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Integration of Carbon Nanotubes in Microelectronics

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1. Introduction

Carbon nanotubes (CNTs) has received much attention since their discovery in 1991 due to unique combination of interesting electrical, mechanical, thermal and other properties, and numerous potential applications (Meyyappan, 2005, Sharma, 2008). Single-wall carbon nanotubes (SWCNTs) can be metallic or semiconducting, while multi-wall nanotubes (MWCNTs) are basically metallic. Semiconducting SWCNTs can be used in nanotubes based field effect transistors (FET-CNT), while metallic SWCNTs and MWCNTs can be employed for electrical and thermal interconnections, in sensors and other micro- and nanodevices. However, the integration of nanotubes into microelectronic circuitry is a very challenging task which requires development of reliable and compatible technologies for controlled synthesis, accurate positioning and contacting of nanotubes and their arrays in new devices. Many difficult issues associated with these technologies have to be addressed. In particular, mechanisms of nucleation and growth of high quality nanotubes still are not well understood. Mechanisms of electrical and thermal conductivity in individual nanotubes and ropes, the role of defects, formation of contacts with metals have to be investigated thoroughly.

2. Electrical properties of carbon nanotubes

SWCNT can be viewed as a single graphite (or graphene) sheet rolled into a cylinder of a nanometer size diameter, and MWCNT as a coaxial array of several single-wall nanotubes separated by approximately 0.34 nm. In graphene layers, $sp^2$ hybridization results in formation of three strong in-plane $\sigma$ bonds between carbon atoms and one $\pi$ bond, the latter corresponding to loosely bound $\pi$ electrons of high mobility that are responsible for a very high conductivity along the graphene plane.

As SWCNTs are essentially one-dimensional structures, at the absence of defects they are characterized by a ballistic transport of electrons (without scattering) at moderate current densities for nanotube lengths up to a few micrometers (Graham et al, 2004). This is in striking contrast to metal (copper) wires where the mean free pass (MFP), determined by the mean grain size, is in the range of a few tens of nanometers.


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Another advantage of nanotubes is that small diameter copper vias are subject to failure due to electromigration at high current densities (>10^6 A/cm^2) while CNTs of the same diameter can sustain current densities as high as 10^9 A/cm^2 (Graham et al, 2004). This makes CNTs very attractive for electrical interconnect applications (especially, vias) instead of currently used copper.

The resistance of a SWCNT (or a shell in a MWCNT) has three components (Tan et al, 2007; Matsuda et al, 2007): (i) a contact resistance associated with one-dimensional systems, given by a quantum resistance \( G_0 = \frac{h}{2e^2} = 12.9 \text{ k}\Omega \) corresponding to one conducting state (a factor of two is added due to two possible spin states), (ii) an intrinsic resistance due to scattering which is length dependent, and (iii) an additional contact resistance associated with imperfect contacts between a metal electrode and CNTs. Metals that form carbides (e.g., Ti) are believed to be an optimal electrode material as it is expected that carbides ensure better electrical coupling with nanotubes (Tan et al, 2007).

It is very challenging to evaluate precisely the contribution of contact resistances due to evident experimental difficulties. Comparison between different experiments is also not straightforward because of wide variation of conditions and particular geometries used for studies (for example, side- and end- contacts, presence of surfactants and other contaminants, metal grain sizes and degree of metal annealing after its deposition over nanotubes, etc.). SWCNTs can be metallic or semiconducting, depending on chirality. The energy gap for semiconducting nanotubes is given approximately by \( E_g(eV) = \frac{1}{d(\text{nm})} \), reducing rapidly with the graphene shell diameter \( d \). Usually, the number of metallic SWCNTs in as-grown samples is close to 1/3, the rest are semiconducting. In contrast, MWCNTs of larger diameters are basically metallic and thus are especially appropriate for interconnects.

