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Advanced Room Temperature Hydrogen Sensor based on Interdigitated Electrodes and Polycarbazole Membranes

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Summary:

In this work, the development of a new hydrogen sensor based on an interdigitated Pt-based electrode fabricated with semiconductor technology and conjugated polymers based on polycarbazole as selective receptor is described. The major novelty of this sensor is the ability to operate and measure at room temperature, which makes it an energy-efficient and safe solution and the mass fabrication of microelectrodes. The H_2 sensor results demonstrate that the sensor is capable of measuring in a wide range of H_2 concentrations from hundreds ppm up to low explosive level (4%) in air, containing different relative humidity, at room temperature with good linearity.

Keywords: planar microelectrodes, interdigitated electrodes, electrodeposition, polycarbazole, hydrogen detection.

Headlines

- New H_2 gas sensor based on a polycarbazole conducting polymer (PCz).
- Microelectrode based on a Pt interdigitated electrode (IDE).
- Room temperature hydrogen gas sensor.
- Detecion of wide H_2 concentration range.
- High selectivity to potential interfering gases.

Background, Motivation an Objectives

Industrial processes involving hydrogen are becoming common, requiring relevant safety measures to prevent hydrogen fires and explosions. Reliable and real-time monitoring of hydrogen gas concentrations is a key element for ensuring safety. Despite this, existing hydrogen sensing solutions have many drawbacks, such as: a) sensors can be potential ignition sources due to requiring elevated temperatures for their operation; b) sensors have a limited range of H_2 concentrations or respond slowly to sudden H_2 concentration changes; c) sensors are cost-intensive in terms of both unit price and operation. In that context, the objective of this work is to address the needs of the "hydrogen industry" by developing an efficient solution for sensing hydrogen gas in industrial conditions that mitigates the above drawbacks.

Interdigitated electrodes (IDE) are attracting due to their distinguished geometry (Fig. 1A) and electrochemical behaviour compared with planar electrodes, whose can be applied in non-conductive mediums like deionized water or air [1]. The IDE surface can be modified with other materials to enhance their selectivity to the target gas.

On the other hand, polymers are widely utilized in literature for sensor modification. Especially noteworthy are those with structures containing aromatic rings, as this renders them soluble in organic solvents, simplifying their synthesis and manipulation. Within this group, carbazole stands out for multiple reasons. It is a fully aromatic unit, providing a better chemical stability; its nitrogen atom can be easily substituted in order to increase the solubility or modify the optical and electrical properties; and its structure has a bridged biphenyl unit resulting in materials with a lower band gap than traditional poly(p-phenylene)s.

The objective of this study is the fabrication of interdigitated electrodes and their subsequent modification with polycarbazole, aiming to propose a novel alternative method for the determination of hydrogen at room temperature. These sensors will be fabricated using mass production microelectronic technologies allowing the scaling-up of the whole sensor fabrication process.

Methodology

The IDE is fabricated following compatible CMOS silicon technology, at the Institute of Microelectronics of Barcelona (IMB-CNM-CSIC). The technological process is divided into different steps as shown in Fig. 1B, where the conductive material could be Pt or Au.

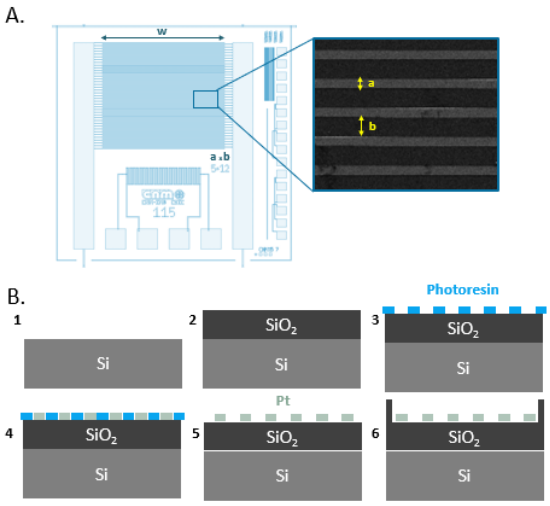

Fig. 1. (A) Design of the IDE where a is the digit width and b the separation between them; (B) Scheme of the fabrication process.

