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Contents

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Digital Sensors and Sensor Systems: Practical Design

Sergey Y. Yurish

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- Practical orientation
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Thermal Sensitivity of Solid Polymer Coated Surface Transverse/Love Wave Based Resonators on AT-cut Quartz for Sensor Applications

I. D. AVRAMOV and K. D. ESMERYAN
Georgy Nadjakov Institute of Solid State Physics,
72 Tzarigradsko Chaussee Blvd.
1784 Sofia, Bulgaria
Tel.: + (3592) 979 5762

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Abstract: - Thermal and mass sensitivity data from solid polymer coated shear horizontal (SH) wave two-port resonators on AT-cut quartz for sensor applications are measured and compared with data from identically coated Rayleigh surface acoustic wave (RSAW) based resonators of a similar design operating at the same acoustic wave length. The acoustic devices were coated at 6 different thicknesses in the 0 to 300 nm range with the chemosensitive solid hexamethyldisiloxane (HMDSO) polymer film obtained in a plasma polymerization process. It was found that above a certain film thickness the SH-wave resonators simultaneously support two modes – the surface transverse wave (STW) mode and the 1st order Love mode. Major differences in the quasi parabolic temperature frequency characteristics (TFCs) over the (-50 to +120) °C range of these two modes are identified and discussed.

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Keywords: Thermal sensitivity, Solid polymer films, Shear horizontal waves, Love waves, Sensors.

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1. Introduction

One of the most exciting applications of surface acoustic wave (SAW) devices is chemical gas and liquid sensing [1-8]. A chemosensitive layer that absorbs the chemical analyte to be measured is added to the surface of a SAW based sensor device (delay line or resonator) operating on an appropriate for the particular application acoustic wave mode. In the process of absorption the physical properties of the layer change and this affects the way in which the acoustic wave interacts with the layer. Thus variations in the concentration of the measured analyte result in variations of the propagation velocity and amplitude of the acoustic wave [2]. This process is illustrated in Fig. 1 which shows how the resonant peak of a Rayleigh SAW (RSAW) two-port resonator coated with a chemosensitive layer of polyisobutilene (PIB) shifts down when tetrachloroethylene vapors of 6500 ppm concentration are applied to the surface of the RSAW sensor device. In this case, the sensing PIB layer becomes heavier as a result of vapor sorption and mass loading at the sensor surface increases. Accordingly, a sound propagation velocity decrease results in a frequency downshift of 580 ppm. A propagation loss increase by 2 dB is also observed. If the RSAW sensor resonator from Fig. 1 is connected to the feedback loop of a sensor oscillator whose frequency is adjusted at the resonance peak then the gas concentration proportional frequency shifts of the sensor can be measured with a high precision using a high-resolution frequency counter [9].

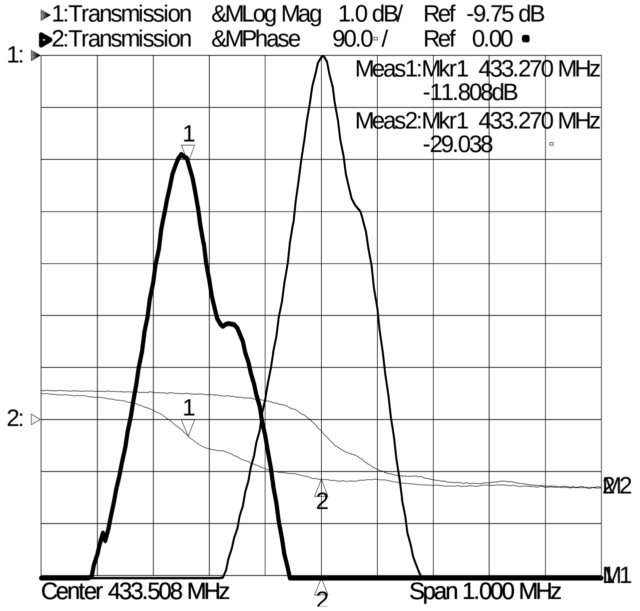


Fig. 1. Frequency responses (upper curves) and phase responses (lower curves) of a PIB coated 433 MHz RSAW two-port resonator before (right) and after (left) tetrachloroethylene vapors are applied to the sensor surface.

An important issue associated with the practical application of a SAW based sensor system that, unfortunately, has not received adequate attention so far is the thermal sensitivity of the film coated sensor device. This problem exists even in sensor devices fabricated on temperature compensated Y-cuts of piezoelectric quartz known for its lowest thermal sensitivity among piezoelectric materials commonly used for SAW devices. Temperature compensated quartz cuts feature parabolic temperature frequency characteristics (TFC) which can cause serious thermal drifts especially at the edges of the operating temperature range [10]. In portable sensor systems operated over a large temperature range the measurement error caused by these thermal drifts can exceed 100 % [11]. The thermal sensitivity problem is aggravated by the fact that the sensing layer will not only cause a thickness dependent shift of the TFC but will also change its slopes. This requires precise knowledge of the sensor's TFC at the selected for maximum chemical sensitivity thickness of the sensing layer so that the sensor reading can be corrected for thermal drifts at the temperature of the measurement [11].

The surface acoustic wave modes most commonly used in chemical sensors are the classical RSAW mode featuring elliptic motion and the surface transverse wave (STW) mode and its solid film guided version – the Love wave (LW) mode, both featuring shear horizontal (SH) motion. The RSAW mode provides excellent results in gas-phase environments while the STW/LW mode works well in both –

gas-phase and liquid environments [2]. While the thermal sensitivity of RSAW devices coated with solid chemosensitive layers has been sufficiently well documented [11-14], little is known about the thermal behavior of solid film coated STW/LW-based sensors, although very attractive features and advantages of the STW and LW modes for sensor applications have been demonstrated. For example, STW resonant devices on temperature compensated rotated Y-cuts of quartz tolerate solid chemosensitive layers much better and provide substantially higher gas sensitivities and lower flicker noise than their RSAW counterparts [9]. On the other hand, solid film coated LW mode devices on quartz demonstrate excellent mass sensitivity when operated as biosensors in water based liquid environments [15].

