UNIVERSITI PUTRA MALAYSIA

ASSEMBLY AND ALIGNMENT OF LONG CARBON NANOTUBE BRIDGES USING THICKNESS-CONTROLLED AIRFLOW-ASSISTED DIELECTROPHORESIS FOR SENSOR APPLICATIONS

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By

ABDULLAH ABDULHAMEED GUMAAN ABDULHAMEED

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This work is dedicated to my beloved parents, wife, daughter, siblings, family and friends for their love and support. This work is also dedicated to the fond memories of Dr. Nurul Amziah Mohd Yunus (1975-2019).

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

ASSEMBLY AND ALIGNMENT OF LONG CARBON NANOTUBE BRIDGES USING THICKNESS-CONTROLLED AIRFLOW-ASSISTED DIELECTROPHORESIS FOR SENSOR APPLICATIONS

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December 2021

Chair : Izhal Abdul Halin, PhD
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Carbon nanotubes (CNTs) have recently attracted significant interest due to their unique combination of properties, including high mechanical strength, distinct optical characteristics, and high thermal and electrical conductivity, which make them suitable for a wide range of applications. In order to apply CNT in devices, they have to be aligned and placed precisely in a location within the device. Examples of such devices are sensors, biosensors and transistors. In sensor devices, the aligned CNT transport the charge carriers faster than random CNT, which amplifies the sensor response.

Currently, the main challenge in manufacturing CNT-based devices is the inability to precisely control the CNT placement in the desired location. Dielectrophoresis (DEP) is a method that can be applied to assemble and align CNT across electrode structures with great precision. However, the DEP method is limited to small-scale fabrication and fails to assemble CNTs across wide electrode gaps (>50µm) due to the presence of the Electric Double Layer (EDL) and Joule Heating effects. These effects cause medium drag velocity, which moves the CNTs away from the deposition area. Furthermore, there are difficulties in maintaining the alignment quality of long CNT bridges during the fabrication process, which reduces the reproducibility in manufacturing CNT-based devices.

In this thesis, a new DEP setup is introduced that is able to minimize the medium drag velocity and preserve the quality of the aligned CNT bridges during assembly and alignment. The setup is based on the standard DEP setup with two mean differences: first is the ability to control the channel height through a top glass cover to minimize the medium drag velocity caused by Joule Heating

and EDL effects, and second is the use of hot airflow to preserve the alignment quality during the drying process. In addition to these two modifications, a computational framework based on the Finite Element Method (FEM) is employed to find the ideal combination of the signal parameters and medium properties that result in minimum Joule Heating and EDL effects.

Experimental work focusing on fabrication of electrode structures, preparation of CNT suspensions and design controllable signal supply system was carried out to prove that the effects of Joule Heating and EDL can be minimized to allow for a longer alignment of CNTs. The proposed system is able to align CNT bridges with lengths up to 125μm when using a sine wave signal of 20 volts peak to peak and 2.5MHz. The aligned CNT bridges are formed across Interdigitated Electrodes (IDE) made of Indium Tin Oxide (ITO) material because it is suitable for transparent sensors. We also fabricated three sensor devices: pH sensor, Hydrogen sensor, and temperature sensor using the new DEP setup. The response of the aligned CNT bridges towards the pH value, temperature variation, and Hydrogen molecules is measured and analyzed. The sensitivity of the aligned CNT toward the pH value of a solution is between 170% (pH=4) to 2000% (pH=10) and response times between 4 to 6 seconds. The temperature coefficient of the aligned CNT bridges after the assembly is -0.19572%, where the resistance increased from 550Ω to 575Ω when the temperature is reduced from 70^{o}C to 30^{o}C. The aligned CNT bridges are also exposed to Hydrogen gas at different concentrations. The sensitivity of the Hydrogen sensor is different based on the functional groups attached to the CNTs and the gas concentration.

