

SnO₂:Sb – A new material for high-temperature MEMS heater applications: Performance and limitations

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Abstract

MEMS micro heater devices capable of long-term operation at temperatures up to 1000 °C are presented. The enhanced long-term stability has been achieved by employing antimony-doped tin oxide (SnO₂:Sb) as a substitute for the conventionally used noble metal heater resistors. A detailed investigation of its high-temperature stability reveals that degradation is caused by out-diffusion of Sb impurities from the SnO₂ film. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

MEMS micro heater devices are key components of chemical sensing microsystems. Possible areas of application include modulated thermal infrared (IR) sources, flameless ionisation detectors and surface ionisation devices [1–5]. Such applications call for micro heater devices that can withstand prolonged operation at temperatures up to 1000 °C. In previous work we have demonstrated thermal IR emitters employing antimony-doped tin dioxide (SnO₂:Sb) as a novel heater material and showed that such devices exhibit a high-temperature stability that is superior to a whole range of metallic and semiconductor heater materials [6].

In the present work we present the results of a detailed investigation including electronic transport, optical absorption, X-ray diffraction and X-ray photoelectron spectroscopy (XPS) measurements to reveal the high-temperature degradation mechanisms that limit the lifetime of SnO₂:Sb heater materi-

als. We find that – unlike most other heater materials – the high-temperature stability of SnO₂:Sb is not limited by electromigration but rather by outdiffusion and evaporation of Sb dopants from the SnO₂ bulk.

1.1. MEMS heater devices operating at $T > 800$ °C

Before turning to the solid state properties of SnO₂:Sb we present a high-temperature MEMS heater device that employs this material as a resistive heater element and that was designed to work as a thermal IR emitter. Considering this device we want to acquaint the reader with those arguments that have led to the decision to consider SnO₂:Sb as a MEMS-compatible heater material.

The general layout of the IR emitter device is shown in Fig. 1. The top view on the left-hand side shows that the device exhibits a hotplate architecture featuring a thin silicon membrane suspended by four silicon bridges within a massive chip frame. This kind of layout was chosen to achieve low power consumption and a short thermal response time – two features which are essential for thermal IR emitters [7].

The outer chip dimension is 5 mm × 5 mm. The heated membrane itself measures 1.5 mm × 1.5 mm being suspended within a massive Si frame with an inner opening of 2 mm × 2 mm. The

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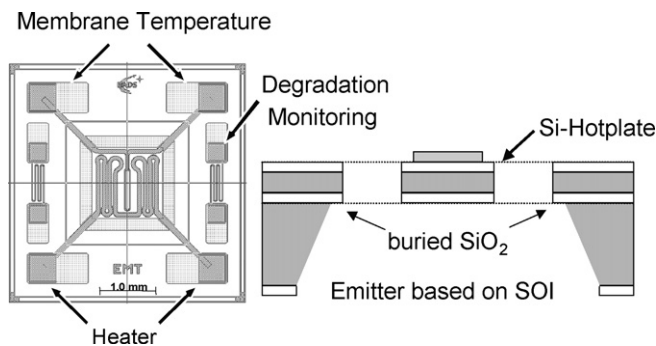


Fig. 1. Mask layout of a thermal IR emitter with on-chip temperature sensing elements for controlling the membrane temperature and for assessing the long-term drift of the heater meanders; right-hand side: cross section through the device exhibiting an etch trough and a hotplate suspended within a massive silicon rim.

Si suspensions have a length of $350\text{ }\mu\text{m}$ with a width of about $150\text{ }\mu\text{m}$. On top of the membrane a heater meander is placed with an overall length of about 12 mm and a width of $100\text{ }\mu\text{m}$. The cross sectional view on the right-hand side shows that this hotplate has been fabricated from silicon-on-insulator (SOI) wafers with a thin top silicon layer of about $6\text{ }\mu\text{m}$ sitting on top of a $350\text{ }\mu\text{m}$ thick handle wafer. Both silicon layers are separated by an intermediate SiO_2 layer, about 300 nm thick.

