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Iridium interdigital transducers for high-temperature surface acoustic wave applications

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Abstract— Iridium is investigated as a potential metal for interdigital transducers (IDTs) in surface acoustic wave (SAW) devices operating at high temperatures. SAW delay lines based on such IDTs and langasite (LGS) substrate are fabricated and electrically characterized. The results show reliable frequency responses up to 1000°C. The strong increase of insertion losses beyond this temperature, leading to the vanishing of the signal between 1140 and 1200°C, is attributed to surface transformation of LGS crystal, consisting in relevant gallium and oxygen losses, as evidenced by scanning electron microscopy, energy-dispersive X-ray spectroscopy and secondary ion mass spectroscopy.

Keywords-high temperature; SAW; langasite; iridium

I. INTRODUCTION

In the last fifteen years, surface acoustic waves (SAW) devices have paid a lot of attention regarding the possibility to make wireless sensors working at high temperature (up to 1000°C) using this technology. Indeed, SAW devices are passive components requiring no embedded electronics neither power source to be wirelessly interrogated [1]. The choice of the constitutive materials is critical regarding the fabrication of such sensors. Concerning the piezoelectric substrate, langasite ($\text{La}_3\text{Ga}_5\text{SiO}_{14}$ or LGS) is currently the most appropriate candidate. In fact, it has been intensively studied, demonstrating a good stability at high temperature, while being commercially available. In particular, Hornsteiner *et al.* recorded a SAW signal on LGS at temperatures as high as 1085°C for some minutes [2]. Another remarkable result was obtained by Da Cunha *et al.* who successfully operated LGS-based SAW devices for more than 5½ months at 800°C

[3]. Regarding the electrodes, numerous studies have shown the limitations of pure Pt thin films which experiment agglomeration phenomena from 700°C on, whatever be the nature of the adhesion layer [4-6]. Several successful strategies were proposed by Da Cunha *et al.*, such as the use of a Pt/10%Rh alloy or co-deposited Pt/10%Rh/ZrO₂ composite, to avoid or at least slow down this process which turns the electrodes into a collection of separate beads [3]. They all help hinder self-diffusion of Pt which is the microscopic mechanism leading to agglomeration, by adding some well-chosen impurities into the Pt layer. Another way to obtain similar results might come from the use of more stable metals than Pt from a physical point of view, which means metals with a higher melting point and thus lower self-diffusion coefficients [7]. Qualitatively, volume diffusion phenomena, which lead to complete agglomeration in a matter of hours or less, are known to start around the Tammann temperature, which is defined as half the melting temperature of the material (both expressed in Kelvin) [5,8]. In this context, iridium could be a material of first choice as it is a noble metal as platinum, with a melting temperature almost 700°C higher (2440°C versus 1770°C), which means a Tammann temperature close to 1100°C against 750°C in the Pt case. The main drawback of Ir is its tendency towards oxidation above 700°C in oxidizing environments [9]. Recently, we confirmed the superiority of Ir to Pt with respect to agglomeration phenomena, as Ir interdigital transducers (IDT) on LGS substrate showed great stability after an annealing of 20h at 900°C under vacuum (5.10⁻⁵ mbar of pressure), contrary to Pt ones [5].

In this letter, we study Ir/Ti/LGS SAW delay lines at temperatures going from the ambient to 1200°C, by in-situ radio-frequency electrical measurements, coupled to scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDXS) and secondary ion mass spectroscopy (SIMS).

II. EXPERIMENTAL

To perform IDTs, 10 nm thick titanium as adhesion layer and 100 nm thick iridium films as electrodes were deposited by e-beam evaporation onto Y-cut LGS substrates (Witcore Company, Jinan, China). SAW delay lines were then fabricated by photolithography and ion beam etching. The devices were constituted of two identical IDTs, with 50 pairs of fingers and a wavelength λ fixed to 14 μm . The IDT center spacing,

acoustic aperture, and finger width to space ratio were 65λ , 70λ , and 1:2, respectively. The SAW propagation path was along the X direction. The S_{21} parameters of these devices were recorded between room temperature and 1200°C under vacuum conditions (pressure: $2 \cdot 10^{-4}$ mbar). To do so, a specific setup was used [10]. The morphology of the device surface after high-temperature exposure was observed by SEM (Jeol 6500F), while the corresponding chemical composition was obtained by EDXS and SIMS (Cameca IMS 7F).

