AlN-based solidly mounted resonators on glass substrates for high temperature applications

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Abstract—Acoustic wave resonators have been widely investigated for processes monitoring under harsh environments, particularly high temperatures, due to their low cost and good performance. One of the most explored devices are langasite-based surface acoustic wave sensors. Although they have proven to fulfill high temperature requirements, their long and narrow metallic strips can still suffer from destructive agglomeration. As an alternative, we propose using AlN-based solidly mounted resonators (SMRs). In previous works we proved these devices to sustain temperatures up to 1000°C without degradation, however we have never tested their in-situ behavior at high temperatures. In this work we not only electrically characterize them in-situ (while annealing), but we prove Si substrates are not adequate when metallic strips are present on the resonator surface or these are integrated in filters or arrays, since Si becomes conductive above 250°C and large capacitive parasites appear. To solve this, we used Corning glass substrates, which have proven to preserve the performance of SMRs up to 400°C.

Keywords—solidly mounted resonator, high temperature, AlN, glass substrates, in-situ measurements.

I. INTRODUCTION

Sensors capable of operating at high temperatures are increasingly demanded in many industrial fields, which includes the petro-chemical sector, concrete processing, and aerospace or automotive sectors. For example, in the aerospace sector, there is a need for sensors able to monitor the gases emitted by propulsion jet engines; engines, tires or brakes also need to be controlled in the automotive field. The possibility of measuring temperature or pressure in harsh environments is also fundamental in chemical industry.

Within the different sensing techniques [1], piezoelectric acoustic sensors appear to be especially interesting [2] because their output response is usually a frequency variation, which allows for remote interrogation, eliminating the need for interconnecting wires. In particular, one of the most explored approaches is that of surface acoustic wave (SAW) resonators [3-5], although high temperature applications have been also proven for other devices, such as Lamb wave resonators [6]. A SAW device is based on an interdigital transducer (IDT) usually placed on a piezoelectric single crystal. Many studies have

focused on the selection of the materials needed for such critical applications [7,8]. Regarding the piezoelectric substrates, their main limitations are the phase transitions they can experience that can turn them into non-piezoelectric materials. Single-crystal langasite (LGS) seems to be the most frequently used. Pt with different adhesion layers or as part of composites has proven to be a suitable metal for the IDTs [9, 10]. A serious drawback of high-temperature SAW devices is the transducer topology. At high temperatures the IDTs, composed of long and narrow metallic strips, suffer from destructive agglomeration. Solidly mounted bulk acoustic resonators (SMRs) based on thin AlN piezoelectric films can be a good alternative to overcome the electrodes and power handling limitations of SAW resonators [11] and to push operating frequency towards the GHz range.

Recently we demonstrated that AlN-based SMRs manufactured over standard Si substrates could sustain temperatures as high as 1000°C without significant degradation of their piezoelectric response after the annealing process [12]. However, the devices were not in-situ characterized during the heat treatment, i.e. we did not assess their frequency response during their operation at high temperature, which we intend to monitor in the present work.

Undertaking the monitoring of the frequency response of acoustic devices at high temperatures possess two main challenges. The first one is the experimental setup. In many cases it has been solved with the use of an RF cable specifically designed for high temperatures or using remote interrogation. The second challenge involves the device itself. First, the multilayered structure must sustain the heat treatment without degrading, overcoming problems associated to thermal stresses (lack of adhesion between layers, micro-cracking), microstructural changes (grain size increase, new crystalline phases,) or chemical reactions (oxidation). In addition, the electrical response of the resonators should remain stable upon increasing the temperature, in the sense that they should deliver a similar output signal in response to the same stimulus. For example, typical high resistivity Si substrates ($\rho > 4000 \text{ } \Omega \cdot \text{cm}$) used in SMRs production become conductive at 250°C, since the intrinsic concentration exceeds $3.3 \times 10^{14} \text{ cm}^{-3}$, and the resistivity decreases to $10 \text{ } \Omega \cdot \text{cm}$. Therefore, intrinsic Si substrates do not

behave as insulators at high temperatures, which seriously compromises the frequency response of resonators containing metallic lines on their surfaces or the independent operation of resonators on the same substrate like filters or arrays owing to the parasitic capacitances that appear between the electrodes and underlying conductive layers [13]. To overcome this problem true insulating substrates like ceramic Al_2O_3 or AlN , or quartz or glass plates can be used. In addition, insulating acoustic mirrors are also required; acoustic reflectors composed of alternated layers of low acoustic impedance (SiO_2) and high acoustic impedance (Ta_2O_5 or WO_x) have already proven their suitability [14,15] but they degrade at high temperature.

