AlN-Solidly mounted resonators sustaining up to 1000°C with TCF compensation

Teona Mirea, Jimena Olivares, Marta Clement, José Luis Olivera, Jesús Sangrador, and Enrique Iborra

Abstract—Surface acoustic wave (SAW) devices are currently the most exploited acoustic wave devices for high temperature applications due to their straightforward fabrication (few photolithographic steps) and ability for wireless interrogation. However they have some drawbacks like the destructive agglomeration of their electrodes, leading to low power handling, or their low operating frequencies (MHz range). In this work we present AlN-based solidly mounted resonators (SMRs) as an alternative to SAWs for high temperature applications. They offer the advantages of simplicity, high power handling and high operating frequencies. We prove that, with a proper design, SMRs can sustain temperatures as high as 1000°C with temperature coefficients of frequency significantly lower than SAW devices, which is crucial for sensing applications at high temperatures.

Keywords—AlN-based SMR; high temperature; TCF compensation.

I. INTRODUCTION

Surface acoustic wave (SAW) devices employing langasite (LGS) substrates are the most frequently acoustic devices used for sensing applications in harsh environments. Particularly, they have been proven to operate at temperatures above 1000°C [1]. Their success for this particular application lies on the fact that LGS is one of the most stable piezoelectric substrates at such high temperatures and on the possibility of the wireless interrogation of SAW devices, mainly in the form of delay lines. Recently, AlN-based SAW devices employing sapphire substrates have been demonstrated for operation at 1050°C [2]. AlN retains its piezoelectricity up to 1150°C [3], being chemically stable up to 1040°C [4]. Compared with LGS-based SAWs, AlN-based SAW devices can reach higher frequencies, at which LGS presents propagation losses. AlN-based SAW devices showed temperature coefficients of frequency (TCF) of -68 ppm/°C at 20°C and -80 ppm/°C at 500°C.

Although SAW-based devices are the preferred choice for high temperature applications due to the wireless interrogation possibility, they still present the drawback of the destructive agglomeration suffered by the long, thin and narrow strips of the interdigital transducers (IDTs) at such high temperatures. Many efforts have been devoted to the development of suitable metals for IDTs, being Pt and Ir alloys the best choice [5], [6]. Nevertheless, metal destruction at high temperatures still limits the power handling capabilities of such devices.

In this work we present an alternative to SAW-based devices, based on thin film bulk acoustic wave resonators (FBARs), namely AlN-based solidly mounted resonators (SMRs). As suggested by Aubert et al. [7], AlN-based SAW devices can be a promising alternative to LGS-based ones. Likewise, we propose to use AlN films but in this case in SMRs, to offer a solution to the power handling limitation in SAW devices and the possibility of operating at frequencies of several GHz. In FBARs the destructive agglomeration of the electrodes can be minimized by increasing their thickness at expenses of reducing that of the AlN film in order to keep the resonant frequency constant. This approach cannot be envisaged in SAW-based devices, since their resonant frequency is set by the IDTs pitch. The metal/AlN/metal sandwiched structure of FBARs allows then for high power handling. Additionally, if sensing applications others than temperature sensing are pursued, FBARs appear as the potential choice owing to the low TCF that can be achieved (<-10 ppm/°C).

In a previous work we presented 2.5 GHz AlN-based SMRs particularly designed to sustain temperatures as high as 700°C [7]. In this work we extend that study by testing two types of acoustic reflectors: (1) made of SiO₂/Mo, and (2) fully insulating reflectors made of SiO₂/AlN, and proving that these devices can sustain temperatures up to 1000°C.

II. METHODS

As mentioned, two types of acoustic reflectors have been used to fabricate the SMRs (Fig. 1). A partially conductive reflector composed of five alternating layers of SiO₂/Mo (620/629 nm-thick, respectively) (Fig. 1(b)), and a fully insulating one made of seven alternating layers of SiO₂/AlN (516/916 nm-thick, respectively) (Fig. 1(c)). The structure of the SiO₂/AlN reflector was, in some cases, modified to achieve a better TCF compensation by modifying the thickness of the outermost films. Since these structures are multilayered, special attention was paid to the sputtering deposition conditions of the materials. These were tuned to improve adhesion between layers and reduce stress to values below < 200 MPa. In this context, the first SiO₂ layer of both types of reflectors was thermally grown to improve adhesion with the underlying Si substrate. With the same purpose, a thin (15 nm-thick) sputtered Ti film was placed between each layer of the SiO₂/Mo reflector.
Fig. 1. Structure of the SMRs. (a) Optical top view of an actual device, (b) sketch of a device using a five layers SiO$_2$/Mo acoustic reflector and (c) sketch of a device using a seven layers SiO$_2$/AlN acoustic reflector.

