Stabilized Pt Interdigitated Electrodes for High-Temperature SAW Sensors

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***Abstract*— The standard working temperature of Surface Acoustic Wave (SAW) device does not exceed 150°C, although high-temperature device demand is increasing. Commonly, Interdigitated Transducers (IDTs) for high-temperature SAW devices are fabricated by means of deposition of metal films by evaporation at room temperature and lift-off process. A bunch of different stacks of materials has been tested in order to optimize IDT stability at high-temperature: adhesion layer/noble metals, interlayering oxide/noble metal IDTs, composite with oxide particles incorporated in noble metal, metal alloys (note that a capping layer is also usually used). Such heterostructure implies many structural defects, small grains, and chemical instability which favor agglomeration, recrystallization and interdiffusion during annealing. Our previous work demonstrating a complete elimination of interdiffusion issues by direct growth of Pt IDTs onto Langasite (LGS) without adhesion layer. In this work, the growth of Pt films at high temperature was considered including the development of entire microfabrication process to demonstrate the feasibility of stable pure Pt electrodes.**

Keywords—IDTs, SAW, sensors, high temperatures, micro-fabrication, platinum

I. Introduction

SAW devices are widely used under ambient conditions as radio frequency filters, sensors (pressure, gas, chemicals…), oscillators. The most usual way to fabricate metal IDTs for SAW devices is a lift-off process of noble metal films deposited by means of e-beam evaporation onto a piezoelectric substrate. Although the demand for devices working at high temperature (>600°C) is increasing, the fabrication methods remain the same, and most of the research works deal with the use of different materials and stacks in order to stabilize the device. Typically, the commonly used aluminum IDTs are replaced by noble metal (Pt) ones and LGS piezoelectric crystal family, able to withstand temperatures >1000°C, is used instead of quartz or lithium niobate. Different strategies have been considered to optimize the stability of this stack [1]–[5]:

- Alloys of noble/noble or noble/transition metals;
- Insertion of oxide grains into the noble layer;
- Interlayering noble metal and oxide;
- Using an oxide as the adhesion layer and a capping layer to protect the IDTs;
- A mix of all these approaches.

Unfortunately, such heterostructures grown by e-beam evaporation at room temperature contains big concentration of defects / small grains, and present chemical instability which favor agglomeration, recrystallization and interdiffusion. Therefore, the recrystallization of the materials takes place during annealing at high temperature. Indeed, the more different materials are present in the stack, the higher possibility is for restructuring, mixing, or oxidation due to the affinity between the materials themselves or with the oxygen present in the atmosphere/stack, the energetic interfaces (grain boundaries, crystalline phases). Although, these phenomena have been studied widely in the literature, from a device point of view, the shift and non-reproducibility of the frequency due to material instability at high-temperature is not mastered yet and requires a development of a robust solution.

Following our previous study concerning the direct deposition without adhesion layer of pure Pt IDTs onto LGS [6], we report the improvement of Pt film quality thanks to high-temperature magnetron sputtering (at 630°C). Evidently, such deposition method does not allow to use lift-off process with photoresist (PR) masks. Therefore, an entire microfabrication process comprising deposition, patterning of IDTs by means of Reactive Ion Etching (RIE), and wire-bonding has been developed. In addition, an in-situ RF measurement have been done up to 1000 °C. Note that cladding layer has not been used intentionally here in order to accelerate Pt degradation and to demonstrate the effect of Pt quality itself.