The number of conducting states per graphene shell depends on its chirality, it can be 0 or 2 for small diameter semiconducting and metallic SWCNTs, respectively, and it increases linearly with the shell diameter for larger nanotubes (Naeemi and Meindl, 2007). MFP also was shown to increase with MWCNT diameter. The theoretical limit for resistance of high-quality MWCNTs (diameter of 15 nm, 10 walls) in a ballistic regime can be lower than 0.1 k\Omega (for lengths smaller than MFP), compared with the resistance of about 1 k\Omega for a 150 nm long, 10 nm diameter copper damascene wire (Graham et al, 2004). The model of MWCNTs as electrical conductors developed by Naeemi and Meindl, 2007, predicts that they can outperform copper wires for lengths exceeding 5-10 \( \mu \)m, depending on diameter. Bundles of SWCNTs were shown to have potentially superior performance at smaller lengths (<1 \( \mu \)m), however for this, dense nanotube packing is essential which is a very difficult practical task.

Recently, it has been reported by Jun et al, 2007, that the AC conductance of high quality PECVD (plasma enhanced chemical vapor deposition) grown MWCNTs decreases gradually with increasing frequency (frequencies up to 50 GHz were studied), indicating that nanotubes can be used not only for DC but also in a microwave range.

### 3. Synthesis of carbon nanotubes

It is important to emphasize that properties and quality of carbon nanotubes depend strongly on the fabrication method. There are two main groups of CNTs synthesis methods: (i) high-temperature processes like arc discharge and laser evaporation where the process temperature can reach \( T = 2000-4000 \) °C, and (ii) chemical vapor deposition (CVD) processes
performed at much lower temperatures: in the range of 500-1000 °C for thermal CVD and even lower for plasma-enhanced CVD. In high-temperature processes, higher quality nanotubes can be obtained, however the process output is a CNT containing soot which needs to be further processed (dispersed, purified and in some cases functionalized) before applications. The low-temperature CVD methods can be compatible with microelectronic technologies and therefore attract most attention. Note that activation by plasmas in PE-CVD processes can promote formation of higher quality nanotubes at lower temperatures, and thus PE-CVD is a promising technology for microelectronics applications. Electric fields built-up in the plasma, can also be used to provide directional nanotube growth. Studies of nucleation mechanisms (Moshkalev & Veríssimo, 2007) and search for new methods of synthesis, compatible with microelectronics technologies, must continue to provide better control on the properties, location, growth direction and quality of nanotubes.

4. Measurements of CNT resistances

Experimental measurements of individual nanotube resistances can be performed using 2- or 4-points methods. To deposit nanotubes over pre-fabricated metal electrodes, an AC dielectrophoresis (DEP) method (Krupke et al, 2007; Vaz et al, 2008) can be applied, see Fig. 1a. As contact resistances obtained after DEP are frequently very high, further improvement of CNT-electrode contacts using metal (e.g., Ni or Pd) deposition by electroless methods, usually followed by annealing, is required (Vaz et al, 2008; Liebau et al, 2003). For 4-points measurements, additional electrodes can be made using platinum (Pt) deposition induced by focused ion or electron beams, see example in Fig. 1b.

![Fig. 1. (a) Individual MWCNT deposited by AC dielectrophoresis over Pd electrodes and then cover by Ni electroless process. (b) For 4-points measurements, 2 intermediate Pt electrodes are fabricated by electron beam induced deposition.](www.intechopen.com)
diameter): ~20 kΩ per Pd or Ni electroless contacts. It should be noted that distinctly different values of MWCNT resistances were obtained for relatively short MWCNTs (nanotube lengths < 1 μm) using 2 and 4 points methods: ~30 kΩ/μm and 100 kΩ/μm, respectively. This was attributed to different contact geometries: in the former, all-around contacts were formed during electroless metal deposition over nanotubes, while in the latter, nanotubes were only side-contacted thus the contribution of internal shells to the measured conductance was considerably smaller. This finding emphasizes the importance of careful evaluation of the measurement conditions, particularly in terms of nanotube/metal contacting.

