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Is AlN/Sapphire bilayer structure an alternative to Langasite for ultra-high-temperature SAW applications ?

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Abstract— This paper explores the potentiality to use AlN/sapphire structure as alternative piezoelectric material to langasite (LGS) for high-temperature SAW applications. *In situ* SAW measurements performed in vacuum up to 1050°C attest that AlN/Sapphire is more stable than LGS in such conditions. While in the case of LGS, the signal is completely lost after 8 hours at 1050 °C, the device based on AlN/sapphire stays alive for 60 hours at this extreme temperature. Moreover, the degradation is attributed not to AlN but to the agglomeration phenomena undergone by the Iridium IDTs. The AlN/Sapphire structure shows also, between room temperature and at least 1050°C, a great sensitivity to temperature as well as a good linearity, which is very suitable for temperature sensor applications.

I. INTRODUCTION

In addition to being small, simple and robust, surface acoustic wave (SAW) devices have the advantage of being passive (batteryless), remotely requestable (wireless) and inexpensive if fabricated on a large scale. The use of SAW devices as passive and wireless sensors allows them to operate in extreme conditions such as those with high levels of radiation, high temperatures up to 1000°C, or electromagnetic interference, in which no other wireless sensor can operate. This is obviously conditioned by the fact that the materials constituting the device can withstand these harsh conditions.

Langasite (LGS) is currently the reference as piezoelectric material for high-temperature SAW applications up to 850°C [1]. However, some drawbacks such as rapidly increasing propagation losses with temperature and frequency as well as surface deterioration after high-temperature exposure have been reported [2-4]. There is a real need for an alternative material to meet scientific and industrial specifications for harsh environments.

AlN/Sapphire layered structure is expected to be a potential substitute to LGS for high frequency applications. Indeed, AlN/Sapphire structure exhibits low propagation losses, good stability at high temperature and high acoustic velocity required to perform high frequency SAW devices [5]. This paper investigates the suitability of AlN/Sapphire structure to operate at very high temperatures, up to 1050 °C.

II. BACKGROUND AND STATE OF THE ART

Aluminium nitride brings many qualities that make it a material of great interest for electronic and opto-electronic devices. It has a large bandgap of 6.2 eV, high electrical resistivity of 10^{13} Ω .cm combined with a high thermal conductivity of 320 W.mK⁻¹ [6-7] and the highest SAW velocity (5700 m/s) among all piezoelectric materials [8], allowing the fabrication of high frequency SAW devices up to 10 GHz when combined with high velocity substrate such as diamond or sapphire [9-12]. Unlike conventional piezoelectric materials AlN cannot be obtained by crystal growth methods, but must be deposited on a substrate. Several methods have been applied to obtain AlN thin films: MOCVD [11,13], pulsed laser deposition [6], magnetron sputtering [14], etc. The main advantage of such layered structure is the possibility to control device properties like TCF, electromechanical coupling (K^2), velocity, etc, thanks to AlN film thickness and/or the choice of substrate.

Aluminium nitride can be used in high temperature conditions. It melts at 3214°C but starts to decompose chemically in vacuum at 1040°C following the reaction $2\text{AlN}(s) = 2\text{Al}(s) + \text{N}_2(g)$ [15]. In an N₂-rich atmosphere, the equilibrium will be shifted towards AlN formation, and the decomposition temperature will be higher. Thus, Pattel and Nicholson have measured a piezoelectric activity for AlN up to 1150°C in such environment [16].

For high-temperature conditions, sapphire shows the best choice as substrate to be combined with an AlN film. Indeed, it is commercially available with a moderate price, can withstand very high temperature, at least up to 1600°C [17], and the lattice mismatch with AlN is reasonable allowing an epitaxial growth [18]. Moreover, its acoustic wave velocity is very close to that of AlN [19], avoiding large velocity dispersion and ensuring high operating frequencies with conventional lithography.

