In order to fabricate novel infrared sensors, dense films of multi-wall carbon nanotubes have been grown on silicon and alumina substrates, using Plasma enhanced Chemical Vapour Deposition technique. The physical and optical characteristics of the devices were studied using a variety of methods. The carbon nanotube on silicon device showed a peak response at a wavelength of 1.02 μm. The infrared response of the carbon nanotube on alumina device showed that the detection behavior was consistent with a bolometric effect. Measurements have also revealed a wide spectral response from the visible to the long-wave infrared for that sample.

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1. Introduction

Infrared imaging capability is a major tool of modern defense organizations. It is a critical capability for many operations; from dismounted soldier portable systems to more complex airborne systems installed on unattended air vehicles. The different wavelength bands of interest for defense and security can be divided in the following way and are, to a large
of microwave power. After 10 min of H2 plasma pretreatment, the CH4 gas is then introduced into the chamber for the 30 minutes. The temperature is kept constant for another 10 min, before the ignition of the H2 plasma at 4 Torr and 900 W magnetron sputtering. A 99.5% pure TiN target is used and the Si substrates are heated to 500 [2].

activates the catalyst for the subsequent growth process [9]. To assure process reproducibility, the chamber is pumped to 0.72 mTorr. Deposition parameters are set to obtain typical catalyst layer thicknesses of 10 nm. Thicknesses are measured by ellipsometry. After the deposition of TiN and Ni layers, the resulting Ni/TiN/Si material is then transferred in air into the formation of NiSi [8] and then.

by ellipsometry. After the deposition of TiN and Ni layers, the resulting Ni/TiN/Si material is then transferred in air into the MPECVD reactor.

The 2.45 GHz microwave generator of the MPECVD system could be operated with the power up to 1.0 kW. The H2, CH4 and Ar process gases were introduced independently in the chamber through electronically controlled mass flow controllers. The substrates can be heated up to 800 °C and maintained at constant temperature.

In order to explore the possibility of using CNTs for photonic and bolometer type infrared detectors, two different test devices were fabricated. In this paper, we present results on the infrared response of those two prototypes.

2. Experimental details

2.1. Carbon nanotubes on silicon

The general experimental conditions for the growth of carbon nanotubes have been described previously [7], but we include it here with more details. We use prime grade N-type Sb doped Si substrates with (100) orientation in these experiments. The TiN interlayer has a dual functionality in our sensor structure. It acts as an electrode, which provides the electrical contact for the CNTs, and it also forms a protective barrier layer against diffusion of the Ni catalyst and formation of NiSi. This silicide formation occurs at temperatures above 300 °C [8]. The TiN layer is deposited by DC-magnetron sputtering. A 99.5% pure TiN target is used and the Si substrates are heated to 500 °C for the deposition in a reactive Nitrogen atmosphere at 1.2 mTorr. A TiN thickness of 100 nm is used in our experiments. The Ni catalyst layer is then deposited by RF-magnetron sputtering using a 99.9% pure Ni target at ambient temperature under Ar atmosphere at 0.72 mTorr. Deposition parameters are set to obtain typical catalyst layer thicknesses of 10 nm. Thicknesses are measured by ellipsometry. After the deposition of TiN and Ni layers, the resulting Ni/TiN/Si material is then transferred in air into the MPECVD reactor.

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We use a synthesis procedure that includes a H2 plasma pretreatment step. The pretreatment reduces NiOx and then activates the catalyst for the subsequent growth process [9]. To assure process reproducibility, the chamber is pumped to 10^-6 Torr prior to introducing the H2 gas. The substrates are gradually heated to the growth temperature of 700 °C in about 30 minutes. The temperature is kept constant for another 10 min, before the ignition of the H2 plasma at 4 Torr and 900 W of microwave power. After 10 min of H2 plasma pretreatment, the CH4 gas is then introduced into the chamber for the CNT synthesis at 900 W. The total pressure in the chamber is maintained at 5 Torr during the 20 minutes growth process. A CH4:H2 ratio of 1:4 is used. Ar refilling of the chamber is performed at the end of the growth process to cool the samples down to ambient temperature. It should be noted that the substrate temperature could increase during experiment, as a result of contact with the plasma. However, these variations are identical for all samples. Carbon nanotubes layer produced using this method are 10 μm thick, dense and vertically aligned. The nanotubes themselves are multi-wall with a diameter of about 20 nm.

