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High Voltage Electrophoretic Deposition of Aligned Nanoforests for Scalable Nanomanufacturing of Electrochemical Energy Storage Devices

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Abstract. High voltage electrophoretic deposition (HVEPD) has been used to obtain forests of aligned multi-walled carbon nanotubes (MWCNTs) on long strips of flexible, conductive substrates. Successful design and integration of a continuous HVEPD setup has enabled scalable fabrication of electrodes for electrochemical energy storage. The mechanism of continuous HVEPD has been investigated to ensure appropriate alignment. Well-aligned forests of MWCNTs were obtained using a conductive holding layer which helped reduce internal resistance and enhance the electrochemical performance of the electrodes.

Introduction

Vertically-aligned one-dimensional (1D) nanomaterials (nanoforests) are desirable for electrochemical energy storage and conversion. Alignment can help to take full advantage of the excellent mechanical and electrical properties of 1D nanomaterials along their axis, while providing larger electrochemically active area and greater packing density [1-3]. Vertically aligned carbon nanotubes have been shown to have superior electrochemical properties than their randomly oriented counterparts [4-6] and have been investigated extensively as electrode materials for batteries and supercapacitors.

However, it is still a great challenge to obtain vertically-aligned nanoforest on large-area, flexible, conductive substrates. For example, there have been significant efforts to scale up the synthesis of aligned carbon nanotubes [7-9]. In spite of the limited success with catalytic vapor deposition methods [10], the high process temperatures have seriously limited their substrate compatibility, cost effectiveness and scalability. On the other hand, the post-growth alignment methods [11-13] are yet to achieve satisfactory alignment angle, density and scalability for electrochemical applications.

Electrophoretic deposition (EPD) is a simple, low-cost, room temperature technique with great promise for nanomanufacturing [14]. The deposition works in two steps: the charged particles in the colloidal dispersion first move towards the oppositely-charged electrode and then get deposited due to surface interactions [15, 16]. It is also known that a high voltage can be used during EPD to align carbon nanotubes [17-19]. It was shown in our previous work that high voltage EPD (HVEPD) can be used to obtain well-aligned MWCNT forests on conductive substrates by the co-deposition of a $Mg(OH)_2$ holding layer [5]. The ease of EPD and its capability to produce aligned structures shows great promise for the development of scalable processes to obtain large electrodes with aligned nanostructures.

In this work, we report a scalable fabrication setup to continuously print aligned MWCNT forests on long strips of flexible metal foils. It is also found that the use of a conductive holding layer would decrease the internal resistance and help improve the electrochemical performance of electrodes with aligned MWCNT networks. Alignment of MWCNTs using a conductive holding layer is first investigated on small substrates to find the optimal parameters following which large-scale production of nanoforest electrodes is demonstrated by using the continuous HVEPD method. Electrochemical testing is performed to compare the performance of samples with conductive and non-conductive holding layers and the long strip using the scalable setup.

