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Effective platform for the generation of aerosol droplets and application in evaluating the effectiveness of a MEMS-based nanoparticle trapping device

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ABSTRACT

The purpose of this study is two-fold: firstly, the development of a cheap, easy-to-construct and effective nanoparticle generator for testing nanoparticle sensors; secondly, the use of such a generator to test the effectiveness of a sensor device in trapping aerosolised nanoparticles. In this study, we have constructed an effective aerosol generator platform, based on aerosol-assisted chemical vapour deposition technology. Under well-controlled experimental conditions, this platform is capable of depositing aerosolised sodium chloride particles homogeneously on a substrate very effectively. Deposited aerosol droplets were subsequently dried and shown to form nanosized cubic crystals that are free from impurities. This platform was employed to test the effectiveness of a MEMS comb device in the electrostatic trapping of nanoparticles. Upon applying a DC bias (0.5 V) to the MEMS device, results showed an increase in nanoparticle deposition on the surface of the device, due to electrostatic precipitation. The presence of an electric field was shown to affect crystal formation upon drying of the aerosol droplets on the substrate; this caused a blotchy appearance on the SEM image, which was not observed in the absence of electric field.

1. Introduction

There is an urgent need to develop smaller nanoparticle sensor devices, for improved portability, in order to monitor airborne nanoparticles (Maynard & Aitken, 2007). MEMS-based devices have significant potential to be incorporated into stand-alone sensors that can be worn easily as a badge or placed throughout a building (Tantra & Cumpson, 2007). The small size, relatively low cost (using standard semiconductor fabrication techniques) and low power consumption of MEMS devices makes them ideal for such a purpose (Dohn, Svendsen, Boisen, & Hansen, 2007). A very promising platform design is based on a comb drive capacitor design that can be electrostatically actuated, with a resulting natural frequency. If the device is to be used as a sensor, then the sensing principle will be based on measuring the resonance frequency shift of the micromechanical structure due to added mass bound on the surface of the resonating structure (Goeders, Colton, & Bottomley, 2008). An additional, attractive feature of such device is the possibility of electrostatically precipitating the particles on the surface of the sensor during its actuation; the magnitude of this attraction will be dominated by several factors, to include the magnitude of the applied voltage.

To test the effectiveness of such devices a platform for generating nanoparticles effectively is required. Although ranges of techniques have been developed for the generation of fine aerosolised particles (Kang & Park, 1996), there are very few that are cheap, easy-to-construct, portable and that generate safe particles during testing. The ideal particle generator would have the following characteristics:

(a) ability to generate nanosize particles;
(b) ability to control size/shape and concentration (number of particles per unit area) of the particles deposited;
(c) ability to deposit particles uniformly on the surface;
(d) ability to generate particles free of impurities that could significantly affect particle characteristics;
(e) ability to deposit biologically safe particle materials (e.g. water soluble biologically compatible salts).

To fulfil such requirements, we present a nanoparticle generator based on aerosol-assisted chemical vapour deposition (AACVD) technology (Parkin, Price, Hibbert, & Molloy, 2001). The particle generator we present is a modification of the set-up described in a paper by previous workers (Shaw, Parkin, Pratta, & Williams, 2005). The purpose of a "mini-fogger" is to thus provide cheaper

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and smaller alternative to what have been previously used in the past by Parkin and co-workers. The design will not only substantially reduce cost but will offer portability. In addition, the goal here is to use this platform not to produce a thin film but to generate non-toxic nanoparticles in the form of sodium chloride crystals. Sodium chloride was chosen as it offers not only low toxicity but also crystals with simple cubic structures. In this study, we will test the effectiveness of this apparatus in producing nanoparticles, against the set of criteria detailed above.

The platform will also be used to test the effectiveness of a MEMS comb drive device as an electrostatic precipitator in the presence of a DC bias. Our objective is to evaluate the effect of trapping nanoparticles electrostatically on to the MEMS device. It is envisaged that more particles will be trapped in the presence of an applied voltage than would otherwise impact and adhere to the surface of the sensor. We have assessed the performance of the MEMS device in nanoparticle deposition by analysis using a scanning electron microscope (SEM). Analysis of the purity of the deposited nanoparticles was made via elemental analysis made through using energy-dispersive X-ray (EDX) spectrometry.

2. Methods and materials

2.1. Aerosol generator: set-up and testing

The design of the aerosol vapour deposition set-up is shown in Fig. 1. It consists of several elements. The primary element was a mist generator using a mini-fogger mist sonicator (Maplin Electronics). The mist was created from sodium chloride solution (25 mL, Sigma–Aldrich saline 1 M stock, diluted to appropriate concentrations using deionised (DI) water) contained in the two-neck round bottom flask (50 mL capacity, Sigma–Aldrich). This flask was immersed in a beaker (100 mL capacity) containing water (75 mL). The bottom of the round bottom flask was placed over the centre of the mist generator, which was observed to be the most effective position for the generation of fine aerosols. The fine mist produced was then carried using nitrogen gas into an airtight polypropylene sample chamber, which held the sample substrate. The flow-rate of nitrogen gas was controlled by using the needle valve on a gas flow meter (Roxspur, with measuring range up to 1 L/min). Experiments were performed inside a fume-cupboard.

