



Multi-walled carbon nanotubes as high temperature carbon monoxide sensors

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ARTICLE INFO

Article history:

Received 18 February 2008

Received in revised form 14 May 2008

Accepted 6 June 2008

Available online 12 June 2008

Keywords:

Combustion sensors

Harsh environment sensors

CO sensors

ABSTRACT

Heat treatment of acid-treated multi-walled carbon nanotubes (MWCNTs) produces profound changes in their electrical properties. Below 600 °C the resistance of a thick film of MWCNT (~100 μm) was below 200 Ω while at 700 °C, the resistance increased to over 20 MΩ. This process was irreversible. TEM showed that for a heat treatment ≤600 °C, the tube nature prevailed, but above 600 °C, nanoparticles of carbon materials with graphitic layers as well as tubes are present. The resistance changes upon interaction with carbon monoxide were monitored for materials heated at 600 and 700 °C. For materials heated at temperatures ≤600 °C, the largest changes in resistances (p-type) were observed at 400 °C with CO, with no measurable resistance changes at 100 and 600 °C. For materials heated to 700 °C, p-type resistance changes were observed for both CO and O₂ between 600 and 700 °C, with no changes at 800 °C, and background resistances approaching 95 MΩ at 500 °C. MWCNTs are demonstrated as potential materials for carbon monoxide sensing over the temperature range of 600–700 °C, but not very suitable for sensing between 100 and 400 °C, primarily because of the drift in the background.

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1. Introduction

The discovery of multi-walled carbon nanotubes (MWCNTs) [1] and single walled carbon nanotubes (SWCNTs) [2,3] has generated considerable interest due to their extraordinary physical, chemical, structural, mechanical and electronic properties [4–6]. They are being considered for a variety of potential applications, including hydrogen storage, chemical sensors, one-dimensional quantum wires, field emitters in display technology, nanoprobe in scanning and atomic force microscopies, electrodes for rechargeable batteries, resistors and interconnects. Depending on their chirality, diameter and functionalization, carbon nanotubes (CNTs) exhibit metallic, semi-metallic or semi-conducting properties [7–9].

CNTs without intentional doping behave as p-type semiconductors. The origin of the p-type character has been subject to a number of interpretations, including interaction with atmospheric oxygen [10] or metal electrodes [11] or due to impurities and defects introduced during synthesis or processing [12]. In the presence of oxygen, the p-type behavior has been proposed to arise from removal of electrons from the CNT by chemisorbed oxygen, thereby creating hole carriers [10].

CNTs possess remarkable adsorptive capacity due to their increased surface area and hence the interest in these materials for gas and vapor detection [13]. For instance, interactions with NH₃, NO₂ or O₂ and aromatic compounds have been shown to alter the electrical behavior of CNTs [10,13–18]. Vibrational spectroscopic studies on the interaction of NH₃ and NO₂ with SWCNTs were consistent with gas adsorption in interstitial channels in nanotube bundles [14].

There is considerable interest in development of high temperature CO sensors for optimization of combustion processes. Because of the thermal stability of MWCNTs, we explore here the use of such materials for high temperature sensing. The change in resistance of a thick film of acid-treated MWCNTs in the range of ambient to 700 °C is examined. At 700 °C, the material was irreversibly transformed into a high-temperature semiconductor, while heat treatment at 600 °C still maintained the metallic properties of the tubes. The resistance change with CO is best described by p-type semiconducting electronic properties. MWCNT heated to 600 °C exhibited resistance changes with CO between 200 and 500 °C and those heated to 700 °C showed sensitivity to oxygen and CO between 600 and 700 °C.

2. Experimental

2.1. Material preparation and characterization

Multi-walled CNTs (95+ % purity, OD = 40–70 nm, ID = 5–40 nm, length = 0.5–2 μm) were obtained from Aldrich. The as-received

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MWCNTs were heated in concentrated nitric acid under reflux for 5 h and washed with distilled water. The acid-free NTs were dried at 80 °C overnight and used for the resistance measurements.

