



Selective gas detection using CNTFET arrays fabricated using air-brush technique, with different metal as electrodes



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ABSTRACT

This paper deals with the tests of carbon nanotube field effect transistors (CNTFETs) for gas sensing applications, exploiting an original sensing technique to dramatically improve selectivity. Such devices exploit the extremely gas-sensitive change of the Schottky barrier heights between carbon nanotubes (CNTs) and drain/source metal electrodes. This effect is at the origin of the change of the CNTFET transfer characteristics. Indeed the main effect is related to the gas adsorption creating an interfacial dipole that modifies the Fermi levels alignment and so the bending and the height of the Schottky barrier at the contacts with the CNTs. This change is strictly dependent on the metal/CNTs junction and on the gas involved. We have fabricated on the same chip an array of four CNTFETs composed of four different metals (Pd, Pt, Au, Ti) as electrodes and we have demonstrated that each CNTFET interacts in a very specific way, identifying a sort of electronics fingerprinting. This array has been tested after exposure to NO₂, NH₃ and di-methyl-methyl-phosphonate (DMMP, a sarin gas simulant) with gas concentrations varying from 10 ppb to 10 ppm using air as gas carrier.

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1. Introduction

The first paper demonstrating the efficiency of carbon nanotubes field effect transistors (CNTFETs) for gas sensing applications was published in 2000 by Kong et al. [1]. Since then, many teams have obtained very interesting results concerning the sensitivity of this new kind of sensors. We can mention, for example, the results obtained by Qi et al. [2] who were able to detect 1 ppt of NO₂ and, the same year, Snow and co-workers who were able to detect 1 ppm of di-methyl-methyl-phosphonate using transistors made with random networks of carbon nanotubes [3,4]. These results seem to prove that the main issue for this kind of sensors is not the sensitivity. Indeed the real concern consists in finding a suitable technique to achieve a highly selective sensor that could be used in every-day life applications or in operational context. Various methods have been proposed so far. Among these, the deposition of polymers on the CNTFETs (functionalization) is extensively studied and very promising results have been already obtained [see ref. [2]]. However, the use of polymers could present several

drawbacks such as increasing the sensor response time and decreasing its lifetime as methods currently used to desorb gas molecules (thermal anneal and UV exposition) should degrade these polymers. Another issue is the lack of knowledge on the real physical effect of polymers: up to now the choice of polymer continues to be empirical. Another approach is the bio-functionalization, using DNA sequences of the single-walled-CNTs (SWCNTs) to improve the sensitivity for specific gases, performed by researchers at Pittsburg University [5]. Finally we can mention the approach developed jointly by researchers at Nanomix Inc. and Pittsburgh University [6,7]. They deposited nanoparticles of different metals on networks of SWCNTs connecting, by percolation, two Pd electrodes (“metal decoration”). Therefore they fabricated an array of CNTFETs each one characterized by a different metal “decoration”. They exposed this array to several gases (NO, H₂, CO, CH₄, H₂S, NO₂, NH₃) and they observed a specific change of the transfer characteristics of each transistor as a function of the nature of the nanoparticles and of the gas. These results made researchers think that large arrays of “metal-decorated” CNTFETs could be used in order to recognize univocally the gases, identifying their electronic fingerprinting. All the approaches that we have presented above appear to be very interesting from a scientific point of view but the technological steps for the fabrication of the sensors (e.g. polymer/bio-functionalization or metal decoration) are quite complex and their industrial exploitation has not been

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realized yet. Our approach for solving the selectivity problem is to make a sensor comprising an array of SWCNT-based transistors where each transistor will be fabricated using different metals as source/drain electrodes. Considering that no cheap and rapid method, up to now, exists for separating semiconductor from metallic SWCNT specimens, we decided to use SWCNT mats as transistor channel. There are two principal advantages in doing so. Firstly the use of SWCNT mats instead of individual SWNTs (facilitating methodology) results in percolation networks with a better overall electrical control of the system. Secondly, using the dynamic spray-gun technique (patented by Thales) we can achieve uniform and highly controlled SWCNT densities which allows us to obtain CNTFETs with highly reproducible electrical characteristics [8,9]. Moreover, this technique allows us to fabricate large array of sensors with a method very simple, rapid, totally compatible with existing CMOS technology and therefore suitable for industrial exploitation at relatively low-cost. We have chosen to diversify the metals electrodes in order to exploit the different effects of the interaction of the gas with the metal/SWCNTs junction on the Schottky barrier. As a consequence after gas exposure the Fermi level alignment between metal and CNT at the contact will change, modifying the characteristics of the Schottky barrier at the junction. Actually, we cannot talk about a “real” Schottky barrier in our case because we do not deal with a junction exhibiting an intimate contact (like a classical Schottky barrier between semiconductor and metal, where the Fermi Level is pinned by interface defects as explained by Leonard and Tersoff [10]). In our case we have a “non-intimate” contact because the SWCNT is simply deposited on the metal without annealing. This model was firstly suggested by Yamada [11,12] of the Ames Research Center in 2004 for Au/SWCNT contacts, generalized by Bondavalli et al. [13] to all the metal/SCWNT junctions and, for network based CNTFETs, further improved by Peng et al. [14]. In this contribution we will demonstrate that, after being exposed to different gases (NO_2 , NH_3 and DMMP) at concentration from 10 ppb to 10 ppm, each transistor will be influenced in a very peculiar way: this influence on the barrier is strictly related to the interaction between a specific gas and a specific metal/CNT junction [15]. Indeed, the gas adsorption will change the metal work function and so the junction electrical behavior. This is not possible for traditional semiconductor/metal junctions because the Fermi level is pinned by interface defects, as quoted above [10]. Using this method, we will be able to demonstrate that each gas will be “fingerprinted” and so unambiguously identified.