Working Mechanism

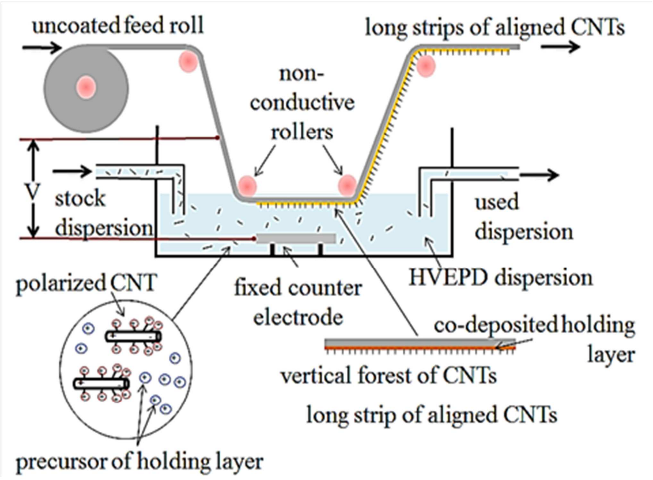


Fig. 1, Schematic of continuous HVEPD

As Fig. 1 shows, a setup was designed and fabricated to achieve continuous HVEPD of aligned MWCNT forests on long strips of flexible stainless steel (SS) sheets (Fig. 1). Flexible SS sheets (2.54 cm wide) were mounted on a roller and then fed through the continuous HVEPD setup. A DC motor was used to pull the long strips of these sheets at a speed of 2.54 cm/minute, so that every spot on the strip could get deposition for 1 minute, the optimal deposition time found in our previous work [5]. The non-conductive EPD vessel was filled with 100 ml of the EPD dispersion and two pumps were used to keep the concentration and volume of the EPD bath constant. The counter electrode (2.54 x 2.54 cm^2) was fixed at the bottom of the EPD vessel. The working electrode was held in tension by two rollers above the EPD bath and two rollers (2.54 cm apart) within the EPD bath. DC voltage was applied across the fixed and moving electrodes for the continuous HVEPD process and a pump flow rate of 8 ml/min was used to replenish the dispersion. The HVEPD process involves three key features; high voltage for alignment, low concentration of nanomaterials to avoid bundle formation and simultaneous deposition of a holding layer to maintain the alignment after the removal of the electric field [5]. It was important to make sure that the optimal parameters found to achieve well-aligned MWCNT forests on smaller substrates [5] were not disturbed during the continuous deposition process. To maintain the concentration of the EPD bath constant, a simple mass balance for the Ni salt and MWCNTs was used as shown below. The HVEPD deposited films on small SS substrates (2 X 2.54 cm^2) were found to consist of 0.1 mg Ni and 0.6 mg CNTs. The volume of EPD bath was maintained at 100 ml. For each minute, the mass balance was calculated as:

$$\text{Mass in deposit} + \text{Mass being pumped out} = \text{Mass required to be pumped in.} \qquad (1)$$

Aligned MWCNT forests were obtained using a Ni salt concentration of 0.035 mg/ml with a MWCNT concentration of 0.05 mg/ml. The concentration of the stock dispersion required to be pumped in was calculated to be 0.0465 mg/ml for Ni salt and 0.119 mg/ml for MWCNTs. Alignment of CNTs at high voltages takes place due to the dipole moment created by polarization of the nanotubes under the influence of the electric field [19]. The influence of fluid flow on the torque required for alignment was explored to check if the flow would impair alignment. We treated the flow between parallel plates as a plane Couette flow as shown in Fig. 2. The Reynolds numbers for the top and the bottom plates were calculated to be 4.468 and 0.584 respectively using:

$$\mathrm{Re} = \rho UL / \mu . \qquad (2)$$

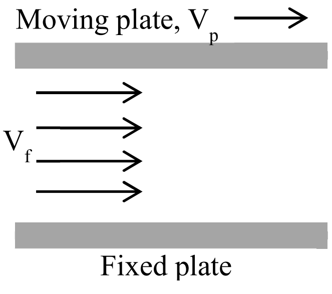


Fig. 2, Flow configuration

Here U is the free stream velocity given by: U = V_f for the bottom plate; U = $V_p + V_f$ for the top plate. Amongst other terms, ρ is the density of the fluid (IPA), L is the length of the plate, μ is the dynamic viscosity of the fluid (IPA). Such small Reynolds numbers indicate creeping flows for which the boundary layer thickness is considered to be infinite. This indicates that the flow between the parallel plates can be considered as plane Couette flow with a fully developed velocity profile, wherein the shear stress and the drag force remain constant [20]. The fluid flow would thus exert only a constant force yet no additional torque on the MWCNTs in the dispersion.

Experimental procedure

The MWCNTs (MER Corporation, Arizona) in this study were used as purchased with a diameter of 140 ± 30 nm and a length of 7 ± 5 µm. We previously reported using the HVEPD process to deposit aligned MWCNTs and β-MnO_2 nanorods along with a non-conductive holding layer [5]. The non-conductive holding layer, employed to hold the MWCNTs in the aligned position, caused high internal resistance and thereby impaired the electrochemical performance. In this work, we co-deposited Ni to serve as the conductive holding layer to reduce the internal resistance and achieve improved electrochemical performance.

