High Overtone Bulk Acoustic Resonators for High Temperature Sensing Applications

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Abstract — **High overtone Bulk Acoustic Resonators have been developed for radio-frequency application such as oscillator stabilization, but also as an alternative to surface acoustic wave resonator for sensor development. In the present work, the possibility to operate such devices at temperature up to 800°C is investigated experimentally. Devices built using Aluminum Nitride deposited on Silicon with Platinum electrodes have been manufactured and resonance frequencies near the 434-MHz centered ISM band have been characterized from room conditions to 800°C. Although the exposition to such a temperature yields changes in the device response, it turns out that the operation is partly reversible and that these HBARs could operate without major defects for several tens of hours at such regimes. The development of wireless temperature sensors on this base then reveals accessible.**

Keywords-Sensor; BAW; High overtone Bulk Acoustic Resonator – HBAR; High temperature; AlN; Silicon; Sapphire

I. INTRODUCTION

The development of elastic wave sensors capable to be remotely interrogated without on-board energy has yield new markets rise. The corresponding users challenge these sensors for several extreme applications such as operation at temperature above 500°C. Although the use of Langasite was the most promising approach, Aluminum Nitride (AlN)-based structures were also considered as a potential solution [1]. It was demonstrated that high overtone bulk acoustic resonators (HBARs) can be applied for wireless sensing as an alternative to SAW resonators [2]. As AlN is well-suited for such devices and exhibit a melting point in excess of 2000°C, the idea to exploit AlN-based HBAR for high temperature operation was considered in the present work.

AlN-based HBARs have been manufactured in a Sigma-Trikon sputtering machine, depositing 1µm thick layers onto a (111) Ti/Pt backside electrode deposited atop 530µm thick Sapphire and 400µm thick Silicon substrates. The classical figure of HBAR response was observed, with a frequency separation of about 8 to 9 MHz depending on the substrate and a maximum resonance of the layer alone near 2 GHz. The resonances between 400 and 500 MHz (close to the 434 MHz centered ISM band) have been focused for assessing the operation of the devices at temperature larger than 500°C.

These devices have been submitted to temperature cycles up to 600°C for 48 hours in an oven developed at IMTEK for calibrating acoustic devices [3], and their electrical response were monitored at various frequencies during the experiment. The observation of the device showed minimal de-wetting effects in form of single droplets atop the surface, as it is currently the case for inter-digitated-based transducer devices. The material lattice was found stable enough to withstand the temperature induced tensions as the thermal grooving and the resulting forming of droplets leaving micro cavities in the metallization layer is observed but limited and do not prevent the device operation. The experiments then were extended successfully up to 800°C.

In the first section of the paper, basic considerations on materials and sensor structure are discussed. The fabrication of the test vehicles then is described and finally test results of the HBAR under temperature ranging from room to 600°C and 800°C are reported.

II. HBAR MATERIALS AND STRUCTURE FOR HIGH TEMPERATURE APPLICATION

A. Materials

Quartz, Lithium Tantalate ($LiTaO_3$) and Lithium Niobate ($LiNbO_3$) are currently the most common piezoelectric crystals for the development of acoustic wave sensors. However, their maximum admissible operation temperature is limited by intrinsic properties, i.e. phase transitions and material decomposition (see for instance [4]). Gallium Orthophosphate and Langasite have both been used for high temperature purposes and revealed their robustness at temperature in excess of 800°C. The main point is that the SAW devices built on such materials suffer from important degradation when exposed for several tens of hours to temperature in excess of 600°C [3][4].

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On the other hand, Aluminum Nitride (like all III-V nitrides) exhibits a high thermal conductivity and a high stability at elevated temperatures. Patel et al. [1] were able to measure an ultrasonic response of AlN at temperatures up to 1150°C, whereas a constant signal strength was observed up to 900°C. AlN exhibits the highest bulk acoustic wave velocity of all known piezoelectric materials, as well as an electromechanical coupling high enough for the generation of high overtones in HBAR structures. The basic idea then is to use such a structure which does not suffer from electrode fragility as it is the case for SAW to operate it at temperature in excess of 600°C. Platinum was considered specifically here as top and bottom electrodes because of its compatibility with high quality AlN deposition and its well known resilience to temperature.

