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On-chip fabrication of surface ionisation gas sensors

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ABSTRACT

Microelectromechanical systems (MEMS) with integrated heaters, originally designed for the readout of the resistance of metal oxide (MOX) layers, were configured to observe surface ionisation (SI) gas signals. Interdigital platinum (Pt) electrodes on top of the dielectric membranes acted as ion emitting layers while flat-plate counter electrodes, positioned at a short distance above them, were used for the current readout. In this work, we show that this device configuration leads to SI responses orders of magnitude higher than those previously reported for thin-film, flat-plate devices and fully comparable to the performance of parallel-nanowire devices (PNDs). The high ionisation efficiency of our MEMS devices, which are suitable for large-scale production and further integration steps, is attributed to the electric field enhancement that takes place at the sharp edges of the Pt electrodes.

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1. Introduction

Surface ionisation (SI) gas sensors [1–6] are an interesting alternative to the widely employed resistive (RES) detectors [7–10]. Both kinds of devices rely on the adsorption of target gases on heated sensitive surfaces, but they strongly differ in the subsequent steps that actually lead to electrically detectable output signals. Whereas in RES sensors the key reaction is surface combustion [7–10], it is the transfer of valence electrons from adsorbed analytes to empty electron states inside the adsorbent solid what matters in SI devices [1–6].

Significant differences also exist in the electrical readout configuration of RES and SI technologies. In RES sensors the in-plane electronic resistance of metal oxide (MOX) layers is monitored. On the contrary, SI sensors detect positive ion currents that can be extracted from heated MOX or noble metal surfaces when easily ionisable analytes adsorb on them. The so-formed analyte ions travel thus through an air gap towards a negatively biased counter electrode, positioned at a short distance near the ion emitting layer [4–6].

Previous work on SI fundamentals showed that this technology also follows very different selectivity rules towards gases than conventional RES sensors. Whereas it is the ease of combustion which matters in the RES case [7–10], ionisation energy and proton affinity of target gases determine the SI response [4–6]. The latter two criteria explain why SI detectors are highly sensitive and selective with regard to amines and hydrocarbons with amine functional groups. Recently, these features enabled the design and implementation of very sensitive, selective and simple detectors for illicit drugs [11,12].

Ion extraction from a heated solid relies on the presence of strong electric fields acting perpendicular to the emitter surface. In our previous works we used platinum (Pt) and tin oxide (SnO_2) thin films as ion emitters together with flat-plate counter electrodes directly facing the emitter surfaces (air gap = 1 mm) [4–6]. These parallel-plate-capacitor-type devices with active areas of about 0.1 cm^2 lead to amine ion currents in the order of hundreds of nanoamps (10^{-7} A). Emitter temperatures ranged between 500 and 600 °C and extraction potentials up to 1000 V. Nanostructured SI devices, on the other hand, which consisted of parallel SnO_2 and Pt nanowires in a planar configuration, exhibited higher ion current efficiencies at lower temperatures (~300 °C) and moderate extraction potentials (~5 V) [13,14]. The enhanced level of current emission in parallel-nanowire devices (PND) was explained by the crowding of electrical field lines at the emitter surfaces in this particular geometry [14]. A disadvantage of PNDs, however, is that their production requires multi-step, time-consuming fabrication

