

A Multi-Surface Acoustic Wave Sensor platform for the Detection and Identification of Toxic Gases

Mariem Ben Hadj Abdallah, Christine Mer, Jean-Philippe Poli, Franck Badets, Edwin Friedmann, Venceslass Rat, Thierry Laroche, Samuel Saada

To cite this version:

Mariem Ben Hadj Abdallah, Christine Mer, Jean-Philippe Poli, Franck Badets, Edwin Friedmann, et al.. A Multi-Surface Acoustic Wave Sensor platform for the Detection and Identification of Toxic Gases. 2023 IEEE SENSORS, Oct 2023, Vienna (AUSTRIA), Austria. pp.1–4, 10.1109/SEN- $\rm{SORS}56945.2023.10324973$. $\rm{cea}\text{-}04487892$

HAL Id: cea-04487892 <https://cea.hal.science/cea-04487892v1>

Submitted on 4 Mar 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

A Multi-Surface Acoustic Wave Sensor platform for the Detection and Identification of Toxic Gases

Mariem Slimani*, Christine Mer-Calfati*, Jean-Philippe Poli*, Franck Badets[†],

Edwin Friedman[∗] , Venceslass Rat† , Samuel Saada[∗] and Thierry Laroche‡

[∗]Universite Paris-Saclay, CEA, List, F-91120, Palaiseau, France, {name.surname}@cea.fr.

†University Grenoble Alpes, CEA, LETI, F-38054 Grenoble, France, {name.surname}@cea.fr.

[‡]SOITEC company, F-25000 Besançon, France, {name.surname}@soitec.fr.

Abstract—In this paper, we present a platform based on a modular approach coupled with powerful algorithms for accurate detection and identification of chemical compounds. The system relies on multi-SAW (surface Acoustic Wave) sensors that are functionalized differently, resulting in multi-responses that collectively constitute the fingerprint of the chemical compound. A prototype has been developed and the overall system, including the design of SAW module, the acquisition system, learning algorithms and online recognition of various compounds, has been tested and validated. The results showed a reliable and accurate system with a perfect score of 100% recognition of DMMP.

Index Terms—Saw sensors, chemical sensors, toxic gases detection, recognition algorithms.

I. INTRODUCTION

Detecting and identifying toxic gases is a critical concern for a wide range of applications including public safety, environmental monitoring, and industrial processes. Surface Acoustic Wave (SAW) devices have emerged as a promising technology for toxic gas detection due to their compact size, cost-effectiveness, high sensitivity, and rapid response times [1] [2]. The sensitivity of a SAW sensor refers to its ability to detect and quantify specific molecules even at low concentrations. A proper design of the SAW device including operating frequency selection, Interdigital Transducers (IDTs) configuration, quality factor optimization can further enhance its sensitivity and its selectivity [3]. The selectivity is the second key factor characterizing the SAW sensor's performance refering to its ability to discriminate the target analyte from potential interferents or cross-reactive substances. It relies on the selection of the sensing layer that can selectively interacts with the target analyte. Mass loading effects can compromise the selectivity of SAW sensors by introducing interference from non-target molecules. This is particularly significant in scenarios involving complex gas mixtures or environments where multiple gases coexist [4]. To overcome this limitation, several strategies have been proposed in the literature. One approach involves designing an optimized sensing layer using materials that minimize non-specific adsorption or exhibit higher affinity for the target analyte [5] [6]. Another approach relies on what is called "e-nose", where an array of sensors with different chemical affinities are used to generate a unique chemical signature of the gas identified by appropriate signal processing algorithms and pattern recognition techniques [7].

In this study, we introduce a muti-SAW sensor platform that offers both high selectivity and sensitivity. Our platform is built upon a modular design concept, combining four distinct modules, each featuring unique chemical functionalizations. Within each module, multiple SAW resonators operating at a high frequency of 427 MHz are employed. An FPGA-based acquisition system has been implemented enabling real-time data acquisition and transmission through UART interface to the user. The acquired data is subjected to multi-parametric analysis techniques and machine learning algorithms for accurate recognition and classification of the gas nature. In this paper, we describe a bottom-up presentation of the platform.

