SURFACE ACOUSTIC WAVE REFLECTIVE DELAY LINE AS PASSIVE COOPERATIVE TARGET FOR WIRELESS SUB-SURFACE SENSING IN LIQUID USING GROUND PENETRATING RADAR

Jean-Michel Friedt[1*], David Rabus[1,2], Lilia Arapan[1,3], Vincent Luzet[1], Frédéric Chérioux[1]

[1]Université de Franche-Comté, FEMTO-ST, CNRS, 25000 Besançon, France
[2]Current affiliation Statice SAS, 25000 Besançon, France
[3]Current affiliation Silmach SA, 25000 Besançon, France
**jmfriedt@femto-st.fr*

Keywords: surface acoustic wave, cooperative target, ground penetrating radar, lithium tantalate, sensor

Abstract

Ground Penetrating RADAR (GPR) is a classical geophysical tool for detecting sub-surface dielectric interfaces. In this context, we complement this measurement by quantitatively sensing various quantities including temperature, stress, and most importantly, the concentration of chemical compounds in a liquid phase. This is achieved using dedicated cooperative targets designed as reflective surface acoustic wave delay lines which are manufactured on lithium tantalate oxide (LTO) piezoelectric substrates. LTO is chosen for detecting sub-surface chemical compounds in liquid phase due to its ability to generate and propagate pure shear acoustic waves, resulting in minimal insertion losses even in the presence of water. Furthermore, the high permittivity LTO leads to minimal impact of the presence of water over interdigitated transducer electrodes, removing the need for challenging microfluidic packaging otherwise needed to prevent capacitive short circuit. The reflective delay line design operating in the 100 to 500 MHz range induces echoes delayed beyond subsurface interface echoes – here associated to clutter – whose time difference is solely representative on the quantity under investigation, whether temperature, stress or chemical concentration depending on the surface functionalization, independently on the distance from the GPR to the sensor. This concept is demonstrated with the direct detection of protein physisorption without the need for sensor packaging.

1 Introduction

Ground Penetrating RADAR (GPR) is a classical geophysical tool for detecting shallow sub-surface dielectric interfaces in the decimeter to hundreds of meter range depending on the conductivity properties of the sub-surface media and the operating frequency typically in the tens to thousands of megahertz depending on the targeted resolution and range [1, 2].

While GPR is well-suited for mapping sub-surface interfaces, including voids, the commonly available instrument cannot measure subsurface quantities such as temperature, stress, or the presence and concentration of chemical compounds.

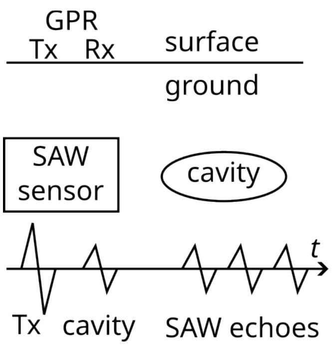

Fig. 1 Experimental setup: Surface Acoustic Wave (SAW) transducers buried underground act as wireless passive sensors probed by Ground Penetrating RADAR (GPR). Despite being at the same depth than sub-surface passive interfaces such as cavities, the echoes induced by the delay line (bottom) are beyond clutter thanks to the slow acoustic wave delaying the SAW echoes well beyond the passive interface backscattered signal despite millimeter-long piezoelectric substrate dimensions.

Surface Acoustic Wave (SAW) transducers are classical radio-frequency signal processing components [3, 4]. In SAW transducers, the incoming electromagnetic signal is converted into an acoustic wave by a piezoelectric substrate patterned with interdigitated transducer (IDT) electrodes. This acoustic wave propagates on the surface of the piezoelectric substrate before being either converted back to an electromagnetic wave in a transmission mode transducer, or being reflected by mirrors patterned as electrodes on the surface to propagate back to the IDT and radiated back as electromagnetic waves. Therefore, although SAW transducers appear as electrical components to users, the underlying physical principle relies on the propagation of acoustic waves, whose velocity depends on the mechanical properties of the piezoelectric substrate used in the SAW device. This velocity is influenced by temperature, stress or boundary conditions when chemical compounds are adsorbed on the free surface between IDTs or IDT and mirrors.

