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Selective Detection of Hydrogen with Surface Acoustic Wave Devices Using Palladium Layer Properties

Meddy VANOTTI¹, Virginie BLONDEAU-PATISSIER¹, David RABUS¹,
Jean-Yves RAUCH², Sylvain BALLANDRAS¹

¹Time and Frequency Department, FEMTO-ST Institute, Besançon, France

²Optic Department, FEMTO-ST Institute, Besançon, France

meddy.vanotti@femto-st.fr

Abstract: For an increasing number of applications, hydrogen represents a solution of the future as it is the most common element in the Earth. However, due to its unstable properties in gas phase, a particular care must be dedicated to control possible gaseous leaks close to tanks and facilities using this resource. In this paper, surface acoustic wave sensors are proposed for detecting gaseous hydrogen in standard environmental conditions (atmospheric pressure and room temperature). The proposed Surface Acoustic Wave sensors consists in two Rayleigh-wave delay lines built on Quartz, one equipped with a palladium overlay and the other exhibiting a free path between the two interdigitated transducers. A specific gas test cell has been developed to test various sensor configurations submitted to hydrogen-composed atmospheres. A particular care was paid to avoid hydrogen leakage in the working environment and to perform the regeneration of the gas absorbing layer. The developed device allows for identifying different concentrations of hydrogen (in the 1-4% range) diluted in nitrogen and is also able to detect hydrogen in current atmosphere. Surface Acoustic Wave devices exploiting hydrogen absorption capabilities of palladium thin films have been here used to make the detection and the identification of hydrogen concentrations in the 1-4% range and the influence of outer parameters such as temperature and relative humidity variations on the sensor operation is also reported.

Keywords: Gas sensors, Hydrogen, Palladium layer, SAW device, Rayleigh waves.

1. Introduction

The raising shortages of fossil energy resources added to the increasing concern towards environmental issues have led to consider hydrogen as one of the most promising energy resource. This odorless and colorless gas being highly explosive over 4% concentration in air, the availability of a fast and accurate detection system close to storing facilities and equipping hydrogen-operated machines is mandatory for obvious security reasons. Such a system must exhibit a

significant selectivity as it must detect the presence of gaseous hydrogen in air with concentrations smaller than the above-mentioned critical limit at standard conditions (room temperature and atmospheric pressure) as well as in harsher environment (very low or significantly high temperature). Although, some solutions have been proposed [1] [2] [3] [4] [5] [6] [7], the current availability of such a detection system meeting modern specifications of hydrogen use and storage is still questionable. The main improvements for such sensors are their sensitivity, their selectivity and their reliability together with sensor size, cost

reduction, energetic needs and response time [8]. Many methods of detection of hydrogen and a comprehensive review can be found in the literature [9], providing a substantial material base to try and address the above challenges. Among the possibilities, SAW (Surface Acoustic Wave) sensors have been widely studied in the last decades because of their attractive capabilities [21] [26] [27]. Indeed, SAW devices exhibit high sensitivity to surface perturbation since most of the energy propagates in a region whose thickness is a few times the wavelength of the propagating acoustic wave. It is also a mature technology, SAW devices do exhibit limited size (less than 1 cm²) and they allow for wireless use [10]. Initial works were made by D'Amico et al. [11] using the properties of palladium layer to trap the targeted gas. Since this pioneer work, innovations concerning the selectivity and stability of sensitive layers versus external parameter have been proposed to improve hydrogen detection using SAW devices [12] [13] [14] [15] [16] [17] [18] [19]. In this paper, a SAW sensor is proposed for detecting gaseous hydrogen in standard environmental conditions (atmospheric pressure and room temperature). The proposed SAW sensor consists in two Rayleigh-wave delay lines built on quartz, one equipped with a palladium (Pd) overlay and the other exhibiting a free path between the two interdigitated transducers (IDTs) used to excite and detect the acoustic wave. These IDTs are built using aluminum electrodes, as this metal is of low density and acts as a limited acoustic reflector. An innovative aspect of the proposed sensing system consists in the open-loop strategy for phase changes monitoring [20]. Moreover, delay lines are monitored in parallel using a synchronous detection approach that provides high frequency measurement resolution and that permits a systematic characterization of the device before starting measurement. Along this approach, the impact of changes of intrinsic properties of the devices such as working frequency drift with aging can be minimized. These sensors have been tested in a specific gas cell developed to experiment with various sensor configurations submitted to hydrogen composed atmospheres. A particular care has been dedicated to avoid hydrogen leakage in the working environment and to promote the gas absorbing layer regeneration. The developed device allows for identifying different concentrations of hydrogen diluted in N₂ and is also able to detect H₂ in current atmosphere. The first section of the paper presents the SAW sensor as well as the exploited monitoring system. Experimental validation of H₂ detection then is reported, with a description of the chemical test bench and detection results for various H₂ concentrations. An analysis of the influence of H₂ adsorption on SAW propagation is proposed to provide routes for the proposed device optimization. In the same purpose, the last section of the article is devoted to characterizing the influence of external parameters such as temperature and relative humidity variations on the sensor operation, yielding conclusive discussions.

