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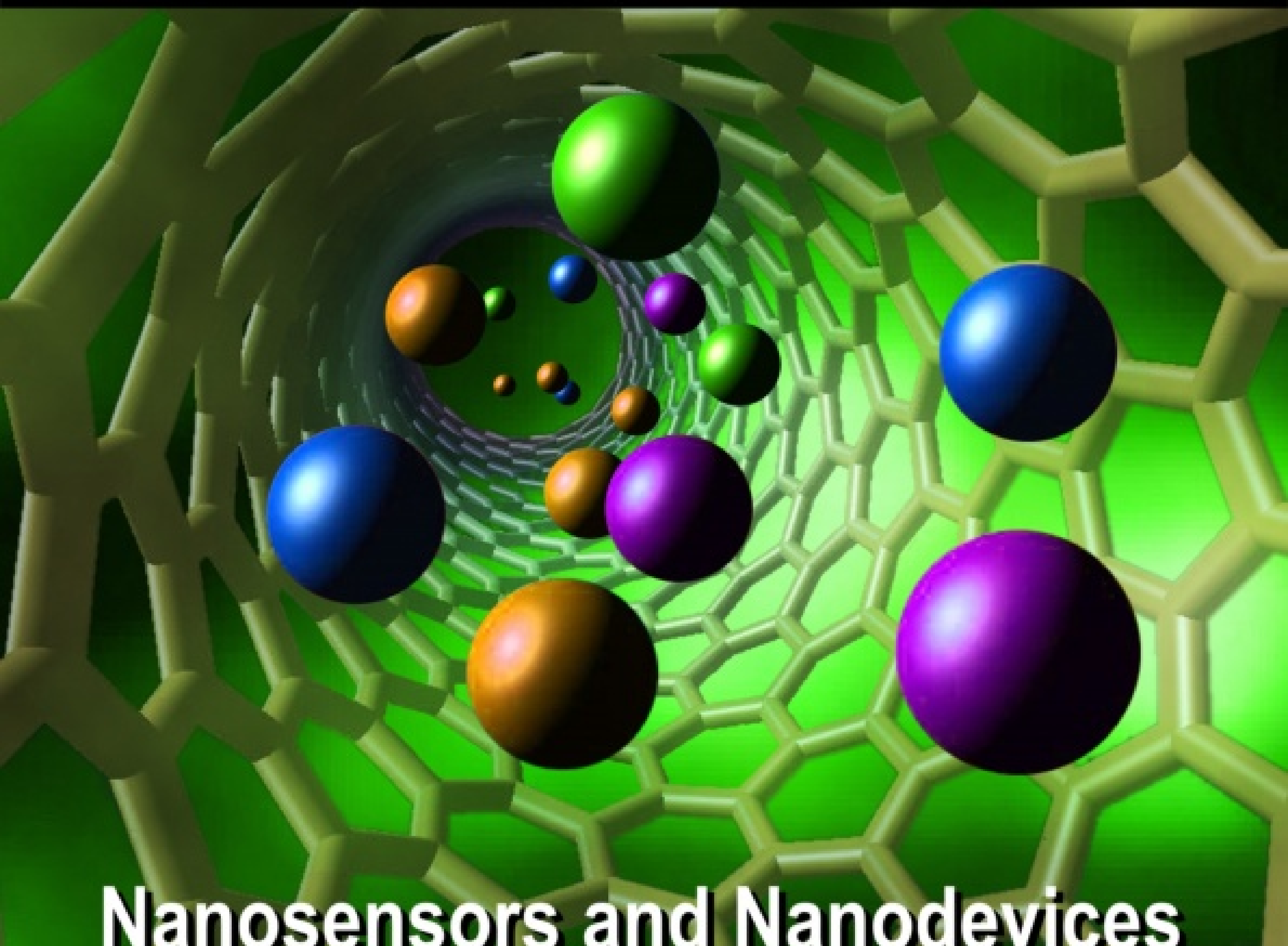

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Focused Ion Beam Nanopatterning for Carbon Nanotube Ropes based Sensor

