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I, Bradley M Ruff, hereby submit this original work as part of the requirements for the degree of Master of Science in Mechanical Engineering.

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Carbon Nanotube and Nanoparticle Materials for Electromagnetics Applications

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Carbon Nanotube and Nanoparticle Materials for Electromagnetics Applications

A Thesis submitted to the
Graduate School
of the University of Cincinnati
in partial fulfillment of the
requirements of the degree of

Master of Science
in the Department of Mechanical Engineering
of the College of Engineering
by
Bradley Ruff
i. Abstract

The primary goal of this thesis was to do preliminary investigation into a variety of promising ideas that could lead to the development of Carbon Nanotube (CNT) and nanoparticle materials that would have applications in electromagnetic devices and motors. Recommendations on which ideas should be investigated further are made based on the qualitative and quantitative results. The use of electrostatic charge can be used to control the diameter and spacing or Fe/Co catalyst particles. This could be used to help in the consistent production of spinnable CNT arrays which rely on a specific structure that involves the consistent spacing of high quality CNTs. These spinnable arrays can then be used to produce CNT fibers. The fiber structures can be modified to a higher density and more consistent diameter using a controlled chemical densification process. These changes in structure are consistent with improved mechanical and electrical properties. In addition, a magnetic polymer nano composite can be made by mixing Iron Oxide nanoparticles into commercial polymer matrixes to provide a ridged or flexible magnetic composite that could possess superparamagnetic properties. These new materials have the potential to change the way that electromagnetic devices and motors are designed. A proposed design is shown that takes advantage of the desired properties of both the CNT thread and magnetic composite materials.
ii. Acknowledgements

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1. Introduction

1.1. Introduction to Carbon Nanotubes and Nano Materials

One of the biggest differences between the engineering in the modern world and engineering in ancient Rome is materials. The biggest advances that support our civilizations and lifestyles are supported by the advancements in materials. Skyscrapers were enabled by steel, bullet proof vests by Kevlar, computers by silicon technology. With our world racing at an ever increasing rate in a direction of unsustainability, materials development will become even more important. Many of the things that we take for granite in the 21st century are undeniably finite. Oil, for example, is the trendiest of these. Regardless of the debate over global warming and greenhouse gasses, petroleum reserves are not unlimited and we are consuming them at an increasing rate. Even though we likely have several decades of oil reserves we may soon hit peak oil production and the price will start increasing. Many of the resources we consume are not considered ‘rare’, but they are finite.

There is very little materials development for electric motors done today. This leaves the area prime for innovation. The late Richard Smalley envisioned infinitely long cables of carbon nanotubes used to transmit power across the world [1]. This is due to the fact that CNTs have the potential to be more conductive than any metal wire could be [2]. This is not only an advantage for power transmission but could also be revolutionary in electric motors. If copper could be replaced with a conductor that has lower resistance that would increase both efficiency and power in all types of motors.

Individual CNTs possess incredible properties that, if realized, could change everything from airplanes to power lines. Individual CNTs have been shown to have up to 1 TPa modulus of elasticity, and 50 GPa strength [2]. This could lead to super strong composites that allow airplanes and cars to be stronger and lighter. In addition to incredible mechanically properties, CNTs have been shown to have electrical
conductivities higher than copper[2]. High conductivity means that electrical power can be transmitted across long distances more efficiently, as well as the production of more efficient and powerful electrical devices. The motivation to mass produce a high quality CNT fiber is more than evident and the attention that is given to the research in these areas is well deserved.

1.2. Current Electromagnetic Device Technology

Electrical power is a leading candidate to solve the fossil fuel problem. It’s an easy and logical step to remove the combustion motor and gas tank, and replace it with an electric motor and batteries. This is the best commercially viable configuration to date. With electric motors and batteries being the popular choice for future machinery most material people tend to focus on the later. Battery technology still has a lot of room for improvement so a large number of resources are devoted to the development of new battery technology. In contrast, electric motors work pretty well. Many operate with greater than 90% efficiency. The best materials used for this are extremely well established, copper conductors, iron magnetic cores, and ‘rare earth’ permanent magnets. These materials have been relatively unchanged in electric motors for the last decade.

Another major component of most electric motors is a ferromagnetic core material that increases the strength of the magnetic field. Iron works well because it supports very high magnetic field strength. One problem with iron magnetic core materials is they suffer from parasitic hysteresis and eddy current losses that limit their efficiency. They combat this by making thin laminations of iron and stacking them with insulating materials. This eliminates the electrical conduction between layers and minimizes eddy current losses. However, in high frequency operations the magnetic losses can still be considerable. The other disadvantage of iron is that it is a very dense material and adds considerable weight to any device. So a lighter material with very low conductivity could be extremely valuable as an Iron replacement in electric motors. Many modern electric motors use permanent magnets as part of the machine.
Permanent magnets have become increasingly popular as so called ‘rare earth magnets’, which are much stronger than traditional magnets, have become more affordable. Rare earth magnets use different elements[3]. However there are some political reasons why these might not be the best path for the future.
2. Materials Background