II. Process and fabrication details

PLASSYS MP450S sputtering machine has been used for the deposition of 300 nm thick Pt films at 630 °C. It allows the use of different gas as Ar and O_2 which is important for Pt growth as shown in the literature [7]–[9]. Pt structure has been investigated using the D8 Advance BRUKER X-ray diffractometer. Pt patterning was performed by RIE with the CORIAL 200R and a chromium hard mask deposited by means of evaporation (PLASSYS MEB600). The roughness was measured by the Stylus Profilometer DEKTAK XT. The materials surfaces/interfaces were studied by using focused ion beam (FIB) cross-section cut, scanning electron microscopy (SEM) with the Helios Nanolab 600i. Pt wire-bonding has been performed using the Wire bonder TPT 16 from TPT. The in-situ RF measurements was done by using

the LINKAM TS1000 heating stage. Single-port SAW resonators working at 254 MHz (room temperature) have been successfully fabricated. The RF measurements were done with the ROHDE and SCHWARZ Vector Network Analyzer (VNA).

III. Results and Discussion

A. High-quality Pt deposition

The Pt deposited at room temperature presents very small grain size and tends to recrystallize at high temperature. In order to avoid the recrystallization phenomenon, the Pt layers have to present high crystalline quality/big grains and preferably the layers have to be grown at or above the operational temperature. High-temperature Pt deposition was done by means of magnetron sputtering at 630°C (targeted operational temperature is 600 °C). In order to increase the crystalline quality and grain size, the layer growth rate was reduced to 5 nm/min (the growth rate of 55 nm/min was used for depositions at room temperature). The influence of other deposition conditions such as pressure and presence of oxygen have been considered as well. Ideally, we have been looking for the conditions offering epitaxial growth on LGS substrates, offering the highest crystalline quality.

Fig. 1 compares the grain size and texture quality of platinum layers deposited at room temperature (RT) and optimized ones grown at 630°C. XRD pole figures showed

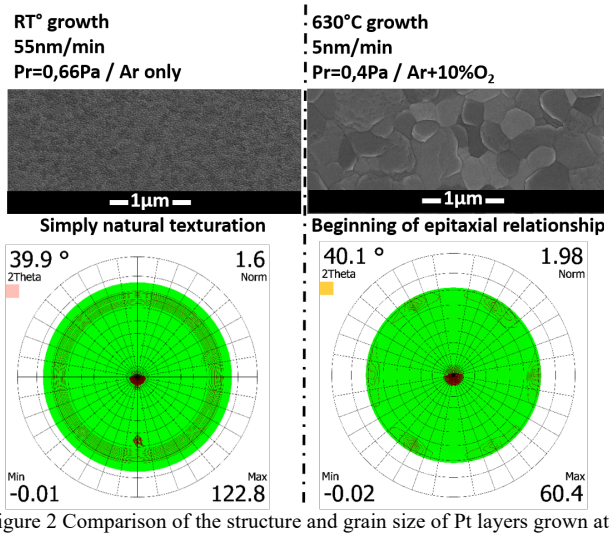

Figure 2 Comparison of the structure and grain size of Pt layers grown at room temperature with a fast recipe (left) and at 630°C with an optimized one (right).

that both films grew along the (111) direction which is the natural orientation of dense planes for face-centered cubic lattice. The pole figures are pointing out different growth behaviors: a simple textured growth without any alignment in the plane with the substrate at RT (circle observed at χ = 60-70°) and a certain orientation in the substrate plane – initiation of the epitaxy at 630 °C (large spots at χ = 60-70°).

Furthermore, the grain size is significantly bigger in the layers grown at high temperature. Fig. 2 plots the distribution of grain size as a function of the growth parameters and also after annealing of sample grown at room temperature. The mean grain size has been increased up to 300 nm by increasing the deposition temperature up to 630 °C and by reducing the growth rate. In addition, the grains grown at 630°C are twice bigger and the roughness is found to be 3 times lower than the grains grown at room temperature and annealed for 48h at 800 °C. This confirms, once again, the inefficiency of the

