Characterization of materials for the design of wireless SAW sensors in a high temperature environment

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***Abstract*—In this work, we describe an experiment performed to analyze the behavior of materials deposited on an oxidized silicon substrate submitted to high temperature conditions (650°C to 1000°C). This process developed in FEMTO-ST institute aims to characterize materials of potential interest for wireless SAW (Surface Acoustic Wave) sensors in a "high temperature" environment. The analysis indicates that three kinds of sublayer/electrode pairs (W/Mo, Ta/W and Ta/Mo) are good candidates for further developments in actual sensing devices.**

Keywords— MEMS; high temperature; SAW; sensor.

I. INTRODUCTION

At present, the ability to interrogate wireless SAW (Surface Acoustic Wave) devices in a high temperature environment (600°C to 1000°C) represents a serious challenge. Despite the publication of many results about this subject [1-5], finding the right configuration for actual devices subject to a harsh environment is still problematic.

The aim of our work is to focus not only on the melting temperature of the materials used, but also on others parameters occurring before this precisely defined point. This should result into a selection of materials that can withstand a high temperature environment. To test the obtained configuration, we designed and realized a specific pattern in our laboratory.

II. MATERIALS SELECTION

A. Context

According to the literature [6] the materials undergo two "stages" during a sustained increase of temperature. The Fig.1 sketches these two stages.

The first one is the Hüttig temperature (T_H) which is equal to 0.3 T_m (melting temperature). This stage creates some surface diffusion on the material which means that the atoms become highly mobile on the surface. This phenomenon corresponds to a recrystallization of the material surface. Then a strong interfacial atomic diffusion appears from the surface and the grains boundaries.

The second stage is the Tamman temperature (T_T) which is equal to 0.5 T_m. This stage creates some volume diffusion in the material, allowing the atoms or molecules of the solid to gain enough energy to become mobile.

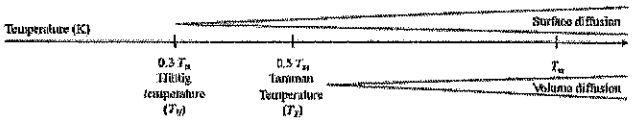


Fig.1: Volume and surface diffusion in function of the temperature

The combination of these two stages may cause the separation of the grains of the material. In this case, they gather in "islands" called crystallites. This phenomenon is called "dewetting". An example of such dewetting can be seen on Fig.2. The picture was obtained on a sample of Ta/Pt sublayer/electrode pair annealed during 20 h at 900 °C. Then the observed system no longer works, therefore it is necessary to avoid the appearance of these crystallites.

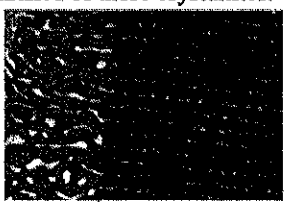


Fig.2: Dewetting of a device Ta/Pt exposed 900 ° C for 20 h.

B. Selection of the tested materials

To perform the tests we selected the materials (Table 1) according to the temperature parameters T_H, T_T and their electrical conductivity. We also retained platinum which is well-known for its temperature resistance and low impedance despite its low Tamman temperature (884.5°C). The selected materials are Mo, W, Ta and Pt.

Chosen materials	T_m(°C)	T_H(°C)	T_T(°C)	Conductivity (μΩ.cm)
W	3380	1014	1690	5.5
Ta	3017	905.1	1508.5	13.1
Mo	2623	786.9	1311.5	5.3
Pt	1769	530.7	884.5	10.6

Table 1: Selected materials

III. DESIGN OF THE TESTING DEVICES

The testing devices were designed to address several issues such as passivation, impedance of a finger network, development of the electric contact and ability to obtain a clear view of the diffusion in the substrate. The diagram of the sectional view can be seen on the Fig.3.

Fig.3: Diagram of a sectional view of the material test structures

These complete devices (Fig.4) are 4 mm x 4 mm. The finger width dimensions available on the design are 1, 1.5, 2, 2.5 and 2 µm. A large pad is designed in the center of the structure to facilitate the required analyses to verify the diffusion of the different materials.

