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Behavior of Platinum/Tantalum as Interdigital Transducers for SAW Devices in High Temperature Environments

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Abstract—In this paper, we report on the use of tantalum as adhesion layer for platinum electrodes used in high-temperature SAW devices based on langasite substrates (LGS). Tantalum exhibits a great adhesive strength, and a very low mobility through the Pt film, ensuring a device lifetime at 900°C of about one hour in air atmosphere and at least 20 hours under vacuum. The latter is limited by morphological modifications of platinum, starting by the apparition of crystallites on the surface, and followed by important terracing and breaking of the film continuity. Secondary neutral mass spectroscopy (SNMS), Auger electron spectroscopy (AES), X-ray diffraction (XRD) measurements, as well as the comparison with iridium-based electrodes allowed us to show that this deterioration is likely intrinsic to platinum film, consisting in agglomeration phenomena. Finally, based on these results, we present a solution that could enhance significantly the lifetime of Pt-based IDTs placed in high-temperature conditions.

Keywords-high temperature; platinum; SAW; langasite; tantalum; iridium

I. INTRODUCTION

It is now well established that SAW devices can be used as pressure, gas or temperature sensors [1,2]. Those devices are particularly interesting for applications in harsh environments where no electronics can survive. Indeed, because they can be remotely powered and interrogated, embedded electronics is not required for wireless applications. Many industrial sectors, such as aeronautics, metallurgy, automotive, are looking forward those small, robust and wireless devices [3,4]. Efforts are now focused on selecting materials able to withstand harsh conditions, such as high temperatures up to 1000°C.

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3 Regarding to the substrate, several studies have shown that langasite ($\text{La}_3\text{Ga}_5\text{SiO}_{14}$) is well suited for
4 this kind of applications [5]. It is only limited by its relative important propagation losses, especially at
5 high frequencies, making it unsuitable for applications above 1 GHz under high-temperature conditions
6 [6], which restrains its use for wireless applications to the 434 MHz ISM band or, in the best case to the
7 869 MHz one, excluding the more promising band at 2.45 GHz. Now, the main challenge is to find a
8 reliable combination of materials to realize an IDT structure with high stability and good performances.
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12 Up to now, most of the studies have been conducted on platinum electrodes [3,4]. Indeed, this noble
13 metal has a relatively high melting temperature (1773°C), and the largest chemical inertness among the
14 noble metal family, essential at high temperature. However, this property has a drawback: the consequence
15 of the platinum weak chemical affinity for others elements is a very poor adhesion to oxide substrates such
16 as quartz or langasite. The solution consists in using a very thin (10 to 20 nm) intermediate layer between
17 the substrate and the platinum film, so-called “adhesion layer”. It must be constituted by a refractive metal,
18 more reactive than platinum, such as titanium, zirconium, tantalum, etc. Bonding between the adhesion
19 layer and the platinum film is ensured by alloying [7].
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35 Several studies have shown the limitations of titanium as adhesion layer at temperatures above 600°C
36 [8]. The diffusion of titanium in the platinum film is actually large, leading to the formation of oxide
37 precipitates (TiO_2) in the Pt grain boundaries. The consequences are the modification of the electrical
38 properties of the IDTs and the progressive vanishing of the adhesion layer leading to delamination.
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44 Pt/Zr combination has shown far better performances until 700°C . In particular, AES (Auger electron
45 spectroscopy) measurements have highlighted that Zr has not diffused at all through Pt after 8h of
46 annealing at 700°C [9]. Nonetheless, it has been shown that this combination is inadequate for continuous
47 operation beyond about 700°C , because of an important degradation attributed to de-wetting phenomena
48 [5].
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55 More recently, Da Cunha *et al.* have studied alternative solutions, replacing platinum by a Pt/10%Rh
56 alloy, while maintaining the adhesion layer of zirconium [5]. This combination exhibited a very good
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3 stability until 950°C, which is a considerable progress. This stability was even slightly improved by co-
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6 depositing the alloy with ZrO₂, and by using a SiAlON protective over-layer [10].
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8 In this work, our purpose is to investigate tantalum as adhesion layer for platinum electrodes. This
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10 combination has only been studied yet in other fields than SAW applications [11]. Maeder *et al.*, in
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12 particular, have noted that its performance is very close to that of zirconium after an annealing of 10
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14 minutes in a low pressure O₂ atmosphere at 620°C [12]. The second aim of this study is to understand the
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16 mechanisms leading to the potential deterioration of Pt electrodes, in order to consider appropriate
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18 solutions.
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20 21 22 II. EXPERIMENTAL 23