2. Experimental

2.1. CNTFET fabrication

Our CNTFET arrays have been fabricated using n-doped Silicon substrates (common gate electrode) covered with 50 nm of thermally grown SiO_2 . The electrodes have been prepared by UV photolithography: the metal deposition has been performed using evaporation technique and lift-off. This step has been repeated sequentially for each metal in order to achieve a final chip ($0.5 \text{ cm} \times 0.5 \text{ cm}$) composed by 16 couples of electrodes. We have prepared two series of eight couples of electrodes with a distance of $10 \mu\text{m}$ and a width respectively of 1 and 3 mm. In this way we can modulate the final current value between Drain and Source: the wider electrodes will be able to provide higher current because the number of SWCNT chains linking the two electrodes is higher. The metal electrode thickness was 35 nm. A 5 nm Ti thick layer has been added in order to improve the layer adhesion of Au, Pd, Pt and Ti layers on the substrate (see Fig. 1).

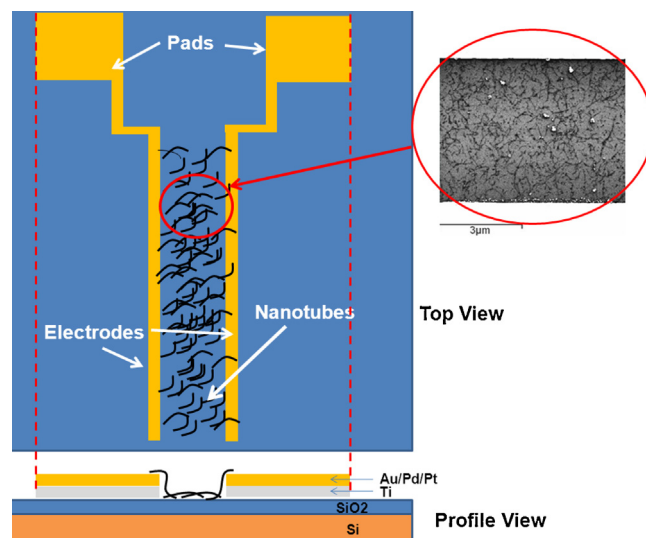


Fig. 1. Top and bottom schematic view of one of the transistors. In the inset a SEM image of the SWCNTs based mat acting as channel in the CNTFET.

2.2. SWCNT deposition

The SWCNTs that we have used, have been supplied by South West NanoTechnologies, and were CoMoCat SG65 composed by 90% of semiconducting SWCNTs [16]. In order to obtain stable suspensions, we have put them in solution using N-methylpyrrolidone (NMP) as solvent [17]. SWCNT bundles and residual impurities (e.g., catalyst particles) have to be eliminated from the original SWCNT suspension. This can be accomplished by sonication to “break” the bundles (1 h), followed by centrifugation (two phases of 10 min at 3000 rpm) and careful recuperation of the supernatant part of the suspension. The final solution of SWCNTs has been deposited using a dynamic air-brush technique developed at Nanocarb Lab. at Thales Research and Technology (patented) (Fig. 2).

This method relies on the SWNT deposition from the suspensions using a spray-gun which can be moved along the three axes ($35 \text{ cm} \times 35 \text{ cm} \times 35 \text{ cm}$). In order to achieve uniform mats of nanotubes on large surfaces in an extremely reproducible way, the substrates, on which the solutions are deposited, are heated to avoid the so-called “coffee ring” effect (the tendency of nanoparticles to move to the borders of the drops when evaporating at ambient conditions [18,19]).

Indeed, in our specific case the substrate was heated to a temperature higher than $202 \text{ }^\circ\text{C}$ (evaporation point for NMP) in order

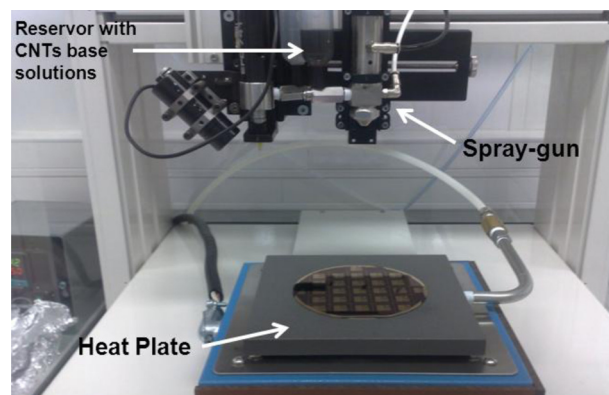


Fig. 2. Overall view of the set-up for the deposition of solutions of CNTs using the dynamic air-brush deposition technique.