To preserve the fully insulating nature of the SiO$_2$/AlN reflector the Ti interlayer was not employed in this case; this is crucial if SMRs with electrical extensions are needed [8]. On both types of reflectors we mounted a piezoelectric sandwich composed of a 120 nm-thick Ir bottom electrode, a 1 µm-thick AlN piezoelectric, and a 150 nm-thick Mo top electrode (Fig. 1). A thin Ti layer (15 nm-thick was added underneath the Ir bottom electrode). The piezoelectric AlN was deposited in an UHV sputtering system and its stress was controlled by lowering the bias applied to the substrate [9].

The finished SMRs were annealed at 1000 ºC for in HV (10$^{-6}$ Torr) for two hours. The frequency response of the devices was measured before and after the heat treatment. The electrical response of some SMRs made of partially conductive reflectors was also in-situ monitored during annealing, in this case at atmospheric pressure and temperatures up to 550ºC.

In order to check whether the films had experienced any inter-diffusion during the high temperature treatments, several SMRs, some of them as-grown and others subjected to the heat treatment, were assessed by Rutherford Backscattering Spectrometry (RBS) using a 5 MeV tandem accelerator. Since the technique only gives information of the first surface layers (around 3 to 5 µm, depending on the experimental conditions), in some cases several layers were removed before the measurement in order to assess deeper layers. The RBS spectra were measured using a 4He beam at 2 MeV impinging normally on the samples and detecting the backscattering ions with the surface barrier detector set at 170.5°. The spectra were fitted with a model to obtain the composition, atomic ratio and depth distribution of the layers composing the SMRs (SiO$_2$, AlN, Ir, Mo, Ti).

III. RESULTS AND DISCUSSION

A. TCF characterization of as-deposited SMRs

In a first step, we assessed the TCF of the fabricated devices at low temperatures (25°C to 100°C) by monitoring the real part of their electrical admittance, Re($\mathcal{Y}$) (i.e. the maximum of the peak). Table I shows the measured TCF values of devices with the regular SiO$_2$/Mo and SiO$_2$/AlN reflectors, and additionally that a TCF-compensated SiO$_2$/AlN reflector. In the latter the thicknesses of the first and second layers of the reflector were set to 1.3 µm for the SiO$_2$ and 700 nm for the AlN. The obtained values show that regular SiO$_2$/AlN reflectors display lower values of the TCF than SiO$_2$/Mo reflectors, due to the lower coefficient of thermal expansion of AlN (4.5·10$^{-6}$/°C), compared to that of Mo (4.8·10$^{-6}$/°C). Additionally, the TCF of the SMRs can be further reduced to -10 ppm/°C by using compensated SiO$_2$/AlN reflectors, without drastically worsening the device frequency performance.

<table>
<thead>
<tr>
<th>Acoustic Reflector</th>
<th>Compensation</th>
<th>TCF [ppm/°C]</th>
</tr>
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<tbody>
<tr>
<td>SiO$_2$/Mo</td>
<td>NO</td>
<td>-19 ± 1</td>
</tr>
<tr>
<td>SiO$_2$/AlN</td>
<td>NO</td>
<td>-16 ± 0.9</td>
</tr>
<tr>
<td>SiO$_2$/AlN</td>
<td>YES</td>
<td>-10 ± 1</td>
</tr>
</tbody>
</table>

B. Effects of the heat treatments

The electrical response of devices made with regular SiO$_2$/Mo and SiO$_2$/AlN reflectors, before and after annealing them in vacuum at 1000°C for two hours, are presented in Fig. 2. These results clearly prove that the electrical performance of the devices is not significantly affected after the high temperature treatment. Their quality factors are preserved as well as the piezoelectric activity of the AlN films (their electromechanical coupling coefficient barely varies).

![Fig. 2. Electrical impedances before and after 2h annealing at 1000°C of the SMRs made of (a) the SiO$_2$/Mo reflector and (b) the SiO$_2$/AlN reflector.](image_url)

The RBS spectra of three types of structures, with and without annealing treatment, are presented in Fig. 3. These structures are: the entire SiO$_2$/Mo reflector (Fig. 3(a)), the entire SiO$_2$/AlN reflector (Fig. 3(b)), and a complete device on a SiO$_2$/AlN reflector after having removed the Mo top electrode (Fig. 3(c)). By comparing the spectra of the acoustic reflectors (Fig. 3(a) and (b)) we can only detect inter-diffusion in the SiO$_2$/Mo reflector (Fig. 3(a)). These is evinced by the
Fig. 3. RBS spectra of three different samples (insets): (a) a complete SiO$_2$/Mo reflector, (b) a complete SiO$_2$/AlN reflector, and (c) a device without the Mo top electrode made of the SiO$_2$/AlN reflector.