We have previously solved the issue of the integrity of the multilayered structure at very high temperatures for silicon substrates [12]. In this paper we tackle the in-situ characterization and electrical response challenges. On one hand, we adapt our RF probes to sustain high temperatures following an already employed procedure [16]. On the other hand, we use devices containing metallic electric lines at their surface, which would be a realistic situation in many scenarios. Therefore, the SMRs are manufactured over electrically insulating glass plates and acoustic mirrors. We discuss aspects related to the device fabrication, like the choice of the substrates and materials, the experimental setup for high temperature measurements and compare the frequency response at high temperature of SMRs manufactured over both glass and Si substrates.

II. DESIGN AND MODELING OF THE SMRS

We used SMRs operating at around 2.4 GHz consisting of an AlN active layer sandwiched between two metallic electrodes and grown over electrically insulating acoustic reflectors deposited on glass substrates, as shown in Fig. 1. The top electrodes of the devices were purposely extended by means of electrical lines to set the contact pads away from the active area of the resonator. These electrical extensions were designed like a $50\ \Omega$ microstrip waveguide. The bottom electrode was patterned to avoid the formation of parasitic capacitors under the metallic lines of the electrical extension. In addition, to prevent the formation of other parasitic capacitors in parallel with the resonator that might significantly degrade their frequency response at high temperatures, the conventionally used Si substrate was substituted by an electrically insulating plate. Among the possible choices, ceramic plates were discarded because they are too rough and hard to polish. Likewise, quartz plates were also discarded because their price is excessive for production purposes. Finally, we opted for boron-silicate glass plates from Corning (7059) that can endure temperatures up to 639°C before softening. However, high resistivity Si substrates were also used for comparison purposes. The designed acoustic reflector was composed of seven $\lambda/4$ alternating layers of SiO_2/AlN with thicknesses of 516 nm and 916 nm, respectively; As for the piezoelectric capacitor, we opted for refractory metal electrodes (Ir and Mo), with an Au capping to prevent the Mo top electrode from oxidizing during the heat treatment.

Fig. 1. Scheme of the device. a) Cross section and b) top view photo.

III. EXPERIMENTAL

A. Production of SMRs

The multilayered SiO_2/AlN acoustic reflector was sputtered on glass plates and high-resistivity Si wafers. The uppermost SiO_2 layer of the acoustic reflector was mechanically polished to a roughness below 1 nm. Then, the Ir bottom electrode was e-beam evaporated at a substrate temperature of 400°C over a 10 nm-thick Ti film used as adhesion layer. The Ti/Ir bilayer was patterned by Ar ion milling using a Mo hard mask which was removed afterwards. The piezoelectric AlN layer was then sputtered in an ultra-high vacuum under a high-energy process yielding highly c-axis oriented films with rocking curves around the $(00\cdot2)$ XRD reflection narrower than 2° . Finally, the Mo top electrode was sputtered and capped with a thin Au film. The deposition conditions for all the layers were adjusted to reduce internal stress in each layer ($< 200\ \text{MPa}$) to preserve the mechanical integrity of the structure, even at high temperature.

B. Characterization of SMRs

The frequency response of the SMRs was assessed by measuring the S_{11} reflection coefficient at variable frequency with a network analyzer from which the electrical impedance spectrum was derived. The resonator characteristics (resonant frequencies, coupling factors and quality factors) were obtained by fitting of the experimental frequency response of the impedance to Mason's model [17] and by modeling the effect of the extended electrodes by an inductance and a series resistance. In fact, the extended electrodes designed as $50\ \Omega$ microstrip waveguide behaved as an inductance, owing to their long length compared to the wavelength of the acoustic wave.