Once the chips are manufactured, they are packaged in a PCB and the electrical parts protected allowing them to be used in aqueous environments. Modification of the IDS can be carried by means of the electrodeposition of polycarbazole (Fig.2.) with cyclic voltammetry technique.

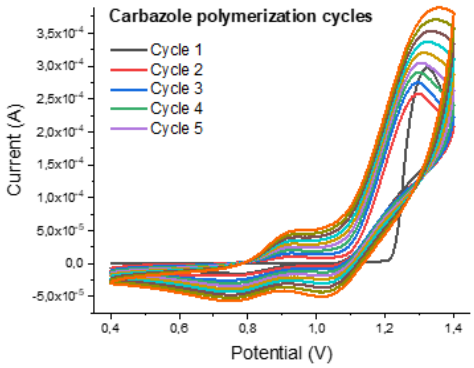

Fig. 2. Carbazole polymerization via Cyclic voltammetry.

Gas sensing experiments were performed using a mass flow controller-based system with constant H_2 flow rate in a measurement chamber at room temperature (23°C). The humidity level (RH=30%) was adjusted using bubbler humidifier. Measurement cycles consisted of 20 min flow of the carrier gas and 10 min flow of the carrier gas with the indicated and constant concentration of H_2. The resistance of the sensors was measured using Keysight DAQ970A.

Results

After modifying the IDE with polycarbazole polymers, a morphological and electrical characterization is carried out using SEM-FIB and electrochemical impedance spectroscopy (EIS). The morphological analysis reveals the uniformity of polycarbazole electrodeposition and the presence of pores in the membrane, facilitating H_2 diffusion to the transducer.

An example of the Pt IDE-PCz sensor response for repeated cycles of H_2 exposition is presented in Fig.3. The calibration curve for resistance data obtained is shown in Fig.4. The results show high efficiency of H_2 sensing with satisfying dynamics and good linearity of responses.

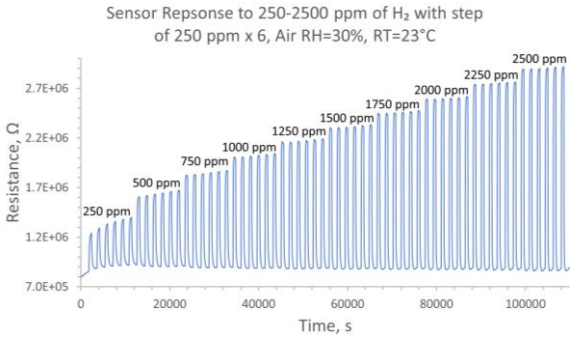

Fig. 3. Recording of sensor resistance changes for several H2 exposition cycles.

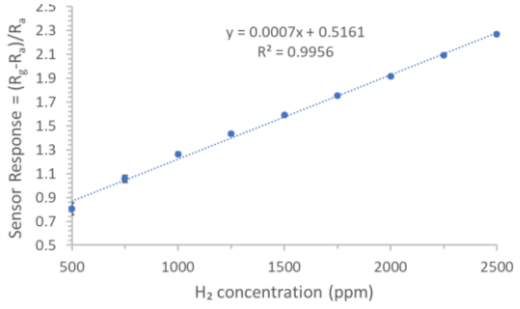

Fig. 4. Sensor calibration curve for a H_2 concentration range of 500-2500 ppm.

Reference

[1] A. Stolarcyk, T. Jorosz, M. proccek, Room Temperature Hydrogen Gas Sensing via Reversible Hydrogenation of Electrochemically Deposited Polycarbazole on Interdigitated Pt Transducers, *Sensors* 19, 1098 (2019); doi 10.3390/s19051098