Since acoustic waves are about 10^5 times slower than electromagnetic waves, the resulting transducer dimensions are reduced by the same factor with respect to the electromagnetic wavelength: IDT electrodes operating in the 50 to 1000 MHz range are patterned using optical lithography with dimensions in the 25 to 1.25 µm (assuming an acoustic velocity of 5000 m/s), well within the capabilities of cleanroom lithography technologies. Furthermore, thanks to this slow acoustic velocity, generating an echo in the microsecond range to delay the sensor response beyond sub-surface interfaces only requires a 5 mm-long (assuming an acoustic velocity of 5000 m/s and a delay of 10^{-6} s), acoustic path, leading to a compact sensing element easily fitted to sub-surface utilities such as pipes or cables (Fig. 1).

We, and others, have previously demonstrated how SAW transducers made on the strongly coupled lithium niobate oxide (LNO) piezoelectric substrate allow for wireless sub-surface sensing [5-10], assuming the GPR instrument is designed with a stable enough timebase in order to allow for fine time-of-flight delay analysis of the echoes returned by the transducer [11, 12]. However, LNO propagates a Rayleigh wave with an out-of-plane component that would radiate in the liquid of the sensing area if the transducer is coated with such a medium, leading to a propagating wave in the liquid and a vanishing surface acoustic wave. Therefore, LNO is not compatible with chemical sensing in a liquid environment The current investigation aims to replace LNO with LTO in order to replace the Rayleigh SAW with a pure shear wave that hardly interacts with the fluid, yet provides high gravimetric sensitivity. The acoustic wavelength in the micrometer range is well suited for detecting chemical interactions when the sensing areas between IDT and mirrors are coated with organic thin films deposited with similar thicknesses as achieved with cleanroom spin-coating techniques. Furthermore, while low-permittivity piezoelectric substrates, such as quartz, have been susceptible to capacitive short circuits when coated with high-permittivity fluids, such as water, the high-permittivity LTO is resistant to such effects, and the fraction of the electric field leaking into the water only marginally affects the insertion losses.

2. Methodology

Based on the analysis presented above, we have designed a dipole reflective delay line compatible with the pulsed mode GPR as implemented in most commercial instruments. To redirect a portion of the incoming acoustic energy back toward the IDT, the number of electrodes in the mirrors is adjusted to fine-tune the reflection coefficient. Multiple mirrors are patterned on the LTO substrate, defining multiple sensing areas. Each of those areas is coated with a different thin organic film for sensing purpose. One of these areas is coated with a chemically inert sensing layer to provide a reference delay and compensate for the delay induced by the distance between the GPR and the sensor.

To achieve the optimal conversion of the incoming electromagnetic wave into an acoustic wave, the number of electrodes in the IDTs is selected to be equal to the inverse of the piezoelectric substrate electromechanical coupling coefficient [3]. This typically involves using a few tens of electrodes for electrochemical coefficients in the few percent range, such as found in LTO or LNO (Fig. 2). The mirrors are apodized to control the shape of the reflected echoes and avoid energy spreading in time according to the sinc() function that would result from a uniform overlap of the IDT.

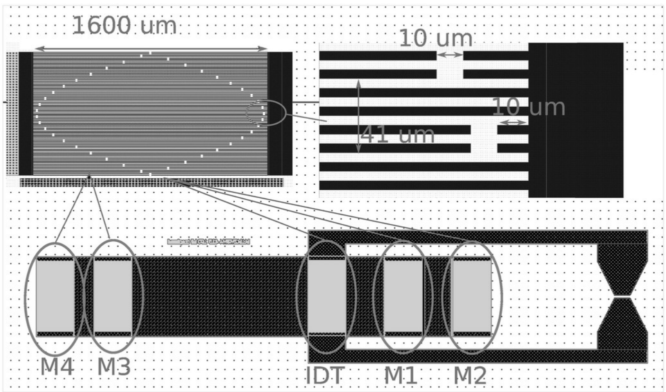

Fig. 2 Interdigitated transducer (IDT) and mirrors (M1 to M4) layout, with the dark areas representing metal patterned on the lithium tantalate substrate.