2.1. Carbon Nanotube growth

The popularity of Carbon Nanotubes as a research topic started with two publications that both appeared in 1993. Iijima and Ichihashi [4] independently both published articles that they had independently created and observed single wall Carbon Nanotubes. There were previous discoveries of nano scale 1 dimensional carbon structures dating back to 1952. However, they were all large diameter (~15 nm) and Multiwall (15-20 walls) CNT [5]. The major reason why this discovery did not make a major impact on the scientific community is that it was published in the Journal of Physical Chemistry of Russia and not translated into any other language. Despite the lack of proliferation of this discovery, Radushkevich and Lukyanovich were the first to observe nano-size, hollow, carbon structures [6].

One important step for CNTs was the discovery of buckminsterfullerene by Kroto et al. in 1985 [7]. They found that after laser evaporation of carbon that there was a reoccurring molecule with 60 carbon atoms. They later discovered that this molecule was extremely stable, round and symmetrical. The structure was that similar to that of an American Soccer ball. This discovery of a third allotrope of Carbon attracted a large amount of attention in nano-scale engineering and science. It was also the time when advances in transmission electron microscopy allowed for images with true nanometer resolution. During the 21st century Carbon Nanotubes have been a major area of scientific research. There are now many different ways to synthesize CNT and each has its own advantages and disadvantages. The approaches that are focused on here are for mass production of macro amounts of CNT materials, namely aligned vertical arrays. Growth of CNT is a truly bottom up approach to material fabrication.

2.1.1. Chemical Vapor Deposition

Chemical Vapor Deposition is a process where gaseous compounds are flowed into a reaction chamber where the conditions are controlled to facilitate the construction of different micro and nano scale
structures. At Nanoworld in the University of Cincinnati, there are two different CVD furnaces that are used to grow CNT arrays. The first is a First Nano EasyTube 1000, which has a 2” quartz tube. The Easy Tube 1000 has the option of using 4 different gases which is generally Argon, Hydrogen, Ethylene, and then Argon run through a bubbler to pick up water vapor. It only has one temperature zone that can be heated to 1000 deg Celsius by an electric resistance heater. Unlike some other CVD machines it has no ability to control or measure the pressure in the reaction chamber. The reactor doesn’t have the level of control that the new reactors have so it is generally used more for proof of concept experimentation. However, the EasyTube 1000 at UC was used to grow the world record length vertically aligned CNT Array [8].

![Figure 2.1. First Nano CVD EasyTube 1000 furnace. First Nano EasyTube 300.](image)

The other Furnace that is used at Nanoworld laboratories is a First Nano EasyTube 3000. This furnace has a much greater level of control over the EasyTube 1000. It has a larger tube so that it can handle larger substrates. It also features a secondary vacuum loading chamber that allows the substrates to be loaded into the furnace at a more controlled environment by limiting contamination. The main purpose of this furnace at UC is to produce highly aligned arrays for drawing or spinning into threads and sheets. These spinnable arrays are a very specific subset of vertically aligned CNT arrays that are only made with CVD synthesis. All the factors that contribute to an arrays spinability are not fully understood. One feature that is widely believed is that the CNTs in the array must be highly order and aligned. This can be accomplished by adjusting the growth parameters or recipes through extensive trial and error.
2.1.2. Plasma Enhanced CVD and Electric Field CNT alignment

CVD is used often in the processing of silicon and Integrated circuits. Some of the components that require CVD can't handle the high temperatures that are associated with traditional CVD. So Plasma Enhanced CVD (PECVD) was developed to allow lower temperature processes, down to room temperature in some cases [9]. It uses DC or AC plasma, usually driven by microwaves, to give the vapor molecules the energy they need to dislocate and form the desired chemical structure. These machines can be used to grow CNTs just like the traditional CVD machines. Using PECVD, CNTs have been grown as low as 500 °C [9].

It has been shown before that electric fields can be used align CNTs both during growth and while suspended in solution [10–12]. The CNTs are excellent conductors and the electrons to flow freely in the tube. In the presence of an electric field, the electrons are all pulled towards the positive electrode and this creates a di-pole. See Figure 2.2. The resulting force of the di-pole in the electric field aligns the CNTs with the field. There is also evidence that the type of CNT and some other growth parameters can be controlled by the electric field strength and polarity [13,14]. The PECVD system provides this field in either DC or AC and has been shown to grow individual, free standing vertical CNTs, which are not supported by neighboring tubes [9]. However the process and the role of the plasma aren’t well understood and the resulting arrays are on the order of tens of microns tall.

2.2. Carbon Nanotube Thread Spinning

When something is considered ‘Nano’ that means that at least one dimension is on the nanometer scale. In the case of CNTs the diameter of the tubes is in nanometers, and the length can be in centimeters. This leads to aspect ratios on the order a million. These structures have been used for various applications in there as-produced form. Raw CNTs can be used in composites and for structures like in electronic circuits or battery electrodes[12,15]. These take advantage of the vastly superior strength, and electrical properties of the individual CNTs. However, powder CNTs can only be used in very small volume fractions in traditional composites. Likewise the use of CNTs in electronics is application limited not yet cost effective compared to traditional materials.

