**UNIVERSITI PUTRA MALAYSIA**

***FABRICATION AND CHARACTERIZATION OF CARBON NANOTUBES/POLYDIMETHYLSILOXANE NANOCOMPOSITE FOR LOW PIEZORESISTIVE PRESSURE SENSOR***

**SAMAN AZHARI**

**ITMA 2016 4**

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**By**

**SAMAN AZHARI**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science**

**June 2016**

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**DEDICATED**

*This thesis is dedicated to my beloved parents for their endless love, supports and encouragement*

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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**SAMAN AZHARI**

**June 2016**

**Chairman : Associate Professor Mohd Nizar Hamidon, PhD**
**Institute : Advanced Technology**

Pressure sensors can vary drastically in technology, design, performance, application suitability and cost. A conventional estimate would be that there may be over 50 technologies and at least 300 companies making pressure sensors worldwide. However considering the number of companies there are still certain range of pressure, scale and applications that are being under developed. Being able to detect pressure at micrometer and nanometer scales would be of an advantage.

In recent years conductive and piezoresistive pastes fabricated via nanomaterials are attracting a lot of attention. Polydimethylsiloxane (PDMS) is the most common silicon based organic polymer. Mechanical and electrical properties of PDMS make it an ideal polymeric material for Nano fluidic, wearable and flexible sensory applications.

One of the most common issues that are not fully resolved is CNTs tendency to aggregate while being mixed in polymeric matrix. Our goal for this work is to fabricate a homogenous CNTs/PDMS nanocomposite with Piezoresistive properties for applications in flexible electronics and sensory devices. The first step taken in this work is to characterize the CNTs.

In this work two common solvents to prepare CNTs/PDMS nanocomposite have been compared. Moreover the electrical properties of samples prepared by solvent mixing and the effect of mixing techniques and curing time on aggregation of CNTs were discussed. Different purification processes and advantages of functional CNTs were studied and functional CNTs were utilized to fabricate a homogenously dispersed CNTs/PDMS nanocomposite.

Based on the results obtained, purification of CNTs increases the crystallinity and functionalization enhances the dispersion rate of CNTs in PDMS. There is a direct relation between mixing method and curing time with aggregation rate of CNTs in PDMS; THF displays better dispersion in comparison with chloroform and CNTs/PDMS nanocomposite exhibits measurable conductance at above 10 wt%. Piezoresistive measurements indicate this material could be a suitable replacement for piezoresistive materials used for sensory devices due to their high sensitivity, accuracy and flexibility in addition to their adjustable size.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

**PEMBANGUNAN CARBON NANOTUBES/POLYDIMETHYLSILOXANE KOMPOSIT NANO; TERPAKAI UNTUK NANO APLIKASI SENSING TEKANAN PIEZORESISTIVE**

Oleh

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Sensor tekanan boleh berubah-ubah secara drastik dalam teknologi, rekabentuk, prestasi, kesesuaian permohonan dan kos. Anggaran konvensional mungkin terdapat lebih daripada 50 teknologi dan sekurang-kurangnya 300 syarikat membuat sensor tekanan seluruh dunia.Walau bagaimanapun memandangkan bilangan syarikat masih terdapat pelbagai tertentu tekanan, skala dan aplikasi yang masih dalam pembangunan.Dapat mengesan tekanan pada micrometer dan nanometer skala akan menjadi satu kelebihan.

Dalam tahun-tahun kebelakangan ini peskonduktif dan piezoresistive direka melalui bahan nano yang menarik banyak perhatian. Polydimethylsiloxane (PDMS) adalah silikon yang paling biasa berasaskan polimer organik. Sifat-sifat mekanikal dan elektrik PDMS menjadikan ia satu bahan polimer yang sesuai untuk nano fluidic, guna semula dan aplikasi fleksibel deria.

Salah satu isu yang paling biasa yang tidak dapat diselesaikan sepenuhnya adalah kecenderungan untuk agregat ketika bercampur dalam matriks polimer. Matlamat kami untuk kajian ini adalah untuk mereka homogen CNTs / PDMS nanokomposit dengan sifat piezoresistive untuk aplikasi dalam elektronik fleksibel dan peranti deria.Langkah pertama yang diambil dalam kerja-kerja ini adalah untuk mencirikan CNTs.