To test the effectiveness of the set-up in the delivery of aerosolised nanoparticles on to a substrate, sample substrates used were pieces of silicon (1.5 cm × 1.5 cm, thickness of 0.38 mm), cut from a silicon wafer (Goodfellow). Prior to use the silicon pieces were washed using 2% Hellmanex (Hellma) in an ultrasonic bath for 5 min and then rinsed using DI water. The steps required to deposit the particles on the silicon substrate involved taking an individual piece of silicon and placing it inside the chamber. After closing the chamber the nitrogen flow was turned-on to the desired rate and then the sonicator switched on (up to the desired sonication exposure time). After turning off the sonicator, the gas was allowed to flow for another 1 min and then the chamber lid was taken off. The substrate was left undisturbed for another 2 min before it was taken out for further analysis; this was enough to ensure that the aerosol droplets have sufficiently dried-out.

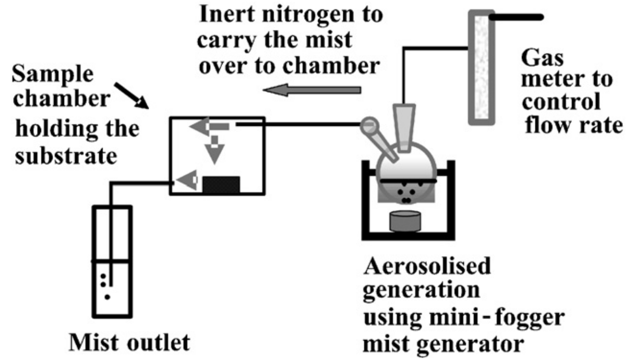

Fig. 1. Schematic set-up of equipment used to generate aerosolised nanoparticles.

The effectiveness of the particle generator was tested under various conditions and changes in the concentration of sodium chloride, the rate of nitrogen flow and exposure time of the substrate were investigated. In order to determine the optimal operating conditions for deposition, it is important to systematically investigate the influence of the various parameters in turn. To conduct this successfully, one parameter was varied whilst keeping the other factors constant; from the series of results, an operating condition that resulted in nearly homogeneous nanosized particles was identified. This approach, of varying one parameter whilst keeping all others constant, was repeated with the other parameters, until the optimal conditions for deposition were found.

Aerosol generation and subsequent deposition were all carried out under normal laboratory conditions, at a temperature of ~20 °C.

2.2. MEMS device

The MEMS device employed in this study was made from silicon and is based on "comb drive" type device. The uses of an electrostatic comb drive design in MEMS devices are quite common and have been previously described in the literature (Banks, 2006). The device used in this study consisted of a moveable platform common to four-comb drive actuator units (each comb drive consists of a pair of interdigitated fingers); an SEM image of the device can be found in Section 3 below (Fig. 5). The MEMS device was fabricated using the polyMUMPs process (from MEMSCAP, North Carolina), a polysilicon surface micromachining process.

The device was loaded into the aerosol sample chamber cell (Fig. 1) and the appropriate electrical connections were made to apply a DC bias to the MEMS device. This was achieved by attaching two separate bond pads of the chip; wire bonding connects the gold pads to both the positive/negative components of the applied voltage (from an external power supply), so that it can actuate the pairs of fingers. One set of electrodes was connected to the positive terminal of a DC power supply, whilst the other set was earthed.

In the study, two MEMS devices (of identical design) were placed side by side inside the cell chamber and only one of the two devices was connected to a DC power supply. Hence, the effect of nanoparticle deposition upon applied voltage can be observed. Deposition of the aerosolised particles was carried out using the following experimental conditions: 600 cm^3/min flow-rate, exposure time of 5 s and 0.1 M of saline solution (25 mL).

2.3. Characterisation techniques employed

Deposited particles on the substrates (silicon wafer or MEMS devices) were characterised using a Carl Zeiss Supra 40 scanning electron microscope with built-in energy-dispersive X-ray spectroscopy (EDX) analysis capability. An accelerating voltage of 5–20 kV was used. Images were recorded with an in-lens secondary electron detector (potentially with spatial resolution of a few nanometres). The deposited particles were evaluated in terms of: particle size, shape, concentration and homogeneity across the substrate, upon varying the different experimental conditions in the deposition protocol. Elemental analysis using the EDX technique was used to indicate the purity of the deposited particles on the substrate.