If a bulk material could be made that reflected the mechanical and/or electrical properties of the individual tubes it would have endless applications. The material could be either pure CNTs or a composite material. On the other hand, having building blocks of this scale can cause major technical
challenges in the production of macroscopic bulk materials. There are several different methods that have been established to make CNT based fibers. The first successful method of making continuous fibers was done through a solution process [16]. However this leaves residue of the compounds used for spinning in the thread. Another method of spinning was discovered that involves pulling the CNTs from the side of an array and twisting it to make a fiber[17]. This process produces a pure CNT fiber. One method that has already reached full commercialization involves growing CNTs via a floating catalyst method. The floating CNTs, also known as Aerogel, are then coagulated and pulled out of the furnace to produce a continuous fiber. Only the first two methods of spinning are performed at the University of Cincinnati and will be discussed here.

2.2.1. Liquid Spinning

The first successful method of producing continuous CNT fibers was done using a liquid coagulation method. Liquid coagulation is currently used to produce Kevlar and other commercial fibers. It involves dissolving the CNTs in a solution and the extruding that solution into another that forces the CNTs to agglomerate and form a fiber[2]. This process makes very neat fibers but is limited to using short tubes currently. There are many different varieties that have been proven by different groups. The first method that was published used Sodium Dodecyl Sulfate to disperse the tubes and then injected the CNT solution out of a syringe into a bath of beaker PVA solution[16]. This process was scaled up to be able to produce continuous fibers[18]. Instead of a syringe they used co-flowing streams of the two solutions through a long tube and a collection spool on the other end. There have been other variations of this method but they all use similar methods and produce similar results[2]. The downfall of this method is that they always leave a polymer residue in the thread that limits the mechanical and electrical properties of the thread.
There is a different type liquid spinning that doesn’t have the same residue problems as the polymer based processes. It involves using an acid to dissolve the tubes. This has been shown to produce very high quality CNT thread [19]. The original method used 102% concentration sulfuric acid also known as ‘super acid’. This super acid/ CNT solution is then extruded in to a bath through a capillary tube and then collected. This process has yielded the highest conductivity thus far for CNT fibers[20]. The acid does cause some defects in the tubes and is limited to short tubes. So this method shows good potential but still has room for improvement.

![Fibers form in coaxial flow field.](image)

**Figure 2.3.** Continuous liquid spinning done at University of Texas Dallas. Super acid spinning done at Rice University. [1] Adams W. W., Green M. J., Behabtu N., and Pasquali M., 2008, “revolutionary enough to save the world ? Professor Richard E . Smalley,” Engineering.

### 2.2.2. Dry Spinning

One of the most basic ways to make a cotton thread is to pull the boll of a cotton plant and the individual cotton fibers begin to align and form a long thin strand that can be twisted into a fiber. In 2002, Jiang et. al. discovered that highly aligned vertical arrays could be pulled from the side and they would form a continuous web that could be twisted to make a fiber[17]. This method produced a fiber that contained nearly 100% CNTs without inducing any defects and also a high degree of alignment. The type of highly aligned vertical array that can be spun into a thread is referred to as a spinnable array. These spinnable arrays are have a certain structure  that allows the CNTs to slide relative to each other
yet pull the next adjacent tube to produce a continuous web. That structure is very specific and not well understood so it is a challenge to produce in a repeatable manner. Because of the potential of this method, a huge effort from the engineering community has taken place to increase the properties and scale up the process[2].

At the University of Cincinnati there are two different style of spinning machine. The older one, shown in Figure 2.4, is a clevis that contains a reeling motor and then the whole clevis spins to produce twist. This machine is simple and reliable. It uses a small dc gear motor to do the reeling. This gear motor has a limited range of speeds. The twist is powered by a brushless dc motor and controlled manually by adjusting a dial on a motor controlled. This machine is limited in speed by the rotation speed. The motor can only spins so fast and the slip ring that supplies power to the reeling motor is only rated to a certain speed. The machine could be upgraded to spin faster but because the reeling rotation axis is perpendicular to the twist axis it introduces dynamic, gyroscopic forces that would make the machine vibrate and introduce high forces on the bearings and other components. These speed limitations mean that only moderate twist to length ratio and production speeds can be achieved with this machine.

The newer one, shown in Figure 2.4, is a ring spinning machine that uses a ring rotating with the collection bobbin. The differential rotation speed of the ring and the bobbin produces the reeling of the thread. This machine can operate at much higher speeds because the reeling axis and the twist axis are concentric. This can allow the machine to pull the thread faster and still maintain high twist to length ratios which the older machine cannot do. The twist axis is driven by a synchronous AC motor and geared to increase the speed via a timing belt and pulleys. The motor that controls the reeling is a small stepper motor that sits inside the shaft. The two axes are concentric so the reeling motor has very low stress on it during spinning.
Figure 2.4. Low speed spinning machine at UC. High speed spinning machine at UC Built by Charles Dandino and Joe Sullivan.