III. RESULTS AND DISCUSSION

Taking into account the high Tammann temperature of Ir and the previous successful tests at 900°C [5], the first runs were focused on the determination of the highest operating temperature of the Ir/Ti/LGS SAW devices. The temperature was then increased as fast as possible (temperature ramp: $100^{\circ}\text{C}/\text{h}$) to the maximum temperature reachable in the oven (1200°C). Figure 1 shows the measured S_{21} frequency response at different temperatures. One can observe that insertion losses are quite constant up to 1000°C , with variations below 3 dB. Beyond this temperature, they increase quickly, inducing the loss of SAW signal after 1140°C . Consequently, as shown in figure 2, some other samples were heated from room temperature to 1050°C , just below the Tammann temperature of Ir, and then withstood a plateau at this level for several hours

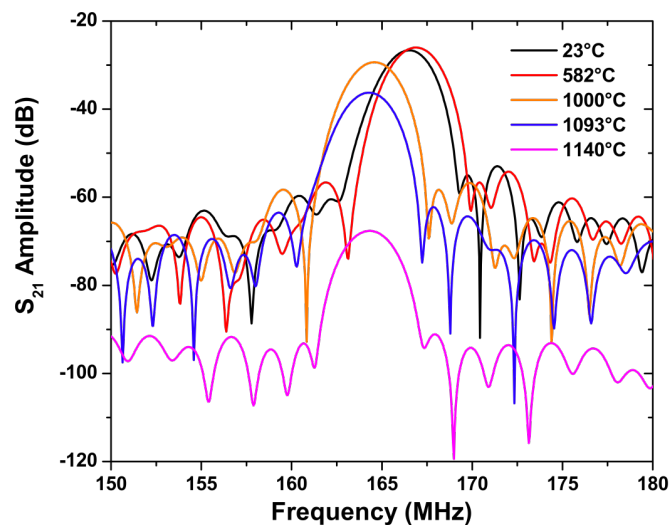


Fig. 1. Ir/Ti/LGS SAW delay line frequency response (S_{21} magnitude) at different temperatures

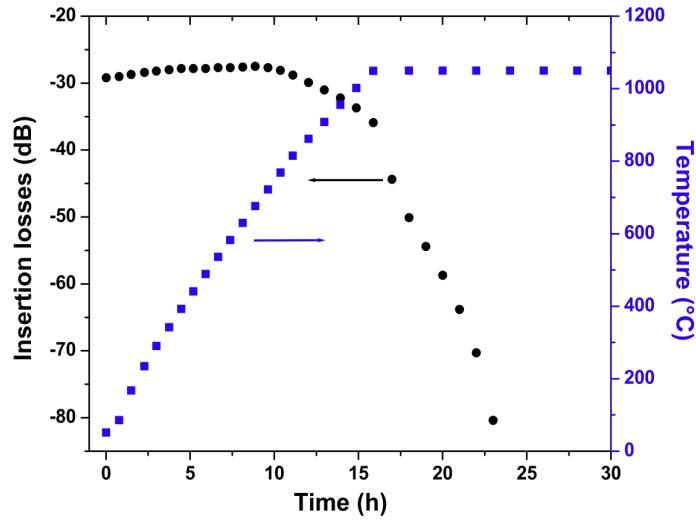


Fig. 2. Evolution of delay lines insertion losses with time (circles) and temperature profile (blue squares)