The in-situ high-temperature measurements were carried out using a modified RF probe from Picoprobe®. We employed the method suggested by Schwartz et al. [16] to condition the probe for high power dissipation, consisting in attaching a Cu heat spreader to the RF probe (see Fig. 2). This way we could measure the samples placed on the hot stage at temperatures up to 420°C (limited by the heater). The heater was a homemade stainless steel holder housing a double coil of Inconel sheathed heating cable from ThermSys. The heater temperature was set by means of a PID controller through a LabVIEW application that also controlled the network analyzer. A typical measurement cycle consisted in increasing the temperature from room temperature to 425°C in steps of 50°C and registering the frequency response after reaching the steady state. The whole

cycle lasted typically 30 min. The calibration of the network analyzer was carried at room temperature. This calibration remained relatively stable during the heating cycle, with only small drifts that barely affected the measurement of the resonators characteristics. After the cooling down, the experimental frequency response was almost identical to that recorded before the thermal cycle.

Fig. 2. RF probe station modified for high temperature operation. A Cu cap is used to prevent temperature dissipation (bottom).

IV. RESULTS AND DISCUSSION

The performance of SMRs grown over glass substrates resulted similar to those grown over Si. To verify our previous experiments with Si-based SMRs [12] we annealed glass-based devices up to 500°C. Their post-annealing assessment revealed no frequency response (Fig. 3), nor structural degradation.

Fig. 3. Modulus of the electrical impedance of a typical SMR deposited on glass substrate at room temperature (blue line) and after a thermal annealing at 500°C for 2 h (orange).

For the in-situ characterization we used both types of devices: Si-based and glass-based. We tested them in ambient atmosphere at variable temperature from 25°C to 400°C by using the modified probe described previously. The measurements were corrected by de-embedding a series resistance and a series inductance, resulting from the extended electrodes. Then, the

resonant and anti-resonant frequencies, electromechanical coupling factors, and quality factors at anti-resonant frequency were derived as a function of the temperature.

Fig. 4 shows the real part of the impedance around the resonant frequency as a function of the temperature for both types of SMRs manufactured with identical SiO_2/AlN acoustic reflectors over the two different substrates: a high-resistivity Si wafer (Fig. 4a) and a 7059 Corning glass plate (Fig. 4b)). The degradation of the response of the resonator operating over Si substrates can be clearly observed in Fig. 4a, where the increasing conductivity of the Si substrate produces a severe attenuation of the impedance at resonance as the temperature increases, almost vanishing for temperatures above 220°C. Contrarily, the resonance peak in the SMR manufactured on a glass substrate maintains a significant amplitude even at higher temperatures. The ripples appearing in the high-temperature measurements are due to the mentioned drift in the network analyzer calibration.

Fig. 4. Spectra of the real part of the impedance for a resonator deposited on a) silicon and b) glass at different temperatures.

Fig. 4 also reveals that in both cases, the resonant frequencies are shifted towards lower values as the temperature is increased, which was an expected result owing to the negative value of the TCFs of most of the materials involved in the multilayered structure. In order to clarify this effect, we have depicted in Fig. 5 the evolution of the antiresonant frequency (frequency at the maximum of $\text{Re}(Z)$) as a function of temperature for the two devices. The resulting TCFs for silicon-based and glass-based SMRs are -14 ppm/°C and -12 ppm/°C, respectively. This low

values are due to the four SiO₂ layers included in the acoustic reflectors.

Fig. 5. Resonant frequency variation of SMRs deposited on a high resistivity silicon substrate (blue symbols) and on a glass substrate (orange symbols).

The attenuation of the resonant peak observed in Figs 4a and 4b is nothing but the decrease of the quality factors at antiresonant frequency of the resonators. To highlight this effect, Fig 6 depicts the maximum of $\text{Re}(Z)$ as a function of the temperature for both resonators. This maximum is directly related to the quality factor of the device. It is worth noting that the quality factor of the Si-based SMR dramatically drops with temperature whereas that of the glass-based SMR experiences only a moderate steadily decrease.

Fig. 6. Height of the $\text{Re}(Z)$ peak (Quality factor at anti-resonant frequency) for the resonators of Fig 4.

CONCLUSIONS

We have demonstrated that common high-resistivity Si substrates are not appropriate for SMRs intended for high temperature (>200°C) operation since they can strongly affect the performance of devices or enhance the cross talk between them in complex structures like filters or arrays. This is due to the decrease in the resistivity of Si at high temperature due to the increase in the intrinsic carrier density. This problem can be solved by using an insulating substrate operative at high temperature and a fully insulating acoustic reflector, if no metallic layer patterning is desired. High temperature glass plates, like 7059 Corning, can be a good choice in front of rough

ceramic plates or expensive quartz substrates. The in-situ high-temperature electrical characterization carried out on Si wafers and glass substrates clearly sets the severe degradation suffered by the Si-based devices compared to the glass ones.