The worldwide effort to increase the properties and scale of dry spinning has been fruitful to a degree. There are a handful of labs that can produce these unique arrays and all have developed methods for spinning them. Many of these labs use a spinning style known as array spinning [21,22]. Array spinning requires a machine that is much less complex than other types of spinning. The twist comes from spinning the array on a platform and then reeling the thread on to a bobbin that is separate from the twisting platform. This also lends its self well to inline processing of the CNT yarn. By passing the fiber around a series of rods it increases the tension during twisting and increases the strength [21,22]. It is also possible to infiltrate polymer during this spinning [23]. The strongest dry spun fiber produced to date had an ultimate strength of 3.3 GPa. What made this fiber so strong was that it was spun from a very tall (1mm) spinnable array [24]. The dry spinning method is still being improved and may prove to be the preferred method of producing CNT based materials in the future.

2.3. Carbon Nanotube Sheet

Carbon Nanotubes are referred to as one dimensional carbon structure because they generally have a length that is much larger than their diameter. This makes them ideal for making one dimensional macro sized materials, like threads and yarns. However, there are some times when one dimensional materials,
like threads and yarns, don’t suit the application as well as a two dimensional material, like a sheet or film. Threads can be woven into a fabric to form a two dimensional material like cotton fabric used for clothes. This is extremely time consuming and uses a large amount of thread material. Bucky Paper is a thin layer of randomly oriented CNTs that is made by dispersing CNTs in a solution and filtering it to remove the liquid. This produces in plane isotropic electrical conductivity and strength. This can be good for filters and electromagnetic shielding. There is another method of making aligned CNT sheet that starts with drawing the CNTs from the side of the array, similar to dry spinning. Instead of twisting the web into a fiber the web is wound onto a belt or mandrel continuously until a solid sheet is made. The alignment of the CNTs in this sheet method produces a much stronger sheet in the direction of the tube alignment than in the transverse direction.

### 2.3.1. Bucky Paper

Bucky Paper is a name for a very thin layer of randomly oriented CNTs that form a paper like structure. It is usually fabricated by dispersing CNTs in a solution with surfactants and then filtering that solution through a PTFE filter paper. Once the solution is filtered the remaining network is rinsed with solvents to remove the surfactants. This process is used to fabricate up to 50 um thick layers of CNTs[25]. Another method of making CNT sheet material is done by Nanocomp. They use a floating catalyst method that can be used to make yarns as well. They collect the aerogel of CNTs onto a rotating conveyor belt and this forms a film of CNTs with random orientation[26]. See Figure 2.5 for images of different bucky papers.
Figure 2.5. SEM image of Bucky paper made with filtering. Nanocomp sheet manufacturing.


These sheets have good electrical and mechanical properties in their as made form. However, there are some additional techniques that can be done to increase the strength and/or electrical properties of the sheets. The random oriented CNTs in the bucky papers can have up to 70% air space in them. This space can be filled with a polymer to make a composite material [25]. One method of infiltration is to dilute epoxy resin in acetone and then soak the bucky papers in the solution. These Buck Paper/Epoxy composites can have Elastic Moduli up to 15GPa [27]. The bucky papers possess very isotropic properties in the in-plane directions of the sheet. This is good for some applications but for others anisotropic strength maybe more desirable. The Nanocomp sheet is around 100 MPa in strength in both in-plane directions when it is fabricated. They have a stretching process that allows the strength in one direction to be increased to 1000 MPa [26].

2.3.2. Aligned Carbon Nanotube Sheet

There are some methods of making Bucky Papers with a degree of alignment. These include application of strong electrical or magnetic fields [28], as well as mechanical methods of combing or stretching [26].
The process of dry drawing CNTs from an array can also be used to produce two dimensional sheets [29]. When the web is pulled from the side of the array it is then wound onto a conveyor or mandrel. The conveyor is then wound until the desire thickness or number of layers is achieved. This method was first demonstrated by Zhang et. al. where they created meter long free standing transparent webs of CNTs. These webs showed good strength and electrical properties as well as hydrophobicity and the ability to produce polarized light [28]. The CNT webs have been shown to have commercial applications in thin film transparent conductors used in touch screens, TEM grids, artificial muscles and even loud speakers [30].

