Atomic Layer Deposition of Aluminum Oxide Films for Carbon Nanotube Network Transistor Passivation

Kestutis Grigoras1, *, Marina Y. Zavodchikova2, Albert G. Nasibulin2, Esko I. Kauppinen2, Vladimir Ermolov3, and Sami Franssila1, 4

1Department of Micro and Nanosciences, Aalto University School of Science and Technology, P.O.Box 13500, FI-02015 TKK, Finland
2Nanomaterials Group, Department of Applied Physics and Center of New Materials, Aalto University School of Science and Technology, Espoo, Finland
3Nokia Research Center, Helsinki, Finland
4Department of Materials Science and Engineering, Aalto University School of Science and Technology, Espoo, Finland

Ultra-thin (2–5 nm thick) aluminum oxide layers were grown on non-functionalized individual single walled carbon nanotubes (SWCNT) and their bundles by atomic layer deposition (ALD) technique in order to investigate the mechanism of the coating process. Transmission electron microscopy (TEM) was used to examine the uniformity and conformality of the coatings grown at different temperatures (80 °C or 220 °C) and with different precursors for oxidation (water and ozone). We found that bundles of SWCNTs were coated continuously, but at the same time, bare individual nanotubes remained uncoated. The successful coating of bundles was explained by the formation of interstitial pores between the individual SWCNTs constituting the bundle, where the precursor molecules can adhere, initiating the layer growth. Thicker alumina layers (20–35 nm thick) were used for the coating of bottom-gated SWCNT-network based field effect transistors (FETs). ALD layers, grown at different conditions, were found to influence the performance of the SWCNT-network FETs: low temperature ALD layers caused the ambipolarity of the channel and pronounced n-type conduction, whereas high temperature ALD processes resulted in hysteresis suppression in the transfer characteristics of the SWCNT transistors and preserved p-type conduction. Fixed charges in the ALD layer have been considered as the main factor influencing the conduction change of the SWCNT network based transistors.

Keywords: Single Walled Carbon Nanotubes, Nanotube Networks, Atomic Layer Deposition, Field Effect Transistors, Conformal Coating, Passivation.

1. INTRODUCTION

Atomic layer deposition (ALD) offers excellent conformality and precise layer thickness control on the nanometer scale.1–4 This is based on the self-terminating reactions during each cycle of the growth.5 Application areas of the ALD process are rapidly extending, and the main drivers for that include the continuous dimensional downscaling of microelectronic devices, requirements for lower film growth temperatures (especially in the case of polymer substrates), and growing list of different materials obtainable by the ALD technique (oxides, nitrides, metals, and even polymers5).

Field-effect transistor (FET) based on carbon nanotubes (CNTs) have recently attracted large attention.6–7 This great interest is due to the unique properties of CNTs, especially high mobility of charge carriers in semiconducting nanotubes.8 However, tube to tube variations in chirality, position and orientation require rather complicated device fabrication steps, for example, an atomic force microscopy or high resolution electron scanning microscopy to identify the nanotube, then an electron beam lithography to pattern the contacts. Thus, sorting and placement of individual nanotubes still remain the main challenges for the application of individual CNTs in active devices. On the contrary, a network of randomly distributed CNTs enables one to attain reproducible electrical behaviour and uniformity by statistical averaging over a
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High gate switching efficiency in FET can be achieved by a strong gate coupling. A gate-all-around structure, where the gate is wrapped around the transistor channel, was shown to be the ideal geometry to maximize electrostatic gate control of the conducting channel and was successfully applied with the ultrathin body of an individual CNT. The ALD process exhibited to be the most suitable technique to obtain conformal coatings with a superior thickness control and uniformity. Coating of multiwalled CNTs by ALD is rather straightforward, but the surface of SWCNTs is known to be chemically inert to ALD precursor molecules, and continuous ALD coating cannot be achieved without a supporting substrate or without functionalization of the tubes. Recently, several functionalization techniques have been proposed, resulting in a successful ALD coating. Most of them have been applied for the coverage of a single nanotube and individual SWCNT transistors. In the case of a SWCNT network, the introduction of some type of functional layer, for example, deoxyribonucleic acid (DNA) as in Ref. [16], could affect the charge transport between the nanotubes resulting in deteriorated electric characteristics of the device and reduced carrier mobility. Furthermore, non-covalently adsorbed monolayer around the nanotube after its exposure to nitrogen dioxide and trimethylaluminium (TMA) vapor, which was shown to make the surface of SWCNTs susceptible to ALD precursors, could result in the growth of a coaxial aluminum oxide coating which is free to slide along the CNTs, revealing bare nanotube areas.

