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Effect of the Deposition Conditions of Platinum Electrodes on their Performance as Resistive Heating Elements

Andrei Ionut Mardare[a], *Cezarina Cela Mardare*[a], *Ednan Joanni*[a,b]*

[a]*INESC-Porto, Unidade de Optoelectrónica e Sistemas Electrónicos, Rua do Campo Alegre 687, 4169-007 Porto, Portugal*
[b]*Universidade de Trás-os-Montes e Alto Douro, Departamento de Física, 5001-911 Vila Real, Portugal*

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The performance of different platinum electrodes used as resistive heating elements was studied. Pt films having different thickness were deposited by RF magnetron sputtering at room temperature followed by post-deposition annealing at 700 °C or made in-situ at 700 °C. The Pt films were deposited over oxidized silicon, using Ti or Zr buffer layers. The resistance dependence on temperature was studied by applying increasing currents (up to 2A) to the Pt films. Changes in the microstructure of the Pt films account for the changes in the temperature coefficient of resistance as a function of the deposition parameters. The maximum substrate temperature (675 °C) was obtained when using 200 nm Pt films deposited at 700 °C over Ti, with a power consumption of only 16 W.

Keywords: *joule heating, platinum, thin films, bottom electrodes*

1. Introduction

Post deposition heat treatments on thin films deposited at low temperature are an important step in obtaining ceramic films with the required crystalline phases. Different heating methods can have a very big influence on the final material properties, therefore many efforts are made to find the optimum conditions for heat treatment. For instance, slow furnace annealing and rapid thermal annealing methods can give distinct results regarding grain size and phases present in the films[1-3].For high temperature crystallization heat treatments platinum bottom electrodes are important due to their high stability and oxidation resistance. Platinum films are also used as heating elements in resistive heaters, and their temperature coefficient of resistance is exploited in temperature sensors, gas sensors and anemometers[4-7]. In a previous report we presented a simple method for crystallizing ceramic thin films using the platinum bottom electrodes as resistive heating elements[8].This bottom electrode crystallization (BEC) method is simple and has several advantages over the other crystallization techniques, such as very low power consumption and the possibility of using high heating rates with precise temperature control.

In this paper we report the effect of the preparation conditions of the platinum thin films and adhesion layers on their performance as resistive heating elements for the crystallization of ceramic thin films using the Joule effect.

2. Experimental

The samples used for the study of the platinum heating elements for the BEC method consisted of Pt/Ti(Zr)/SiO_2/Si stacks. For sample preparation, silicon wafers with (100) orientation were cut into 1 × 1 cm^2 pieces and oxidized in wet oxygen flow for 72 h at 950 °C in order to obtain a 2 μm layer of SiO_2. On those substrates, titanium and zirconium films 30 nm thick, deposited at 200 °C, were alternatively used for enhancing the adhesion between the silica and the platinum films. All the metallic layers were deposited by RF magnetron sputtering in argon atmosphere. The targets used had a 2 in diameter and the deposition power and pressure were kept constant at 150 W and 10^{-2} mbar respectively. The thickness of the films was measured in real time using an oscillating quartz crystal thickness monitor. All the platinum films were heat treated at 700 °C in order to minimize subsequent microstructural changes during the experiments. One series of Pt films was deposited *in situ* at 700 °C while the other series

*e-mail: ednan@utad.pt

was deposited at room temperature and heat treated in air at 700 °C for 10 min using a 1000 W halogen lamp heater with 10 °C/s heating and cooling rates. The preparation conditions for the electrodes are presented in Table 1.

Electrical contacts were made on two opposite edges of the platinum films using 0.5 mm diameter tungsten wires; the distance between the contacts was 8 mm. This setup allows a bigger contact area compared with the one previously reported[8], avoiding localized hot spots and making unnecessary the use of silver paste. The samples were placed over a high temperature ceramic fiber insulator. The temperature obtained due to the Joule effect was monitored in real time using a type K thermocouple with 0.3 mm thick wires. The setup is shown schematically in Fig. 1.