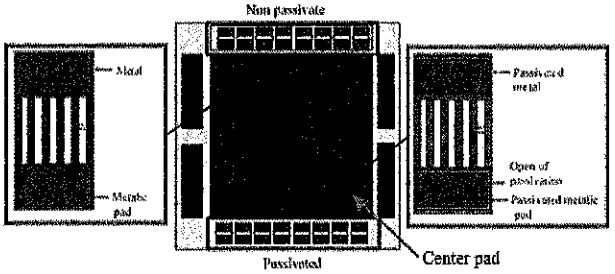


Fig.4: Diagram of a top view of the test structures

IV. TESTS AND RESULTS

The first step of the testing process consisted in the characterization of the investigated materials (W, Ta, Mo and Pt) with the design of the structure of the Fig.4. Different configurations of materials were subjected to different durations (1 h to 20 h) and annealing temperatures starting from a base of 600°C to 1000°C in steps of 100°C. Three materials combinations showed a satisfactory behavior during the annealing: W/Mo, Ta/W and Ta/Mo. All observations were carried out under microscope and electrical tests were realized by probing the devices to measure the resistivity of them. This last operation can be interesting despite the fact that the material was returned to room temperature after the measurements. Indeed, it allows to hihlight the power cuts suffered by the device because of the dewetting effect or the oxidation of the material.

A. W/Mo

This configuration showed good performance at different exposures. Passivated pads were found the most resistant. We observed that the strength of this stack depends on the temperature exposure. The higher the temperature, the higher is the strength of the materials increases. Indeed we observed that during annealing at 600 ° C for 1h Mo top layer peels off. This peeling off of the Mo layer can be observed on Fig.5.

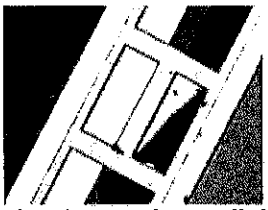


Fig.5 : Observation of a W/Mo sample annealled during 1h at 600°C

Although this configuration showed good performance at different exposures., the reaction of this layer was different during annealing at higher temperatures. A dark stain appears on the central pad at its center after 20 hours of annealing at 900 °C (Fig.6).

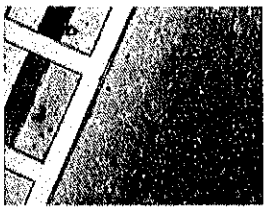


Fig.6 : Observation of a W/Mo sample annealled during 20h at 900°C

The bonding layer of tungsten had beneficial effects on keeping molybdenum fingers unlike the configuration Ta/Mo. It showed no surface deformation but a dark stain in the center of the central pad was still present. In addition, there were no appearance of cavity or fracture at the base of the fingers, whatever the passivation case. We observed a beginning of dewetting after exposure for 20 h at 1000 °C in the case without passivation. The passivated fingers resisted to the annealing (Fig.7).

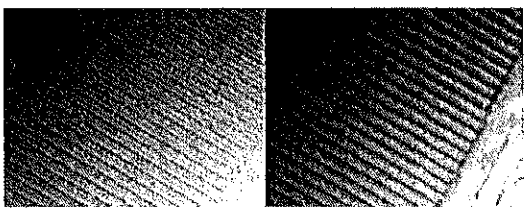


Fig.7: Behavior of the W/Mo finger networks with passivation (a) and without (b) after a 20h annealing at 1000°C

The three graphs below show the different thickness variations during 20 h annealing at 600°C, 700°C and 1000°C. On the graph of Fig.8 we see that the thickness has decreased since a measure of the total thickness of material on a reference sample revealed a thickness of 297 nm after the return at room temperature

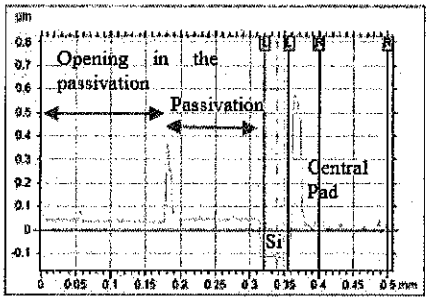


Fig.8 : Observation of the profile of a sample of W/Mo on the area passivated after a 20 h annealing at 600 ° C.