An important aspect of the operation of SWCNT-based FETs is stability. Passivation of the channel of bottom-gated SWCNT FET structures helps to stabilize its electrical characteristics against environmental variations. Environment is known to be one of the causes of hysteresis in the transfer characteristics of bottom-gated SWCNT FETs. Hysteresis in SWCNT FETs has been attributed to various charge-trapping mechanisms, such as adsorbed water molecules or oxygen from air in the vicinity of the SWCNT. Hysteresis is one of the major obstacles in the development of SWCNT based transistors. In earlier studies, chemical vapour deposited (CVD) silicon nitride and polymethylmethacrylate (PMMA) layer have been used for both passivation and hysteresis suppression. However, plasma deposition of nitrides can degrade the nanotube properties, and PMMA passivation does not protect properly from ambient humidity. ALD has been widely applied as a suitable technique for SWCNT FET passivation and top gate oxide deposition mainly due to conformality and uniformity of the obtained coating. In addition, the possibility to grow the coating layer at low temperature is important when polymer substrates are used. Herein, we explored the effect of the aluminum oxide coating by ALD at various conditions on the electrical performance of SWCNT-network based FETs.

Naturally the SWCNT FETs exhibit a p-type electronic character in ambient air which is explained by the presence of electron-accepting adsorbates that downshift the Fermi level towards the valence band, or large Schottky barriers at the metal–nanotube interface which prevent the injection of electrons, resulting in a dominant p-type behaviour. Therefore, by choosing a metal with a small work function, the n-type SWCNT FET can be obtained, as it was shown in Ref. [27] using Ca for contact electrodes. It was also shown that silanol groups present on the SiO2 surface are responsible for suppression of electron conduction in devices fabricated on SiO2/Si substrates. Interestingly, ambipolarity in SWCNT FETs has been observed under vacuum and with certain top gate oxides. Switching the channel conduction into n-type was also achieved by electrostatic doping, using a dual-gate device configuration, where the potential of the bottom gate was used to shift the Fermi level in SWCNT closer to the conduction band. Recently, it was shown that electrostatic doping due to positive fixed charges introduced by the ALD layer can lead to the reduction of the Schottky barrier thickness for electrons and cause n-type conductivity.

Majority of the research work focuses on the coating of individual SWCNTs. At the same time, the concept of random SWCNT network FETs is highly promising for low-cost larger scale production and is currently being widely investigated. Typically, bundles, consisting of a different number of individual SWCNTs, are forming the network. To our knowledge, research on the coating of SWCNT bundles by means of the ALD technique and its influence on the device operation is almost non-existent. In this study we have examined the possibility to overcome the inertness of the SWCNT surface by varying the growth temperatures and precursors for oxidation during the ALD process in the attempt to avoid the functionalization step. Moreover, the coating of thin SWCNT bundles (2–7 tubes/bundle) by means of the ALD at different growth conditions was investigated. The SWCNTs were obtained by means of direct deposition from the aerosol chemical vapour deposition (CVD) reactor. The conformality of ultra-thin ALD coatings (2–3 nm thick) was

number of individual tubes in the network. Moreover, CNT networks allow to circumvent the difficulty of differentiating between semiconducting and metallic nanotubes, since as-synthesized SWCNTs always contain a mixture of metallic and semiconducting tubes. Standard lithographic techniques are applicable for the SWCNT network based transistor fabrication. However, the electrical transport of SWCNT network based transistors is more challenging than that of individual SWCNT FETs due to the presence of multiple tube-to-tube contacts, with Schottky barriers formed at the junctions of semiconducting and metallic tubes as well as at the interface of the metal contacts.

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examined by TEM for two different precursors (water and ozone) and two different process temperatures (80 °C and 220 °C). SWCNT network based FETs were fabricated, and their passivation by thicker alumina layers (20–30 nm) was investigated for different ALD process conditions. The observed changes in the conduction type of ALD-coated SWCNT network based FETs are also discussed.