2. SAW sensor and associated monitoring system

Selective detection of hydrogen at room temperature and pressure have been achieved using SAW delay lines exploiting Rayleigh waves on AT-cut quartz, as the corresponding first order temperature coefficient of frequency (TCF) is close to zero, yielding frequency-temperature compensation for the above-mentioned operating conditions. The sensor structure correspond to a differential set-up in, which a sensitive track is achieved by depositing a Pd layer in between two IDTs whereas the reference track surface is left free to detect nonspecific gas/surface interaction. Along this approach, one can significantly increase the sensitivity of the device and its robustness to correlated perturbations (temperature, vibration, non-specific adsorption). The configuration of both generation and detection IDTs used for the sensor consist in 50 fingers pairs with a grating period of 10μm and path length of 5mm (the reactive surface). The wavelength is 40μm, yielding a frequency operation in the vicinity of 78MHz as the wave velocity approaches 3100m.s⁻¹. The Pd film was deposited by thermal evaporation on a single run and shaped by a lift-off technique. Its length along the propagation path was 3mm and its thickness equal to 300nm. The device configuration is shown on Fig. 1. Using a network analyzer, the transfer function of the device can be easily determined and hence the phase shift induced by gas absorption has been first monitored that way. However, the use of a dedicated electronics has been implemented and delivers similar information. That is represented on Fig. 2. This system includes two separate phase measurement schemes: a rough characterization stage using the AD8302 phase and magnitude detector, and a high sensitivity phase measurement using dedicated phase detectors and variable gain, low noise operational amplifiers as presented in another work [20].

By increasing the gain on the operational amplifier, one can gradually improve the phase measurement resolution while remaining at a constant operating point. However, this approach also reduces the phase range due to the limited voltage range of the analog to digital converter (ADC). While the AD8302 is tuned to generate a voltage within the ADC range as the phase rotates from +90° to -90°, increasing the gain of the high sensitivity stage yields measurement ranges

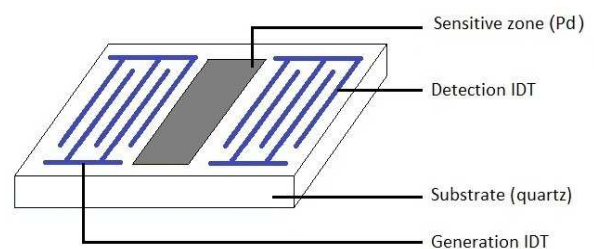


Fig. 1. Scheme of a SAW delay line using Rayleigh surface acoustic wave at 78MHz.

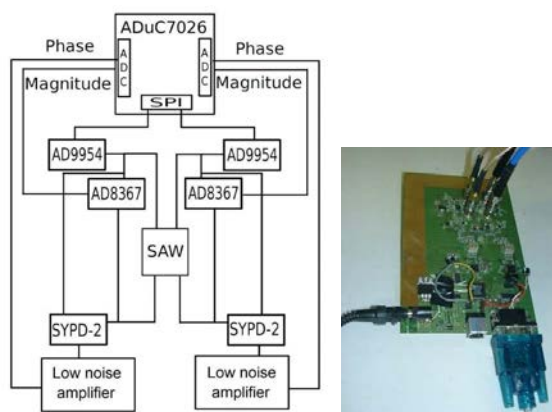


Fig. 2. Scheme and photograph of a SAW devices probing card.

reduced to a fraction of the angle range. Thus, a tracking scheme is implemented in which the phase to frequency (locally linear) relationship is identified, and whenever the phase reaches a condition close to the boundary of the operating range of the ADC, the RF source frequency is tuned so that the phase moves back to the middle of the ADC range. This implementation is similar to a digital oscillator, since the frequency tracks a phase condition, with the added advantage that a precise phase measurement is provided as long as the voltage remains within the operating range of the ADC. The sensitivity of the set-up allows for some tens millidegrees resolution and is easily transportable. The response of the bare device and the functionalized one are respectively measured. This configuration has been used so as to make a systematic characterization of each new device used for H₂ detection. Fig. 3. illustrates the way the phase shift measurement is achieved.