We get here a thickness of 121 nm. It remains greater than the thickness of the 20 nm bonding layer of W. The second profile measurement was conducted on a sample annealed for 20 h at 700°C (Fig.9). We observe that the thickness increases and reaches 267 nm. We also see a rise of the peaks which reach a height of 700 nm in Fig.8. On Fig.9 we see that these peaks reach now 1.3 µm.

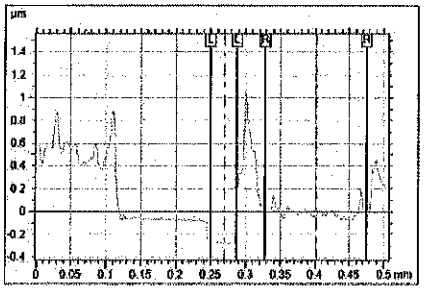


Fig.9: Observation of the profile of a sample of W/Mo on the area passivated after a 20 h annealing at 700 ° C.

The latter observation is that of a sample annealed for 20 h at 1000 ° C (Fig.10). We note that unlike previous observations there is no presence of peaks. The surface of the pads is irregular but maintains an average value of 726 nm. This value is almost 2.5 times greater than that of the reference sample. We can therefore assume that some oxidization of Mo occurs.

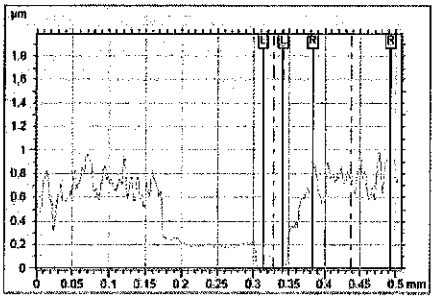
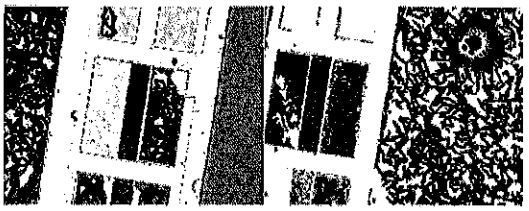


Fig.10: Observation of the profile of a sample of W/Mo on the area passivated after a 20 h annealing at 1000 ° C.

During the study of the profile of the devices, we see that as the temperature increases, the thickness of the material increases once the device returns to room temperature. This configuration of W / Mo material seems usable for the production of devices in high temperature environments. Nevertheless, one must take actions to prevent the growth of molybdenum oxide which could preclude the reliability of the electrical contacts on the pads.

B. Ta/W

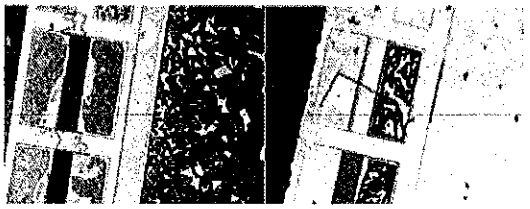
This stack showed an unexpected dark coloring of the fingers after the development in cleanroom. Yet the fragility came from the pads as in the case of Ta/W. They dissociated themselves to form a pattern similar to that observed in the case of the Ta/W pair (Fig.11).



a. b.

Fig.11: Sample of Ta/W without passivation (a) and with passivation (b) after a 1h annealing at 600°C

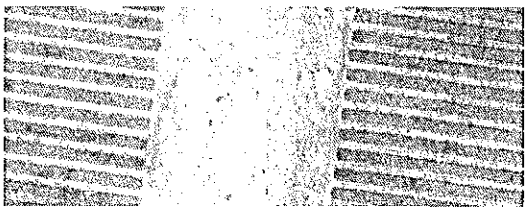
Passivated and non-passivated tests were performed up to a temperature of 700°C for 20 hours. The appearance of the samples after these test is shown on Fig.12.



a. b.

