for $\alpha = 2$.

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169 3. Sensitivity

Changes in the boundary condition of the waveguide due 170 to the sensing event modify phase and group velocities. As 171 consequence, the transit time of the delay line and the phase of 172 the transfer function are modified. The sensing event is quan-173 tified by recording the phase shift at a fixed frequency (open 174 loop configuration) or the frequency shift at a fixed phase 175 (closed loop configuration). This quantification gives rise to 176 the concept of sensitivity. The sensitivity is not an unique 177 concept for acoustic sensors because various parameters in-178 fluence the acoustic velocity. As example of such parame-179 ters, there is the density and the viscosity of liquid solutions 180 and adsorbed biomolecules film when the device is used as 181 biosensor. The sensitivity is the most important parameter in 182 design, calibration and applications of acoustic waveguide 183 sensors. Its measurement must be carefully addressed in or-184 der to extract the intrinsic properties of the sensor. 185

186 3.1. Definitions of the sensitivity

The *velocity sensitivity S_V* is defined by the change of
 phase velocity as a function of the surface density change at
 a constant frequency. Its mathematical expression is given by
 [10]:

¹⁹¹
$$S_{\rm V} = \left. \frac{1}{V} \frac{\partial V}{\partial \sigma} \right|_{\omega}$$
. (8)

The definition reflects the velocity change in the sensing area only while outside this area the velocity remains unmodified. The expression is general because the initial velocity V of the sensing part does not need to be equal to V_0 ; this situation occurs in practical situations where the sensing part has a selective coating with its own mechanical properties, leading to an initial difference between V and V_0 .

¹⁹⁹ To link the sensitivity (caused by the unknown veloc-²⁰⁰ ity shift) to the experimental values of phase and frequency ²⁰¹ shifts, we introduce two additional definitions related to the ²⁰² open and the close loop configurations, respectively. The ²⁰³ *phase sensitivity* S_{ϕ} is defined by

$$S_{\phi} = \frac{1}{kD} \frac{\mathrm{d}\phi}{\mathrm{d}\sigma},\tag{9}$$

and the *frequency sensitivity* S_{ω} is defined by

$$S_{\omega} = \frac{1}{\omega} \frac{\mathrm{d}\omega}{\mathrm{d}\sigma}.$$
 (10)

207 3.2. Phase differentials without interferences

In order to point clearly the effects of the electromagnetic interferences on the different sensitivities presented in the previous section, we calculate the phase differentials in the ideal case of no interferences. For that case, the phase of the transfer function is a function of the frequency and of the velocities in the different parts of the sensor, themselves function of the frequency and of the surface density:

$$\phi(\omega, V(\omega, \sigma), V_0(\omega)) = -\omega\tau \tag{11}$$

214

217

$$\phi(\omega, V(\omega, \sigma), V_0(\omega)) = -\omega \left(\frac{D}{V} + \frac{L - D}{V_0}\right). \tag{12}$$

Therefore, the total differential of the phase is

$$d\phi = \left(\frac{\partial\phi}{\partial\omega}\Big|_{V,V_0} + \frac{\partial\phi}{\partial V}\Big|_{\omega,V_0} \frac{\partial V}{\partial\omega}\Big|_{\sigma} + \frac{\partial\phi}{\partial V_0}\Big|_{\omega,V} \frac{dV_0}{d\omega}\right)d\omega + \frac{\partial\phi}{\partial V}\Big|_{\omega,V_0} \frac{\partial V}{\partial\sigma}\Big|_{\omega}d\sigma$$
(13)

$$\mathrm{d}\phi = \left. \frac{\partial\phi}{\partial\omega} \right|_{\sigma} \mathrm{d}\omega + \left. \frac{\partial\phi}{\partial\sigma} \right|_{\omega} \mathrm{d}\sigma. \tag{14}$$