II. DESIGN OF MULTI-SAW MODULE

A. SAW device

A SAW device is composed of a piezoelectric material on which two interdigitated transducers (IDTs) are deposited at opposite ends of a cavity covered with a sensing layer. The IDTs allow the conversion of electrical signal into acoustic wave that propagates along the surface of the piezoelectric substrate. The cavity is designed to be resonant at a specific frequency. When gas molecules interact with the sensing layer, the propagation of the acoustic wave is disturbed inducing a shift in the frequency of the measured electrical signal.

The sensing layer of a SAW device is a key element that influences its selectivity and sensitivity. There have been rapid developments for suitable sensing materials to improve sensing performance of SAW devices [8] [9]. In this paper, we used a new sensing layer, proposed in [10], involving the use of diamond nanoparticles deposited on the piezoelectric substrate. This approach offers a great versatility in terms of functionalization due to the diversity of carbon chemistry, resulting in a diamond layer that can be easily modified to suit specific detection requirements.

B. Multi-SAW module

A multi-SAW module in quartz was fabricated. It consists of 8 aligned measurement SAWs, with 4 on the left and 4 on the right, as well as two SAWs located at the top of the module, as shown in Fig. 1(a). These laters are non-functionalized SAWs and serve as reference oscillators, while the remaining eight SAWs are functionalized, acting as gas sensors. The sensing and reference SAWs are designed to exhibit a resonance

Fig. 1. (a) Multi-SAW module, (b) resonance frequency.

Fig. 3. Saw oscillator architecture.

frequency offset of approximately 1.5 MHz. The resonance frequency is determined by considering the maximum value of the magnitude of parameter S_{12} , as depicted in Fig. 1(b). The quality factor (Q-factor) is determined at resonance by considering the slope of the phase of parameter S_{12} at that point. The designed modules have a high quality factor around 10,000. Four modules were functionalized through a process involving the deposition of a thin film of diamond nanoparticles (approximately 20 nm) using deep coating [10]. Subsequently, they were plasma-fixed through Microwave Chemical Vapor Deposition (MCVD) and further functionalized using various methods, including immersion and plasma treatments with three different surface terminations: -OH, COOH, and -CH3.

III. MULTI-SENSING PLATFORM

The developed multi-sensing platform is shown in Fig. 2. It consists of a mainboard capable of accommodating 4 daughter boards. Each daughter board is designed to integrate ten oscillators each associated to one of the ten SAW devices on the multi-SAW module shown in paragraph II-B. The system measures the frequency difference between the reference and the sensing oscillators.

Fig. 2. Multi-sensing platform.

Note that within the same module, only one sensing SAW device and one reference SAW device are activated simultaneously. An FPGA-based acquisition system is implemented to

control the oscillations of the SAW devices and enable simultaneous measurement of the four signals from the four daughter boards. Different functionalizations were incorporated on the multi-SAW modules of each daughter board to obtain distinct responses (i.e., different frequency shifts) to a specific gas. The measured responses form a unique fingerprint of the target gas, which can be leveraged by AI algorithms for accurate gas recognition. In the following subsections, we provide a detailed description of the key parts of the developed multisensing platform including the design of the oscillator, the implementation of the acquisition system and the software framework.

