High precision pressure sensors based on SAW devices in the GHz range

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A B S T R A C T

In this paper, an AlN/free-standing nanocrystalline diamond (NCD) system is proposed in order to process high frequency surface acoustic wave (SAW) resonators for sensing applications. The main problem of synthetic diamond is its high surface roughness that worsens the sputtered AlN quality and hence the device response. In order to study the feasibility of this structure, AlN films from 150 nm up to 1200 nm thick have been deposited on free-standing NCD. We have then analysed the influence of the AlN layer thickness on its crystal quality and device response. Optimized thin films of 300 nm have been used to fabricate of one-port SAW resonators operating in the 10–14 GHz frequency range. A SAW based sensor pressure with a sensibility of 0.33 MHz/bar has been fabricated.

1. Introduction

For the last few decades, surface acoustic wave devices have been used not only for wireless communication systems [1], but also for gas [2–4], temperature and pressure sensors [5–7] due to their intrinsic energy confinement in a narrow region underneath the surface. Furthermore, the interest in tyre pressure monitoring systems (TPMS) for the automotive industry has increased in recent years. First generation of TPMS was based on MEMS pressure sensors and battery-powered active transmitters positioned in every wheel [8]. The use of SAW sensors offers the most promising solution for batteryless second generation TPMS [9,10]. In a longer term, implantable blood pressure sensors are envisioned.

Different factors are under consideration in the selection of a suitable substrate for SAW sensors, including the electromechanical coupling coefficient (k^2), the surface acoustic wave velocity, and the temperature coefficient of delay (TCD). Moreover, the sensitivity increases for higher frequencies [11]. The central frequency of a SAW resonator, given by $f = v/\lambda$, depends on many physical parameters. Each material has a phase velocity, v, and λ is the wavelength corresponding to the period of the interdigital transducers (IDTs). These are not the only parameters that can be changed in order to modify the central frequency. In a slow on fast structure such as the one used for this work, the substrate material and the piezoelectric film thickness play an important role to determine the resonance of the device under test, as discussed previously [12].

Eqs. (1) and (1a) show the linear approximation of the SAW resonator frequency dependence on pressure when the temperature is kept constant.

$$f(p) = f_0 + S^*(p - p_0) \tag{1}$$

$$S^* = f_0 S \tag{1a}$$

where p_0 is a reference value and S is the first order pressure coefficient [13]. The higher the central frequency, the higher the shift in the frequency for the same pressure change.

In order to achieve higher frequencies, either the selection of a suitable material with higher phase velocity or a smaller IDT period must be considered [12]. Aluminium nitride (AlN) deposited on chemical vapour deposition (CVD) diamond is a very attractive structure for high frequency SAW devices. AlN has a temperature coefficient of −25 ppm/°C [14], a k^2 between 0.2 and 4%, and a phase velocity of 5600 m/s [15,16]. On the other hand, CVD diamond is an appropriate material to work in harsh environments [17] and its SAW velocity is approximately 12,000 m/s [18], the largest of all materials. The temperature coefficient of delay (TCD) of the AlN/diamond system is between −21 and −9 ppm/°C, according to [18]. These properties make CVD diamond combined with AlN an attractive structure for the processing of high frequency SAW sensors. Thus, apart from the enhanced sensitivity at higher frequencies as discussed in [11], the use of the AlN/Diamond bi-layer system is also justified by the possibility of using this structure in harsh environments and by its low TCD.