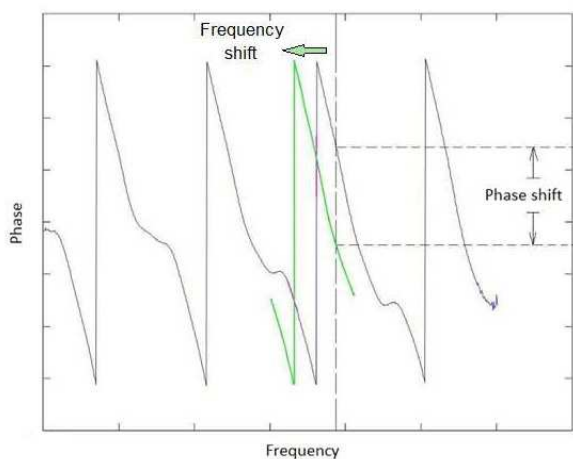


Fig. 3. Phase shift measurement principle using a dedicated instrumentation.

2.1 Detection of hydrogen at room temperature and atmospheric pressure

Hydrogen detection in the percent order has been achieved at room conditions. Figs. 4 and 5 present experimental results when using either nitrogen or air

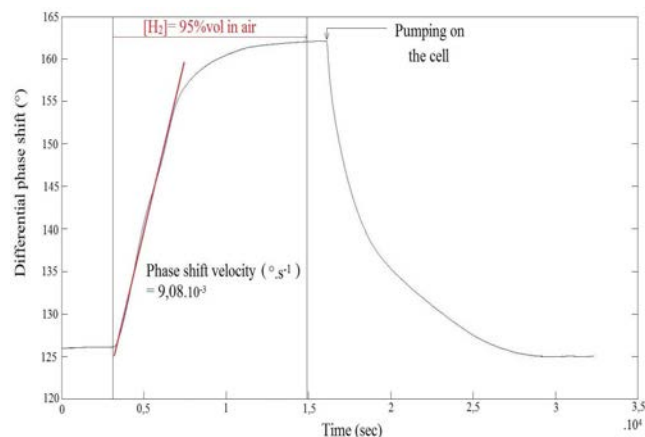


Fig. 4. Detection of hydrogen at high concentration in air.

as carrier gas. As it can be observed in Fig. 5., the detection of about 95%vol of hydrogen in air at 35% RH and 20°C can be achieved with a response delay of about 20 seconds considering that the determination of the H₂ concentration in the melting gas is derived from the phase shift velocity during the exposure and not from the steady state. It is notable that this value depends on a large part on the performance of the hydrogen generation setup. Therefore, the intrinsic response delay of the device is expected to be shorter. Fig. 4. shows that the device allows for identifying different concentrations of hydrogen diluted in a nitrogen flow of 100sccm.

3. Analysis of hydrogen absorption on Pd films and its influence on SAW propagation

Absorption of hydrogen into Pd layer induces modifications on the SAW propagation conditions. The effective physical characteristics of the wave then vary with the amount of absorbed H₂ (especially its phase velocity). It has been shown [21] that the two main parameters that influence the phase velocity change of elastic waves on quartz are mass- and elastic-loading as it can be seen on the equation below [21]. Since only devices using quartz substrates have been used here, changes in electrodes conductivity are not considered as a possible origin of the observed phase velocity drift and therefore were not taken in account in the proposed analysis. Electromechanical coupling of Rayleigh waves on quartz substrates is actually small enough to consider conductivity changes negligible. The following equation has been derived for materials whose crystal structure is “cfc” and does not directly apply for the experiment presented in this paper but it provides a clear illustration of the two main principles governing the observed variations and an (although simplified) analytical formula for estimating the importance of each contribution (elastic and mass loadings).