24 Platinum and tantalum thin films were deposited on Y-X (Euler angle: 0°, 90°, 0°) langasite substrates
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26 (Witcore Co. Ltd.) by sputtering method in argon gas (pressure: 10⁻² mbar), under a DC power discharge of
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28 60 W for Ta and 15 W for Pt. The thickness of the adhesion layer was 10 nm, while that of platinum film
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30 was 100 nm. SAW delay lines operating at 97 or 167 MHz were then processed using photolithography
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32 and ion beam etching (IBE). All devices were then electrically characterized with a probe station before
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34 and after annealing. These were performed in a tube furnace under air atmosphere or vacuum (5.10⁻⁵ mbar
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36 of pressure), at 900°C for a duration varying between 30 minutes and 20 hours. The samples were quickly
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38 inserted in the pre-heated furnace, and pulled out of the hot zone as fast as possible. Langasite substrates
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40 have shown great resistance to this thermal shock treatment. Some samples with Ir/Ti electrodes (which
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42 means 10 nm of titanium as adhesion layer and 100 nm of iridium as electrode) were also fabricated to
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44 compare their behavior to that of Pt/Ta IDTs.
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48 The structure and the morphology of the thin metal films were studied by X-ray diffraction (XRD) and
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50 scanning electron microscopy (SEM). Compositional depth profiles were obtained by secondary neutral
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52 mass spectroscopy (SNMS) and AES. These analyses were conducted in the bus bar region, to avoid
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54 averaging effects. The dimensions of the holes were 200 x 200 μm in the SNMS case and 12 x 12 μm for
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AES. The latter has in addition given us the chemical mapping of the surface of our devices. The size of the probe was close to 50 nm.

III. RESULTS AND DISCUSSION

A. Air annealing

First of all, the adhesion of the Pt/Ta films to the substrate was very good at room temperature as well as after high-temperature treatment. The frequency response shows no deterioration after an annealing at 900°C for 30 minutes. We can even note a slight decrease in the insertion losses (Fig. 1). SEM images have confirmed this result, revealing very limited modifications, which consist in the apparition of crystallites at the surface of the platinum film (Fig. 2).

To study the limitations of this kind of device at high temperature, we made it undergo an annealing process at 900°C for 2 hours. The result was no appeal: the SAW signal vanished while SEM images revealed that the crystallites have almost disappeared, leaving a substantial terracing and some broken IDTs fingers, which explains the loss of the SAW signal (Fig. 3). In order to understand the origin of this problematic limitation, some Pt/Ta/LGS samples were investigated after heating treatment, by SNMS (Fig. 4), AES (Fig. 5 and Fig. 6) and XRD (Fig. 7).

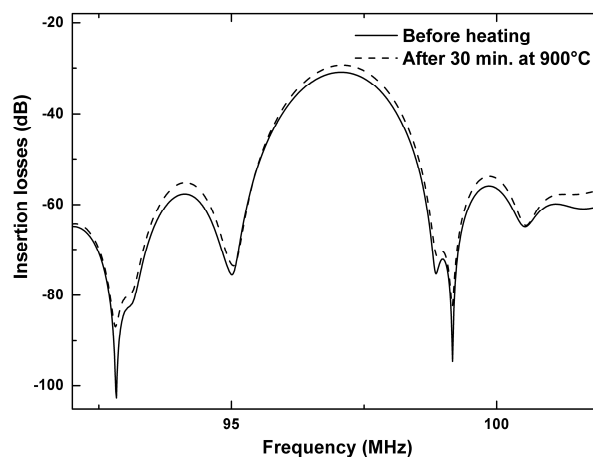


Fig. 1. Frequency response ($|S_{21}|$) of a Pt/Ta/LGS SAW device before and after annealing for 30 min. at 900°C