However properly designed GPR instruments aim at emitting a single pulse, leading to a broader spectrum than the coupling bandwidth of the transducer and additional losses due to the fraction of the electromagnetic energy effectively converted to acoustic energy. On the other hand, the multitude of periods in the returned echoes allow for averaging and fine measurement of the time delay measured as the phase of the backscattered signal. Either a polynomial fit of the cross-correlation of the measurement echo to the reference echo or a difference of the phase of the Hilbert transform of the returned signal at the delays of the reference and measurement echoes allow for a time-of-flight measurement with better resolution than the sampling period. Considering a few tens of ppm/K temperature sensitivities of the piezoelectric substrate, a temperature variation of 1 K leads to a relative delay of 10^{-5} (at 10 ppm/K) or for an echo delayed by 1 µs, a variation of 10 ps. Such a tiny delay variation is experimentally measured as a phase variation: since the phase (in degrees) introduced by a time delay T at center frequency f is 360xfxT, then at a 1 µs delay at 100 MHz the accumulated phase rotation is 36000° and the 10 ppm relative variation is measured as a phase variation of 0.36°, well within the capabilities of vector network analyzers (VNA). When the equivalent sampling rate of the stroboscopic GPR receiver is 1 GS/s, the signal processing improvement on the time delay measurement over the sampling period must hence been 1000-fold.

In the case of chemical sensing, the SAW propagating on the piezoelectric substrate only provides a sensing capability since the acoustic velocity is affected by the boundary condition driven by the layer density (slowing down the wave and hence increasing the delay) and stiffness (speeding up the wave and hence reducing the delay). The selectivity is however brought by the chemical thin film functionalizing the surface to specifically react with the targeted analyte and avoid interference from unwanted compounds.

3 Results

Although the objective of the cooperative target is to be interrogated by GPR, frequency domain characterization using a VNA of the sensor response over time allows for recovering the echo delay using the inverse Fourier transform [13-14]. While embedded commercial GPR are commonly designed around a pulsed mode stroboscopic interrogation thanks to the low power consumption of the avalanche transistor emitter and audio frequency analog to digital converter, using a VNA allows for better control of the spectrum occupation of the transmitted signal, especially under laboratory experimental conditions [15]. The phase of the inverse Fourier transform of the reflection scattering coefficient S_{11} is representative of the fine measurement of the acoustic velocity and hence chemical interaction of the sensing layer with the surrounding molecules. Since the timestep after inverse Fourier transform is determined by the bandwidth which, when probing a SAW transducer, is determined by the electromechanical coupling coefficient, all VNA parameters are set from the SAW sensor characteristics:
* center frequency and bandwidth are determined by the IDT period selected by the transducer design and electromechanical coupling coefficient respectively
* the number of samples is determined by the longest delay after inverse Fourier transform.
As an example of numerical application, a SAW sensor with center frequency 100 MHz and electromechanical coupling coefficient of 7% will require a bandwidth of 7 MHz. If for safety a 20 MHz bandwidth is selected, then the timestep after inverse Fourier transform is the inverse of the bandwidth or 50 ns, and the number of samples allowing to detect a backscattered echo at 5 µs delay at maximum is only 100

samples (Fig. 3). The intermediate frequency bandwidth of the VNA determines the inverse of the duration for each samples acquisition and hence the total sampling duration. A successful embedded, low power implementation of the VNA measurement is the NanoVNA which meets all the requirements of the presented application: such an instrument was used on the field for on-site measurements [15].