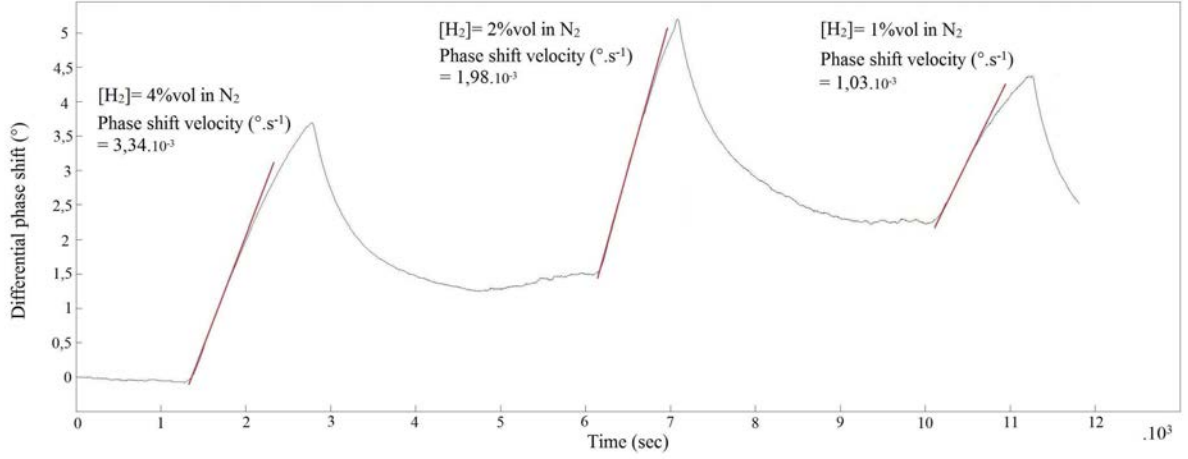


Fig. 5. Detection of hydrogen in the range 4% to 1%vol in N₂.

$$\frac{\Delta\nu}{\nu} = \left(\frac{\pi h}{2\lambda}\right) \left[-\frac{\Delta\hat{\rho}}{\hat{\rho}} \{(A_x^2 + A_y^2 + A_z^2)\hat{\rho}\nu_0^2\} \text{ mass-loading term} \right. \\ \left. + \frac{\Delta\hat{C}_{44}}{\hat{C}_{44}} \{(4A_z^2 + A_x^2)\hat{C}_{44}\} \text{ elastic-loading term} \right. \\ \left. + \left\{ \frac{\left(1 - \frac{\Delta\hat{C}_{44}}{\hat{C}_{44}}\right)^2}{\left(1 - \frac{\Delta\hat{C}_{11}}{\hat{C}_{11}}\right)} - 1 \right\} \left(4A_z^2 \frac{\hat{C}_{44}^2}{\hat{C}_{11}}\right) \right] \text{ elastic-loading term} \quad (1)$$

In case of H₂ adsorption on Pd layer of thickness h through which a SAW of wavelength λ propagates, both the mass density ρ and the elastic constants C11 decrease, whereas the C44 constant increases. The values of normalized mechanical displacement A_i for palladium are reported in [22] and [23]. Numerical calculations of hydrogen absorption in Pd layers [21] predict an increase of C44 elastic constant, yielding an increase of the phase velocity of the Rayleigh wave propagating under such an overlay. These calculations are in agreement with the experimental observations presented here.

4. Theoretical study and simulation results

Accurate calculations have been achieved, based on a set of programs dedicated to the analysis of stratified structures used as elastic wave guides. The basics of the corresponding developments have been presented in [24] [25]. The calculation principle is based on the derivation of a surface or interface effective permittivity yielding numerous information about the wave propagation characteristics such as the wave velocity, the mode coupling and possible propagation losses. The model accounts for all the known linear properties of the materials constitutive of the layered structure which directly condition the accuracy of the results as no assumption is made on the device operation (provided flat interfaces). Here are presented simulation results about mass loading effect. One can see on Fig. 6. the influence of a

growing water film at the surface of the sensor. A phase decrease is observed. It is noticeable that the phase shift observed on Fig. 7. that is relative to the exposure of the sensor to humidity is consistent with the theoretical result. Indeed, that phase decrease corresponds to a reduction of the velocity of $133.10^{-3} \text{ m.s}^{-1}$ which would correspond to the presence, at the top, of the device of a water film that thickness is 4nm which is realistic.

