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INNOVATIVE SURFACE ACOUSTIC WAVE DEVICES: A SOLUTION FOR REAL TIME MONITORING AND SELF-CLEANING CASCADE IMPACTOR

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Abstract: This paper presents a comparative study of three types of surface acoustic waves sensors (SAW) used for particulate matter (PM) detection.

These sensors are placed in a cascade impactor as impaction plates. Monitoring their phase variation allows us to know the quantity of fine particles present in the environment with high accuracy. Until now, the sensors used in our prototype are built on quartz substrate and present a good sensitivity to fine particles. One major concern in our application is the fouling of the sensor's surface with particles upon long periods of exposure. This shaped our drive to develop a self-cleaning sensor relying on other substrates with a stronger electromechanical coupling coefficient (K^2). Hence, the aim of this study is to demonstrate the possibility of using strongly coupled piezoelectric substrates for an accurate PM detection as well as a self-cleaning process.

Keywords: Cascade impactor, SAW sensors, Lithium Niobate, Quartz, Particulate matter, PM10, PM2.5.

1. INTRODUCTION

Nowadays, outdoor pollution is rising in all areas causing around 7 million premature deaths worldwide yearly, according to the World Health Organization [1], majorly due to PM penetrating into human lungs. The toxicity of particles is directly linked to their size. The smaller are the particles the deeper they penetrate in the human lung. Exposure to PM10 and PM2.5 has a proven connection with death due to cardiovascular and respiratory diseases such as asthma, chronic obstructive pulmonary disease (COPD), pulmonary fibrosis, pneumonia, and lung cancer [2]–[4]. For these reasons, there is a great need for continuous air monitoring to ensure the respect of health-based standards. SAW sensors are widely used in diverse sensing applications such as biosensors [5], gas sensors[6], temperature sensors [7], humidity sensors [8] and light detectors [9], [10]. SAW technology has been also attracting attention for particle measurement [11], [12]. Among the most used systems for PM measurements, we name the cascade impactor. Despite its good performance, this equipment does not provide real-time measurements. The impaction plates should be weighed before and after sampling, which is time consuming. For that reason, our team attempted to equip the impaction plates with SAW sensors [13], [14] to ensure real-time measurements. However, a problem regarding prolonged exposures to particles persisted and caused progressive fouling of the sensor's surface, from which the need of an integrated regeneration system that does not require any dismantling of the impactor.

Melvin Paquit *et al.* [15] have shown in a previous study that the use of Rayleigh waves with a delay line based on 128° Y-cut $LiNbO_3$ substrate allow, due to their elliptical motion, to significantly displace particles from the substrate's surface. The displacement is achieved by applying a radio-frequency signal with a power level higher than 30 dBm (1 W). The particles tested were issued from a candle smoke (fig 1) with an average diameter smaller than 2.5 µm. 30 seconds was a sufficient period to move the particles off the surface under 30 dBm power. Particles of silicon carbide (SiC) smaller than 5 µm have also been tested and successfully removed from the acoustic track after 20 seconds at 31 dBm (1.25 W) (fig 2).

In this paper, we investigate the possibility of using high electromechanical coupling factor substrates to build sensors. Accordingly, two SAW sensors that have been selected for this study; Rayleigh wave based on 128° Y-cut $LiNbO_3$ and PSAW wave based on 41° Y-cut $LiNbO_3$. The sensitivity of these sensors is compared with that of a Love wave sensor based on the AT-quartz substrate that is currently used in our system. The gravimetric sensitivity of these sensors was studied numerically and the sensitivities of the fabricated sensors were estimated experimentally.