The experimental results show that the newly proposed DEP setups are able to precisely align CNTs more than 50% longer than the conventional DEP setup, and the produced CNT bridges are suitable for use in the fabrication of electronic devices.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMASANGAN DAN PENJAJARAN JEJAMBAT TIUB NANO KARBON PANJANG MENGGUNAKAN KAWALAN KETEBALAN DENGAN BANTUAN ALIRAN UDARA DIEKTROFORISIS UNTUK PENGHASILAN PENDERIA

Oleh

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Karbon Nantotiub telah menarik minat yang penting kebelakangan ini disebabkan oleh terdapatnya ciri-ciri kombinasi yang unik, termasuklah tinggi kekuatan mekanik, ciri-ciri optik yang berbeza, dan tinggi daya pengkonduksian terma dan elektrik, yang membuatkan ia sesuai untuk digunakan dalam aplikasi yang pelbagai. Untuk menggunakan CNT, ianya perlu dijajarkan dan diletakkan dengan tepat pada lokasi tertentu dalam sesebuah peranti.Contoh peranti-peranti tersebut ialah kepelbagaian jenis penderia electronik, penderia-bio dan transistor.

Buat masa ini, cabaran utama yang dihadapi untuk membuat peranti-peranti ini ialah kesukaran untuk mengawal dengan jitu penempatan CNT pada lokasi yang tepat sambil CNT tersebut dijajarkan dengan susunan yang panjang. Dielektroforosis (DEP) ialah kaedah yang terbukti untuk Menyusun dan menjajarkan CNT di antara sepasang elektrod dengan tepat. Walaubagaimanapun, kaedah DEP terhad kepada penyusunan CNT yang agak pendek (>50μm) kerana kehadhiran kesan Elektrik Dwi-Lapisan (EDL) dan Pemanasa Joule. Kesan-kesan ini menyebabkan halaju seret bahan, iaitu suatu pergerakan medium yang menggerakkan CNT dari Kawasan deposit yang dikehendaki.

Dalam tesis ini, sebuah radas DEP yang baharu diperkenalkan. Radas ini mampu mengurangkan halaju seret bahan, oleh itu meningkatkan kualiti jejambat CNT yang dibina sewaktu proses pemasangan dan penjajaran. Radas yang dicadangkan adalah berdasarkan radas DEP piawai tetapi mempunyai dua perbezaan utama. Perbezaan pertama adalah kebolehan mengawal ketinggian medium dengan kehadiran sebuah penutup kaca yang dapat mengurangkan

kesan halaju seret bahan yang disebabkan oleh Pemanasan Joule dan EDL. Pembaharuan kedua ialah penggunaan udara panas untuk mengekalkan kualiti penjajaran sewaktu proses pengeringan. Sebagai tambahan terhadap kedua-dua modifikasi ini, kedua-dua modifikasi ini dapat dibuat keatas radas penajajaran CNT berasaskan DEP hasil dari kerangka pengiraan bersandarkan kaedah elemen finit (FEM) untuk mendapatkan kombinasi yang ideal terbaik parameter isyarat dan medium agar kesan Pemanasan Joule dapat dikurangkan.

Kerja-kerja eksperimen yang tertumpu kepada pembikinan elektrod, penyedian ampaian CNT dan rekabentuk sistem litar pengeluar isyarat dilaksanakan untuk membuktikan bahawa kesan Pemanasan Joule dan EDL dapat dikurangkan agar penjajaran CNT yang lebih Panjang berhasil. Sistem yang dicadangkan mampu Menyusun CNT sepanjang 125μm dengan penggunaan isyarat sinus dengan ayunan 20V pada kekerapan 2.5MHz. Jejembat-jejambat CNT dihasilkan diantara elektrod bergerigi (IDE) yang dibuat dari Oxida Timah Indium (ITO) kerana ianya sesuai untuk pembikinan peranti lutsinar. Kami juga menghasilkan tiga buah peranti iaitu peranti pH, peranti gas Hidrogen dan peranti suhu menggunakan radas DEP baharu tersebut. Respon peranti-peranti tersebut terhadap nilai pH, kehadhiran molekuln Hidrogen dan keamatan suhu direkod dan dianalisa. Hasilnya menunjukkan kepekaan CNT yang sejajar terhadap nilai pH larutan adalah antara 170% (pH = 4) hingga 2000% (pH = 10) dan masa tindak balas antara 4 hingga 6 saat.. Koefisien suhu daripada penjajaran jejambat-jejambat CNT selepas himpunan ialah - 0.19572%, yang mana rintangan menaik daripada 550Ω kepada 575Ω apabila suhu dikurangkan daripada 70^{o}C kepada 30^{o}C. Penjajaran jejambat-jejambat CNT juga didedahkan kepada gas hidrogen dengan nilai kepekatan yang berbeza. Daya kepekaan pengesan hidrogen berbeza berdasarkan kumpulan terfungsi yang melekat kepada CNT dan juga kepekatan gas.

