Reliability of AlN/Sapphire bilayer structure for high-temperature SAW applications

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Abstract—**This paper explores the possibility to use AlN/sapphire bilayer structure as piezoelectric material for high-temperature SAW applications in air atmosphere. In order to verify the temperature stability of AlN, homemade AlN/Sapphire samples were annealed during 2 to 20 hours, at temperatures going from 700 to 1000°C, before being characterized by *ex situ* X-ray diffraction (XRD), ellipsometry and secondary ion mass spectroscopy (SIMS). *Ex situ* SAW measurements were used to transfer the obtained results to the SAW field, while *in situ* investigations were conducted up to 500°C to observe the effect of temperature on the SAW signal.**

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 remotely requestable and 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 one of the challenges to face to realize such sensors. Indeed, there are very few piezoelectric materials capable of withstanding high temperatures above 500°C [2]. Up to now, the most promising results have been obtained 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 is also characterized by some weaknesses. In particular, its acoustic propagation losses increase drastically with frequency and temperature, which restrains its use in wireless mode to the 434 MHz-ISM band, excluding the more promising 2.45 GHz one [5]. Furthermore, the high-temperature properties of LGS have been mainly investigated in air atmosphere, but its behavior is unknown in most industrial gas environments (NH_x, SO_2, H_2, etc). For these two reasons, alternatives to LGS would be of great interest.

Aluminium nitride (AlN) thin films could be one of these. It has been identified as a potential candidate for high-temperature SAW applications more than 10 years ago [4,6]. Indeed, this material is chemically stable up to 1040°C in vacuum [7], 1200°C in N_2/H_2 mixture [8] and is expected to oxidize in air atmosphere in the 800°C-range [9]. 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 [10]. Eventually, some high-quality AlN films show acoustic propagation losses well lower than those of LGS (Tab. 1).

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 [11]. Now, the goal of the present study is to investigate the reliability of the AlN/Sapphire bilayer structure for long time applications at high temperatures, in order to verify its potential as an alternative structure to the LGS family, especially for high frequency applications.

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

	SAW velocity (m/s)	**Electromechanical coupling coefficient K^2 (%)**	**Acoustic propagation losses at 1 GHz (mdB/λ)**
Langasite (0°,138.5°,26.6°)	2700	0.44	0.7
(002)AlN/(0001)sapphire	5700	0.3	4

II. EXPERIMENTAL

1,3 µm-thick hetero-epitaxial (002) AlN thin films were deposited by reactive magnetron sputtering onto (0001) sapphire substrates. The microstructure of the films was investigated by X-ray diffraction (XRD) and transmission electron microscopy (TEM), while atomic force microscopy (AFM) gave access to its morphology.

To study the temperature stability of AlN in air atmosphere, the AlN/Sapphire samples were annealed at temperatures between 700 and 1000°C, for periods going from 2 to 20 hours. The effects of high-temperature exposure were then studied by XRD, ellipsometry and secondary ion mass spectroscopy (SIMS). To confirm and transfer these results to the SAW field, the SAW signals coming from delay lines with aluminium electrodes, operating at 560 and 710 MHz, performed on both as-deposited and annealed samples, were compared.

Pt/Ta IDTs (which means 10 nm of tantalum as adhesion layer and 100 nm of platinum as electrode [12]) were also implemented in order to achieve *in situ* SAW measurements with a probe station, up to 500°C.

III. RESULTS AND DISCUSSION

A. Hetero-epitaxial AlN thin films on sapphire

XRD measurements and TEM revealed that the (002) AlN films are stress-free, while being 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°, as well as in the plane (Fig. 1). The value of the grain size, obtained from XRD measurements via the Scherrer formula [13], confirmed by AFM, is close to 30 nm. The latter also give the roughness of the films which value (Rms), equal to 8Å, is suitable for SAW applications.