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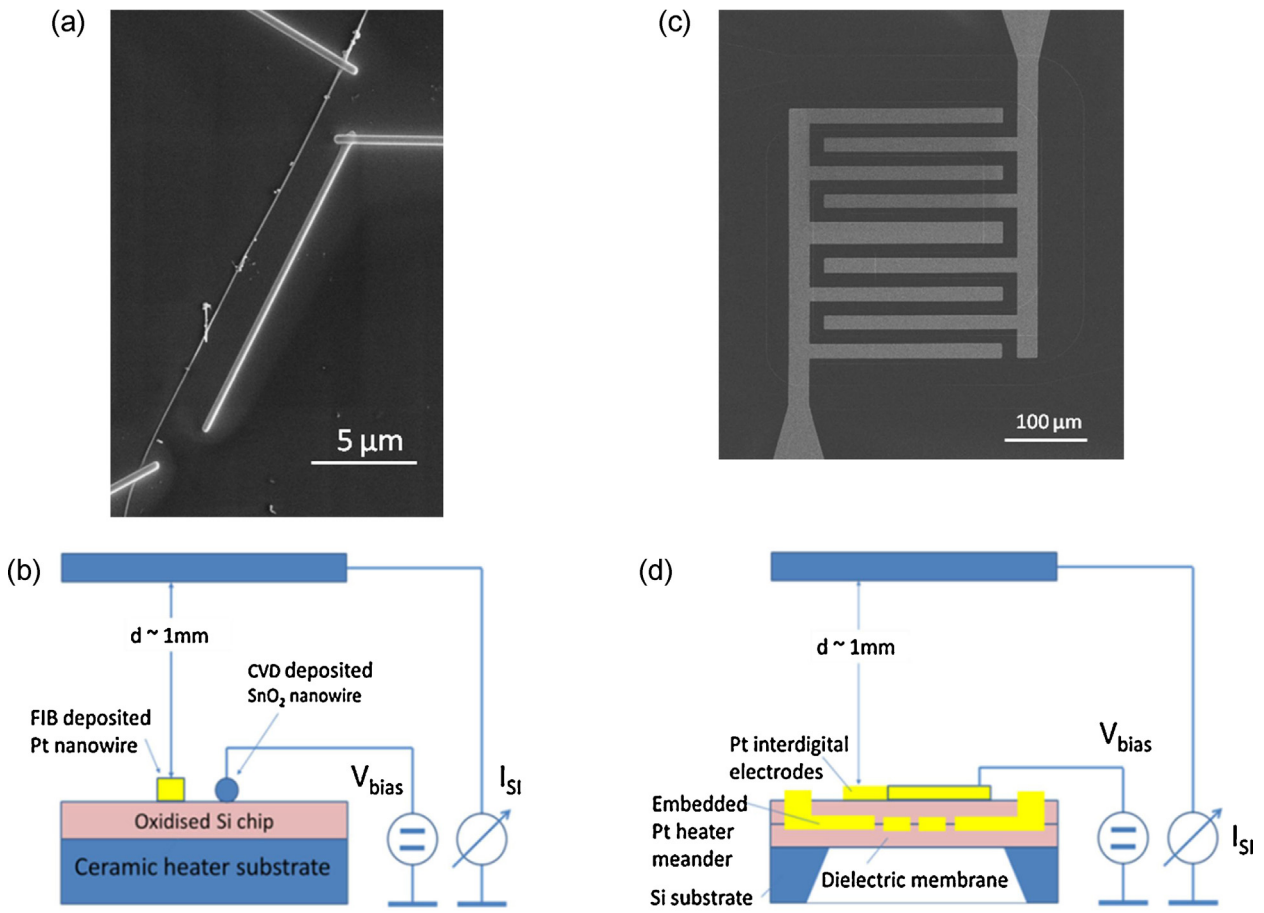

Fig. 1. (a) Parallel nanowire device (PND) featuring a SnO_2 nanowire and a FIB-deposited Pt-strip. (b) Vertical SI readout architecture applied to the PND in (a). (c) General view of a pair of interdigital Pt electrodes onto a MEMS hotplate. (d) Vertical SI readout architecture applied to the MEMS chip shown in (c).

processes and complex techniques, such as Focused Ion Beam (FIB) lithography, which limits their use to the role of research prototypes [15].

In this work, we present new high efficiency SI devices with gas sensing performances comparable to PNDs but suitable for large-scale production. Interdigital Pt electrodes, formed on MEMS micro heater devices by standard photolithography were used to extract ions (emitter) while working with vertical readout architecture similar to the one already employed in SI flat-plate devices [4–6]. The superior efficiency of our present MEMS approach is also explained by the crowding of electrical field lines that takes place at the sharp edges of the photo-lithographically patterned Pt-electrodes.

2. Materials and methods

The device architectures investigated in this work and the different SI current readout configurations are illustrated in Fig. 1. A scanning electron microscope (SEM) image of a PND featuring an individual SnO_2 nanowire running parallel to a FIB-Pt strip deposited at a distance of about 2 µm from the metal oxide nanowire is shown in Fig. 1a. Both elements were positioned on the surface of an oxidised silicon chip with pre-patterned macroscopic Au/Ti/Ni electrodes to facilitate the electrical contact to the external circuitry. The nanofabrication steps necessary to obtain such devices were described in previous works [13–15]. To enable the SI readout either from the SnO_2 or the Pt nanowire, a cold, flat-plate counter electrode was positioned at a distance of about 1 mm from the chip surface. Electrical tests were performed with the help of a Keithley 6517A Source Meter (Fig. 1b).

The design of the MEMS devices, which are the subject matter of this paper, is presented in Fig. 1c and d. They are based on commercial MEMS hotplates with integrated heaters and a pair of interdigital electrodes at the membrane surface that were initially designed for the RES readout of custom-specific MOX sensing layers [16]. Here, the Pt-interdigital electrodes were used as ion emitters, which were connected to external detection circuits as shown in Fig. 1d.

Heating of the devices was performed as illustrated in Fig. 1 using macroscopic ceramic substrates with screen-printed heater meanders and temperature sensors on their backside [4–6]. For gas measurements, the sensor chips were mounted inside a custom-built stainless chamber with heated chamber walls (T_{wall} = 80 °C). The chamber contained a gold-plated counter electrode for the SI readout. Precise positioning of this electrode relative to the emitter surface was done with the help of a micrometre screw. Premixed gases could be guided through the system using a customized gas test rig. To evaluate the SI response of those analytes which do not naturally abound as gases under normal ambient pressure–temperature conditions, such as the easily ionisable fluid dibutylamine (DBA), the test rig was also equipped with an automated syringe injector for dosing and vaporizing liquids [17].

3. Results and discussion

3.1. Gas response characteristics of MEMS emitters

Fig. 2 shows that the MEMS emitters exhibited SI responses similar to those of our PNDs operated in the vertical readout

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