If a large number of studies has been devoted to AlN-based SAW multilayer structures [11,13,19], few of them were intended for use in high-temperature conditions and the rare published works are very recent. Pisano group has experimentally demonstrated that lamb wave device based on AlN film could be operated up to 700°C in air atmosphere [20]. Bruckner *et al.* have experimentally studied AlN/Sapphire structure at various temperatures up to 858 °C in vacuum. They observed that delay lines patterned on AlN/Sapphire structure could be successfully used at 680 MHz and temperatures of 630°C at least. At 858°C, failure is observed and attributed to the degradation of the Pt electrodes. Post-analysis shows no degradation of the AlN surface. Unfortunately, no intermediate measurement was performed between 630 and 858°C which would have clarified the operating temperature limit [21]. Note however that these results are in perfect agreement with some previous studies about the limits of platinum IDTs estimated at 750 °C [22]. Aubert *et al.* have also studied AlN/Sapphire layered structure as a potential substrate for SAW devices for high temperatures up to 900 °C under air atmosphere [23]. Frequency characterizations of the delay lines based on this structure indicate a slight increase in the insertion losses after annealing for 30 min. at 900 °C and post-analysis (SEM, AFM, XRD) suggest that these losses are due to the deterioration of the Pt/Ta electrodes and to a slight oxidation of the AlN film.

III. EXPERIMENTAL

1.2 μm-thick (002) AlN films were epitaxially grown on c-plane sapphire substrate by magnetron sputtering method [24]. To perform IDTs circumventing the limitations of platinum, 10 nm thick titanium as adhesion layer and 100 nm thick iridium films as electrode, were deposited by e-beam evaporation onto the AlN/Sapphire substrate. SAW delay lines operating at 392 MHz were fabricated using optical lithography and ion beam etching. The delay line is consisting of two identical IDTs with 50 finger pairs and a spatial period (λ) of 14 μm. The gap between IDTs, the aperture, and the finger width to space ratio, were respectively fixed to: 70λ , 65λ , and 1:2. SAW propagates along X-axis of the sapphire substrate. S_{21} scattering parameters were measured using a network analyser (Rohde & Schwarz ZVC), at numerous temperatures ranging from 20°C to 1050°C, under vacuum conditions (pressure: $2 \cdot 10^{-4}$ mbar). To do so, a specific setup was used [25]. Vacuum was chosen as ambient environment because of the tendency of both Ir and AlN towards oxidation from 700°C in

air atmosphere [5,26]. The temperature was increased linearly from 20°C to 1050°C during 20 hours and was maintained at this maximum temperature during 60 hours.

To compare AlN/Sapphire performances with those of langasite, identical delay lines were performed on YX-cut LGS substrates ((Witcore Company, Jinan, China) and characterized simultaneously [27].

Finally, scattering electron microscopy (SEM, Jeol 6500F) was used to get the morphology of the devices surface after high-temperature exposure.

IV. Results and discussion

Frequency responses (S_{21} magnitude) measured at different temperatures and time show how promising the AlN/Sapphire bilayer structure for high-temperature applications in such conditions is (Fig. 1). Indeed, insertion losses are quite constant from room temperature to 1050°C, with variations below 3 dB. A significant deterioration is only recorded after several hours at this last extreme temperature.

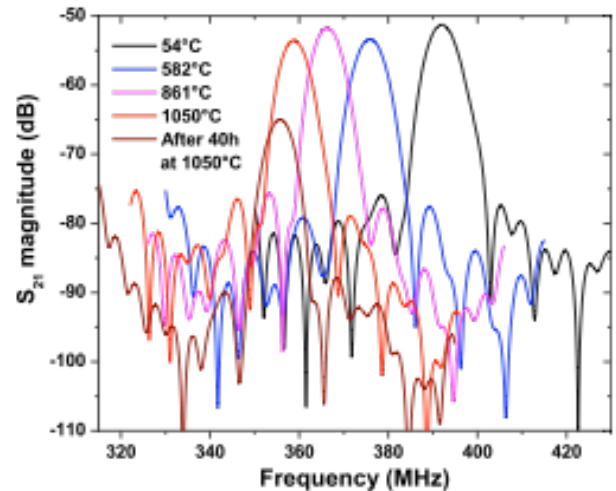


Figure 1. Ir/Ti/AlN/Sapphire SAW delay line frequency response (S_{21} magnitude) at different temperatures.