A tiny change in the slope of the Mo signal appearing around channel 400 and corresponding to the first SiO$_2$/Mo interface. Adjusting the spectra yields interfaces widths of around 30 nm. In the case of the sample containing the Ir bottom electrode of Fig. 3(c), intermixing between the Ir bottom electrode and the Ti and SiO$_2$ underneath layers is clearly noticeable.

It is important to remind that the as-deposited and heat-treated samples assessed by RBS are actually different dice taken from the same silicon wafer. The different widths displayed by some signals in the spectra cannot be attributed to inter-diffusion effects arising from the thermal treatments, but rather to inhomogeneity in the thickness of the different films across the 100 mm silicon wafer resulting from the sputtering process.

Both, the lack of homogeneity and the inter-diffusion effects in the acoustic reflectors do not significantly affect the performance of the SMRs after the high temperature treatments.

C. In-situ characterization at high temperatures

The in-situ electrical characterization of SMRs is performed at atmospheric pressure, hence the devices being passivated with a thin SiO$_2$ capping layer, and it requires a specific set-up since regular RF cables cannot be used. In this work we present a first home-made set-up for this scope (Fig. 4). It is composed of:

- An open oven with a thermocouple temperature controller (Fig. 4(a)).
- A home-made rigid 50 ohms coaxial RF cable made of a 1.5 mm in diameter Cu core, a quartz tube forming the dielectric filling, and a 4 mm inner diameter stainless steel external tube (Fig. 4(b)). The Cu core and the exterior tube were welded to a 1 mm in diameter Au probes for the signal and ground contacts, respectively. The contact to the device was achieved by bonding the device to a 50 ohms Au co-planar micro strip. The device was placed on a 5 mm-thick ceramic support (Figs. 4(c) and (d)).
- A portable NA (Keysight Fieldfox N9912A) connected to the rigid RF cable through a regular low loss flexible one (Fig. 4(a)). It offers a simple calibration option for non-standard adapters (in this case the home-made RF cable) to connect the device under test.

Fig. 4. Measurement set-up for the high temperature in-situ characterization of SMRs: (a) whole set-up composed of the open oven, the home-made rigid RF cable connected to the low loss flexible one, and the portable NA; (b) close view of the rigid RF cable; (c) rigid cable holder where its Au probes are connected to an Au coplanar micro-strip to which the device is bonded, and (d) close view of the bonded devices where the SiO$_2$ capping layer is observed.

Fig. 5 shows the in-situ frequency response of SMRs using SiO$_2$/Mo reflectors up to 550ºC. While increasing the temperature, parasitic effects, which we attribute to variations of the rigid cable and set-up materials properties, appear. These parasitic effects can be cleaned by employing the port-extensions compensation option of the portable NA. Parasitic effects are, to a certain extent, cleaned in these measurements, however, above 450ºC the signal starts suffering severe degradation. This is attributed to the contact loss that appears at those temperatures between the Al wire
of the ultrasonic bonding we needed to use and the top electrode of the device. The best wire materials found in the literature for high temperature bonding are Pt or Inconel [30], [32], however we did not have the means for using them in this work. In all cases, a good adhesion between these metal wires and the top electrode should be guaranteed.

Despite the signal degradation above 450°C, the extracted shifts in resonant frequency (Fig. 6) are in good agreement with the TCF calculated for this type of device at low temperatures (Table I).

IV. CONCLUSIONS

We have designed AlN-based SMRs with both, SiO₂/Mo and SiO₂/AlN reflectors, for operation at very high temperatures and proved that they can sustain up to 1000°C. Their electrical characterization before and after annealing them for 2h proved that their integrity and performance is maintained. RBS characterization proved that inter-diffusion between the device layers only occurs in SiO₂/Mo reflectors and between the Ir bottom electrode and the thin Ti layer underneath. Both SiO₂/Mo and SiO₂/AlN reflectors provide the devices with TCF of -19 ppm/°C and -16 ppm/°C, respectively. These can be improved by TCF compensation to a value of -10 ppm/°C without drastically compromising the device performance.

Additionally, we have performed in-situ electrical characterization of SMRs composed of SiO₂/Mo reflectors up to a temperature of 550°C. This temperature limitation was imposed by our home made measurement set-up, whose improvements are ongoing. Nevertheless, we have proved that the TCF of this devices is preserved also at very high temperatures.

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REFERENCES