2. EXPERIMENTAL DETAILS

2.1. SWCNT Synthesis and Deposition

SWCNTs were synthesized by the aerosol CVD technique. Nanotubes were produced in the gas-phase via thermal vapour decomposition of ferrocene, forming catalyst nanoparticles. Carbon monoxide (CO) was utilized as a carbon source. The temperature in the reactor was kept at 1000 °C. The length of the grown individual nanotubes was about 300 nm. Previous studies showed that individual nanotubes entangle with each other in the cooling zone of the reactor, forming bundles (from 2 to 7 SWCNTs in a bundle). The growth process is described in more details in Ref. [36].

As-grown SWCNTs were collected from the gas phase by means of an electrostatic precipitator (EPS) at room temperature directly downstream of the synthesis reactor. The substrate was placed on the electrode inside the EPS biased at 6 kV. Due to spontaneous charging of SWCNTs and their bundles in the reactor, the high electric field improves the collection efficiency of SWCNTs on the substrate reducing the deposition time. The main advantage of this technique is the direct dry deposition at room temperature, allowing to avoid time-consuming liquid-containing transfer steps and to use heat-sensitive polymer substrates, which is impossible with the direct CNT growth approaches.

2.2. Atomic Layer Deposition

Aluminum oxide layer was grown in a Beneq TFS-500 reactor using TMA as a metal precursor and water or ozone as a precursor for oxidation. TMA-water is the most established ALD process, and aluminum oxide growth can be performed at low temperatures. The main reason for using ozone-based process is a higher activity of ozone as an oxidizer—important for growth on the inert surface of SWCNTs. Both ALD processes were carried out at 220 °C (standard temperature) and 80 °C (temperature suitable for most type of polymers). The pressure in the reactor was kept at about 4 Torr. Nitrogen was used as a carrier gas to transport precursor vapors to the sample surface and purge reaction gases from the reactor during each reaction half-cycle. 1 s precursor pulses and 3 s purge pulses (the same for both precursors) were used. The growth-per-cycle rate was calibrated by ellipsometer data obtained on the flat Si substrates and compared with layer thickness evaluation from TEM measurements.

2.3. Sample Preparation

For TEM observation a holey carbon film (400 Mesh Cu (50)) grids have been used. SWCNTs were collected on the grid using electrostatic precipitation technique. During the ALD coverage step, the grid with deposited nanotubes was carefully placed between the supporting silicon wafer pieces and gently fixed by a smaller silicon piece on top (to prevent from blowing away during the gas pulses). The silicon pieces contacted the grid only at the edges, and the central grid area remained open from both sides to ensure the ALD precursor and purge flows.

Bottom-gate FETs were fabricated on a highly boron doped Si substrates (resistivity 0.01 Ohm cm) with 100 nm thick thermal oxide layer. Therefore, the substrate acted as a bottom gate. A 200 nm thick aluminum layer was sputtered on the back-side to improve the contact to the gate. The transistor channel was formed by photoresist patterning, SWCNTs deposition and subsequent lift-off step. The density of the SWCNT network was controlled by the deposition time. The final lithography step defined the source and drain electrodes by means of photoresist patterning, metal evaporation (5 nm of titanium and 45 nm of gold) and lift-off in acetone. Afterwards, the devices were passivated with aluminum oxide layer grown by ALD.

Fig. 1. (a) Schematics of the SWCNT network FET fabrication: (1) lithographic patterning of photoresist (PR) on Si/SiO₂ substrate, (2) SWCNT network deposition, (3) lift-off in acetone, (4) Ti/Au evaporation and patterning. (b) SEM image of the SWCNT network covered with 25 nm ALD aluminum oxide.
SWCNT network FET main fabrication steps are shown in Figure 1 and described in details in Ref. [4].

2.4. Measurement Techniques

Uniformity of ultra-thin ALD coatings was examined by TEM (Philips CM200 FEG). The thickness of the ALD layers was calculated from the known growth per cycle value for the ALD of aluminum oxide and was also confirmed by ellipsometer measurements (PLASMOS SD 2300). Alumina passivation (20–35 nm) of FET channels was characterized with Zeiss SUPRA™ 40 field emission scanning electron microscope (SEM). An HP 4155A semiconductor parameter analyzer was used for the electrical measurements of the devices at ambient conditions.

