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.
- Where any part of this dissertation has previously been submitted for a degree or any other qualification at this University or any other institution, this has been clearly stated.
- Where I have consulted the published work of others, this is always clearly attributed.
- Where I have quoted from the work of others, the source is always given. With the exception of such quotations, this dissertation is entirely my own work.
- I have acknowledged all main sources of help.
- Where the dissertation is based on work done by myself jointly with others, I have made clear exactly what was done by others and what I have contributed myself.

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

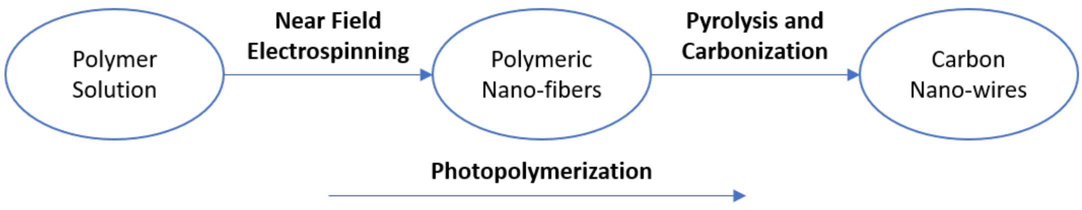


FIGURE 1.1: Fabrication process and characterization techniques of conductive carbon nano-wires to achieve through the dissertation.

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

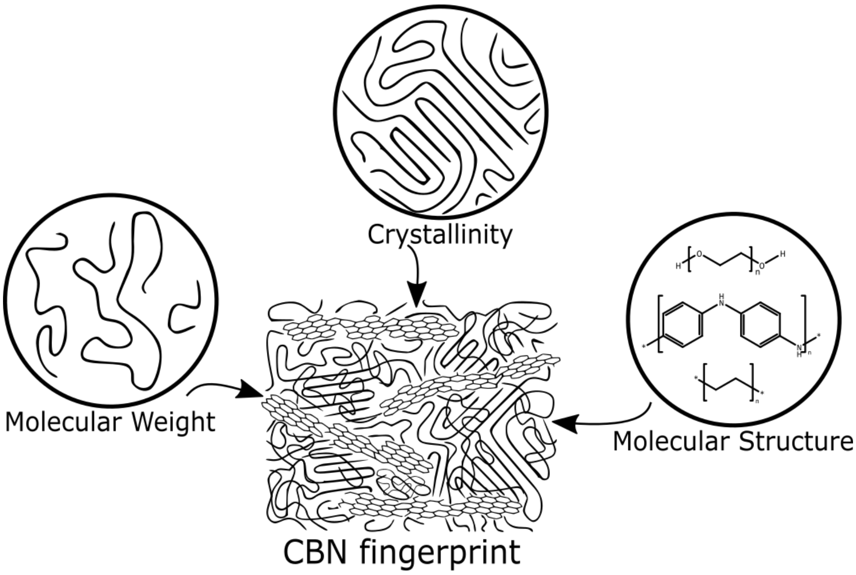


FIGURE 1.2: Molecular to mesoscale structural features of synthetic polymers influence the emergence of specific microstructural features in polymer-derived carbon materials after pyrolysis.

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

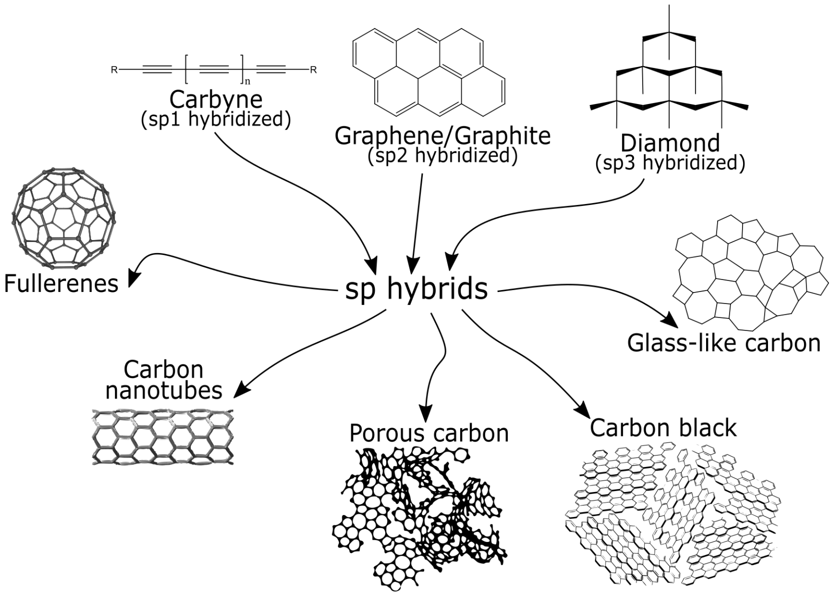


FIGURE 1.3: Three carbon allotropes (diamond, carbyne and graphene) are the building blocks of additional deriving carbon types such as fullerenes, porous carbon and glass-like carbon.