More detailed studies of the MWCNT resistance as function of nanotube length (Moshkalev et al, 2008) have shown a non-linear behavior for tubes longer than 1-2 μm, in both 2 and 4 points measurements (Fig. 2). This is likely due to increasing conduction to internal walls as tube length grows. The data can be interpreted using the model of a nanotube as a resistive transmission line consisting of two parallel linear conductors (Bourlon et al, 2004). From the model, one can evaluate the resistance of an external shell ρ1, of internal shell ρ2 (only two outermost shells are considered in the model) and the intershell conductance g. For MWCNTs produced by arc discharge method, characteristic values ρ1 ~ 10 kΩ/μm, ρ2 ~ 0.1 x ρ1 and g = (10 kΩ)⁻¹/μm were obtained by fitting using the model (Bourlon et al, 2004).

In our study for CVD grown MWCNTs, the following data were obtained, using 2 and 4 points configurations:

i. 2-points, Pd all-around contacted nanotubes (Fig. 2, left): ρ1 ~ 37 kΩ/μm, ρ2 ~ 4 kΩ/μm, g ~ (100 kΩ)⁻¹/μm;

ii. 4-points, side-contacted nanotubes (Fig. 2, right): ρ1 ~ 100 kΩ/μm, ρ2 ~ 22 kΩ/μm, g ~ (100 kΩ)⁻¹/μm.

As discussed above, the difference in measured resistances can be explained by different contact geometries.

The data presented show that resistances of MWCNTs produced by different methods are still far from theoretical limits and thus are not yet suitable for interconnect applications.
microelectronics. Better quality (lower resistance) is characteristic of nanotubes produced by high-temperature (arc, laser) methods compared with a conventional thermal CVD. Further optimization of growth and contacting technologies aiming to obtain lower nanotube resistances and better contacts (in particular, direct contact to internal walls) is strongly required.

5. Contacts of nanotubes with metals: theoretical and experimental approaches

At the most fundamental level, the resistance of a metal contact to a nanotube requires a calculation of the quantum mechanical transmission between the two objects (Lan et al, 2008). Such theories usually assume an ideal interface between a nanotube and the metal contact, which in practice is frequently contaminated with different impurities. For calculations, usually the interface between graphene (flat graphitic monolayer) and metal is considered. Graphene, the building block for other graphitic materials such as the 3D graphite (stacked graphene planes), 1D carbon nanotubes (rolled graphene sheets), and 0D carbon buckyballs (wrapped graphene specks), consists of a flat monolayer of carbon atoms tightly packed in a two-dimensional honeycomb lattice. It is important to note that despite long known in the literature, with the electronic structure of graphite well established since the 40’s, it was the groundbreaking article of Novoselov et al., 2004, which brought up interest in graphene as a potential material for a number of applications. One of important applications is in microelectronics, where graphenes (in a form of nanotubes and, more recently, of few-layer graphites or FLG) with their highly unusual properties deriving from its two-dimensional geometry open new opportunities. Since then graphene has been intensively investigated both theoretically and experimentally as reviewed by Geim & Novoselov, 2007. Several experimental groups have focused on new field effect transistors where silicon is replaced by graphene as the channel material (Novoselov et al, 2004; Wang et al, 2008). These devices take advantage of graphene’s high and nearly temperature independent mobility of carriers leading to ballistic transport in the submicrometer scale, its linear I x V characteristics, and its unusually large sustainable currents (> 10^8 A/cm^2). To create a graphene-based transistor, graphene is typically deposited over some substrate, usually SiO_2 (Ishigami et al, 2007), or grown on top of some carbon-based substrate such as SiC (Berger et al, 2004). Because the substrate may alter graphenes electronic properties, these groups have investigated if and how this interaction happens, and the impact it causes (Akcoltekin et al, 2009).

Theoretical studies of single- and multi-layer graphene have employed the tight binding model which describes their band structures through the Dirac formalism (Castro Neto et al, 2009). In the presence of other chemical species such as dopants or adsorbates, or for graphene on substrates, under gate dielectrics, or on/under metal contacts, graphene may be structurally and/or electronically affected depending on the nature of the species involved. In this context, many-body effects such as electronic exchange and correlations may play an important role in describing correctly the band structure, requiring ab initio techniques for the simulation of such systems.