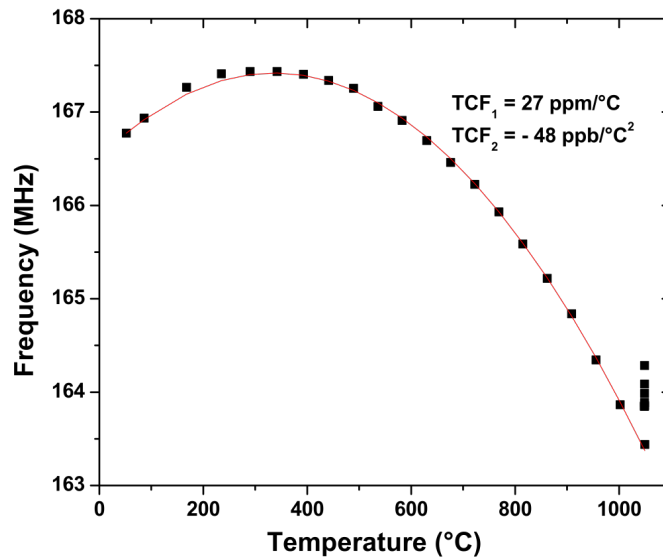


Fig. 3. Evolution of SAW delay lines central frequency with temperature

Figure 3 shows the corresponding frequency-versus-temperature curve. The latter is close to perfect parabola with temperature coefficients equals to $+27 \text{ ppm}/^\circ\text{C}$ and $-48 \text{ ppb}/^\circ\text{C}^2$ for the first and second order respectively (reference temperature: 52°C). Quick aging of devices during this plateau can be deduced from the strong frequency drift visible on Fig. 3, coupled with an increase of the insertion losses at a rate of 6 dB/h , leading to the vanishing of the signal after 8h at 1050°C (Fig. 2).

SEM images performed on heated samples after both electrical characterizations revealed

comparable damages. In addition to showing a significant increase in roughness (Fig. 4a), langasite crystal surface also has numerous cracks (Fig. 4b). The chemical contrast between the substrate and the IDTs is very low, indicating likely the vanishing of the iridium thin film (Fig. 4b). EDXS confirmed this hypothesis, as only traces of Ir can be found on finger electrodes initial position (Tab. I). The presence of manganese and iron can also be noted. These elements are probably coming from the steel mantle of the RF cables used to interrogate devices. Last but not least, EDXS indicates that langasite crystals underwent huge losses in gallium. Such crystal degradation in vacuum was recently reported by Bardong *et al.* [11]. Schulz *et al.* observed as well this effect in air atmosphere, by surface exchange measurements, and attributed its origin to high Ga vapor pressure of gallium-containing compounds at high temperatures [12].

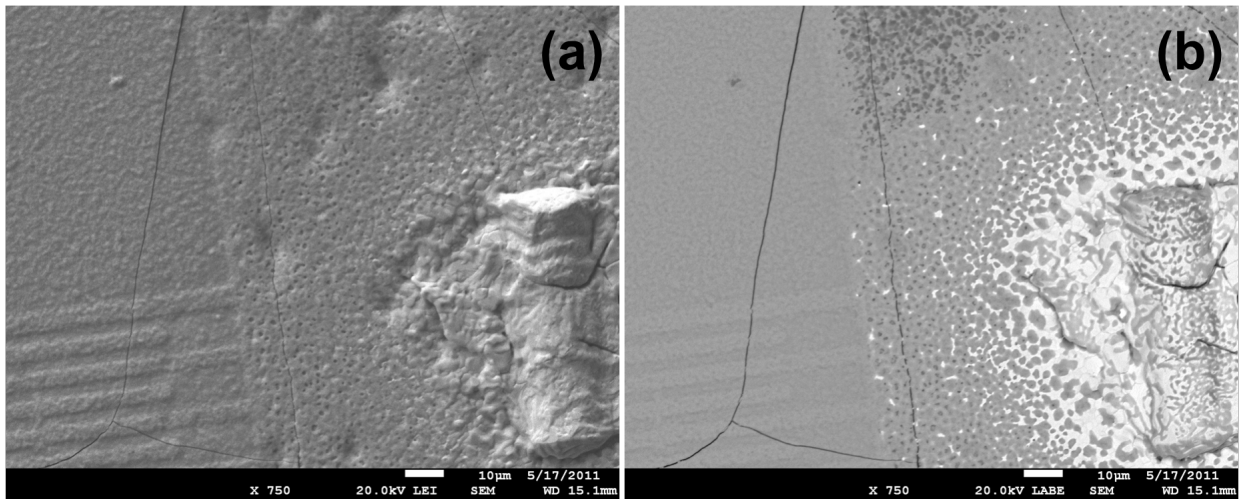


Fig. 4. SEM images of an Ir/Ti/LGS SAW device after 60h at 1050°C under vacuum conditions ((a) topography/(b) chemical contrast). IDT finger width is 2.3 µm. On the right half of the image is the bus bar on which the tip of a bonded Pt microwire is clearly visible (bottom-right corner).

