INSTITUTO TECNONÓLOGICO Y DE ESTUDIOS SUPERIORES DE MONTERREY

Campus Estado de México
School of Engineering and Sciences

Fabrication of Suspended Nanowires Through Mechano-Near-Field Electrospinning of Polymers in Solution for the Production of Glass-like Carbon

A thesis presented by:
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Declaration of Authorship

I, Antonio Osamu Katagiri Tanaka, declare that this thesis titled, "Fabrication of Suspended Nanowires Through Mechano-Near-Field Electrospinning of Polymers in Solution for the Production of Glass-like Carbon" and the work presented in it are my own. I confirm that:

- This work was done wholly or mainly while in candidature for a research degree at this University.
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Antonio Osamu Katagiri Tanaka
Estado de México, Atizapan de Zaragoza, December 02, 2020

"Carbon is a simple element but ....

One branch of chemistry is devoted to its compounds!

One branch of science is devoted to the many forms of the element as a solid material.

The best of this is that although most carbon materials are grey or black to the naked eye and the uninitiated, a closer examination reveals the form, beauty and even color of carbon science."

Marsh, Harry
Universitat d'Alacant, Alicante, Spain
scopus.com

Dedication

Thanks for all your unconditional confidence, support, patience, and encouragement. You were my main motivation for pushing through this work.

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Fabrication of Suspended Nanowires Through Mechano-Near-Field Electrospinning of Polymers in Solution for the Production of Glass-like Carbon

by Antonio Osamu Katagiri Tanaka

Abstract

Carbon nano-wires are versatile materials composed of carbon chains with a wide range of applications due to their high conductivity. Regardless of the high interest in the implementation of carbon nano-wires in several applications and devices, no feasible processes have been developed to fabricate carbon nano-wires with spatial control at a reasonable cost. Carbon nano-wires have been fabricated with the use of a photoresist, but little is known about polymers that can produce more conductive carbon nano-wires after pyrolysis. Various polymer solutions have been tested in near field electrospinning (NFES) and photopolymerization separately, however, few have been tested for nano-wire fabrication purposes through pyrolysis. The intention behind the thesis proposal is to implement rheology analyses of different polymer solutions to determine if they can be easily electrospun at low voltages and then fabricate nano-wires with them. This thesis work arises from the need to test a greater variety of polymers with the goal to design a polymer solution to fabricate carbon nano-wires with better conductivity than the current SU-8 polymeric nano-fibers. The research process will include the design of polymer solutions that can be electrospun, photopolymerized, and then pyrolyzed into conducting carbon nanowires. On the other hand, it is intended to engineer a newly designed polymer solution to achieve mass scale manufacturing of conductive carbon nano-wires in an inexpensive, continuous, simple and reproducible manner as central components for nano-sensors.

keywords: nanotechnology, carbon, nano-wires, Near-Field Electrospinning, NFES

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Chapter 1

Introduction

Carbon nano-materials are subjected to great interest for research purposes due to their various potential applications in diverse areas that take advantage of the nano-scale properties. Carbon nano-materials are suitable for catalysis, adsorption, carbon capture, energy and hydrogen storage, drug delivery, bio-sensing, and cancer detection. Some matchless properties that allow carbon nano-materials to be utilized within multiple functionalities include high porosity, distinguished structures, uniform morphologies, high stability, high magnetic properties, and high conductivity. [1–8]

This document bestows a thesis project to perform research to engineer a polymer solution to achieve mass scale manufacturing of high conductive carbon nano-wires with a reduced diameter in an inexpensive, continuous, simple and reproducible manner. The research intends to involve several manufacturing processes such as near field electrospinning, photo-polymerization, pyrolization, and carbonization, as they have shown to be promising methods for the fabrication of carbon nano-materials. [9] See Figure 1.1. A number of processes have been developed for specific purposes of polymeric nano-fibres, some include surface deposition, composites, and chemical adjustments. Polymeric nano-fibers must be also pyrolyzed to generate carbon nano-wires with conductive capabilities [10] for electrochemical sensing and energy storage purposes.