Density functional theory (DFT) is particularly suitable to simulate large systems, which is typical of graphene/substrate interface models (Zhou et al, 2007). DFT has been employed to investigate the interaction of graphene with metal contacts (Chan et al, 2008; Giovannetti et al, 2008; Ran et al, 2009) and other substrates. In the specific case of metal-graphene
contacts it is important to understand how the two materials interact at the interface, since this information will help to optimize device operation. For example, a large electrical contact resistance degrades device performance. Because good contacts are usually formed under chemical interaction, knowing the bond strength at these interfaces is crucial to the comprehension of the device transport characteristics. Here we mention the results of Chan et al., 2008, obtained with first principles DFT within the generalized gradient approximation (GGA), which show that ionic bonds are formed between graphene and metals from groups I-III, while covalent bonds are formed between graphene and transitional, noble, and group IV metals. Another study by Giovanetti et al, 2008, found that metal/graphene contacts can be divided in two groups (p and n) by observing the Fermi level change with respect to the Dirac point in the band structure. In a more recent study, Ran et al., 2009, claim, also based on first principles DFT calculations, that there exist two groups of metal/graphene contacts depending on the strength of the interaction between d-orbitals in metals and p_z orbitals in graphene.

In the first group (typical example: Ti) strong chemically bonded contacts are formed through the attractive interactions between the 3d electrons of the metal and the p_z states in graphene, while the second group (example: Au) comprises of weak physically bonded contacts. Both situations are essentially determined by the electronic configurations of the metals. The authors also perform transport simulations and their results suggest that metals which form chemical contact with graphene might be best as electrode materials in graphene-based electronics.

Resuming, theoretical studies have indicated that Ti contacts have lower resistances followed by Pd, Pt, Cu and Au (Matsuda et al, 2007), basically confirming the experimentally observed trends. However, it should be noted that the high reactivity of Ti may lead to its oxidation and distortion of a nanotube structure in the contact region.

Finally, theoretical simulations involving single- and multi-layer graphene in contact with different materials are an important tool to investigate these systems, helping to pave the way for the next generation of electronic devices.

In practice, determining the contact resistance is usually a very difficult task, and requires a great number of experiments to give statistically averaged results. An interesting approach to measure the contact properties between an individual multi-wall nanotube and thin metal layer has been recently developed by Lan et al, 2008. For this, sequential cuts by a focused ion beam (beam diameter of ~10 nm) in the area of contact (reducing the contact length) were utilized. Then, from the measured dependence of 2-terminal resistance on the contact length, both specific nanotube resistance and contact resistance can be evaluated. For PE-CVD grown MWCNTs with diameters in the range of 50-60 nm and thin Ag metal film deposited by evaporation, following parameters were obtained: 1) nanotube resistances ~ 4.5 kΩ/µm, 2) specific contact resistances \( r_c \) were shown to depend strongly on the thickness of the Ag film, being of 38 kΩ µm and 1.6 kΩ µm (i.e., 6.4 kΩ for 2 contacts of 0.5 µm length each) for Ag layers of 23 and 63 nm, respectively. From the relation \( r_c = \rho_c / (\pi d^2 / 2) \), where \( \rho_c \) is the specific contact resistivity for a nanotube of diameter \( d \), \( \rho_c \) values were determined: 35 and 1.3 μΩ cm^2, for 23 and 63 nm thick layers of Ag, respectively. In this case, the contribution of contact resistances (inversely proportional to its length) to a total 2 terminal resistance becomes insignificant for contact lengths exceeding 1 µm. Much higher values for thinner metal films are due to non-complete coverage of the nanotubes.
Note that in the measurements using the transfer length method (TLM) by Jackson & Graham, 2009, the specific contact resistance between a thin film single wall carbon nanotube electrode and a deposited silver contact were found to be considerably higher: 20 mΩ cm². The same method was used by Liu et al, 2008, but the test structures for TLM were produced using densified carbon nanotube strips formed from vertically-aligned CNT forests and various metal films. Contact resistances of Ti/CNT, Pd/CNT, Ta/CNT, and W/CNT contacts with the same nominal contact area were extracted to be 40, 49, 108, and 160 Ω, respectively. This corresponds to even higher specific contact resistivity values for the nominal contact area ~0.144 mm². The high resistivity is explained by the geometry of the experiments, where intertube tunneling is the main mechanism of lateral conduction. It is also argued that actual metal/CNT contact area can be much smaller than the nominal contact area, so that ρc values cannot be accurately calculated. These results show that much care should be taken while comparing data obtained using different methods and specific experimental conditions. Speaking more generally, considerable contributions still should be developed in the area of metrology of measurements involving nanostructured materials, in particular nanotubes.