The derivative of the phase velocity as a function of the frequency comes from the definitions of phase and group velocities; at constant surface density, we have from [11]: 221

$$\frac{\partial V}{\partial \omega}\Big|_{\sigma} = k^{-1} \left(1 - \frac{V}{V_{\rm g}}\right); \tag{15}$$

$$\frac{\mathrm{d}V_0}{\mathrm{d}\omega} = k_0^{-1} \left(1 - \frac{V_0}{V_{\mathrm{g}0}} \right). \tag{16}$$

The other partial differentials are obtained by differentiation of Eq. (11): 224

$$\left. \frac{\partial \phi}{\partial \omega} \right|_{V,V_0} = -\tau; \tag{17}$$

$$\left. \frac{\partial \phi}{\partial \omega} \right|_{\sigma} = -\tau - \omega \left. \frac{\tau}{\omega} \right|_{\sigma} \tag{18}$$

$$\left. \frac{\partial \phi}{\partial \omega} \right|_{\sigma} = -\tau_{\rm g}; \tag{19} \quad {}_{228}$$

$$\frac{\partial \phi}{\partial V}\Big|_{\omega, V_0} = \frac{\omega D}{V^2}; \tag{20} \quad 229$$

$$\left. \frac{\partial \phi}{\partial V_0} \right|_{\omega, V} = \frac{\omega(L-D)}{V_0^2}.$$
(21) 230

The time of flight τ_g introduced in Eq. (19) is calculated ²³¹ as ²³²

$$\tau_{\rm g} = \frac{D}{V_{\rm g}} + \frac{L - D}{V_{\rm g0}}.$$
(22) 233

3.3. Open loop configuration

In the open loop configuration, the input transducer is excited at a given frequency while the phase difference between output and input transducers is recorded. This configuration with a constant frequency has $d\omega = 0$ in Eq. (13); related 236 237 238 238 239 239 239 239 239 239

phase variations caused by surface density variations are ob-tained by

$$_{^{241}} \quad \frac{\mathrm{d}\phi}{\mathrm{d}\sigma} = \left. \frac{\partial\phi}{\partial V} \right|_{\omega, V_0} \left. \frac{\partial V}{\partial\sigma} \right|_{\omega} \tag{23}$$

$$_{^{242}} \quad \frac{\mathrm{d}\phi}{\mathrm{d}\sigma} = \left. \frac{\partial\phi}{\partial V} \right|_{\omega, V_0} VS_{\mathrm{V}}.$$
(24)

In the absence of interferences, phase variations obtained experimentally are directly linked to velocity changes by the product kD involving the geometry of the sensor as seen by replacing Eq. (20) in Eq. (24):

$$_{247} \quad \frac{\mathrm{d}\phi}{\mathrm{d}\sigma} = k D S_{\mathrm{V}}.\tag{25}$$

In other words: $S_{\phi} = S_{V}$ when there are no interferences. 248 In a first approximation k is assumed equal to $k_{\rm T}$, an as-249 sumption valid as long as the phase shift is evaluated close 250 to the synchronous frequency and for waveguides with low 251 dispersion. The wavelength is only known when the sensing 252 part extends over the transducers (D = L). In that case, the 253 transfer function of the IDTs is modified accordingly to the 254 velocity changes. In practice, the value of the sensitivity is 255 slightly underestimated to its exact value since $k \leq k_{\rm T}$, the 256 error being less than 5%. 257

In the case where interferences occur, the partial differential of ϕ with respect to the velocity is obtained by differentiation of Eq. (4):

$$_{261} \quad \left. \frac{\partial \phi}{\partial V} \right|_{\omega, V_0} = \left(\frac{1 + \alpha \cos(\omega \tau)}{1 + 2\alpha \cos(\omega \tau) + \alpha^2} \right) \frac{\omega D}{V^2}, \tag{26}$$

and the phase sensitivity is obtained by combining the latter
 equation with Eq. (24):

$$S_{\phi} = \left(\frac{1 + \alpha \cos(\omega \tau)}{1 + 2\alpha \cos(\omega \tau) + \alpha^2}\right) S_{V}.$$
 (27)