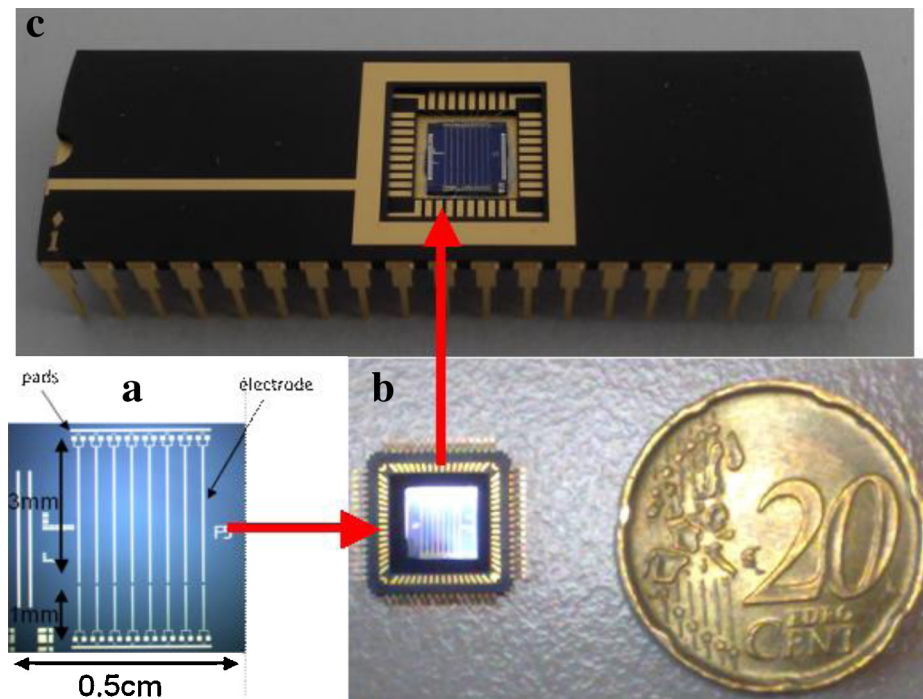


Fig. 3. (a) Chip containing 16 CNTFETs achieved using four different metals as electrodes (four for each metals), (b) Chip dimensions compared to a 20 cents coin, (c) Chip mounted on the C-DIL used for tests.

to evaporate instantaneously the solvent droplets hitting the substrate: in this way the drops will instantaneously “collapse” and the SWCNTs are pinned at the impact zone. The advantage of this technique, compared with similar technologies such as electrospray, is that it is not necessary to apply an electric field to deposit the nanomaterials. Actually, this electric field can damage the nanomaterials' functionalization and the nanomaterials have the tendency to be mainly distributed along the electric field lines (and so not uniformly). Other teams utilize static spray, i.e. motion-less deposition method. This technique does not permit to obtain extremely uniform mats on large surface areas and the thickness is not precisely tuned. Moreover this static spray technique is not scalable and so not suitable for industrial exploitation.

2.3. Experimental set-up

Our test set-up measures the electrical characteristics (current between drain and source as a function of gate voltage) of the SWCNT based transistors and, the changes over time, before and after exposure to a specific gas. The sensor chip is mounted in a ceramic-dual-in-line package (C-DIL), model KD-S78346-C, and the drain and source contacts are wire bonded (see Fig. 3). The currents and voltages are controlled and measured thanks to 3 Keithley Source Meters 2400. Indeed considering that we have to test four transistors at the same time (for each different metal electrode couples) we need four source measurement units (SMUs) for the four drain contacts, one for the source contacts (in common and connected to ground) and one for the back gate (see Fig. 4). To carry out the electrical measurements, instead of standard probe tips, we have used an electronic card on which is implemented a socket. Then the C-DIL package with the sensor chip, is plugged onto the socket. After plugging the chip housing to the socket, the electronic card is inserted inside the gas enclosure through an aperture made for this purpose. The samples are placed inside a chamber where we can control the gas composition under which the sample is exposed.

The gas input tube is positioned right above the sample (the chip with the CNTFETs). The design of the input tube is performed

to keep a laminar flux on the sample and so to control, during all the test phase, very accurately the gas concentration (see Fig. 5).

To manage the gas composition inside the chamber, we use a set of mass flow meters, a bubbler (for those materials, which are liquid at room temperature), tubes connected to gas bottles on one side and to the measurement enclosure on the other side. This set-up allows us to dilute gases up to 1000 times and so to perform tests with concentrations potentially varying between 10 ppb and 10 ppm. The gas carrier used for our tests was dry synthetic air and the flux value corresponded to 300 sccm. Indeed, the sample is in a chamber where ambient air is present. We have worked at ambient conditions and so at ambient pressure. Thanks to

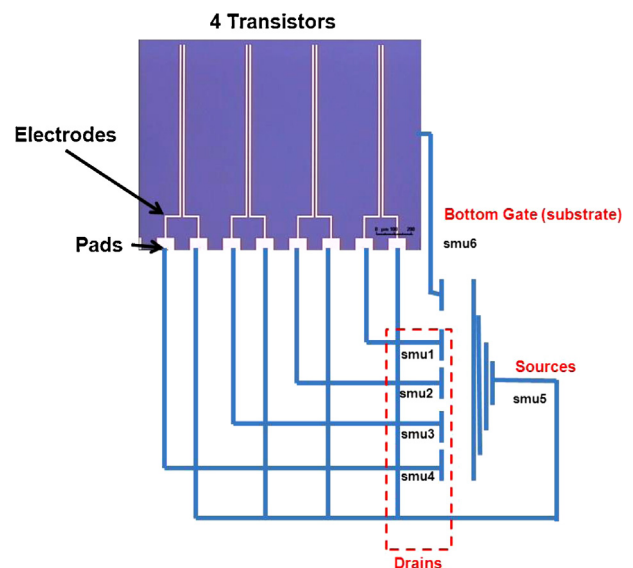


Fig. 4. Schematic view of the set-up connections. Smu 1,2,3,4 corresponds to the transistors drains. Smu 5 to the sources which are grounded and connected together. Smu 6 corresponds to the bias applied to the substrate that acts as bottom gate.