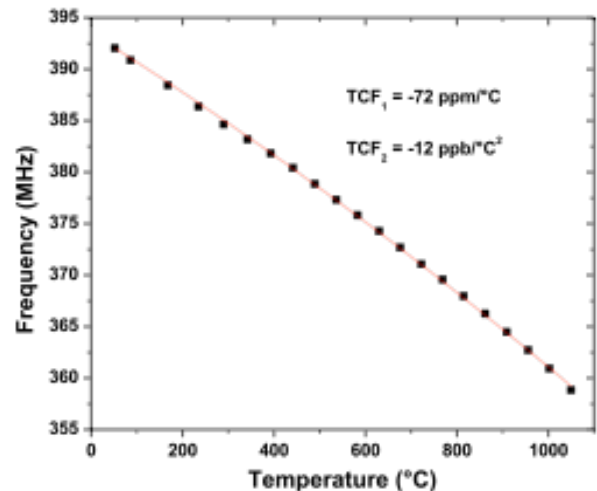


Figure 2. Ir/Ti/AlN/Sapphire SAW delay line frequency-temperature law.

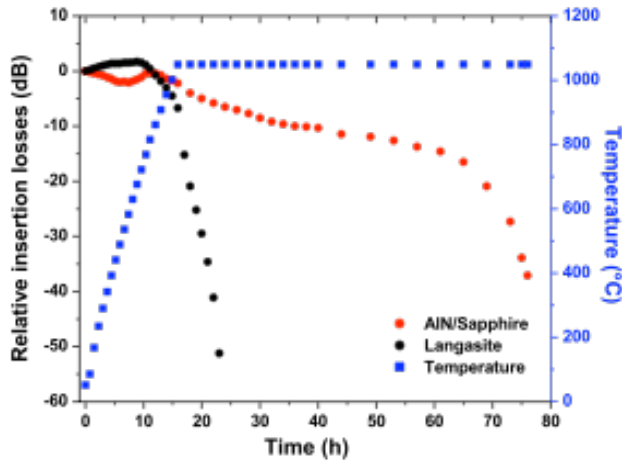


Figure 3. Evolution of insertion losses with time for LGS (black circles) and AlN/Sapphire-based (red circles) delay lines as well as temperature profile (blue squares).

The experimental frequency-temperature law is depicted on figure 2. Note that the studied structure shows either a large sensitivity to the temperature and a remarkably good linearity in the full investigated range with temperature coefficients of frequency (TCF) equals to $-72 \text{ ppm}/^{\circ}\text{C}$ and $-12 \text{ ppb}/^{\circ}\text{C}^2$ for the first and second order respectively (Reference temperature: 50°C). These properties are highly suitable to perform temperature sensors able to cover a wide range of temperature.

Figure 3 shows the variation of insertion losses (IL) with time of both kinds of delay lines (AlN/Sapphire and LGS-based) as well as the temperature profile used during the experiment. One can see that for the LGS delay lines IL decrease slightly with temperature between room temperature and 700°C . Between 700°C and 1050°C a large increase of IL (around $3 \text{ dB}/100^{\circ}\text{C}$) is observed. The rate of this deterioration is even amplified during the plateau at 1050°C where it reaches $6 \text{ dB}/\text{hour}$, leading to signal loss after 8h at this level. The AlN devices show a completely different behaviour. It can be divided in four steps. First of all there is a slight increase of IL with temperature up to 500°C . Note that this observation is consistent with what we already obtained in a previous work where the characterization was made up to 500°C . In the latter case, the environment was air atmosphere and the IDTs were made from platinum [5]. A second phase between 500 and 900°C shows an unexpected decrease in IL which get back to their initial value. Beyond 900°C , IL increase again but at a very small rate compared to the one recorded for LGS. During the plateau at 1050°C , the increase is only of $0.3 \text{ dB}/\text{hour}$ during the first 50 hours. Afterwards, we see a radical change in behavior that results in the loss of the signal after 60h at 1050°C . SEM images performed on the samples after annealing confirm the difference in behavior concerning both kinds of devices. In the case of LGS, one can observe a strong increase in the substrate roughness accompanied by the apparition of numerous cracks (Fig. 4a), as well as the vanishing of the Ir IDTs (Fig. 4b). Such evolution is attributed to the high Oxygen and Gallium losses experienced by the LGS substrate at such high temperature [27].