A. SAW oscillator

Usually, resonator oscillators are implemented using onetransistor topology like Colpitts, Pierce or Hartley oscillators. Nevertheless, the rather high motional resistance and the rather high parasitics capacitance of the SAW resonator prevent the use of this kind of architectures. Instead, a classical feedback oscillator has been chosen where the SAW resonator is placed in series with an amplifier and a phase shifter circuit as depicted in Fig. 3. The amplifier has to provide enough gain to counteract the worst case 23 dB loss of the SAW resonator and the attenuation of the phase shifter. In order to get sufficient headroom a 40 dB gain amplifier has been designed using two Infineon BGA614 bipolar amplifiers in series. This amplifier brings a 20 dB fixed gain in a tiny package similar to discrete bipolar transistor package. This helps in compacting the oscillator design and minimizing both inductive and capacitive parasitics of the oscillator wires. The phase shifter is basically a LC high pass filter whose components values are set in order to bring the necessary phase shift to satisfy Barkhausen criteria, following a similar methodology as described in [11]. A daughter board has been designed to integrate ten oscillators, connected to each SAW device, generating an output signal at the desired specific frequency and ensuring sustained oscillation within the SAW device. As mentioned earlier, only one sensing oscillator and one reference oscillator are activated at a time. The "sel" signals are generated by the FPGA to control the activation of each oscillator. The difference of frequencies between the sensing oscillator and the reference one is obtained using a mixer and a low pass filter. The resulting signal pass then through a comparator to obtain a signal with CMOS compliant levels allowing seamless utilization by the FPGA.

B. Implementation of acquisition system

The acquisition system was implemented using a commercial board equipped with a Xilinx Zynq FPGA, which combines an Arm processor and an FPGA. This architecture enables parallel communication with all daughter boards: the control signals allow simultaneous activation of the corresponding SAW sensors on the four daughter boards and the four square wave signals, generated from the comparator output on each daughter board, are simultaneously sent back to the FPGA where reciprocal counters were implemented to accurately measure their frequencies. The measured signals are then transmitted via a UART connection upon user demand. Additionally, the implemented architecture provides access to valuable information stored in a dedicated EEPROM memory on each individual daughter board. This information could enhance the performance and effectiveness of recognition algorithms.

Note that the sensing oscillators within the same board are sequentially activated one after another. Moreover, the oscillators demonstrate a latency period for startup and shutdown. These time delays must be carefully considered, both for accurate frequency calculation and for the proper activation of the SAW devices. Firstly, the frequency measurements should be performed during a stable oscillation period to ensure reliable results. Secondly, it is essential to ensure that the SAW oscillators are not simultaneously active since they share the same output. Therefore, it is necessary to wait for one oscillator to completely shut down before activating the next one.

C. Software

We divide the software into two parts. On the one hand, the embedded software is in charge of the communication with a client, regarding a dedicated protocol. This communication protocol allows operations such as reading the array of sensors, configuring the sensors, or asking for information about the cards plugged onto the mother board.

On the other hand, the client software has been thought as a wizard. First, the user is invited to collect data into a database and to label them. The data is then the input of the next part, where the user is able to chose different preprocessing, different features to extract and different machine learning models to train. The training is achieved regarding a validation technique, among cross-validation, leave-one-out validation, etc. It is also possible to search for the optimal hyperparameters. At that time, only a grid search can be used. Once a workflow is constituted, it can be compared with other workflows or tested on a validation set. The client software then displays a window with all the main metrics for model

Fig. 4. Confusion matrix of the experiments.

performances. Finally, a workflow can be loaded and exploited in real-time to recognize gases.

In [12], the authors describe a usual worflow as consisting of preprocessing steps, feature extraction, dimension reduction and a classifier (machine learning). They also present an extensive benchmark of the most useful algorithms in each category. Since then, new features have been introduced specifically for SAW array of sensors [13] and explainable artificial intelligence has been applied to such sensors [14].

IV. RESULTS AND DISCUSSIONS

For the qualification tests of the prototype, a set of four compounds was utilized in liquid form and securely placed in pillboxes. A pump upstream of the system draws the vapors routed to the sensor modules. The tested gases included DMMP (a sarin simulant), as well as water, ethanol and shower gel, strategically chosen as interferents to assess the system's selectivity. The results are given in Fig. 4 as a confusion matrix over 80 acquisitions with a fuzzy classifier as suggested in [14].

V. CONCLUSIONS

This scientific paper presents the development and qualification of a multiplexed surface acoustic wave (SAW) sensor system for gas detection. The system incorporates functionalized modules with multiple SAW sensors, allowing for enhanced sensitivity and selectivity. The prototype was tested using various compounds, including a sarin simulant (DMMP), as well as interferents such as water, ethanol, and shower gel. The collected sensor responses were processed using specialized software tools for feature extraction, dimensionality reduction, and model development. The qualification tests demonstrated the system's robustness, achieving 100% recognition rate for DMMP even in the presence of interferents. Further analysis using fuzzy logic will provide additional insights into sensor drift and optimization of surface functionalization for improved gas detection performance. Moreover, ongoing research

is exploring alternative surface functionalization methods such as polymers or chromogenic materials, allowing the exploration of novel materials and their potential applications in various domains.