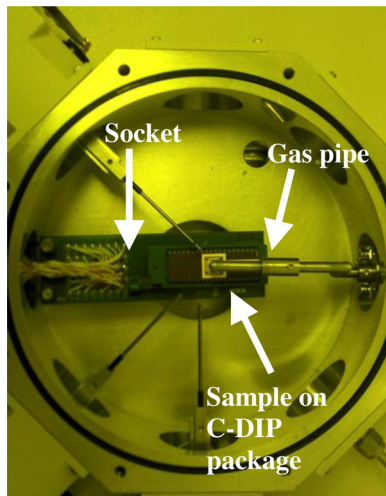


Fig. 5. Detail of the chamber used to perform measurements.

home-made software, we can control the gas input composition (through flow meters) which we can change over time with pre-programmed sequences, and we can both send routines to the Keithleys and save data (transistor characteristics, and drain current over time) on a computer.

3. Experimental results

After fabricating chips, we have performed measurements exposing chips to three different gases, namely NO_2 , NH_3 and dimethyl methylphosphonate (DMMP). We have exposed each time one single chip to the gas. In this way we were able to record the change of the transfer characteristics of four CNTFETs, composed by four different metals (Au, Pt, Pd, and Ti) as electrodes, at the same time. The first measurements have been performed exposing the CNTFETs to concentration values from 100 ppb to 10 ppm for NO_2 and NH_3 (see Fig. 6 for schematics explaining the

modification of the Schottky barrier height by dipole formation at the injecting contact when the CNTFET is exposed to NO_2 or NH_3). In this way we have tried to evaluate the limit of sensitivity of each metal. The variations of the I_{DS} current during the exposure cycles (for $V_{\text{GS}} = -16 \text{ V}$ and $V_{\text{DS}} = -1.6 \text{ V}$) are shown in Figs. 7 and 8 for NO_2 and Fig. 9 for NH_3 . The exposure time was of 300 s to gas and 600 s to air.

All the CNTFETs fabricated show a p-type behavior because the metal work functions are larger than the CNT work function (around 3.7 eV). However Ti has a lower marked p-type behavior because its Fermi level approaches the CNT's one. Moreover Ti-based electrodes are naturally oxidized and so the current is quite low compared to the other metals. The CNTFETs have been exposed during 300 s to the gas, followed by 600 s to air. From the measurements, it is highlighted, as shown in literature, that the behavior of the CNTFETs is exactly the opposite in the case of the two gases. As shown in previous papers [13–15] in case of short channel configurations (less than $100 \mu\text{m}$) and gas concentration lower than around 200 ppm (for the most reacting gases [20]), the main effect of gas on the CNTFET is at the metal/CNT junctions.

Indeed, if we consider that the contact resistance between two SWCNTs in the random mat varies after being exposed to a gas, this effect will be negligible if the channel transistor is short ($<100 \mu\text{m}$). In fact, the metal/SWCNTs contact resistance is around three orders of magnitude larger than the resistance between two SWCNTs with the same electronic character. Therefore, the metal/SWCNT behavior at S/D contacts will dominate the overall sensor behavior. This predominance will be strongly reduced using long channel transistors ($\sim >100 \mu\text{m}$) with a number of SWCNT/SWCNT contacts (in each chain linking the two electrodes) larger than 100. In this case, the mat resistance will attain a value comparable to the metal/SWCNT contact resistance [4,21].

Indeed considering that NH_3 is a so-called “electro-donating” gas, it increases the Schottky barrier at the metal/CNT interface (for all the metal electrodes) as if electrons were transferred in the SWCNT (moving up the Fermi level) and so it reduces the passing hole current. Exactly the opposite behavior is observed for NO_2 , which is an “electro-withdrawing” gas (the Schottky barrier is

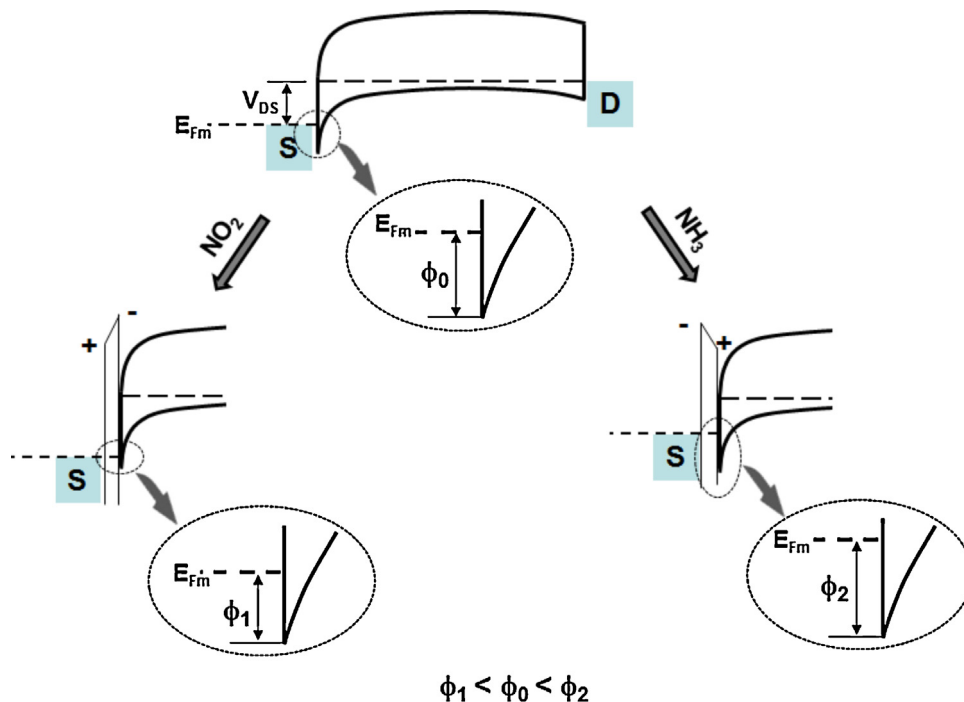


Fig. 6. Effect of the gas molecules of NO_2 and NH_3 on the alignment of the Fermi Levels of CNTs and metal electrodes.