2.2. Characterization

SEM micrographs were taken on a FEI Phillips XL 30 ESEM FEG. TEM micrographs were taken on a HR-TEM, FEI Tecnai™-20. The grid used for the TEM has carbon and Cu as received from supplier (PELCO® Support Films of Formvar, 200 mesh copper with ultra thin carbon). Raman spectra were recorded on a Renishaw™ inVia Raman Microscope using 514.5 nm argon ion laser and 50× objective.

2.3. Electrical measurements

The acid-treated MWCNTs were screen-printed on 15 mm × 10 mm planar interdigitated electrodes on alumina substrates (fabricated by Case Western Reserve University Electronics Design Laboratory, Cleveland, OH, USA). This device was then placed in an oven and heated from ambient to 700 °C and the resistance was monitored. In another set of experiments, the screen-printed MWCNT was heated to 600 °C for 3–4 h in a N₂ atmosphere, and then its resistance change with CO at temperatures of 100–600 °C was monitored. In a third set of experiments, the device was thermally annealed at 700 °C for 4 h in N₂ and then its resistance change with CO between 500 and 800 °C was examined. Acid-treated MWCNTs were also heated as a powder at 700 °C in nitrogen atmosphere for 10 h and then screen-printed on the alumina substrate and the resistance of the device in the presence of CO between 500 and 800 °C was examined. Both the 700 °C heat-treated materials exhibited similar resistance changes with CO.

The screen-printing ink was prepared as follows. About 50 ml absolute ethanol was mixed with 7.5 g V-801 glue and 2.5 g RV-507 solvent (Heraeus Circuit Materials Division, Conshohocken, PA, USA). Following this, 5 ml of the thoroughly mixed liquid was delivered to a vial containing 0.5 g dry MWCNT powder. The vial was sonicated to suspend the particles uniformly in the vehicle. Ethanol was evaporated from the samples in a vacuum oven at room temperature or with mild heating. The resulting paste was screen printed on alumina substrates with interdigitated gold electrodes. Screen specifications were selected to give a film thickness of ~0.1 mm. Press parameters were adjusted to give a uniformly smooth thick film with sharp edges and no obvious voids. After drying at 110 °C for 30 min, two Au wires were attached to the interdigitated wires and then placed in a quartz tube (4 cm diameter and 40 cm length). The electrical measurements were made with a HP multimeter data acquisition system interfaced with BenchLink software.

CO gas concentrations of 250, 500 and 750 ppm in a carrier gas N₂ were used. The N₂ source was a 99.998% cylinder obtained from Praxair. Resistance was also measured in varying oxygen levels: 0%, 2%, 5% and 10% balanced with the N₂ carrier gas. For each temperature regime, the device was allowed to equilibrate for 12 h before CO gas was introduced. The net gas flow was set at 100 cm³ min⁻¹ for all experiments.

3. Results

The MWCNT was subjected to acid treatment to disperse the fibers and introduce defects into the tubes [19]. Fig. 1 shows the change in resistance of acid-treated MWCNT from 100 to 700 °C in steps of 100 °C, each temperature being maintained for a 12-

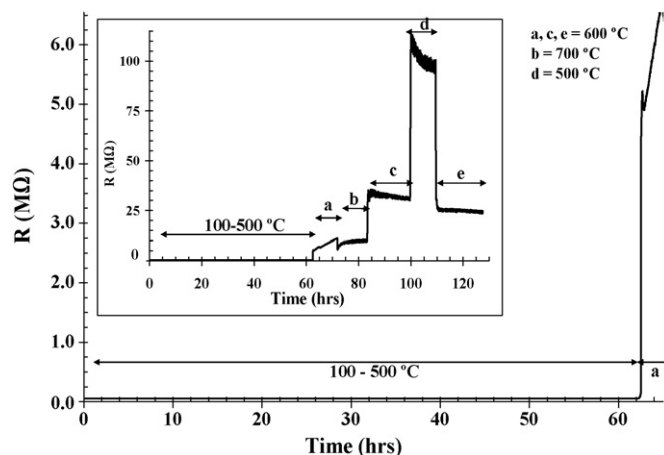


Fig. 1. Resistance versus time profile of material obtained from acid-treated MWCNTs that had been previously heat-treated at 500 °C for 4 h before exposure to various temperature regimes.