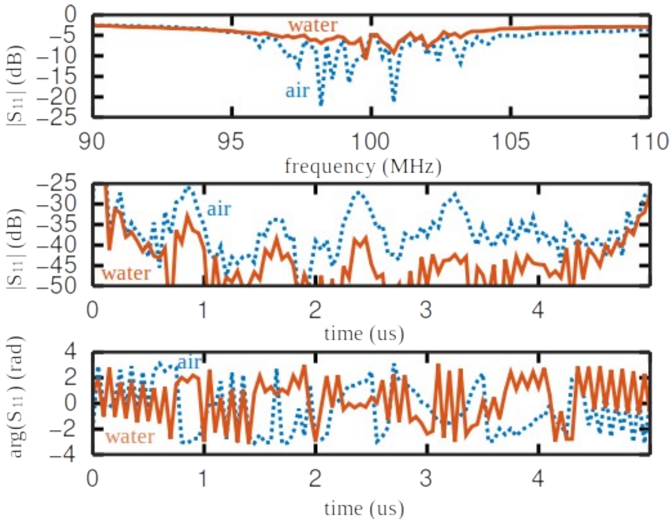

Fig. 3 Frequency domain (top) and time domain (middle and bottom) of a LTO SAW characterized using a NanoVNA vector network analyzer. The dotted line is the measurement in air and the solid line in water with no microfluidic packaging: Notice how the echo magnitude in the time domain (middle chart) only drops by 6 dB when inserting the sensor in water. The phase (bottom) is only defined (flat line) when the returned power is significant.

After demonstrating the remote measurement principle and designing the sensor, all individual components are integrated to achieve complete detection of sub-surface chemical compounds in a liquid phase. In this particular example, we focus on detecting toluene as a pollutant in the water table [17]. Polyepichlorohydrin (PECH) is known to form thin films that can reversibly change mechanical properties when exposed to organic solvents [18]. We deposited 600-nanometer-thick films using spin-coating onto an LTO substrate, which confines the shear acoustic wave in a Love mode, enhancing gravimetric sensitivity by increasing the acoustic energy density near the surface.

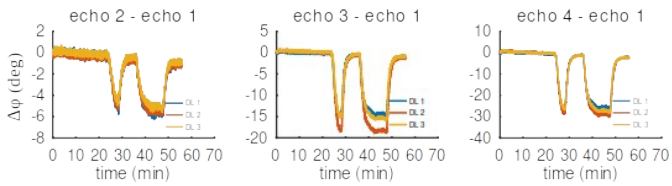

Fig. 4 Time evolution of a 600-nm thick polyepichlorhydrine (PECH) coated lithium tantalate sensor exposed twice to toluene: notice how reversible is the reaction when returning to pure water at time 25 and 50 minutes, and how the longer echo delay (from left to right) magnifies the impact of the toluene interaction with PECH.

Fig. 4 displays the phase of the reflection coefficient as a function of reaction time, with toluene injected around dates 25 and 35 after a stable baseline was reached. The impact of toluene is observed as a decrease in the phase difference of the reference echo 1 with the other four echoes. The response increases with increasing delay since the phase is the operating frequency times the time of flight so the phase variation is magnified with longer time of flight.

4 Conclusion

We illustrate the design considerations for sub-surface cooperative targets probed from the surface using Ground Penetrating RADAR, without the need for any local energy source. In addition to measuring physical quantities such as temperature and stress, the use of lithium tantalate as piezoelectric substrate for converting the incoming electromagnetic wave to a surface acoustic wave allows for detecting chemical compounds in water. Coating the sensor with dedicated polymer thin films enhances the selectivity of the reaction with a specific pollutant during the interaction of the Love-mode acoustic wave with the surrounding media, providing high sensitivity due to the acoustic energy confinement near the surface.

5 Acknowledgments

This work is supported by the French National Agency through the project UNDERGROUND (ANR-17-CE24-0037) and by the TotalEnergies company. We also acknowledge partial support of the French RENATECH network and its FEMTO-ST technological facility. We thank N. Nief, G. Pucheu, B. Segues, S. Dehez, and J.-R. Ordonez-Varela (TOTALenergies) for fruitful discussions.