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REFERENCES

- [1] M. R. Werner and W. R. Fahrner, "Review on material, microsensors, systems and devices for high-temperature and harsh-environment applications", *IEEE Transactions on Industrial Electronics*, vol. 48, pp. 249-257, April 2001.
- [2] X. Jiang, K. Kim, S. Zhang, J. Johnson and G. Salazar, "High-temperature piezoelectric sensing", *Sensors*, vol. 14, pp. 144-169, 2014.
- [3] D. W. Greve, T.-L. Chin, P. Zheng, P. Ohodnicki, J. Baltrus and I. J. Oppenheim, "Surface acoustic wave devices for harsh environment wireless sensing", *Sensors*, vol. 13, pp. 6910-6935, 2013.
- [4] T. Aubert and O. Elmazria, "Stability of langasite regarding SAW applications above 800°C in air atmosphere", *2012 IEEE International Ultrasonics Symposium Proceedings*, pp. 2098-2101, 2012.
- [5] J.A. Thiele and M. Pereira da Cunha, "High temperature LGS SAW gas sensor", *Sensors and Actuators B*, vol. 113, pp. 816-822, 2006.
- [6] T.-T. Yen et al., "Characterization of aluminum nitride Lamb wave resonators operating at 600°C for harsh environment RF applications", *2010 IEEE International Conference on MEMS*, pp. 731-734, 2010.
- [7] J. Hornsteiner, E. Born, G. Fischerauer and E. Riha, "Surface acoustic wave sensors for high-temperature applications", *1998 IEEE International Frequency Control Symposium*, pp. 615-620, 1998.
- [8] D. Damjanovic, "Materials for high temperature piezoelectric transducers", *Solid State & materials Science*, vol. 3, pp. 469-473, 1998.
- [9] M. N. Hamidon et al., "Fabrication of high temperature surface acoustic wave devices for sensor applications", *Sensors and Actuators A*, vol. 123, pp. 403-407, 2005.
- [10] E. Çiftçiyörek, K. Sabolsky and E. M. Sabolsky, "Platinum thin film electrodes for high-temperature chemical sensor applications", *Sensors and Actuators B*, vol. 181, pp. 702-714, 2013.
- [11] B. Ivira et al., "Solidly mounted resonators aging under harsh environmental conditions", *International MEMS Conference 2006, Journal of Physics*, vol. 34, pp. 668-673, 2006.
- [12] T. Mirea, J. Olivares, M. Clement, J.L. Olivera, J. Sangrador, and E. Iborra, "AlN-Solidly mounted resonators sustaining up to 1000°C with TCF compensation", *2017 Joint Conference of the European Frequency and Time Forum and IEEE International Frequency Control Symposium (EFTF/IFCS)*, pp. 519 - 22
- [13] M. DeMiguel-Ramos et al., "Influence of the electrical extensions in AlN-BAW resonators for in-liquid biosensors," in *2014 European Frequency and Time Forum (EFTF)*, 2014, pp. 301-304.
- [14] J. Capilla, J. Olivares, M. Clement, J. Sangrador, E. Iborra, and A. Devos, "High-acoustic-impedance tantalum oxide layers for insulating acoustic reflectors," *IEEE Trans. Ultrason. Ferroelectr. Freq. Control*, vol. 59, pp. 366-372, . 2012.
- [15] M. DeMiguel-Ramos, B. Diaz-Duran, J. Munir, M. Clement, T. Mirea, J. Olivares, and E. Iborra, "Tungsten oxide layers of high acoustic impedance for fully insulating acoustic reflectors," *IEEE Trans. Ultrason. Ferroelectr. Freq. Control*, vol. PP, no. 99, pp. 1-6, 2015.
- [16] Z.D. Schwartz, A.N. Downey, S.A. Alterovitz, and G.E. Ponchak, "High-Temperature RF Probe Station for Device Characterization Through 500°C and 50 GHz", *IEEE Transactions on Instrumentation and Measurement*, 54, pp. 369-373, 2005.
- [17] J. F. Rosenbaum, *Bulk Acoustic Wave Theory and Devices* (Artech House, New York, 1945)