3. RESULTS AND DISCUSSION

In order to follow precisely the ALD layer formation over the nanotube, we considered 2–5 nm thick coating to be optimal: thinner coatings have not been considered to act as good electrical insulating layers. Our SWCNT network contained both individual and bundled SWCNTs (with 2–7 tubes/bundle). The TEM images of SWCNTs coated with a thin aluminum oxide layer grown by the ALD process using water and ozone at temperatures 80 °C and 220 °C are shown in Figures 2 and 3, respectively. It is visible that the coating obtained for water-process ALD at low temperature, is rather poor (Fig. 2(a)). In this case, the grown aluminum oxide layer on both individual SWCNTs and bundles is not uniform, rough, revealing bare non-coated areas. On the other hand, increase of the temperature of ALD process results in a smooth and uniform coating of larger SWCNT bundles (Fig. 2(b)). However, the individual SWCNTs were found to be coated only at the open ends or close to the contact with another tube/bundle (even a short part of a small bundle (containing just 2 nanotubes) seems to be uncoated, as seen in Figure 2(b), but this special event was just an exception from many TEM observations). This confirms the inertness of the SWCNT surface to ALD precursor molecules.

From TEM images it is possible to evaluate the layer thickness of the ALD coating. The obtained value of 5 nm (for high temperature water-process) agrees well with the thickness values measured using an ellipsometer (on a Si substrate) and calculated from the growth-per-cycle rate. This indicates that the layer growth on SWCNT bundles starts from the first ALD cycles, without any incubation period, which is characteristic for a hydrogen-terminated silicon surface.39, 40 Uniform thickness of the layers grown by high temperature water-based ALD process confirms this “immediate” start: a delayed growth at some positions would reveal thinner coating of these areas, which is characteristic for the low temperature water-based ALD process.

Comparing the water and ozone ALD processes, it can be concluded that ozone’s oxidizing activity does not help to overcome the chemical inertness of the SWCNT surface: most areas of individual tubes were found to remain uncoated, both for low and high temperature ozone-based ALD processes (Fig. 3). This confirms that without functionalization the surface of individual SWCNT is not susceptible to ALD precursors, and in those cases where direct ALD coating was reported 14, 15—the layer was grown over the nanotube only with the assistance of the supporting substrate. Comparing the ALD ozone-processes performed at two different temperatures, it was observed that lower temperature growth resulted in a perfect coating of SWCNT bundles as processed at a higher temperature, similarly leaving open areas in the coating.
the individual SWCNTs in the bundle.\(^{41}\) The precursor molecules can pre-adsorb in the interstitial nanospaces, initiating the layer growth from there. With more nanotubes joined into a bundle, the relative part of their inert surface “open” to the ALD precursor becomes smaller, at the same time, the area of interstitial spaces increases, supporting the initiation of the layer growth. To our knowledge, this is the first attempt to evaluate and to explain the ALD coating of SWCNT bundles.

Further, we examined the effect of all the four types of aluminum oxide ALD coatings on the electrical performance of SWCNT network FETs. Since 2–5 nm thick alumina layers did not change the transfer characteristics of our devices significantly, we performed the device coating with 20–35 nm thick ALD aluminum oxide layers. Recently, we have shown that by increasing continuously the aluminum oxide layer thickness grown by ALD, the monotonic suppression of the hysteresis can

of individual SWCNTs (Figs. 3(a and b)). Thus, one can conclude that ozone is a more active precursor than water for the coating of SWCNT bundles, being therefore more suitable for the low temperature processing on polymer substrates with strong thermal limitations.

The principal difference in the ALD coating of bundles and individual nanotubes can be explained by their different surface conditions, with certain adsorbents formed on the SWCNT surface during the bundle formation in the synthesis process.\(^{37}\) The presence of adsorbents on the SWCNT surface after the bundle formation and their influence on the mechanism of the ALD layer growth is a subject of our current investigation. Another explanation for the higher activity of the bundle surface for the ALD precursors is the formation of interstitial pore spaces between

Fig. 3. SWCNTs coated with alumina by TMA + ozone ALD at (a) 80 °C and (b) 220 °C, with the thickness of 2.5 nm and 3 nm, respectively. The uncoated individual nanotubes are visible near the coated bundles.