The influence of electromagnetic interferences on the 265 phase sensitivity is simulated in Fig. 6 versus the relative 266 frequency for different values of α . The phase sensitivity is 267 always different compared to the velocity sensitivity. For the 268 threshold value $\alpha = 1$, the phase sensitivity present a sin-269 gularity and is undefined; for higher values of α , the phase 270 sensitivity is always underestimated to the velocity sensitiv-271 ity. 272

The interference peaks permit a direct evaluation of α because at these points $\cos(\omega \tau) = -1$ and Eq. (27) becomes linear with α :

276
$$\alpha = 1 - \frac{V^2}{\omega D} \left. \frac{\partial \phi}{\partial V} \right|_{\omega, V_0}$$
 (28)

$$\alpha = 1 - \frac{S_{\rm V}}{S_{\phi}}.$$
 (29)



Fig. 6. Phase sensitivity at constant frequency as a function of the relative frequency for different values of simulated interferences obtained by Eq. (27).

3.4. Closed loop configuration

In the closed loop configuration, the frequency is recorded while a feedback loop keeps the phase difference between output and input transducers constant. The configuration at constant phase has $d\phi = 0$, the variation of the frequency as a function of the mass change is given by introducing this condition in Eq. (14):

$$\frac{\mathrm{d}\omega}{\mathrm{d}\sigma} = \left(\left.\frac{\partial\phi}{\partial\sigma}\right|_{\omega}\right) \left(\left.\frac{\partial\phi}{\partial\omega}\right|_{\sigma}\right)^{-1}.$$
(30) 285

The upper term is replaced by Eq. (24). The phase slope as a function of the frequency at constant mass is obtained by differentiation of Eq. (4): 288

$$\left. \frac{\partial \phi}{\partial \omega} \right|_{\sigma} = -\left(\frac{1 + \alpha \cos(\omega \tau)}{1 + 2\alpha \cos(\omega \tau) + \alpha^2} \right) \tau_{\rm g}. \tag{31}$$

We can establish a finalized equation taking into account the electromagnetic interferences by combining Eqs. (24), (26) and (31) in Eq. (30): 292

$$S_{\omega} = \frac{DS_{\rm V}}{V\tau_{\rm g}}.$$
(32) 293

At the opposite of the open loop configuration, the fre-294 quency sensitivity is not influenced by the interferences. 295 However, as indicated by Eq. (32), the frequency sensi-296 tivity is strongly dependent of the structure of the sen-29 sor and the dispersion characteristics of the delay line. 298 As result, the link between the frequency sensitivity and 299 the velocity sensitivity is difficult to exploit although it 300 can be noticed that $S_{\omega} \leq S_{\rm V}$ since $V_{\rm g} \leq V$ for Love mode 30 devices.

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302 4. Experimental results

For the practical consideration of the described and mod-303 eled behavior, we investigated a Love mode sensor. It was 304 fabricated and tested under liquid condition in the open loop 305 configuration to evaluate the influence of the electromagnetic 306 interferences. In a first part, the sensor fabrication and in-30 strumentation is described, followed in a second part by the 308 application of the model to these results to demonstrate the 309 influence of the interferences on the sensitivity of the sensor. 310

311 4.1. Sensor fabrication and instrumentation

The Love mode was obtained by conversion of a surface 312 skimming bulk wave (SSBW) launched in the direction per-313 pendicular to the crystalline X axis of a 500 µm thick ST-cut 314 (42.5° Y-cut) quartz substrate. The conversion was achieved 315 by a 1.2 µm thick overlayer of silicon dioxide deposited on 316 the top side of the substrate by plasma enhanced chemical 317 vapor deposition (Plasmalab 100 from Oxford Plasma Tech-318 nology, England). Via were etched in the silicon dioxide layer 319 using a standard SF₆/O₂ plasma etch recipe. This process 320 stopped automatically on the aluminum contact pads of the 321 transducers. 322

The transducers consist of split fingers electrodes etched in 200 nm thick sputtered aluminum. The fingers are 5 μ m wide and equally spaced by 5 μ m. This defines a periodicity $\lambda_{\rm T}$ of 40 μ m. The acoustic aperture defined by the overlap of the fingers is equal to $80\lambda_{\rm T}$ (=3.2 mm), the total length of each IDT is $100\lambda_{\rm T}$ (=4 mm) and the distance center to center of the IDTs is $225\lambda_{\rm T}$ (L = 9 mm, D = 5 mm).