h interval. The device showed high electrical conductivity until 600 °C. However, at 600 °C, there was a steep rise in the baseline resistance, as depicted in the region labeled “a” in Fig. 1. The parts labeled “a” “c” and “e” are the baseline resistances of the material at 600 °C. It can be seen that resistances at “c” and “e” are significantly higher than the resistance in region “a”. Regions “c” and “e” were generated after the material had been heated to 700 °C (region “b”, insert in Fig. 1), suggesting that the material after exposure to 700 °C is clearly altered from the material in region “a”. Note that when the temperature was decreased to 500 °C (region d), the baseline resistance increased even further towards 100 MΩ. A further decrease in temperature resulted in a signal beyond the range of the multimeter. Thus, two materials were distinguished in this study, MWCNT-600, which was heated to 600 °C and MWCNT-700, which was heated to 700 °C and form the focus of the experiments.

3.1. Materials characterization

Fig. 2(a and b) shows the SEM images of the CNTs before and after acid treatment. The images indicate that the surface morphologies are similar except for a decrease in entanglement of the tubes in the acid-treated MWCNTs [19]. For the MWCNT-600, the electron microscopy images were similar to Fig. 2b, indicating no major morphological changes. For MWCNT-700, tubes as well as fragments of CNTs are seen, as depicted in the TEM images shown in Fig. 3. The elemental analysis showed that the MWCNT-700 contains carbon, nickel and oxygen (Fig. 3c). The nickel particles can be seen as randomly dispersed dark spots. The Cu arises from the TEM grid.

The Raman spectra of the as-received and MWCNT-700 are shown in Fig. 4. The “G” peak at 1580 cm⁻¹ is a high-frequency E_{2g} first-order mode that is characteristic of crystalline graphite. Contribution from a disorder-induced “D” peak can be seen around 1350 cm⁻¹ for the as-received MWCNTs. The D band is attributed to the presence of defects such as pentagons and heptagons in graphite or amorphous carbon. This disorder-induced peak is observed to increase as a result of heat treatment. The D/G ratio is 0.63 for the as-received samples and 0.97 for the MWCNT-700. It has been shown that defects introduced in CNTs increase the D/G band intensity ratio [20]. Also, an intense band related to two-phonon scattering (D*-band) at 2705 cm⁻¹ and a weak signal of three-phonon scattering (G*-band) around 2932 cm⁻¹ can be seen in the spectra.

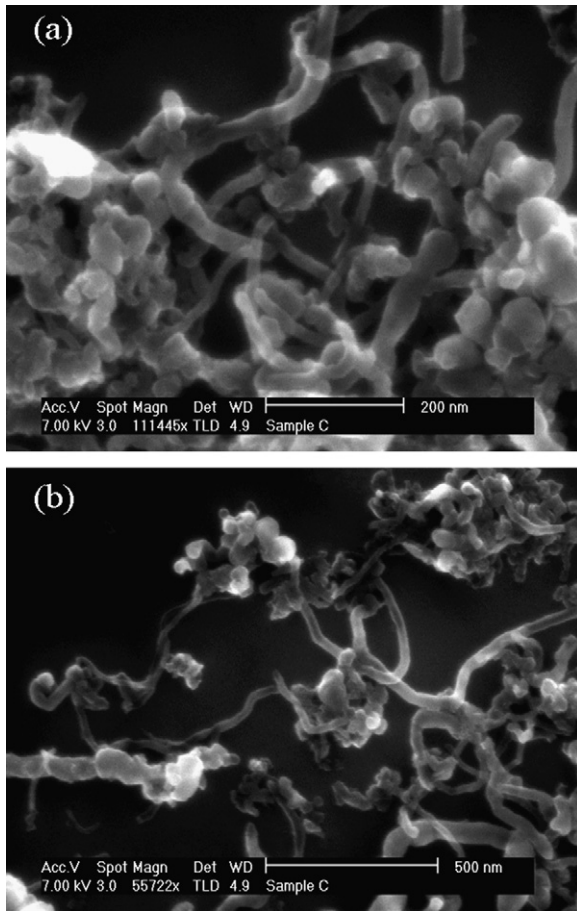


Fig. 2. Electron micrographs of as-received (a) and acid-treated (b) MWCNTs before heat treatment.