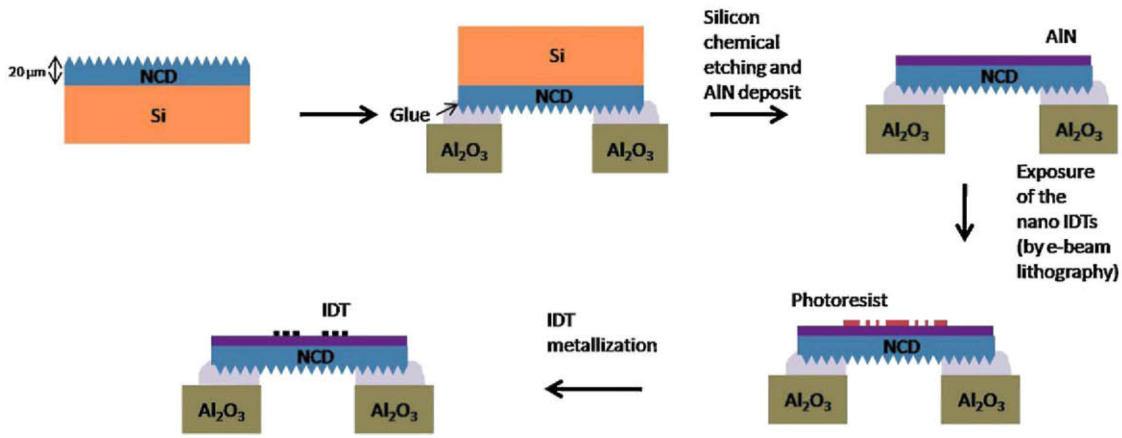

Fig. 1. Schematic process followed to obtain the free-standing diamond used to deposit AlN and process SAW device sensors.

CVD nano-crystalline diamond on silicon has been used in this work to deposit AlN films by reactive sputtering. The principal disadvantage of this synthetic diamond is its high surface roughness. Several methods have been proposed to overcome this problem [19]. Mechanical polishing of the diamond surface has been used for microcrystalline diamond (MCD) in order to solve it [20,21]. However, this procedure is expensive, time-consuming and not yet optimized for NCD. Some works have proposed alternative methods in order to overcome these disadvantages. These consist of using the smooth unpolished nucleation side of freestanding diamond layers [22–24]. This solution requires the whole Si substrate to be sacrificially etched. At least 100 μm thick films are required for structural stability, and thus this method is expensive, as the growth of a free-standing diamond film is a long duration process (approx. 1 μm/h). However, in this work, an alumina substrate was used to get a more robust structure so the thickness requirement was alleviated; in this case, 20 μm thick diamond films were employed. Moreover, the achieved frequency for the devices processed on 100 μm films is below 1 GHz, far less than reported here.

Apart from the surface roughness, different technological requirements must be satisfied in order to obtain high frequency devices. The quality of the *c*-axis AlN film and hence its piezoelectricity is thickness dependent [25]. Furthermore, low surface roughness and small grain size are closely connected with low propagation losses [26].

In this work, we use a 20 μm thick NCD, free standing membrane, whose silicon substrate was removed by chemical etching, to fabricate high frequency SAW resonators for mechanical sensors. This structure not only allows deposition of highly *c*-axis oriented AlN due to the low surface roughness of the nucleation side, but also the membrane is free to move so it provides pressure sensitivity. Furthermore, in some sensor systems, the property to be analysed can be more precisely controlled if a cavity can be fabricated in the structure [27].

On the other hand, the choice of the piezoelectric film thickness implies a compromise between the frequency and the intensity of the targeted resonances, as discussed previously [12]. Here, the influence of the AlN thickness on the SAW response of these free-standing structures is studied. The optimum AlN thickness was found to be 300 nm and allows manufacturing high performance and high frequency SAW resonators working above 10 GHz, with similar performance as those achieved before on polished MCD [28]. A sensitivity of 0.33 MHz/bar was observed under pressure conditions.

2. Experimental

NCD substrates were used to fabricate a free-standing structure by using the nucleation side of the NCD to deposit the AlN thin films. The procedure is summarized in Fig. 1. A home built balanced magnetron sputter deposition system was used to deposit the piezoelectric film. NCD substrates were grown on silicon according processes published elsewhere [29]. The rough side of the NCD was glued with an epoxy adhesive to an alumina substrate with a hole in the middle. The silicon substrate was removed by $HF:HNO_3$ 2:1 chemical etching at room temperature. NCD films from 2 μm- to 20 μm-thick were used to obtain free-standing diamond. The 20 μm-thick NCD is the only film that did not break during the processing to obtain the free-standing diamond.