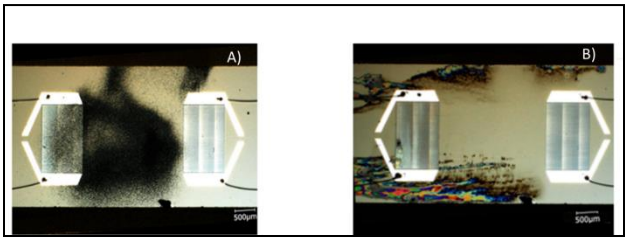

Figure 1: View of SAW delay lines covered with particles smaller than 2.5 µm from a burning candle (A) before and (B) after high-power RF cleaning

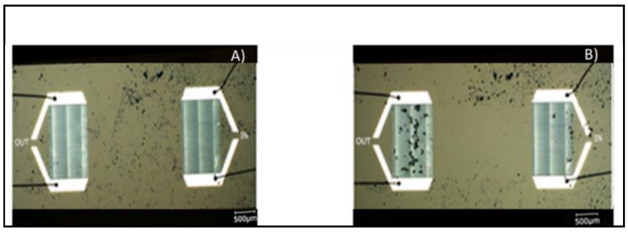

Figure 2: View of SAW delay lines covered with particles smaller than 5 µm from SiC (A) before and (B) after high-power RF cleaning.

2. MATERIALS AND METHODS

2.1. Cascade impactor

Our system is a customized cascade impactor [13] of three stages working at 3 Lpm flow rate in which two impaction plates have been replaced by SAW sensors (fig 3). The first stage is equipped with an impaction plate for the collection of coarse particles with aerodynamic diameters higher than 10 µm. These particles are not measured since the toxicity of suspended particles is essentially due to particles with a diameter less than 10 µm. The last two stages are equipped with SAW sensors as impaction plates coupled with a monitor and aim at filtering particles having a diameter less than 10 µm and 2.5 µm in the second and third stage, respectively.

2.2. SAW sensors

SAW delay lines, used in this study, are based on piezoelectric substrates to generate waves at the surface. The quantification of particles is based on gravimetric sensitivity. To overcome the perturbations due to outer parameters such as temperature, pressure and humidity, a differential configuration relying on two delay lines was used.

Each sensor is composed of two delay lines as shown in the zoomed part of figure 4. By positioning the holes of the impactor aligned with the sensing area of a specific delay line (the measurement line), the particles are collected only on this latter.

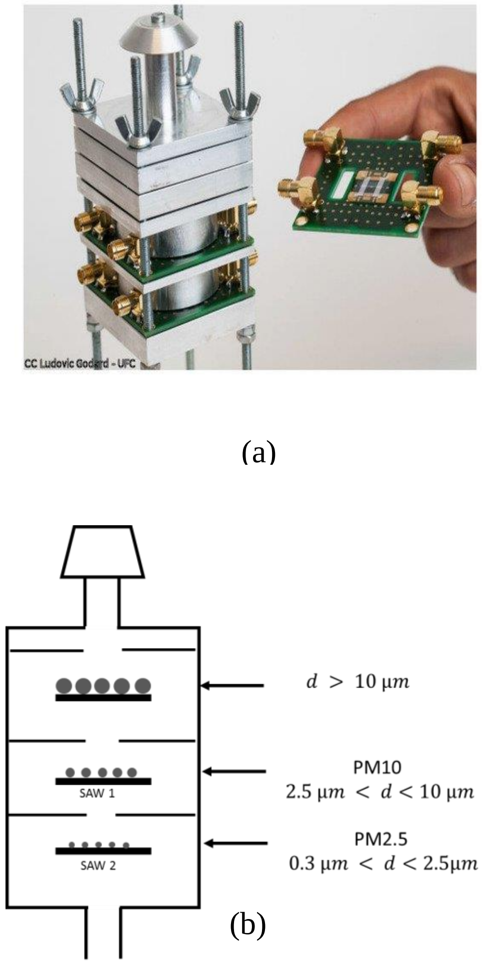

Figure 3: (a) A photograph and (b) a schematic figure of our 3 Lpm cascade impactor.

The second one (the reference line) remains free of particles and is used as a reference. By subtracting the phase response of the reference line from that of the measurement line, we can accurately obtain the variation induced by the gravimetric effect as a result of PM deposition.