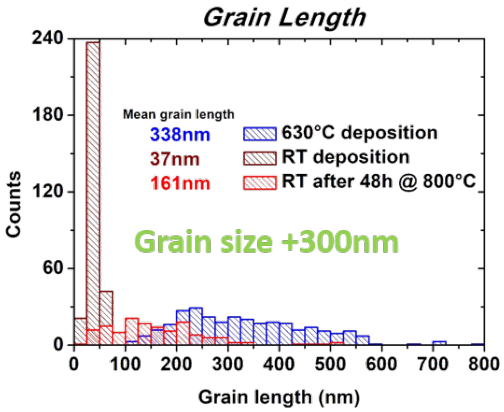

Figure 1 Distribution of the grain size of samples grown at room temperature (as deposited and annealed for 48 h at 800 °C) and film grown at 630 °C.

annealing processes of the layers grown at RT and the necessity of the growth of Pt layers at high temperature in order to obtain high-quality crystallinity.

B. Pt patterning

Pt deposition at high temperature was performed in full-plate mode directly onto our LGS substrate. Therefore, Pt was patterned by means RIE. A set of experiments has been tested in order to find the best parameters to get a nice wall-shape without (or with the minimum) of redeposition:

- PR mask (positive and negative resist) with different thickness, wall shape tuning, hardbake (in total 6 different PR with parameters tuning for each);
- Hard mask using wet-etching and lift-off for the patterning (deposited with direct current sputtering and e-beam evaporation, respectively).
- Pt patterning etching with different parameters: power, gas (Ar, SF_6, C_2F_6, CHF_3, O_2), pressure, susceptors.

Considering everything together, the best combination was found using a 120 nm thick mask of Cr patterned with lift-off and the following Pt etching conditions: pressure of 15mTorr, power of 150W, graphite susceptor, gas: Ar/SF_6 pulsed (cycles of 10 s at 50%)/C_2F_6 with the proportion 5/50/5

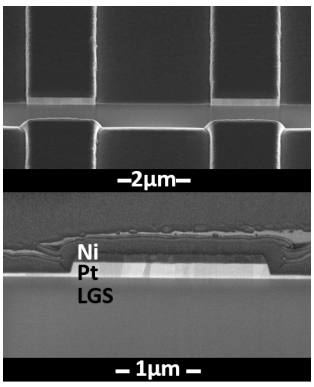

Figure 3 SEM tilted micrograph (top) and cross-section micrograph (bottom) of IDTs after Pt patterning using a nickel hard mask.

(in sccm). Note that the use of C_2F_6 just allows to dilute the Ar but does not play a major role in the etching. Fig. 3 is an illustration of successful patterning of Pt IDTs by means of RIE. Indeed, the etching does not induce any roughness (checked by the profilometer), redeposition is limited (not even clearly observable) and side-wall shape is good considering the verticality.

C. *Pt wire-bonding*

The heater stage of TPT 16 provides temperatures up to 250°C. To do measurement up o to 1000°C, Pt wire bonding is mandatory, therefore we used Pt wires of 25 µm diameter. Unfortunately, we were not able to do a proper bonding in these conditions, even tuning all the different parameters of the bonder. The 2 critical criteria in thermosonic bonding are the temperature and the force [10]. In this case, the temperature was insufficient.

LINKAM heating stage has been fit to the bonder. And successful bonding of Pt was succeeded at 425°C. Moreover, the bonding was easily reproducible and robust. The strength of the bonding allowed us to manipulate and weld several times the same device. During measurement and annealing, we proved that the wires easily withstand 1000°C for 10 hours (at least, annealing time was limited by our heating stage) which is way more than the capacities of any device. Fig. 4 is showing SEM pictures of a wire-bonded device after a simple annealing at 800°C for 3h.

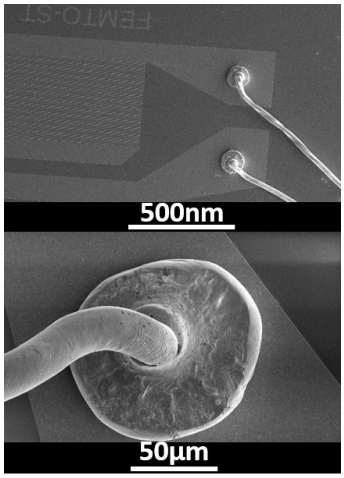

Figure 4 SEM micrographs of a wire-bonding after annealing for 3h at 800°C.

