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To cite this version:

Thierry Aubert, Omar Elmazria, Badreddine Assouar, Eloi Blampain, Ahmad Hamdan, et al.. Investigations on AlN/Sapphire piezoelectric bilayer structure for high-temperature SAW applications. IEEE Transactions on Ultrasonics, Ferroelectrics and Frequency Control, 2012, 59 (5), pp.999 - 1005. 10.1109/TUFFc.2012.2285 . hal-00783993

HAL Id: hal-00783993

https://hal.science/hal-00783993v1

Submitted on 20 Feb 2023

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Investigations on AlN/Sapphire Piezoelectric Bilayer Structure for High-Temperature SAW Applications

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Abstract—**This paper explores the possibility to use AlN/Sapphire piezoelectric bilayer structure for high-temperature surface acoustic wave (SAW) applications. In order to determine the temperature stability of AlN, homemade AlN/Sapphire samples are annealed in air atmosphere for 2 to 20 hours at temperatures going from 700 to 1000°C. *Ex situ* X-ray diffraction measurements reveal that the microstructure of the thin film is not impacted below 1000°C. Otherwise, ellipsometry and secondary ion mass spectroscopy investigations attest that AlN/Sapphire is reliable up to 700°C. Beyond this temperature, both methods evidence ongoing surface oxidation of AlN. Furthermore, Pt/Ta and Al interdigital transducers are patterned at the surface of the AlN film. The resulting SAW devices are characterized up to 500°C and 300°C respectively, showing reliable frequency response and a large, quasi-constant, temperature sensitivity, with a first order temperature coefficient of frequency around -75 ppm/°C. Besides, between room temperature and 300°C, both electromechanical coupling coefficient K^2 and propagation losses increase, so that the evolution of delay lines insertion losses with temperature strongly depends on the length of the propagation path.**

Keywords-high temperature; AlN; sapphire ; SAW; langasite

I. INTRODUCTION

Surface acoustic wave (SAW) devices are key components of telecommunication systems for more than thirty years. They also constitute a very promising solution for sensors applications, as they are very sensitive to the external environment, while being passive. This last property is particularly interesting in

harsh environments, such as high temperatures up to 1000°C, where this technology offers the possibility to achieve wireless sensors [1].

The choice of the piezoelectric substrate, which is an essential part of SAW devices, is critical to realize such sensors. Indeed, there are very few piezoelectric materials capable of withstanding high temperatures above 500°C [2]. The most promising results have been obtained so far with langasite ($La_3Ga_5SiO_{14}$ or LGS). In particular, Pereira da Cunha *et al.* have successfully tested this material for 5½ months at 800°C [3]. Moreover, Hornsteiner *et al.* have measured a SAW signal on an LGS-based device up to 1085°C [4]. Last but not least, good quality LGS-wafers are commercially available. However, LGS has also some drawbacks. In particular, its acoustic propagation losses increase drastically with frequency and temperature, which restrains its use in ISM wireless mode to the 434 MHz-band, excluding the more promising 2.45 GHz one [1]. Furthermore, several authors have observed at high temperature a significant increase in the surface roughness of LGS substrates, which onset temperature depends on the gas environment [5]-[7]. This phenomenon is related to chemical transformations happening in the vicinity of the surface of LGS crystals. Thus, Bardong *et al.* observed Ga losses from 650°C under light vacuum environment (pressure = 0.4 mbar), using energy dispersive X-ray spectroscopy [5]. With the help of secondary ion mass spectroscopy (SIMS) measurements, Shulz *et al.* have quantified those losses in air atmosphere. The quantity of Ga atoms that would leave the crystal through its surface during a period of 100 days at 750°C is in the order of 10^{16} per centimeter square [7]. These results show that it is mandatory to find new piezoelectric materials which could overcome the limitations of langasite for ultra-high temperature (over 800°C) and ultra-high frequency (over 1 GHz) SAW applications.