B. *Device characteristics*

The schematic cross section with the specific stack details of this kind of HBAR is given in Figure 1 as well as the metallization layers and thicknesses. For the processing of the layers themselves, standard processes as described in section III where the buried electrode and top electrode layers were vapor deposited and the transducer layer was sputtered. A standard (100) silicon substrate was used for this type of HBAR. The main electrode layouts are also depicted in Figure 1. The top electrode was not protected anyhow as there were no fine electrode design structures but only plain surface area.

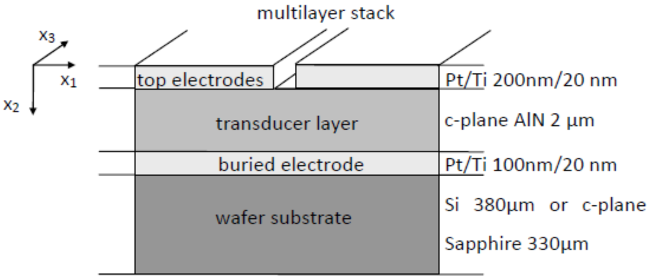

Figure 1 Scheme of HBAR stack structure with design and material parameters for the specific stack used in this work

III. FABRICATION OF THE RESONATORS

AlN-based HBARs have been manufactured in a Sigma-Trikon sputtering machine, depositing 1µm thick layers onto a (111) Ti/Pt backside electrode deposited atop 530µm thick Sapphire and 400µm thick Silicon substrates. The qualitative observation of the layer (**Figure 2**) indicates a dense layer with the expected textured columnar structure.

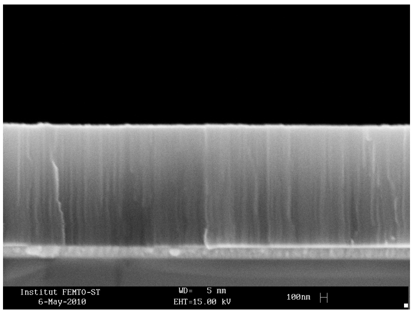

Figure 2 SEM observation of the trench of an AlN layer deposited on a Ti/Pt electrode on silicon

The Ti/Pt bottom electrode was deposited by sputtering and then thermally annealed to obtain proper orientation promoting the AlN deposition. This thermal process was achieved in the sputtering machine just before the AlN deposition to minimize potential surface pollution. Figure 3 shows a view of the devices after top electrode patterning. Although these devices were built for double-port laterally coupled HBAR application, they could be used without difficulties as single port resonators by simply leaving one of the port open.

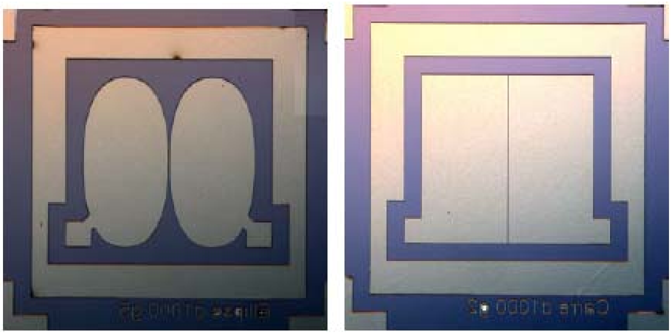

Figure 3 Photos of two top electrode designs for the manufactured HBAR devices, here, devices processed atop c-plane AlN on (100) silicon

IV. EXPERIMENTAL CHARACTERIZATION OF HBAR AT HIGH TEMPERATURE

The classical figure of HBAR response was observed, with a frequency separation of about 8 to 9 MHz depending on the substrate and a maximum resonance of the layer alone near 2 GHz. The resonances between 400 and 500 MHz (close to the 434 MHz centered ISM band) have been focused for assessing the operation of the devices versus temperature. The devices were first exposed to 500°C for tempering the platinum layer and for the following high-temperature measurements at 600°C and 800°C (along the profile of **Figure 4**), the different devices and their frequency characteristics were measured afterwards and evaluated in comparison to un-tempered devices.