Aiming at high operation temperatures the hotplate structure should consist of materials capable of operating at temperatures up to $1000\text{ }^\circ\text{C}$ for prolonged periods of time. A suitable base material is mono-crystalline silicon. In ambient atmosphere silicon tends to form a natural oxide whose growth is diffusion-controlled and thus self-limiting. Such oxide layers also perform very well as high-quality insulator layers. The crystalline silicon base structure itself possesses a high thermal conductivity, which is good for achieving short thermal response times and thus a high modulation depth when MEMS heaters are operated in a discontinuous ac mode [1]. High thermal conductivity, however, is not compatible with low electrical power consumption [8,9]. For this latter reason, closed membrane designs cannot be used; hotplate structures such as the one shown above need to be used to limit the cross sectional area of the heat conduction paths from the heated hotplate to the cold silicon rim [10–14]. On top of the hotplate the thermal IR emitter device features a heater meander for electrical heating and a temperature sensing resistor for monitoring the temperature of the IR emitting surface. Two additional resistors on the cold chip frame have been included for referencing against the heater meander resistance and the temperature sensing resistor on the hotplate. Making such comparative measurements, high-temperature induced changes in the heater and the hotplate temperature sensing resistors can be detected [15].

Having chosen crystalline silicon as a base material for the hotplate architecture, the next questions relate to materials that are suitable for serving as resistive heater elements, chip thermometers and electrical insulation layers. In this context it is not only sufficient to choose materials that can serve these purposes as such; rather it is extremely important that these materials are also compatible with the silicon substrate material and its pro-

cessing. In order to find such materials, an extensive survey for the right materials and material combinations had been made [6]. To this end MEMS heaters with different heater materials on top had been produced and tested for their operational performance. During high-temperature operation the heater materials not only suffer from the high-temperatures themselves but also from the mechanical stress generated by the thermal expansion mismatch between substrate and thin film materials on top and from the high current densities applied. Aiming at low operation voltages the most obvious choice for a heater material are noble metals such as platinum (Pt). Unfortunately, Pt exhibits a relatively large thermal expansion mismatch with respect to Si, SiC and SiO_2 , i.e. with the substrate material chosen and compatible passivation and insulation layers. A better match can be obtained using metallic alloys such as CrSi_xN [16]. The price to be paid for the better thermal expansion mismatch is a significantly lower electric conductivity and an increased tendency towards oxidation, which results in a relatively large long-term drift. Accepting a lower electrical conductivity, heavily doped SiC would be an ideal choice with regard to minimizing the thermal expansion mismatch [17–22]. The problem with high-temperature oxidation, however, would still remain [23]. Keeping these constraints in mind we found that an optimum choice for a high-temperature resistive heater element might be heavily doped SnO_2 [24,25]. Attractive properties of SnO_2 are, that as a wide-bandgap semiconductor ($E_g \sim 3.5\text{ eV}$), SnO_2 stays extrinsic up to extremely high temperatures ($\sim 1500\text{ }^\circ\text{C}$) and, as a stoichiometric oxide, SnO_2 is inert towards further oxidation. Furthermore, this material can be doped by adding group V elements such as Sb to obtain conductivities comparable to degenerately doped Si.

Degenerately doped $\text{SnO}_2\text{:Sb}$ layers were produced by e-beam evaporation of mixtures of SnO_2 and Sb powders. Annealing is performed in order to activate the antimony dopants inside the deposited layer. Here, it was carried out in a standard furnace in ambient air with the temperature being ramped up slowly to $1050\text{ }^\circ\text{C}$. Thereafter, the temperature is kept constant for about 4 h. Systematic doping experiments revealed that with this annealing treatment, average conductivities of about $4500\text{ }\mu\Omega\text{ cm}$ can be obtained when Sb concentrations of about 5% are employed. Structuring of these layers into heater meanders is performed by depositing the $\text{SnO}_2\text{:Sb}$ onto pre-patterned photoresist masks and subsequent lift-off. This requires $\text{SnO}_2\text{:Sb}$ to be deposited at room temperature as an amorphous layer and to be structured prior to the activation anneal. The deposited $\text{SnO}_2\text{:Sb}$ film thickness was determined to be 950 nm . Fig. 2a presents a scanning electron micrograph of a $\text{SnO}_2\text{:Sb}$ heater element and Fig. 2b an IR emitter device operating at about $1000\text{ }^\circ\text{C}$.

In order to assess the long-term performance of such heater elements, accelerated degradation tests had been performed. In these tests the MEMS heaters were operated at a constant dc power input, i.e. at constant membrane temperature for a period of 30 min. Thereafter, the heating power input was increased in 50 mW steps until the heater finally failed. In order to maintain constant power consumption, the dc current was permanently measured and the supply voltage adapted accordingly. The appropriate membrane temperatures had been ascertained by a

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