Dalam kerja-kerja ini, kami telah berbanding dua pelarut biasa untuk menyediakan CNTs / PDMS Kompositnano. Tambahan lagi ,sifat-sifat elektrik sampel yang disediakan oleh pencampuran pelarut dan kesan pencampuran teknik dan masa pengawetan pada pengagregatan CNTs telah dibincangkan. Proses pembersihan yang berbeza dan kelebihan CNTs berfungsi dikaji dan CNTs berfungsi telah digunakan untuk menghasilkan sebaran yang seragam CNTs / PDMS Kompositnano.
Berdasarkan keputusan yang diperolehi, pembersihan CNTs meningkatkan penghabluran dan fungsian meningkatkan kadar penyebaran daripada CNTs dalam PDMS. Terdapat hubungan langsung antara kaedah campuran dan masa pengawetan

dengan kadar pengagregatan CNTs dalam PDMS; THF memaparkan penyebaran yang lebih baik berbanding dengan kloroform dan CNTs / PDMS komposit nano bersifat kealiran diukur lebih dari 10%.Ukuran piezoresistive menunjukkan bahan ini boleh menjadi pengganti yang sesuai untuk bahan-bahan piezoresistive digunakan untuk peranti deria kerana kepekaan yang tinggi, ketepatan dan fleksibiliti di samping saiz yang boleh diubah.

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I certify that a Thesis Examination Committee has met on 30 June 2016 to conduct the final examination of Saman Azhari on his thesis entitled "Fabrication and Characterization of Carbon Nanotubes/ Polydimethylsiloxane Nanocomposite for Low Piezoresistive Pressure Sensor" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| 0D | Zero dimensional |
| 1D | One dimensional |
| 2D | Two dimensional |
| 3D | Three dimensional |
| ACCVD | Alcohol catalytic chemical vapor deposition |
| C | Carbon |
| CNTs | Carbon nanotubes |
| COOH | Carboxyl |
| CVD | Chemical vapor deposition |
| D-band | Disordered-band |
| EDS | Energy dispersive X-ray Spectroscopy |
| FESEM | Field emission scanning electron microscopy |
| G-band | Graphitized-band |
| HP | Hot Plate |
| HRTEM | High resolution transmission electron microscopy |
| MEMS | Micro Electro Mechanical System |
| MS | Magnetic Stirrer |
| MWCNTs | Multiwalled carbon nanotubes |
| NEMS | Nano Electro Mechanical System |
| Ni | Nickel |
| O | Oven |
| OH | Hydroxyl |
| PDMS | Polydimethylsiloxane |
| RBM | Radial breathing mode |
| RT | Room Temperature |
| SWCNTs | Single walled carbon nanotubes |
| THF | Tetrahydrofuran |
| UB | Ultrasonic Bath |
| UH | Ultrasonic Homogenizer |

# CHAPTER 1

# INTRODUCTION

Sensor is a transducer used to detect certain changes -correlated to design and application- in its surroundings. Automotive industry, telecommunication, electrical and electronics, chemistry and many other fields of science and engineering as well as industries would not be where they are now without these devices [1]. We are surrounded by sensors all around us; our current daily life depends on them.

There are different types of sensors available on the market. These devices could be categorized based on the modes of detection. Electrical, optical, mechanical, chemical, physical and biological methods introduce a wide variety of sensors [2], [3]. A certain phenomena could be detected by different types of sensors; for example optical, electrical and mechanical sensors each could be used to detect pressure. A sensor is preferred based on the application and the environment [4].

A pressure sensor is a transducer that converts an applied pressure force to electrical signal. There are number of MEMS devices which are able to do so; in scales as big as few micron. Although one of the major disadvantages would be their cost; furthermore the process of making such sensory devices are not as simple as one would suspect. As an example of such devices micro Cantilever and silicon based Piezoresistive pressure sensors could be mentioned [1], [5], [6].