The substrates were cleaned using a standard procedure before each deposit: rinsing in pirrolidone for 5 min at 80 °C, acetone for 5 min at 60 °C and methanol in an ultrasonic bath. The sputtering conditions were optimized in order to obtain highly *c*-axis oriented AlN at low temperature, determined by X-ray diffraction (XRD) as described elsewhere in [12]. The crystallographic structure of these

Table 1
Processing conditions used during the reactive sputtering of AlN on diamond substrates.

Parameter	Value
Power [W]	700
Base pressure [mbar]	$<5 \times 10^{-8}$
Gas composition Ar/N_2 [sccm]	3/9
Process pressure [mTorr]	3
Substrate temperature [°C]	25
Target substrate distance [mm]	45
Target diameter [mm]	101.6
Target thickness [mm]	6.35
Deposition rate [nm/min]	80

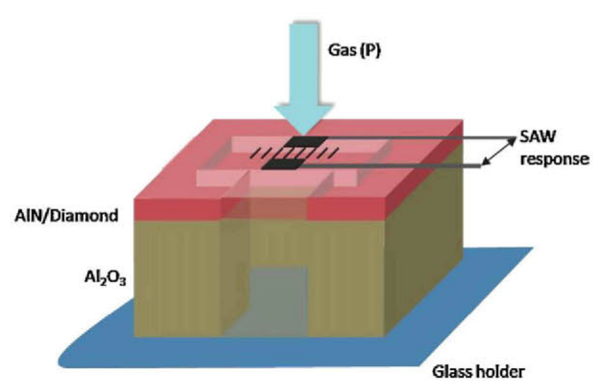

Fig. 2. Schematic of the set-up used to characterize the SAW based pressure sensor.

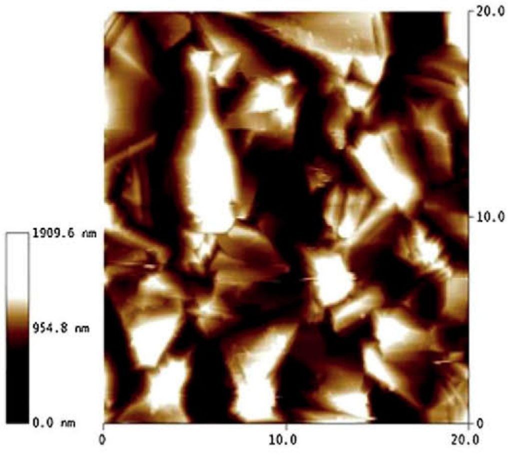

Fig. 3. AFM micrographs of rough (a) and nucleation side surface (b) of NCD.

films was examined using a Phillips X-Pert Pro MRD diffractometer. Although the diamond grains were randomly oriented, only the peak corresponding to the (1 1 1) direction appeared in the spectra. AlN films of approximately 150 nm-, 300 nm-, 600 nm- and 1.2 µm-thick were deposited on free-standing NCD substrates under the conditions shown in Table 1.

Aluminium IDTs were fabricated by e-beam lithography with Crestec CABL-9500C equipment. The periodicity of the finger pairs was nominally 800 nm, i.e. finger width and pitch of 200 nm. The number of periods was 100 for both, the IDT and the reflector. Once the process was optimized, the reflection function of the devices was measured with an Agilent N5230A vector network analyzer using coplanar-waveguide probe tips and standard calibration techniques.

Nitrogen gas in a pressure range of 2–5 bars was used to measure the pressure sensitivity of the SAW sensor. A schematic illustration of our SAW pressure measurement configuration is shown in Fig. 2. The SAW sensor was located on the edge of a 5 mm^2 diaphragm and a pressure controlled nitrogen gas flow was located approximately 5 mm above the membrane. The pressure underneath the AlN/diamond structure was kept constant at atmospheric pressure. The pressure applied to the membrane results from the difference (Δp) between the pressure applied on top ($P1$) and the atmospheric pressure underneath the AlN/diamond membrane ($P2$).