connected to the outer surface of the solid, in other words a 'open pore' is a '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

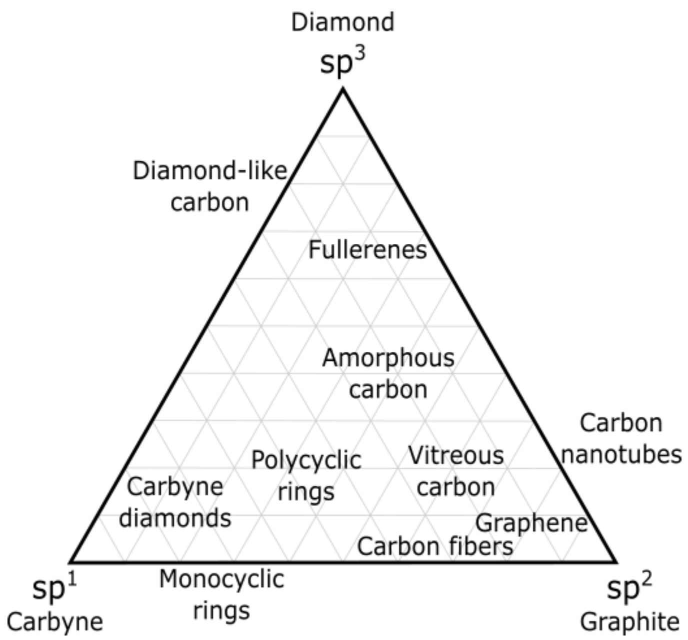


FIGURE 1.4: Ternary phase diagram of amorphous carbon regions based on hybridization degree. Adapted from [16–22].

electrochemical and mechanical properties. [23, 24, 29] In this regard, carbon nano-structures, such as nano-wires [30, 31], have been fabricated to achieve improved electrochemical characteristics.

### 1.1.2 Carbon nanowires

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 (CNF) have diameters of several micrometers and are different from carbon nano-tubes (CNT). [33–37]

### 1.1.3 Nanowires synthesis

Numerous methods to prepare nanowires, which include template-assisted synthesis [30], vapor–liquid-solid (VLS) [31], electrodeposition [32], electrospinning [33], hydrothermal [34], also hierarchical arrangement

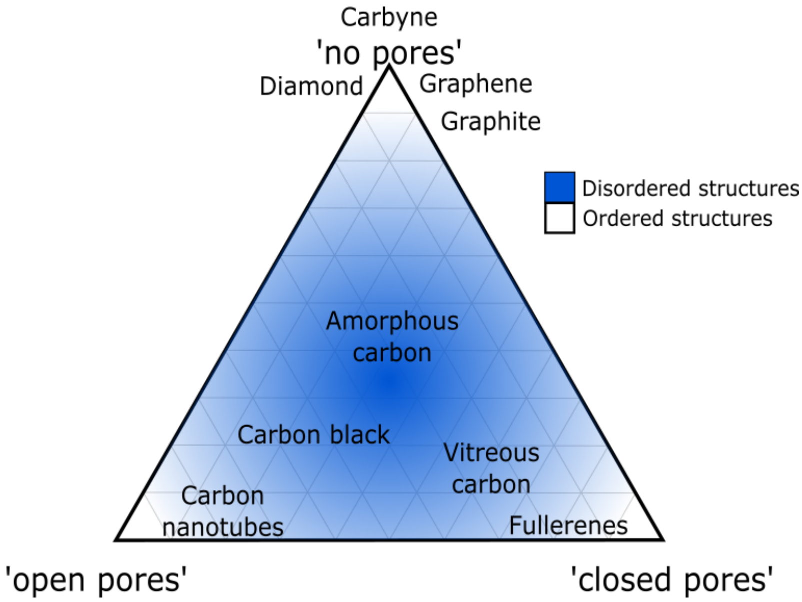


FIGURE 1.5: Ternary phase diagram of amorphous carbon regions based on structure order and porosity. Regions are colored by the degree of crystalline order within the carbon structure. White represents highly ordered structures, whereas white represents disordered structures. [23, 24]

techniques [35–37] to organize the nanowires have been studies in the last ten years. Nanowires based on organic, inorganic or hybrid materials have been applied in order to get single or composites nanomaterials for innumerous purposes, such as chemical and biochemical sensing devices, thermoelectric, optical, magnetic and electrical application. In this section, we are focusing on the synthesis of conducting polymer and composites to develop materials in nanowires architectures.

**Synthesis of conducting polymer nanowires**

### 1.1.4 *conclude that NFES is the way to go*

## 1.2 Problem definition and motivation

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. [38] Photopolymerization techniques deliver patterning resolutions with nano-scale tolerances through two-photon lithography for the production of highly detailed structures [39].

On the other hand, electrospinning has been acknowledged as a process with promising results at nano-structure fabrication [38], 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 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. [40]

## 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 carbon nano-wires in an inexpensive, continuous, simple and reproducible manner; by the integration of mechano-electrospinning technique.

### 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**erre**y** |
| **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 |
|---|---|---|
| $\omega$ | 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.