Aluminium nitride (AlN) thin films could be one of these materials. It has been identified as a potential candidate for high-temperature SAW applications more than 10 years ago [4], [8]. Indeed, this material is chemically stable up to 1040°C under ultra-high-vacuum conditions [9], 1200°C in N_2/H_2 mixture [10] and is expected to oxidize in air atmosphere in the 800°C-range [11]. Furthermore, showing the highest SAW velocity among the piezoelectric materials, it is particularly suited to carry out devices operating at high

frequencies such as 2.45 GHz [12]. Eventually, some high-quality AlN films show acoustic propagation losses well lower than those of LGS (Tab. I).

Despite all these potentialities, AlN has hardly been experimentally studied as piezoelectric material for high-temperature SAW applications. We have recently demonstrated that IDT(Pt/Ta)/AlN/Sapphire SAW structure withstands temperatures as high as 900°C for short-time exposure of 30 minutes [13]. Now, the goal of the present study is to investigate the reliability of the AlN/Sapphire bilayer structure for longer time under such environment.

TAB. I. COMPARISON BETWEEN SAW PROPERTIES OF LGS AND AlN/SAPPHIRE BILAYER STRUCTURE [2]

Material	SAW velocity (m/s)	Electromechanical coupling coefficient K^2 (%)	Acoustic propagation losses at 1 GHz (mdB/λ)
Langasite (0°, 138.5°, 26.6°)	2700	0.4	4
(002)AlN/(0001)Sapphire ($kh_{AlN} = 2$)	5600	0.3	0.7

II. EXPERIMENTAL

1.3 µm thick (002) AlN thin films were epitaxially grown by reactive magnetron sputtering onto 3" (0001) sapphire wafers [14]. X-ray diffraction (XRD) measurements as well as transmission electron microscopy revealed that the (002) AlN films are stress-free and highly-textured out of the plane, with a full width at half-maximum (FWHM) of the (002) rocking-curve (RC) as low as 0.26°. Both methods also evidenced that these films are textured in the plane as well. The value of the grain size, deducted from XRD measurements and atomic force microscopy, is close to 40 nm. The latter method also gave access to the surface roughness of the films which value, equal to 8 Å (root mean square), is suitable for SAW applications.

To study the temperature stability of the AlN/Sapphire structure in air atmosphere, the 3" wafers were cut in small pieces (20 x 20 mm). The resulting samples were annealed at temperatures between 700 and 1000°C, for periods going from 2 to 20 hours in a tubular furnace with open ends. The effects of high-

temperature exposure were studied by doing XRD (θ-2θ and rocking-curve scan modes, PANalytical X'Pert Pro MRD), ellipsometry (Horiba Jobin Yvon Uvisel) and SIMS (primary ions: Cs^+, Cameca IMS 7F) measurements.

Furthermore, to perform interdigital transducers (IDTs) withstanding high-temperature conditions, 10 nm thick tantalum as adhesion layer and 100 nm thick platinum films as electrodes, were deposited by sputtering method on some AlN/Sapphire substrates [15]. SAW delay lines were then fabricated by photolithography and ion beam etching. The devices were constituted by two identical IDTs, with 50 pairs of fingers and a wavelength λ fixed to 16 µm. The IDT center spacing, acoustic aperture, and finger width to space ratio were 175λ, 70λ, and 1:2, respectively. The SAW propagation path was along the Sapphire X-direction. These devices were electrically characterized up to 500°C in air atmosphere using a Network Analyzer (Agilent PNA 5230A, Santa Clara, CA) and RF prober station (Signatone S-1160, Gilroy, CA) equipped with Signatone thermal probing system S-1060 Series so that it can withstand temperatures up to 600°C. However, we restricted our characterization to 500 °C due to the limitation imposed by the use of cooled RF probes (Z-Probe Suss Microtech). Finally, to get the evolution of the electromechanical properties of the AlN/Sapphire bilayer structure with temperature, conventional 100 nm thick Al IDTs were used to minimize the influence of the electrodes on the devices response. These measurements were conducted up to 300°C, considering the electromigration phenomena that aluminium undergoes from this temperature [16]. Namely, the propagation losses behavior was studied from delay lines with the same design as the one described above, except that the IDT center spacing was comprised between 55λ and 110λ in that case. Some synchronous resonators were also fabricated to access the electromechanical coupling coefficient K^2 of the structure, *via* the following equation [17]:

where G_o is the conductance value at the central frequency f_0, C the capacitance between IDT finger pair and N the number of finger pairs in the IDT. To do so, the wavelength was reduced to 6 µm so that the

SAW is more confined in the piezoelectric film, leading to a higher value of K^2, and by the way to a better accuracy in the determination of its evolution.

III. Results and Discussion

A. Temperature stability of AlN in air atmosphere

On the figures 1 and 2, the RC of the XRD (002) AlN peak and the c-lattice parameter of the AlN films are respectively plotted as function of the annealing duration for temperatures going from 700 to 1000°C. The c-lattice parameter was deduced via XRD measurements, from the Bragg angle θ of the (002) AlN peak, using the Bragg formula [18]. Its evolution is related to the one of the stress in the film. One can see on these two figures that neither the texture nor the stress of the AlN layer is modified by high-temperature exposure, provided the temperature is lower than 1000°C. These results evidence the physical stability of the AlN/Sapphire bilayer structure under such environments. On the other side, the intensity of the XRD (002) AlN peak decreases all along the annealing process. Figure 3 illustrates this phenomenon in the case of a 2h-long annealing at 1000°C. As this cannot be related to a decline of the film crystalline quality (texture or grain size), its origin has to be found in the shrinking of the AlN amount, related to its progressive oxidation from the surface [11].

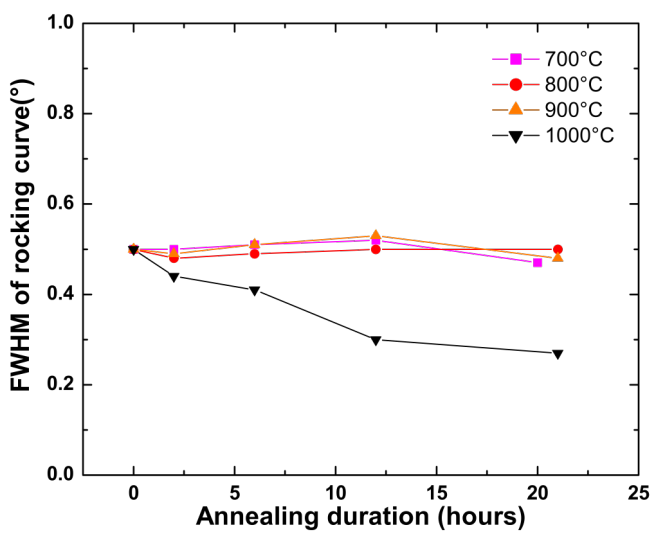

Fig. 1. FWHM of the rocking-curve of AlN (002) as a function of the annealing duration

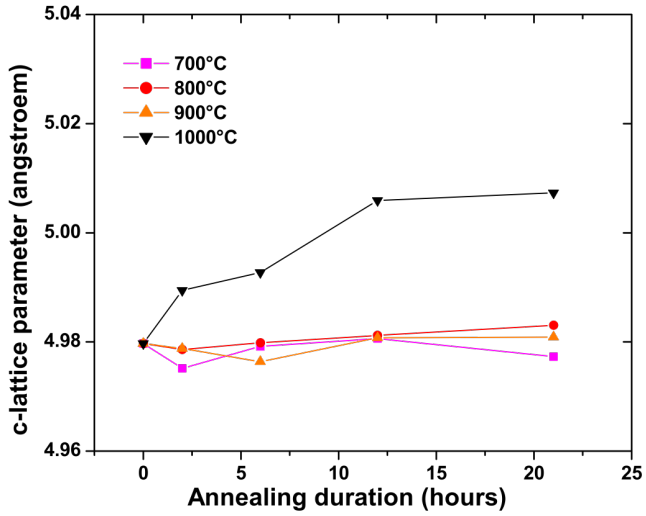

Fig. 2. c-lattice parameter of the AlN film as a function of the annealing duration