The sensing area was defined by covering the space left 330 between the edges of the IDTs by successive evaporation and 331 lift-off of 10 nm of titanium and 50 nm of gold in a first ex-332 periment, and 200 nm of gold in a second experiment. The 333 fingers were protected against liquid by patterning photosen-33 sitive epoxy SU-8 2075 (Microchem Corp., MA) defining 335 120 µm thick and 80 µm wide walls around the IDTs. Quartz 336 glasses of $5 \text{ mm} \times 5 \text{ mm}$ were glued on top of the walls to 33 finalize the protection of the IDTs [14]. 338

The device was mounted and wire-bonded to an epoxy 339 printed circuit board and its transfer function was recorded 340 on a HP4396A Network Analyzer. This setup corresponds to 341 the open loop configuration. Epoxy around the device cov-342 ered and protected it and defined a leak-free liquid cell. The 343 sensing area was immersed in a solution of KI/I₂ (4 and 1 g, 344 respectively, in 160 ml of water) that etched the gold away 345 of the surface [15]. The transfer function of the device was 346 recorded every 4 s (limited by the GPIB transfer speed) dur-347 ing the etching of the gold with a resolution of 801 points 348 over a span of 2 MHz centered around 123.5 MHz. The ini-349 tial transfer function of the device is presented in Fig. 7 with 350 and without gold. The transfer function during etching of the 35 200 nm is shown at two moments (44 and 356 s after etching 352 start) in Fig. 8. The total time for this etching was approxi-353 mately 620 s.



Fig. 7. Initial aspect of the experimentally recorded transfer function of the Love mode sensor with (dashed line) and without (solid line) an overlayer of 200 nm of gold. This device presents an initial phase $\phi_0 = \pi$, leading to a vertical offset by π compared to the simulated phase curve represented in Fig. 3.

4.2. Correlation of the results with the model

The correlation of the experimental results with the model is presented in two steps. In the first step, we show the calculation of the phase velocity from the interference peaks; and in the second step, we evaluate the mass sensitivity in the open loop configuration by the delay phase angle and the phase velocity variations recorded during the gold etching.

The record of the interference peaks frequency f_n during a sensing event permits to follow the evolution of the phase velocity in the sensing area either for constant and integer values of the interference mode numbers *n* as given by Eq. (6),



Fig. 8. Aspect of the experimentally recorded transfer function at two different moments of the etching of 200 nm of gold (solid line after 44 s and dashed line after 356 s). The solid line shows a value of α close to 1 around 123.5 MHz.

⁶

хÒ



Fig. 9. Interferences mode in the amplitude of the transfer function as a function of time and frequency.

either by sampling the mode numbers at a constant frequency. Fig. 9 plots the interference peaks versus time and frequency for the etching of the 200 nm thick gold layer; the interference mode numbers *n* were attributed according to Eqs. (6) and (7) with $V_0 = 4940$ m/s (given by the synchronous frequency of $f_T = 123.5$ MHz times the transducers periodicity λ_T).

The evolution of the velocity in the sensing area with time 372 is representative of the etching rate of the gold layer and 373 is plotted for three different frequencies (123.5, 123.75 and 374 124 MHz) in Fig. 10. At three different frequencies, the values 375 of velocity should differ as a function of the group velocity. 376 This effect is seen better when the probing frequencies are 377 taken far away from each others and for a strongly dispersive 378 delay line, which is not the case for the experimental device 379 presently used. 380

At constant frequency, the peaks are spaced by an unit variation of n, therefore the velocity difference measured be-

4950



Fig. 10. Evaluation of the acoustic velocity on the sensing are during the etching of the gold as a function of time for different values of frequency.



