

# Electrical Characterization of Ammonia Carbon-Based Sensors

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A sensor response parameters of the ammonia sensors which are prepared by using composite of graphene oxide and poly(4-styrenesulfonic acid-co-maleic acid) sodium salt derived carbon are presented. Using the self-designed interdigitated electrode on the experimental setup, we were able to determine the capacity for gas sensing as a sensor response for low ammonia concentrations (20, 50, and 100 ppm).

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## 1. Introduction

The porous carbon and its oxidized counterpart have been investigated previously as the adsorbents of ammonia [1–3]. It has been shown that a combination of chemisorption and physisorption processes governs the adsorption of ammonia on these materials. The structural and chemical surface properties of activated carbons make it possible to use them as a sensing element of the gas sensors. The most important aspects of investigation of a gas sensor are the ability of the sensor to measure the low concentration (sensitivity) of chosen gas (selectivity) and to maintain sensor measurement parameters (stability). The solutions of all these problems need a studies on novel sensing materials [4–6], the improvement of the measurement techniques by control of the sensors structure [7–11] and their fabrication techniques [12–18].

The aim of this work is to design and to build experimental setup for ammonia detection that is based on the adsorption of gas on nanoporous carbons as a adsorbent. High contact area of the electrodes with addition of conductive graphene to composite structure can improve the sensing response [19, 20].

A critical parameter of a sensor is the magnitude of its response [21]. Sensor response, expressed in % (SR(%)), is defined as a ratio of a relative change of a initial resistance ( $R_0$ ) and a resistance measured in  $\text{NH}_3$  atmosphere after  $\text{NH}_3$  exposure ( $R_{\text{NH}_3}$ ) to the initial value  $R_0$ .

$$\text{SR}(\%) = \frac{|R_0 - R_{\text{NH}_3}|}{R_0} \times 100.$$

Decrease [22, 23] of  $R_0$  value for the discussed ammonia sensors was a main goal of this work. We propose how to enhance sensor parameters by using self-designed interdigitated electrode (IDE) and by improving of the bulk conductivity of the sensing materials.

## 2. Materials

Poly(4-styrenesulfonic acid-co-maleic acid) sodium salt derived carbon was applied to produce the ammonia sensor. It was chosen based on its high ammonia sorption capacity [24]. Moreover that material can be easily used as a porous matrix in carbon composites. Ammonia sensing composites of graphene oxide and nanoporous poly(4-styrenesulfonic acid-co-maleic acid) sodium salt-derived carbons were prepared with 1 and 10 wt% of graphene oxide [25]. They are referred to as C1 and as C2, respectively. Poly(4-styrenesulfonic acid-co-maleic acid) sodium salt was used as a nanoporous carbon precursor during synthesis. Graphene oxide (GO) was obtained using an improved Hummers method [20]. The mixture was carbonized at 800 °C for 40 min under nitrogen, in horizontal furnace. The heating rate was 10 °C/min. The resulting composites were washed in water in a Soxhlet apparatus to remove an excess of water-soluble inorganic salts. The details on the initial sample preparation are described in Refs. [26–28].

## 3. Experimental

Owing to the large contact area between sample and electrodes, self-design interdigitated electrodes (IDE) (Fig. 1) were used as the gas detecting chip [29, 30]. The interdigitated electrodes were fabricated using a standard photolithography method; a single finger of the electrode has a width and gap size of 10  $\mu\text{m}$  and 6.5 mm length, respectively. The electrodes have rectangular total area of 6.5 mm  $\times$  2.5 mm. The IDE fingers were made by thermally evaporating 10 nm Ti and 50 nm Au on a layer of silicon dioxide ( $\text{SiO}_2$ ) thermally grown on a silicon wafer. Our sensing materials were deposited on the electrodes as a suspension of a grinded sample in ethanol by using micropipette and spin coating method, and then dried at 120 °C for 24 h. The schematic of procedure for preparation of the sensing chip is presented in Fig. 2. The measurements were done with four point probe attached to the chip.

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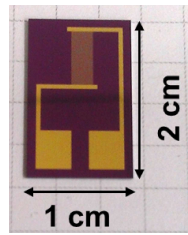


Fig. 1. Gold interdigitated electrodes on silicon substrate.

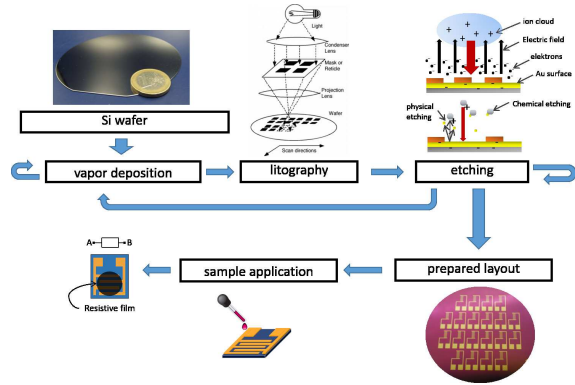


Fig. 2. Procedure for sensing chip preparations: from Si wafer to ready chemiresistor sensor.