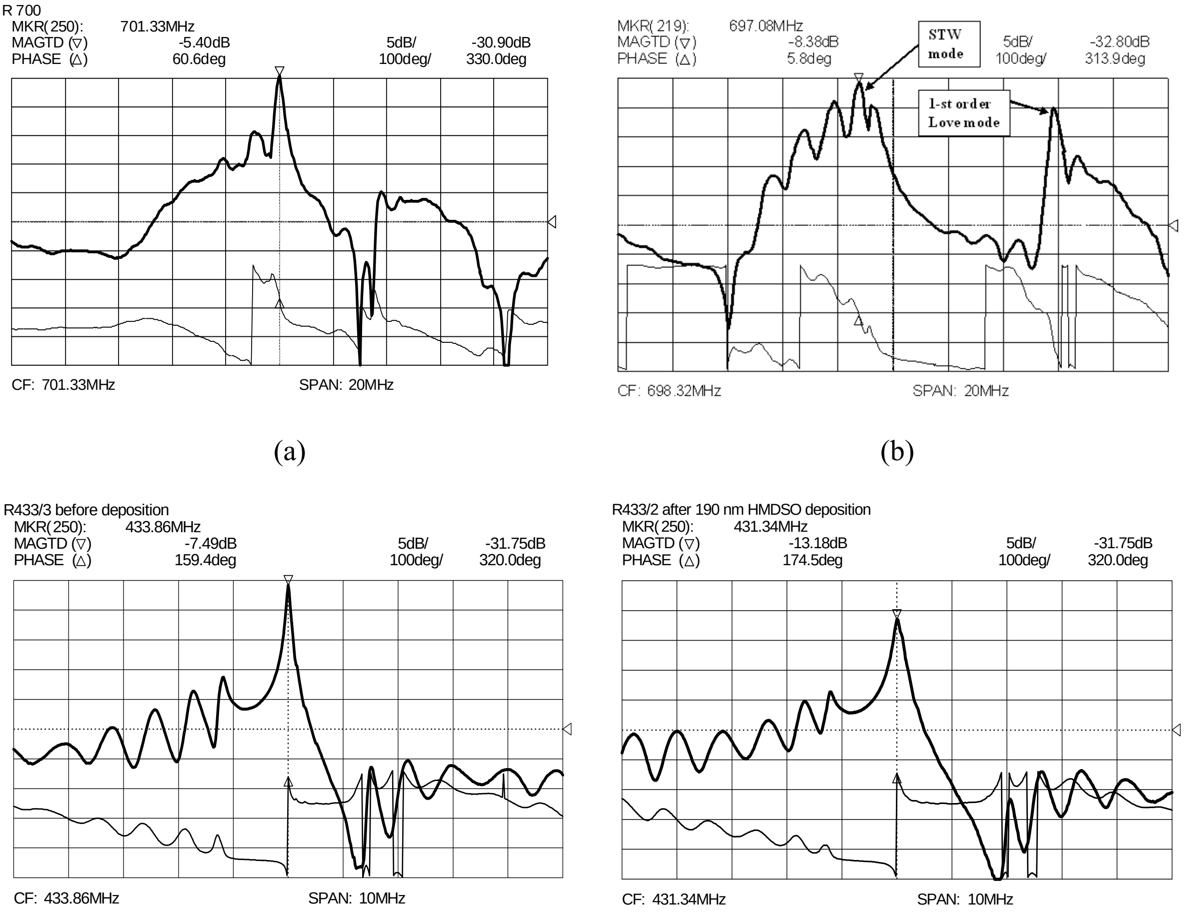
This paper provides systematic experimental data on the thermal behavior of two-port resonators on AT-cut quartz supporting the STW and LW modes as a result of solid chemosensitive polymer coatings as used in a variety of gas and liquid-phase sensors. Hexamethyldissiloxane (HMDSO) is used as a chemical interface. The influence of the HMDSO thickness on the TFC and the overall thermal stability of the sensor is studied in detail and compared to the behavior of identically coated RSAW sensors on AT-cut quartz operating on the acoustic wave length. The thermal sensitivity of the 1-st order Love mode that is simultaneously excited with the STW mode in devices with sufficiently thick HMDSO layers is also measured and discussed. It is shown that solid chemosensitive polymer films can cause significant improvement in the thermal stability of STW resonant sensors compared not only to the uncoated STW device but also to their RSAW counterparts.

2. Solid Film Coating behavior of SH and RSAW Resonators

Before addressing the temperature performance of coated STW/LW resonators it is first necessary to study their film coating behavior and compare it to RSAW two-port based resonators of nearly identical device geometry to identify peculiarities and major differences. The devices we used for this comparison feature the classical two-port resonator structure consisting of two identical interdigital transducers (IDTs) with a center waveguide grating in between and two reflector gratings on both sides of the IDTs to form the standing wave pattern. Further design details are provided in [16]. For a fair comparison, the 433 MHz RSAW and the 701 MHz STW devices that we used in these coating experiments both operate at the same acoustic wavelength of 7.22 μm on AT-cut quartz. This cut is the 36 degree singly rotated Y cut providing quasi parabolic temperature frequency characteristic for both acoustic wave modes. The propagation velocities for the RSAW and STW modes in the uncoated state is 3133 m/s along the X-axis and 5064 m/s perpendicular to the X-axis, respectively, and are slightly lower than the free surface propagation velocities of 3159 for the RSAW and 5100 m/s for the SH modes, accordingly. This is attributed to the mass loading effect of the periodic Al strip structure forming the transducers and reflectors [16].