Nanotechnology has led to the study of different polymer patterning techniques to integrate carbon nano-wires structures. One technique is known as far-field electrospinning (FFES), a process in which electrified jets of polymer solution are dispensed to synthesize nano-fibres which are then pyrolyzed at high temperatures. One sub-technique derived from

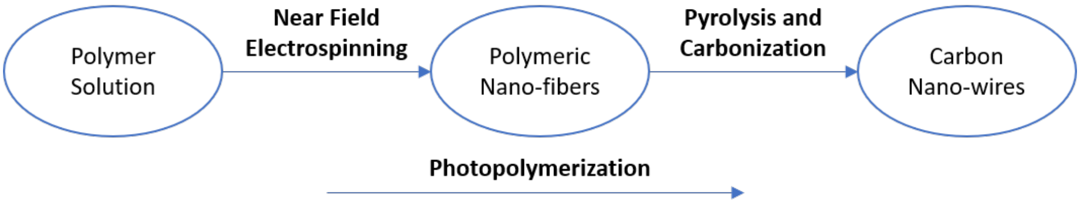

FIGURE 1.1: Fabrication process and characterization techniques of conductive carbon nano-wires to achieve through the dissertation.

electrospinning is near-field electromechanical spinning or NFEMS. Unlike FFES, NFEMS has proved to deliver high control in patterning polymeric nano-fibres. [9]

The proposal is to continue the previous work done in regards to the synthesis of carbon nano-wires. Previous work includes the fabrication of suspended carbon nano-wires by two methods: electro-mechanical spinning and multiple-photon polymerization with a photoresist. [9, 11] This work is intended to focus on electro-mechanical spinning processes only, to bring off polymer solutions that can be electrospun by NFEMS, photo-polymerized and pyrolyzed into conducting carbon nano-wires. The polymer solutions described by Cárdenas and Flores [9, 11] are to be amended to achieve the goal mentioned in the previous statement.

Traditional near-field electrospinning or NFES allows large scale manufacturability combined with spatial control of material deposition. [10] However, the reported efforts required the use of electric fields in excess of 200 kV/m for continuous operation, resulting in limited control for nano-fiber patterning in traditional NFES processes. Madou et al. [10] conclude that the current state-of-the-art synthesis processes for polymer nano-fibers lack to yield precise, inexpensive, fast, and continuous manufacturing properties.

1.1 Carbon Nanowires Research Developments in Terms of Published Papers, Synthesis and Fabrication

Nanotechnology ability to control and piece together materials at the nano-scale has enabled the development of various carbon nano-materials and carbon nano-structures, such as nano-dots, nano-fibres, nano-tubes and nano-wires. [12–15] This chapter bestows on the applications at the micro-scale and nano-scale levels, as well as the current research of carbon-based nano-materials (CBNs).

1.1.1 Carbon and carbon-based nanomaterials

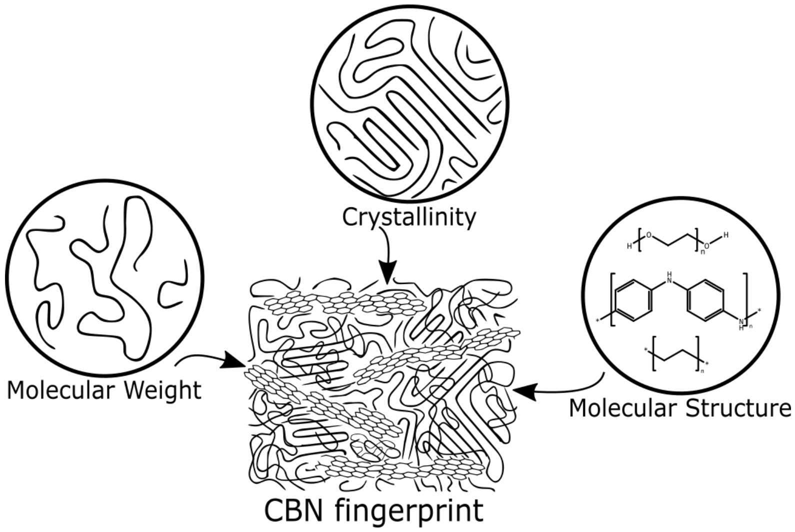

FIGURE 1.2: Molecular to meso-scale structural features of synthetic polymers influence the emergence of specific micro-structural features in polymer-derived carbon materials after pyrolysis.