3. Results and discussion

3.1. AlN sputtering on free-standing diamond

Highly oriented (0 0 0 2) AlN is a requisite for piezoelectric films and thus, to transfer the electrical signal efficiently into a SAW propagating on the diamond. In particular, the narrower the full width at half maximum (FWHM) of the (0 0 0 2)-rocking curve the better; according to our and previous results on sputtered AlN [30], it should typically be smaller than 4° in order to yield a strong piezoelectric response. Moreover, as described elsewhere [12,25], the surface roughness hinders the synthesis of *c*-axis oriented AlN. In order to overcome this difficulty, the nucleation side of a free-standing NCD layer is proposed here. Fig. 3 shows the height AFM micrographs of the top surface (a) and the bottom nucleation side (b) of NCD films. The average surface roughness improves from 262 nm root mean square (rms) for the rough NCD face, to 2 nm rms for the free-standing NCD presented here.

XRD analyses, in the $\theta/2\theta$ and rocking curve scan mode, were carried out to determine the crystallinity and orientation of the films. AlN films with thickness ranging from 150 nm to 1.2 µm were deposited on free-standing NCD. Fig. 4 shows the XRD spectra in the $\theta/2\theta$ scan mode of these AlN films. The AlN films are highly oriented in the (0 0 0 2) direction, i.e. perpendicular to the substrate surface. The intensity of this peak is reduced when reducing the film thickness. This behaviour is also observed on the AlN sputtered on polished MCD [12]. As it is shown in [31], a similar behaviour occurs when the AlN is sputtered on free-standing NCD; the AlN layer grows along the (0 0 0 2) direction but showing a two-domain structure with two in-plane orientations. This explains the co-existence of the main peak at 36.16° and the one at 35.3° corresponding to the (0 0 0 2) direction. For the substrate grains, only the peak corresponding to the diamond (1 1 1) direction appears on the spectra.

In order to evaluate the crystal quality and hence the piezoelectricity of these AlN films grown on free-standing NCD, the (0 0 0 2)-rocking curve was analysed. Fig. 5 shows the FWHM of the rocking curve peaks obtained in several films, which is reduced for thicker AlN layers, down to 4° for the 1200 m- and 600 nm-thick layers.

3.2. AlN thickness influence on the SAW response

Fig. 6 shows the reflection coefficient of a SAW resonator of $\lambda = 800$ nm fabricated on 1200 nm-, 600 nm-, 300 nm- and 150 nm-thick AlN sputtered on the free-standing NCD (solid line) and on

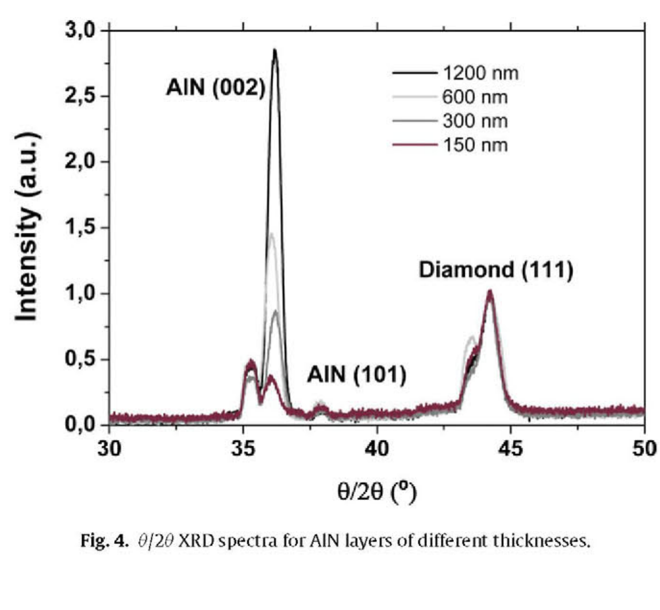

Fig. 4. $\theta/2\theta$ XRD spectra for AlN layers of different thicknesses.