In this work we coated the resonators with the solid polymer film hexamethyldisiloxane (HMDSO) which is deposited in a radio frequency (RF) plasma reactor. The plasma polymerization process we used for deposition provides good reproducibility and control over the film thickness. It is described in Ref. [11] in detail. The reason why we used HMDSO is that it is a typical representative of the solid chemosensitive polymer family and is often used in a variety of SAW based sensors [9]. Furthermore, HMDSO provides excellent adhesion to the substrate surface, is very stable over temperature and time and does not change its mass sensitivity over many probing cycles. It is our belief that the HMDSO based data that we discuss here may be indicative also for other solid polymer films with similar viscoelastic properties and featuring surface sorption as used in SAW based sensors.

The HMDSO coating behavior of the STW vs. RSAW devices is shown in Fig. 2 for 200 nm film thickness.



(a) (b)

(c) (d)

Fig. 2. Frequency (upper curves) and phase (lower curves) responses of a 700 MHz STW two-port resonator (upper row) (a) prior to and (b) after 200 nm HMDSO deposition. The data on the lower row applies to the 433 MHz RSAW device (c) prior to and (d) after HMDSO deposition of the same thickness in the same deposition process.

From those data it becomes evident that both modes respond quite differently to identical mass loading by the solid polymer film. The RSAW device (see Fig. 2 c) and d)) shifts its resonance frequency down by 2.5 MHz (3560 ppm), its insertion loss increases by 5.7 dB and its loaded Q decreases from 6000 to about 2000 as a result of film deposition. The STW device shifts its resonance down by about 4 MHz (6100 ppm) meaning almost twice as high mass sensitivity while its insertion loss decreases just by 3 dB. On the other hand, a second SH mode arises at about 7 MHz higher than the main STW resonance, (see Fig. 2 b)) which now appears fairly distorted due to film degraded phase conditions along the propagation path. According to [2] this second mode is the 1-st order Love mode while the left one is the 0-th order Love mode that is also called by many authors “STW mode” for simplicity. The 1st order Love mode can exist in a layered structure consisting of a substrate and a guiding layer only if the shear velocity in the layer is smaller than in the substrate. The fact that such a Love mode is clearly observed in Fig. 2 b) indicates that a HMDSO coated STW two-port resonator supports this mode and the SH wave velocity in the HMDSO film is smaller than in the substrate. In this case the layer slows down the acoustic shear mode in the substrate, thus decreasing the penetration depth and confining the acoustic energy to the surface [2]. At zero or very small thickness of the guiding layer (0 nm in the uncoated STW device from Fig. 2 a)) the acoustic field deeply penetrates into the substrate. When thickness increases guiding becomes more efficient, the penetration depth decreases and a larger fraction of the total wave energy propagates in the layer. For very thick layers almost the entire energy is trapped in the guiding layer. As shown in [2] at a high thickness several Love modes are supported in a delay line structure on rotated Y-cut quartz. The data in Fig. 2 b) clearly indicate that such multimode behavior is quite efficiently supported also by two-port STW resonator structures on AT-cut quartz even at fairly thin HMDSO films (200 nm in this case) that are close to the optimum for maximum mass sensitivity [9]. This implies that STW/LW resonators on quartz may become an attractive alternative to LW delay lines for biosensor applications in liquid phase environments [2-7] since two-port resonators retain much lower loss and higher Q compared to their delay line counterparts. This feature is of major importance to low sensor noise levels [9].

3. Temperature behavior of HMDSO Coated STW/LW Resonators Versus RSAW Devices

In the past, substantial effort was dedicated to the thermal sensitivity of uncoated SH-wave resonant devices. In most cases these devices support only the STW mode due to the fairly weak mass loading effect caused by the metallization and/or etched grove structure used for excitation and guiding the wave energy at the device surface for maximum efficiency and loaded Q [16-18]. Major goal of those efforts has been to achieve temperature compensation for minimum thermal sensitivity over a desired temperature range in STW devices whose periodic metal electrode and/or groove structure has been optimized for optimum electrical performance. Ballandras, et. al. used a perturbation method to predict the existence of temperature compensated quartz cuts for periodic groove and metal strip gratings [19]. They also showed that the wave guiding periodic perturbation on the surface of the STW device can significantly shift its turn-over temperature (TOT) and predicted the thermal sensitivity as a function of the perturbation parameters – metal thickness and groove depth [20].

As shown in the previous sections, for the excitation of the 1-st order Love mode, especially for sensor applications, an additional guiding layer is required. This layer will cause a serious change in the surface perturbation parameters and the temperature coefficients, accordingly [15]. In the next sections we will discuss the influence of film thickness on the TFCs of HMDSO coated STW/LW resonators and will compare their thermal and mass sensitivities with identically coated RSAW sensors from previous work.

3.1. Experiments and Temperature Stability Evaluation Criterion

In this work we measured the TFCs of 6 STW two-port resonators as the ones from Fig. 2 a) and b), HMDSO coated at 6 different film thicknesses: 0, 50, 100, 150, 250 and 300 nm. The controlled plasma polymer process for film deposition and the TFC measurement setup are the same as used in Ref. [11] for the RSAW devices from Fig. 2 c) and d). Each resonator was connected to the feedback loop of an oscillator whose frequency was adjusted at resonance and recorded with a frequency counter. For sufficiently thick film coated devices supporting both the STW and the LW modes the oscillator was first excited on the STW mode for its TFC measurement. Then, for the LW measurement, a half wavelength loop adjust line was added to the loop to excite the oscillator on the LW mode which is by 180 deg. phase reversed compared to the STW mode (see phase responses for both modes in Fig. 2 b)). In both cases stable oscillation was obtained over the entire measurement temperature range from -50 to +120 °C.