The electrical characterizations of the sensors were carried out by using self-designed experimental setup (Fig. 3). It consisted of toxic gas tank, air compressor, air dryer chamber, two flow meters, homemade gas chamber with sensing chip, source meter (Keithley 2400), PC, wires and gas tube sets. Setup operating at room temperature and  $\text{NH}_3$  concentration between 0 and about 1000 ppm. Flow rate and ammonia concentration were controlled by two flow meters, first one for controlling flow of ammonia ( $\text{NH}_3$ ) and the second for controlling flow of dry air. The experimental setup was protected from the water vapor.

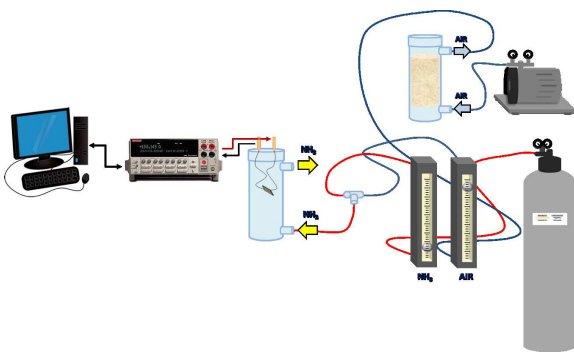


Fig. 3. Experimental setup for  $\text{NH}_3$  detection.

External voltage of 1 V was applied on every tested chip. Sensors were acting as a chemiresistors with a variable resistance of an electrical circuit. The experimental setup for the gas sensing was manually calibrated

to ensure a constant air flow during the electrical measurements. The chips were exposed to the total flow rate of 500 ml/min of ammonia mixed with air, with an ammonia concentrations fixed to 20, 50, or 100 ppm. Between exposure of ammonia the sample was purging by air.

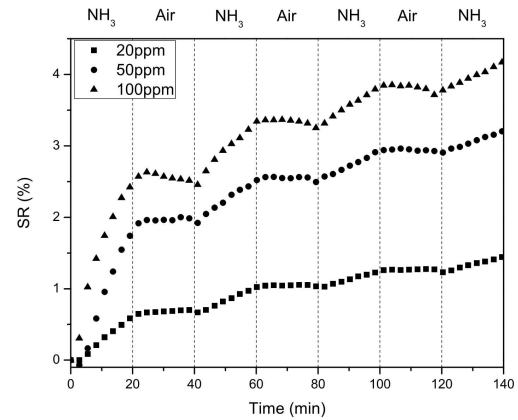


Fig. 4. Sensor response for ammonia concentration of 20, 50, and 100 ppm for C1 porous carbon.

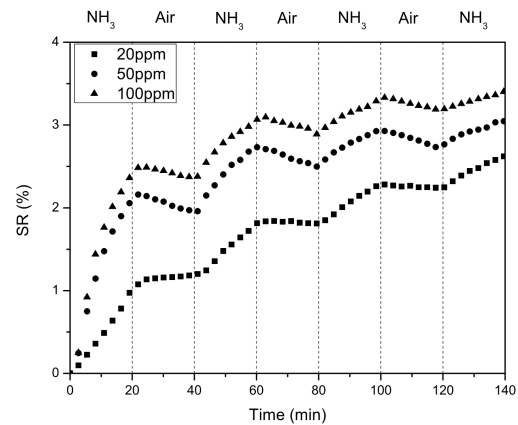


Fig. 5. Sensor response for ammonia concentration of 20, 50 and 100 ppm for C2 porous carbon.

The changes in the sensor response upon various ammonia concentrations, for the composites containing 1% and 10% graphene oxide (C1 and C2) are shown in Fig. 4 and Fig. 5, respectively. The initial resistance of the C2 chip was  $15.4 \Omega$  and for that of C1 —  $25.5 \Omega$ .

The results presented show that the C1 and C2 carbon based sensors give a strong response to  $\text{NH}_3$  at three different concentrations. For both samples the response was larger for a higher ammonia. The C2 carbon showed more pronounced signal change upon the  $\text{NH}_3$  exposure than C1 carbon, especially for a low gas concentration. The results presented show that C1 and C2 carbon based sensors give a strong response to  $\text{NH}_3$  at three different concentrations, at the first 20 min of ammonia exposure. The response was higher for higher ammonia concentration in both samples. The sensors recovery were not observed.

#### 4. Conclusions

We can conclude that the interdigitated electrodes designed in this study are suitable for measuring gas sensing on carbonaceous material chips. In our experimental setup low ammonia concentration could be detected. We also observed that the response of sensors for low ammonia concentration is larger for the samples with larger weight % of conductive graphene (GO) in the carbonized polymer matrix.

Presented here interdigitated electrodes can also work as a simple and reproducible sensor platform for detection of various types of gases in other kinds of nanomaterials, such as single walled carbon nanotubes [29], functionalized graphite or zinc oxide [21] at the room temperature.

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