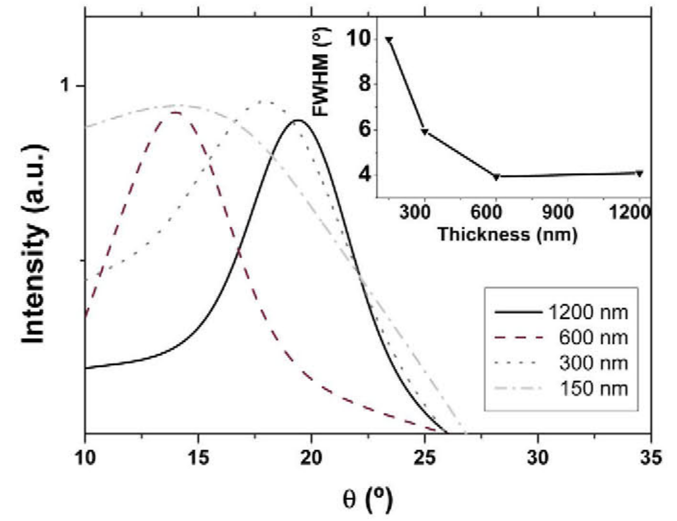

Fig. 5. Rocking curves on the 0 0 0 2 direction of different thickness of AlN sputtered on free-standing NCD. The inset shows the FWHM of the peak.

polished MCD (dash line). The film-thickness-to-wavelength ratio (H/λ) are $kH = 2\pi H/\lambda = 9.42$, 4.71, 2.36 and 1.18, respectively. As it was presented elsewhere [28], the electromechanical coupling coefficient (K^2) and the propagation velocity of the acoustic waves depend on the H/λ. By decreasing the piezoelectric film thickness (small H/λ), its crystal quality and orientation decrease, thus the piezoelectricity worsens. However, the SAW extends far into the diamond substrate, and the phase velocity (v) approaches the large

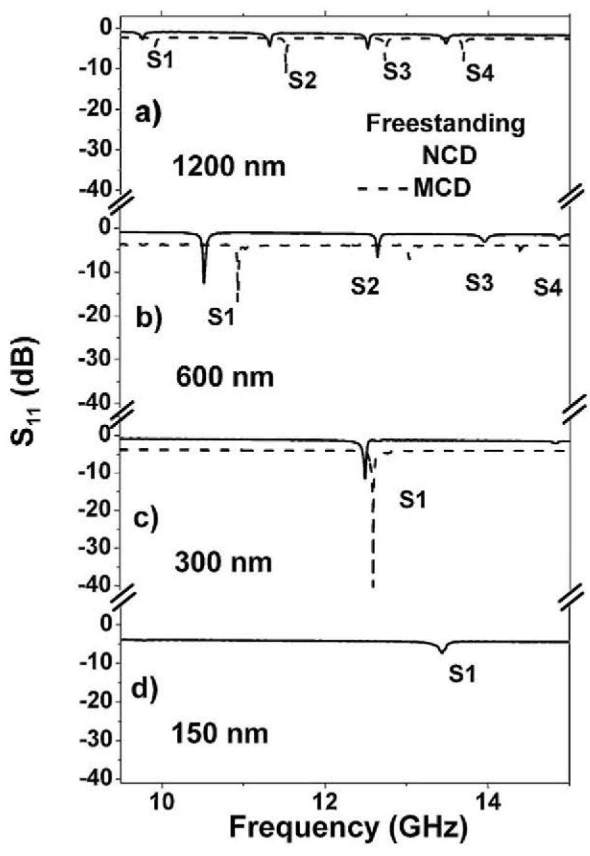

Fig. 6. Reflection coefficient (S_{11}) spectra for identical ($\lambda = 800$ nm) one-port SAW resonators on (a) 1200 nm-, (b) 600 nm-, (c) 300 nm-, and (d) 150 nm-thick AlN films on free-standing nanocrystalline diamond and polished microcrystalline diamond. R and S_I denote the Rayleigh and Sezawa modes, respectively.

velocity value of the bare substrate. When the AlN thickness is higher than one period (800 nm), the crystal quality improves but the SAW concentrates in the piezoelectric film region, thus K^2 is large but v is much smaller. This effect can be observed on Fig. 6. For the device fabricated on free-standing NCD, the v obtained for the first Sezawa mode, S_1, increases from 7816 m/s for the 1200 nm-thick AlN, to 8425 m/s for the 600 nm-thick AlN, 9995 m/s for the 300 nm-thick AlN and 10,751 m/s for the 150 nm-thick AlN. In addition, the out-of-band rejection and quality factor Q also improve when the AlN thickness is reduced until 300 nm. Furthermore, for the 150 nm-thick AlN device, the response is degraded due to the fact that the crystal quality also worsens for this narrow film thickness range, a behaviour common to all the observed modes. The difference observed in all frequency modes in the devices processed on polished MCD, when compared to those in free-standing NCD, is due to differences in the AlN thickness.

