Study of field emission of multiwalled C nanotubes from arc discharge

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Keywords : Field emission, carbon nanotubes, arc discharge

ABSTRACT

Carbon nanotubes (CNT) were synthesized by DC thermal plasma method. After optimizing the synthesis parameters like the pressure, current etc., the synthesized products especially the anodic deposit was characterized by electron microscopies like Scanning Electron and Transmission Electron microscopies. The morphology of the product was ascertained to be multiwalled carbon nanotubes in large ratio. The diameters of these nanotubes were of the order of 30 – 50 nm and the lengths extending upto a micron and sometimes even 2 or 3 microns.

These nanotubes were then studied for its field emission properties. The CNTs were deposited on a metal stub which acted as the cathode. Care was taken to ensure complete covering of the stub to remove any possibility of emission from the metallic stub itself. The emission studies were performed in a stainless steel chamber under a dynamic vacuum in the range of 10^{-8} torr. The field emitted current was detected using a phosphor coated ITO (Indium tin oxide) glass. The phosphorous coating also helped in imaging the tips of the nanotubes. This was crucial in estimating accurately the emitting area and thus the field enhancement factor. The I-V curves for the field emission were recorded for various distances between the electrodes. These results will be shown in the presentation.

1 INTRODUCTION

Since its discovery in 1991 [1] carbon nanotubes are increasingly attracting both scientific and technological fields. This is basically due to its significant advantages over most of the existing materials' properties [2]. One dimensional nanostructures like nanotubes, nanowires etc. are considered to be ideal field emission electron sources due to their nanosize, high aspect ratio, low extracting field, high current density and probably long operating time [3]. In fact, carbon nanotubes based electron field emitters are projected as the next generation cold cathode materials for a variety of vacuum electronic devices [4]. Carbon nanotubes of various shapes and sizes have been used as field emission sources in several different devices, including flat-panel displays. The current emitted by a nanotube depends on the field strength at its free end which is stronger than the applied macroscopic field [5]. It is important that the field emission source has a low turn-on voltage as well as durable emission current [6]. In the recent past field emission properties of various materials like Diamond [7], Si [8], GaN [9], ZnO [10] and AlN [11] apart from carbon nanotubes have been studied.

There are many reports on the field emission properties of carbon nanotubes [12, 13, 14, 15] including the above references. However, we are reporting here the quantitative study of the dependence of the distance between the CNT and the anode to the turn-on field as well as the emitted current density.

2 EXPERIMENT

Carbon nanotubes (CNT) were synthesized by DC arc discharge between two graphite electrodes. A current of 100 A was applied under a DC voltage of ~20 V. The base pressure of the chamber was typically 4×10^{-4} mbar. The operating pressure was increased to 700 mbar after filling the chamber with He gas. The arc was run for duration of ~ 4 minutes and the chamber as well as the electrode holder was cooled by flowing water (for a comparison with carbon nanotube samples synthesized in our past activity, see [16, 17]).

After allowing the chamber to cool, the cathode was removed from its holder and the deposit at the edge of the cathode consisting of nanotubes was used as the sample for analysis as well as field emission studies. These samples were analyzed by scanning electron microscopy for study of the morphology.

The sample was then loaded into the field emission chamber and evacuated to a pressure typically of the range of 6 - 7 $\times10^{-8}$ torr using turbo pump with rotary pump backing. The field emission current was recorded for various applied voltages ranging from 0 V to 5 kV. An ITO

coated glass plate served as the anode/current collector. The coated glass plate has an intrinsic resistivity of 25 ohm-cm.

Carbon nanotubes were studied for its field emission properties in two different modes. Namely, directly inserting the cathodic deposit of diameter 6 mm (of which the CNT covered an area of approximately 4 mm diameter) and length of 5 mm which was affixed on an aluminum stub and this was loaded into the chamber. In the second mode a tungsten wire of diameter 0.2 mm was coated with the CNT and this was used as the emitting source (cathode).

The distance between the electrodes (CNT cathode and ITO coated conducting glass plate anode) were varied in the millimeter range (typically from 0.5 mm to 2.0 mm) and the corresponding emission current recorded. Experiments were performed to check the variation of the turn-on voltage against various distances between the electrodes. The emitted current was measured with Keithley 6485 picoammeter. The fluorescent image formed on the phosphor coated ITO glass plate anode was captured.