In addition to ultra-thin conductors and support structures, CNT sheets have the ability to have high strengths. This can be done in the form of pure CNT sheets [29,31,32] or in the form of a polymer composite [33]. One process of producing aligned CNT sheets from spinnable arrays is done by winding the web of CNTs on to a cylindrical mandrel. The array is moved back and forth to determine the width of the sheet and the circumference of the mandrel determines the length [31,32]. This is an effective way to produce small to moderately sized sheets. Generally the mandrel is covered by a Teflon film that can be removed from the mandrel to produce a flat sheet of CNTs and also allows for easier removal of the CNTs from the mandrel. This process inherently produces a pure, aligned structure of CNTs that is not very dense and contains a large volume of empty space. This space can be removed by a densification process that involves soaking the sheet in a volatile organic compound (VOC) and allowing the VOC to evaporate. The surface tension pulls the CNTs together to reduce the empty space and to increase CNT interaction[29,31]. Another possibility is to fill these voids with a polymer to create a composite. This can be done with a simple spray method where a mixture of VOC and polymer is sprayed on the sheet while is being assembled. Once the layering of the CNTs is done and the VOC is evaporated, the sheet is mechanically compressed by another mandrel to remove voids and densify the sheet.
At The University of Cincinnati the process of making aligned CNT sheets from spinnable arrays has been improved and is not be limited by the diameter of a cylindrical mandrel. Instead the CNTs are wound onto a Teflon sheet that is wrapped around a pair of rollers like a conveyor belt. This allows different lengths of sheet to be made on the same machine by varying the spacing of the rollers[34]. It also allows additional processing of the material due to the conveyors flat surface.

*Figure 2.6. First CNT Sheet machine. Second Sheet machine. Jayasinghe C., 2011, “Synthesis and Characterization of Carbon Nanotube, Threads, Yarns, and Sheets,” University of Cincinnati.* [34]
3. Materials Development

3.1. Electrostatic and Magnetic Assisted Synthesis

When growing CNT arrays using CVD, there are a huge number of factors that dictate the morphology of
the array and the CNTs that comprise it. There has been a huge effort in the past decade to fine tune all
the parameters like temperature, gas concentration, carbon source, catalyst, substrate, etc to produce
the desired CNT arrays. Different combinations of these ingredients, also known as recipes, will produce
CNT arrays with different structures. However, the recipes do not always produce perfectly repeatable
results. There is some very small variation in the gas mixtures, temp, substrate preparation, etc. that
leads to variation in the final products. There is also an unavoidable small variation between reactors. All
of this adds up to a constant need to tune and retune each specific reactor for any desired array
morphology.

Engineers and scientists with chemistry and material backgrounds are the front runners in the area of
CNT synthesis research. This is a logical and necessary pairing when trying to construct structures on the
atomic level. However, due to the lack of atom level control available in even the most advanced CVD
reactors there is always slight variation in the as produced arrays in each run. The other problem with
traditional CVD methods is that the mechanism of control over the structure of the arrays, tube
diameter, tube length, wall count, forest density, etc., is not fully understood. There is too much
interaction of parameters to simply modify one aspect of the synthesis method. For example, if two
arrays were desired to be exactly the same except for smaller tube diameter it would require a huge
amount of effort to tune two different recipes independently.

Cross discipline research has many advantages for innovation and breakthroughs. Science and
engineering have advanced to a level where it is becoming more difficult for one person have a broad
enough knowledge base to make a huge impact or breakthrough in a particular area of science. At
Nanoworld this cross discipline research environment is epitomized by close collaboration between mechanical and chemical engineers and students. This collaboration led to the idea that there could be additional parameters introduced that would give additional control over the morphology of the CNT arrays without having to modify the normal CVD parameters. It was hypothesized that the presents of electrical charge, electric field, and magnetic field may offer additional control over the growth of CNT arrays.

The use of electric field in the alignment of CNT arrays has been studied in the past [12,14]. However, the experiments were performed on the micron size scale and with only a few CNTs at a time. There is also Plasma Enhanced CVD where the substrate is bombarded with microwaves and the tubes align in the direction of the alternating electric field. The concepts put forth by the Nanoworld Nanodevice team were that the alignment, tube diameter, and possibly the chirality of the CNTs could be controlled by using electric and/or magnetic fields. Figure 3.1 shows diagrams of the initial concepts proposed. This first and most basic idea was that a static charge could control the diameter of the CNTs by preventing the catalyst particles from combining. If the CNT substrate had a static charge then if a second substrate were placed on the above the array then a parallel electric field would be formed in the direction of the CNTs in the array. This would hopefully make the tubes grow in a straight, parallel fashion along the Electric field lines as it did in Figure 2.2. Next using a set of coils there could be an aligned magnetic field introduced in the direction of the CNTs. Also a variety of alternating electric fields could be used to attempt to induce small currents in the CNTs. In this thesis there will only be the exploration of the electric field concepts. In the future the magnetic field/CNT interaction will be investigated.
3.1. Substrate Charging

Introduction

The first step in the series of experiments to investigate how electric field affect the growth of CNT is to apply a static charge to the substrate. The first hypothesis is that the charge will influence the catalyst to break down into smaller particles when it’s heated. In order to grow a CNT array a silicon substrate is generally prepared by first depositing 10 nm of Alumina (Al₂O₃) to the polished side of the silicon substrate. At UC they then deposit approximately 1 nm of iron and 1 nm of cobalt via E-beam on top of the Alumina [34]. The iron-cobalt layer is the catalyst for the growth of CNTs. A basic understanding of CNT growth starts with the thin film of metal catalyst that becomes semi-solid as it’s heated to a
temperature below the melting point of the metal. The film then breaks into small droplets that are not liquid but have enough energy to be mobile on the alumina surface. These droplets then absorb carbon from the feed gasses. The iron/cobalt particle then gets saturated with carbon and expels excess carbon in the form of a Carbon Nanotubes.