A DC power supply (Kepco 60-2 DM) with an upper current limit of 2A was used for applying currents to the platinum films. A multimeter (HP 34401 A) was used for reading the voltage from the thermocouple. The setup was controlled using a LabView® program which was set to increase the applied current from 0 to 2 A with a step of 0.02 A and to measure the voltage for each value of the current in order to obtain the value of the resistance as a function of temperature. For ensuring a thermal equilibrium of the whole setup during the resistance measurements, the current values were modified periodically at every 20 s.

The microstructures of the samples were analyzed using X-ray diffraction (Siemens D5000) and scanning electron microscopy (JEOL JSM-6301F).

3. Results and Discussion

X-ray diffraction scans performed on all samples showed only the presence of the Pt (111) peak. Fig. 2. shows the resistance as a function of temperature for Pt films 200 nm, 300 nm and 400 nm thick deposited at room temperature over Ti and heat treated at 700 °C in air. With the increase of the thickness of the Pt films, both the room temperature resistance and the temperature coefficient of resistance decreased. As a consequence, the highest temperature that could be achieved with the maximum current (2 A) decreased from 650 °C for the 200 nm film to 125 °C for the 400 nm film. The power consumption necessary for achieving the maximum temperature with the 200 nm film was 15 W, showing the high energy efficiency of the BEC method due to the small mass of the sample and the direct contact between the Pt film and the Si substrate.

The results obtained for Pt films deposited at 700 °C over Ti and Zr buffer layers are shown in Fig. 3. Comparing films with the same thicknesses, the room temperature resistance and the temperature coefficient of resistance for the Pt films deposited over Ti were higher than those deposited over Zr. The maximum temperature achieved using Ti was 675 °C, with a power of 16 W, while the maximum temperature for the corresponding Zr film was 450 °C. The lowest value of

Table 1. Preparation conditions for the electrodes.

Sample name	Adhesion material	Platinum thickness (nm)	Platinum deposition temperature (°C)	Post deposition heat treatment
Ti200	Ti	200	700	-
Ti300		300	700	-
Ti400		400	700	-
Ti200H		200	room	10 min at 700 °C
Ti300H		300	room	10 min at 700 °C
Ti400H		400	room	10 min at 700 °C
Zr200	Zr	200	700	-
Zr300		300	700	-

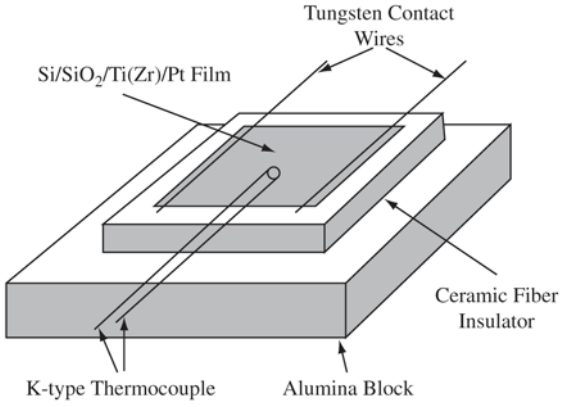

Figure 1. Experimental setup for measuring the resistance of the films as a function of temperature.

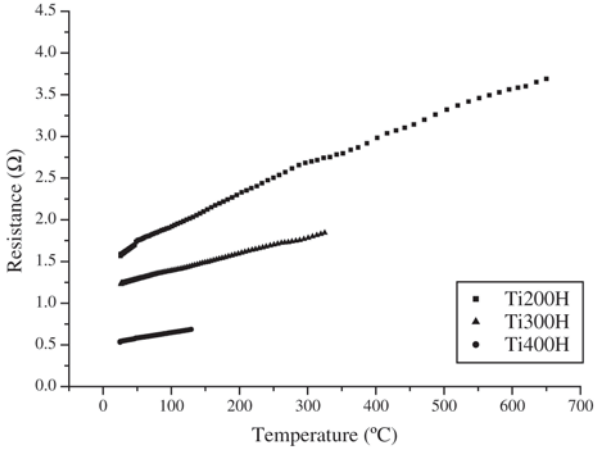

Figure 2. Variation of the resistance as a function of temperature for Pt films 200 nm, 300 nm and 400 nm thick deposited at room temperature over Ti and heat treated for 10 min at 700 °C.

temperature recorded at the maximum current of 2 A was 125 °C and it was measured on the 400 nm Pt film deposited at 700 °C over Ti. The room temperature value of resistance for this film was low, being measured as only 0.45 Ω

The scanning electron microscope photographs of the surfaces of two Pt films deposited over Ti are shown in Fig. 4. All the films deposited at room temperature and heat treated for 10 min at 700 °C were porous and in Fig. 1a can see the voids between the grains. In contrast, the films deposited at 700 °C were denser and had slightly bigger grain sizes as illustrated in Fig. 1b.