However, XRD does not allow to evidence the formation of an aluminium oxide overlayer below 1000°C, likely because this layer is amorphous if the annealing temperature is too low [19]. Figure 4 summarizes the evolution of the intensity of the (002) AlN peak with time for temperatures going from 700 to 1000°C. As expected, the kinetics of this oxidation reaction strongly depends on the annealing temperature. It follows an exponential law which leads to an asymptotic regime in a matter of hours. This kind of behavior is related to the fact that the diffusion of the oxygen molecules through the oxide overlayer, responsible for further oxidation, is getting more and more difficult as the thickness of the latter increases [20]. After 20h of annealing, the asymptotic regime is reached except for the highest temperature in this test, i.e. 1000°C. One can observe that after such duration, the intensity of the (002) AlN peak is less than 10% of its initial value at 1000°C, around 50% at 900°C and more than 90% at 800 and 700°C. As this intensity is directly related to the AlN amount [21], it means that the order of magnitude of the oxide overlayer thickness is respectively of 1 µm, 500 nm and some tens of nanometers. Thus, for the lower temperatures, the variations in thickness of the oxide overlayer are likely too small to be correctly quantified by *ex situ* XRD measurements, as attests the important dispersion of the results at 700°C visible on figure 4.

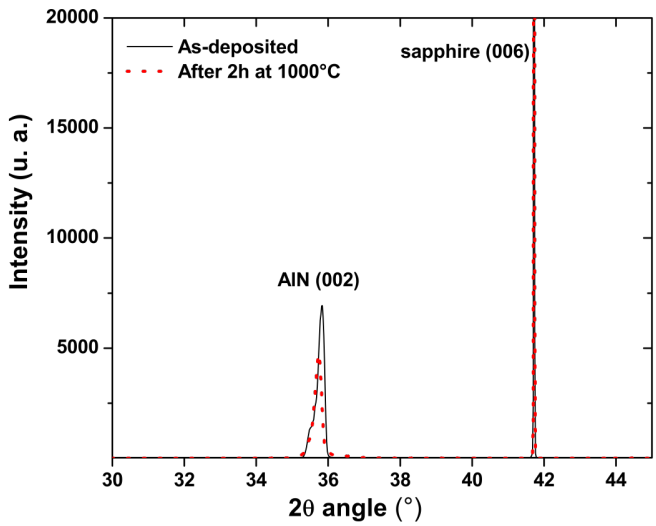

Fig. 3. θ-2θ XRD scan mode of as-prepared and 2h annealed AlN/Sapphire samples

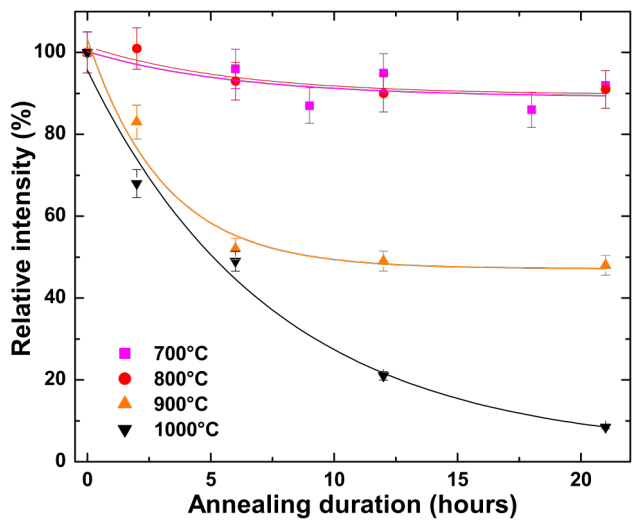

Fig. 4. Relative intensity of the (002) XRD AlN peak as a function of the annealing duration