Hasil-hasil dari ekseprimen ini dapat membuktikan bahawa kaedah penyusunan dan penjajaran CNT yang dicadangkan dalam tesis ini mampu menyusun dengan tepat CNT yang lebih dari 50% kali panjang dari CNT yang disusun menggunakan kaedah DEP yang sedia dan CNT tersebut sesuai digunakan dalam pembikinan peranti-peranti elektronik.

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Finally, very special gratitude and appreciation to my beloved family for their support and being patient along my journey. Without them, this journey would be almost impossible. Their sacrifices in time and endless love encouraged me to complete this journey

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research and the writing of this thesis were done under our supervision;
- supervisory responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2015-2016) are adhered to.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AA-DEP	Airflow-assisted Dielectrophoresis
ACEO	AC Electroosmotic
AC-EP	Alternating Current Electrophoresis
ART	Alignment Relay Technique
BAW	Bulk Acoustic Waves
B-DEP	Basic Dielectrophoresis
CD-DEP	Ceiling Deposition DEP
CM	Clausius–Mossotti
CMC	Carboxymethyl Cellulose
CNT	Carbon Nanotube
CNTFET	Carbon Nanotube Field-Effect Transistor
CNTs	Carbon Nanotubes
CoMoCat	Cobalt Molybdenum Catalyst
CVD	Chemical Vapor Deposition
DCE	1,2-Dichloroethane
DCEO	DC Electroosmotic
DC-EP	Direct Current Electrophoresis
DEP	Dielectrophoresis
DIW	Deionized Water
DLS	Dynamic Light Scattering
DMF	Dimethylformamide
DWCNT	Double-Walled Carbon Nanotube
EBL	Electron Beam Lithography
EDL	Electric Double Layer

EP	Electrophoresis
ETH	Electrothermal
F-MWCNT	Functionalized Multi-walled Carbon Nanotube
F-SWCNT	Functionalized Single-walled Carbon Nanotube
FEM	Finite Element Method
FESEM	Field Emission Scanning Electron Microscopy
FIB	Focused Ion Beam
FTIR	Fourier Transform Infrared
GG	Global Gate
HiPco	High-Pressure Carbon Monoxide
HRTEM	High-Resolution Transmission Electron Microscopy
IC	Integrated Circuits
IDE	Interdigitated Electrodes
IPA	Isopropyl Alcohol
IR	Infrared
ITO	Indium Tin Oxide
LoC	Lab-On-Chip
LB	Langmuir–Blodgett
LG	Local Gate
LS	Langmuir–Schaefer
M-SWCNT	Metallic Single-Walled Carbon Nanotube
MWCNT	Multi-Walled Carbon Nanotube
NMP	N-Methyl-2-Pyrrolidone
PC	Polycarbonate
PDMS	Polydimethylsiloxane
PET	Polyethylene Terephthalate

PPE	Parallel Pairs Electrodes
PS	Polystyrene
SAW	Surface Acoustic Waves
SC	Sodium Cholate
SD	Standard Deviation
SDBS	Sodium Dodecyl Benzene Sulfonate
SDS	Sodium Dodecyl Sulfate
S-SWCNT	Semiconducting Single-Walled Carbon Nanotube
SWCNT	Single-Walled Carbon Nanotube
TC	Temperature Coefficient
TC-DEP	Thickness Controlled Dielectrophoresis
TEP	Teslaphoresis
TW-DEP	Traveling Wave Dielectrophoresis
UV-Vis	Ultraviolet-Visible

CHAPTER 1

INTRODUCTION

1.1 General Overview

Discovering Carbon Nanotubes (CNTs) in 1991 by Iijima was a breakthrough in the field of material science (Iijima, 1991). CNT applications have not seen widespread because of the anisotropic nature of CNTs and the difficulty in placing them into the desired location in an aligned form. Another reason is that CNTs are synthesized in a high-temperature environment, which is not compatible with most current fabrication methods that are used to manufacture electronic devices such as CNT-based sensors and transistors (Z. Chen et al., 2016; Gao et al., 2012; F. Yang et al., 2014).