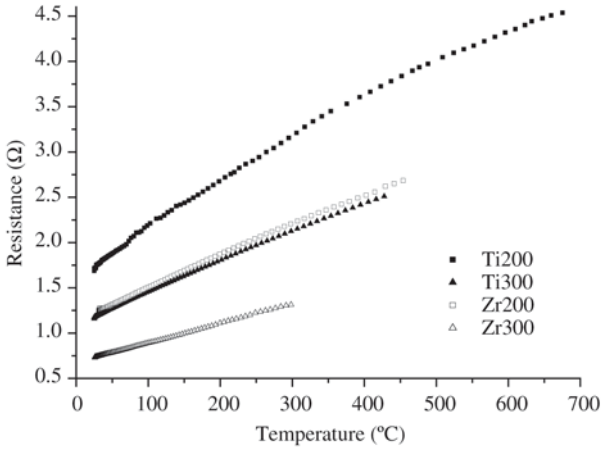

Figure 3. Variation of the resistance as a function of temperature for Pt films 200 nm and 300 nm thick deposited at 700 °C over Ti and Zr.

Considering the microstructure of these films, the results shown on Figs. 2 and 3 for the Pt films deposited over Ti can be explained as follows: in the Pt films deposited at room temperature, Ti diffusing through the Pt grain boundaries during the post deposition heat treatment is oxidized. The volume expansion due to TiO_x formation force them to open as shown in Fig. 4a[9]. Due to thermal expansion, when currents are applied to these films, the volume of the grains increase, having as a result an increase in the contact surface between the grains. As a consequence, the temperature coefficient of resistance is lower than in the case of the Pt films deposited in argon at high temperature. For the Pt films deposited at 700 °C (Fig. 4b), the films are denser and the Ti is not oxidized because the deposition is carried out in argon, without breaking the vacuum between the deposition of the metals. Only when the current is applied to these films in air, the Ti in the grain boundaries oxidizes, increasing both the resistance of the films and their temperature coefficient of resistance.

Figure 5 shows the surface of a Pt film deposited at 700 °C over Zr. In this case the Pt is denser and has bigger grains than the ones deposited over Ti. Accordingly, the resistance of these films is lower and the temperature coefficient of resistance is also lower due to the fact that Zr does not diffuse through the Pt film, as previously reported[9].