HVEPD in still dispersion was first conducted to find the suitable concentrations of Ni salt at which good MWCNT alignment could be achieved, while serving as control experiments for continuous HVEPD. Stable dispersions of MWCNTs and Nickel chloride hexahydrate ($NiCl_2.6H_20$) in isopropyl alcohol (IPA) were prepared by sonication for 45 minutes. Parallel stainless steel electrodes (McMaster, Illinois), with an exposed area of 2 x 2.54 cm^2 and a gap of 0.7 cm, were used for the control experiments. A DC voltage of 150 V was applied for 1 minute using a high voltage power source (Stanford Research Systems, California) for the HVEPD process. Optimal parameters at which well-aligned MWCNT forests can be obtained were then found by SEM observation and checking the electric bridging effect [5]. The optimal concentrations for Ni and MWCNTs were found as 0.035 mg/ml and 0.05 mg/ml respectively and these parameters were then used to conduct HVEPD on gold coated Si wafers. The substrates were prepared in-house by sputtering 80-100 nm gold on Si wafers. The Si wafers were then cut and mounted on a vertical sample holder, to observe the cross-sectional view of the film deposited by HVEPD using a Hitachi S-4700 FESEM. HVEPD of MWCNT with non-conductive holding layer achieved in earlier work [5] was also conducted on such substrates to compare the cross-sectional views.

Continuous HVEPD was then conducted on flexible SS sheets (McMaster, Illinois) using the setup described in the working mechanism. The optimal HVEPD parameters used to achieve alignment were maintained for continuous deposition over a long strip. The long strips were then cut out into smaller strips for FESEM visualization and electrochemical testing.

Electrochemical testing was conducted using a Swagelok-type cell where two aligned MWCNT samples were sandwiched along with a filter paper (Whatman, New Jersey) separator. The Swagelok-type cell was then immersed in 2M KCl electrolyte. A potentiostat (Princeton Applied Research, Tennessee) was used to conduct cyclic voltammetry and galvanometric charge-discharge cycles. Cyclic voltammetry was conducted within a window of -0.5 V and 0.5 V with a voltage scan rate of 50 mV/sec. The galvanometric scans were conducted within 0 V and 1 V at a current of 2 mA. 200 cycles were run for SS substrates (2 x 2.54 cm^2) coated with aligned MWCNT forests (both non-conductive and conductive holding layers) in still dispersion. The final data was collected after cycle stability was reached. A cut-out section from the long strip, with Ni holding layer, was then tested the same way to check for any deviation in performance.

Results and discussions

Samples with good alignment results during HVEPD in a still dispersion were characterized by FESEM visualization and electrochemical testing before continuous HVEPD was conducted.

FESEM visualization. Fig. 3 shows the cross-sectional views of aligned MWCNT forests with non-conductive ($Mg(OH)_2$) and conductive (Ni) holding layers respectively. The alignment with the conductive holding layer was found to be similar to earlier alignment results [5] and well-aligned nanoforests with a conductive holding layer were seen in the FESEM images. The observed alignment was fairly uniform over the whole substrate. The cross-sectional views are at the center of the substrate to give a representative account of the alignment of the deposited MWCNT forests. Visualization at the center of the substrate was important to ensure that alignment was achieved across the entire substrate. The insets in Fig. 3 show magnified views of the aligned MWCNTs right where the Si wafer was cut.

Electrochemical testing. Cyclic voltammetry (CV) curves for the aligned MWCNT forests are shown in Fig. 4a. The CV curve for the aligned CNT forests with a non-conductive holding layer has a greater slope in the middle of the curve than when a conductive holding layer is employed. Also, the total area under the curve increases in case of a conductive holding layer. A smaller slope in the middle of the curve qualitatively indicates that the aligned MWCNT forests with the conductive holding layer have lower internal resistance and the larger area shows its capability for higher capacitance. This was quantitatively measured using the galvanometric charge-discharge cycles shown in Fig. 4b. The areal specific capacitance was calculated as:

$$\text{Sp. C} = I \,/\, A\ (\mathrm{d}v/\mathrm{d}t), \qquad (3)$$

where I is the constant current at which the cycles are run; A is the area of the electrode; dv is the drop in voltage (1 V); dt is the time taken for the discharge cycle. We also measured the IR drop, which is the sudden drop in voltage for one time step at the beginning of the discharge cycle to gauge the internal resistance.

