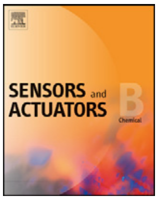
Sensors and Actuators B xxx (2012) xxx–xxx

Contents lists available at SciVerse ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb



Portable cantilever-based airborne nanoparticle detector

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ARTICLE INFO

Article history:
Received 14 July 2012
Received in revised form
19 September 2012
Accepted 22 September 2012
Available online xxx

Keywords:
Cantilever resonator
Airborne engineered nanoparticle
Electrostatic sampler
Mass sensitivity

ABSTRACT

A portable cantilever-based airborne nanoparticle detector (CANTOR) was designed and manufactured for detecting engineered nanoparticles (ENPs) in workplace air by monitoring the resonant frequency shift induced by the mass of the particles trapped on the cantilever resonator. The CANTOR consists of two main modules, i.e., a silicon resonant cantilever sensor and a miniaturized electrostatic ENP sampler. Tested in 15-min aerosol sampling with ∼100 nm carbon-based ENPs having a concentration of ∼6000 NPs/cm^3, the sensor exhibited a mass sensitivity of 36.51 Hz/ng when the second resonant mode was used. Two simple cleaning methods, i.e., dry and wet cleanings, to remove the attached ENPs were successfully demonstrated in order to extend the operating life of the sensor.

1. Introduction

Nanotechnology is a rapidly growing field with many potential benefits to humans. It finds diverse use in many applications, such as electronics, displays, batteries, automobiles, aerospace, construction, and healthcare. With these applications, unprecedented paths of exposure to engineered nanoparticles (ENPs) in humans are likely. Moreover, a high awareness should be given to the workers who produce and handle ENPs in large quantities in the manufacture of commercial products. Exposure through inhalation of ENPs becomes a primary apprehension for worker health and safety because of the sensitivity of the respiratory system. The airborne ENPs, which in general terms are defined as engineered structures with diameters of <100 nm mixed in air, are found to be more hazardous because nano-sized particles can bring substantially greater toxic effects than larger particles of the same materials [1,2]. The inhaled airborne ENPs can be deposited efficiently in all regions of the respiratory tract. In experimental conditions, such nuisance types of ENPs, e.g., titanium dioxide (TiO_2) and carbon black, were found to be harmful to the lung [3,4].

Although the effect of ENPs in the respiratory tract is mainly related to the surface, most epidemiological studies suggest to the inhaled mass. Guidelines as defined by the World Health Organization for PM_{10} and $PM_{2.5}$ in ambient air also use mass related units [5]. Thus, direct mass sensing of airborne ENPs is very useful for the assessment of personal and location dependent monitoring at the workplace and the indoor environment. To assist on this evaluation, some micro/nano-electro-mechanical system (MEMS/NEMS) based resonant mass sensors had been used to determine mass concentration and size distribution of aerosol particles [6–9]. From the previous work, a self-sensing silicon cantilever was investigated in an air pollution sampling of ∼20 nm carbon ENPs which resulted in a mass sensitivity of 8.33 Hz/ng [7]. Nevertheless, the used commercial nanometer aerosol sampler (NAS, TSI 3089) was too bulky and heavy to be implemented in a portable sensing system [10]. Therefore, a low-weight electrostatic sampler was homebuilt to overcome this issue [11]. In this work, a silicon resonant cantilever sensor was integrated with a portable electrostatic nanoparticle sampler and operated in the second bending mode to improve the quality factor, mass sensitivity and sampling efficiency in air. To select the settlement of ENPs, an electrostatic-directed ENP deposition method was implemented to the novel designed cantilevers which were first tested in an aerosol chamber. Moreover, simple cleaning methods were successfully demonstrated to detach the ENPs from the cantilever, which definitely could extend the operating life of the sensor.

2. Portable system description

A portable cantilever-based airborne nanoparticle detector (CANTOR) was developed to fulfill the demand for personal airborne ENP monitoring. In general, the portable system comprises

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http://dx.doi.org/10.1016/j.snb.2012.09.074

Please cite this article in press as: H.S. Wasisto, et al., Portable cantilever-based airborne nanoparticle detector, Sens. Actuators B: Chem. (2012), http://dx.doi.org/10.1016/j.snb.2012.09.074