Since each film thickness provides not only a shift of the TFC along the temperature axis but also changes its slopes, for better comparative evaluation of the overall thermal sensitivities we introduced the thermal sensitivity criterion that we call “100 K temperature stability” (100 KTS). As shown in Fig. 3 the 100 KTS represents the overall temperature induced frequency shift over a span of 100 K, symmetrically plotted on both sides of the TOT point, regardless of the TFC position w.r.t. the temperature axis. In the example of Fig. 3 the TFC has a TOT of 50 °C and the 100KTS is 135 ppm.

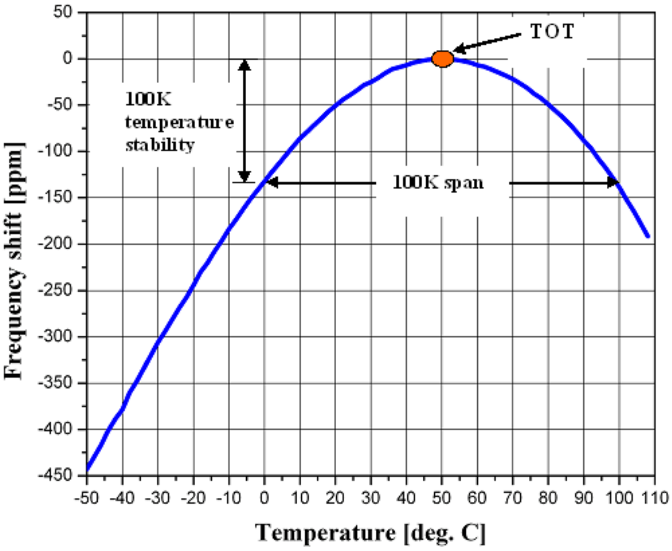


Fig. 3. Definition of the 100 K temperature stability illustrated on one of the tested STW devices.

3.2. Results and Discussion

Fig. 4 compares the TFCs of both uncoated RSAW and STW devices from Fig 2 a) and c), accordingly. For a better visual comparison, the device pair has been selected with nearly identical TOT point in the 30 to 35 °C range. It is clearly seen that in the uncoated state the STW device features steeper slopes of the TFC on both sides of the TOT and provides 50 ppm thermal instability over a 55 K temperature range in this measurement. These data are consistent with the theoretically predicted behavior in [17] and [21]. With 50 ppm drift over a wider 80 K span the uncoated RSAW device clearly demonstrates superior thermal stability compared to its uncoated STW counterpart. However, as shown in Fig. 5, this advantage is almost lost when both devices are coated with 250 nm HMDSO.

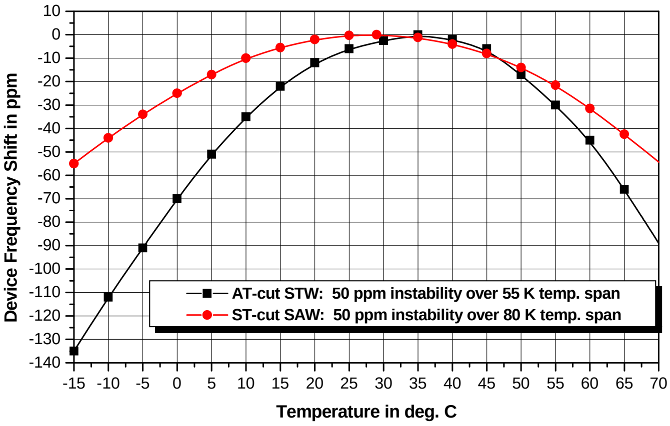


Fig. 4. TFCs of the RSAW vs. STW modes in the uncoated state.

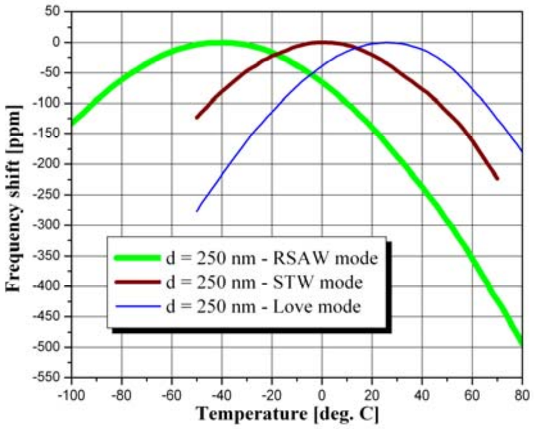


Fig. 5. TFC comparison of the RSAW, STW and LW modes at 250 nm HMDSO thickness.

In that case the 100 KTS becomes 100 ppm for the RSAW vs. 110 ppm for the STW device. Fig. 5 contains also the data plot for the LW mode supported by the STW device at the 250 nm thickness for comparison. The 100 KTS for the LW mode reads 150 ppm and is worst among the three modes. This

LW mode behavior is explained by the fact that the 1-st order LW mode propagates much deeper than the STW mode [2] and its thermal sensitivity is dominated by the quartz substrate. The STW mode on the other hand is confined in the HMDSO film and its temperature behavior is dominated by the film, accordingly. Obviously, the thermal sensitivity of the HMDSO film is substantially lower than the quartz substrate and as a result of that this polymer provides a thermal compensation to the STW mode improving its thermal stability. This compensation effect seems to be much weaker with the RSAW mode which even degrades its 100 KTS from 70 to 100 ppm in the uncoated vs. the 250 nm coated case, respectively. This means that the RSAW thermal sensitivity is always dominated by the quartz crystal which is to be expected since the RSAW energy in this case penetrates several wavelengths into the substrate and the thin (compared to the wavelength) 250 nm HMDSO film has only a small influence on the overall RSAW thermal sensitivity. The 30 ppm degradation of the 100KTS in this case we attribute to surface stress caused by the HMDSO film which does not seem to be so pronounced in STW devices due to the different type of motion. The thermal sensitivities of the STW mode for all 6 HMDSO thicknesses are shown in Fig. 6.