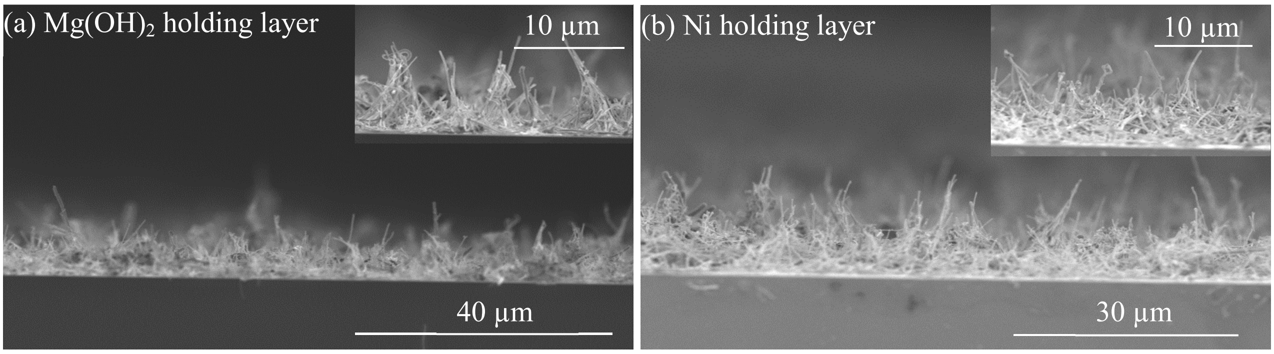


Fig. 3, FESEM images of aligned MWCNT forests achieved by HVEPD (a) with non-conductive (Mg(OH)$_2$) holding layer, (b) with conductive (Ni) holding layer.

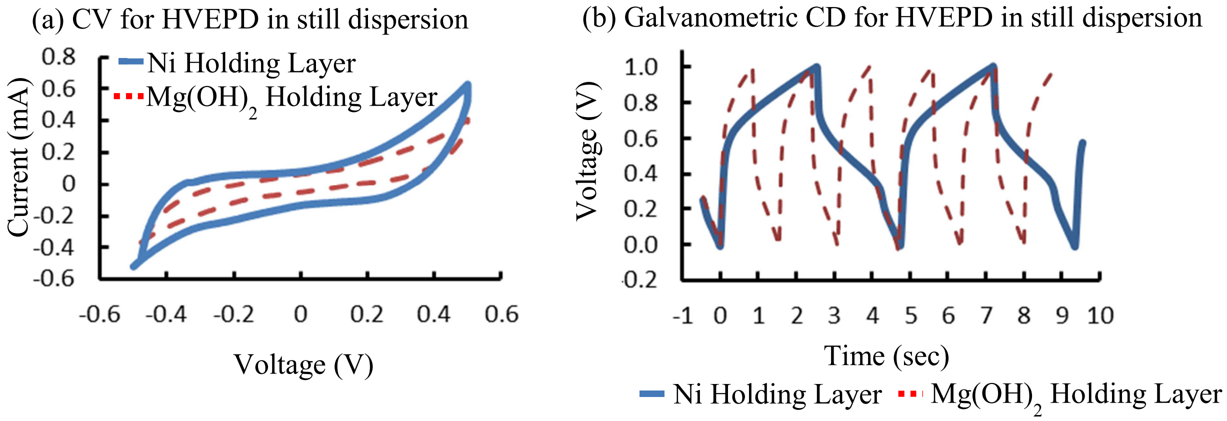


Fig. 4, (a) Cyclic voltammograms and (b) galvanometric charge-discharge curves for small SS substrates (2 x 2.54 cm^2) with aligned MWCNT forests.

The specific capacitance for the aligned MWCNT forests with the conductive holding layer was found to be around 846 µF/cm^2. This was significantly higher than the capacitance of the aligned MWCNT forests with the non-conductive holding layer, which was found to be 295 µF/cm^2, with an IR drop value of 0.479 V. On the other hand, the IR drop reduced to 0.249 V for the aligned MWCNT forests with the conductive holding layer indicating a lower internal resistance and a much higher capacitive performance.

FESEM visualization of continuous HVEPD sample on long strips. Alignment was achieved on a long strip of about 12 inches using the setup described earlier. The long strip was cut into smaller strips for imaging in the FESEM. Fig. 5 shows the long strip (with a ruler of 6 inch as reference) and the FESEM image of a section of the long strip. A tilted (8°) side view was observed to check for alignment. Well aligned MWCNT forests were found on different sections of the long strip indicating uniform deposition over a large length. In the initial stages of continuous HVEPD, the first inch does not receive a deposition of 1 minute and has sparse deposition as compared to the rest of the strip. As the strip moves along, from the second inch onwards, it receives a 1 minute deposition, resulting in uniform coating.