REFERENCES

- [1] L. Panes-Ruiz, L. Riemenschneider, M. Al Chawa, M. Löffler, B. Rellinghaus, R. Tetzlaff, V. Bezugly, B. Ibarlucea, and G. Cuniberti, "Selective and self-validating breath-level detection of hydrogen sulfide in humid air by gold nanoparticle-functionalized nanotube arrays," Nano Reasearch Journal, vol. 15, pp. 2512–2521, 2022.
- [2] G. Singh, S. Kim, and K. Lee, "Development of a highly sensitive and portable particulate matter SAW sensor and interface electronics," Sensors And Actuators A: Physical, vol. 343, pp. 113641, 2022.
- [3] D. Mandal, and S. Banerhee, "Surface acoustic wave (SAW) sensors: Physics, materials, and applications," Sensors Journal, vol. 22, pp. 820, 2022.
- [4] J. Devkota, P. Ohodnicki, and D. Greve, "AW sensors for chemical vapors and gases," Sensors Journal, vol. 17, pp. 801, 2017.
- [5] A. Afzal, N. Iqbal, A. Mujahid, and R. Schirhagl, "Advanced vapor recognition materials for selective and fast responsive surface acoustic wave sensors: A review," Analytica Chimica Acta, vol. 787, pp. 36–49, 2013.
- [6] H. Yuan, N. Li, W. Fan, H. Cai, and D. Zhao, "Metal-Organic Framework Based Gas Sensors," Advanced Science, vol. 9, pp. 2104374, 2022.
- [7] J. Gardner, and P. Bartlett, "A brief history of electronic noses," Sensors And Actuators B: Chemical, vol. 18, pp. 210–211, 1994.
- [8] A. Ayankojo, A. Tretjakov, J. Reut, R. Boroznjak, A. Opik, J. Rappich, A. Furchner, K. Hinrichs, and V. Syritski, "Molecularly imprinted polymer integrated with a surface acoustic wave technique for detection of sulfamethizole," Analytical Chemistry, vol. 88, pp. 1476-1484, 2016.
- [9] J. Rodriguez-Madrid, G. Iriarte, D. Araujo, M. Villar, O. Williams, W. Muller-Sebert, et F. Calle, "Optimization of AlN thin layers on diamond substrates for high frequency SAW resonators," Materials Letters, vol. 66, pp. 339-342, 2012.
- [10] E. Chevallier, E. Scorsone, and P. Bergonzo, "New sensitive coating based on modified diamond nanoparticles for chemical SAW sensors," Sensors And Actuators B: Chemical, vol. 154, pp. 238-244, 2011.
- [11] R. Schmitt, J. Allen, and R. Wright, "Rapid design of SAW oscillator electronics for sensor applications," Sensors And Actuators B: Chemical, vol. 76, pp. 80-85, 2001.
- [12] O. Hotel, J. Poli, C. Mer-Calfati, E. Scorsone, and S. Saada, "A review of algorithms for SAW sensors e-nose based volatile compound identification," Sensors And Actuators B: Chemical, vol. 255, pp. 2472- 2482, 2018.
- [13] O. Hotel, J. Poli, C. Mer-Calfati, E. Scorsone, and S. Saada, "SAW sensor's frequency shift characterization for odor recognition and concentration estimation," IEEE Sensors Journal, vol. 17, no. 21, pp. 7011- 7018, 2017.
- [14] E. Friedmann, J.P. Poli, O. Hotel, and C. Mer-Calfati, "Fuzzy Classifiers for Chemical Compound Recognition from SAW Sensors Signals," in IEEE 32nd International Conference on Tools with Artificial Intelligence (ICTAI), pp. 917-922, 2020.