Heat capacity determination of metallic thin films using temperature profiles at room conditions: experimental


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ABSTRACT

Based on the results of an analytical model developed for a heating/cooling process of a film/substrate system, an experimental method to measure the heat capacity (C) of nanometric films by means of electrical micropulses between 100 and 500 µs is discussed. The duration of the applied pulse will depend on the time constant of the studied film/substrate system. The corresponding changes of temperature on films during the micropulse application are measured by means of the changes in their electrical resistance. The micropulses were controlled with an electronic circuit designed with a NTP45N06 MOSFET switch, a 2N222A transistor driver, and a NI-USB-6216 card, which are driven with home-made software developed in LabView 2009.

An HP6543A power-supply was used for the electrical pulse. Voltage signals measured on films were acquired with a Tektronix digital oscilloscope phosphor DPO-4054 working at 2.5 Giga samples/s. Gold thin films (100 nm, thickness) were deposited on Kapton substrates (125 µm, thickness) for experimentation. The heat capacity (C) of gold films was estimated by measuring, during the micropulse application, the increase of temperature (ΔT), the applied electrical power (Q), and the lapsed-time (Δt) of the applied micro-pulses on films through the simple relation C=(Q * Δt)/(ΔT). The mean value of C for the gold film (100 nm) analyzed was 950 ± 30 nJ/K, as obtained with a pulse of 250 µs, in good agreement with the values reported in literature.

Keywords: heat capacity, micropulse, thermal properties, gold films.

1. INTRODUCTION

The development of new micro-electronic devices is intimately linked with new materials and reduced dimensional sizes. In the last decade the reduction in size of microelectronic devices has been mainly due to the reduction of their components in the order of nanometers [1]. Different nanoscale materials can be used to obtain new applications such as water repellents thin films, anti-reflective layers, screens for computers, photo-cameras, and protecting layers.
The physical properties of these materials at these dimensions can vary in some cases in several orders of magnitude with respect to their bulk properties. Some physical properties that change with the size are the thermal properties. Previous works have reported the resistive thermal coefficient (RTC) [2] of gold films which varies between 0.0017 and 0.0047 °C⁻¹, when the film thickness changes between 110 nm and 880 nm, respectively.

The thermal properties characterization of materials in thin film geometry are performed using complicated and expensive methods, such as the differential nano calorimeters [3] where chemical etching to obtain complex nano-bridges and nano-switches is required. In this case an electrical pulse of some ms is used to determine the heat capacity of copper thin films among others [4]. Different ultra-fast scanning calorimeters have been developed, such as differential scanning calorimeter for thin films [5], differential nano-calorimeter for high-temperature [6], and ultrafast and non-adiabatic nano-calorimeter [7].

In this work we propose an experimental technique to measure the heat capacity of metallic thin films using an electronic circuit for applying an electrical micropulse at room temperature and atmospheric pressure. In the proposed technique the very short pulses applied on the metallic thin films increases its temperature without affecting the substrate. On the other hand, when a voltage is applied on a film during a specified time it is possible to measure with high resolution the changes in the electrical resistance of the film such that the corresponding changes on temperature can be obtained and consequently, the heat capacity C of the film. The next simple relation will be used to determine the heat capacity of metallic films.

\[
C = \frac{Q}{\Delta T/\Delta t} \quad (1)
\]

Where Q is the power applied on the metallic film and \(\Delta T/\Delta t\) is the initial slope of the heating profile (or cooling) of the films when an electrical pulse is applied. The duration of the electrical pulse to be applied on films, can be estimated from the analytical model proposed on a previous work [9].

2. EXPERIMENTAL SETUP

By using the constant time (\(\tau\)) values as proposed with a previous theoretical model [9] obtained when a metallic film is heated with an electrical micropulse, we developed an electronic circuit capable to apply controlled pulses between 100 and 500 µs and measure the changes of resistance on the metallic film with the time of the pulse. With these results, we can obtain the corresponding changes of temperature and the heat capacity of the film.

Figure 1 shows the electrical diagram of the electronic circuit proposed. The electronic circuit consists of a 2N2222A transistor as a handler for the opening and closing of a NTP45N06L MOSFET, which acts as a switch to an electric pulse. The circuit is operated by a USB 6216 data acquisition card which controls the transistor, which in turn drives the opening and closing of the MOSFET.
Figure 1 shows the two stages of the circuit: the sample load stage (dotted rectangle) which contains a Wheatstone bridge and the pulse stage. The sample load stage consists of the resistance $R_1$ ($2.26 \, \Omega$), two calibrated resistances $R_4$ and $R_5$, ($10 \, \Omega$) and a variable resistor $R_6$ used to balance the resistance of the sample in the Wheatstone bridge. The pulse stage involves the 2N2222 transistor, the MOSFET, the USB Card 6216 and the resistors $R_2$ and $R_3$, which values were calculated to obtain the voltage and currents needed to open and close the MOSFET switch. The values of $R_2$ and $R_3$ were selected in order to provide a difference of current between points 3 and 2 in the transistor, taking as a condition that the current in $R_3$ needs to be greater than switch 2. Equation (2) shows the calculation of these values.

$$I_3 = \frac{V_2}{R_2} \frac{V_{BE}}{R_3} = 2 \, mA$$

$$I_2 = \frac{I_{Load}}{R_{vth}} = 5 \times 10^{-9} \, A$$

Where, $V_{BE}$, is the nominal voltage of the transistor. The current $I_3$ is produced in the resistance to apply the potential for the power supply of USB-6216, $I_2$ is the saturation current of the transistor, given by $5 \times 10^{-9} \, A$, such that if $I_3 > I_2$, the transistor switch can work without problems.