6. MWCNTs for sensing applications

Another interesting application of carbon nanotubes is for gas sensing (Star et al, 2006; Zhao, et al, 2007). However, bare nanotubes do not show appreciable sensitivity to some gases, and recently demonstrated decoration of CNTs by nanoparticles (NPs) (Kong et al, 2001) sensitive to the gases of interest (electron-donating or electron-withdrawing) opened the way to CNT/NP based gas sensors with improved performance and wider area of applications. CNT/NP hybrid nanostructures can be selectively sensitive towards various species in a gas or vapor. Nanoparticles of metals like Pd, Al, Pt, Sn, Pd and Rh have been used to decorate CNTs, allowing selective detection of gases like H₂, NH₃, NO₂ (Kim et al, 2006), CH₄ (Lu et al, 2004), H₂S and CO (Star et al, 2006). CNT/NP based gas sensors in different configurations (e.g., CNT-FET, chemical resistors) can have extremely high selectivity due to high aspect ratio, fast time response and extremely low power consumption (μW range). Currently, considerable research efforts are concentrated on development of technologies (among them: electroless, sputtering, reflux, hydrolysis, super-critical CO₂ and others) capable to decorate both SWCNTs and MWCNTs with different metals and their oxides, selectively sensitive to different gases. Other nanostructured materials, metal oxide nanowires (NWs) have been recently implemented as gas sensing elements with high surface-to-volume ratios that allow for considerable improvement of sensitivity and reduction of response/recovery times and power consumption (to ~10⁻³ W, at typical bias of 5-10 V) (Kolmakov and Moskovits, 2004). Furthermore, in experiments with SnO₂ NWs, self-heating by Joule effect has been shown to provide local NW temperatures high enough (~200-300 °C, Prades et al, 2008) to avoid the use of external heating in gas sensing experiments. Note that an external heating (and high power consumption) is usually required for conventional sensors based on metal oxide thin films. However, two functions, sensing and self-heating, are coupled in the same element: the NW resistance can be changed significantly under exposure to the gas, in turn this will change the power dissipated on a NW and thus its temperature. This may result in non-linearities in the sensor response and requires appropriate calibration procedures.
CNT/NP hybrid structures represent other alternative of nano-scaled gas sensors that can operate at low voltage and power consumption (Gelamo et al, 2009). Depending on the type of nanotube, basically two different sensor configurations are currently under intensive studies: field effect transistors (FETs) and chemiresistors (CRs). FETs using semiconducting single-wall carbon nanotubes, have shown to be very sensitive to various gases however fabrication of these devices is technologically more challenging than those based on CRs. Thin films of mixed metallic and semiconducting SWCNTs deposited between arrays of interdigitated electrodes in a CR configuration, were shown to be very sensitive to gases like NO₂ and CH₄ (Lu et al, 2006). Room temperature methane detection was demonstrated for SWCNTs decorated with Pd clusters even at room temperature and a few mW power consumption (Lu et al, 2004). For multi-wall carbon nanotubes, a CR configuration has been studied (Meyyappan, 2005). In principle, the MWCNT based sensors must be less sensitive than those based on SWCNTs, as the measured current (and the associated noise) is supposed to pass through the whole volume of a MWCNT including all internal walls, whereas the reaction with gases should affect mainly the current fraction through the outermost wall. However, as discussed above, for distances shorter than ~1 μm, current redistribution between graphitic shells is small (Moshkalev et al, 2008), i.e., for side contacted MWCNTs and short gaps between electrodes the major fraction of current passes through the outermost wall. In terms of sensing configuration, this effectively transforms a short side-contacted MWCNT in a big-diameter “single-wall” metallic CNT, providing higher signal-to-noise ratio in gas sensing. Self-heating by Joule effect has been observed in nanotubes also, and so can be successfully employed in the case of CNTs based sensors, increasing sensitivity of hybrid CNT/NP systems to gases under interest, see below. Figure 3 shows some examples of MWCNTs decorated with SnO₂ nanoparticles for sensing applications, and Fig. 4 presents an individual MWCNT and MWCNT film deposited over metal electrodes by DEP and decorated by Ni (electroless) and SnO₂ (hydrolysis) nanoparticles, respectively.