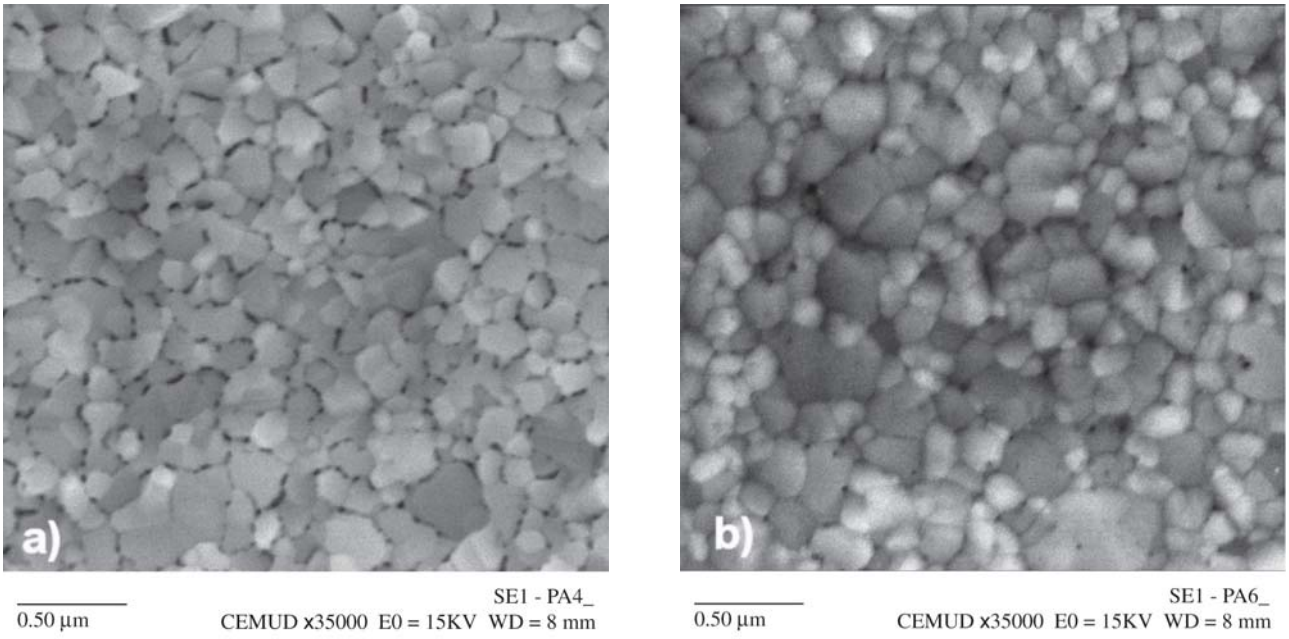

Figure 4. a) Scanning electron microscope image of the surface of the Pt film deposited over Ti at room temperature and heat treated at 700 °C; b) Scanning electron microscope image of the surface of the Pt film deposited over Ti at 700 °C.

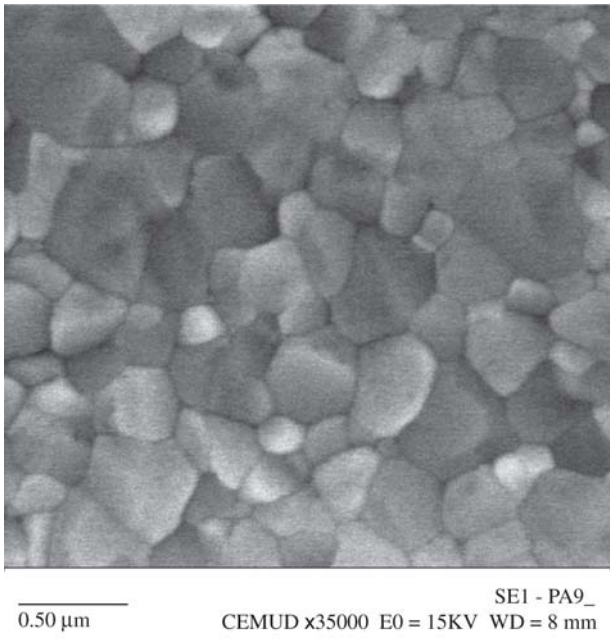

Figure 5. Scanning electron microscope image of the surface of the Pt film deposited over Zr at 700 °C.

4. Conclusions

In summary, the effect of different deposition conditions of Pt/Ti(Zr) thin-films on the maximum temperature achievable on low-current heating elements was studied as a function of the thickness of the platinum films and of the buffer layers used (Ti, Zr). Increasing the current up to 2 A, the resistance of the films made under different conditions was analyzed as a function of the temperature and the results were correlated with the microstructure of the films. The maximum temperature, 675 °C, was obtained when 200 nm Pt films were deposited at 700 °C over Ti adhesion layers.

The platinum films 200 nm thick are more suitable for the crystallization of ceramic thin films due to the fact that higher heating rates and/or higher temperatures can be achieved using a low current power supply. The Zr underlayer has the advantage of having a low diffusion coefficient in Pt, avoiding unwanted contaminations in the ceramic film to be crystallized when deposited over the platinum electrodes. On the other hand, from the point of view of thermal performance of the heating element, the Ti films give better results. The BEC method is very efficient, with a very low (16W) power consumption recorded at 675 °C. Work is now in progress for applying the BEC method to the crystallization of PZT thin films made by different deposition techniques.

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