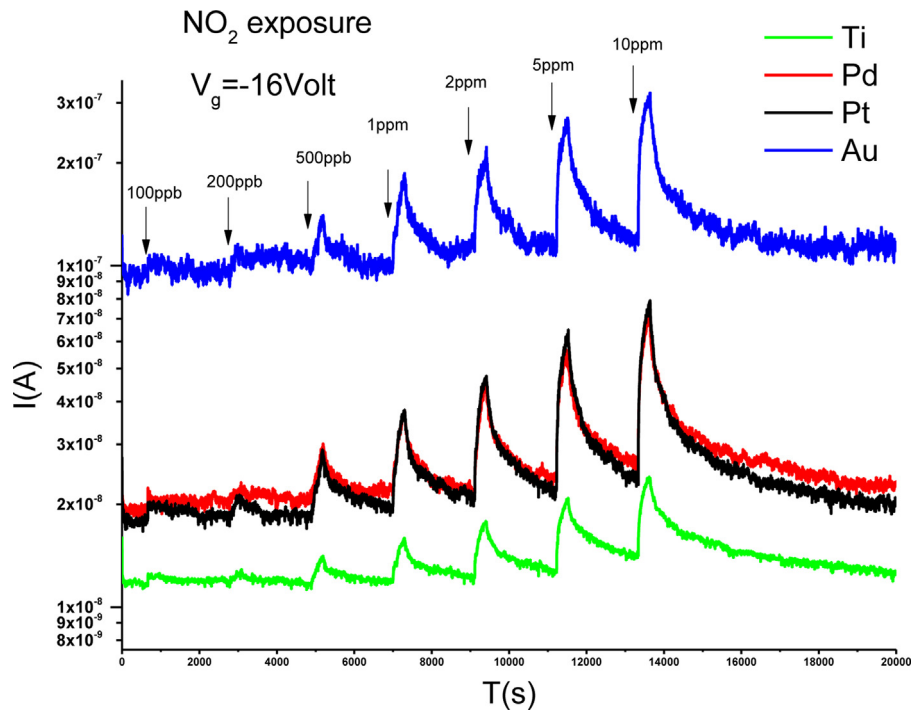


Fig. 7. Change of the I_{DS} current as a function of time for $V_{GS} = -16V$ and $V_{DS} = 1.6V$ for concentrations between 100 ppb and 10 ppm of NO_2 . The exposure time to NO_2 is of 300 s and to air of 600 s.

reduced by the downwards displacement of the Fermi level in the SWNT see Fig. 6). We can also see that the CNFETs seem to be more sensitive to NO_2 . We can observe a clear change in the I_{DS} for concentrations as low as 100 ppb for Pt and Au, and for all the metals at 200 ppb (Figs. 7, 8). For exposition to NH_3 , Au and Pt show a change in I_{DS} for 100 ppb but in order to see a clear change for all the CNFETs, we have to reach a concentration of 1 ppm. These results are at the state of the art considering that our chip has not been functionalized (using chemical or biological species) and the measurements have been performed using air as carrier gas at ambient pressure and temperature.

3.1. Testing selectivity

The most important issue for gas sensors is the selectivity. Our approach is to use different metals as electrodes because the interaction of each gas with each metal is specific. In this way we are able to identify a sort of fingerprinting of each analyte. This idea is not simply based on the fact that for “short” channel configurations the main effect of the gases is on the metal/CNT junctions. Indeed, considering that the channel (composed by CNT-based mats) is the same one for each transistor, the only difference in the reaction of a particular gas with the CNFET array can only originate

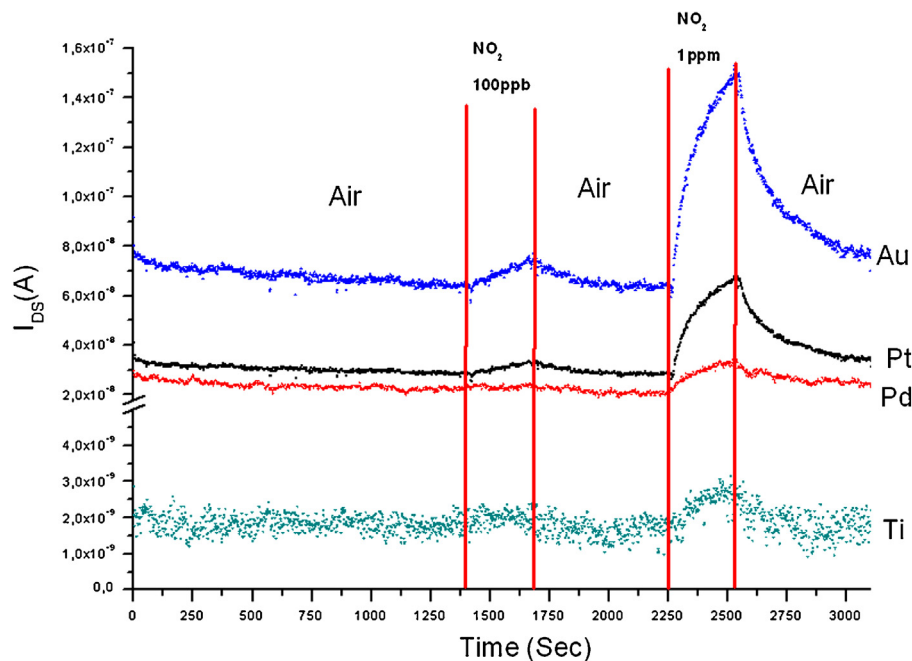


Fig. 8. Detail of the change of the I_{DS} current as a function of time for $V_{GS} = -16V$ and $V_{DS} = 1.6V$ at 100 ppb and 1 ppm. The exposure time to NO_2 is of 300 s and to air of 600 s.