Then, more sensitive methods, such as SIMS or ellipsometry, were used to overcome this limitation. As AlN and sapphire are both excellent insulators, it was necessary for performing SIMS measurements to deposit after annealing an additional 50 nm thick conductive Pt layer at the surface to minimize charging effects. The intensity of the signals related to Pt^+ and CsO^- secondary ions are plotted versus etching time on figure 5, in the case of a sample annealed for 2h at 700°C. This graph clearly evidences the presence of an oxide layer just below the conductive Pt film. On figure 6 can be seen the CsO^- signal for several samples (Pt^+ signals have been removed for better clarity). It is possible to deduct the qualitative evolution

of the oxide layer thickness from the one of the FWHM of this SIMS peak [22]. One can observe the presence of a thin native oxide layer at the surface of the as-deposited AlN films. Its thickness slightly increases after 2 hours of annealing at 700 and 800°C but remains close to that of the native one, which is not the case at higher temperatures, thereby confirming XRD measurements. The most important result appears after 20 hours of annealing. Whereas the thickness of the oxide layer continues to slightly increase at 800°C, it seems that the initial minor oxidation passivates AlN films from further aggression at 700°C.

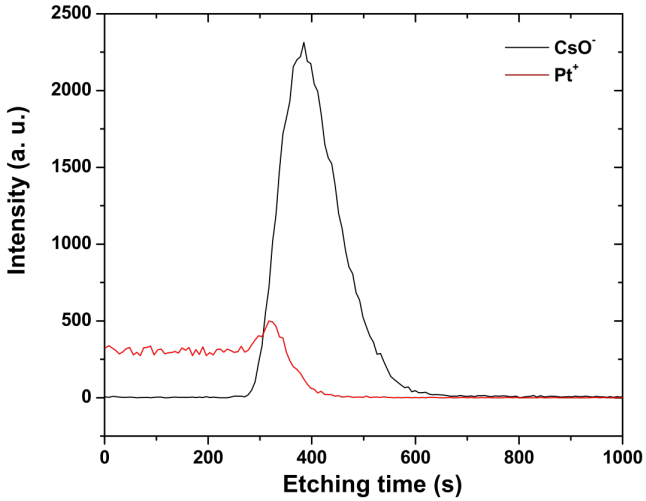

Fig. 5. SIMS depth profile signal of an AlN/Sapphire sample after a 2h long annealing process. A thin conductive Pt layer was sputtered at the surface to reduce charging effects.

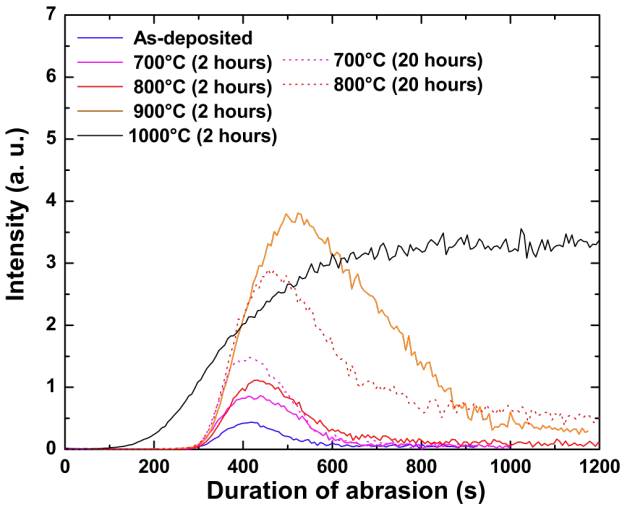

Fig. 6. Comparison of SIMS depth profile signals related to oxygen coming from different samples