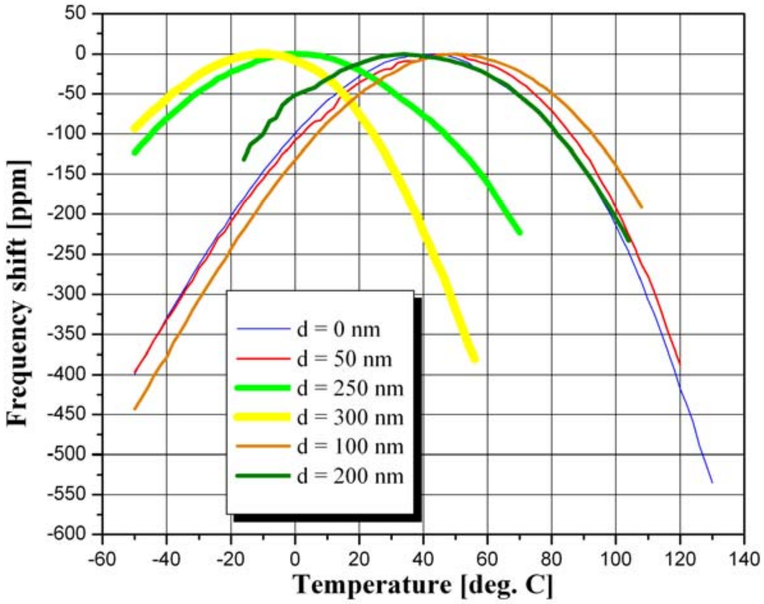
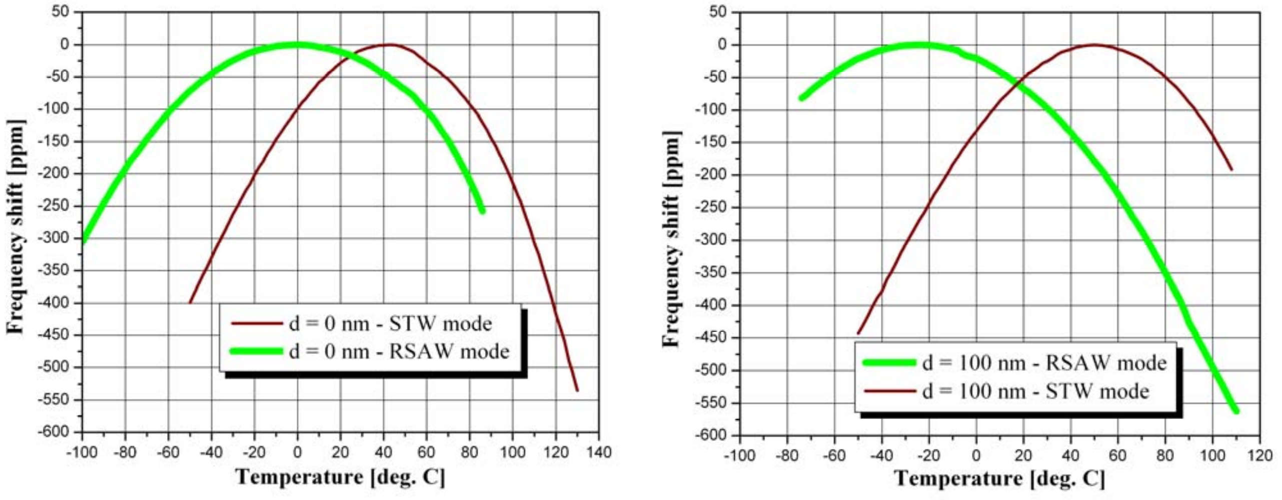


Fig. 6. TFCs of the STW mode at 6 different HMDSO thicknesses.

Here an interesting phenomenon is observed. When the thickness increases from 0 to about 100 nm the TOT shifts up from 40 to about 50 °C and then rapidly decreases for thickness values above 100 nm. As shown in Fig 7 a) and b), this TOT behavior is quite different from the RSAW mode and similar to the one observed in [20] with grooved resonators. It is again attributed to the fact that the thermal properties of the STW mode are dominated by the guiding layer where most of the wave energy is confined. This phenomenon may be of great importance to the design of practical STW based sensors in which small variations of the solid polymer thickness within the maximum mass sensitivity range would allow for precise adjustment of the TOT at the desired value for minimum thermal sensitivity. This procedure is much easier and less expensive than fabricating new sensor devices on a corrected quartz cut. From the data in Fig. 7 it becomes evident that the TOT of the RSAW mode is much more sensitive to film loading than its STW counterpart. When the thickness increases from 0 to 100 nm the TOT of the STW mode shifts up by 10K versus 28K down shift for the RSAW mode. Fig. 8 represents the TOT behavior of the STW and RSAW modes as a function of the film thickness. It is clearly seen that, with increasing film thickness, the STW TOT point first moves towards higher temperatures and then shifts down towards lower ones. The TOT of the RSAW mode follows only a downward direction.



(a)

(b)

Fig. 7. TOT sensitivity to HMDSO deposition in RSAW vs. STW modes at (a) 0 nm, and (b) 100 nm thickness.