Coulomb’s Law that states that like charged particles repeal each other. For more information on charged particle interaction see Section 5.1. This could be applied to the catalyst as its heated and forms droplets. If the film had a net charge as I broke up, it might form smaller droplets on surface of the alumina. This might allow the size of the particle formation to be determined by the applied voltage, i.e. more charge leads to more repulsion and a greater number of smaller catalyst particles. A second effect of charged catalyst is that the CNTs are excellent conductors so they should take on the charge of the substrate. If the individual CNTs also repel each other, then this could influence them to be straighter and less entangled. Entanglement is one major factor in producing arrays that can be continuously pulled and spun to make CNT fibers.

In order to investigate the effect on catalyst particles, substrates were heated to a typical growth temperature and then cooled and removed. A charge was supplied from a DC power supply to the substrate via an insulated nickel wire and a vacuum chamber electrical feed through. The most difficult aspect of the substrate charging experiment was how to get the charge to the catalyst. The regular silicon that is used for CNT growth is not very conductive. In addition, the alumina layer is insulating so it would be difficult to get the charge through the silicon to the catalyst. It is also difficult to make a fixture that can survive the environment of the CNT furnace without mechanical or electrical failure.

**Proof of Concept**

Using the First Nano 1000 CVD reactor, a recipe for CNT was established that lead to high quality arrays with low amorphous carbon and high alignment. The recipes owe their origins to the work done by
Jayasinghe [34]. The recipe was capable of growing different height arrays by varying only the growth time. See Figure 3.3 for the Recipe used. Initially, there was an attempt to grow arrays on conductive, doped silicon wafer without an alumina layer. No arrays could be grown on the conductive wafers with or without the alumina layer. In order to progress on the experiments it was decided to continue with the traditional substrates and alumina layer. The recipe was used without change for the entirety of the proof of concept experiments.

![Figure 3.2. Recipe used for the initial charging investigation.](image)

Initially, the nickel wire was belt into a paper clip style configuration. See Figure 3.4. This allowed the charge to be applied to the surface of the substrate in order to bypass the insulating alumina layer and silicon substrate under the catalyst. The direct contact to the top of the substrate had the advantage of directly applying charge to the catalyst layer. One disadvantage is that the wire had a lot of elasticity and the substrate had very low weight. It was nearly impossible to position the substrate in the same position for each run. Another problem is that the wire was physically in the way of the CNT growth. It also blocked gasses from feeding the catalyst so the CNTs wouldn’t grow around where the clips were touching as shown in Figure 3.3. This approach could allow us to study the effect of charge on the catalyst formation, but would not be able to test how well the CNTs grew with charge.
In order to provide charge to the catalyst but not interfere with the growth of the CNT, the charge needed to come from the bottom of the silicon wafer. The conductive, doped silicon wafers were used without an alumina layer initially. However, without the insulating alumina layer, CNT array growth was decreased or eliminated and randomly oriented instead of a tall highly aligned array. It was then found that the traditional silicon wafers with an alumina layer still had enough conduction to transfer most of the charge through the substrate to the catalyst. This is due to the conductivity of the plain silicon being high enough to transfer static charge with minimal loss and the thin layer of the alumina likely having a small amount of defect or thin spots that allows the charger to transfer through it.

Using a polished strip of stainless steel as a base works well because of the corrosion resistance and the smooth surface maximizes the surface area of contact with the substrate. See Figure 3.4. When 50.5V were applied to the stainless steel base approximately 46-49V could be measure on the top surface of the substrate with a digital multi meter. So this setup could also be used to study the growth during charging. The initial results of the charged growth showed that positive charge increased the growth rate and negative charge decreased the growth rate. The SEM images of the sides of the arrays are shown in figures 3.5, 3.6, and 3.7. It also lead to a smaller catalyst particle formation shown in figure 3.8. These results were very encouraging even though they were based on a qualitative analysis and only a
few runs. In order to be confident that the results were accurate and to quantify them further experiments were needed.

**Figure 3.4** The stainless steel base configuration. The left is a picture before synthesis. The right picture shows the substrate after one run and the carbon that precipitates out of the gases.