D. *In-situ RF measurement set-up*

First of all, a commercial LINKAM TS1000 has been adapted to RF measurements, so we had to completely remove the electrical circuit for the measurement in order to input a coaxial cable as shown in Fig. 5. A Printed Circuit Board (PCB) card has been cut to get only 3 ports (1 signal in the middle and 1 ground on each side). In the latter, contact picks have been welded to provide a contact for the wires. Note that inside the metallic support, we introduced insulating foam to avoid shortcuts. The set-up was cooled with a simple closed water circuit (the water temperature increased up to 30°C at 1000°C for at least 1h).

Obviously, doing the calibration in such a difficult set up is very difficult since we have to take care that the wires have the same size as the calibration kit. To overcome this critical parameter, we have welded together two 100 Ω resistances and try to leave the right length for their legs. The calibration was performed by welding the resistances on the contact picks. By plugging a Sub-Miniature A connector (SMA) on the contacts, and then a calibration kit, we were able to check the quality of our signal. In this set-up it was not achievable 30 dB level (considered as a good value) and the measurements have been done with -20 dB roughly.

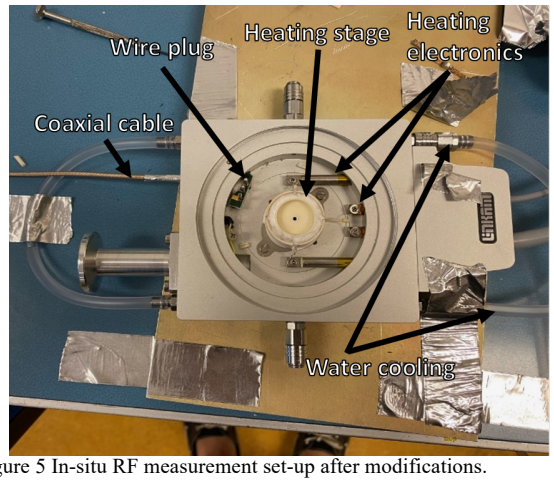

Figure 5 In-situ RF measurement set-up after modifications.

Once the device is place in the middle of the heating stage, we grab the long wires (5cm) and weld them right onto the contact picks. The difficulties of the calibration, added with the use of a device design with different Pt thickness led to the mismatch between the calibration and the device which was totally expected. Nevertheless, we were able to observe the frequency response as a function of temperature (most critical parameter) although the quality factor needs future improvements. The frequency response is not affected by the calibration and not optimized design but the quality factor is. Eventually, a new design will be done in future to optimize the device performance.

E. *Meaurements and ageing*

At first experiment, the frequency response was measured during 3h at 800°C and indicated very limited Pt restructuration. The shape of the IDTs got rounded but it did not cause major change in the properties of the IDTs. Indeed, the measurements with the VNA showed an oscillation of 1.6 ppm between the 3h of work, which remains in the error range. Afterwards, this sample was cycled at 1000 °C. During the first cycle, the frequency at 800°C was still in the range of the previous measurements (249.4912MHz) which proves that the device was still working properly and is stable.

During cycles at 1000°C, the restructuration of the Pt IDTs shape lead (Fig. 6a) to frequency shift (Fig. 6b) and collapse of quality factor (Fig. 6b inset). We remind that the Pt was not covered by any cladding layer intentionally to accelerate the Pt degradation as our heating stage was limited in annealing time at high temperature and that the device does not really fit the 50 Ω calibration.