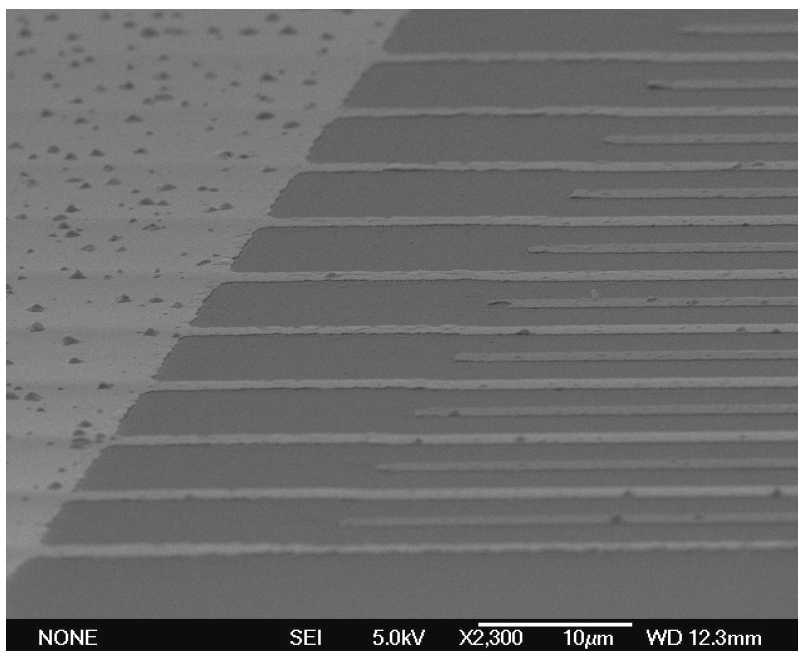


Fig. 2. Grazing SEM image of a Pt/Ta IDT after 30 min. at 900 °C

SNMS depth profile curves point out that there is no significant diffusion of tantalum through the Pt film during heating, while it moves quite well into the LGS substrate at such high temperatures (Fig. 4). It would be interesting to re-evaluate this phenomenon with a langatate ($\text{La}_3\text{Ga}_{5.5}\text{Ta}_{0.5}\text{O}_{14}$) substrate, which could act as a barrier towards tantalum, as it already contains a certain amount of this element. SNMS measurements also reveal that the amount of oxygen in the Ta layer increase significantly during heating, which likely means that **it has been oxidized**. AES measurements confirm these results (Fig. 5). Last, it seems that there is no oxygen in the platinum film, as well as at its surface as one can see on the AES cartography of the IDTs given for platinum and oxygen (Fig. 6): none of our analyses evidence the formation of platinum suboxides as described by Stancovski *et al.* [13]. Thus, we can assume that neither tantalum nor oxygen play an essential role in the deterioration of the Pt layer, implying that this phenomenon is likely intrinsic to platinum: whatever be the nature of the adhesion layer and the ambient gas mixture, it will take place.

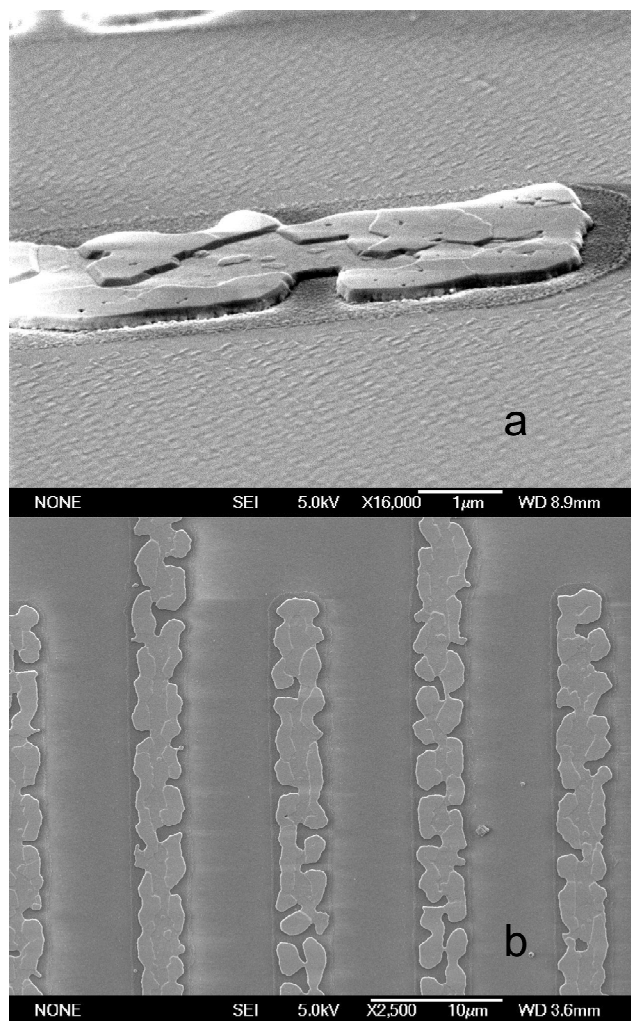


Fig. 3. SEM images of a Pt/Ta IDT after an annealing of 2 hours at 900°C: grazing view of a finger tip (a) and plane view of several finger electrodes (b)