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Abstract: Focused Ion Beam (FIB) technology has been used to realize electrode patterns for contacting Single Walled Carbon Nanotubes (SWCNTs) ropes for chemical gas sensor applications. Two types of transducers, based on a single rope and on bundles, have been realized starting from silicon/Si_3N_4 substrate. Electrical behaviour, at room temperature, in toxic gas environments, has been investigated and compared to evaluate contribution of a single rope based sensor respect to bundles one. For all the devices, upon exposure to NO_2 and NH_3, the conductance has been found to increase or decrease respectively. Conductance signal is stronger for sensor based on bundles, but it also evident that response time in NO_2 is faster for device based on a single rope. FIB technology offers, then, the possibility to contact easily a single sensitive nanowire, as carbon nanotube rope. *Copyright © 2007 IFSA.*

Keywords: Focused ion beam, Nanopatterning, Chemical gas sensor, SWCNTs Rope

1. Introduction

Recently, nanoparticles and nanowires are receiving an increasing attention in the field of chemical sensors. High surface to volume ratio and enhanced reactivity of nanosensors [1] improve device response, even at room temperature, and limit the device power consumption. It has been reported that when metallic nanowires or nanoparticles are exposed to specific analyte molecules, a change in their electrical resistance is recorded [2]. Analogous effects have been observed for single walled carbon nanotubes [3, 4] and for metal oxide semiconducting nanostructures [5-7].

In order to overcome the obvious complexity related to nanodevice fabrication, several special tools have been, and still are, under investigation. In this respect, one of the most promising apparatus is the

Focused Ion Beam (FIB). The use of high energy FIB is an already well established technique in microelectronics and micro-fabrication for mask-less patterning. Moreover milling and deposition by FIB have already found applications in micro-electromechanical and micro-optomechanical systems, micro-sensors and actuators [8, 9].

In this work, we present how the FIB technology can fabricate different types of electrode patterns. Two transducers have been micromachined. For the first a single rope of carbon nanotubes has been isolated on a silicon/Si_3N_4 substrate and then, at its ends, two platinum microelectrodes have been deposited by means of FIB; for the latter 1 μm width channel has been milled by FIB transversally to a Cr/Au pad, previously e-beam evaporated, on which bundles of SWCNTs ropes have been deposited. Electrical behaviour at room temperature in toxic gas environments has been investigated and compared to evaluate contribution of a single rope in a sensor device respect to bundles one. For all the devices, upon exposure to NO_2 and NH_3, the conductance has been found to increase or decrease respectively. Conductance signal is stronger for sensor based on bundles, but it also evident that response time in NO_2 is faster for device based on a single rope. FIB technology has been then used to evaluate the contribution of a single carbon nanotube rope from bundles.

2. Experimental

The system (FEI – Quanta 200 3D), used to realize electrode patterns, is a Dual Beam which integrates an high resolution FIB, a scanning electron microscopy (SEM) and a gas injection system (GIS) composed by a Pt organometallic precursor.

Starting from a silicon substrate coated with a Si_3N_4 layer, 100 nm deposited by plasma enhanced chemical vapour deposition (PECVD), we have realized two different transducers on which a SWCNTs suspension has been deposited. SWCNTs, used in this work, have been prepared by laser ablation arranged normally in the form of bundles shaping ropes of about 50-80 nm thickness and 3-4 micron length [10]. Ropes are bundles of nanotubes packed together in an orderly manner (see Fig. 1 as an example).

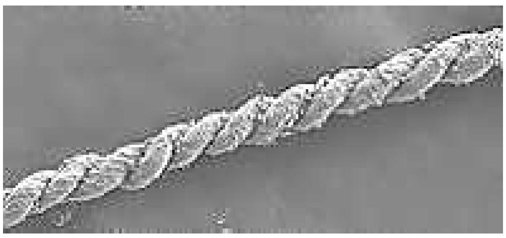


Fig.1. SEM image of a single SWCNTs rope [11].