On another hand, calculations of theoretical velocity variation of the wave consecutive to changes of elastic properties of the palladium layer have been made. Simulation results presented on Fig. 8. were obtained on the basis of [21] results. We can see here the influence of each elastic parameter on the velocity of the acoustic wave. According to [21], we considered a decrease of 0,1% and 10% of the density (red solide curve) and the C11 (green triangles) coefficient of the palladium layer respectively. A increase of 40% of the C44 (blue circles) and the induced modification of non diagonals parameters (black squares) were also simulated. The global modification of the acoustic wave velocity is shown on the dashed black curve. One can notice that the velocity increases, upon a complete hydrogenation

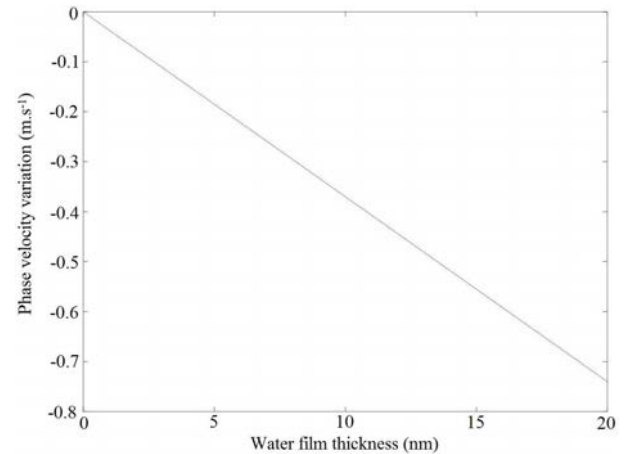


Fig. 6. Simulated condensation of water on the sensitive zone.

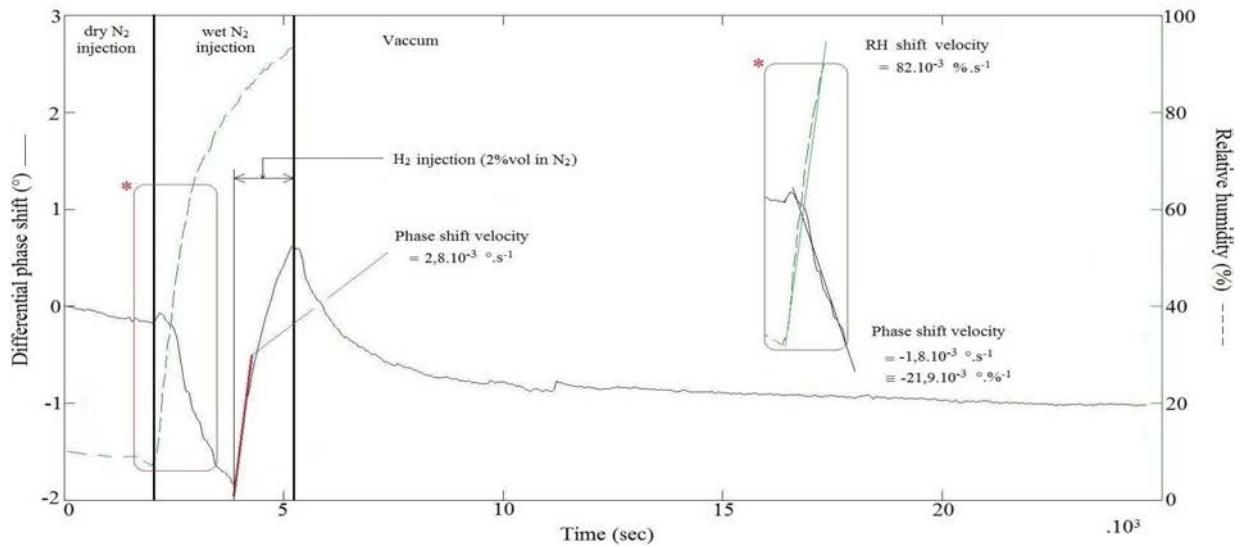


Fig. 7. Influence of humidity on the detection of hydrogen at constant room temperature (19.3 \pm 0.1 $^{\circ}$ C).