Several methods have been used to assemble and align CNTs within materials and devices. The methods differ from each other based on the alignment scale and the force used to achieve the alignment. For example, twisting and compressing are mechanical forces used to align CNTs in fibers up to a few meters long (S. Zhang et al., 2019). Electrophoresis (EP) is a method used to align CNTs in polymer composites on a larger scale (mm-cm) using electric force (Engin C Sengezer et al., 2017).

Dielectrophoresis (DEP) is the electrokinetic movement of dielectrically polarized particles in non-uniform electric fields (Morgan & Green, 2003). DEP is a popular method in microfluidic systems and Lab-On-Chip (LoC) technology, where it is used to manipulate particles such as cells and bacteria (Çetin & Li, 2011; Lapizco-Encinas et al., 2004; Voldman, 2006). DEP is also applied in other research areas, such as nanomaterial manipulation, nanowires assembly, and CNT alignment (Garciá Núez et al., 2020; Ralph Krupke et al., 2006; Linbo Liu et al., 2019).

The DEP setup that is used to assemble and align CNTs consists of three main components, which are the electrode geometry, medium and supplied AC signal. The electrode geometry is usually designed based on the application requirements, while the medium is a stable and easy to process solvent containing well-dispersed CNTs. The supplied AC signal provides an electric field to the system in order to generate the DEP force (B. Yang et al., 2014). The assembly and alignment of carbon nanotubes across narrow electrode gaps using the explained setup are relatively easy because CNTs require low voltage signals to be polarized. Furthermore, the lengths of the CNTs are comparable to the gap width, which means that the CNTs can bridge the electrode gap directly and do not need to chain to each other to form complete bridges. Recent studies reported the alignment of CNTs across gap widths of 1µm (Böttger, Schulz, et al., 2016), 5µm (Gorshkov et al., 2018), 10µm (Shao et al., 2021), 20µm (M. Lee & Kim, 2016), 30µm (M.-C. Hsu & Lee, 2014), 40µm (T. Zhou et al., 2021), and 50µm (Xiaowei Li et al., 2017).

The alignment of CNTs across gap widths beyond 50µm is challenging using the current setup due to several reasons. First, stronger electric fields are required in order to polarize enough CNTs to coat the wide gaps. Strong electric fields are also responsible for the appearance of side forces in the DEP system, such as the Electrothermal (ETH) force and the AC Electroosmotic (ACEO) flow (Burg et al., 2010). These forces induce medium drag velocity obstructing the CNTs' motion and preventing them from reaching the deposition area. Furthermore, maintaining the quality of long aligned CNT bridges during the drying process is critical due to the weak contact between individual CNTs.

There are two roads to overcome the problems related to alignment across wide electrode gaps. The first road focuses on reconfiguring the DEP setup and optimizing its components to minimize the drag velocity resulting from the strong electric field. For example, Beigmoradi et al. used a microfluidic channel with a specific thickness to control the separation of different types of CNTs (Beigmoradi & Aghamiri, 2019). The second road focuses on introducing external forces besides the DEP or combining two alignment methods to achieve high-quality, long-aligned CNTs bridges. Optically-induced dielectrophoresis (O-DEP) (G. Bin Lee, 2010), heating-enhanced DEP (HE-DEP) (Gu et al., 2017), and light-assisted DEP (L-DEP) (W. Li et al., 2014) are examples of external forces used to assist the DEP to align CNTs across microelectrodes. However, the main challenge in introducing these external forces to the DEP setup is increasing its complexity. Combining the DEP with less complex forces or alignment methods such as airflow or vacuum filtration is more promising and easy to scale up solutions (Hedberg et al., 2005)(Oh et al., 2015).

In this work, thickness-controlled airflow-assisted DEP is utilized to assemble CNTs from a solution and align them across electrode structures with gap widths up to 125µm. DEP is chosen over other methods because it is an electrode-based method, where the CNTs can be assembled and aligned directly to the required location across the electrodes. DEP is also a room temperature method and has a flexible hardware setup, making it compatible with other fabrication methods used to fabricate CNT-based devices (Kimbrough et al., 2020).

1.2 Problem Statement

The deposition of CNTs across electrode structure using the DEP force has two steps which are assembly and alignment. The assembly is where the CNTs are attracted from the medium toward the deposition area (electrodes gap), while the alignment is forming organized CNTs bridges parallel with the electric field lines.