3.2. Resistance measurements with CO (100–600 °C)

The resistance of the MWCNT-600 films was measured between 100 and 600 °C while changing CO concentration from 0 to 500 ppm CO in a background of N₂. Fig. 5a shows that the material is non-responsive to CO at 100 °C. Fig. 5b shows a response in the presence of CO gas at 200 °C. A p-type semiconductor response to CO is observed, i.e., an increase in resistance in the presence of a reducing gas. In Fig. 5(c–e), the responses toward CO gas at 300, 400 and 500 °C, respectively are shown, with the response at 400 °C being the highest. Fig. 6 shows the response to repetitive cycles of 500 ppm CO at 400 °C. In the insert of Fig. 6, the data was obtained from the same sample after a 7-day heat treatment at 400 °C. In all of these measurements, a drift in the background towards higher resistances is observed with time. The data shown in Fig. 6 suggests that though the background increased from ~100 Ω to 30 MΩ after a 7-day heat treatment at 400 °C, the sensitivity to CO is still maintained. No response was observed at 600 °C (data not shown).

3.3. Resistance measurement with CO (500–800 °C)

Fig. 7(a–c) shows the resistance-time profile of MWCNT-700 at 600–800 °C for varying concentrations of CO gas in different oxygen partial pressures and in the presence of nitrogen. The sensors show p-type behavior for both O₂ and CO. The sensor response to CO at 600 and 700 °C increases with decreasing levels of O₂ in the background gas, the highest sensitivity as well as background stability being observed at 600 °C. The response and recovery time is

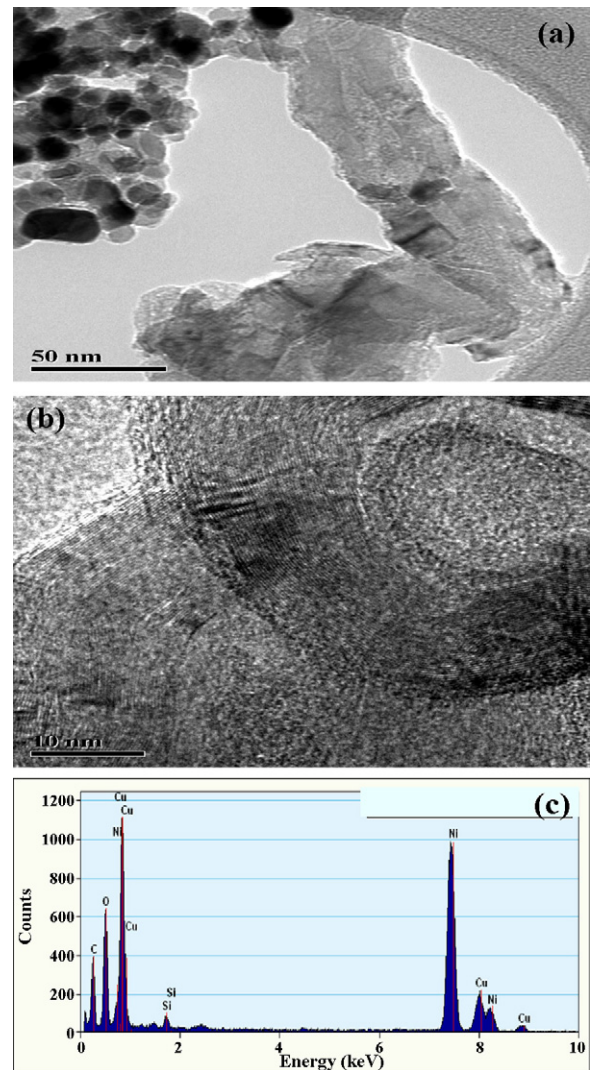


Fig. 3. (a and b) TEM images of materials obtained from MWCNT-700. (c) X-ray fluorescence of MWCNT-700.