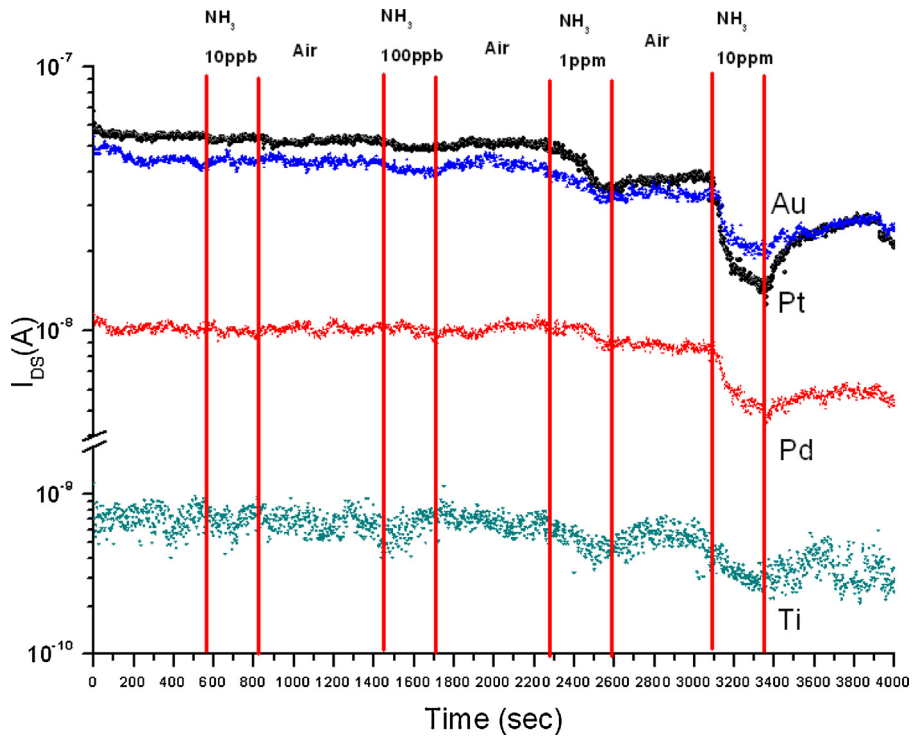


Fig. 9. Change of the I_{DS} current as a function of time for $V_{GS} = -16V$ and $V_{DS} = 1.6V$ for concentrations between 10 ppb and 10 ppm of NH_3 . The exposure time to NH_3 is of 300 s and to air of 600 s.

from the S/D contact junctions (fabricated using different metals as electrodes). In order to verify this hypothesis, we have checked that the response of the chip is not only different for two gases of two different families (like NH_3 , NO_2), but also for gases with the same electronics character. For this reason we have performed different measurements exposing the same sensor chip to DMMP,

which is an “electro-donating” gas (like NH_3). To obtain concentrations from 1 ppm to 20 ppm, we have bubbled dry air through the liquid DMMP. We have not been able to test the sensitivity for lower concentrations because the saturation pressure of DMMP is ~ 1600 ppm at ambient conditions, and with our system (max dilution of 1/1000) we can only reach a maximum concentration

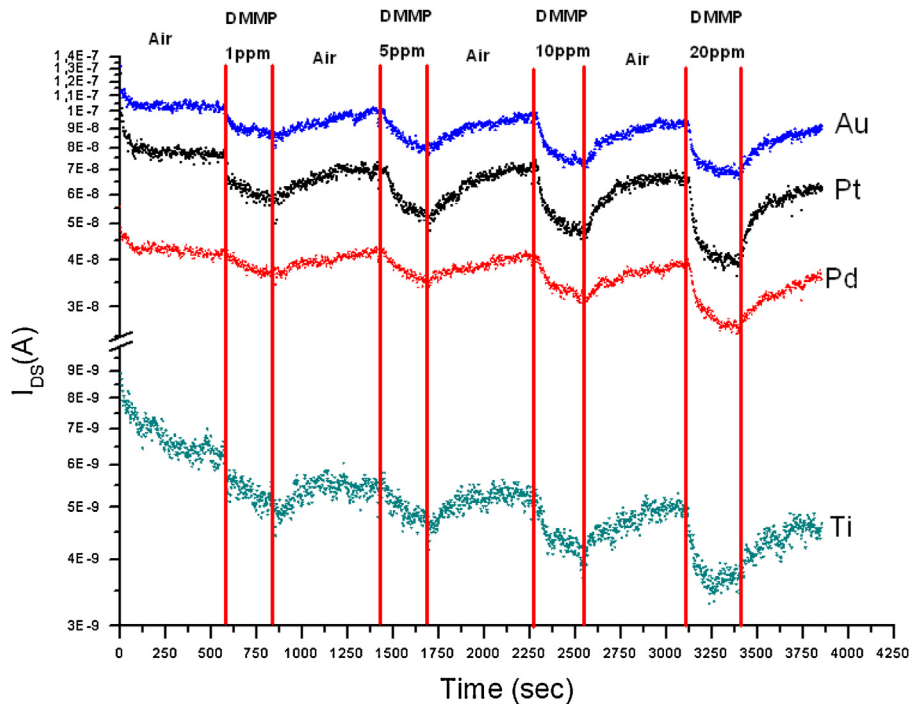


Fig. 10. Change of the I_{DS} current as a function of time for $V_{GS} = -16V$ and $V_{DS} = 1.6V$ for concentrations between 1 ppm and 20 ppm of DMMP. The exposure time to DMMP is of 300 s and to air of 600 s.