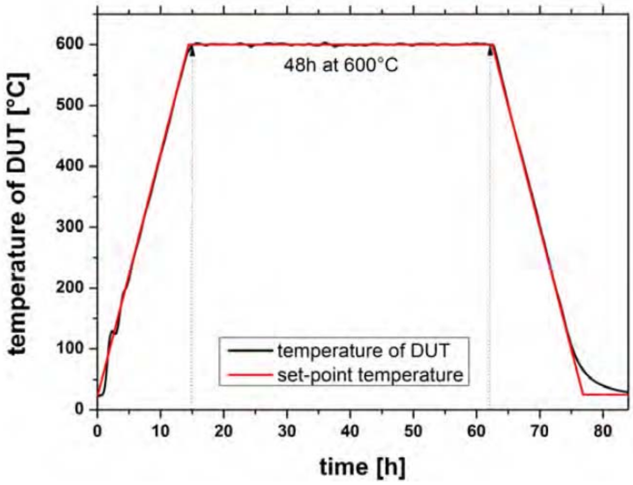

Figure 4 Temperature profile for the characterization of the temperature behavior and stability, the set-point temperature measured by a PT100 is the oven temperature

The on-wafer level measured conductance of the two test designs of HBARs are shown in Figure 5. In the frequency band of interest from 400 to 450 MHz (including the 433MHz-centered ISM band), five well-defined longitudinal mode signatures are observed as well as six very weak signals attributed to shear polarized modes. The resonance frequency of the different modes of one device at one frequency clearly does not change before and after being exposed to 600°C for 48h. Only the amplitude as a figure for the quality factor decreased, possibly due to thermal induced defects at the interfaces between the transducer, layer adhesion and the device surface degradation (not protected during the tests).

During the this first temperature test up to 600°C, the devices were continuously measured by acquiring the resonance frequency measuring the S_{11} parameters automatically every 15 min. using a specific but un-calibrated high-temperature resistant device holder. Therefore one recognizes a varying frequency dependant offset of electrical parasitic effects (see Figure 6).

As the devices did operate well during the whole thermo cycle, the characterization experiment has been extended to 800°C. The devices were heated-up to 800°C and hold at this temperature again for 48h and cooled-down to room-temperature. Comparing the linearity of the frequency of the heating-up phase to the cooling down phase, the frequency shift is about 110 kHz. This corresponds to relative aging rate of 5.3 ppm per hour at 800°C. The linearity during heat-up and cool-down phases and the steadiness of the resonance frequency at the maximum temperature during the hold phase were also evaluated. The results are shown in Figure 7. The temperature coefficient of the frequency (TCF, in some way the sensor “sensitivity”) was measured as shown in Figure 8. The TCF then is estimated between -40 and -42 ppm/K for the complete temperature range and -49 ppm/K at 800°C.