Fig. 3. SEM (left) and TEM (right) images of MWCNTs decorated by SnO₂ nanoparticles.

Multi-wall carbon nanotubes decorated by Ti nanoparticles were used for gas (N₂, Ar, O₂) and pressure sensing at low temperatures (Gelamo et al, 2009). Chemiresistor sensor configurations with supported and suspended nanotubes were tested. For the latter, cuts between electrodes were produced by a focused ion beam before deposition of nanotubes by dielectrophorersis.
As can be seen in Fig. 5, two gas sensing mechanisms (chemical, for O\textsubscript{2}, and electrothermal, for chemically inert Ar and N\textsubscript{2}) were demonstrated. For the former, current decreases, and for the latter, increases during pulsed gas injection. The contributions of these mechanisms were shown to depend strongly on the CNT heat balance. The electrothermal mechanism is due to changes of the CNT electrical resistance (Kuo et al, 2007). Metallic MWCNTs can be self-heated considerably by current (in the way similar to NWs), and this leads to a rise of resistivity, with the temperature coefficient of resistivity (TCR) \( \sim 0.1\% \, ^\circ\text{C}^{-1} \) (Kawano et al, 2007). Further, when a gas is injected in the vacuum chamber, fast CNTs cooling by the gas may result in a measurable current increase. This effect was first observed by Kawano et al, 2007. For suspended nanotubes (and attached nanoparticles), heating by Joule effect is much stronger, resulting in strong enhancement of chemical sensitivity to gas (oxygen).

Finally, a CNT/NP hybrid material has been successfully applied for low-pressure gas sensing applications in chemical resistor configuration. In this configuration, multi-wall
carbon nanotubes serve as a conductive channel (for electrical signal acquisition), a heating element (for local heating of attached nanoparticles), and a substrate for NPs deposition (for selective gas sensitivity), whereas nanoparticles are employed to provide selective sensitivity to specific gases.

7. Conclusion

Many potential applications of carbon nanotubes in microelectronics are now being investigated extensively in many laboratories. Just a few specific applications are considered in the present work in more detail, showing some current problems and achievements. Some earlier expectations have failed, but many new opportunities arise constantly and, in many cases, unexpectedly. Recent introduction of new related nanocarbon material, graphene, is just one such example. For successful large-scale integration in new microdevices, development of reliable and compatible technologies that provide well controlled synthesis, positioning, characterization, manipulation and modification of nanotubes properties is still a great challenge.

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9. References


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The evolution of solid-state circuit technology has a long history within a relatively short period of time. This technology has lead to the modern information society that connects us and tools, a large market, and many types of products and applications. The solid-state circuit technology continuously evolves via breakthroughs and improvements every year. This book is devoted to review and present novel approaches for some of the main issues involved in this exciting and vigorous technology. The book is composed of 22 chapters, written by authors coming from 30 different institutions located in 12 different countries throughout the Americas, Asia and Europe. Thus, reflecting the wide international contribution to the book. The broad range of subjects presented in the book offers a general overview of the main issues in modern solid-state circuit technology. Furthermore, the book offers an in depth analysis on specific subjects for specialists. We believe the book is of great scientific and educational value for many readers. I am profoundly indebted to the support provided by all of those involved in the work. First and foremost I would like to acknowledge and thank the authors who worked hard and generously agreed to share their results and knowledge. Second I would like to express my gratitude to the Intech team that invited me to edit the book and give me their full support and a fruitful experience while working together to combine this book.

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