3 RESULTS AND DISCUSSION

The SEM images as shown in Figure 1 indicate that the CNT formed are quite dense with an average of >85% nanotubes (and the rest amorphous carbon). These nanotubes are found to have typical diameter of 30 – 50 nm and lengths varying between a few hundreds of nanometers to even a few microns. These are in fact found to consist mostly of MWNT and some quantity of SWNT bundles.

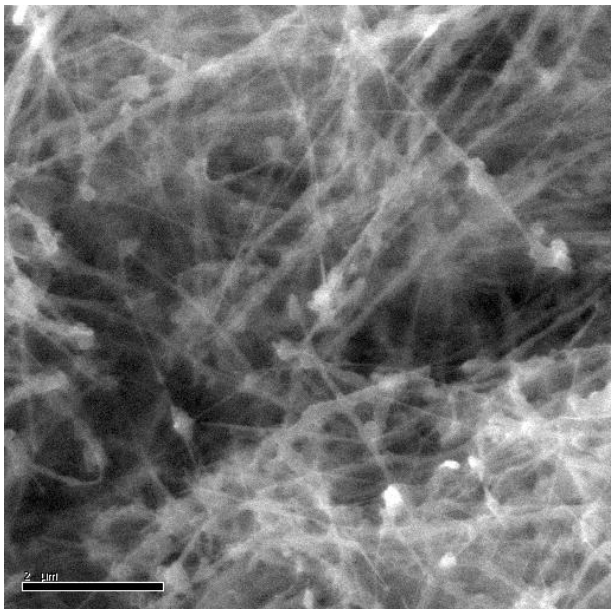

Fig. 1: SEM image of the CNT synthesized by DC arc discharge

For the directly loaded cathodic deposit the field emission current is seen to increase drastically (by more than six orders of magnitude) from a fraction of nano amperes to milli amperes for applied voltages of up to a few kV. As can be seen in the semi-log plot of Figure 2, the turn-on voltage also varies as the distance between the electrodes is increased. The turn-on voltage for an electrode distance of 0.5 mm is just a few tens of volts and this value increases to 2250 V for a distance of 2 mm. The increase in the electrode distance increases the turn-on field systematically. These results will help in estimating the ideal distances (for a specified electric field) of the CNT based field emitters for various applications.

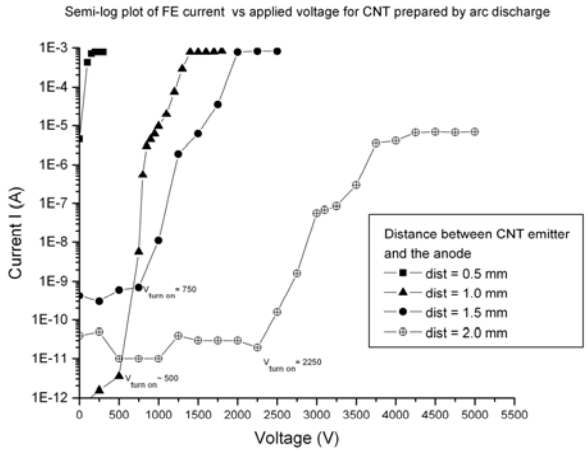

Fig. 2: Semi-log plot of Field emission current vs. applied DC voltage for various electrode distances

The turn-on field was estimated to be 500 V/ mm and a typical current density of 6.4 mA/cm^2 (for an applied electric field of 1350 V/mm) (assuming an emitting surface of 4mm diameter).

The phosphorescence glow of the anode due to the field emitted electrons is shown in Figures 3 and 4 (below). The emission glow threshold was observed to be in micro amperes (the glow was seen for emitted currents of micro amperes or more). Above the glow threshold it was noticed to be in pulses for lower applied voltages indicating possible charging and discharging of the nanotubes. This could be due to the nanotubes being semiconducting.

Also, when the applied voltage was increased to higher values (and the emitted current increases to tens of microamperes) the emission glow was continuous. The image in Figure 3 was captured at an emitted current of ~ 12 micro amperes and the glow at this juncture was in pulses. Rather, the image in Figure 4 was obtained when the emitted current was > 40 microamperes and the glow was continuous.

Fig. 3: Fluorescent image of the field emission electrons on the phosphor coated anode when the emitted current was ~ 12 micro amperes.