Although thin film has given us the chance of making flexible devices, it is yet incapable of reducing cost and increasing reliability. MEMS devices did not perform well below a micro scale [7]; as an example, vascular complication happen at different regions in human body, so detecting the specific area of these complications accurately would be an advantage, but due of the uneven shape of body parts and scale of these vessels, it is not possible to find these complications at its specific region, using existing devices.

Since the discovery of graphene [8] and report regarding the growth of carbon nanotubes (CNTs) [9], nanomaterials have attracted a lot of attention. Although it still has a long way to display its full potentials. Nanotechnology is manipulation of matter in atomic and molecular scale. Nanotechnology has given us the opportunity to overcome such barriers as well as reducing the scale. Considering these researchers from all fields of science have been working together to elevate their knowledge and to devise applicable Nano-scale sensory devices; that could overcome the shortcoming of MEMS technology [10].

In recent years conductive pastes of conductive nanomaterials and non-conductive polymers are attracting a lot of attentions. Conductive pastes are being developed by numerous research groups to overcome such shortcomings of MEMS devices. The fabricated pastes vary in relation to the materials used to develop them. Copper (Cu), Iron (Fe), Aluminum (Al), CNTs and their composite are some of the main nanomaterials used to develop such pastes. Although the tendency of these materials to aggregate during the fabrication process is still an issue that need to be resolved.

## 1.1 Problem statement

Pressure sensors can vary drastically in technology, design, performance, application suitability and cost. A conventional estimate would be that there may be over 50 technologies and at least 300 companies making pressure sensors worldwide. However considering the number of companies there are still certain range of pressure and applications that are being under developed such as low range pressure sensors.

Therefore, being able to detect pressure at micrometer and nanometer scales would be of an advantage. Nano scale pressure sensor is developed to measure the pressure at precise points of micro and nano scale system. Measuring pressure at such small scale needs even smaller devices.

This study is dedicated to characterize the commercial CNTs in addition to devising a flexible Nano-scale sensory device capable of measuring the pressure at very small scale with high accuracy and sensitivity through fabrication of CNTs/Polymer nanocomposite [11]. Flexible CNTs/Polymer Nano-composite is fabricated to develop such device. Piezoresistive effect of CNTs and elasticity of polymer is combined to construct an applicable device. Due to the scale of CNTs specific and accurate results could be obtained at small scales; hence detecting the pressure at micro and nano scales.

## 1.2 Objective

In order to successfully fabricate a Piezoresistive pressure sensor using CNTs/polymer nanocomposite the following objectives will be considered:

1- To characterize the purified and functionalized CNTs; verify their quality and structure.
2- To determine the fabrication technique and effective factor for polymer/CNTs paste.
3- To fabricate a flexible Piezoresistive sensor.

## 1.3 Scope

The challenges of this study are to homogeneously disperse the CNTs in polymer matrix as well as achieving conductivity besides piezoresistivity and finally, to fabricate an ultra low range (0 Psi to 14 Psi) flexible sensor using screen printing technique. Raman Spectroscopy, Energy dispersive X-ray spectroscopy (EDS), Ultraviolet visible spectroscopy (UV/Vis), Fourier Transform Infrared spectroscopy (FTIR), two point probes, Transmission electron microscopy (TEM) and FESEM were used to characterize the CNTs and nanocomposite. Piezoresistivity was measured after fabrication of the sensor.

## 1.4 Thesis content

The layout of this thesis is presented as follows:

Chapter 1 presents an introduction to MEMS technology, sensory devices, their application and flaws; and nanotechnology as well as the scope, objectives and problem statement.

Chapter 2 discusses significant literature review concerning nanocomposites, their applications and properties, CNTs structure and properties, polymers properties and applications, piezoresistivity, percolation threshold, methods for mixing and fabricating of CNTs/polymer nanocomposite, purification and functionalization of CNTs; electrode design and sensor application.

Chapter 3 mainly discusses materials used for the work, methodology and the flow of this work to achieve the objectives as well as characterization techniques. The measurement setup and electrodes design are discussed.

Chapter 4 discusses the experimental results from the characterization techniques and analyses the outcome. The results indicate importance of purification and functionalization; solvent compatibility and quality of the materials, for achieving the objectives.

Chapter 5 highlights the conclusions obtained in relation to objectives. In addition it provides recommendation for further studies.

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