Carbon is a versatile element capable to form a number of bonds with other elements or with itself. Cabon-based nano-materials (CBNs) exist in diverse forms, depending on the precise values of each degree of freedom that specify the material proclivity at multiple scales. Hybridization, crystallization, percolation, anisotropy, porosity, impurities and imperfections are some of the relevant features that determine the CBN set of properties. The combination of these features at the micro- and meso-scale burst a variety of macro-scale properties that comprise the CBN fingerprint (1.2). The interminable collection of possible CBN fingerprints range from soft, conductive lubricants to very hard, low conductivity solids; and from black colour, bulks to transparent, disordered thin films. [1] Figures 1.3 and 1.4 shows the existence of different types of allotrope as carbon orbitals have the ability to hybridize in sp1, sp2 and sp3 configurations, assembling different types of allotropes.

In terms of porosity, CBNs exhibit different properties according to the degree of 'open' and 'closed' pores. A 'closed pore' is a void or empty space in solid materials where a discontinuity is present within the array of atoms and molecules. On the other hand, an 'open pore' refers to a void which is connected to the outer surface of the solid, in other words a 'open pore' is a

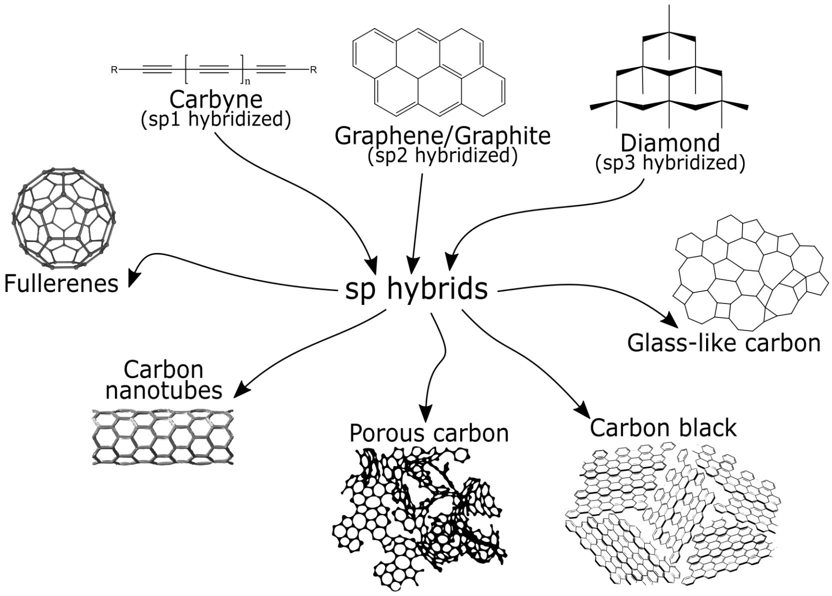

FIGURE 1.3: Three carbon allotropes (diamond, carbyne and graphene) are the building blocks of additional deriving carbon types such as fullerenes, porous carbon and glass-like carbon.

'closed pore' with an opening to the external surface. [23] Figure 1.5 shows a classification of carbon allotropes according to their porous content.

Thermal conductivity and electrical conductivity decrease with increasing porosity due to the reduced amount of material to conduct electrons and energy. Furthermore, porosity negatively affects the mechanical properties like strength and elastic modulus as it reduces the volume in which stresses are distributed. Moreover, stresses are concentrated at the pores which makes the material prone to mechanical failure. [23, 24]

Due to the versatility and variety of CBNs, CBNs have been fabricated and implemented for various purposes. [2, 4–8]. For instance, field effect transistors (FET) have been studied by Novoselov [25] and Heersche et. al. [16]. Carbon FET devices have reported field-effect mobility one order of magnitude higher than that of silicon FETs. Other literature suggests CBNs to be favorable to detect a variety of gases and bio-molecules. [26, 27] As molecules are absorbed by the CBN, the carrier density and electrical resistivity of the carbon material changes. Moreover, CBNs have showed good performance in applications in energy (prevent wastage of energy), water (purification) and diagnostics (lab-on-chip systems and nano-sensors). [15, 28] As mentioned above, the morphology of CBNs has an impact on the electrochemical and mechanical properties. [23, 24, 29] In this regard, carbon