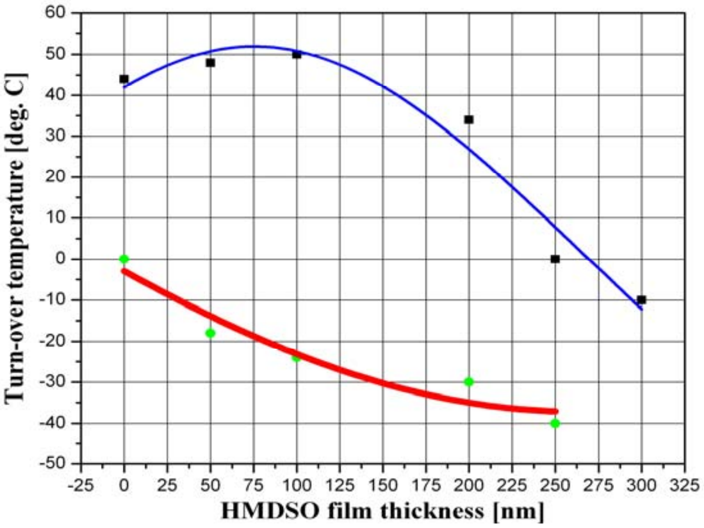
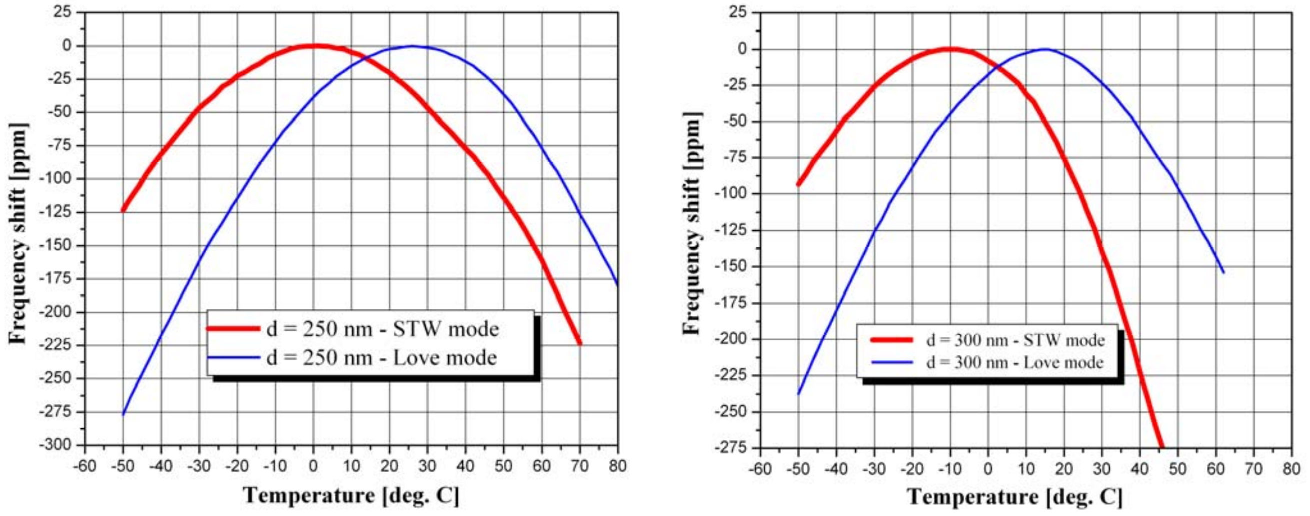


Fig. 8. TOT shift vs. film thickness for the STW (upper curve) and RSAW (lower curve) modes.

The Love mode that starts building up at thicknesses exceeding 100 nm, becomes strong enough at about 200 nm as shown in Fig. 2 b) and at 250 nm and higher its magnitude becomes equal to the STW mode. This makes it fairly easy to excite the measurement oscillator on either of both modes just by adding or subtracting 180 deg. of phase from the oscillator loop with a piece of coaxial cable. The data plots in Fig. 9 a) and b) compare the LW mode's thermal sensitivity with the STW mode at 250 and 300 nm, respectively. Two characteristic features are observed here. On one hand, the slopes of the TFCs for both modes become steeper indicating degradation in thermal stability when the thickness increases from 250 to 300 nm. Also the TFC of the STW mode becomes asymmetric with the right slope much steeper than the left one (compare also the 300 nm curve with the lower thickness ones in Fig. 6). This indicates that at very large thicknesses the STW temperature dependence changes shape and degenerates from parabolic into cubic with an expected second inversion point at very low temperatures. A similar behavior has also been reported with uncoated metal strip STW resonators in [18]. Here again, we attribute this phenomenon to the dominating role of the solid guiding layer. On the other hand, both TFC curves in Fig. 9 a) and b) shift by 10 K towards lower temperatures, however, the TOT points for both modes remain spaced by 25 K at both thicknesses. This means that the presence of the film affects not only the guided STW but also the substrate dominated 1-st order Love mode.



(a) (b)

Fig. 9. LW vs. STW thermal sensitivities at (a) 250, and (b) 300 nm HMDSO thickness.

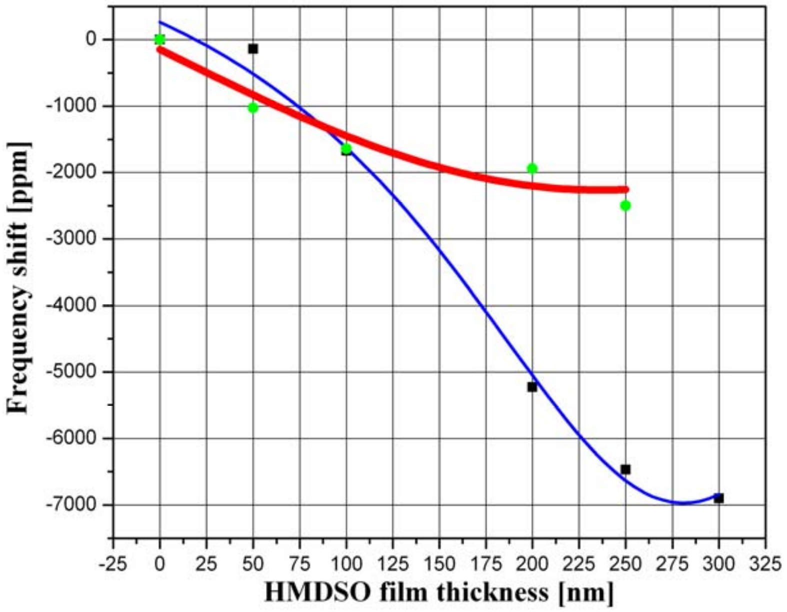


Fig. 10. Mass sensitivities of HMDSO coated RSAW (upper curve) and STW (lower curve) two-port resonators operating at the same acoustic wavelength.

