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## Image processing of non-thermal glow discharge plasma during gas ionization process as a novel hydrogen detection system at parts per billion levels



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### ABSTRACT

A new hydrogen (H<sub>2</sub>) detection system is introduced based on the image processing of nonthermal glow-discharge plasma of the ionization of H<sub>2</sub> at parts per billion (ppb) levels based on gas ionization process under vacuum condition (~8 torr). The system setup consists of a charge coupled device camera as detector. For this purpose, the blue component of the photographic image related to ionization-generated plasma is analyzed during applying a 853-V direct current potential to a two-electrode system including Al disk as cathode (diameter:  $2.4 \pm 0.1$  mm) and multi-walled carbon nanotubes-modified disk (diameter:  $6.5 \pm 0.1$  mm) as cathode with 700  $\pm 10 \,\mu$ m inter-electrode distance. Figures of merits of the fabricated sensor reveal two linear dynamic ranges between 5–36 and 36–180 ppb. Relative standard deviation of at most five replicate analyses during introduction of 90 ppb of H<sub>2</sub> standard solution (in air as solvent) is found as 2.29%. Also, the detection limit was evaluated to ~2.25 ppb. Except acetylene, no significant interfering effect is observed when introducing at least 1000 folds excess (mass/mass) of different foreign species. Reliability of the sensor is also evaluated via determination of H<sub>2</sub> in different industrial gas samples.

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### 1. Introduction

Nowadays, hydrogen (H<sub>2</sub>) is applicable in various fields such as in the design of fuel cells [1], in welding and galvanic plating [2,3], in coal mines during methane or coal-dust explosions [4], in the hydrogen engine cars [5], etc. [6]. The reason of the great applications of H<sub>2</sub> is that, compared to explosive gases such as methane, propane or gasoline, H<sub>2</sub> has a number of unusual properties such as very low density (0.0899 kgm<sup>-3</sup>), small boiling point (20.39 K), large diffusion coefficient (0.61 cm<sup>2</sup> s<sup>-1</sup> in air), high buoyancy [7], low minimum ignition energy (0.017 mJ) [8], high heat of combustion (142 kJ g<sup>-1</sup>) [8], etc. [9]. Consequently, introduction of reliable, robust and accurate H<sub>2</sub> sensors with acceptable detection limit is seriously needed.

Most commercial  $H_2$  sensors are based on ohmicresistance [10], solid-based potentiometry [11], field effect transistor [12], optical [13] and ionization-based sensors

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[14,15]. Among these probes, ionization sensors due to their significant advantages such as high selectivity and capability for simultaneous monitoring ambient contaminating gases [16], have been considered as suitable detection system. In the ionization sensors, usually ionization current during applying a fixed breakdown potential is considered as detection system [14].

The limit of the ionization current related to the previously reported gas ionization sensors is usually ranged in the scale of nA or µA per parts per million (ppm) [17,18]. Therefore, shortcomings such as low sensitivity, high electrical power consumption and operation at high temperatures have seriously limited the development of these gas ionization sensors [19]. It seems that focusing of new detection systems such as image processing can solve these limitations. For this purpose, hereby in this work a new glow discharge plasma-based sensor is fabricated for selective detection of H<sub>2</sub> at parts per billion (ppb) levels via image analysis of the glow dischargegenerated ionization process using a charge coupled device (CCD) camera under vacuum condition ( $\sim$ 8 torr).

#### 2. Experimental

#### 2.1. Chemicals

All the reagents including gas species as well as carbon nanostructures were from their analytical grades. In this study, different concentrations of fresh H<sub>2</sub> in air as solvent at ppb levels were prepared via operating a calibrated mass flow controller (MFC, model: 1661, SN: All43024300, USA) [20]. The setup of the electrical arc/discharge system used to synthesize different kinds of carbon nanostructures such as singlewalled carbon nanotubes (SWCNTs), multi-walled CNTs (MWCNTs), carbon nanofibers (CNFs) as well as various CNTdoped metal nanoparticles such as Al, Cu, Ni, Fe, etc. is shown in Fig. 1. Scanning electron microscopic (SEM, XL-30 FEG, Philips, 20 KV) and transmission electron microscopic (TEM, CM-10, Philips, 100 KV) images of the arc-synthesized CNTs have also shown in Fig. 1. As schematically observed in Fig. 1, to have hydrogen sensing sensor based on the ionization process on the surface of carbon nanostructures, a

length of  $\sim$  2.0 cm of the CNT-supported graphite disk is cut and used as ionization gas support.

Gases including nitrogen (99.98%), argon (99.98%), helium (99.99%), acetylene (99%) and carbon dioxide (99.0%) were purchased from "Parsbaloon, Iran". Hydrogen (99.9996%) and carbon monoxide (99.9997) were from "Linde, Germany". Highly purified (99%) SWCNTs with 3-8 nm internal diameter (i.d), MWCNTs (i.d.: 40-60 nm) and CNFs (i.d.: 250-300 nm) were synthesized by electrical arc/discharge method. For this purpose, briefly, a direct current (DC) potential (20-30 V) was applied to a two-graphite electrode system with inter-electrode distance of <1.0 mm at Ar/He atmosphere under vacuum condition ( $\sim 0.01$  torr). Also, graphene nanopowders were synthesized by exfoliation method according to the procedure reported in Ref. [21], followed by casting onto the graphite electrode by dropcoating method [22].

#### 2.2. Apparatus of the hydrogen sensor

The fabricated hydrogen sensing setup includes a twoelectrode system (Al rod as anode and graphite electrode as cathode) that has been situated horizontally next to each other with inter-electrode distance of 700  $\pm$  10  $\mu m$  via controlling using a stepper motor through a mechanical interface (digital caliber, mode: Z22855). The schematic of the instrument used for H<sub>2</sub> sensing is shown in Fig. 2. To control the pressure inside the glass tubing chamber (volume:  $\sim$ 40 mL) containing the electrode system to the optimum value (8 torr), briefly, a vacuum pump (model: VC 9200) and vacuum meter (model: Edward/E2M2) are added to the setup system. The electrode-applied DC high potential was also controlled using switching power supply (model: Cold Amp - SPS80HV). The applied potential is scanned via controlling the input voltage using a variac (model: TGC2-3KVA) by a stepper motor.

A mass flow controller (MFC, UFC-1661, SN: All43024300, USA) is also utilized to prepare the H<sub>2</sub> standard solutions, introduced through the H<sub>2</sub> cylinder. Different concentrations of H<sub>2</sub> at both ppm and ppb levels were also standardized using MQ-8 H<sub>2</sub> reference sensor and gas chromatography (GC) with thermal conductive (TC) detector, respectively.

The detection systems such as ionization currents or image processing are detected using a digital multi meter (model: AT9995) or a charge coupled device (CCD), positioned

Rotameter





Fig. 2 - Schematic of the system setup of the fabricated hydrogen sensor.



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