To have a good nanotube dispersion, SWCNTs have been dispersed in dimethylformamide (DMF), 0.2mg/ml concentration, and sonicated, at room temperature, in an ultrasonic bath (*Elma*, Transsonic DIGITAL S model T 490 DH) for 2 hours at 70 W, obtaining visually, at naked eye, a homogeneous solution.

The first device has been realized depositing, directly onto Si/Si_3N_4 substrate, 2μL suspension and evaporated under vacuum at 70°C overnight. After DMF evaporation, we have put the sample in vacuum chamber of Dual Beam apparatus. We have explored the surface by SEM, selecting one of the ropes and, leaving the sample in vacuum chamber, we have started with platinum deposition. By means of FIB, we have deposited platinum microelectrodes at each single rope end, adjusting ion beam work conditions at 30 kV accelerating voltage and 10 pA emission current. Each electrode, 300 nm in width and 500 nm in height, has a length of a few microns, ending in 0.25 cm^2 gold pads, previously deposited by e-beam. Emission current has been set at 1 pA near the rope to reduce possible platinum redeposition on its surface. In Fig. 2 a FIB image of device based on a single carbon nanotube rope contacted by platinum microelectrodes is shown together its scheme (see inset in Fig. 2).

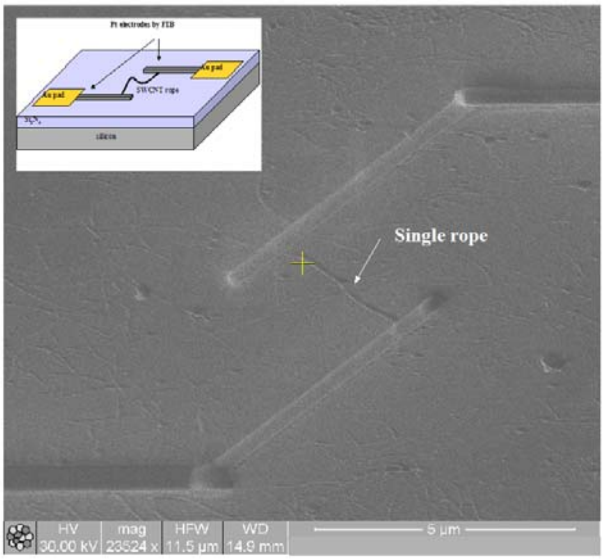


Fig. 2. Ion image of a single rope on the silicon/Si_3N_4 substrate contacted by two Pt microelectrodes deposited by FIB. In the inset the device scheme is reported where Pt microelectrodes have been deposited on Si/Si_3N_4 substrate, ending on Cr/Au pads pre-patterned.

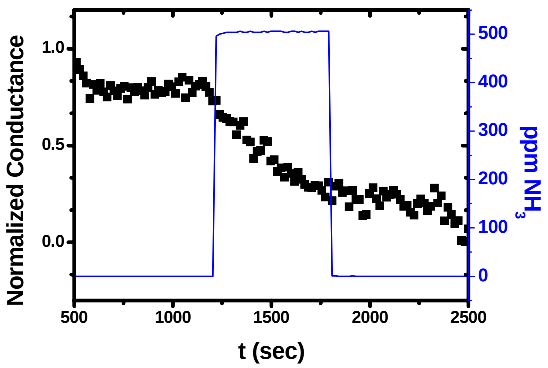
The second device has been realized, always by FIB, milling a Cr/Au pad, previously e-beam evaporated on Si/Si_3N_4 substrate, and realizing 1 micron channel on which 2μL suspension have been deposited and evaporated under vacuum at 70°C overnight. FIB operating conditions (accelerating voltage of 30 kV and emission current of 30 pA) have been chosen to obtain a clean channel without damaging nitride layer and avoiding metal redeposition. In Fig. 3 a FIB image of device, after DMF evaporation, is shown. Several carbon nanotube ropes are present onto the channel.

Fig. 3. A channel, 1 µm width, has been milled by FIB transversally to a Cr/Au pad, previously evaporated by e-beam. SWCNT ropes/DMF suspension has been deposited on channel.