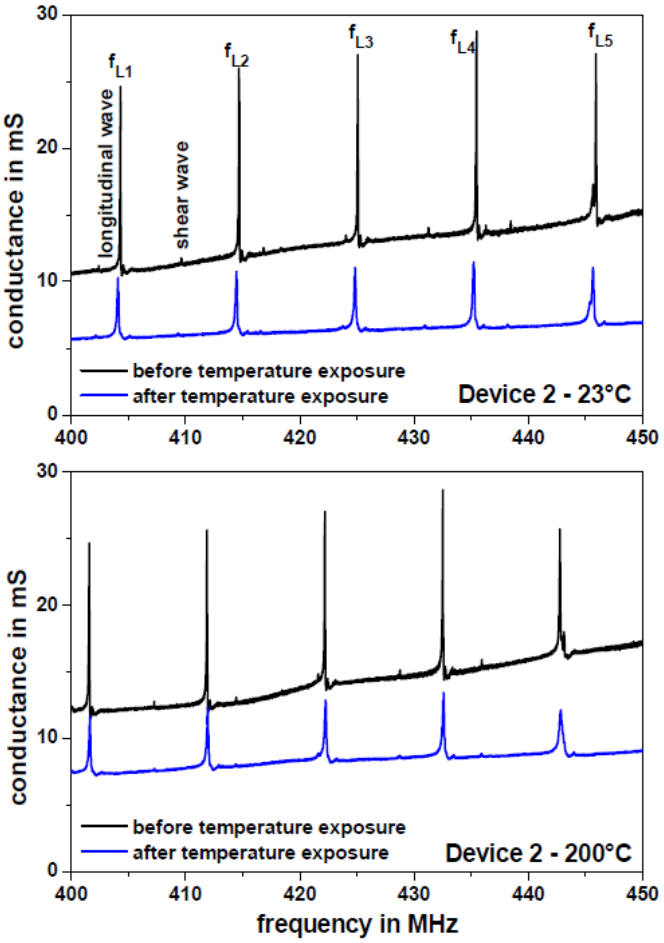

Figure 5 On-wafer measured conductance of the tested HBARs before and after being exposed to 600°C for 48h at room condition and 200°C

At platinum surfaces, as shown in Figure 9, minimal de-wetting effects in form of single droplets atop the surface are observable, showing that the (111) orientation is not fully homogeneous but the lattice is stable enough to withstand the temperature induced tensions as the thermal grooving and the resulting forming of droplets leaving micro cavities in the metallization layer is limited.

VI CONCLUSION

It has been demonstrated that AlN/Si-based HBARs with Ti-Pt electrodes are capable to operate at temperature up to 800°C for several tens of hours. Some signal characteristics are of course observed as the direct observation of the device reveals obvious but limited defects compared to SAW devices operated at such temperature. The importance of an initial tempering process (electrode annealing) has been emphasized to keep a stable device response on the whole temperature range. An almost linear average TCF was measured near -45 ppm.K^{-1} representative of Silicon thermal sensitivity. Furthermore, as the use of HBAR for wireless applications was already reported, future work will be engaged to develop remotely controlled passive sensors based on the presented principles.

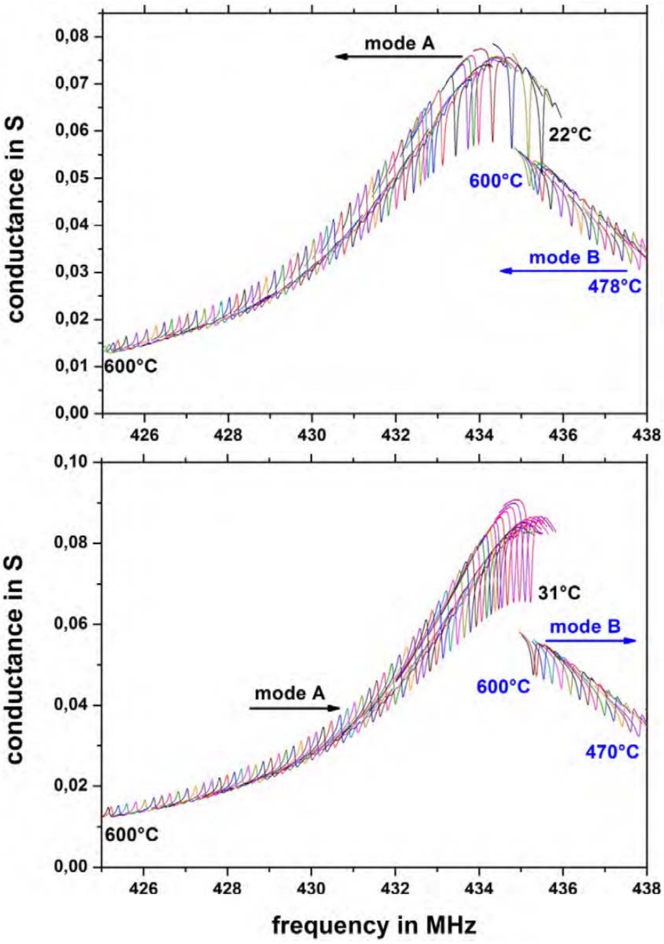

Figure 6 Measured conductance of a test HBAR during heating-up to 600°C (upper graph) Measured conductance of a test HBAR during heating-up to 600°C (upper graph) and cooling down again to room temperature (lower graph) around the 433 MHz ISM band showing two different modes marked as A and B. The flashes indicate the direction in dependence of the temperature gradient