Fig.12: Sample of Ta/W without passivation (a) and with passivation (b) after a 20h annealing at 700°C

This configuration showed some strength in the pattern conservation despite the apparent state of the pads. Fingers exhibited a good resistance with and without passivation after an annealing of 900 ° C for 20 h (Fig.13).



a. b.

Fig.13: Sample of Ta/W without passivation (a) and with passivation (b) after a 20h annealing at 900°C

We performed measurements on two samples. One was submitted to 20 hours of annealing at 700 °C and the second one was annealed during the same time at 900 °C. The first sample annealed at a temperature of 700 °C (Fig.14) shows a thickness of 400 and 500 nm between pads. This is twice the value of the deposited thickness of the reference sample. We can assume that this is due to the appearance of tungsten oxide.

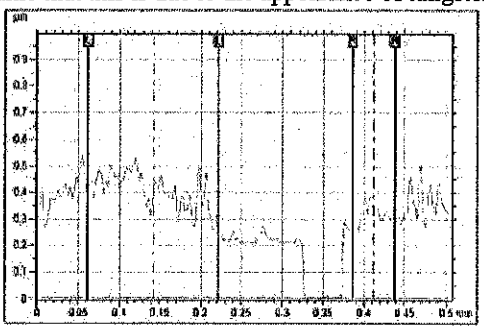


Fig.14: Observation of the profile of a sample of Ta/W on the area passivated after a 20 h annealing at 700 ° C.

Observations of the second sample of Ta/W (Fig.15) show us a slightly decrease about ten microns of the thickness of the material layer.

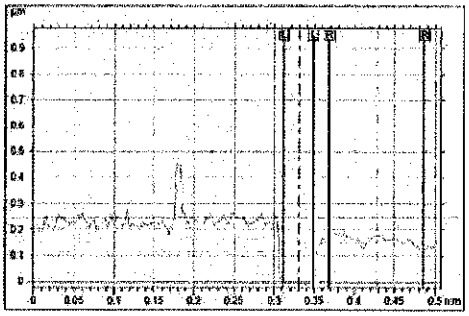


Fig.15: Observation of the profile of a sample of W/Mo on the area passivated after a 20 h annealing at 900 ° C.

This stack of materials Ta/W can also be considered as a possibility for the realization of high temperature systems.

C. Ta/Mo

During the rise in temperature, the devices remain viable up to 600 °C for 20 h. The first signs of deterioration of the device appeared after exposure for 1 hour at 700 ° C. Then small blisters appear on the pads (Fig.16).

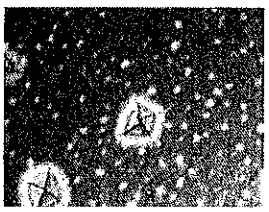


Fig.16: Emergence of small blisters on the Ta/Mo pads after a 1h annealing at 700°C

After exposure for 20 h at 700 °C, we observe that the top layer of the pads peels off and wraps on itself forming micro-rollers (Fig.17).

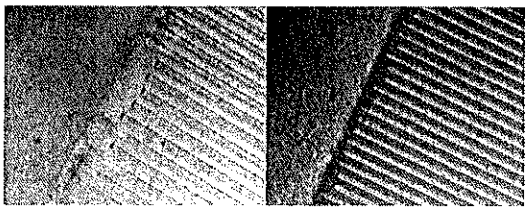
a. b.

Fig.17: Observation of the peeling off of the pads of Ta/Mo into micro-rollers after a 20h annealing at 700°C

Beyond this threshold, "rollers" melt and create a "crack" – looking pattern on the surface of the pads. Upon closer look, this pattern actually consists of a network of mounds on the surface (Fig.18).

Fig.18: Crack pattern on the surface of the Ta/Mo pads resulting from the melting of detached pads of Mo pads after a 20 h annealing at 900 ° C

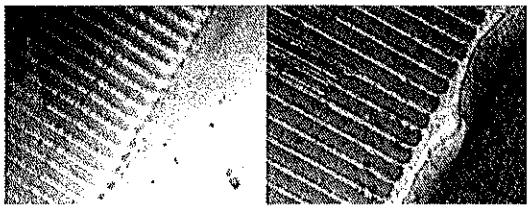
However the network of fingers, passivated or not, between the pads do not seem to have been affected by this peel off. This is a positive point because the fingers look almost the same in the case of Al_2O_3 passivation as without. During some exposures (eg for 1 h at 800 ° C or 20 h at 900 ° C) it is possible to observe the lift-off of non-passivated fingers at their junction with the pads (Fig.19). The passivated fingers are always connected to the pads but a cavity is observable in the same place together with a thinning of the fingers at their base.



a. b.