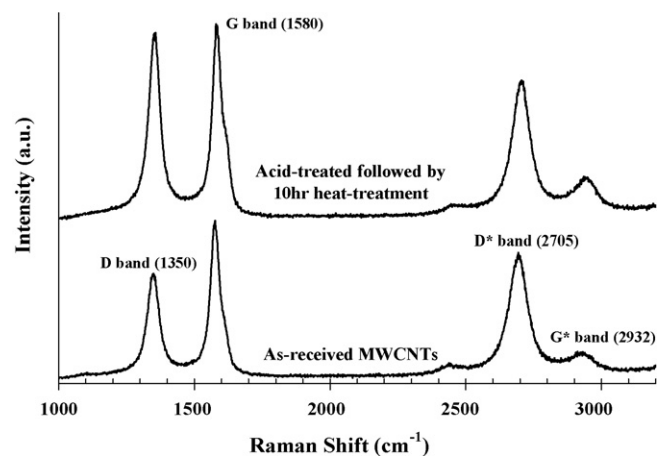


Fig. 4. Raman spectra of MWCNTs in as-received state and MWCNT-700.

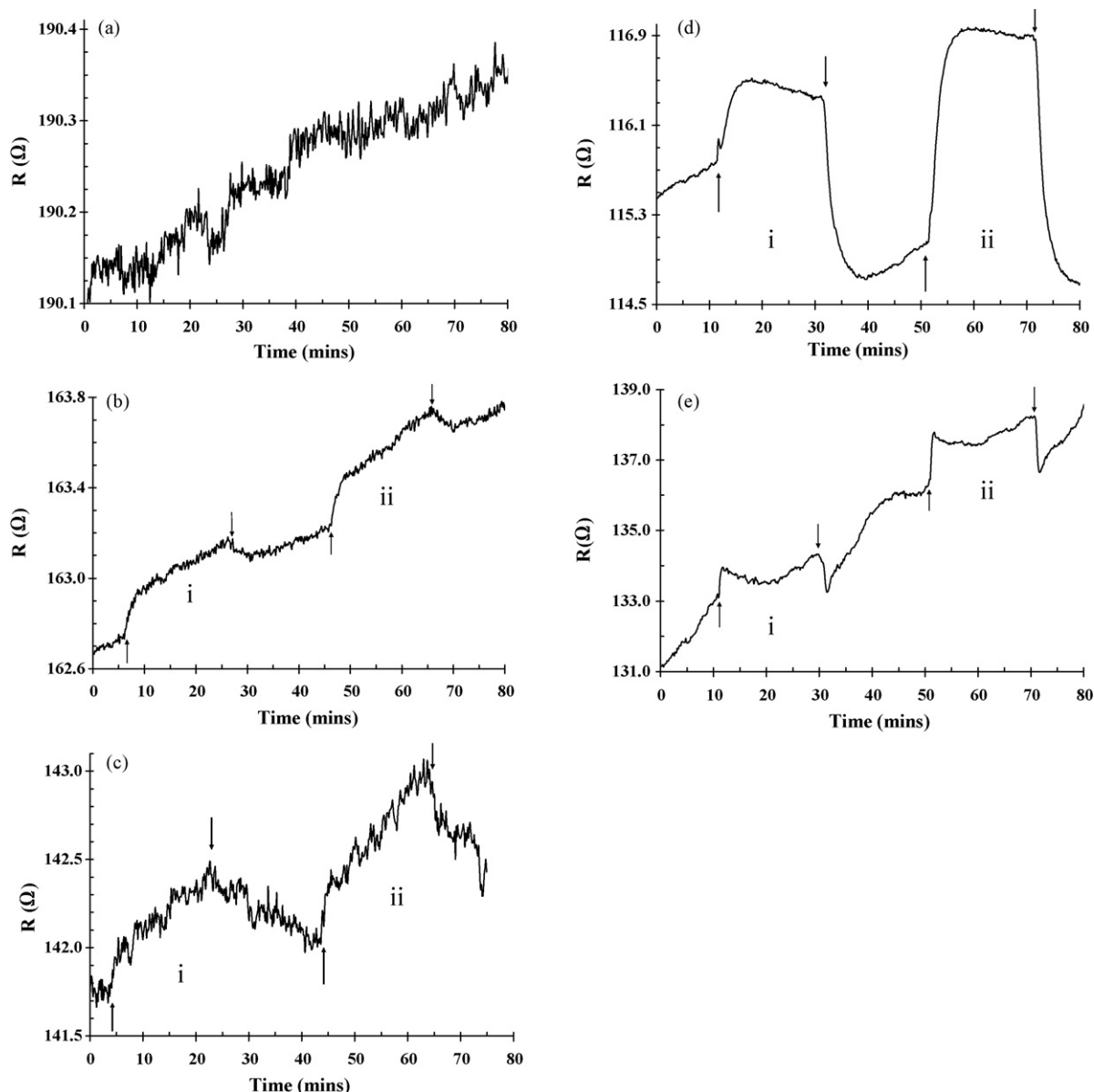


Fig. 5. Resistance versus time response of MWCNT-600 in the presence of CO at (a) 100 °C; (b) 200 °C; (c) 300 °C; (d) 400 °C; (e) 500 °C. \uparrow = CO on and \downarrow = CO off. [CO]/ppm: i = 250; ii = 500.

of the order of a minute and is limited by the time response of the sensing setup. Fig. 7c shows that no response is observed at 800 °C. However, the response to CO does recover upon cooling to 700 °C in 5% O₂ (Fig. 7d).

4. Discussion

There is a profound change in the electrical properties of MWCNTs upon heat treatment (Fig. 1). For samples heated to 600 °C, MWCNTs have remained mostly unchanged, both in morphology and its electrical properties as compared to the starting material. Upon heating to 700 °C, irreversible changes take place. MWCNT-600 remains relatively conducting between 100 and 600 °C, whereas MWCNT-700 is an insulator in this temperature range, and exhibits semiconducting properties at temperatures >600 °C.

Much is known about the electrical properties of MWCNTs, they tend to exhibit metallic properties at low temperatures, though as

synthesized, they are a combination of metallic and semiconductor forms [5,11]. SWNTs in the form of buckypapers exhibited a seven order of magnitude increase in resistance around ~400 °C [21]. Thus, heat treatment at 700 °C is leading to the loss of the metallic form.