In order to process the resulting film, the carbon nanotubes were encased into polyimide (PI-2737 from HD Microsystems). To achieve this, first, the polyimide solution is applied on the nanotube layer, enough to entirely cover the tubes. A soft-bake is then performed at 55 °C for 75 minutes. The sample is later cured at 350 °C for 60 minutes in nitrogen. After
those heat treatments, the polyimide is mechanically polished, exposing the tip of the nanotubes, before the deposition of a 10 nm thick gold electrode by vacuum evaporation. The gold film is deposited through a shadow mask, defining a 5 mm × 5 mm square. Fig. 1 presents the structure of the device.

2.2. Carbon nanotubes on alumina

The nanotube layers on alumina substrates are grown in similar conditions except that the TiN buffer layer and the polyimide are omitted. Also, the Ni catalyst layer is deposited through a 1 cm × 1 cm shadow mask, limiting the growth to a clearly defined area. The nanotubes grown on alumina did not show vertical alignment. Gold electrodes are also deposited by vacuum evaporation through a shadow mask. Contrary to the CNT on silicon device, the electrical contact geometry is parallel to the sample surface. Fig. 2 presents the structure of the device on alumina.

3. Results and discussion

3.1. Infrared response of the CNT on silicon sample

In order to perform infrared response measurements, the device is mounted in a dewar, under vacuum, and cooled to a temperature of 100 K. A ZnSe window enables the illumination of the sample. The illumination source is a wideband glowbar located 12 cm away from the device. Fig. 3 shows the I–V curve of the CNT on silicon device with and without illumination. It can be observed that the bias voltages for which the difference in current between the dark and illuminated conditions is most evident are located between 0 and −1 V. The rise time of the device is measured using a 1.04 μm laser source, a mechanical chopper and an oscilloscope. A value of about 1 ms is found.

The spectral response of the device is measured by using a tungsten lamp in a quartz bulb as the source of illumination. The light is modulated by a mechanical chopper and fed into an infrared monochromator. The device is located at the exit slit. The response of the device is measured by a lock-in amplifier, using the reference frequency of the mechanical chopper. Fig. 4 presents the spectral response of the device. It can be observed that the peak response occurs at about 1.02 μm. This is a similar result than the one observed by Straus et al. [10]. The peak detectivity of the device is evaluated to be equal to $5 \times 10^7$ cmHz$^{1/2}$/W. The cut-off wavelength is equal to 1.15 μm. The measured spectral response is analogous to the one of silicon. It is quite likely that the observed infrared response of the device is in fact the response of the substrate. The
Fig. 3. Current-voltage behavior of the CNT on silicon device at a temperature of 100 K with and without illumination.

Fig. 3. Comportement courant-voltage du dispositif à base de NTC sur silicium à une température de 100 K avec et sans illumination.

3.2. Infrared response of the CNT on alumina sample

Fig. 5 presents the temperature coefficient of resistance (TCR) as a function of the temperature of the sample. The absolute value of TCR is below 0.15% /K at a temperature of 300 K. If the sample is cooled to 100 K, the TCR increases to 0.42% /K. This is lower that what can be measured for YBCO films, which can have absolute TCR values in excess of 2.5%/K at room temperature [11].
The infrared response of the sample is also measured under vacuum and different temperatures using a wideband glow-bar. To examine the role of the TCR values on the infrared response, the variation in current induced by infrared illumination is plotted as a function of TCR (see Fig. 6). If all parameters stay constant, we would expect a linear relation if the detection mechanism was a bolometric effect. This is what is observed in Fig. 6 even though the linear relation is not perfect. The TCR is one important factor in the response of a bolometer, another crucial parameter is the thermal conduction of the active element with the substrate. This last parameter could also change with temperature, thus explaining some of the non-linearity. The fact that the detection mechanism is thermal is consistent with the long response time measured which is in the order of the second.

Using optical filters, it is found that the sample responds to radiation in the visible, mid-wave and long-wave infrared bands, which is expected since the CNT layer should have a broad absorption spectral range. Also, the overall detectivity is equal to about 6000 cmHz$^{1/2}$/W. Obviously, this is much lower than state-of-the-art VO$_2$ or amorphous silicon microbolometers arrays which have detectivity values in excess of $10^8$ cmHz$^{1/2}$/W, but it should not be seen as an upper limit on the performance of this approach since very little optimization is performed in this study.

4. Conclusions

Multi-wall carbon nanotube layers were grown on silicon and alumina to explore strategies for the fabrication and characterization of infrared sensors based on this material. A CNT on silicon device was successfully fabricated using conventional micro-fabrication methods. The I–V curve and the spectral response were also measured. For the carbon nanotube layer on alumina, the temperature coefficient of resistance of the nanotubes was evaluated. The infrared response was found to be consistent with a bolometric effect. Also, the device showed a wide spectral response. For future work, it would be...
interesting to produce vertically aligned layers of single-wall carbon nanotubes and characterize the infrared response of a device incorporating this material.

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