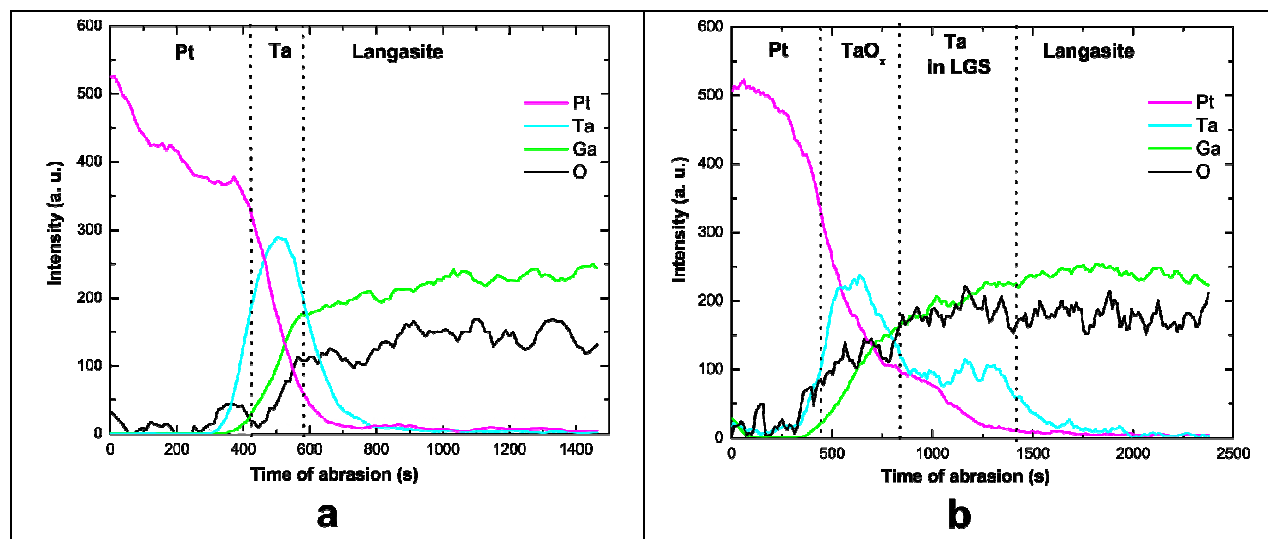


Fig. 4. SNMS depth profile of a Pt/Ta/LGS sample before annealing (a) and after 30 min. at 1000°C (b)

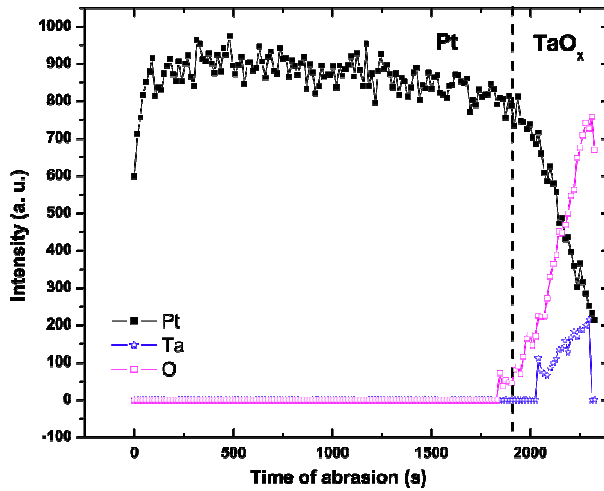


Fig. 5. AES depth profile of a Pt/Ta/LGS sample after 2h at 900°C (up to 1800s, the amount of oxygen is below the detection threshold which is about 3% for the atomic concentration).

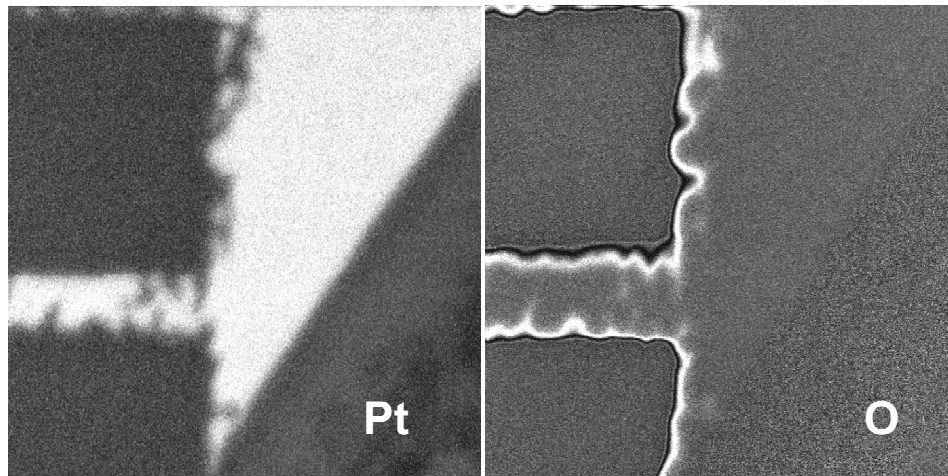


Fig. 6. AES surface mapping of a Pt/Ta IDT after 2 hours at 900°C. Bright zones indicate the presence of the element. The right-bottom corner is covered with silver paint