An HP-6543A power supply was used to apply the electrical pulse to the sample.

To measure the changes in the electrical resistance through two copper electrodes attached to the film with silver paint, we proposed the electric circuit shown in Figure 2. In Figure 2 is included, as a part of the characterization system, a DPO-4054 Tektronix digital phosphor oscilloscope working at 2.5 Gigasamples/s used to capture the voltage signals with high resolution and real time. In order to measure the reduced variations in the electrical resistance of the sample, a Wheatstone bridge was included in the same Figure 2.
Figure 2. Implemented circuit including a Wheatstone bridge. The sample resistance R6 is included as a component of the bridge.

A voltage difference is applied between electrodes in points a and b. Previously, the bridge is balanced by means of the variable resistor R1, such that by applying the electrical pulse will suffer a misbalance, due to the variation of the sample resistance, meanwhile the other resistors maintain at constant temperature. The electrical current flowing through the sample is determined by the voltage measured in resistance R5 of the Wheatstone bridge by using the Ohm's law. Thus, we can know the variation of film resistance during the time of application of the electric pulses.

3. RESULTS
Figure 3 shows the implemented electrical circuit used to apply the micropulses. The Wheatstone bridge and the USB 6216 acquisition card to control the micropulse is shown.

Figure 3. Electrical circuit designed for pulses application. The main components are shown.
The circuit is used and calibrated with different gold thin films (100 nm thickness) deposited on polymeric Kapton substrates by thermal evaporation technique. We used flexible substrates in order to manipulate the gold film without suffering fractures, and give some facilities to measure the electric potential and manipulate the sample-electrodes system.

Figure 4 shows the behavior of the different voltages measured in three points of the circuit, during the application of an electric pulse of 300 mW during 250 µs. It is shown that the input voltage (Vi) to the Wheatstone bridge is higher as compared with the voltages measured in the points a and b. These last voltages correspond to the sample and the variable resistor, respectively. The voltages Va and Vb are very similar, although during the first 50 µs some differences can be seen, may be by the transitory regime of the Au due to the change of temperature during the pulse. Thus, the voltage will be constant until the film sample heats uniform.

Using equation 3, the change of the electric resistance of the film sample can be calculated by:

\[
R_0 = \frac{(V_{u1} - V_{u2}) + (R_1 + R_2)R}{(R_1 V_1 - V_{u1}(R_1 + R_2))}
\]  

(3)

Figure 5, shows the corresponding changes of electrical resistance vs time of the pulse. An increase in the electrical resistance of the Au film is shown, due to the Joule effect. It also shown the small dispersion of the data. Thus, the measured values during 250 µs have high resolution, and greatly improve the recently reported resolution in the literature [8].
Figure 5. Electrical resistance vs time of the pulse applied during 250 µs in the Au (100 nm)/Kapton system.

\[ \Delta R_x = 0.240 \pm 0.012 \, \Omega \]

Figure 6 shows the temperature profile of the Au sample during the electrical micropulse. Note the dispersion estimated from the profile, and the density of points measured during the short pulse of 250 µs.

Figure 6. Temperature evolution during the application of the pulse, in Au(100 nm)/Kapton system.
Taking into account the changes of resistance of the sample and the known relation \( \Delta T = \Delta R_x / \alpha R_0 \), it is possible to calculate the difference of temperature during the time of application of the pulse, where \( \alpha \) is the resistance thermal coefficient, \( R_0 \) is the initial resistance, and \( \Delta R_x \) is the change in resistance of the Au film. The increment in temperature measured on the analyzed Au sample was about 50 K, in good agreement with the values reported in the literature [8].

Using the profile of temperature, the applied power and the initial slope of the heating time (\( \Delta T / \Delta t \)) during the micro pulse application, the \( C \) value can be calculated by the next relation: \( C = Q / (\Delta T / \Delta t) \). Figure 7 shows the behavior of the heat capacity for the Au sample during the applied pulse of 250 \( \mu \)s. From Figure 7 it can be shown that during the first 50 \( \mu \)s of heat application, the values of \( C \) are very large because the film was in the transitory heating; however, with time its value tends to reach a stable value. The estimated value of \( C \) for the Au sample with 100 nm-thickness was 950 \( \pm 30 \) nJ/K, which agrees with data reported in the literature (980 nJ/K) [8].

![Figure 7. Heat capacity \( C \) of Au (100 nm) film as a function of the time of the micropulse for a Au/Kapton system.](image)

**Figure 7.** Heat capacity \( C \) of Au (100 nm) film as a function of the time of the micropulse for a Au/Kapton system.

4. CONCLUSIONS
Heat capacity \( C \) of Au films deposited on Kapton substrates was obtained by using an experimental system working at room temperature and atmospheric conditions. The novel experimental system is based in applying an electronic micro pulse which duration depends basically on the film thickness, but can be in the range of 30 to 300 \( \mu \)s. An electronic circuit including a 2N2222 transistor and a NTP45N06L MOSFET was implemented to generate the very short pulse with high resolution. The slight variations in the electrical resistance of the Au film were measured by means of a Wheatstone bridge, where the resistance of the film is a component of the bridge. The control of the pulse was done with a USB-6216 card. Heat capacity values close to those reported in the literature as obtained with microcalorimeters for samples of Au films was obtained with our proposed method.
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REFERENCES