Finally, for the sake of completeness, Fig. 10 compares the mass sensitivities of the STW and the RSAW modes in the HMDSO coated two-port resonators from Fig. 2. At the same acoustic wave length, the STW mode demonstrates up to 2.5 times higher mass sensitivity than its RSAW counterpart. This is consistent with data from other solid and semisolid polymer films presented in [9]. The better mass sensitivity is another advantage of the STW mode operating with solid chemosensitive films in sensors.

The results of this experimental study have been summarized in Tables 1, 2 and 3 for the 100KTS, the TOT and mass sensitivity, respectively.

Table 1. HMDSO thickness dependence of the 100KTS for the RSAW, STW and LW modes.

		HMDSO thickness in nm						
		0	**50**	**100**	**150**	**200**	**250**	**300**
100KTS	**RSAW**	70 ppm	67 ppm	80 ppm	85 ppm	n/a	100 ppm	n/a
	STW	150 ppm	140 ppm	130 ppm	n/a	120 ppm	110 ppm	175 ppm
	LW	n/a	n/a	n/a	n/a	n/a	140 ppm	160 ppm

Table 2. HMDSO thickness dependence of the TOT for the RSAW, STW and LW modes.

HMDSO thickness in nm								
	mode	**0**	**50**	**100**	**150**	**200**	**250**	**300**
TOT	**SAW**	0 °C	-18 °C	-24 °C	-30 °C	n/a	-40 °C	n/a
	STW	40 °C	48 °C	50 °C	n/a	34 °C	0 °C	-10 °C
	LW	n/a	n/a	n/a	n/a	n/a	26 °C	16 °C

Table 3. HMDSO thickness dependant resonance frequency shift (mass sensitivity) of the RSAW and STW modes.

HMDSO thickness in nm								
		0	50	100	150	200	250	300
Mass sensitivity	**RSAW**	0 ppm	1025 ppm	1643 ppm	1943 ppm	n/a	2500 ppm	n/a
	STW	0 ppm	138 ppm	1670 ppm	n/a	5230 ppm	6470 ppm	6900 ppm

4. Summary and Conclusions

In this work we have presented results from systematic experimental studies on the thermal sensitivity of solid polymer coated two-port SH resonators supporting the STW and 1st order Love wave mode in AT-cut quartz substrates and have compared them with data from identically coated RSAW devices operating on the same acoustic wave length. Plasma polymer HMDSO films of 6 different thicknesses have been used for coating. Major differences in the coating and thermal sensitivity behavior of the RSAW vs. STW/LW modes are attributed to the wave guiding effect of the solid film in the SH wave devices which is not present in the RSAW ones. Film guiding affects not only the STW but also the deeper penetrating 1-st order Love wave mode whose TFC shifts only down in temperature when film thickness increases. A similar TFC downshift is observed also with coated RSAW devices. We also found that HMDSO substantially improves the thermal stability of the coated STW sensor by reducing the slopes of its TFC thus providing partial temperature compensation. On the other hand, the film has a negligible effect on the temperature stability of the RSAW mode which is attributed to the large penetration depth of this mode into the quartz substrate dominating the temperature performance in this case. An interesting effect of the film coating on the turn-over temperature of coated STW devices is also observed. When HMDSO film thickness increases from 0 the TOT point first shifts towards higher temperatures and then moves downwards. This provides an elegant and cost effective way of positioning the TOT point at a desired temperature, thus minimizing the thermal sensitivity of the coated sensor just by small variations of the guiding layer thickness within the optimum mass sensitivity range of the STW sensor. This is not possible with the RSAW mode whose TFC moves only downwards with increased film thickness. Thus another advantage of solid film coated STW sensors has been identified – along with better mass sensitivity, they provide an easy way for temperature compensation and TOT adjustment avoiding the necessity to fabricate new sensor devices on corrected quartz cuts.

It is the authors' belief that the results obtained with the HMDSO films will apply also to other solid chemosensitive films used for sensor applications and will help those working in the sensor area to design better STW/LW sensors on temperature compensated cuts of quartz. Further work is necessary to improve the efficiency of such types of sensors in water based liquid environments.