(corresponding to $X=0.66$ (PdH_X)), by $1.7\text{m}\cdot\text{s}^{-1}$. This value takes in account the modification of both C_{ij} and density modifications of the palladium layer. It is also necessary to take into account the gravimetric effect due to H_2 adsorption. This phenomenon, shown on Fig. 9., results in a decrease of the wave velocity of $0.5\text{m}\cdot\text{s}^{-1}$ when considering a complete hydrogenation. As a consequence, the predicted global velocity change is an increase of about $1.2\text{m}\cdot\text{s}^{-1}$. This result is in accordance with the observation made on Fig.4. Indeed, the phase shift observed on this figure corresponds to an increase by $2.6\text{m}\cdot\text{s}^{-1}$ of the acoustic wave velocity. The orders of magnitudes are consistent and the difference between these to values ($2.6\text{m}\cdot\text{s}^{-1}$ for the experiments and $1.2\text{m}\cdot\text{s}^{-1}$ from the simulation) can be explained by the fact that the palladium layer used here has quite different geometrical properties than the one that was used in [21] for the determination of the elastic properties modifications. Moreover, the sensitive layer has been here deposited using sputtering method contrary to [21] where thermal evaporation was used.

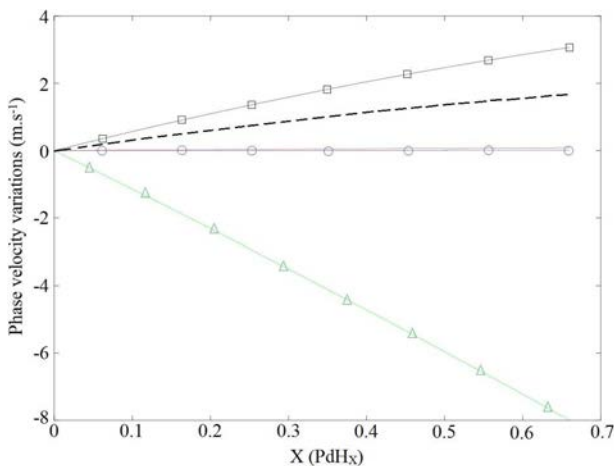


Fig. 8. Wave velocity shift induced by changes in C11 (green triangles), C44 (blue circles), C12 (black squares), density (red solid line), global shift (black dashed line).

5. The influence of humidity and temperature on hydrogen detection

As SAW devices are known to suffer from interference due to humidity and temperature, the influence of these parameters on the sensor operation have been experimentally investigated and the corresponding results are exposed here. The observations reported in this section are expected to provide information allowing for the improvement of the differential acquisition setup. One can see in Fig. 7. that an increase of the relative humidity (RH) of the injected gas causes a massloading effect that results in a decrease of the measured phase of the delay line. Indeed, the condensation of water onto the surface of the device naturally leads to a raise of the mass at the surface of the device (mass loading) yielding the decrease of the experimentally observed phase velocity. As said in the previous section, that behavior is sustained by theoretical observation. Furthermore, a damping phenomenon can clearly be seen on the

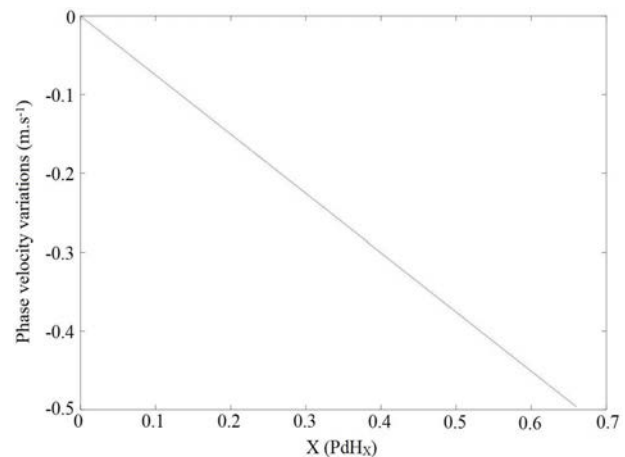


Fig. 9. Phase velocity variation due to hydrogen massloading effect.