Fabrication process

The SAW sensors are composed of two interdigitated Transducers (IDTs). The first one is used as input delay line and allows the generation of the acoustic wave while the second IDT enables the detection of the acoustic wave. The IDTs consist of double finger pairs of Aluminium with thickness 300 nm made by a lift off process. For sensors based on AT-cut Quartz substrates, a silica guiding layer is necessary and was deposited on top of the IDTs using a PECVD process to allow the propagation of Love-mode acoustic wave at the surface of the device. To link the two ports of the delay line to the probing electronics, the electrical connection has to be opened using Deep Reactive Ion Etching (DRIE). The working frequency are 125 MHz, 100 MHz and 100 MHz, respectively, for sensors based on AT-cut quartz, 128° Y-cut $LiNbO_3$ and 41° Y-cut $LiNbO_3$.

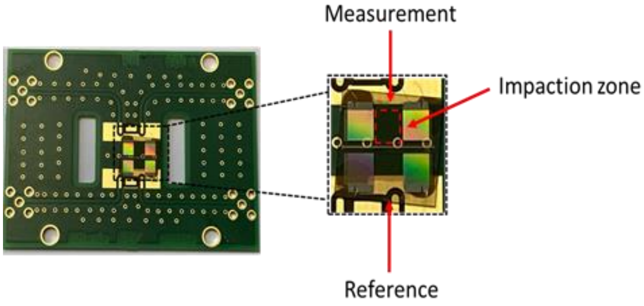

Figure 4: A photograph of SAW sensors mounted on a PCB.

2.3. Particles generation and experimental process

In order to experimentally determine the sensitivity of the three sensors, a test bench has been developed (fig. 5). It enables us to generate particles with controlled concentrations. A particle generator AGK 2000 purchased from Palas® was used to produce fine particles from SiC solutions for PM10 and NaCl solutions for PM2.5 by nebulization. The size distribution and concentration of the generated particles depend on the concentration of solution and the air pressure at the generator input. Therefore, the solutions were carefully calibrated and a digital pressure controller from Bronkhorst was coupled with the generator. An optical particle counter (OPC) FIDAS 100 ® was used as a reference system to measure the concentration inside the test chamber in order to correlate it with the measurements obtained from SAW sensors. For particle sensing, the baseline phase of the SAW delay lines was stabilized under typical working conditions ($T \approx 25°, RH \approx 30\%$). Then, concentration in the chamber was stabilized around the target concentration. It was noticed that the particles generator produces the concentrations with a fluctuation of ±20 µg⁄m^3.

3. SENSITIVITY CHARACTERISATION

3.1. Theoretical approach

To predict the gravimetric sensitivity of the SAW sensors in question, a specific software developed in our team [16] was used. It calculates the effective permittivity of a stratified medium from which we extract the propagation velocity of the existing modes as well as the coupling factor K^2. A gold layer was used to mimic the mass of PM at the sensor's surface. Finally, the gravimetric sensitivity is calculated using the Sauerbrey approximation: $S = \frac{\Delta v}{v_0}.\frac{A}{\Delta m}$ with $\frac{\Delta v}{v_0}$ the relative shift in the speed of the wave, Δm the mass variation and A the active surface of the sensor. In our case, the mass variation can be considered as the product of the density, the surface considered in the model and the thickness of the gold layer Δe :

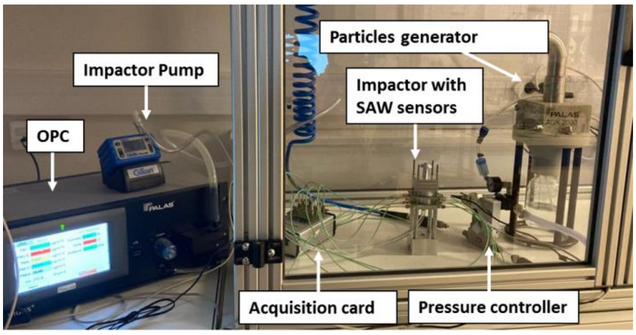

Figure 5: The Experimental test bench for particles generation.

$$\Delta m = \rho . \Delta V = \rho . \Delta e . A$$

From there, the gravimetric sensitivity can be expressed as:

$$S = \frac{\Delta v}{v_0} . \frac{1}{\rho . \Delta e}$$

To begin with, the simulation results were used to estimate the expected sensitivity. These results are shown in figure 6. Thus, the highest sensitivity (250 cm²/g) was obtained with the sensor based on a AT-Quartz, followed by that based on 128° Y-cut $LiNbO_3$ with a sensitivity of 172 cm²/g and finally by the one based on the 41° Y-cut $LiNbO_3$ with a sensitivity of 139 cm²/g. Accordingly, even if the sensors based on $LiNbO_3$ show lower sensitivity values with respect to that of the sensor based on quartz, $LiNbO_3$ substrate remains a strong alternative for measuring mass loading induced by PM in the application considered here.