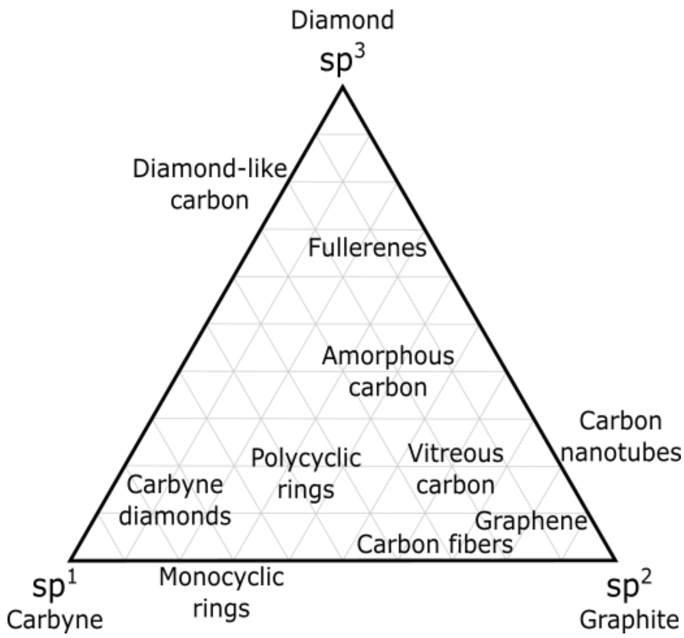

FIGURE 1.4: Ternary phase diagram of amorphous carbon regions based on hybridization degree. Adapted from [16–22].

nano-structures, such as nano-wires [30, 31], have been fabricated to achieve improved electrochemical characteristics.

1.1.2 Carbon Nano-wires

As depicted in Figure 1.4, carbon nano-fibers (CNFs) have been classified as linear, sp2-based structures. [16–22] Nano-fibers own good electrical, optical and mechanical characteristics, however those properties are highly dependent on the morphology of the fibers. [32] The material properties of 1D nano-structures depend on fiber diameter, porosity, crystallization degree and crystallization orientation. Consequently, the fabrication parameters and environment conditions have an impact on the reproducibility of high quality fibers. [32] Carbon nano-fibers (CNFs) have diameters of several micrometers (Figure 1.7) and are different from carbon nano-tubes (CNT). [33–37] Unlike carbon nano-tubes with hollow cores, carbon nano-fibers can be represented as stacked layers along the thread length. [37–39] The stacked geometry of carbon nano-fibers results in unique electrical, chemical and mechanical properties. [40–42] Unlike CNFs, carbon nano-tubes inherent problems such as high cost and low effective surface area, which limit their practical use. [22]

sp2 carbon nano-wires have been used for the improvement of power

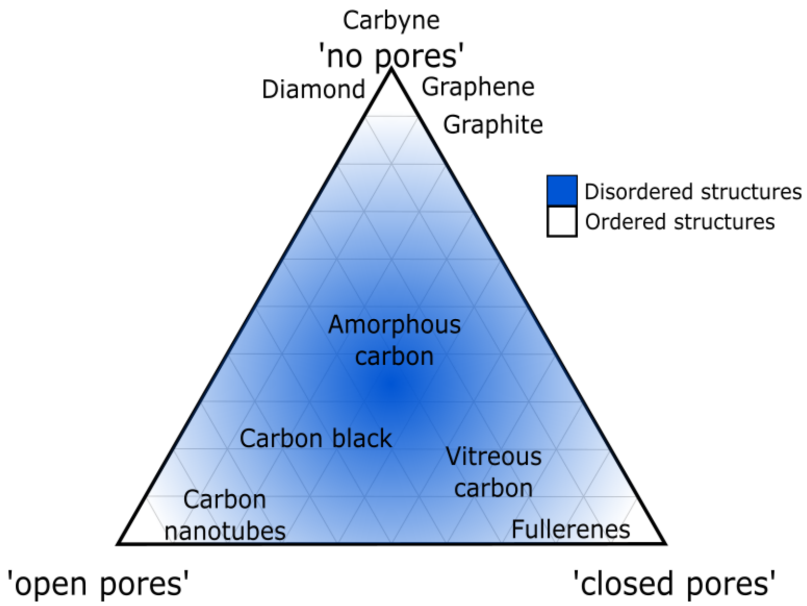

FIGURE 1.5: Ternary phase diagram of amorphous carbon regions based on structure order and porosity. Regions are colored by the degree of crystalline order within the carbon structure. White represents highly ordered structures, whereas white represents disordered structures. [23, 24]