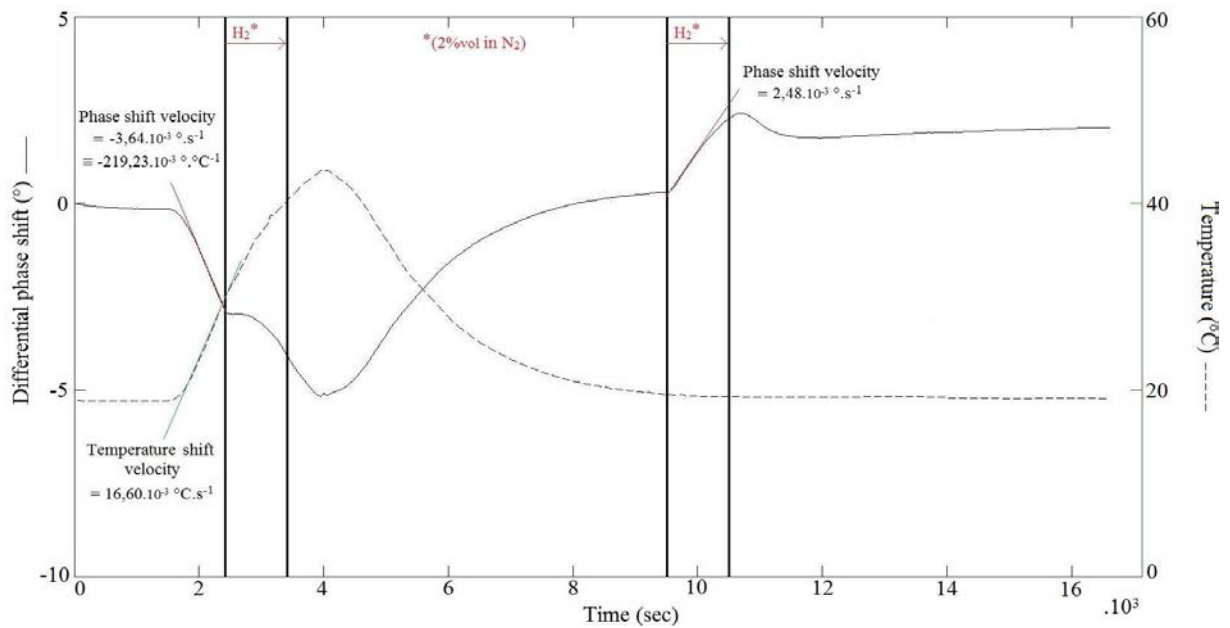


Fig. 10. Influence of temperature on the detection of hydrogen (RH in the 3-8% range).

transfer function of the device when exposed to humidity. Fig. 10. exhibits the impact of temperature variations on the capability of the SAW sensor to detect hydrogen at atmospheric pressure. Temperature changes lead to a decrease of the delay line synchronicity frequency characterized by a sensitivity of $-219.10^{-3} \text{ } ^\circ\text{C}^{-1}$. That phase decrease totally compensates the phase shift toward the high frequency observed when detecting hydrogen in absence of any temperature changes. It appears that the delay line used as a reference does not undergo any phase shift when exposed to heated gas since the device is temperature compensated. However, the TCF of the Pd detection channel notably changes as the Pd overlay tends to lower the 1st order TCF. This behavior currently represents an obstacle to accurate hydrogen detection but can be avoided by the use of an appropriate reference (covered by a layer inert to hydrogen which induces the same TCF change as experienced here for the detection channel). As one can see on the second part of the graph, the detection of H_2 at 2%vol in N_2 is still clearly possible considering a stable operation temperature at room conditions.

6. Conclusion

In this paper, a differential SAW sensor based on Rayleigh wave on quartz has been developed and successfully tested. The sensor consist of two adjacent delay lines, one being used as a reference and the second one being functionalized with a Pd overlay, a metal known to be affected by gaseous hydrogen. The observation of specific phase shifts on the sensitive track compared to reference in presence of gaseous

actually allows segregating the nature of the gas inserted in the reaction cell and provides quantitative information about its composition. The impact of temperature and humidity on the sensor operation also were analyzed. Although the differential nature of the sensor is expected to reject such interferences, it appears necessary to control this parameters at very minimum. Concerning the analysis of the sensor operation itself, some work still has to be carried out even if strong convictions arise from the experiment that the main change is related to the elastic properties of the Pd film due to hydrogen absorption. The crystalline structure of the film having an effective influence on the way hydrogen interact with the metallic film, it still has to be determined in order to improve simulation accuracy of the leverage of hydrogen absorption on the SAW device response. Toward this aim, further work will consist in the elaboration and characterization of palladium layers with different crystalline structures. Simulation of SAW device exhibiting different sensing layers will also be deepened in order to provide a comprehensive analysis of the experimental observations and to predict the structure of the delay-line to use in order to improve the differential acquisition setup. These investigations are expected to enhance the performance of the proposed sensor in terms of sensitivity and selectivity toward outer parameters such as temperature and humidity.

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