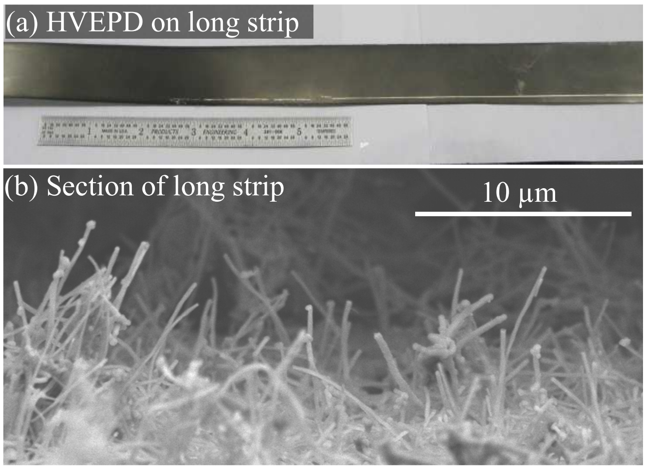
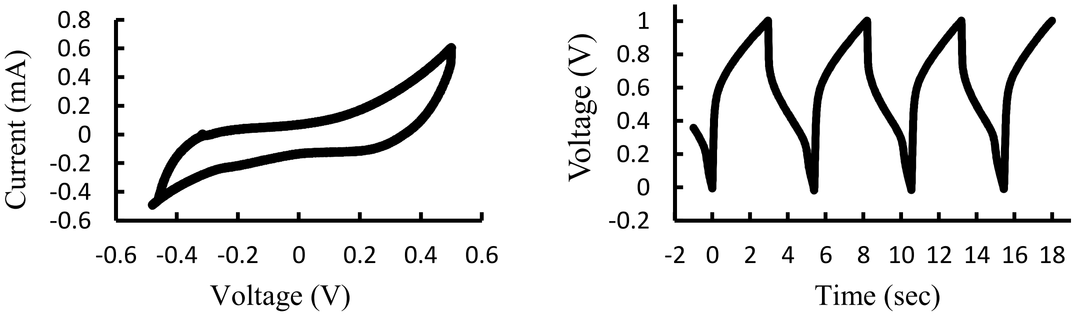


Fig. 5, Aligned MWCNT forests on a long strip

Electrochemical testing of continuous HVEPD sample on long strips. Cut-out sections of the long strip were also used to test the electrochemical performance of the aligned MWCNT forests. It was important to see that there was no loss of capacitance or increase of the internal resistance due to continuous deposition, as compared with the HVEPD with still dispersion. Fig. 6 shows the cyclic voltammograms and the galvanometric charge-discharge curves for sections of the long strip. Calculated values for specific capacitance and IR drop were found to be 847 µF/cm^2 and 0.252 V respectively, almost identical to that of the still HVEPD control samples (846 µF/cm^2 and 0.249 V respectively). The values were consistent over different sections of the strip, proving the uniformity of the continuous HVEPD process.


(a) CV for section of long strip

(b) Galvanometric CD for section of long strip

Fig. 6, (a) Cyclic voltammograms and (b) galvanometric charge-discharge curves for a section of long strip with aligned MWCNT forests

Conclusions

Scalable fabrication of long electrodes with aligned MWCNTs was successfully demonstrated. The nanoforests of MWCNTs, have been formed on long strips of flexible stainless steel sheets using a single step deposition process, namely, continuous HVEPD. Improved electrochemical performance of such electrodes was shown with the use of a conductive holding layer. Continuous feeding of stock solution has proven to be effective in stabilizing the deposition and provide uniform deposition. It has also been found that the motion of the strip and feeding flow does not disturb the alignment during continuous deposition. The electrochemical performance of continuous HVEPD samples is found to be almost identical to the still HVEPD control samples. In conclusion, continuous HVEPD has been shown as a promising method for low-cost, scalable production of high-performance electrodes for electrochemical energy storage devices.

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