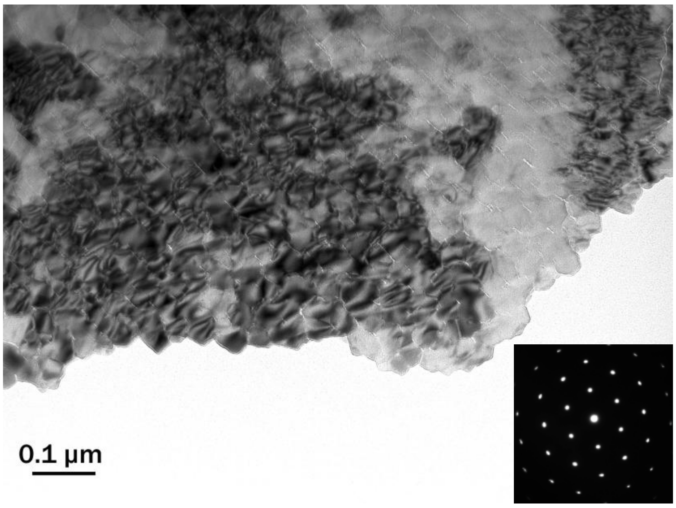

Figure 1. TEM image and SAED pattern of the optimized AlN films

B. Temperature stability of AlN in air atmosphere

XRD measurements point out that, up to 900°C, neither the stress, nor the grain size nor the texture of the films are modified by the high-temperature exposure (Fig. 2). On the other side, the intensity of the (002) AlN peak promptly decrease during the annealing process, from 900°C (Fig. 3). As this phenomenon cannot be related, at least at 900°C, to a modification of the microstructure of the films, its origin has to be found in the shrinking of the AlN amount, associated to a strong oxidation. The results obtained at 700 and 800°C are more uncertain, the formed oxide overlayer being likely too thin to be precisely detected by *ex situ* XRD measurements. Thus, more sensitive methods, such as SIMS or ellipsometry were used to provide a clearer picture of the situation at these lower temperatures.

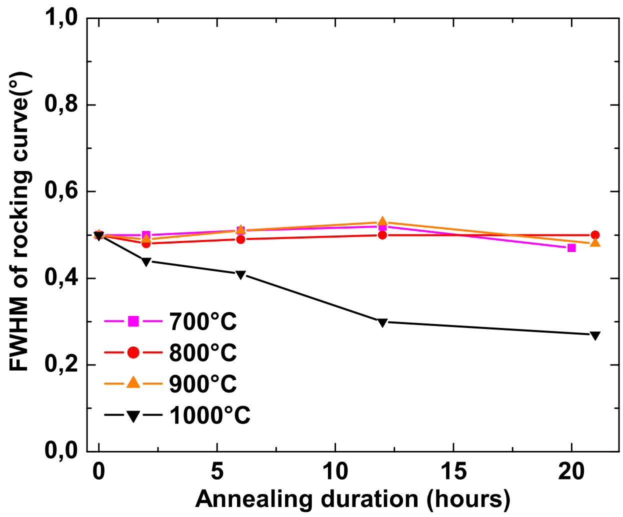

Figure 2. FWHM of the RC (002) as a function of the annealing duration

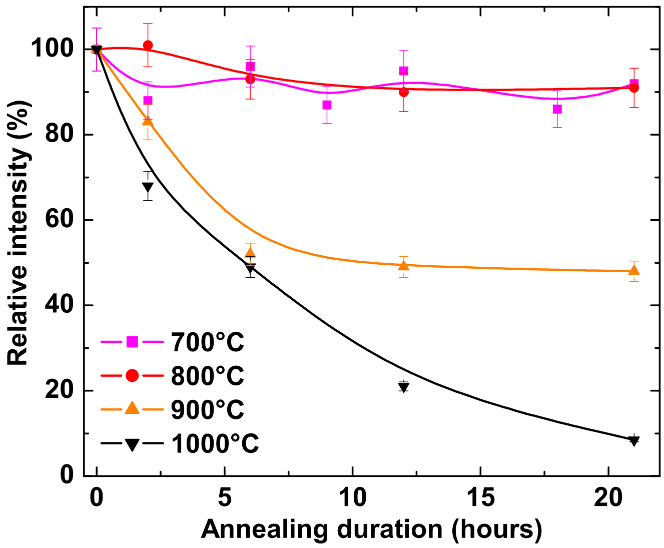

Figure 3. Intensity of the (002) XRD AlN peak as a function of the annealing duration