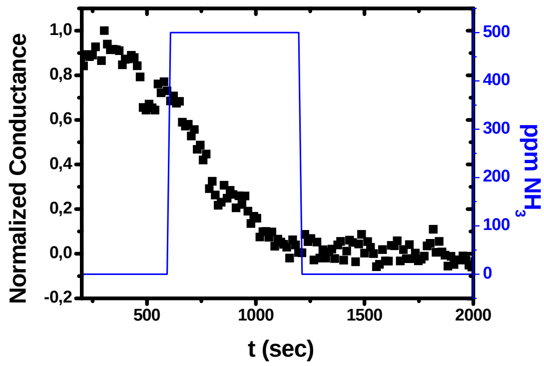
All the devices have been electrically characterized at room temperature in ambient air. For all the series, I-V characteristic is linear between –1 ÷ 1 volt. The device characteristic is ohmic with resistances about 600 kΩ and 3 kΩ for device based single rope and for bundles of ropes, respectively.
Devices have been electrically characterized as chemical sensors measuring their response, at room temperature, towards NO_2 and NH_3. A volt-amperometric technique, at constant bias, has been employed for sensor dc electrical characterization in a controlled gas-flow environment, pre-mixed with dry carrier in the desired percentage by mass flow meters and continuously controlled by means of an in-line Fourier Transform InfraRed (FTIR). All the tested devices have been biased at 0.1V. Total gas flow has been set to 500 sccm. For the measurements here reported, certified bottles containing mixtures of 30 ppm of nitrogen dioxide in synthetic air and 500 ppm of ammonia in synthetic air have been used [12].

3. Results and Discussion

Devices based on SWCNT ropes, realized by means of FIB, have been characterized in presence of toxic gasses and compared each other. It has been possible a comparison between these devices, even if electrode pattern is different. In fact SWCNTs based different geometry transducers, as photolithography, e-beam evaporation and FIB technology, show the same electrical signal [13]. In Fig. 4 the normalized conductance is reported towards the observation time when devices are exposed to 500 ppm of NH_3 in synthetic air.



(a)



(b)

Fig. 4. Normalized conductance vs time in presence of 500 ppm NH_3: a) device based on a single carbon nanotube rope; b) device based on bundles of carbon nanotube ropes.

Fig. 4a and Fig. 4b show the response of sensor device based on a single carbon nanotube rope and on bundles of ropes, respectively. Electrical variation is stronger for sensor based on bundles, 20 times higher than device based on single rope, while both of response times are 10 minutes. In Fig. 5 the normalized conductance is reported towards the observation time when devices are exposed to 30 ppm of NO_2 in synthetic air.

Fig. 5a and 5b show the response of sensor device based on a single carbon nanotube rope and on bundles of ropes, respectively. Electrical variation is always stronger for sensor based on bundles, 20 times higher than device based on single rope, but now the response time of sensor based on a single rope (Fig. 5a) is faster than the other, 3 minutes respect to 8 minutes. This result emphasizes how it is possible by means of Dual Beam (SEM/FIB) to isolate and contact one single carbon nanotube rope for extrapolating its response from bundles contribution.