Fig. 11. Evaluation of α at the position of the interference peak.

tween two peaks is obtained by differentiation of Eq. (6) with respect to *n*: 383

$$\frac{\partial V}{\partial n}\bigg|_{f_n,V_0} = -\frac{4DV_0^2 f_n}{[(2n+1)V_0 + 2(D-L)f_n]^2},$$
(33) 38

which gives a variation roughly equals to -40 m/s between 386 two peaks at the three sampling frequencies. From the acous-387 tic velocity variation (4610 m/s for 200 nm gold to 4940 m/s 388 when all the gold is etched) and by assuming that gold has a density of $\rho = 19.3 \text{ g/cm}^3$, we have an evaluation of S_V 390 equals to $-173 \,\mathrm{cm}^2/\mathrm{g}$. Because the phase loses its period-39 icity for the thick gold layer, we were not able to deter-392 mine a value for the phase variation and consequently we 393 have no value for S_{ϕ} . The Eq. (28) was employed to esti-394 mate the value of α at the interference peak; the result is 395 displayed in Fig. 11 that demonstrates a variation of α with 396 the frequency. Around the synchronous frequency, α equals 397 0.33 and the phase has a periodicity of 2π ; but as the fre-398 quency is far from the synchronous frequency, α clearly 399 change above the critical value of 1 (in the present case, 400 $\alpha = 5.7$). The consequence is seen in the phase that presents 401 at this point of calculation a positive slope and a periodicity 402 below π . 403

We applied the same procedure to the thinner gold layer 404 of 50 nm. Fig. 12 shows the transfer function recorded be-405 fore and after the gold etching; the interference mode num-406 ber 225 has been followed and give a velocity varying from 407 4876.5 to 4940 m/s. The resulting velocity sensitivity is $S_V =$ 408 $-96 \,\mathrm{cm}^2/\mathrm{g}$. This value is lower than the one obtained by etch-409 ing of the thick gold layer since a thicker layer enhances the 410 sensitivity due to a better entrapment of the acoustic energy 411 in the top guiding layer. 412

The phase sensitivity S_{ϕ} could be calculated for frequencies where α remained inferior to the critical value of 1, that is close to the synchronous frequency. The result is plotted versus the frequency in Fig. 13 and compared with the estimated value of $S_{\rm V}$ while the values of α indicated on the 8

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Fig. 12. Transfer function before and after the etching of 50 nm of gold. The arrows indicate the interference mode 225 followed to estimate the velocity sensitivity.



Fig. 13. Phase sensitivity relative to the velocity sensitivity as a function of the frequency and computed from the experimental data obtained by etching 50 nm of gold. Oscillations are attributed to the electromagnetic interferences.

graph have been estimated at the interference peaks thanks to
Eq. (29). The graphs shows that the interferences modify the
value of the sensitivity as given by Eq. (27). A comparison of
the Figs. 6 and 13 shows the correlation between the theoretical modeling of the effects of electromagnetic interferences
on the sensitivity of the surface acoustic waveguide sensor
and the experimental results.

425 5. Discussion

Electromagnetic interferences have a clear effect on the transfer function of the acoustic device because of the ripples they cause. The interaction modeled as a constant factor α is specific to each device and must be identified by a careful inspection of the transfer function. The amplitude of the 430 transfer function peak to peak is supposed to be the product 431 between the transfer function of the transducers and the in-432 terference, and therefore an evaluation of α is possible if the 433 transfer function of the transducers only is known. However, 434 the experiment shows that α is a function of the frequency 435 and the surface density, indicating that finding its exact value 436 is not straightforward. Only the phase indicates whether α is 437 higher or lower than one. 438

In term of sensitivity, when $\alpha \ge 1$ the phase has a periodicity *P* in the range $0-\pi$. We suggest the following correction to the experimental phase sensitivity: 441

$$S_{\phi} = \frac{2\pi}{P} \frac{1}{kD} \frac{\mathrm{d}\phi}{\mathrm{d}\sigma}.$$
(34) 442