The results given by the latter perfectly converge (Fig. 4&5). They both highlight the presence of a thin (≈ 10 nm-thick) native oxide layer at the surface of the AlN films. This thickness slightly increases after 2 hours of annealing at 700 and 800°C but remains comparable 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, reaching approximately 50 nm at the end of the process, it seems that the initial oxidation passivates AlN films from further aggression at 700°C.

In order to validate this key result in the SAW field, we compared the $|S_{21}|$ signal given by delay lines performed with aluminium IDTs on both as-deposited and 700°C-annealed samples. This special procedure has been followed to overcome the limitation of our *in situ* facilities which cannot be used over 600°C. One can observe on Fig. 6 that the technological dispersion makes the interpretation of the results quite difficult. However, it seems that no systematic frequency shift related to the 700°C-annealing process of the AlN/Sapphire samples can be detected. This is a fundamental result in the context of the use of AlN in SAW sensors. On the other side, insertion losses seem increase slightly, probably because of the initial growth of the thin oxide overlayer. Note however that some devices carried out on annealed samples show the same losses as those prepared on as-deposited samples.

C. *In situ SAW measurements*

One can observe on Fig. 7 that, except a slight increase in the insertion losses of 0.01 dB/°C, there is no deterioration of the SAW signal when the device goes from 20°C to 500°C. The latter exhibits a large and quasi-constant sensitivity to the temperature, with 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 the direction of propagation of the SAW was here along the Y-axis of the sapphire substrate, and that the relative thickness of AlN (2π.h/λ, where h is the thickness of the AlN film) was quite limited, with a value of 0.5.