Fig.19: Lift-off of the Ta/Mo finger networks after a 1h annealing at 800°C (a) and a 20h annealing at 900°C (b).

Exposures at 1000 °C (1 hour and 20 hours) seem to deny previous comments on samples strength. Indeed fingers networks remained intact in these conditions (Fig.20).



a. b.

Fig.20: Observation des doigts de Ta/Mo après recuit de 1h à 1000°C et 20h à 1000°C Observation of a Ta/Mo sample annealled during 1h at 1000°C(a) and 20h at 1000°C(b).

We tried an observation of the visible micro-rollers profile in Fig.21. They were very compliant, so that the tip of the profilometer could not measure them. Therefore we oriented the observations on the profile "cracks"–looking patterns shown in Fig.21.

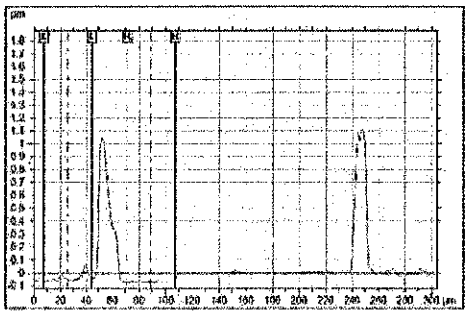


Fig.21: Observation of the profile of the crack patterns on a sample of Ta/Mo after a 20 h annealing at 900 ° C.

We observe that these patterns have an important height (between 1.1 and 1.2 microns) compared to the rest of the sample. We also realized profile measurements on a sample annealed for 20 hours at 1000 °C to see the evolution of the mounds Fig.22).

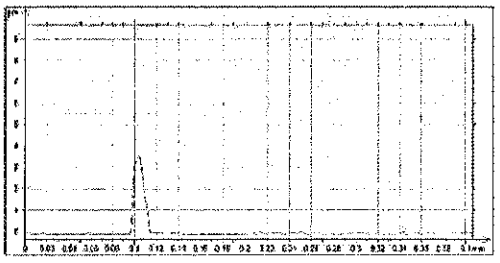


Fig.22: Observation of the profile of the crack patterns on a sample of Ta/Mo after a 20 h annealing at 1000 C.

We observe an increase in the height of the mounds. This confirms the assumed dewetting combined with coalescence thereof.

Observing this configuration Ta / Mo has shown us that it can not be used for high temperature applications.

The main issue was the oxidization of three of the materials (W, Ta and Mo) in the passivation opening, resulting into a cancellation of the electrical contact. This led us to set up the next step of the tests.

This second step consisted in the characterization of the material to assert the quality of the electrical contact at the electrode surface. The requirement was that the contacts do not corrode and do not dewet along the temperature increase. Platinum was a natural candidate for this experiment. As it is a noble material, it was the only one which was not oxidized after the treatment. Nevertheless, due to the small thickness (100 nm) of the investigated layer, it dewetted on the whole surface of the electrode. To avoid this problem the solution was to increase the thickness of platinum (200 nm) (Fig.23).

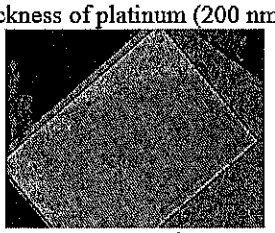


Fig.23: Zoom on a platinum pad

V. CONCLUSION

In this paper, we present the development of a process aimed to select appropriate metals for the realization of SAW sensors in a harsh environment, on the basis of the knowledge of Hüttig and Tamman corresponding temperatures. The design of a test structure allowed us to test the different configurations usable in this kind of environment.

ACKNOWLEDGMENT

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