



Handheld personal airborne nanoparticle detector based on microelectromechanical silicon resonant cantilever



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ABSTRACT

The development and real-time performance test of a fully integrated low-cost handheld cantilever-based airborne nanoparticle (NP) detector (CANTOR-2) are described in this paper. The device is the enhancement of the previously developed cylindrical electrophoretic NP sampler (CANTOR-1), which is used for direct-reading of exposure to airborne carbon engineered nanoparticles (ENPs) in indoor workplaces. All components of the proposed detector can be divided into two main units depending on their packaging placements (i.e., the NP sampler head and the electronics mounted in a handy-format housing). For the NP sampler, a miniaturized electrophoretic aerosol sampler created in a cubical shape and an electrothermal piezoresistive resonant silicon cantilever mass sensor are employed for collecting the ENPs from the air stream to the cantilever surfaces and measuring their mass concentration, respectively. To realize a real-time measurement, a frequency tracking system based on phase-locked loop (PLL) is built and integrated to the device. From the device calibration, a good correlation of the CANTOR-2 data is found with the fast mobility particle sizer (FMPS, TSI 3091) reference at a precision of 8–14%. By having a total device volume of 540 cm³, weight of 375 g, and power consumption of 1.25 W in the current version, this developed CANTOR-2 provides a very good portability for being used as a personal airborne NP monitoring device, which can be easily held or worn by workers during their activities.

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1. Introduction

Nanotechnology has been used across a variety of industries (e.g., electronics, medicine, cosmetics, pharmaceuticals, food packaging, household appliances, and national defense). The interests in this technology are mainly from the novel characteristics and properties of the nanomaterials or nanoparticles (NPs), as scale effects play a role in altering the material properties [1]. Nevertheless, airborne NPs, which are ubiquitous in many workplaces, may produce risks and potential health effects for workmen. These NPs can be formed and released to air through both incidental (i.e., unintentional NPs) and planned manufacturing processes (i.e., engineered NPs (ENPs)). In case of unexpected excessive concentrations of hazardous NPs, the potentially exposed persons should be equipped with a personal airborne NP detector to prevent an overexposure, so that they can leave the rooms

immediately before inhaling too many NPs. In this case, the NP detecting device has to be low-cost, direct-reading/real-time, sensitive and able to detect various types of aerosols, easy to operate, and small/lightweight enough to be either handheld or worn in the work clothing. Moreover, the selected metric should be traceable to the standard international (SI) units. Since the World Health Organization (WHO) suggested air quality standards as mass concentrations in µg/m³ [2,3], direct gravimetric measurements of aerosol mass concentration are preferable for scientific communities. However, these measurements are difficult to realize because of very low NP masses.

From market observation, it is found that most of the commercially available expert NP monitoring systems are expensive and not directly readable. In terms of limit of detection (LOD), the tapered element oscillating microbalance (TEOM) provides sufficient values. However, its instruments for fine particulate matter of particle diameters of less than 2.5 µm (PM_{2.5}) are weighty (34 kg) and costly (€19,500) [4]. As an alternative method, diffusion size classifiers (DSCs) have been frequently used by some companies in Europe to develop their portable devices (i.e., DiSCmini (Matter Aerosol AG, Wohlen, Switzerland [5]),

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NanoTracer (Philips Aerasense, Eindhoven, The Netherlands [6]), NanoCheck (Grimm Aerosol Technik GmbH & Co.KG, Ainring, Germany [7]), and Partector (Naneos Particle Solutions GmbH, Windisch, Switzerland [8]). All those devices basically combine a unipolar diffusion charger and electrometers to charge the particles and measure their transferred currents, which are then converted into their corresponding number concentrations, respectively. Regardless of their fine time resolutions (1 s) and LODs (10^3 – 10^6 pt/cm³), all those mentioned portable particle monitoring devices still cost between €5000 and €10,000 because of the high expenses of their components [9]. As a consequence, the devices are rarely found in the real workplace. Correspondingly, a paradigm shift has been addressed in air pollution monitoring toward lower-cost, easy-to-use, portable, and direct-reading sensors [22]. A step in this direction is the Dylus Air Quality Monitor, which costs less than \$300 and is found to track well with standard commercial monitors [23]. However, since it is based on light scattering, it is limited to the particle diameter range above of 0.5 μm . Real NPs can be detected using mass detectors based on micro/nanoelectromechanical systems (M/NEMS) which in addition offer the necessary potential of miniaturization and cost-effective batch fabrication. Indeed, most of the currently introduced M/NEMS-based NP mass detectors still need to be operated at high flow velocity using a pump and characterized with external bulky, heavy, and vacuum tools [10–16]. In contrast, our second generation of a fully integrated cantilever-based airborne NP detector (CANTOR-2) developed in this work has small size and low weight. Furthermore, it allows direct-reading operation at appropriate flow-rates under atmospheric pressure working conditions and is comprised of low-cost components.