Previous studies have shown that oxidation of defect-free graphite occurs at high temperatures [22], and if the graphite is defective, oxidation occurs at 600–700 °C [23]. Air annealing of CNTs show that at ~385 °C, amorphous carbon is oxidized and at higher temperatures, defect density increases [24]. Defect formation can include loss of shells [22], creation of oxygenated surfaces, including carboxylates, hydroxyl and carbonyl groups [24], and functionalization can destroy the inherent electrical conductivity of CNT [25]. Temperature induced polygonalization is also possible [24].

In this study, heating is carried out under a N₂ atmosphere, with trace levels of oxygen. The Raman spectrum of MWCNT-700 (Fig. 4b) shows increased intensity of the D band, indicating a higher

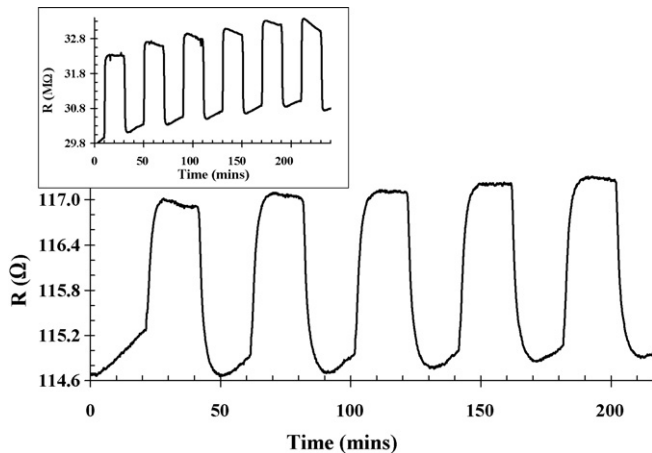


Fig. 6. Response to repetitive cycle of 500 ppm CO at 400 °C for MWCNT-600. Insert is the device response after being maintained at 400 °C for 1 week.

density of defects. The carboxylated CNT's will be decarboxylated at these temperatures and will lead to defects. Morphological changes that are brought upon by the 700 °C heat treatment include partial loss of the tube structure as well as formation of particles with graphitic rings (Fig. 3). Morphological studies have shown that oxidation can lead to loss of the pentagon caps on the tube [26]. The

defective tubes we observe in Fig. 3 have been noted before upon thermal treatment of B-doped CNT [27]. Upon oxidative annealing of CNTs, its increased sensitivity to different vapors has been reported [28].