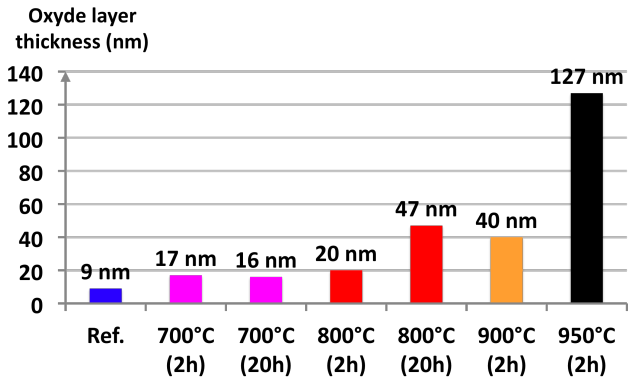

Fig. 7. Thickness (±2 nm) of the oxide overlayer determined by ellipsometry for different samples

A more quantitative estimation of the oxide layer thickness was obtained by performing ellipsometric measurements. To do so, one has to make experimental optical parameters and calculated ones from dedicated softwares (in our case, DeltaPsi2 from Horiba Jobin Yvon) fit together. The accuracy of the method depends mainly on the correct modeling of the sample structure. In our case, reliable results with high correlation coefficients were obtained by simply describing annealed samples as composed by 3 layers one above the other: amorphous Al_2O_3/AlN/sapphire. The oxide layer thickness deducted from this method for the same samples as the ones investigated by SIMS can be seen on figure 7. The correlation between the results given by both methods is very good. In particular, the main information provided by SIMS method is fully confirmed by ellipsometry: the 10 nm thick native oxide layer is growing a little bit by 7-8 nm during the first 2h of annealing at 700°C, and then is stable during longer annealing process at this temperature. As this stability is not observed at higher temperatures, 700°C seems the upper limit regarding the stability of AlN/Sapphire at high temperature in air atmosphere.

B. SAW measurements

The experimental frequency-temperature law of the Pt/Ta/AlN/Sapphire SAW delay lines is plotted on figure 8. The investigated structure is characterized by a large and quasi-constant sensitivity to temperature, with first-order temperature coefficient of frequency (TCF) values going from -68 ppm/°C at 20°C to -80 ppm/°C at 500°C, making it well suited for temperature sensor applications. Note that as the relative thickness h/λ of the AlN layer (where h is the thickness of the AlN film) is quite limited here, with

a value smaller than 0.1, the SAW is mainly located in the sapphire substrate. Thus, this thermal behavior can be mainly attributed to the latter. Figure 9 shows the time-gated frequency response (S_{21} magnitude) of the same delay line at different temperatures between the ambient and 500°C. The main observation is the quasi-constant increase in the insertion losses at a rate close to 1 dB/100°C.