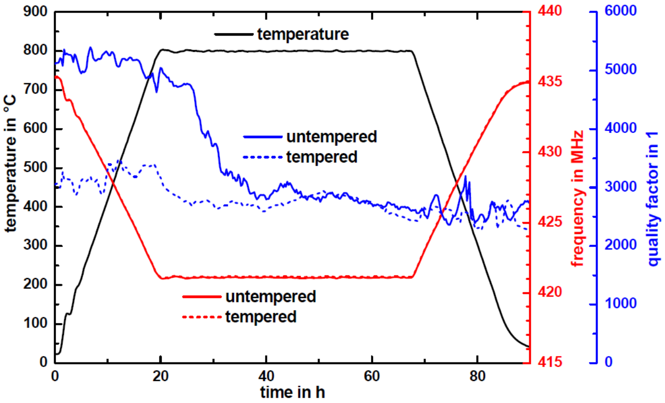

Figure 7 Frequency response and quality factor of un-tempered and tempered HBAR test devices versus time during a measurement cycle of 90 hours with a rising temperature up to 800°C. The quality factor of the tempered devices decreases by a value of about 800 whereas for the un-tempered device the value decreases by 2500

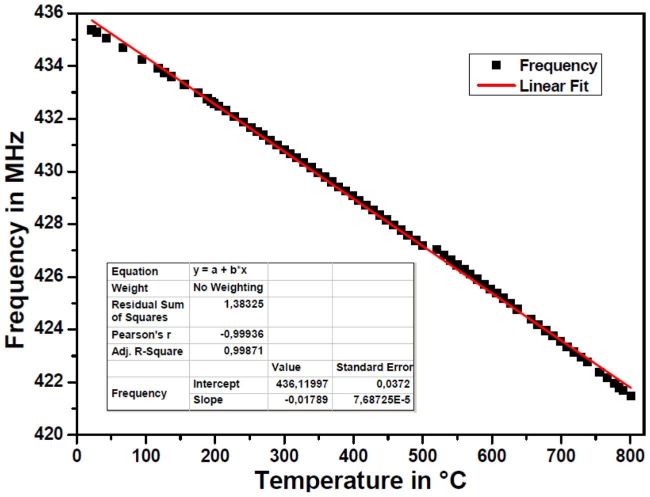

Figure 8 Measured device frequency characteristics of the test device up to 800°C resonance frequency in dependence of temperature with the corresponding linear fit, evaluating the frequency-temperature behavior and its linearity

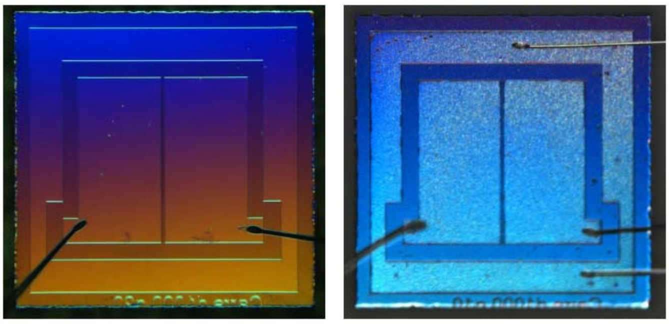

Figure 9 Differential interference contrast LM-micrographs for a visualized surface topography of test devices exposed to temperature. Left-hand side: after being exposed for to 600°C for 48h. Right-hand side: after being exposed to 800°C for 48h.

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REFERENCES

[1] N. D. Patel, P. S. Nicholson, “High-frequency, high-temperature ultrasonic transducers,” Ndt International, vol. 23, pp. 262–266, 1990.

[2] S. Ballandras et al, “High overtone Bulk Acoustic Resonators built on single crystal stacks for sensors applications” Proc. of IEEE Sensors Conference, pp.516-519, 2011

[3] I. Shrena et al, "SAW-relevant material properties of langasite in the temperature range from 25 to 750 °C: New experimental results," Proc. of the Ultrasonics Symposium, pp.209-212, 2008

[4] R. Fachberger et al, “Applicability of LiNbO3, langasite and GaPO4 in high temperature SAW sensors operating at radio frequencies,” IEEE Transactions on UFFC, vol. 51, no. 11, pp. 1427–1431, 2004.