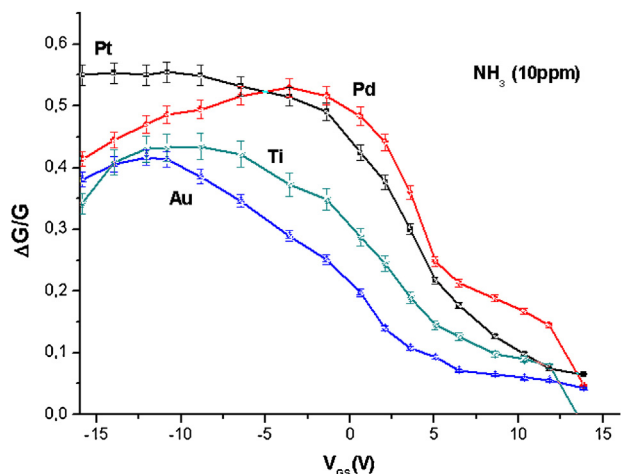


Fig. 11. Relative conductance change as a function of V_{GS} for $V_{DS} = 1.6V$ for a concentration of 10 ppm of NH_3 .

around 1 ppm. The results of several exposure cycles are reported in the following Fig. 10.

In order to deeply analyze the effect of each gas on each CNT-FETs we have calculated the relative change of resistance for a concentration of 10 ppm after 300 s of exposure for $V_{DS} = -1.6V$ (Figs. 11–13). We have chosen this concentration because we have a valuable change of the I_{DS} for all the CNTFETs and all the gases. The values are reported as a function of the gate voltage for each gas. The most important result is that each gas appears to interact in a specific way with each CNTFET fabricated using a different metal. For example for NH_3 the more sensitive CNTFET, for gate voltages higher than $-5V$, is composed by Pd-based electrodes, followed by the Pt, Ti and Au-based ones. For lower voltage values, Pt appears to be more sensitive compared to Pd. For NO_2 sensing, the most sensitive CNTFET is composed by Pt-based electrodes for all the gate voltage values, followed by Pd, Au and Ti for voltages higher than $0V$. For lower voltage values, Au appears to be more sensitive than Pd and Ti. Finally, in the case of DMMP (a gas with the same electronics character as NH_3) the more sensitive metal is Pt on the entire spectrum followed by Pd, Au and Ti. We can observe that the more sensitive metals electrodes are, in the three cases,

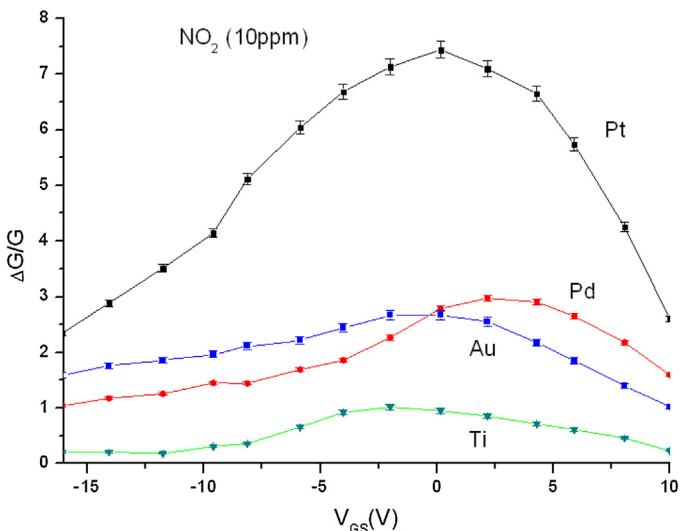


Fig. 12. Relative conductance change as a function of V_{GS} for $V_{DS} = 1.6V$ for a concentration of 10 ppm of NO_2 .

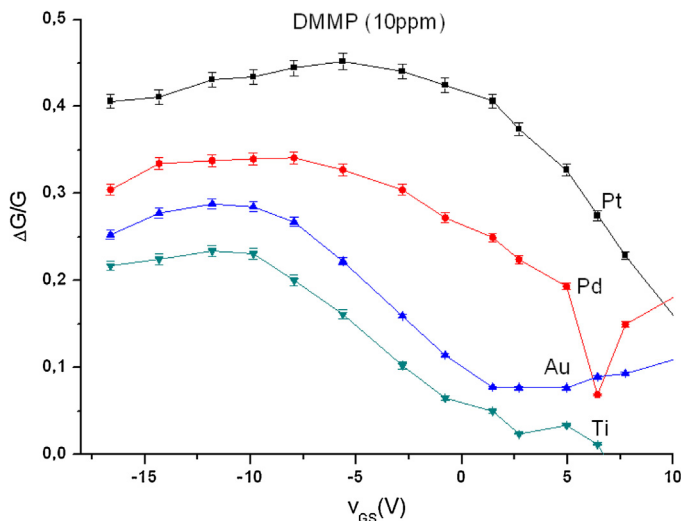


Fig. 13. Relative conductance change as a function of V_{GS} for $V_{DS} = 1.6V$ for a concentration of 10 ppm of DMMP.

not the same for the entire gate voltage spectrum. If we observe more carefully the graphics of the relative conductance change, for each metal we have a maximum at a specific value. This is related to the fact that the gas changes the barrier at the metal/CNT contact and so it translates the $I_{DS}(V_{GS})$ characteristics. In the case of NH_3 and DMMP, considering that they are both “electro-donating” gases, they promote a translation of the $I_{DS}(V_{GS})$ characteristics toward negative bias (the SWNT Fermi level increases and so the threshold voltage of the CNTFETs are more negative, as shown by Kong et al. [1] in case of single nanotube based CNTFETs). In case of NO_2 we observe the opposite effect and the transfer characteristics are translated toward higher gate voltage (the Fermi level of the SWNT decreases see e.g. Fig. 14 for Pt). From this analysis, we can deduce that we can achieve a sort of electronics fingerprinting of the gas (for a defined gate voltage) simply with our chip composed by a CNTFET array with different metals as electrodes. This seems to demonstrate that it is the effect of the gas on the junctions that influences the response of the CNTFETs, considering that, as previously quoted, the channel composed by nanotubes is exactly the same for all the CNTFETs.