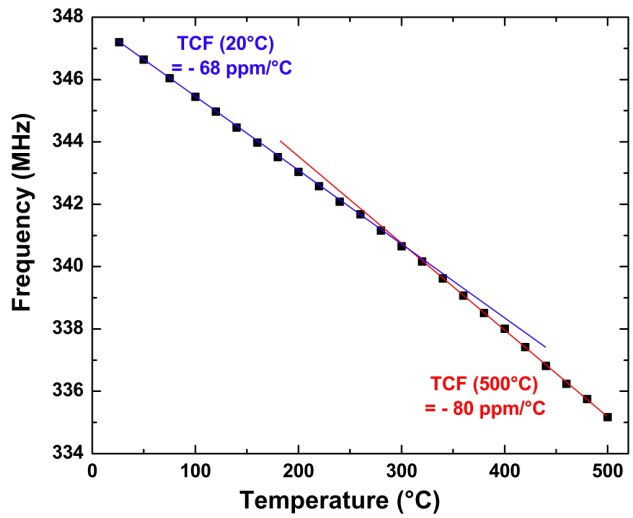

Fig. 8. Temperature dependence of the synchronous frequency of the delay line

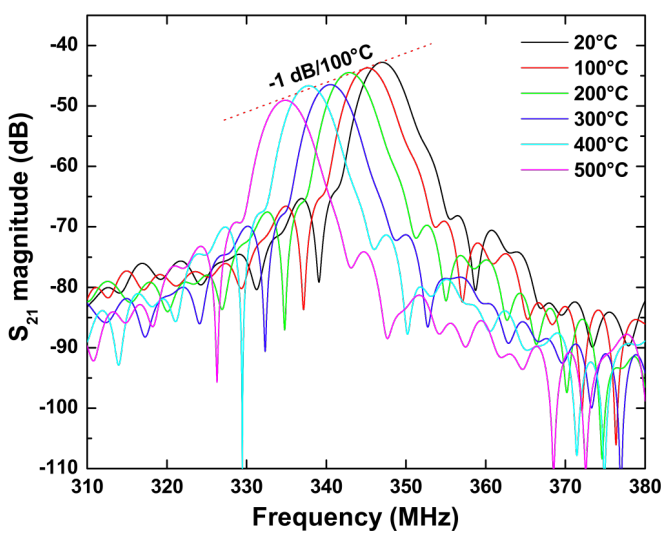

Fig.9. $|S_{21}|$ signal of IDT (Pt/Ta)/AlN/Sapphire SAW delay line from 20 to 500°C

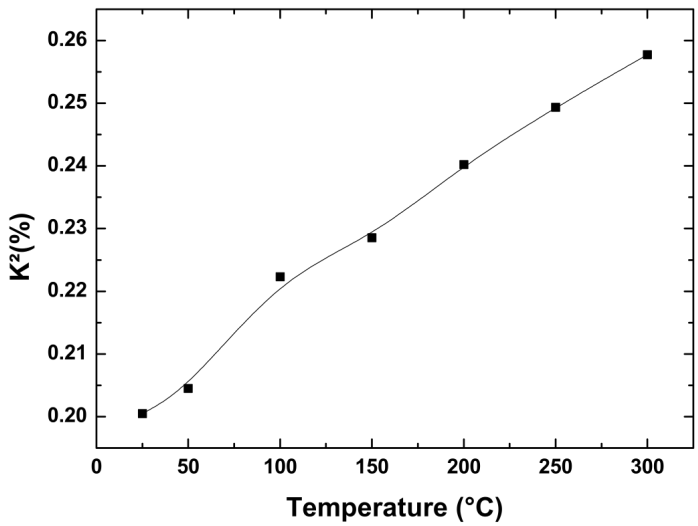

Fig. 10. Evolution of K^2 of AlN/Sapphire structure with temperature

As expected, SAW devices with Al IDTs helped us to clarify the origin of this slight deterioration. The evolution with temperature of the K^2 of the structure, deduced from the characterization of the synchronous resonators, is plotted on figure 10. K^2 increases from 0.2 % at room temperature to 0.26 % at 300°C, which means an expansion of 30 %.

Regarding delay lines, such an improvement should result in a decrease of insertion losses with temperature. However, as it can be seen on figure 11, the evolution of the latter strongly depends on the length of the propagation path. In fact, whereas a significant improvement (insertion losses diminution) can be seen for the shortest delay line between room temperature and 300°C, one can observe a relative stability for intermediate ones and a slight deterioration in the case of the longer one. This means that the propagation losses also increase when the temperature is rising and that this phenomenon can counteract the expansion of K^2 if the delay line is long enough. In the case of the Pt/Ta/AlN/Sapphire delay line investigated up to 500°C, the propagation length was even more important with a value of 125λ, which explains this increase of the insertion losses at a non-negligible rate of 1 dB/100°C.