density and specific energy in lithium-ion batteries. [43–45] Authors posit that the performance and capacity of Li-ion batteries depend on the CNF structure and texture. Through the right combination of electrospinning and carbonization parameters, electrically conductive, mechanically tough and with thin diameter fibers have been achieved by Yoon et al. [39]. Yoon reported 431 mili-ampere-hour per gram batteries with vitreous carbon nanofibers. Yoon states that the battery capacity highly depends on the pyrolysis process parameters as the morphology of the fiber develops pores and hence different surface properties. CNFs supercapacitors have been investigated as energy storage devices due to their high power bearability and long lifecycles. [46–49] The studies' authors posit that carbon nano-fibers can be implemented as high-power supercapacitors due to their large surface area and high electrical conductivity.

On the other hand, the low reactivity and unique morphology of CNFs make them promising catalyst supports for metal nano-particles. [50–52] It is well known that the morphology and nano-structure of the supporting material are the main factors that prevent agglomerations of nano-particles. [53, 54] Moreover, in bone tissue scaffold applications, collagen is the most popular scaffold. However, collagen scaffolds bring xenogenicity issues which

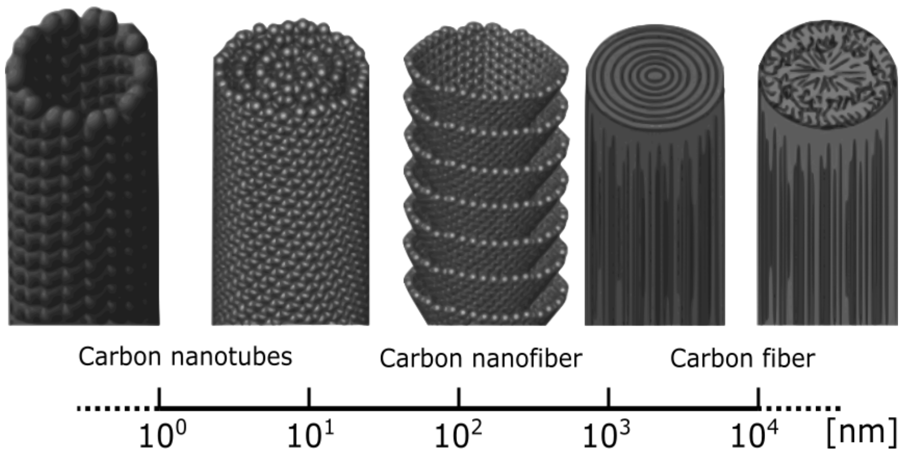

FIGURE 1.6: Various types of fibrous carbon materials bear different characteristics according to their molecular structure. Adapted from [22]

leads to disease transfer or immunogenic reactions, besides its inhability to preserve its shape once placed in the body. [55–62] Currently, carbon fibers have been studied for bone tissue scaffold, however early attempts yield too thick fibers for cell cultivation and tissue regeneration. [63, 64] As depicted in previous research of CNFs for different applications, fiber morphology seems to have a significant impact on their performance.

Typically, carbon nano-fibers (CNFs) are synthesized by a combination of a patterning process and a pyrolysis process. CNFs fabricated by electrospinning of polymer solutions with electrostatic forces. Electrospun CNFs have characteristics such as high surface area, thin morphology with nano-scale diameters. The properties of electrospun fibers allow CNFs to be implemented in nano-sensing devices, energy storage applications, and tissue scafflods. [28, 65–70] Several patterning techniques have been attempted to achieve the desired fiber morphology. In addition to electrospinning, CNFs have been also fabricated by two-photon polymerization (TPP) and photo-lithography techniques. [71] Cardenas et al. implemented TPP and conventional UV lithography to study the fabrication of CNFs within carbon micro-electromechanical systems.

1.2 Problem definition and motivation

The role of carbon nano-fibers in nano-sensor devices play an important role, as portable instruments require light-weight and small-sized components. [28] Table 1.1 lists some advantages of nano-sensors that can be accomplished by the fabrication of CNFs via near-field electrospinning and a thermal treatment in an inert environment.