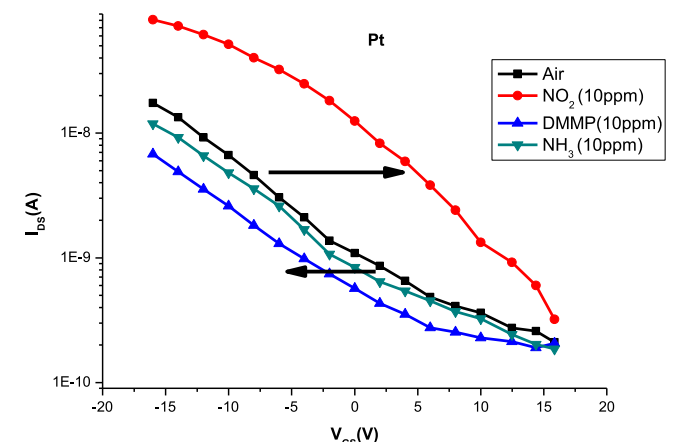


Fig. 14. Transfer characteristics of a CNTFET fabricated using Pt electrodes after exposure to Air, NO_2 , NH_3 and DMMP (10 ppm, each time for 5 min).

4. Conclusions

In this work, we have demonstrated that the NO₂, NH₃, and DMMP gases interact differently with each metal/CNT junction. This is because each gas changes the Fermi level alignment of the SWNTs with the metal at the S/D contacts in a specific way and therefore changes the transfer characteristics of the CNTFETs. This effect does not depend on the initial metal work function. In fact, if we analyze the relative change of the conductance, we have observed that it is not correlated to the starting work function and it is different for each gas. From these results, we think that CNTFET-based arrays could be potentially used to obtain an electronic fingerprinting of specific gases and therefore to perform selective sensing using very simple, relatively low-cost devices. The results obtained in this contribution, concerning the sensitivity of all the transistors on the same chip (200 ppb for NO₂, 1 ppm for NH₃ and around 1 ppm for DMMP), are at the state-of-the-art considering that we have not functionalized chemically or biologically our devices and that we have tested CNTFETs fabricated using CNT mats as channels for the transistors at ambient conditions.

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Biographies

P. Bondavalli, Msc, PhD, Hdr is the Head of Nanomaterial team at Thales Research and Technology (CNRS/Thales, UMR137) and he is a member of the Nanocarb Lab. (joint team Ecole Polytechnique/Thales). Presently his work is focused on the development of new materials (e.g. graphene, cnts, nanowires) for the new generation of electronics devices and for energy storage applications and memristor. Dr Bondavalli has received his Hdr in 2011, at Paris-Sud on a work on “devices based on random network of carbon nanotubes”. He is EU expert, and Vice-Chairman, for Marie Curie Fellowships (EIF, IIF, OIF, CIG, IRSES), NMP and ICT panel, for the French National Research Agency (ANR), EDA, Eureka and reviewer for several international journals. During the last five years, he has participated, also as coordinator, in several EU projects (concerning MEMS, MOEMS, CNTs, graphene, spintronics) and ANR projects. He is involved in the Graphene Flagship initiative (Energy and High-frequency WPs).

L. Gorintin received his PhD in 2011 for his work on the topic of carbon nanotube field effect transistors for gas sensing at Thales Research and Technology in collaboration with Ecole Polytechnique. He is the author of 9 papers on this topic. He presently works at GDF-Suez, dealing with innovating routes for GDF Suez needs on gas sensing.

G. Feugnet, received an engineer diploma from the Ecole Supérieure d’Optique and join Thales TRT in 1991 after a two-year stay in US (Coherent) and has been involved in the development of diode-pumped solid state laser. He published more than 30 papers and holds 15 patents. He also coordinated many multidisciplinary and collaborative projects supported by the European Defence Agency, the European Space Administration or the European Commission.

G. Lehoucq was born in 1984 in Lesquin (France). After receiving her engineer degree from Ecole Centrale de Lille in 2006, she joined Thales Research & Technology (Palaiseau, France) where she prepared her PhD, focusing on the conception, fabrication and characterization of a SiNW-based sensor. She is currently working on the fabrication of nanostructured optical devices.

D. Pribat graduated from Grenoble University (INPG) and from Orsay University, France. He worked for 24 years (1978–2002) within the Central Research Laboratory (LCR) of Thomson-CSF (now Thales Research & Technology) and joined CNRS (Centre National de la Recherche Scientifique, the French National Center for Scientific Research)/Ecole Polytechnique in 2002 and started there an activity on carbon nanotubes and semiconductor nanowires for molecular electronics and field emission applications. In September 2009, D. Pribat moved to Korea and he now holds a full time professor position in Sungkyunkwan University, Suwon, South Korea, participating in a World Class University program. D. Pribat has published more than 160 scientific papers in peer reviewed journals and presented more than 60 invited talks in international conferences. He is also co-author of 3 book chapters and author or co-author of more than 55 patents.