The choice of the piezoelectric film thickness implies a compromise between the frequency and the intensity of the targeted resonances. For the structure presented in this work, the optimal AlN film thickness is 300 nm. The principal resonance for the SAW one port resonator fabricated on this film is 12.5 GHz, corresponding to the first Sezawa mode, and it shows a Q factor of 643 and an out of band rejection of 11 dB. The Q and out of band rejection are better for the device processed on AlN/MCD due to the better quality of the AlN film [28]. Nevertheless, the results obtained for the devices processed on AlN/free-standing NCD are the highest obtained so far on nanocrystalline diamond [32]. Moreover, the achieved frequency is the highest obtained among other free-standing NCD structures [22–24]. In addition, these results are in agreement with the ones obtained before [28], and confirm that the AlN/free-standing NCD structure is an alternative to the more expensive polished MCD for the processing of high performance and high frequency SAW devices.

3.3. SAW pressure sensor

The free-standing structure presented here enables the exploitation of the SAW devices fabricated on top of it as pressure sensors. The SAW resonators processed on the optimal AlN thickness obtained before (300 nm) were characterized in a range of 1–4 bars in steps of 0.5 bar at room temperature i.e. the frequency was swept in the 10.2–11.2 GHz range while the pressure was kept constant at 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 bar. The pressure was applied using compressed nitrogen as shown previously in Fig. 2, in positive (from 2 until 5 bar) and negative (from 5 until 2 bar) variation modes. As the pressure underneath the hole was kept constant at atmospheric pressure, the real range applied to the membrane in both directions was between 1 and 4 bar. Fig. 7 shows the first Sezawa mode of the reflection coefficient and its frequency shift due to the pressure variation for positive (Fig. 7a) and negative (Fig. 7b) directions. Fig. 8 shows the behaviour of the frequency shift of the device for different pressures also for positive (up) and negative (down) direction. The small differences between both behaviours are related to hysteresis effects inherent to piezoelectric phenomena (that lead to a decrease of the piezoelectric coefficient d_{33} [33,34]) and also due to the manometer error (±0.1 bar as shown in the figure). A non linear behaviour was observed when a pressure variation in the range $\Delta P = 0$–1 bar was applied. Differences between air and N_2 density and mainly the dimensions of the AlN/diamond membrane, as well as the location SAW device on top of it are considered to be the most critical factors determining this kind of behaviour. Moreover, a temperature variation, in this case caused by the N_2 flow, leads to a shift of the centre frequency as studied elsewhere [13]. A dual oscillator temperature compensation technique, using two SAW resonators in close proximity [35], or two devices with and without the membrane in the same