Concerning the AlN-based devices, the main information brought by SEM is that the Ir IDTs undergo severe agglomeration phenomena, but without any vanishing. This agglomeration was expected since the Tamman temperature of Iridium (1080°C) is very close to 1050°C . Thus we assume that the signal loss can be attributed to the degradation of the electrodes and not of the AlN film.

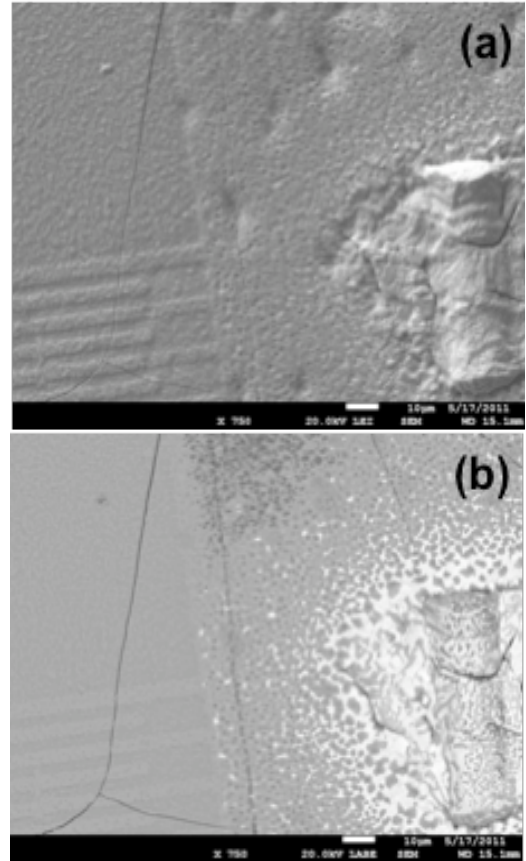


Figure 4. SEM images of an Ir/Ti/LGS SAW device after 60h at 1050°C under vacuum conditions ((a) topography/(b) chemical contrast). IDT finger width is $2.3 \mu\text{m}$. On the right half of the image is the bus bar on which the tip of a bonded Pt microwire is clearly visible (bottom-right corner).

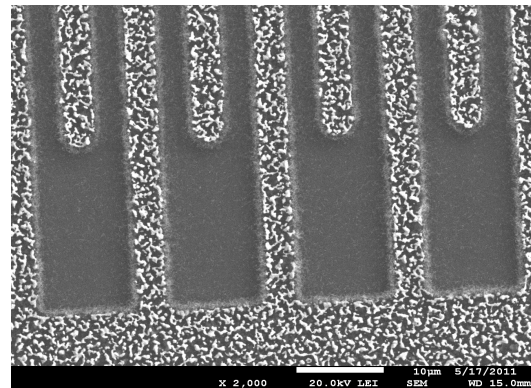


Figure 5. SEM image of an Ir/Ti/AlN/Sapphire SAW device after 60h at 1050°C under vacuum conditions (chemical contrast). The agglomeration phenomena of Ir IDTs is clearly visible.

V. CONCLUSION

In this study, we have shown that SAW devices based on Ir/Ti/AlN/Sapphire structure exhibit a good stability at high temperatures up to 1050°C even better than LGS' one in environments containing small amounts of oxygen. Moreover, the studied structure shows a good linearity of the frequency with temperature in the full studied range (room temperature to 1050°C). All these results combined with previous work about the acoustic propagation losses of the AlN/sapphire structure at high frequency, make this structure a very promising solution to perform wireless SAW sensor operating at high frequency and high temperature.

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