TABLE 1.1: Advantages of Nano-sensors. Adapted from [28]

Advantage	Description
High sensitivity	More accuracy, single molecule detection
Small size	Light-weight, portability, low-power consumption, small sample size, reduced sample preparation, and ease of use
Low response time	High-frequency, real time analysis
Low cost	Disposable devices

Sensors of small size require less time to output a stable signal as signals require less time to travel shorter lengths, hence signal noise is also reduced. Nano-sized sensors allow data collection and measurements to be performed in real time at faster speeds. [28] The nano-scale also allows sensors to increase the active surface area, enabling the absorption and detection of analytes in low concentrations. [28] The integration of small-sized sensing devices lean to lower fabrication costs as large-scale production and reproducible fabrication can be achieved when producing nanosensors. [28] Moreover, conventional sensors are bulky and require higher amounts of power to operate. In gas sensing, neither a large sensing surface or a large sample is required to get a readable output signal from the sensor. Power consumption can be saved by reducing the thermal mass of the sensor. [28] Furthermore, if several gases are to be detected, an array of several gas sensors are to be assembled into an array. A multi-gas sensor array can increase the size and cost, whereas an array of gas nano-sensors (each functionalized to detect a specific analyte) can be implemented into a single device. [28] Nano-sensors can be classified by the kind of energy or physycal phenomena that is detected, as depicted in Table 1.2 for instance: biological, mechanical, thermal, chemical, and optical sensors. [28, 72]

Carbon nanowires have been fabricated with a photoresist by multiple-photon polymerization techniques. However little is known about polymers that can produce conductive carbon nano-wires after pyrolysis, as it is generally believed that most polymers do not form significant amounts of graphitic carbon when carbonized. In the past years, photopolymerization processes have been applied to the fabrication of nano-structures with the use of an epoxy based photoresist. [73] Photopolymerization techniques deliver

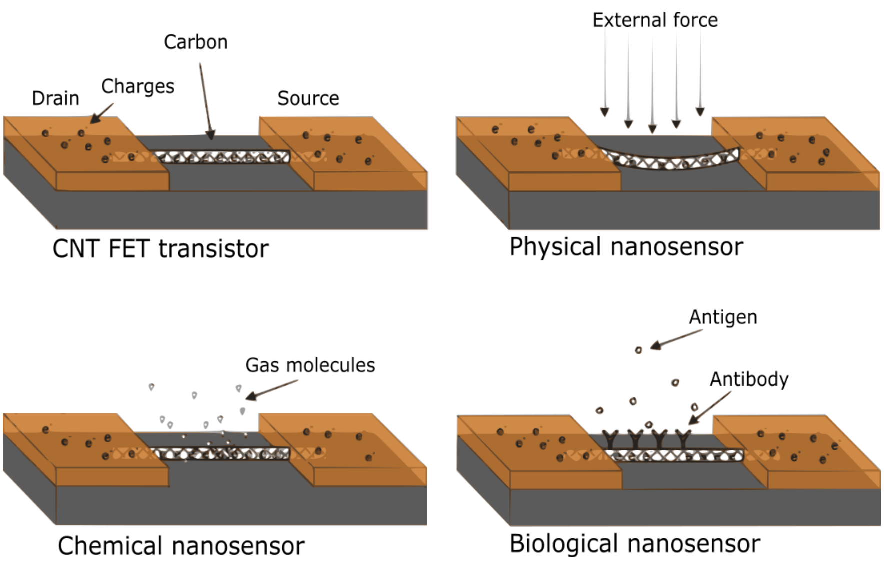

FIGURE 1.7: Diagram examples of carbon-based nano-sensors. Adapted from [72]

patterning resolutions with nano-scale tolerances through two-photon lithography for the production of highly detailed structures [74].

On the other hand, electrospinning has been acknowledged as a process with promising results at nano-structure fabrication [73], yet there is little research regarding the implementation of electrospinning for the fabrication of carbon nano-wires. Electrospinning has the potential to be a more straightforward process for the design and fabrication of nano-structures, as it can achieve mass scale manufacturing in a continuous, simple and reproducible manner. Cardenas [9] showed that electrospinning can be implemented with ease for carbon nano-wire synthesis. Mechano-electrospinning, a new variant of electrospinning shows promising results in the production of ordered carbon nano-wires. As stated in [9], mechano-electrospinning is an early technology invention and brings new challenges, such as the reproducibility of carbon nano-wire production. Furthermore, the study of a new fabrication process to produce carbon nanowires that involves mechano-electrospinning will enable spatial control of the structures' patterning.