In order to evaluate the possibility of self-cleaning out of these sensors, simulations were performed to predict the expected coupling coefficients as a function of the type of substrate used and its orientation. Thus, the obtained values of K^2 coefficients are 0.14 %, 5.5 % and 17 % for AT-quartz substrate, $LiNbO_3$ Y-cut at 128° and for $LiNbO_3$ Y-cut at 41°, respectively (Table 1). Considering the sensitivity and the electromechanical coefficient results, the use of 128° Y-cut $LiNbO_3$ substrate with Rayleigh wave seems to be promising for the development of a sensor capable of detecting fine particles and able to perform self-cleaning after high particle accumulations.

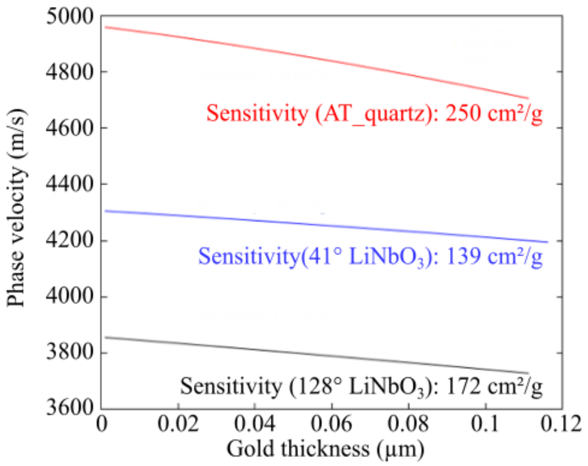

Figure 6: Simulated gravimetric sensitivity of AT-Quartz, 128° Y-cut LiNbO$_3$ and 41° Y-cut LiNbO$_3$.

Table 1: electromechanical coupling factors and gravimetric sensitivity of sensors used in this study.

Substrate	K² (%)	Gravimetric sensitivity (cm²/g)
Quartz-AT cut (Love wave)	0.14	250
128° Y-cut LiNbO$_3$ (Rayleigh wave)	5.5	172
41° Y-cut LiNbO$_3$ (PSAW wave)	17	139

-30%

- 44%

3.2. Experimental Approach

In order to validate the theoretical approach towards the ability of the 3 sensors to detect fine particles in a lightly polluted environment, a follow-up of their phase, during successive exposures to particles, was performed. In this section, we report on the sensor's sensitivities obtained experimentally. Figure 7 shows an example of the phase shift of sensor based on AT-cut Quartz during successive exposures to particles. For this experiment, only particles smaller than 2.5 µm were generated at a concentration of 150 µg/m^3. The total concentration of particles with a diameter <10 µm was about 170 µg/m^3, which indicates that only a concentration of 20 µg/m^3 corresponds to particles with a diameter between 2.5 and 10 µm. As this value represents the fluctuations of the bench, we can consider that the PM10 particles present are due to ambient particles in the chamber. It can be seen that the curve of the PM10 stage does not show any measurable phase shift unlike that of the PM2.5 stage which shows a clear phase shift at each sampling. These results highlight the size separation efficiency of our impactor. The sensor's response is then obtained by determining the derivative of the phase variation during the particles sampling. In order to increase the accuracy, it is worth mentioning that 4 identical measurements were performed. The reported value is thus estimated by averaging these 4 measurements.