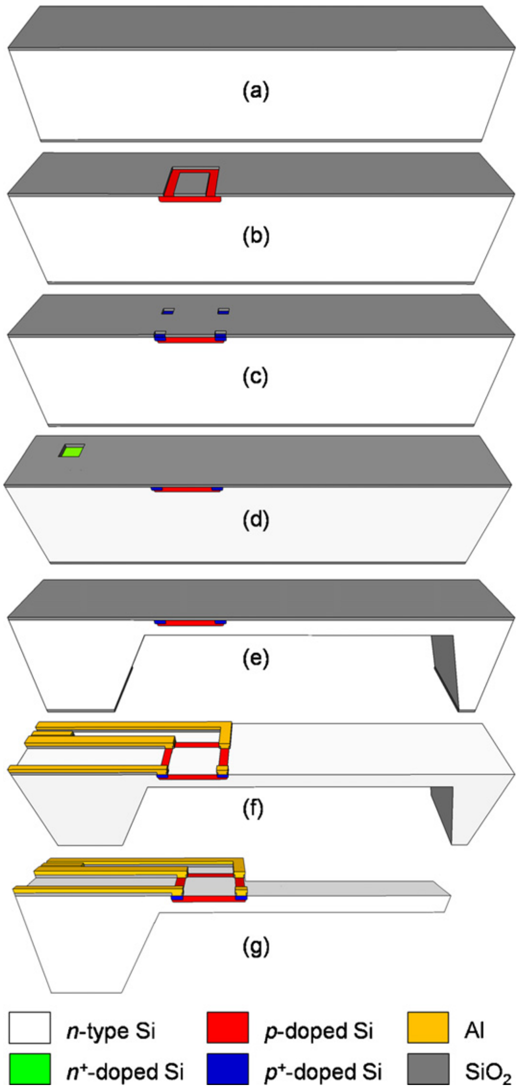

Fig. 1. Fabrication process for silicon resonant cantilever sensors. Bulk silicon wafers are preferred over SOI wafers to be employed in sensor fabrication considering their lower price, high mechanical quality factor, high stability, and high degree of freedom for the geometrical resonant cantilever design. More details of the process steps are given in the text.

two main modules, i.e., a cantilever resonator and a miniaturized-electrostatic sampler.

2.1. Silicon resonant cantilever sensor

The first module of the portable system is a silicon-based cantilever resonator. In order to read the signal output of the sensor, Si cantilever has a full Wheatstone bridge on its clamped end as a piezoresistive element which works based on the strain-to-resistivity change [8]. The cantilevers are manufactured by utilizing a silicon bulk micromachining process based on photolithography, diffusion, and inductively coupled plasma (ICP) cryogenic deep reactive etching (cryo-DRIE) which offers many benefits including precise control of dimensions, integration and miniaturization of devices, and fabrication of an array of devices [12,13].

Fig. 1 shows the process flow of the silicon resonant cantilever fabrication. The process details of the cantilever resonator fabrication can be described as follows:

(a) Firstly, an *n*-type bulk silicon wafer (3–5 Ω cm) of 300 μm thick was put into a furnace to produce a thin layer of silicon dioxide (SiO_2) on the silicon surface. The thermal oxidation process was performed at a temperature of 1100 °C within 1 h. Thus, a SiO_2 layer of ~400 nm was grown successfully coating the whole area of silicon surface.
(b) The oxidized wafer was then covered by photoresist. Using UV lithography, the photoresist mask was patterned resulting in a square geometry. At the next step, the selected SiO_2 area for creating *p*-type piezoresistors was etched using hydrofluoric acid (HF) solution. Afterwards, boron diffusion or implantation was carried out to form a full piezoresistive Wheatstone bridge on the Si wafer.
(c) Subsequently, additional boron diffusion or implantation was used to form p^+-feed lines to the Wheatstone bridge and improve contact formation. Therefore, the contact resistance could be reduced.
(d) Phosphorus diffusion was set to an *n*-type well (n^+) close to one of the piezoresistor contacts in order to have ohmic contact to the silicon substrate. This contact was intended to be used for ENP collection purpose.
(e) Next, the wafer bottom-side oxide layer was patterned and etched using potassium hydroxide solution (KOH) to a residual thickness of 25–40 μm. This KOH-etching had a tolerance of ±0.5 μm which might slightly affect the performance of the cantilever sensors.
(f) After etching the field oxide (SiO_2) for creating contact holes, metallization was then carried out by 300 nm top-side aluminum coating. Moreover, in order to have an easier bonding to the printed circuit board (PCB), the contact pads were designed as 0.75 mm × 1 mm area.
(g) At last, the cantilevers were lithographically patterned and released by cryogenic ICP deep dry etching of silicon from the front side using an SI500C cryogenic dry etcher (Sentech Instruments) with photoresist serving as the etching mask.

In order to actuate the cantilever into resonance an external piezostack adhered to the cantilever supporting frame was used. A sine voltage signal generated from function/arbitrary waveform generator (HP 33120A) with U_{pp} of 10 V was fed into the piezostack. The Wheatstone bridge circuit was supplied by a voltage U_0 of 1 V from a DC power supply (HP E3631A). The signal output of the bridge could be directly read through a digital multimeter (HP 34401A) and a dynamic signal analyzer (HP 3562A), respectively [8]. The measured data were stored to a PC for further analysis.

It has been known that the quality factor (*Q*-factor) of a resonator increases with the mode number which can help to achieve a higher sensitivity of the sensor [14–16]. The *Q*-factor of a damped system can be defined as

$$Q = 2\pi \frac{\text{stored vibrational energy}}{\text{energy lost per cycle of vibration}} = \frac{f_n}{\Delta f_{\text{bandwidth}}} \tag{1}$$

where f_n is the resonant frequency of the mode and $\Delta f_{\text{bandwidth}}$ is the full width at half maximum (FWHM) of the resonance peak in the frequency domain. Dissipation, which is the inverse of the *Q*-factor, may occur during vibration caused by several factors that are either intrinsic factors Q_{int} (e.g., internal material damping Q_{mat} and damping due to the sensor substrate through the cantilever support Q_{sup}) or extrinsic factors Q_{ext} (e.g., viscous damping Q_{vic}). The intrinsic dissipation factors are highly considered when resonators are operated in high-vacuum conditions. However, at atmospheric pressure, the viscous damping or momentum exchange with the surrounding medium is usually dominant among the other damping sources.

Furthermore, using the drag force method (i.e., the force exerted on the vibrating beam by the surrounding medium (e.g., air)), Blom

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