Tab. I. EDXS chemical composition (atomic percentage) of the surface of an Ir/Ti finger electrode on LGS substrate before and after 60h at 1050°C under vacuum conditions. O levels are not indicated since the Ir thin layer seriously interferes with the detection of this light element in the as-prepared sample

Chemical element	La	Ga	Si	Ir	Ti	Mn	Fe
Before annealing	12.3	20.0	3.8	7.1	0.8	0	0
After 60h at 1050°C	14.8	0.9	10.3	0.9	0	3.9	0.7

SIMS measurements clarified this result, showing that there exists a Ga-depleted layer at the surface of the crystal, which is around 400 nm thick (Fig. 5). Moreover, SIMS also highlights significant losses of oxygen. Contrariwise to the Ga case, this lack seems not only to be located at the surface vicinity, but is quite homogeneous all along the 2 μm thick analyzed layer. This difference could be related to the fact that the diffusion coefficient of O in langasite is four orders of magnitude higher than the one of Ga [12], which involves a much smaller time for O to reach equilibrium than for Ga. Such oxygen losses in langasite and more generally in oxide crystals have been intensively studied as they have important consequences on the conductive properties of such crystals [13]. Thus, in poor-oxygen environment, the following reduction reaction takes place, under which double charged oxygen vacancies and compensating electrons are formed in the crystal [14].



(in accordance with the Kröger and Vink notation [15])

This model allows explaining easily the vanishing of the IDTs. Indeed, it appears that because of that crystal decomposition, the iridium thin film is permanently in close contact with oxygen-rich environment, which leads, at such temperatures, to its oxidation in the highly volatile IrO_3 [16].

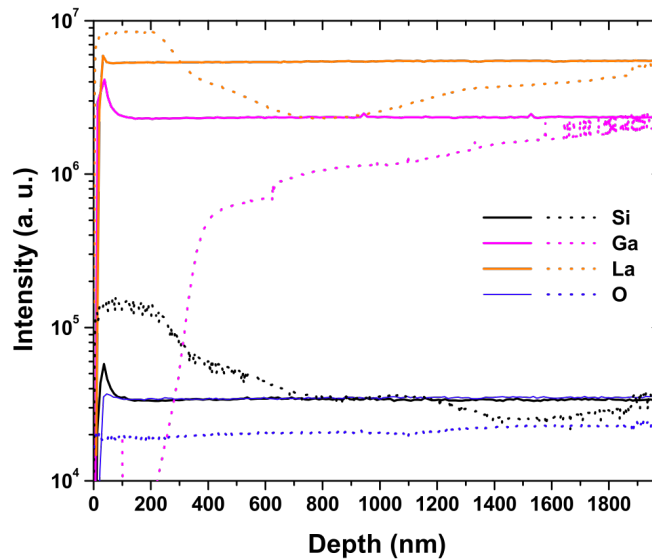


Fig. 5. SIMS depth profile of LGS substrate. Solid and dotted lines refer to the same sample respectively before annealing and after 60h at 1050°C under vacuum conditions

IV. CONCLUSION

In summary, Ir/Ti/LGS SAW devices have been successfully operated at high temperatures up to 1140°C. They also showed a lifetime of more than 7h at 1050°C. Combination of SEM, EDXS and SIMS methods allowed attributing the devices failure to surface chemical transformation of langasite crystals, consisting in oxygen and gallium losses. In addition to altering the substrate properties, it also induces the oxidation of Ir electrodes, leading to their evaporation. Such troubles could be avoided by the use of non-oxide substrate, such as aluminium nitride, which has been shown to be a good candidate for such applications [17]. In that case, Ir could be a first-choice material to make IDTs for SAW applications at high temperatures up to 1000°C. Another possibility to overcome those limitations could come from the use of an Ir/Rh alloy which is potentially usable in air atmosphere, where langasite is expected to be more stable [18].

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