Fig. 4: Fluorescent image for higher applied voltage and the emitted current was recorded to be ~ 45 micro amperes

The sample was analyzed by SEM again after the field emission experiment was completed. Comparing the SEM images of Figure 5 with Figure 1, it can be seen that the density of the nanotubes remains same both before and after the field emission studies indicating that the nanotubes remain strongly adhered to the graphite surface. However a change in the dimensions of the nanotube was noticed. It was found that the diameter of the nanotube was much larger than it was before being used for the field emission experiment. It could be that single strands of the nanotubes gets adhered together to form bundles of MWNT under the influence of the electric field.

Notice that the use of tungsten wire yields an advantage for the measurement, since an emission current can be observed even at large electrode distances (up to 1 cm) for a lesser applied voltage. This in turn helps in obtaining a large magnification of the image formed on the phosphor coated glass plate.

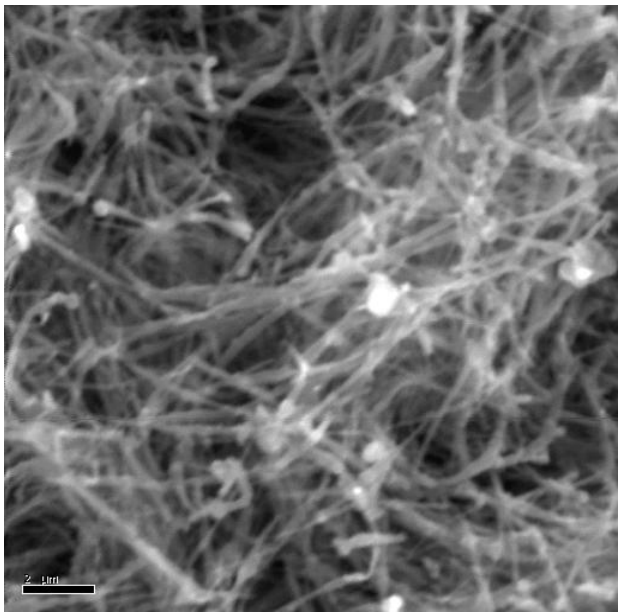

Fig. 5: SEM image of the same sample (as in Fig. 1) after its use in the field emission studies.

The emission occurs even at an electrode distance of 8 mm (*whereas in the previous case of CNT on aluminum stub there was no emission at a distance beyond 2mm for an applied voltage of up to 4 kV*). This is because the effective field is larger in a small wire as compared to the field from a large area cathodic deposit. The emitted current is low - a few nano amperes (see Figure 6). This is because the total number of nanotubes on the wire tip is much less than the number of nanotubes on the large surface of the cathodic deposit. However, this emitted current can be increased to some extent by increasing the applied voltage.

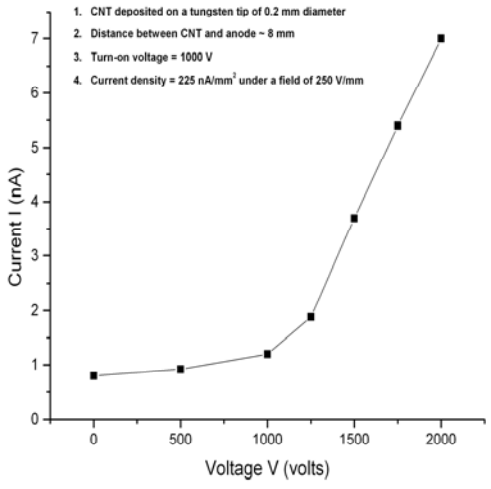

Fig. 6: Plot of field emission current vs. applied voltage from CNT deposited on a tungsten wire of diameter 0.2 mm.

The current density was estimated to be 2.22 mA/cm^2 under a field as low as 280 V/mm.

4 CONCLUSION

Multiwalled carbon nanotubes along with bundles of single walled nanotubes were synthesized by DC arc discharge. These were found to be of a few microns in length and diameters of 30 – 50 nm. The field emission studies were performed on these samples both by directly introducing the large area cathodic deposit as well as small area tungsten wire coated with the nanotubes. The emission studies gave a quantitative idea of the dependence of the emission current and the turn-on voltage as a function of CNT – current collector distance. The turn-on voltage increased from a few tens of volts at 0.5 mm electrode distance to 2250 V at 2.0 mm distance. It was also found that the nanotubes get bundled together under the influence of the electric field during the field emission experiment. The field emission from a carbon coated small area wire resulted in a lower turn-on voltage at larger distances of up to 8 mm.

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