For MWCNT-600, the samples exhibit high conductivity and an increase in resistance in the presence of CO. The CO response increases with temperature between 100 and 500 °C, with optimal response at 400 °C, and no response at 600 °C. This response to CO must arise from the semiconducting part of the MWCNT, and the p-type behavior observed is consistent with previous studies. Molecules, such as ammonia, an electron donor are also reported to cause an increase in resistance of SWNTs [12,14]. The p-type behavior of semiconducting MWCNT is proposed to arise from interaction with oxygen, with the defect sites acting as adsorption centers [5,6,18]. Oxygen adsorption would decrease the resistance of the MWCNT since it is an electron acceptor [16]. The change in resistance with CO will arise from reaction with the chemisorbed oxygen. With increasing temperature, oxygen chemisorption will decrease, though reactivity of CO with chemisorbed oxygen will decrease. Thus, an optimum temperature of 400 °C is observed for largest changes in resistance with CO (Figs. 5c and 6). Previous studies on gas sensing by CNT has focused on temperatures <200 °C, e.g. with NH₃ sensing was observed between ambient and 180 °C, with higher temperatures leading to lower sensitivities [5]. In this study, CO sensing falls off beyond 500 °C with the MWCNT-600. A rising background drift is noted in these measurements and is possibly

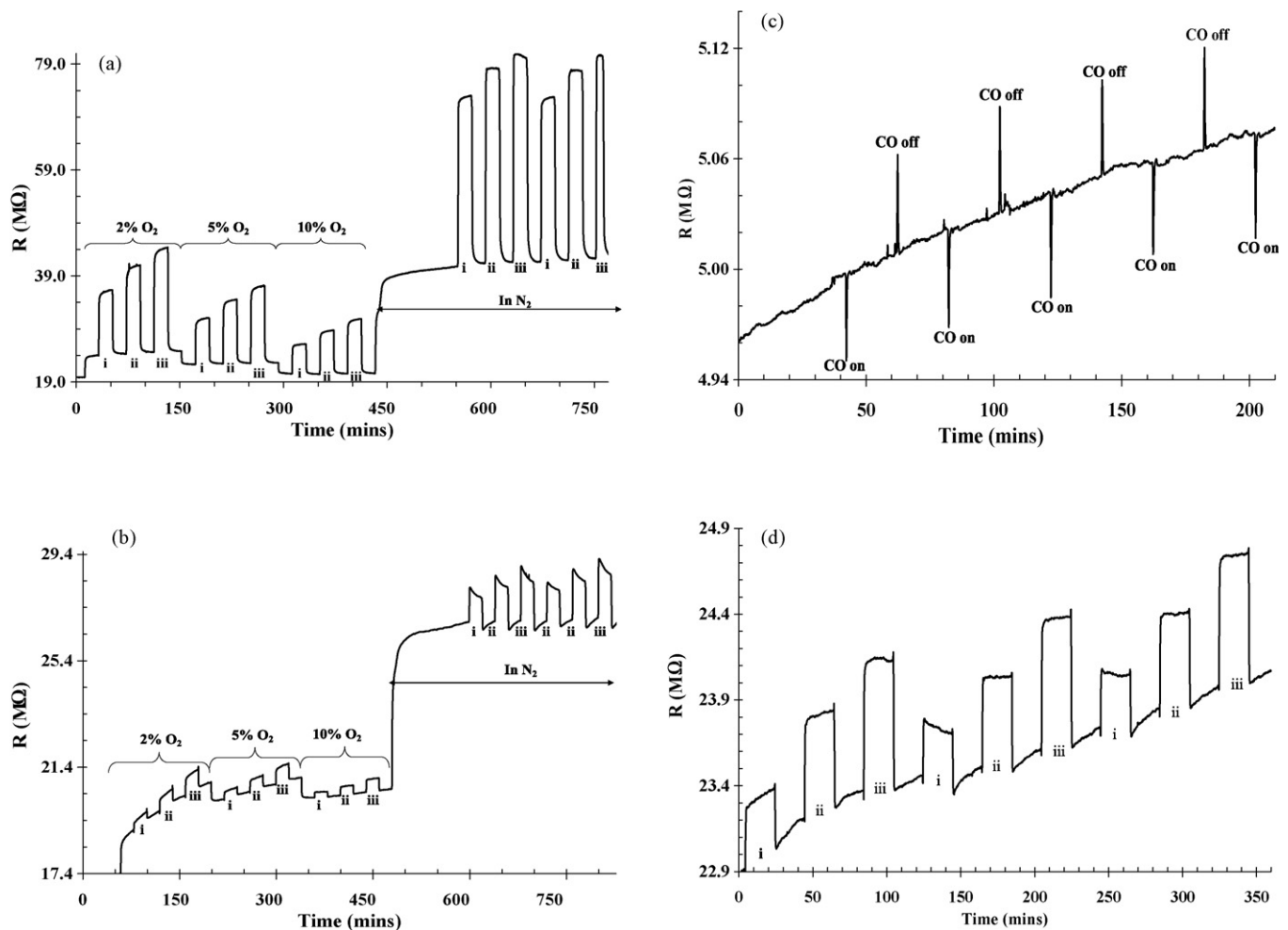


Fig. 7. (a) Response of MWCNT-700 at 600 °C. [CO]/ppm: i = 250; ii = 500; iii = 750. (b) Response of MWCNT-700 at 700 °C. [CO]/ppm: i = 250; ii = 500; iii = 750. (c) Response of MWCNT-700 at 800 °C. (d) Device response to varying concentrations of CO at 700 °C in 5% O₂ after it was tested at 800 °C. [CO]/ppm: i = 250; ii = 500 and iii = 750.

arising from loss of the metallic behavior with continued time of heating. The insert in Fig. 6 shows that the material heated at 400 °C for a week increases in background resistance by almost six orders of magnitude (due to loss in metallic character) and yet still exhibits resistance changes with CO.