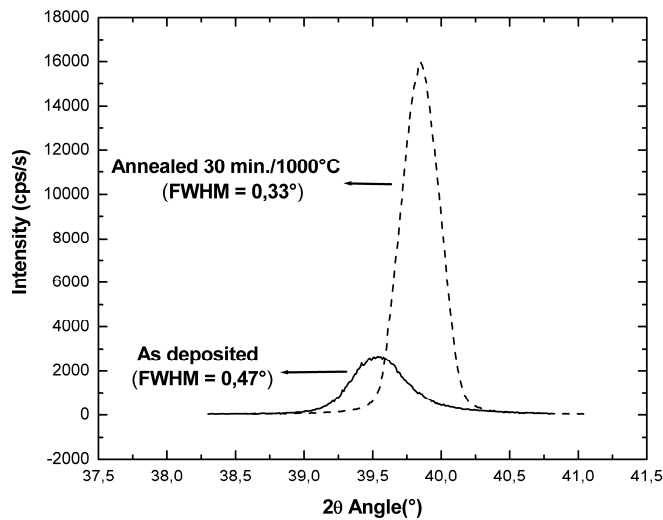


Fig. 7. Pt (111) XRD peak before and after 30 min. at 900°C

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3 XRD measurements strengthen this hypothesis, by showing the modification of the microstructure of
4 the Pt thin film under heating. θ - 2θ scan points out a strong (111) orientation of the platinum films, which
5 is a conventional result for FCC metals, like Pt [14]. The important fact is that the full width at half
6 maximum (FWHM) of this (111) Pt peak decreases significantly while its intensity increases after
7 annealing (Fig. 7). It is well known that the width of diffraction peaks decreases while increasing the size
8 of grains, so we can conclude that the Pt layer strongly recrystallized during annealing, and that the
9 apparition of the big crystallites is a spectacular but instable manifestation of this phenomenon. The slight
10 decrease in the insertion losses observed after annealing (Fig. 1) could be related to this grain growth
11 resulting in fewer grain boundaries and thus better conductivity of the Pt film. Another interesting
12 information is given by the shift of this (111) Pt peak if one compares its position before and after
13 annealing. The peak of as-deposited films is at a slightly lower angle of diffraction ($39,538^\circ$) than its
14 theoretical value ($39,763^\circ$), which means that the interplanar value d_{111} of these films is larger than the bulk
15 one. This indicates that the Pt layer is slightly in compression in the plane of the film. A likely reason for
16 this compressive stress is peening, by which the subsurface of a sputtered film is compressed due to
17 bombardment by reflected neutral atoms [14]. This compressive stress is then removed during the
18 recrystallization process which leaves a completely relaxed film at the temperature of annealing. To the
19 opposite, the peak after annealing has shifted to a much higher value ($39,850^\circ$). This can be explained by
20 the difference of the thermal expansion coefficient α between Pt ($8,8 \cdot 10^{-6} \text{ K}^{-1}$) and LGS ($5,6 \cdot 10^{-6} \text{ K}^{-1}$),
21 which induces, after cooling down at a high rate a strain of the film and an associated “thermal” stress.
22 Here, as α_{LGS} is lower than α_{Pt} , we can deduce that the Pt film is stretched in the plane, implying a lower
23 d_{111} value than for the bulk, and thus a higher diffraction angle than the theoretical value.
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49 Nonetheless, the recrystallization phenomena, alone, cannot explain the important deterioration of the
50 Pt film leading to the break of the IDT fingers. Indeed, in Fig. 8, platinum grain size, derived from FWHM
51 of (111) Pt peak via the Scherrer formula [14], is plotted versus the annealing duration. This figure shows
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3 that the recrystallization is a “fast” phenomenon, which is finished in less than 15 minutes, whereas the
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5 break of the electrodes requires much more than 30 minutes as Fig. 2 attests.
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8 It is important to note that as our Pt films are polycrystalline, the total surface of grains is very large,
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10 and surface diffusion mechanisms probably governs recrystallization phenomena. This hypothesis agrees
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12 with the rule of thumb stating that the onset of surface diffusion is about the Hüttig temperature (T_H). T_H is
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14 defined relatively to the material melting temperature (T_m) by $T_H = 0.3 T_m$ (temperatures are expressed in
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16 Kelvin). For platinum, T_H is about 410°C, well below the temperature considered in this study, i.e. 900°C.
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18 Actually, at such high temperatures, bulk mobility is also activated as these mechanisms are known to
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20 become significant over the Tammann temperature (defined as $0.5T_m$), which value for Pt is about 750°C
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22 [14]. Thus, whereas these experiments are conducted at temperatures well below the melting temperature
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24 of platinum, thermal energy is sufficient to generate driving forces which will work towards reducing the
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26 source of instability of any thin film: its enormous surface to volume ratio [11,15]. Surface area can be
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28 reduced through mass transfer by the diffusion of the platinum nanocrystallites. If this phenomenon goes to
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30 its end, the nanocrystallites agglomerate to form a collection of separate beads, which is the configuration
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32 with the smallest surface to volume ratio (Fig. 9). The break of the electrodes that we have observed after 2
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34 hours of annealing at 900°C is likely a consequence of the first movements of agglomeration (Fig. 3).
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39 *B. Vacuum annealing*