The triangular geometry taken by the Pt while annealing clearly depicts a recrystallization which could be at least limited by a cladding layer. In the same time, one can see that the frequency is shifting (1MHz down between the 1st and the 3rd run). Obviously, several factors are playing a role here: the ratio a/p completely changed and the thickness of the digits increased from 300nm to 700nm on the top of the crystals (SEM cross-section image on the bottom). The unregular shape of the digit after recrystallization leads to collapse of the quality factor from Q=2050 to Q=21.

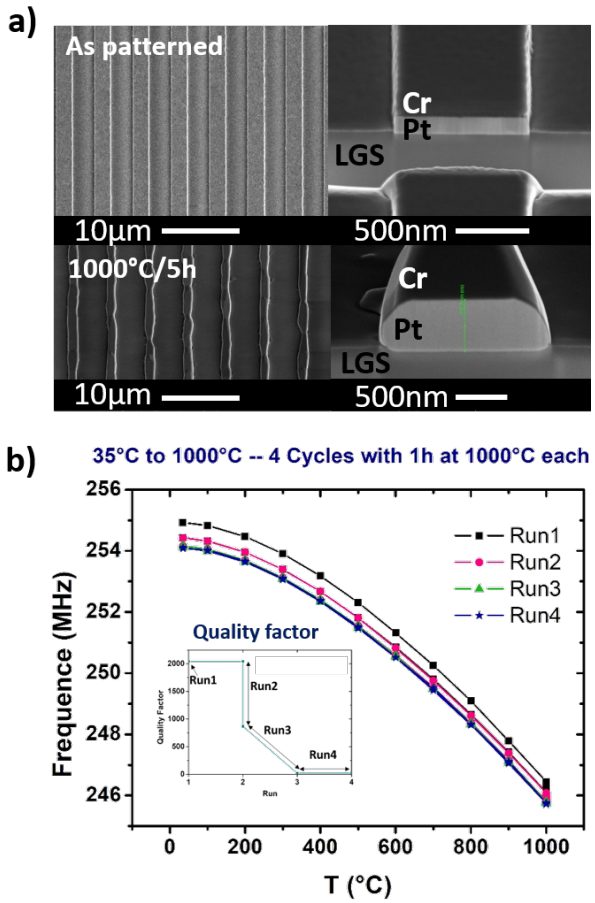

Figure 6 a) SEM micrographs of the surface and cross-section of a device as-fabricated (top) and after 5h at 1000°C (bottom), b) frequency response during 4 heating runs up to 1000°C. Inset shows the quality factor at different runs.

Nevertheless, one can see that the shift between a run and the next one decreases over time. Between the 3rd and the 4th run, the frequency does not shift anymore. We assume this to be due to the restructuration endpoint (at least, it extremely decreased). The observation is quite similar with quality factor, after the 3rd run, it remains at Q=21 and the device is still working. Obviously, the IDTs shape damage is significant, but it exceptional stability for the unprotected Pt layer at 1000 °C and these results are really encouraging for future device elaboration including the cladding layer. In addition, we have to support the fact that this behavior is different from the dewetting observed when there is a big difference of surface energy between the materials which causes the agglomeration and non-geometrical shapes (droplets).

IV. CONCLUSION

In conclusion, this work has shown that the direct deposition of Pt onto LGS at low deposition rate and at high temperature allowed to attain high crystalline quality Pt films showing epitaxial relationship with substrate and with a mean grain size close to 340 nm (ten times bigger grains as compared to films grown at RT). As high deposition temperature is not compatible with standard lift-off process, a new patterning process has been developed using RIE. Pt-wire bonding, stable at 1000 °C for hours, was performed at 425°C. Pt IDTs restructuration was very limited at 800 °C but it led to frequency shift of 1MHz and collapse of quality factor (from Q=2047 to Q=21) at 1000°C. After 3cycles of 1 h at this temperature, the frequency remained stable as well as the quality factor which is assumed to come from restructuration endpoint (or at least close). No dewetting was observed and the IDTs shape restructuration was mainly due to the recrystallization which can be limited by a cladding layer.

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