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Joule heated metallic microwire devices for sub-microsecond *T*-jump experiments

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ABSTRACT

We report a study of the transient heating response of submicrometric gold wires on oxidized Si substrates, highlighting their potential as versatile platforms for creating fast temperature variations (*T*-jumps). To characterize electrically the transient heating response, we developed an original differential resistance setup that excites single wires with current pulses and provides a signal proportional to the ΔT induced. A reproducible sub-microsecond transient heating response (for $\Delta T \leq 80$ °C) was observed for the heaters, regardless the substrate temperature or the presence of a thermal load (polymer film) on the surface. We also developed a simplified mathematical model that reproduced the main experimental observations and provided valuable insights into the dynamics and stability of the heating process. As a proof of concept, our electrical setup was also coupled to a time-resolved optical microscopy setup to detect the transient thermal response of a well-known luminescent thermometer molecule (Rhodamine B).

1. Introduction

Joule heated wires have the potential for becoming submicrometer scale heating elements for implementing diverse thermal excitation and temperature control strategies. Their electrothermal behavior depends on the resistivity of the heater, its temperature coefficient and the heat transfer between the resistor and its surroundings [1]. During the last few decades, micrometric "hot wire" and "hot-strip" approaches based on either static or dynamic electrical excitation have been integrated into diverse characterization techniques in material science, chemistry and biology. For example in static (DC) mode, the thermal profiles of metallic micro/nanowires deposited on oxidized Si wafers have been employed for developing fluorescent micro-thermometry with high spatial resolution using far- [2] and near-field [3] setups in both liquid and dry environments. Also, this configuration has been used to perform three-dimensional imaging of the heat dissipation around a wire in liquid conditions by means of quadriwave lateral shearing interferometry [4]. Such submicronic heaters have also been employed to induce thermal phase transition in thin films and to perform AFM thermomechanical imaging during the phase change process [5]. In a similar configuration, the Joule heating on arrays of Si or Cr nanowires was used to selectively ablate a protective polymer layer from the region right on top of the wires and to expose it for a subsequent localized surface functionalization with chemical linker molecules [6] or to perform other chemical reactions such as polymer cross-linking or direct and localized hydrothermal synthesis of metal oxide nanostructures [7].

On the other hand, the dynamics of Joule heated microwires has also been widely explored with electrical techniques in the time domain (transient regime) and frequency domain. In a basic transient regime experiment, the change of resistance of a heating element after a sharp voltage/current step is studied. The magnitude of ΔR and the characteristic time (relaxation time) at which this variation takes place carries information about the temperature rise and thus, the thermal properties of the heater itself and its surroundings. To this aim closely related techniques have been developed such as: pulse non linearity measurements (PMN) [1,8], transient electrothermal technique (TET) [9], transient hot strip (THS) [10] and ultra-fast transient hot-strip (UFTHS) [11,12] techniques. As for the measurements in the frequency domain, the so-called 3ω method is the most commonly employed. Here, a sinusoidal current is fed through the heater which leads to a

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temperature variation and thus, a resistance change at the second harmonic of the excitation frequency; this distortion gives rise to a voltage component across the heater in the third harmonic. The frequency dependence of the amplitude and phase of this 3ω signal is used to infer the thermophysical properties of the environment around the wire [13]. All these techniques in time and frequency domain have been successfully extended to characterize the thermal conductivities and thermal diffusivities of different solids [13], as a function of temperature [14], in the form of multi-system thin films deposited on a substrate [1,15–17], or suspended conductive and non- conductive microwires [9], as well as the dynamic specific heat of solid or liquids [18–21] and even detecting the phase transition on thin films [22]. Furthermore, these schemes have been integrated into microfluidic chips to measure thermal properties in small liquid volumes [23] and to achieve a real-time characterization on nanoliter samples [24]. Additionally, the alternative temperature variation generated by a microheater in a microfluidic channel combined with fluorescence detection was successfully employed to perform titration based on the principle of a “thermokinetic band-pass filter”. Thus, it was possible to discriminate a species of interest present in a solution with diverse interfering compounds without any separation step [25].