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41 In order to assess our hypothesis stating that the deterioration of the platinum electrodes is a
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43 consequence of agglomeration phenomena, we have conducted a new series of annealing at 900°C under
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45 vacuum on two kinds of devices: the same as above with Pt/Ta electrodes, and others with Ir/Ti electrodes.
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47 Iridium was chosen as it is a noble metal like platinum with a higher melting temperature of 2440°C,
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49 compared to 1769°C for platinum. Thus, for iridium, the Hüttig temperature is about 630°C, while the
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51 Tammann temperature is about 1080°C. This implies that iridium should undergo some recrystallization at
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53 900°C, but no agglomeration (or at a very slow rate compared to platinum). These experiments have to be
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3 conducted in vacuum as above 700°C, iridium slightly oxidizes in surface, and this oxide is highly volatile
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5 [16].
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8 As expected, XRD measurements show that our iridium films are (111) oriented too, and confirm their
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10 recrystallization from 700°C (Fig. 10).
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13 Fig. 11 shows a morphological comparison between Pt/Ta and Ir/Ti electrodes after an annealing of 20
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15 hours at 900°C in vacuum, at a pressure of $5 \cdot 10^{-5}$ mbar. While early manifestation of agglomeration can be
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17 seen in the platinum case, there is no trace of it on iridium, which is coherent with our hypothesis. This
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19 result also points out the high potential of iridium for such applications and qualifies it as a very promising
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21 candidate for the future.
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24 Another important observation is related to the kinetic of the deterioration in vacuum, which is well
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26 lower than in air atmosphere. Whereas this deterioration is well advanced in air after 2h at 900°C, inducing
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28 the loss of the SAW signal, an annealing in vacuum at the same temperature for 20h has a very limited
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30 impact on the frequency response (Fig. 12). This fundamental difference is very difficult to explain on the
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32 basis of our results. According to our interpretation of the deterioration, it should be related to a difference
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34 in the mobility of the moving species. From there, two hypotheses can be proposed to explain this
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36 phenomenon. First, it could be related to a better mobility of the Pt nanocrystallites on **tantalum oxide** than
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38 on Ta [14]. This would mean that the nature of the adhesion layer, as well as that of the surrounding
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40 atmosphere, impact the kinetic of the agglomeration phenomena. The second explanation could come from
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42 the formation of metastable platinum oxides which would be more mobile than platinum.
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47 Anyway, this result confirms that a simple solution to significantly extend the lifetime of Pt-based
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49 electrodes could come from the use of an over-layer. This would play a dual role: in addition to ensuring
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51 its natural protective role (particularly with respect to oxygen), it could allow to mechanically fix the Pt
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53 nanocrystallites, thereby preventing their agglomeration. Indeed, as it can be clearly seen on Fig. 3, the
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55 agglomeration phenomena initially take place at the tip of the platinum film, where the nanocrystallites
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57 undergo fewer forces from their neighbors, and thus where they are less mechanically fixed.
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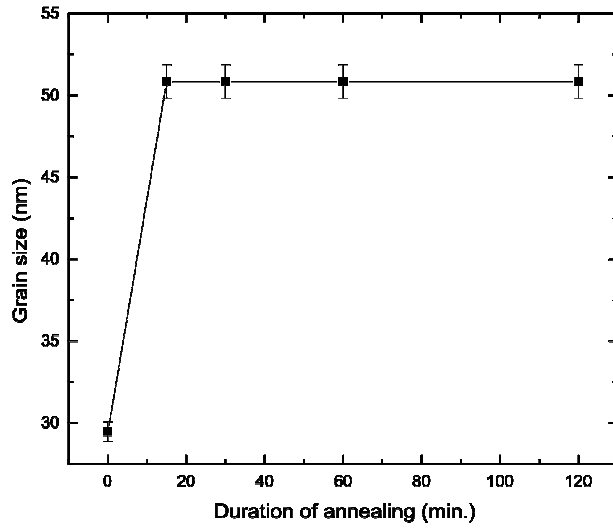