Broadening our inspection to account for PM10, this same measurement protocol was applied to all three sensors at different particles concentrations and by injecting particles having different sizes and generated with NaCl and SiC solutions. Thus, the absolute value of the phase derivative as a function of the particle concentration measured by FIDAS® optical system is presented in Figure 8 (a) and (b). A linear fit has been applied on the obtained curve to determine the sensitivity of the sensor. The correlation coefficient denoted R² has been calculated to estimate the quality of the fit. The phase derivative-PM concentration couple showed strong correlation for all sensors with an R² between 0.8 and 0.9. From there, the sensitivity of the sensors has been determined. For Love wave-based sensors on AT-quartz cut substrates, the sensitivity was estimated to be 50±0.1 µ°/s/µg.m^3 for PM10 and 300± 0.08 µ°/s/µg.m^3 for PM2.5. The Rayleigh wave-based sensors based on 128° Y-cut LiNbO$_3$ showed a sensitivity of 8±0.03 µ°/s/µg.m^3 for PM10 and 100±0.02 µ°/s/µg.m^3 for PM2.5 The PSAW sensors based on 41° Y-cut LiNbO$_3$ showed the lowest sensitivities among the tested sensors and are estimated to be 7±0.08 µ°/s/µg.m^3 for PM10 and 60±0.3 µ°/s/µg.m^3 for PM2.5. (Table 2 and 3). The comparison between the sensitivity to PM 10 and PM2.5 remains possible even if the generated concentration values are different in the two cases since the tested range remains the same (between 40 and 200 µg/m^3). Comparing the experimental sensitivities to PM2.5 for the 3 sensors, we can estimate that there is a 68% decrease in the case of 128° Y-cut LiNbO$_3$ as well as an 80 % decrease with 41° Y-cut LiNbO$_3$ compared to that of AT-quartz. Theoretical estimations of sensitivity (fig.6) revealed an expected loss of 44% and 30% respectively delineating the evolution of the sensitivity for different substrates. Moreover, although the calculations were carried out based on equations considering the deposition of a thin layer of gold, an inhomogeneous deposit of fine particles on a limited surface of the sensitive zone of the sensor was achieved in our experiments. Nevertheless, these estimations were still able to predict the sensitivity leading to results agreeing with our predictions.

Regarding the PM10 and PM2.5 measurement, it is obvious that the measurement is definitely applicable and usable given the linearity of the response even if the sensitivity is weak. Even if they are less sensitive than those of Love wave-based sensors, the Rayleigh wave sensors based on 128° Y-cut $LiNbO_3$ are good candidates for PM measurement. We can notice that SAW sensors show different sensitivities toward PM10 and PM2.5. This can be due to the size and the different morphology of particles.

Particle size plays a crucial role in defining the sensor's response since most adhesion forces are linearly dependent on particle diameter [17].

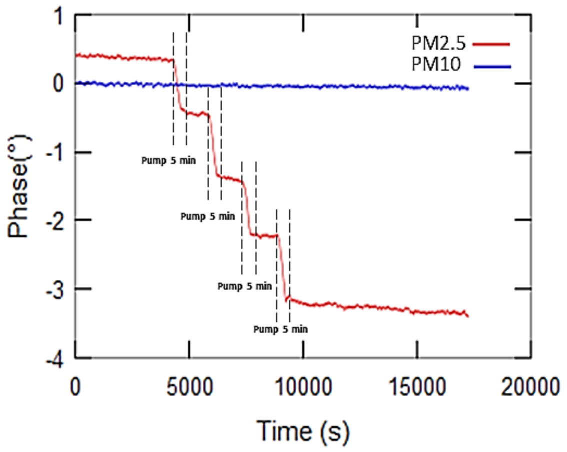

Figure 7: SAW sensor phase shift of PM2.5 stage (red) and PM10 (blue) during successive exposures to particles in the [0, 2.5 µm] range.

Table 2: Experimental sensitivity to PM2.5 of sensors used in this study.

Sensor	PM2.5 sensitivity (µ°.s^{-1}.µg^{-1}.m^{-3})
Quartz-AT cut (Love wave)	300 ± 0.08
128° Y-cut $LiNbO_3$ (Rayleigh wave)	100± 0.02
41° Y-cut $LiNbO_3$ (PSAW wave)	60± 0.3

-68%

- 80%

Table 3: Experimental sensitivity to PM10 of sensors used in this study.