2. Fully integrated cantilever-based airborne nanoparticle detector

The CANTOR-2 is an enhanced version of our previously developed cantilever-based airborne nanoparticle detector (CANTOR-1), which was still in a partially integrated system using a cylindrical electrophoretic NP sampler head, successfully detecting 20 nm carbon ENPs in off-line [14] and real-time measurements [15]. In this novel device, the NP sampler head has been improved in terms of its design and functionality. Its outer shape is created in a cubical shape to overcome the instability of the NP sampler during handling and suited to the final plug-in CANTOR-2 device housing (Fig. 1(a)). Nevertheless, this NP sampler head still consists of three main parts made of aluminum (i.e., inlet, middle, and outlet parts), which is similar to that of CANTOR-1. The inlet part has an opening hole with a diameter of 2 mm allowing the air intake. In the middle

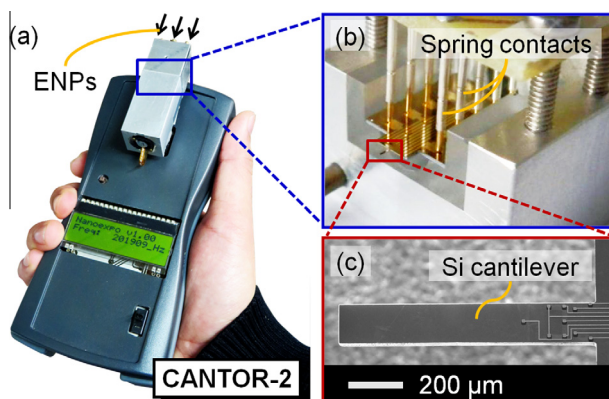


Fig. 1. (a) The developed CANTOR-2 showing (b) the in-plane silicon cantilever mounted with spring-loaded contacts and (c) its SEM image.

part of the sampler head, the mounting of the silicon cantilever resonator was carried out with spring-loaded contacts (Fig. 1(b)) for fast and easy replacement of the MEMS device in case of overload. Moreover, to assure a good electrical contact between the spherical shafts ($d_{\text{shaft}} = 500 \mu\text{m}$) and the cantilever contact pads, the I–V characterizations of the resistors were firstly done before and after pressing. For the outlet part, the identical miniature fan (MF10A03A, SEPA Europe GmbH) as being used in CANTOR-1 is integrated in the system. However, due to the different head design, the airflow rate is reduced in CANTOR-2 to be around 0.3 L/min instead of 0.68 L/min. The airflow rate was measured using KLIMATHERM Anemometer TA3-D. The obtained results from the measurement were then used as references for further simulating the samplers using particle tracing module of COMSOL Multiphysics 4.3b. Thus, the changes of the airflow rate can be determined in respect to the modification of the sampling head shapes.

It should be noted that the sensor employed in the CANTOR-2 is an electrothermal silicon cantilever sensor with integrated heating and strain-sensing resistors connected in a full U-shaped Wheatstone bridge supplied by 1.3 V for in-plane resonant-mode detection (Fig. 1(c)). The heating resistor is positioned off-axis at the fixed end of the cantilever. Superposition of direct current (DC, 2 V) and alternating current (AC, 1 V, amplitude) actuation voltages leads to local heating and deformation of the cantilever exciting in its fundamental lateral bending mode. Therefore, an external piezoelectric stack actuator used in CANTOR-1 is no longer required. This cantilever module has a geometry of $1000 \times 170 \times 19 \mu\text{m}^3$, a weight of $m_0 = 7.53 \mu\text{g}$, a resonant frequency of around $f_0 = 201.8$ – 202.1 kHz, and a quality factor (Q) of 4700 ± 100 which corresponds well to the value of 4200 reported for a cantilever of $400 \times 90 \times 12 \mu\text{m}^3$ [24]. Operation in a lateral mode has the advantage that shear loss to the surrounding fluid is much lower than the viscous damping by the effective mass of the fluid that has to be accelerated in case of vertical cantilever deflection. To fit into the NP sampler head, the cantilever is designed to have large contact pads and membrane supporting frame.

The cantilevers are fabricated by utilizing silicon bulk micromachining processes, which provide many advantages including precise control of dimensions, integration and miniaturization of devices, and low-cost batch fabrication of device arrays. Fig. 2 shows the detailed process steps to fabricate the electrothermal cantilever sensors, which can be described as follows:

- (a) A piece of sample with a dimension of $30 \times 30 \text{ mm}^2$ was cut from an n -type (100) four-inch silicon bulk wafer doped by phosphorus. This sample has a thickness of $\sim 300 \mu\text{m}$ and a resistivity of 1–10 Ωcm . To clean the substrate from the organic residues, a piranha cleaning was firstly carried out by placing the sample inside a quartz glass with an 1:1 mixture solution of hydrogen peroxide (H_2O_2 , 30%) and sulfuric acid (H_2SO_4 , 96%) inside. To increase the cleaning efficiency of this solution, the already sample-filled glass was then boiled on a heating plate with a temperature of 90 $^\circ\text{C}$ within 5 min. This cleaning procedure was frequently used in these sequential sensor processes (e.g., before and after thermal oxidation and prior to dopant diffusions).
- (b) Next, a silicon dioxide layer was grown in a thermal oxidation process. The oxidation was performed in a combined process of dry and wet oxidations. The temperature was set constant to be 1100 $^\circ\text{C}$ during the growth. A constant dry oxygen flow rate of 2.5 L/min was used in the dry oxidation steps with a total duration of 45 min. Whereas, additional wet oxygen with a flow rate of 4.5 L/min was necessarily involved in the wet oxidation within 60 min.

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