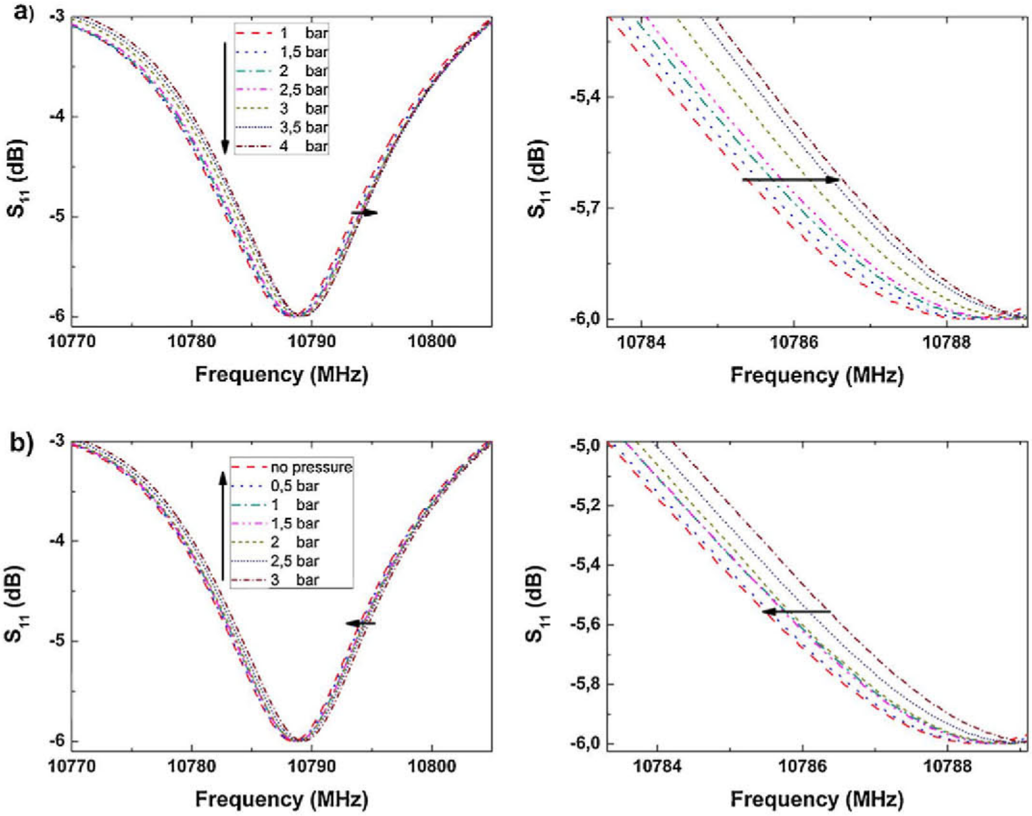

Fig. 7. Frequency response of AlN/diamond SAW resonators under: (a) upwards pressure variation (left), zoom in (right); (b) downwards pressure variation (left), zoom in (right).

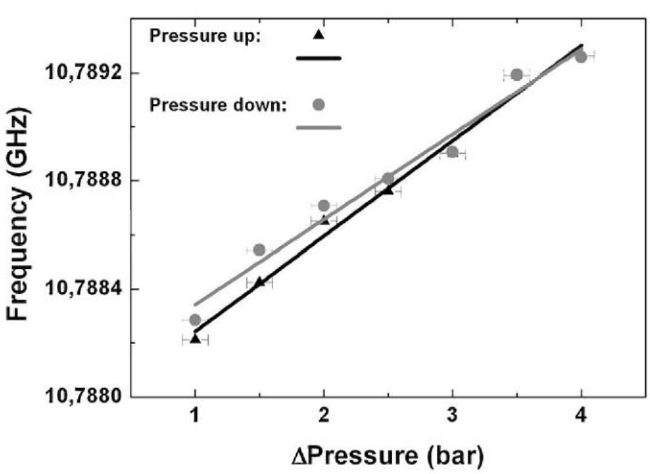

Fig. 8. Experimental frequency shift when pressure is applied in positive (up) and negative (down) direction (symbols) and linear regression for both (solid line).

substrate (together with an optimal membrane size and SAW device location on top of it), could be used in order to overcome the non linear behaviour and eliminate those temperature effects.

The sensitivity obtained by linear fitting is 0.33 ± 0.02 MHz/bar as shown in Fig. 8. The high sensitivity obtained here can utterly be tuned up by optimizing the device location within the diaphragm [35,36].

4. Conclusions

A free-standing NCD structure is proposed as an alternative to polished MCD in order to sputter AlN and fabricate SAW devices for high frequency sensor applications. The influence of the piezoelectric film thickness on the SAW response has been analysed, and the optimum AlN layer thickness in our experiments results in 300 nm. The main resonance of a device obtained using this film with an IDT period of 800 nm is at 12.5 GHz, corresponding to the first Sezawa mode, and it shows a Q factor of 643 and an out-of-band rejection of 11 dB. As the membrane of the structure is free to move, these devices may be used as pressure sensors, which have shown a sensitivity of 0.33 MHz/bar.

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