Fig. 4. \(I_D - V_G\) characteristics of a SWCNT network FET prior to and after coating with alumina by water-ALD process performed at (a) 80 °C (channel length 20 \(\mu\)m, width 50 \(\mu\)m, \(V_{ds} = 1\) V) and (b) 220 °C (channel length 50 \(\mu\)m, width 50 \(\mu\)m, \(V_{ds} = 300\) mV). Low temperature coating causes ambipolar conduction, whereas high temperature ALD reduces hysteresis, preserving \(p\)-type transistor performance.
be achieved. The standard TMA-water ALD process at 220 °C was used in that study. Here, we investigate the influence of lower process temperature and different precursors for the oxidation (water and ozone) during the ALD aluminum oxide growth. The transfer characteristics of SWCNT network FETs have been measured before and after the channel passivation by ALD aluminum oxide, as shown in Figures 4 and 5. Sweeping the gate voltage from −10 V to +10 V and back, the characteristic hysteresis was observed. The influence of the ALD coating can be divided into two groups: high temperature and low temperature coatings.

Layers obtained at 220 °C by water-and ozone-processes resulted in reasonable hysteresis suppression, preserving p-type conductivity of the channel (Figs. 4(b) and 5(b)). This agrees well with our previous results. On the other hand, both coatings performed at 80 °C did not suppress the hysteresis significantly, but resulted in ambipolar electrical transport in SWCNT networks with the pronounced electron conduction (Figs. 4(a) and 5(a)). We assume that the main difference in our aluminum oxide layers grown at different temperatures of ALD reactor is the film quality and density of defects; lower temperature results in more defects. This statement was confirmed by the tests done separately for similar layers grown on silicon: resistance of the low temperature ALD layer against etching (for example, in buffer hydrofluoric acid, BHF) was an order of magnitude lower than that for the layer grown by high temperature ALD. TEM images revealed a rather good coating of SWCNT bundles using low temperature ozone-based ALD process in comparison with low temperature water-based ALD, as discussed earlier (Figs. 3(a and b)). This resulted in a 100% yield of ambipolar device characteristics after ozone-based ALD process, a phenomenon observed in earlier research works with a top gate oxide. However, only 64% of all the measured devices exhibited ambipolarity after low temperature water-based coating, with the remaining 36% being n-type transistors. As discussed in an earlier study, SWCNT transistors exhibit p-type conductivity at ambient conditions because of electron conductivity suppression due to adsorbed species and high temperature annealing in vacuum and subsequent passivation can recover n-type conductivity. The effect of the p- to n-type transition after the device encapsulation with alumina film grown by water-based ALD at 150 °C was also observed earlier for individual SWCNT-based FET operation, which was attributed to a change in the energy barriers at the CNT-metal contacts caused by oxygen desorption during annealing, similar to Ref. [31]. Oxygen removal during the pretreatment phase of the ALD process could be a potential mechanism for the observed conduction change. Although, in our case, high temperature ALD coating (with samples kept for about 1 hour at 220 °C in vacuum before starting the deposition) did not change the conduction type and, vice versa, ALD performed at low temperature and without any annealing resulted in an ambipolar electrical transport.

We believe that the electrostatic doping (ESD), suggested in earlier studies, could be the most possible explanation for the conduction changes in our devices. As reported earlier, gate modulation of the Schottky barriers formed between semiconducting and metallic tubes or metal contacts, as well as conduction through the semiconducting tubes themselves determine the switching mechanism of SWCNT-network FETs. Applying the ESD effect to the case of SWCNT networks, we suppose that the barriers for electrons or holes can be modified by the fixed charges in aluminum oxide layer, in a similar way as discussed in Ref. [15] where the change of the carrier type from p-type to n-type was explained due to the introduction of positive fixed charges during the deposition of gate insulator by ALD. With the reduction of the ALD process temperatures during the growth of amorphous aluminum oxide layers, the number of defects is

![Fig. 5.](image_url)
increased. It was shown that the average density of the grown film is decreased with temperature: from 3.0 g/cm$^3$ at 177 °C to 2.5 g/cm$^3$ at 33 °C. The n-type conductivity of the SWCNT-network FETs becomes more pronounced after low temperature coating (Figs. 4 and 5). We assume that the low temperature ALD layer contains positive fixed charges, possibly due to the large amount of hydrogen (reaching 15% at 80 °C). These charges could reduce the Schottky barrier thickness for electrons and increase the n-type conduction (as obtained in Ref. [15]). Another reason can be the excess amount of aluminum due to not completed oxidation reactions at low temperature ALD process. Thus aluminum can act as a dopant of the SWCNT network.

