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Investigation of the thermal properties of thin solid materials at different temperature levels using a set of microresistors

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ABSTRACT

The measurement of thermal properties of solid materials at different temperatures above ambient is investigated using a set of microresistors. Samples consisted of suspended films with sets of long, parallel resistive wires deposited on their surfaces. One resistive wire was heated by an alternating current. Surface temperature changes in DC and AC regimes were then detected by measuring the change in electrical resistance of the other wires deposited on the surface. The length of wires was chosen so that they may be assumed isothermal and such that heat diffusion acts perpendicularly to their axes. By measuring the dependence of the surface alternating temperature oscillation on the modulation frequency f and on the separation between the heating wire and the probing wires, the thermal diffusivity of the sample was determined. Through adjustment of the alternating current amplitude in the source wire, the temperature at which the thermal diffusivity of the sample was evaluated was finely controlled. For the validation of the method, pure silicon samples were first studied. An experimental bench was set up and resistive source and probes were experimentally characterized. Results obtained from ambient temperature to 500 K for pure silicon are in accordance with reference data found in the scientific literature.

1. Introduction

Characterization of the thermal diffusivity and conductivity of bulk materials and thin films is very important in various application domains (microelectronic, nuclear, materials for aeronautics…). To achieve such a determination, different methods have been developed. They can be classed in two main groups, the non-contact optical methods, also called photothermal methods and the contact methods using contact electrical means.

For the non-contact methods, the thermal source is usually photothermally created. In many cases, the sample is illuminated by a pump modulated laser beam. This laser causes heating of the sample through absorption and subsequent thermalization of energy. By covering the surface with a thin layer that is optically thick at the wavelength of the light, the heating at the sample surface is localized. A spot, a straight line or a domain of the surface can be illuminated. They correspond respectively to cylindrical, plane, and 1D propagation regimes of thermal waves. The analysis of the temperature of the sample surface as a function of the modulation frequency of the thermal source or the distance to the thermal source allows the determination of the sample thermal properties. Pulsed heating sources are used also as in the flash method [1,2]. Through numerous detection schemes available nowadays such as mirage setup [3,4], photoreflectance microscopy [5–8] or photothermal radiometry [9,10], the variations of the surface temperature are measured. Thermal parameters are then extracted from fitting these measurements with curves calculated with the model of heat spreading in the sample.

Considering now the thermal methods using contact electrical means, various methods discussed in Ref. [11] are based on the transient hot strip source technique proposed by Gustafsson [12] and the 3ω method of Cahill [13]. Both methods were initially developed for measuring the thermal conductivity of bulk materials. In these methods, the sample is heated by a thin metal strip deposited on the sample surface. The metal strip is used as a heater and also as temperature sensor. The sample heating is either transient (transient hot strip method) or modulated (3ω method). The measurements of

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the heater temperature are coupled with heat transport modeling. Through these measurements, the sample thermal properties are estimated. The uncertainty in the estimations depends then on the parameters used in the thermal modeling of the experiment. Parameters such as contact resistance between the heating strip and the sample, the design and dimensions of the metal strip and substrate can in particular play a major role and have to be taken into account [11,14]. However, characterization of the thermal conductivity of thin films requires certain adaptation of the methods. Using these methods, sufficient heat should pass through the sample for an adequate study of the thermal properties of the sample and a simplified interpretation using straightforward analytical approaches. To address these issues, solutions include adjustment of the sample geometry or fabrication of sophisticated and delicate suspended structures. Moreover, transient methods such as the membrane method have been developed [15,16]. As the method used in this work, this technique includes separate heater and temperature sensors and has been developed for the identification of the in-plane thermal conductivity of thin films [15,16].

Despite their large applicability, all the methods given above need a particular heavy conditioning of set-ups for measurements at different levels of temperature above ambient [7,15,17–19]. Moreover, they need specific apparatus for these measurements. Based on the principle of the membrane method, this paper approach demonstrates the successful extraction of thermal properties of highly diffusive bulk materials above ambient using only a set of microresistors. Quite simple, it can easily be implemented by other investigators.

Section 2 is dedicated to present the method. Section 3 describes the sample under test and the different steps of fabrication. To prove the method applicability, the case of a sample of pure silicon has been chosen. The numerical simulations used to design the samples and to estimate the uncertainty in the thermal parameters determination are given in Section 4. Section 5 describes the different parts of the experimental set-up with their functionality. Our experimental results are analyzed and compared with data found in the scientific literature. Finally, we conclude and give the perspectives of this work.