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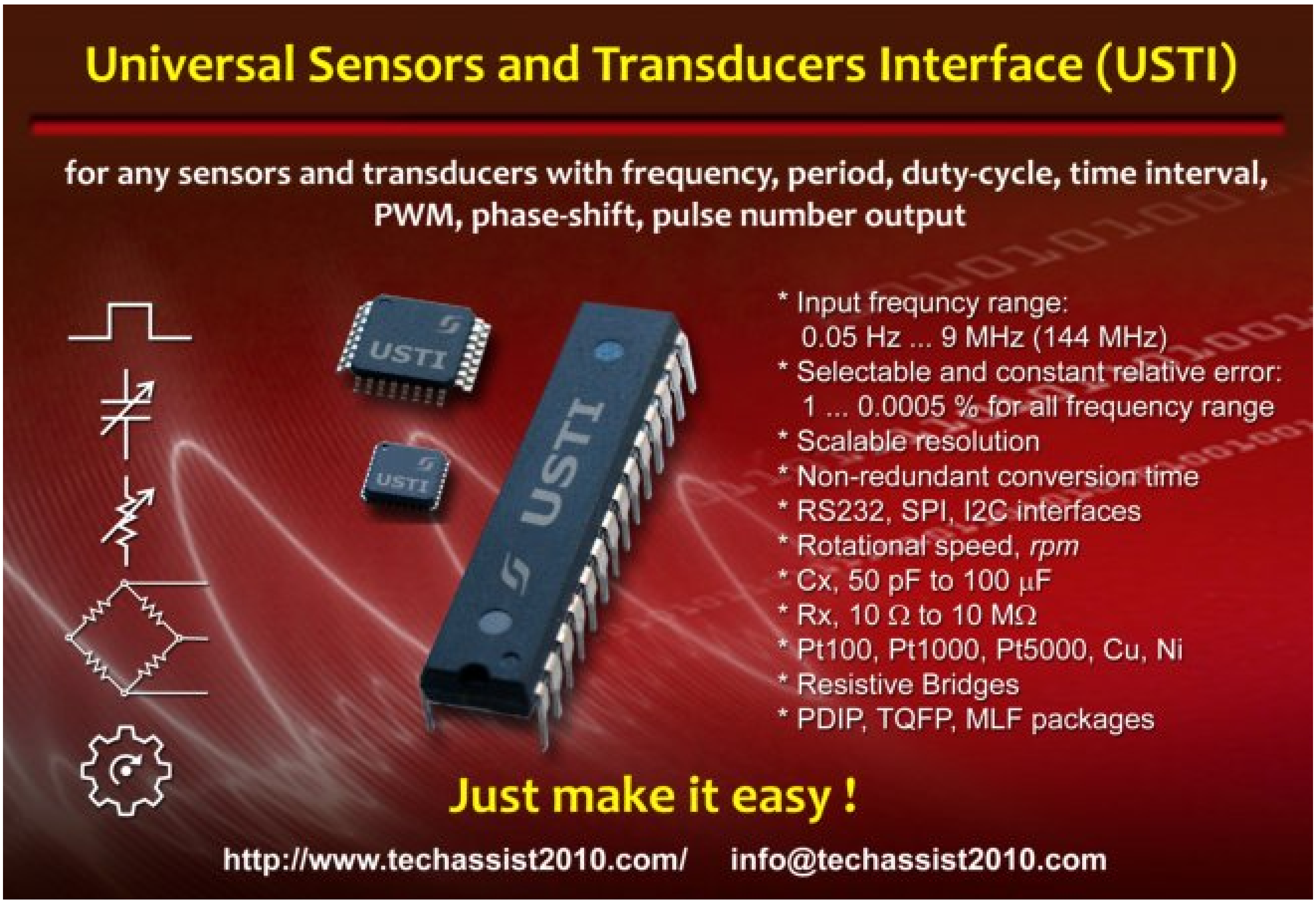

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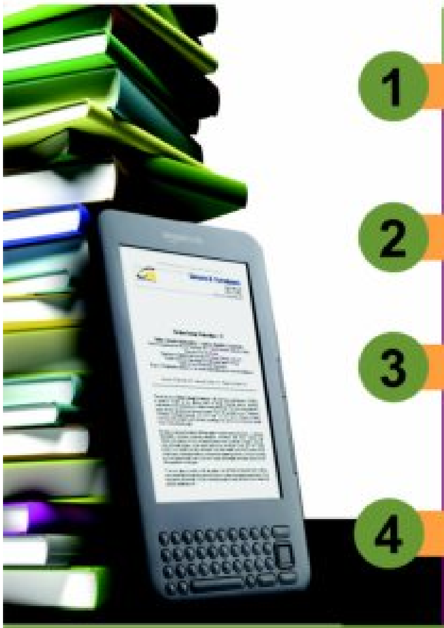

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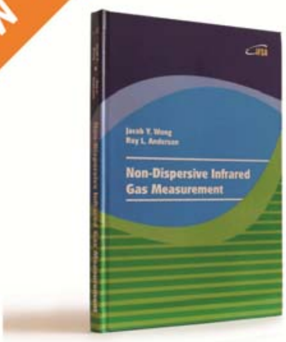

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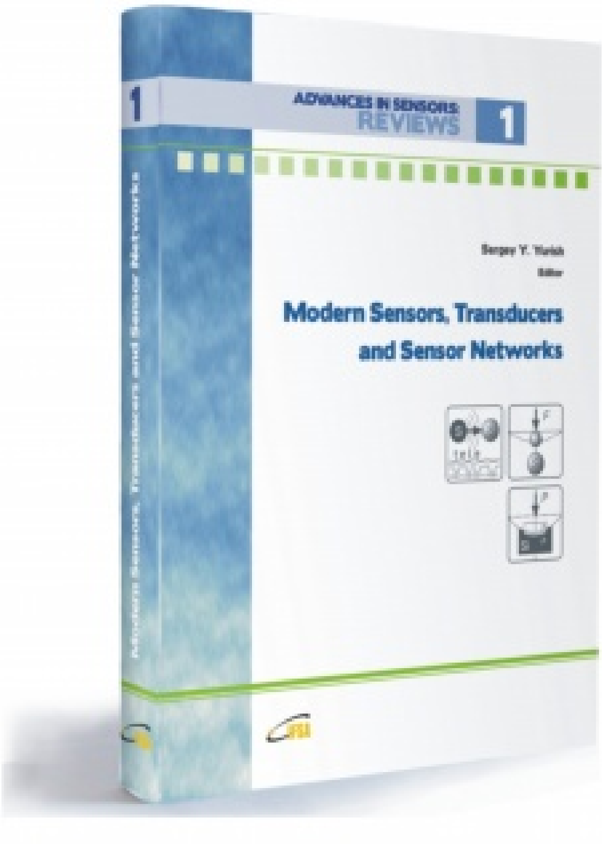

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