**Figure 3.5.** Positively charged +50V. Height of 650,700µm. “SNM GOALI: Carbon Nanotube Superfiber to Revolutionize Engineering Designs”
Figure 3.6. No charge. Height of 360, 375µm

Figure 3.7. Negatively charged -50V. Height of 250, 310µm

Fixture Investigation

The charging base that was used in the proof of concept seemed to work well enough for the concept investigation. However, slight vibrations could cause the substrate to fall off or for the two substrates to make contact with each other. This wouldn’t serve very well for a more detailed experiment or for production use. Building on the original stainless steel base, a second stainless steel base design was used that used 2 long thin strips as electrodes so that the growth conditions could be identical for two samples. A schematic and picture is shown in Figure 3.9. This was effective in transferring the electric potential to the substrate. However, a major problem with the stainless steel strips as a base is that it moved around during the synthesis like the substrate in the clip did. This is again due to the nickel wire going through heat cycles and warping. The stainless steel base is however heavier than the plain array so the weight help to constrict the movement of the base to just sliding back and forth on the loading tray. This still poses a problem if we want to have two electrodes with
different potentials on the loading tray at the same time. If they came to close they would short and create sparks. This lead to the conclusion that, if the base were heavy enough and it had two electrodes that were at a fixed distance apart they would have minimal movement and not contact each other.

![Diagram](image)

**Figure 3.9** Diagram of the first charging base using thin stainless steel strips. Gray is the quartz loading tray. Black is the stainless steel. Red is nickel wire. Green the silicon substrate.

For the next version of the charging base a more complicated design was conceived with inspiration from layer silicon devices. The stainless steel worked well as an electrode however it lead to a significant amount of carbon precipitation after each growth cycle. The furnace tube and tray are made from quartz so this seemed like a good material to use as a base. We purchased some quartz microscope slides from Fisher Scientific to use for material. Then some Omega CC High Temperature Cement was obtained to act as filler and glue for all the pieces. The two stainless steel electrodes were placed between two quartz plates and then filled with the cement. The assembly went together the way that it was planned however the bonding between the cement and the other parts did not last when the assembly was subjected to the CNT growth environment. After one run in the furnace the cement crumbled and lost adhesion to the quartz and stainless steel. This is due to the lack of wetting between the quarts and the cement and also the difference in thermal expansion of the three different materials.
The next attempt used a less constricting method of holding the electrodes that didn’t include cement. It used two pieces of a machineable glass called Macor. These pieces were machined to have slots for the electrode. Then, in order to minimize the effect that the electrode had on the incoming gas, the ends of the electrode were hammered flat. This would minimize the cross section of the electrode at the point where the substrate was in contact with it. This produced a counterproductive result. The areas where the metal was worked spawned the growth of a huge amount of amorphous carbon, shown in figure 4. This must have been due to the irregular and rough surface of the stainless steel. It could also be due to the annealing and oxidizing that takes place during hot working of steel alloys. Whatever the mechanism of amorphous carbon formation it is very undesirable for this experiment. So the move to a smooth surfaced electrode was needed.
In order to keep the desirable material properties of the stainless steel but keep a low profile a stainless steel foil was used. However, the foil is harder to mechanically constrain in the furnace. A method of sowing the foil to a thin plate of Macor is conceived using a quartz thread. This thread is wrapped through holes in a Macor plate and then through corresponding holes in the stainless steel foil. The approach worked well for a short time. The quartz thread and the Macor developed a thin film of carbon after every run so a cleaning cycle was used between runs to avoid a buildup of carbon to the point of electrically connecting the two electrodes. There was another problem with this setup. The stainless steel foil started out extremely flat and smooth. However, after several cycles in the furnace the foil would start to warp due to thermal stresses and then it would not make good contact with the substrate. Instead of reading around 49V at the top surface of the substrate readings around 5-10V were observed. This meant that the electrodes had to be flat, smooth and rigid so that they would retain their characteristics.
In order to have a thin film of metal for conduction, a rigid base that wouldn’t warp after heating and cooling and something that wouldn’t accumulate carbon during a growth cycle it was determined that the best design of a charging base would be a flat quartz slide that had thin layers of metal deposited on it. Quartz was used as the substrate because of its extremely high melting point. It is also same material that is used for the furnace loading platform and tube. The metal electrodes were to be deposited only several hundred nanometers thick. First the quartz slide was masked with tape to create two separate electrodes, one on each side. From conversations with Alvarez and Shanov, chrome was chosen for the base layer because it forms a good interface with the quartz material. A 100 nanometer thick layer was deposited by e-beam on the quartz in the UC clean room. However, after a few experiments, chrome on its own didn’t last very long. It quickly faded in the furnace at the growth temperature of 750 C. In order to have a strong adhesion to the chrome but also protect the chrome from fading a second layer of gold was added to the chrome base layer. Gold was chosen because it is extremely conductive and also has a melting point much higher than chrome and the growth temperature. A diagram and picture of the charging base is shown in Figure 3.13. This configuration worked well in the furnace and was used to carry out more base charging experiments.
Experiment