The challenges in the CNTs assembly increase as the electrode gap increases because the DEP force suffers from high attenuation at regions far from the electrode surface (C. H. Han et al., 2019). The logical solution to this problem is increasing the electric field in order to generate stronger DEP force and polarize enough CNTs to occupy the electrode gap. However, strong electric fields cause a temperature rise in the DEP medium because the current and the charge carriers generate heat when they pass through the medium, which is also termed Joule heating (JH) (Kwak et al., 2019; W. Li, 2017). The temperature rise produces gradients in the medium's permittivity and conductivity, causing a generation of coulombic and dielectric forces. Another coulombic force is generated above the electrode surface due to an Electric Double Layer (EDL) formation at the electrode medium interface. These electrothermal (ETH) and AC electroosmotic (ACEO) phenomena cause medium drag velocity, obstructing the CNTs from reaching the gap vicinity or driving them away from the deposition area (Rabbani et al., 2020).

The challenge in the alignment is the difficulties in maintaining the quality of the alignment of long carbon nanotube bridges due to the poor controllability of the DEP method (T. Zhou et al., 2020). Long CNT bridges, which consist of shorter CNTs, are sensitive to any medium motion because of the weak contact between the chained CNTs (Böttger, Hermann, et al., 2016). For example, the medium motion during drying might destroy or break the aligned bridges causing an increase in the device resistance. The distortion in the aligned CNT bridges caused by the medium motion is the main reason for the resistance variation in CNT-based devices fabricated with an identical DEP procedure (weak reproducibility) (C K M Fung et al., 2004; T. Han et al., 2019).

Overcoming the assembly and alignment challenges discussed above is essential to fabricate long CNT bridges. Current DEP setups can be modified to achieve long alignment either by reconfiguring the system components or introducing new forces to the setup. Reconfiguring the system components such as optimizing the electrode geometry and AC signal parameters or selecting stable CNT suspension with low drag velocity. However, maintaining the system's simplicity should be considered to preserve the DEP controllability and make it suitable for large production of CNT-based sensor devices with strong reproducibility (P. Sharma & Ahuja, 2008).

1.3 Aims and Objectives

The main objective of this study is to produce long well-aligned CNT bridges across electrode structures with gap widths between 50µm to 125µm using the DEP method. The achievement of this primary objective can be reached by achieving the following specific objectives:

1. To investigate the effect of the electrode geometry, medium properties, and AC signal on the dielectrophoretic assembly of CNTs using Finite Element Method (FEM) simulation.

2. To experimentally characterize the solubility and dispersity of treated CNTs in different mediums in order to select suitable suspensions for the dielectrophoretic assembly and alignment of CNTs.

3. To experimentally assemble and align long CNT bridges using modified DEP setups that are able to reduce the drag velocity caused by the ETH forces and maintain the alignment quality during drying.

4. To investigate the effectiveness of the designed DEP setups by fabricating CNT-based sensor devices for pH, temperature, and Hydrogen sensing.

1.4 Scope and Limitations

This work is divided into two phases. In the first phase, a simulation model will be designed to investigate the role of each component of the DEP system in order to predict the CNT velocity during the assembly process. The simulation will focus on three main quantities: the electric field distribution, forces present in the DEP system, and velocities subjected to the CNT. Only 2D simulation will be considered to reduce computation time.

In the second phase, experimental work will be conducted based on the results obtained from the simulation. The experimental work will focus on preparing various CNT suspensions and fabricating several electrode structures with gap widths up to 125μm. The modification in the DEP system will be limited to using a glass cover to control the channel height and introducing airflow to maintain the aligned CNT bridges. The CNT bridges will be used as sensing material in sensor devices to prove their usability. Although CNTs show strong sensitivity to many external stimuli, in this work, the sensitivity testing of the aligned CNT bridges will be limited to pH value, Hydrogen molecules, and temperature variation.

1.5 Report Organization

This report is organized into five chapters. The first chapter presents the introduction, problem statement, objectives, and the scope of the research. The second chapter addresses the literature review and previous work related to the topic of this dissertation. Work on CNT alignment using DEP is discussed, compared, and extensively reviewed. The methodology, including the simulation details, experimental setups, and characterization methods, are explained in chapter three. In chapter four, the findings are presented and discussed in detail, including both simulation and experimental results. The thesis is concluded in chapter five, where the final results and optimum parameters are listed and summarized. Future outlook and suggestions to improve this work are also included.

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