2. Method used

2.1. Principle

For the experiments, the material to be studied is shaped in a flat suspended film. As shown in Fig. 1, an array of parallel and long resistive wires is deposited on the surface of the sample. One of the resistive wires is heated with an alternating current I of magnitude I_{ac} at angular frequency $\omega = 2\pi f$. The power P dissipated in this wire may be written as

$$P = R.I^2 = R.I_{ac}^2/2 + R.I_{ac}^2/2.\cos(2\omega t) \quad (1)$$

where R is the electrical resistance of the heated wire.

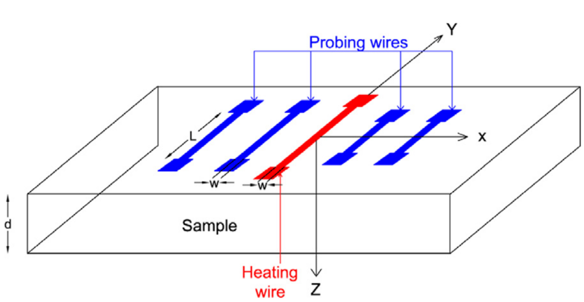

Fig. 1. Schematic of the implemented sample. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

In the material, conductive diffusion of this heating produces a continuous temperature variation called ΔT_{DC} and alternating temperature oscillations at angular frequency 2ω called $\Delta T_{2\omega}$. The length of all the wires is chosen large so that they may be assumed isothermal along their length (in the Y direction in Fig. 1). However, the probing wires are shorter than the heating wire to detect mainly the lateral heat spreading from the heating wire.

By measuring the electrical resistance change of the probing wires $\Delta R_{2w}(x)$, the surface temperature $\Delta T_{2\omega}(x)$ is detected

$$\Delta R_{2\omega}(x) = R_0(x)\beta\Delta T_{2\omega} \quad (2)$$

here $|x|$ is the distance from the heating wire, $R_0(x)$ is the electrical resistance of the probing wire at ambient temperature T_a and β is the temperature coefficient of the electrical resistivity. A direct current i_0 of small magnitude is generated through the probing wires for the $\Delta R_{2\omega}(x)$ measurements. Using a lock-in amplifier, the second harmonic voltage at the probing wires is measured

$$\Delta V_{2\omega}(x) = \Delta R_{2\omega}(x)i_0 = R_0(x)\beta\Delta T_{2\omega}(x)i_0 \quad (3)$$

By measuring the $\Delta T(x)$ dependence on the modulation frequency $F=2f$ and on the distance $|x|$, the sample thermal properties are determined.

2.2. Modeling

The thermal diffusivity of the sample α is given by

$$\alpha = \lambda/(\rho_0 C) \quad (4)$$

where λ, ρ_0, and C are the sample thermal conductivity, density, and heat capacity respectively. The thermal diffusion length μ in the sample material is given by

$$\mu = (\alpha/(2\pi f))^{1/2} \quad (5)$$

Identifying α is all the easier as μ is large compared with the thickness d of the suspended film. This condition is verified while working at small frequencies. In this case, the thermal gradient in the sample thickness may be assumed null (in the Z direction). Moreover, d is chosen so the Biot number defined as $Bi = hd/k$ is much less than 0.1, where h is the convective heat transfer coefficient.

With the fact of $\mu \gg 2\pi w$ where w is the width of the wire, the heating wire is considered punctual. From the resolution of the heat transfer equation, the second harmonic temperature decay measured along a distance x from the heater may then be written as

$$\Delta T_{2\omega}(x) = A\exp(-x/\mu)\exp(-ix/\mu) \quad (6)$$

where

$$A = P/(2dl\lambda)(\alpha/(2\omega))^{1/2} \quad (7)$$

here P is the electrical power dissipated in the heating wire and l is the sample width.

The thermal diffusivity α is deduced from the attenuation of the magnitude $\exp(-x/\mu)$ and the variation of the phase $(-x/\mu)$, both as a function of x. Let α_{am} *and* α_{Φ} the thermal diffusivity extracted from the magnitude attenuation and the phase lag respectively. Then λ could be deduced from A in Eq. (7). By measuring the signal of the probing wires and using Eqs. (3)–(6), the thermal properties of the sample are estimated. This needs a good knowledge of the electrical properties of the material composing the wires. Adjusting the amplitude I_{ac} in the source allows controlling the sample DC temperature at which the thermal properties are identified. Depositing many wires at the sample surface allows the estimation of the DC temperature

$$T_{DCexp} = (1/N)\sum_{i=1}(R(x_i)-R_0(x_i))/(R_0(x_i)\beta) + T_a \quad (8)$$

where N is the number of the probing wires and i is an index for a considered probing wire. A careful design of the sample thickness