MWCNT-700 exhibits markedly different resistance changes with CO. Measurable resistances were observed only at temperatures >600 °C, and baseline resistances were five to six orders of magnitude higher than MWCNT-600. At both 600 and 700 °C, MWCNT-700 exhibited p-type behavior with both O₂ and CO. No response was observed at 800 °C, though the properties recovered upon cooling to 700 °C. The mechanism for resistance changes with the higher temperature heated samples is similar to its counterpart at lower temperature, i.e. oxygen chemisorption on defects and reaction of CO with the chemisorbed oxygen. The defect sites in SWCNT have been shown to increase charge transfer by 1000% [29]. The background drift at 600 °C is minimal and sensor response and sensitivity are clearly in the range to be of practical interest.

This study demonstrates that thermal treatment of MWCNTs has the potential to generate CO sensors for high temperature applications, in particular at 600 °C. There are many low temperature CO sensors, but high temperature sensors necessary for optimization of combustion processes, especially under low oxygen levels are few and far between. Sensors that have been demonstrated to work under these conditions are TiO₂ [30] and Ga₂O₃ [31] and this study adds to that group in the form of thermally treated acid-modified MWCNT.

5. Conclusion

The heating of MWCNT at 700 °C leads to semiconducting materials and loss of metallic behavior. As-received MWCNTs after acid treatment contains a mixture of metallic and semiconducting tubes, which survive thermal treatment up to 600 °C. The semiconducting part of this MWCNT exhibits p-type electrical behavior and in temperature range of 200–500 °C behaves as a CO sensor in a background of N₂ gas, with optimum response at 400 °C. However, the drift in the background makes these sensors impractical. The material heated at 700 °C also exhibits p-type response, but CO sensing in N₂ and O₂ can be carried out between 600 and 700 °C. The defect structure of the material heated to 700 °C exhibits higher disorder, as measured by Raman spectroscopy. The MWCNT heated at 700 °C exhibits defective tube morphology, along with smaller fragments of graphitic material. Defects introduced by chemical and thermal process create site for oxygen adsorption, which provides avenue for the interaction with CO and the resultant change in the electrical signal of the MWCNTs thick film can serve as the basis for a gas sensing device, with optimal behavior at 600 °C.

Acknowledgements

We acknowledge funding from NASA and DOE for this research.

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Biographies

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