Fig. 8. Platinum grain size versus duration of annealing at 900°C

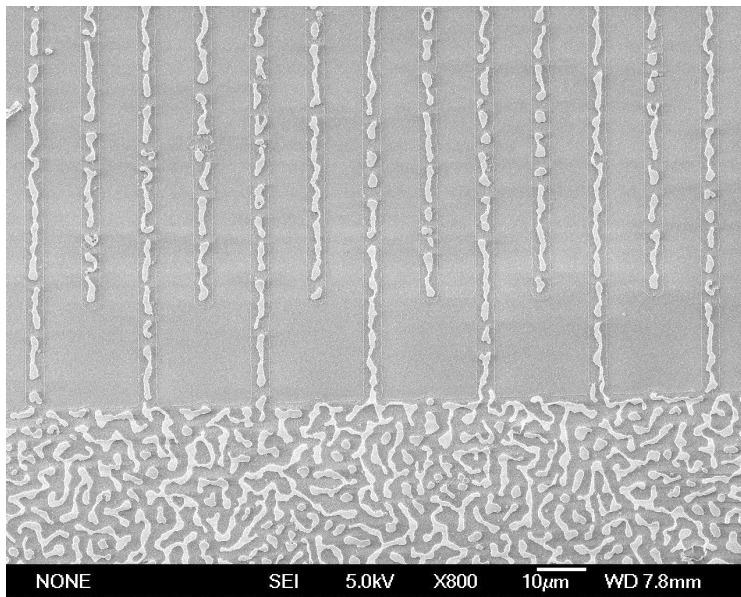


Fig. 9. SEM image of a Pt/Ta IDT after 6h at 1000°C

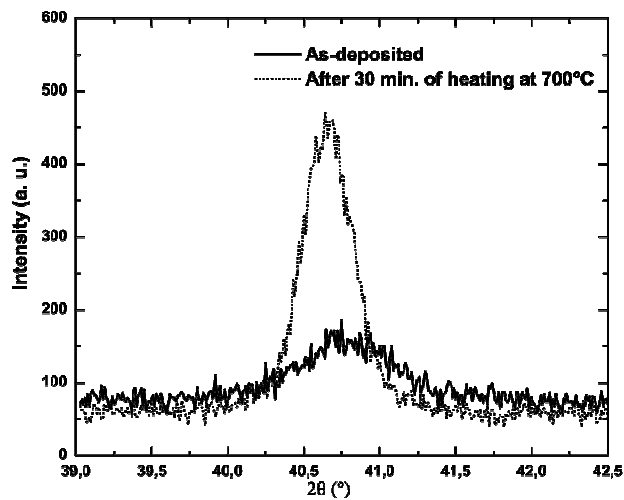


Fig. 10. Ir (111) XRD peak before and after 30 min. at 700°C

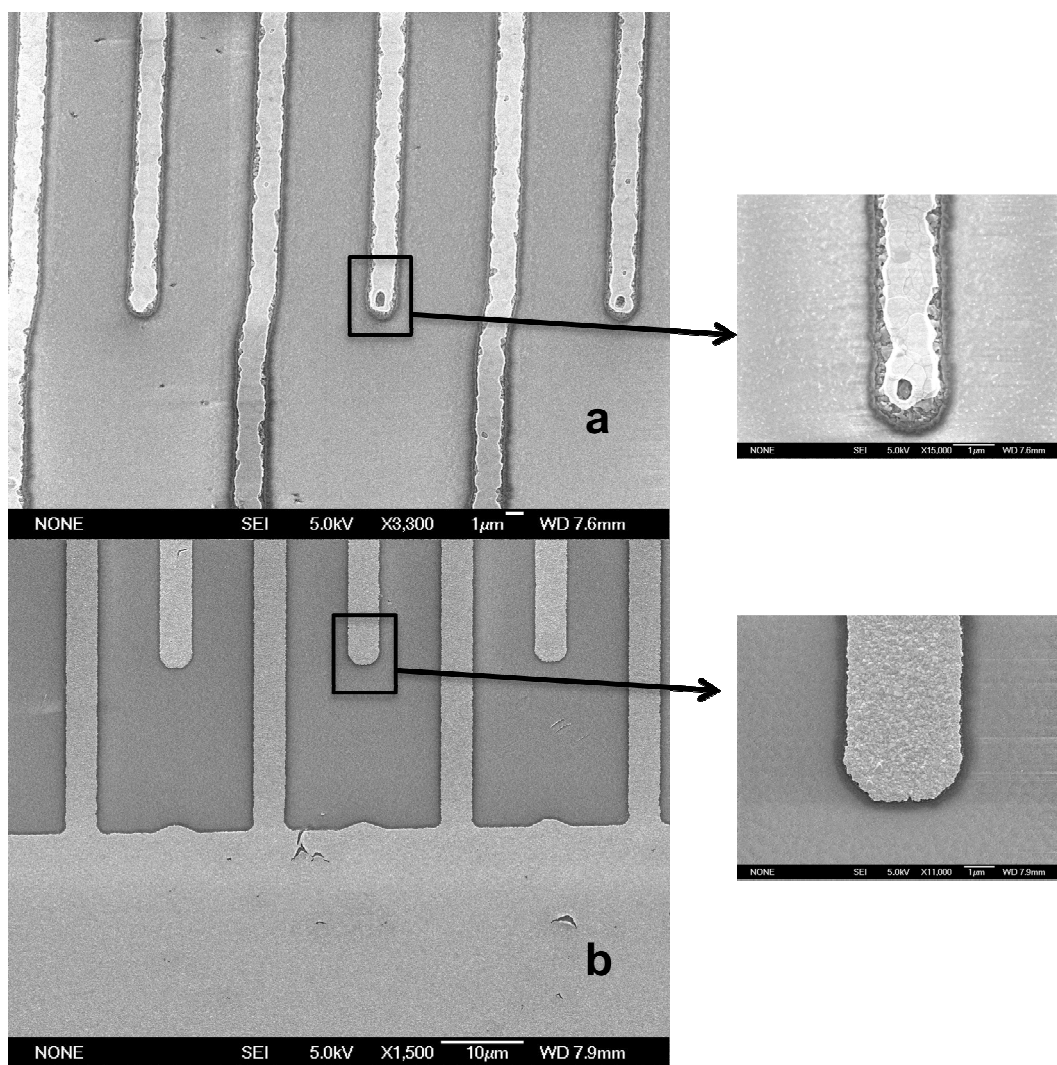


Fig. 11. SEM images of Pt/Ta (a) and Ir/Ti IDT (b) after 20h at 900°C

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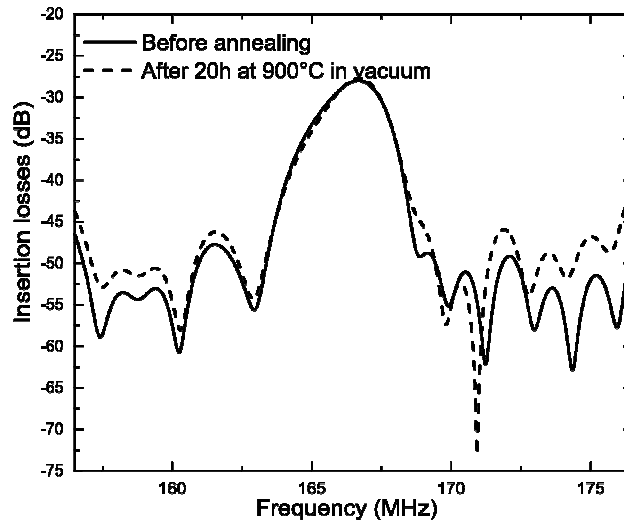


Fig. 12. Frequency response ($|S_{21}|$) of a Pt/Ta/LGS SAW device before and after annealing for 20h at 900°C in vacuum

Eventually, it has to be noted that these results are quite different from that obtained by Bardong *et al.*, who have observed an important degradation of the SAW signal after a relatively short annealing of 2h at 650°C in vacuum [17]. They have attributed this deterioration to a modification of the langasite substrate they used, this presenting a lack of gallium and oxygen after heating. This divergence between their results and ours are quite difficult to explain, but could be related to a difference of surface chemical properties, depending on the cut of the LGS substrate. Indeed, while Bardong *et al.* have used the (0°; 138,5°) cut, our study was realized with the Y cut.

IV. CONCLUSION

To summarize, we have shown that, although it oxidizes in air atmosphere at high temperature, tantalum has the required properties to be used as an adhesion layer for platinum-based IDTs. The deterioration of Pt/Ta electrode can be attributed to agglomeration phenomena in the platinum thin film, leading to the break of the IDTs fingers and the loss of the SAW signal in a matter of hours at 900°C in air atmosphere. This interpretation means that, whatever be the nature of the adhesion layer, the deterioration of the platinum film at high temperature cannot be avoided, as it is intrinsic to it. This is consistent with the fact that similar observations have been done in the case of a zirconium adhesion layer [5]. However, it could be possible to significantly increase the lifetime of such electrodes by using a

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3 protective over-layer which could slow down the agglomeration mechanisms [10]. Another original way
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5 to avoid such thin film-specific problem is to replace platinum by iridium, which exhibits a far higher
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7 melting temperature. Our experiments confirmed this prediction, as Ir/Ti electrodes performed much
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9 better than Pt/Ta ones. Nonetheless, even in this case, the use of a protective over-layer could be
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11 mandatory as iridium oxidizes above 700°C [16].
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