It was reported, that aluminum oxide layers grown by the ALD process at high temperatures usually contain a substantial amount of negative fixed charges. Negative charges are possibly due to residual OH-ions, or tetrahedrally coordinated Al. The fixed charge density can reach $10^{12}$–$10^{13}$ cm$^{-2}$ for about 20–30 nm thick layer. This negative charge can keep larger Schottky barrier for electrons, resulting in a p-type behaviour of our transistors coated with a high-temperature ALD layer. We can therefore assume that in the case of SWCNT networks coated by ALD the electrostatic doping is a prevailing phenomenon influencing the carrier type, as it was shown for individual SWCNTs. The n-type conductivity was not observable in devices with high temperature coating due to stronger effect of the negative fixed charge in the deposited layer. Our results on high temperature aluminium oxide coating agree well with the results obtained in Ref. [14] for SWCNT transistors with 15 nm thick alumina passivation at high temperature, where only the hysteresis reduction was observed without any changes in the conduction type. Authors of Ref. [14] emphasized the quality of their coating, with remarkably low interface trap densities.

We have also examined a much thicker aluminum oxide coating layers grown by high temperature water-based ALD process on the SWCNT network, dry deposited directly after the CNT synthesis reactor onto a Si/SiO$_2$ substrate. The cross sectional SEM picture presented in Figure 6 shows the SWCNT network, conformally coated by 145 nm of aluminum oxide. The “coaxial cables”, visible in the SEM image, are the coated nanotube bundles protruding from the oxidised silicon surface. From this image it is visible that a randomly distributed network of interpenetrating SWCNT bundles is a more challenging material for electrical transport studies in comparison with individual SWCNT devices.

4. CONCLUSIONS

We have investigated the ALD growth of aluminum oxide layers on non-functionalized individual SWCNT and their bundles, used to form networks of SWCNTs. Main attention was paid to the influence of different ALD process temperatures and different precursors for oxidation. We have found that due to a higher activity of ozone as a precursor, even the low temperature ALD process produced reliable coatings of SWCNT bundles, whereas the usage of water resulted in poor coatings at low temperature ALD. On the other hand, individual SWCNTs remained uncoated for all the ALD processes tested. It confirms that conformal coating of individual SWCNTs is impossible without initial functionalization step. The principal difference in layer growth on individual SWCNTs and their bundles has been explained by different surface conditions for the ALD precursor: the interstitial pores formed between the nanotubes in a bundle can act as the initial point for the precursor molecules to adhere and start the layer growth. To our knowledge, the ALD growth on small SWCNT bundles has not been investigated until now. This is an important issue in SWCNT-network transistors, where individual nanotubes are usually grouped in the bundles.

Using thicker aluminum coatings (20–35 nm) for the passivation of SWCNT network FETs we found that electrical characteristics such as hysteresis suppression and conductivity type depend on the quality of the grown film. Transistors with 20–25 nm thick low temperature ALD coatings exhibited mostly ambipolar conduction, where similar coatings grown at high temperature resulted mainly in reduction of the hysteresis, but preserving the p-type conductivity. This was valid for both water and ozone ALD processes. Thus, depending on the temperature and precursors used during the device passivation by the ALD process, the coatings can modify the gate characteristics of SWCNT network FETs, and the capability to control these changes can be highly beneficial for the construction of more complex electronic circuits.

The dependence of the obtained results on several ALD parameters and rather complicated explanation of the possible effects responsible for it means that more experimental data and even simulations are needed in order
to improve the understandings of the passivation mechanism of SWCNT network devices and its influence on the electrical characteristics of these devices.

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References and Notes


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