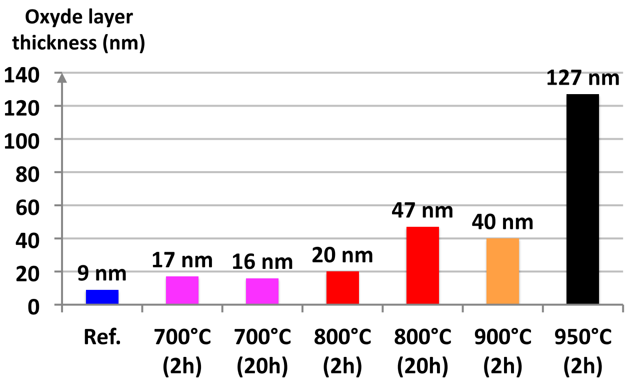

Figure 4. Thickness of the oxide overlayer determined by ellipsometry

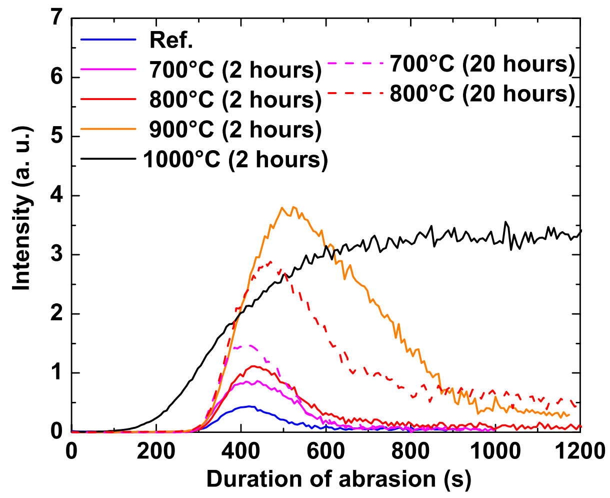

Figure 5. SIMS depth profile signal related to oxygen

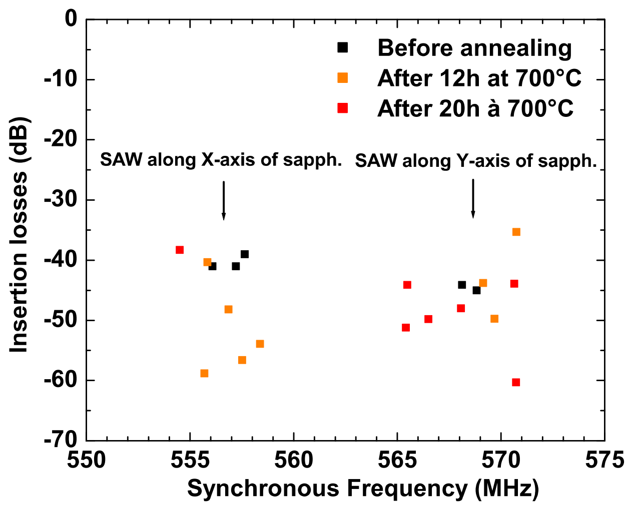

Figure 6. Insertion losses and synchronous frequency of several delay lines carried out on as-deposited AlN films and 700°C-annealed ones

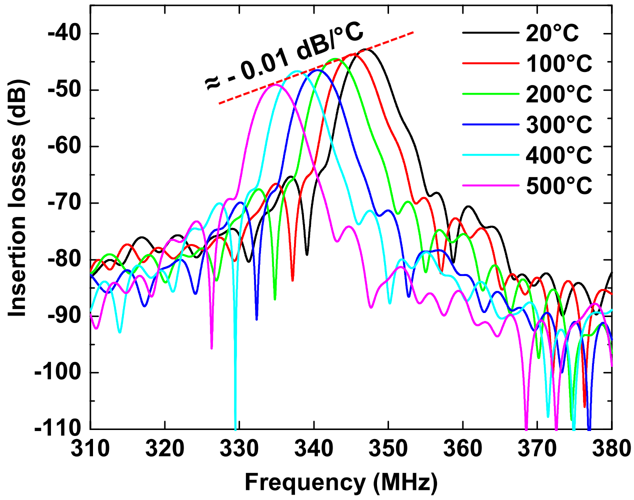

Figure 7. $|S_{21}|$ signal of an IDT (Pt/Ta)/AlN/Sapphire SAW delay line, from 20 to 500°C

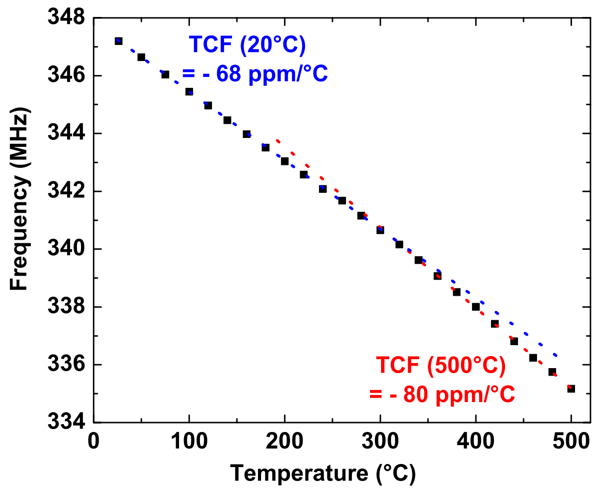

Figure 8. Temperature dependence of the synchronous frequency of the delay line

IV. Conclusion and Outlooks

The results obtained throughout this study suggest that AlN can be used as piezoelectric material for SAW sensors operating up to 700°C in air atmosphere, as the oxidation process seems to be insignificant, 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. Anyway, better stability of AlN is expected in environments less aggressive than air, such as those containing small amounts of oxygen, where temperature as high as 1000°C could be reached [8]. Eventually, 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 are 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 [10].

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