The temperature control, confinement of the thermal perturbation and sensitivity obtained in all the applications mentioned above rely strongly on a thorough design of the electrical excitation setup, the geometry of the heater and the thermal properties of its environment (underlying substrate, passivation layer, *etc.*). In general, the dynamic thermal behavior of this type of heaters has been rationalized with the aim to facilitate the study of the propagation of the thermal wave through the media surrounding the wire. From a different perspective, this article reports a study of the transient heating response of submicrometric gold wires on oxidized Si substrate that highlights their potential as versatile platforms for creating fast temperature variations (T-jumps). Due to their small thermal mass, micro- and nano-wire based heaters are particularly interesting in terms of thermal response time and highly localized heating generated with low power excitation. Here, we demonstrate that under our experimental conditions, such devices can be successfully employed for inducing fast thermal perturbations of ca. 80 °C below the microsecond range even with relatively high thermal loads. The simplicity of this approach facilitates the integration of the heaters into existing on-chip technologies for thermal excitation and observation of temperature dependent processes such as polymerase chain reaction (PCR), temperature gradient focusing (TGF), thermotaxis, thermophoresis, *etc*, [26].

The heaters we studied consist in 50 nm thick, 80 μm long and 500 nm or 1 μm wide gold wires (Fig. 1(a)). They were patterned on an oxidized Si substrate. To characterize electrically the transient heating response of the wires in the sub-microsecond range, we developed an original differential resistance setup that generates current pulses for the excitation of the heaters and provides a signal proportional to the ΔT induced in the wire. Furthermore, we propose a simple mathematical model that reproduces the main experimental observations. This allowed us to gain valuable insights not only on the general dynamic thermal behavior of the heaters, but also on the stability of the heater temperature while exciting with electrical current steps. As a proof of concept our electrical setup was also coupled to a gated CCD camera in order to perform time-resolved luminescence micro-spectroscopy and detect the dynamical thermal response of a well-known luminescent thermometer material (Rhodamine B).

2. Materials and methods

2.1. Fabrication process of the heaters

The fabrication process on oxidized Si wafers involves a mix and match strategy with three levels. In level one, conventional photolithography employing the negative photoresist NLOF, a subsequent thermally evaporated 10 nm Ti/200 nm Au bilayer and a lift-off process in acetone were employed to create the optical alignment marks for the other two levels. The Ti layer works as an adhesive layer between the Au and the SiO_2 passivation layer. In level two, a positive EBL resist layer (PMMA, Polymethylmethacrylate) was spin coated on the surface of the wafer and then it was selectively exposed in the chamber of a RAITH-150 EBL writer. After this, the patterns were developed by immersing the wafer a 1:3 solution of MIBK (Methyl isobutyl ketone): IPA (Isopropyl alcohol) during 45 s. Then, a 10 nm Ti/ 40 nm Au bilayer was thermally evaporated and lifted in acetone to leave only the metal that was in touch with the substrate *i.e.,* the metallic wires. In level three, the chips were completed with a second photolithography employing the NLOF resist, a 10 nm Ti/ 300 nm Au bilayer and a lift-off in acetone to create the connecting pads for the wires. Fig. 1(b) displays an example of one of the heaters under study.

2.2. Finite element simulations

The temperature in our devices was modeled in three dimensions using COMSOL's “Joule Heating Module” coupled, with “Heat Transfer Module”. Basically, the heat transfer across the nanowire can be described by Fourier's law and may involve different heat transfer mechanisms (conduction, convection and radiation).

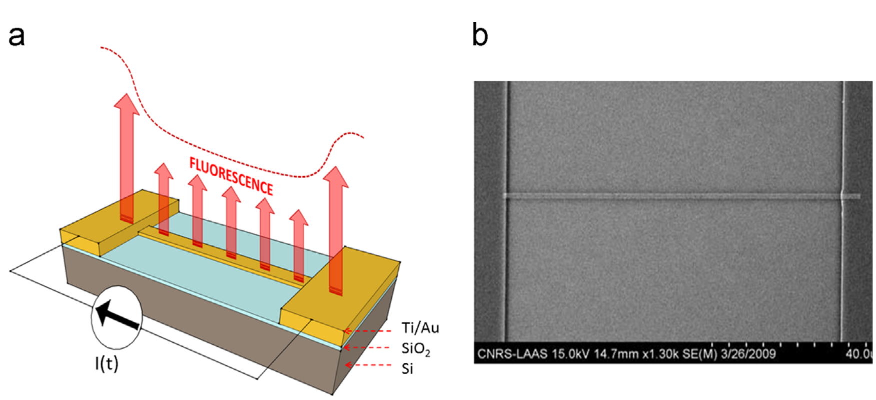

Fig. 1. (a) Scheme of the electro/optical device. A gold microwire connected to two gold electrodes is patterned on a silicon substrate with a 300 nm insulating layer of SiO_2. In order to perform optical measurements, a layer of the Rhodamine B is employed as a fluorescent thermal probe. (b) Scanning Electron Microscopy image of a 50 nm × 1 μm × 80 μm wire.