This modification gives a better evaluation of the velocity 443 sensitivity by stretching the phase of the transfer function to 444 2π . Only the extraction of P is not immediate since it depends 445 upon α . From a physical point of view, α indicates the strength 446 of the electromagnetic wave in comparison with the acoustic 447 wave. For a constant amplitude of the EM wave, a higher α 448 stands for a larger attenuation of the acoustic wave; its precise 449 value is an indication of the actual attenuation of the acoustic 450 wave along the delay line. 45

The observation of the interference peaks in the experi-452 mental part was facilitated by the large velocity change in-453 duced by the gold coating. Indeed, 50 nm of gold corresponds 454 to a surface density of 96.5 μ g/cm², a relatively large shift in 455 comparison to the targeted (bio)chemical recognition appli-456 cation where molecules films surface density are in the order 457 of hundreds of ng/cm² and even lower. The calibration of the 458 sensitivity is best recorded by adding or etching thin layers of 459 materials and that under the operating conditions of the sen-460 sor, especially if liquids are involved [16]. In (bio)chemical 461 measurements, the precision on the velocity measurement de-462 pends upon the assessments on the initial conditions (i.e. V_0 463 and n) but also on the induced variation of velocity, which 464 is function of the velocity sensitivity of the waveguide. The 465 evaluation of the mass sensitivity by the frequency variation 466 of an interference peak is identical to a closed loop measure-467 ment locked on the interference peak instead on a constant 468 value of the phase. For the detection of a minimum value of 469 the surface density $\Delta \sigma$, the frequency shift of an interference 470 peak must be measured with a precision estimated from Eq. 471 (32): 472

$$\Delta f_n = \frac{DS_V f_n}{V \tau_g} \Delta \sigma, \tag{35} \quad {}_{47}$$

that gives $\Delta f_n / \Delta \sigma \simeq -6.5 \text{ cm}^2 \text{ Hz/ng}$ in the present case. The detection of a monolayer of proteins, about 400 ng/cm², requires to detect a frequency variation of 2.6 kHz, which is compatible with the instrumentation of surface acoustic waveguide sensors.

One benefit of our calculation method resides in the possibility to still measure the acoustic velocity in the sensing area even when the electromagnetic and the acoustic waves

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are strongly interfering, in particular for α greater than one. 482 Strong interferences are unwanted in an experimental set-up 483 because they prevent the correct electrical measurement of 484 the sensor. It must be noticed that for these high values of 485 interference, the electromagnetic wave amplifies the acous-486 tic signal as seen in Fig. 5, which could be of interest for 487 the operation of the device even in conditions where the 488 acoustic signal is weak. Finally, the presented method op-480 erates directly on the raw signal of the acoustic device thus 490 avoiding a lost or a modification of the physical information 491 it carries. 492

6. Conclusion 403

We have proposed a model for surface acoustic waveg-494 uides used as sensors. The model shows the influence of elec-495 tromagnetic interferences caused by interdigital transducers 496 on the velocity sensitivity in open and closed loop configu-497 rations. In both cases, the dimensions of the delay line and the sensing part influence the experimental value of phase or 499 frequency shifts. 500

The interference peaks in the transfer function offer an 501 unique possibility to access the information about the acous-502 tic phase velocity in the sensing area. The velocity sensitivity 503 was calculated directly from these peaks. 504

In an open loop configuration and with interferences, the 505 phase shift is disturbed and the sensitivity is over- or under-506 estimated to the value of the velocity sensitivity. For strong 507 interferences, the phase has a periodicity lower than 2π that 508 must be considered when normalizing the phase shift to ob-509 tain a correct figure of the sensitivity. 510

In a closed loop configuration and with interferences, the 511 frequency shift is not disturbed. The frequency shift is pro-512 portional to the sensitivity by the ratio between the length of 513 the sensing area and the distance separating the transducers. 514 In addition, the frequency shift is influenced by the dispersive 515 516 properties of the waveguide.