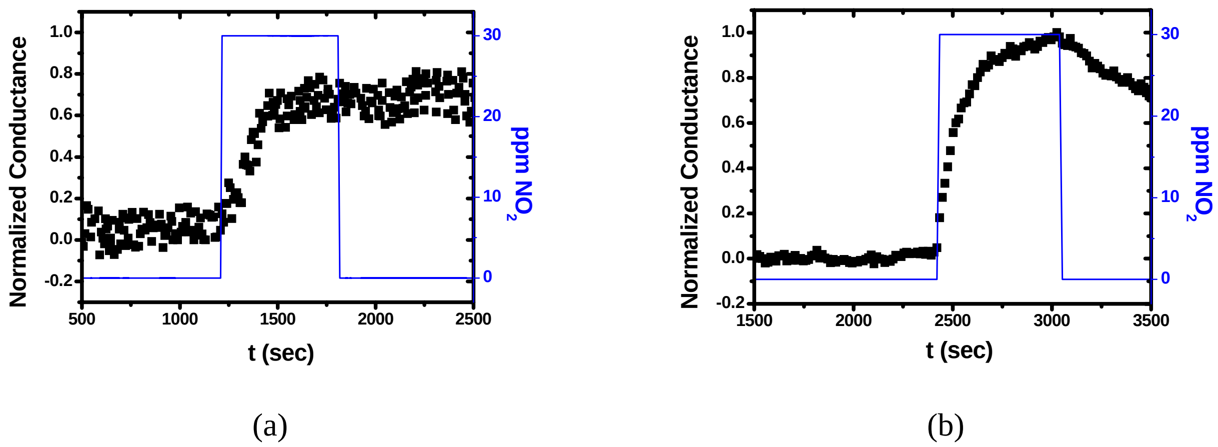


Fig. 5: Normalized conductance vs time in presence of 30 ppm NO_2: a) device based on a single carbon nanotube rope; b) device based on bundles of carbon nanotube ropes.

4. Conclusions

In this work it has been shown how it is possible to use FIB technology for micromachining of electrode patterns for single walled carbon nanotubes ropes based sensor.

Two types of transducers, starting from Si/Si_3N_4 as substrate, have been realized obtaining sensors based on a single carbon nanotube rope and on bundles of ropes. These devices have been electrically characterized in NO_2 and in NH_3 showing different response times and signal intensities.

Peculiarity of FIB technology is the possibility to contact easily sensitive nanowire realizing micro-nanoelectrodes for sensor application.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726- 5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
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- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
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- Nanosensors;
- Microsystems;
- Applications.

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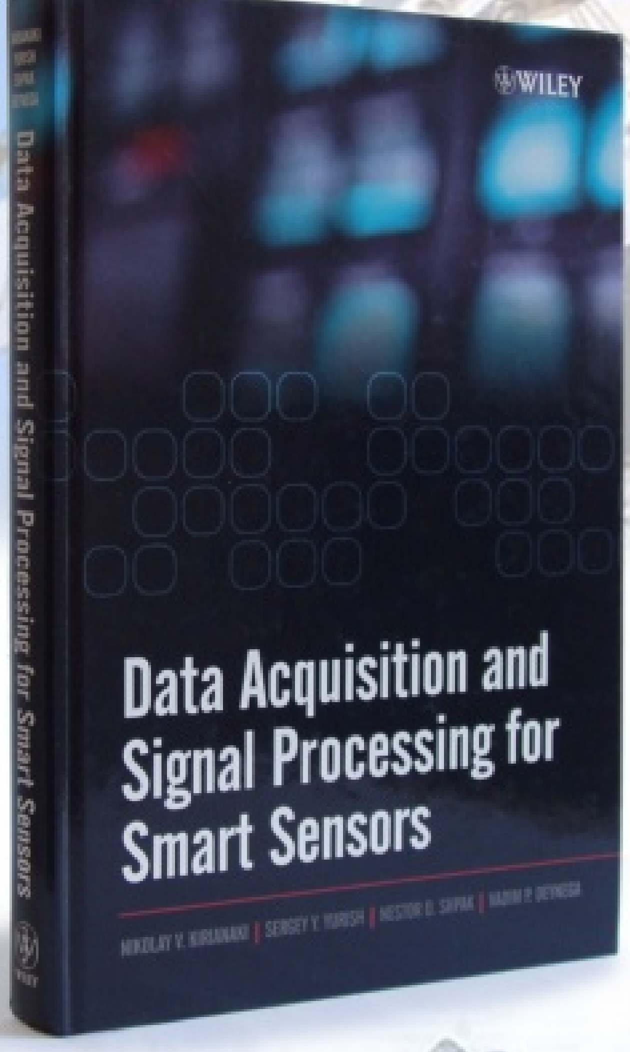

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