Since electrospinning seems to be a better alternative for carbon nano-wire fabrication processes; and for that purpose of its implementation, it is required to develop polymer solutions that can be mechano-electrospun, photopolymerized and pyrolyzed into conducting carbon nano-wires. Carbon nano-materials have been subjected to research due to their various

TABLE 1.2: Classification of Nano-sensors. Adapted from [28]

Classification	Phenomena / Energy
Mechanical	Position, acceleration, stress, strain, force, pressure, mass, density, viscosity, moment, torque
Acoustic	Wave amplitude, phase, polarization, velocity
Optical	Absorbance, reflectance, fluorescence, luminescence, refractive index, light scattering
Thermal	Temperature, flux, thermal conductivity, specific heat
Electrical	Charge, current, potential, dielectric constant, conductivity
Magnetic	Magnetic field, flux, permeability
Chemical	Components (identities, concentrations, states)
Biological	Biomass (identities, concentrations, states)

potential applications in diverse areas that take advantage of the nano-scale properties. [8] Carbon nano-materials are suitable for the catalysis, adsorption, carbon capture, energy and hydrogen storage, drug delivery, bio-sensing and cancer detection. [8] However most applications are not currently feasible due to the lack of a continuous, simple and reproducible fabrication method with inexpensive processes. With the newly designed polymer solution, it would be possible to produce carbon nano-wires in large quantities, and therefore more applications will become feasible. On the other hand, the new technique will overcome some limitations of other methods such as lithography currently has. For instance, patterns created by lithography processes cannot be originated, only replicated, all constituent points of the pattern can only be addressed at the same time, and the process requires the pattern to be encoded into a mask. [75]

1.3 Hypothesis

The rheological properties of polymer solutions along with synthesis parameters (stage velocity, voltage, dispense rate) can be amended through rheological analyses to obtain a low voltage electrospun-able, photopolymerizable and graphitizable fibers for the fabrication conductive of carbon nano-wires with specified dimensions (diameter and length). The rheological properties of polymer solutions along with synthesis parameters are to be amended by replacing the PEO (Poly(ethylene) oxide) component within the existing polymer solutions described in Flores [11] and Cardenas

[9] work. PEO is to be replaced as its only purpose is to allow the electrospinning process to take place, but no benefit is obtained from it after pyrolysis.

1.4 Research Questions

- Is there any evidence of conductive carbon nano-wire fabrication though electrospun-able and pyrozable polymer solutions?
- What are the process parameters to consider/control for the fabrication processes of carbon nano-wires?
- What rheological properties are to be controlled/tested to deliver an electrospun-able and pyrozable polymer solution?
- Are there any efforts employed to the design of polymer solutions that can be electrospun, photopolymerized, and pyrolyzed into conducting carbon nanowires?
- What are the optimal fabrication parameters for the synthesis of carbon nano-wires through near-field electromechanical spinning?
- What materials can be used to ease the electrospinning process and favor the carbon nano-wire properties after pyrolysis?

1.5 Objectives

1.5.1 General objective

Study the practice and feasibility of a new fabrication process to achieve mass scale manufacturing of polymeric micro-wires in an inexpensive, continuous, simple and reproducible manner by the integration of near-field electromechanical spinning (NFEMS) and forward-thinking on pyrolysis processes to convert polymeric micro-wires into carbon nano-wires.

1.5.2 Specific objectives

- Design polymer solutions that can be electrospun by NFES, photopolymerized, and then pyrolyzed.

- Through rheological analyses, determine if polymer solutions can be easily employed for conducting carbon nano-wire synthesis.

- Determine and control the polymer solution rheological properties along with the process parameters of carbon nano-wire synthesis.

- Discover a PEO-similar material to allow the electrospinning process as well as input favourable properties to the carbon nano-wire yield.

1.6 Dissertation Outline

Chapter 2

Near-Field Electrospinning as an Affordable Way to Gain Spatial Control

2.1 Review of Polymer Solutions for NFES with Spatial Control

2.2 *conclude with a NFES fabrication parameter baseline to yield the desired fibres*

Chapter 3

Selection of Compatible Polymer-Solvent Combinations for Near-Field Electrospinning and Pyrolysis

3.1 Selection of Candidate Spunable Polymer Solutions

3.1.1 Rheology of candidate polymer solutions

3.2 Effect of aromatic groups in oxygen-free polymers in NFES and Pyrolysis

3.3 *conclude with a collection of potential spunable polymer solutions*

Chapter 4

Fabrication and Characterization of Polymeric Fibers through Near-Field Electrospinning, and Forward-thinking on Photopolymerization and Pyrolysis