The influence of the electromagnetic interferences on the 517 transfer function of a Love mode sensor operating in liquid 518 conditions was presented for a comparison. From the experi-519 ment it appears that the interferences are function of both the 520 frequency and the surface density. 521

For future investigations, an analytical expression of the 522 electromagnetic-acoustic interaction and the parameters act-523 ing on it have to be identified in order to reduce the influence 524 or, on the opposite, to enhance the velocity sensitivity of sur-525 face acoustic waveguides. 526

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References

[1] C. Campbell, Surface Acoustic Wave Devices and Their Signal Pro-	535
cessing Applications, Academic Press, San Diego, 1989.	536

- [2] B.A. Auld, Acoustic Fields and Waves in Solids, vol. 2, Wiley, New 537 York, 1973. 538
- [3] G.L. Harding, J. Du, P.R. Dencher, D. Barnett, E. Howe, Love wave 539 acoustic immunosensor operating in liquid, Sens. Actuator A Phys. 61 540 (1997) 279 - 286541
- [4] E. Gizeli, Design considerations for the acoustic waveguide biosensor, Smart Mater. Struct. 6 (1997) 700-706.
- [5] F. Herrmann, M. Weinacht, S. Büttgenbach, Properties of sensors 544 based on shear-horizontal surface acoustic waves in LiTaO₃/SiO₂ and 545 Quartz/SiO2 structures, IEEE Trans. Ultrason. Ferroelectr. Freq. Con-546 trol 48 (2001) 268-273. 547
- [6] A. Rasmusson, E. Gizeli, Comparison of poly(methylmethacrylate) and 548 Novolak waveguide coatings for an acoustic biosensor, J. App. Phys. 549 90 (2001) 5911-5914. 550
- [7] G.L. Harding, Mass sensitivity of Love-mode acoustic sensors incor-551 porating silicon dioxide and silicon-oxy-fluoride guiding layers, Sens. 552 Actuator A Phys. 88 (2001) 20-28. 553
- [8] K. Kalantar-Zadeh, W. Wlodarski, Y.Y. Chen, B.N. Fry, K. Galatsis, 554 Novel Love mode surface acoustic wave based immunosensors, Sens. 555 Actuator B-Chem. 91 (2003) 143-147. 556
- [9] Z. Wang, J.D.N. Cheeke, C.K. Jen, Sensitivity analysis for Love mode 557 acoustic gravimetric sensors, Appl. Phys. Lett. 64 (1994) 2940-2942. 558
- [10] B. Jakoby, M. Vellekoop, Properties of Love waves: applications in 559 sensors, Smart Mater. Struct. 6 (1997) 668-679. 560
- [11]G. McHale, F. Martin, M.I. Newton, Mass sensitivity of acoustic wave devices for group and phase velocity measurements, J. Appl. Phys. 92 562 6 (2002) 3368-3373 563
- [12] R.M. White, F.W. Voltmer, Direct piezoelectric coupling to surface elastic waves, Appl. Phys. Lett. 7 (1965) 314-316.
- [13] G. Feuillard, Y. Janin, F. Teston, L. Tessier, M. Lethiecq, Sensitivi-566 ties of surface acoustic wave sensors based on fine grain ceramics, 567 in: Instrumentation and Measurement Technology Conference 1996 568 (IMTC-96), Conference Proceedings 'Quality Measurements: The In-569 dispensable Bridge Between Theory and Reality', vol. 2, IEEE, 1996, 570 pp. 1211–1215. 571
- [14] L.A. Francis, J.-M. Friedt, C. Bartic, A. Campitelli, An SU-8 liquid 572 cell for surface acoustic wave biosensors, Proc. SPIE 5455 (2004) 353-573 363 574
- [15] J.L. Vossen, W. Kern, Thin Film Processes, Academic Press, New York, 575 1978 576
- [16] J.-M. Friedt, L. Francis, K.-H. Choi, F. Frederix, A. Campitelli, Com-577 bined atomic force microscope and acoustic wave devices: application 578 to electrodeposition, J. Vac. Sci. Technol. A21 (2003) 1500-1505. 579

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