Sensor	PM10 sensitivity (µ°.s^{-1}.µg^{-1}.m^{-3})
Quartz-AT cut (Love wave)	50 ± 0.1
128° Y-cut $LiNbO_3$ (Rayleigh wave)	8± 0.03
41° Y-cut $LiNbO_3$ (PSAW wave)	7±0.08

-84%

- 86%

Considering that smaller particles adhere more to the surface, the slowdown of the wave is more significant for PM2.5. The size-related sensitivity of SAW sensors was reported in a recent study [18].

In this latter, the higher sensitivity of SAW sensors toward PM2.5 can be explained by the fact that particles' coupling to the sensor's surface is dominated by the gravimetric effect for smaller particles and by the elastic effect for bigger particles.

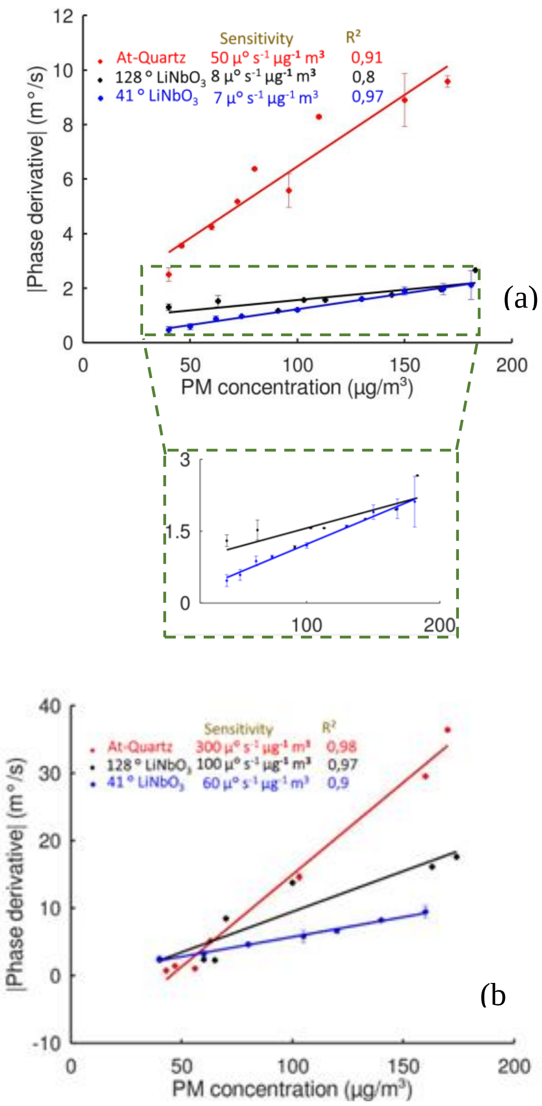

Figure 8: Experimental characterization of the sensors sensitivity a) PM10 and b) PM2.5. (PM concentration measured by Fidas 100®).

On the other hand, the known phenomenon of rebound effect in cascade impactors may also explain the lower sensitivity toward PM10. As outlined by Dahneke [19], a particle sticks to or rebounds from a substrate depending on the balance between the kinetic energy of the particle and other processes such as adhesion and the plastic deformation of the particle and/or the substrate.

4. CONCLUSION AND PERSPECTIVES

In this research, we have investigated the sensitivity of substrates having high electromechanical coupling factor allowing the displacement of particles and compared it to that of the AT-Quartz based SAW sensors used in our current system. The sensitivities of the selected devices were estimated against the presence of PM10 and PM2.5. In light of the simulated and experimental results, we demonstrated the potential of 128° Y-cut $LiNbO_3$ based SAW sensors to measure PM collected by means of a cascade impactor. Despite their lower sensitivity compared to AT-quartz based sensors, these sensors exhibit higher electromechanical coupling factor that are likely to allow the removal of accumulated particles from the surface. Further investigations will be carried out to develop self-cleaning SAW sensors based on 128° Y-cut $LiNbO_3$ substrates for cascade impactors. Furthermore, a study is being carried out to study the effect of a material with lubricating properties on the rebound of particles in order to enhance the quality of measurements for larger particles as well.

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