4.1

4.2

4.3 Fabrication and Characterization of Legacy SU-8 carbon fibers

4.4 Comparison of the Obtained Polymer Fibres Against SU8-based Carbon Fibres and Potential Applications

4.5 *conclude with fibre morphology before and after pyrolysis. determine best pyrolysis process*

Chapter 5

Concluding Remarks

5.1

5.2 Future work

Acronyms and Abbreviations

CEM	**C**ampus **E**stado de **M**éxico
CNWs	**C**arbon **N**ano-**w**ire**s**
DC	**D**irect **C**urrent
EMS	**E**lectro**m**echanical **S**pinning
FFES	**F**ar **F**ield de **E**lectro**s**pinning
ITESM	**I**nstituto **T**ecnonólogico y de **E**studios **S**uperiores de **M**onterrey
MA	**Ma**ssachusetts
MEMS	**M**icro**e**lectro**m**echanical **S**ystems
MNT	**M**aestría en **N**ano**t**ecnología *(Master of Science in Nanotechnology)*
MTY	**M**on**t**errey *or* Campus **M**on**t**errey
NFEMS	**N**ear-**F**ield **E**lectro**m**echanical **S**pinning
NFES	**N**ear **F**ield de **E**lectro**s**pinning
USA	**U**nited **S**tates of **A**merica
UV	**U**ltra**v**iolet

Variables and Symbols

Symbol	Name	Unit
ω	angular frequency	rad

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EDUCATION

TECNOLÓGICO DE MONTERREY
MSc in Nanotechnology
Jan 2019 - Dec 2020 | Estado de México, MX

TECNOLÓGICO DE MONTERREY
BS in Digital Systems and Robotics
Aug2012 - May 2016 | Querétaro, MX
Cum. GPA: 3.6 / 4.0

LINKS

Github:// **katagirimx**
LinkedIn:// **Osamu Katagiri-Tanaka**
Personal Website:// **katagiri-mx.com**

COURSEWORK

GRADUATE

Thermodynamics of Materials
Nano-structured Materials
Plastics and Composites Engineering
Rheology & Electrospinning

UNDERGRADUATE

Sensors
Control Engineering
Digital Systems
Computer Architecture
Embedded Systems
Web Application Design
Microcontrollers
Electric Circuits

SKILLS

PROGRAMMING

Over 5000 lines:
Python • Javascript • LaTeX
Over 2000 lines:
C • C++ • ADA • Verilog • VHDL
Over 1000 lines:
Java • CSS • PHP • Assembly
Familiar:
Android • MySQL

EXPERIENCE

GE AVIATION | Embedded Software Eng.
Jun 2018 - Dec 2019 | Querétaro, MX

- At General Electric's Business & General Aviation Power Software team, I develop and test critical software for Aviation Power products. I have high responsibility in the development and in the documentation of the features and interactions with other systems.

GE AVIATION | SW Edison Engineering Development Program
June 2016 – May 2018 | Querétaro, MX

- EEDP is an intensive program for people who have a passion for technology, a drive for technical excellence, and share in GE's core values. It is designed to accelerate participants' professional development through intense technical training.

GE POWER | Software EID Intern
May 2015 – May 2016 | Querétaro, MX

- Support and improve engineering projects and activities.
- Worked on the analysis and optimization of +20 wind turbines for every GE wind farm worldwide.

RESEARCH

MACROPHOTOSCIENCE RESEACH GROUP | MSc Student
Jan 2019 – Dec 2020 | Nuevo León, MX
Worked with **Phd. Alan Aguirre** and **Phd. Dora Medina** to determine the electro-spunability of various polymer solutions for the fabrication of carbon nano-wires.

AWARDS

May 2018	top 4%	Software EEDP graduate at GE Aviation
Aug 2015	1^{st}/1000	GE 9th Lean Challenge
Nov 2014	1^{st}/50	GEIQ's Robotics Project

PUBLICATIONS

[1] Saeed Beigi-boroujeni, Osamu Katagiri-tanaka, Braulio Cardenas-benitez, O Sergio, and Alan Aguirre-soto. Pyrolytic Carbon from Novolac Epoxy Resin Compressed before Photocrosslinking and Pyrolysis. *Materials Today: Proceedings*, 2020.