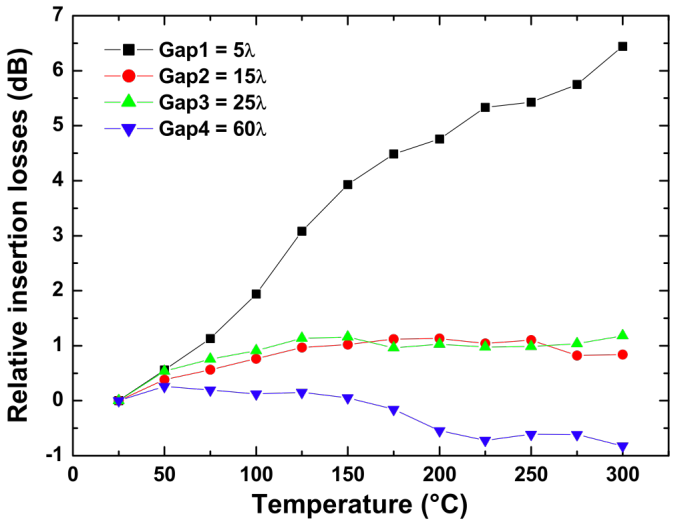

Fig. 11. Delay line length influence on the insertion losses evolution with temperature

CONCLUSION AND OUTLOOKS

The results obtained in the first part of this study suggest that the AlN/Sapphire bilayer structure can be used as piezoelectric material for SAW sensors operating in air atmosphere up to 700°C, as the oxidation process seems quite limited, which is not the case at higher temperatures. This statement will have to be confirmed by *in situ* SAW measurements conducted up to this temperature, in order to determine the impact of this very thin additional oxide overlayer. Anyway, better stability of AlN is expected in environments less aggressive than air, such as those containing small amounts of oxygen, where temperatures as high as 1000°C could be likely reached [10]. Moreover, the AlN/Sapphire bilayer structure could have an enormous hidden potential for high-temperature SAW applications, as the IDTs could be placed below the AlN film. Thus, AlN/IDT/AlN/Sapphire or AlN/IDT/Sapphire configurations are both promising solution leading to protect both IDTs from agglomeration phenomena and active piezoelectric area of AlN from oxidation, or other chemical aggression, not to mention that this would significantly improve the electromechanical coupling coefficient K^2 of the structure [12]. The results presented in the second part of this paper, dedicated to SAW measurements on devices working around 350 MHz, show that the AlN/Sapphire structure is particularly suited for temperature sensor applications

as it shows a large, quasi-constant, temperature sensitivity up to at least 500°C. We also observed that both K^2 and propagation losses are increasing with temperature so that the evolution of the insertion losses of delay lines based on this structure depends mainly on their length. Finally, though this study was conducted at relatively low frequency (around 350 MHz), these results are also encouraging for high-frequency applications (i.e. mainly in the ISM band at 2.45 GHz), for which AlN/Sapphire structure shows great interest over LGS. In that case, if the AlN thickness was kept around 1.3 µm, its relative value h/λ would reach 0.6, leading to a wave more confined into the piezoelectric film and thus to an electromechanical coupling coefficient K^2 likely not too far from the maximum value for this structure, i.e. 0.3%. Concerning the TCF, such an increase in the relative thickness of AlN should result in a reduction of its value to 20-30 ppm/°C at room temperature [23], which is still suitable for temperature sensor applications. On the other side, it is not possible to predict if in that case, the frequency-temperature law will still show a quasi-linear behavior in the 20-500°C range as the thermal behavior of pure AlN is not known. At last, a larger operating frequency means as usual more propagation losses. But, as Uehara *et al.* showed, if the AlN film is of high quality with very low roughness (some Å) and low dislocation density, then the AlN/Sapphire structure could show well-contained propagation losses, even lower than those of lithium niobate [24]. The only remaining question will be then the quantitative evolution of these propagation losses with temperature.

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