6 References

[1] Daniels, J.D.: ‘Ground Penetrating Radar – 2nd Edition’ (The Institution of Electrical Engineers 2004)

[2] Bogorodsky V.V., Bentley C.R.: ‘Radioglaciology’ (D. Reidel Publishing Company 1985)

[3] Morgan, D.: ‘Surface acoustic wave filters: With applications to electronic communications and signal processing’ *(*Academic Press. 2010)

[4] Hashimoto, K. Y.: ‘Surface acoustic wave devices in telecommunications’ (Springer-Verlag 2000)

Journal articles

[5] Rabus D., Friedt J.-M., Arapan L., et al.: ‘Sub-surface H_2S detection by a Surface Acoustic Wave passive wireless

sensor interrogated with a ground penetrating radar'. ACS Sensors, 2020, 5(4), pp.1075–1081

[6] Paquit M., Arapan L., Feng W., et al.: 'Long range passive RADAR interrogation of subsurface acoustic passive wireless sensors using terrestrial television signals'. IEEE Sensors, 2020, 20 (13), pp. 7156–7160

[7] Friedt J.-M.: 'Passive cooperative targets for subsurface physical and chemical measurements: a systems perspective'. IEEE Geoscience and Remote Sensing Letters, 2017, 14(6), pp.821-825

[8] Friedt J.-M., Hugeat A., Lamare S., et al.: `Subsurface wireless chemical sensing strategy compatible with Ground Penetrating RADAR`. Proc. IWAGPR, Edinburgh, Scotland, 2017, pp.1–6

[9] Friedt J.-M., Chérioux F., Lamare S., et al.: 'Acoustic wave H_2S sensor comprising a polymer film comprising carboxylate functional groups and lead or zinc cations and manufacturing process'. USPTO 11185887, November 2021

[10] Shi, R., Zhang, C., Qin, P., Hao, T., Han, T., Chen, B., & Dai, Y.: 'SAW Tags With Enhanced Penetration Depth for Buried Assets Identification'. IEEE Transactions on Industrial Electronics, 2020, 68(9), pp.8863-8873.

[11] Rabus D., Arapan L., Tanguy P., et al.: 'Sampling frequency fluctuations of the Sensors & Software SPIDAR GPR when probing passive surface acoustic wave delay lines for pollution sensing', IEEE Geoscience and Remote Sensing Letters, 2021, 19, pp.3503005

[12] Minary F., Rabus D., Martin G., et al.: 'Note: a dual-chip stroboscopic pulsed RADAR for probing passive sensors'. Review of Scientific Instruments, 2016, 87, p.096104

[13] Seyfried, D., Schubert, K., Schoebel, J.: 'Investigations on the sensitivity of a stepped-frequency radar utilizing a vector network analyzer for Ground Penetrating Radar'. Journal of Applied Geophysics, 2014 111, pp.234-241

[14] Klotzsche, A., Jonard, F., Looms, et al.: 'Measuring soil water content with ground penetrating radar: A decade of progress'. Vadose Zone Journal, 2018, 17(1), pp.1-9

[15] Lambot, S., Weihermüller, L., Huisman, et al.: 'Analysis of air-launched ground-penetrating radar techniques to measure the soil surface water content'. Water resources research, 2006, 42(11).

[16] Very Tiny Palmtop Vector Network Analyzer, https://github.com/ttrftech/NanoVNA, accessed 10 September 2023

[17] Amati, D., Arn, D., Blom, N., Ehrat, M., Saunois, J., & Widmer, H. M.: 'Sensitivity and selectivity of surface acoustic wave sensors for organic solvent vapour detection'. Sensors and Actuators B: Chemical, 1992, 7(1-3), pp.587-591

[18] Bender F., Josse F., Mohler R.E., et al.: 'Design of SH-Surface Acoustic Wave Sensors for Detection of ppb Concentrations of BTEX in Water', Proc. Joint IEEE UFFC, EFTF and PFM Symposium, 2013