Using the charging base shown in Figure 3.13, additional charging experiments were performed in order to confirm and quantify the results shown in Figure 3.8. The polarity of the voltage was found to not affect the catalyst particle formation so only positive voltage was tested in the new experiments. In order to help quantify the effect of the charge on the catalyst a series of voltages (0, 30, 60, 90, and 120) was used. Two experiments were performed at each of the voltages to help verify the results. A list of the experiments and the recipe that was used is shown in Table 3.1 and Figure 3.2 respectively.
<table>
<thead>
<tr>
<th>Date</th>
<th>Run #</th>
<th>Sample #</th>
<th>Charge</th>
<th>Run Time</th>
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<td>1</td>
<td>+30</td>
<td>41</td>
</tr>
<tr>
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<td>2</td>
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<td>41</td>
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<td>3</td>
<td>+60</td>
<td>41</td>
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<td>4</td>
<td>0</td>
<td>41</td>
</tr>
<tr>
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<td>5</td>
<td>+90</td>
<td>41</td>
</tr>
<tr>
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<td>3</td>
<td>6</td>
<td>0</td>
<td>41</td>
</tr>
<tr>
<td>11-Oct</td>
<td>4</td>
<td>7</td>
<td>+120</td>
<td>50</td>
</tr>
<tr>
<td>11-Oct</td>
<td>4</td>
<td>8</td>
<td>0</td>
<td>50</td>
</tr>
<tr>
<td>14-Oct</td>
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<td>9</td>
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<td>43</td>
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<td>41</td>
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<td>18</td>
<td>0</td>
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</tbody>
</table>

|\textbf{Table 3.1.} List of substrate charging experiments performed.|

The substrate images shown in Figure 3.8 were taken using Scanning Electron Microscopy (SEM). However, the images taken at this magnification can be unreliable. The next time that images were attempted with the SEM they didn’t appear as clear and the first images. It was then determined that Atomic Force Microscopy (AFM) would be a better and more reliable method for imaging the substrates. Each of the samples had 3 images taken of it using AFM and the images were cleaned up using the native Nanoscope Analysis software that is intended for use with the AFM images. The Nanoscope Analysis software has many built in functions for cleaning and improving the AFM images for analysis. Each image was flattened to remove any curvature, the streaks and dots were removed, and then finally the images were treated to a median image filter to smooth out the edges. An example set of images that was treated is shown in Figure 3.14.
Once the AFM Image was cleaned up using the Nanoscope Analysis software, it was exported as a 2 dimensional Tiff image. The Tiff image was then imported into an open source image processing program called ImageJ. ImageJ has a built-in function for doing particle analysis. Once the tiff image was imported it was converted to a 32-bit black and white image for easier processing. The image was then inverted to make the raised areas or particles dark instead of light. It is the default of the particle analysis to look for dark particles against a light background. In order to distinguish the particles from the background the threshold tool was used to separate the dark particles from the light background. At this point the darkness of the pixels corresponds to the height. So the 3-D picture is sliced through the

**Figure 3.14.** Original AFM image. Image after flattening. Image after clean up. Image after Median filter
middle and the areas above are turned black and the areas below are turned white. See Figure 3.15 for
the transformation of the Tiff image to the image after the threshold.

![Image](image.png)

**Figure 3.15.** Imported TIFF image. Image after inversion. The threshold tool. Image after threshold.

Once the image has been through the threshold process, it turns the areas that are considered particles
to black and distinguishes them from the white background. The next step is to quantify these particles
so that the effect of voltage on the substrate can be understood. ImageJ has a built in particle analysis
tool, but it’s not as straight forward as just running it. Even after the image processing there are still
areas that are fuzzy or left over imperfections that show through as small dots in the threshold image. We don’t want count defects in the picture as particles so a lower limit of 100 pixels in area was used to only count real particles. See Figure 3.16 for a comparison of particle counts with and without the lower limit. This gives us the amount of area on the substrate that the particles are occupying, as well as a comparison of the number of distinct particles present. This won’t give us an accurate number as to the average size of the particles because many of the particles are overlapping and are considered a single particle with double the size.

![Image after Threshold. Particle count without lower limit, 251 particles counted. Particle count with 100 pixel lower limit, 117 particles counted.](image)

**Figure 3.16.** Image after Threshold. Particle count without lower limit, 251 particles counted. Particle count with 100 pixel lower limit, 117 particles counted.

One problem with imaging the substrates is that many of the particles overlap and form large blobs. If the number of particles and their average size was analyzed there would be a lot of error because some groups of particles would be counted as a single. This would cause the number of particles to decrease and average size to increase. In an attempt to determine if the average particle size is decreased with voltage, which was one of the observations made from the initial experiments shown in Figure 3.8, particles were excluded if they didn’t meet a circular criteria. The circular criterion is a built in function of the particle analysis tool. The criteria was set to only select the particles that had a circularity from 0.5 – 1. This didn’t give a good indication of the total amount of particles but it did reveal the average
size of the particles that happened to be not touching another particle. An example of the effect that the circular criteria had on the analysis is shown in Figure 17. The analysis method that only used a size criterion will be referred to as Method A. The analysis method that used a size and circularity criterion will be referred to as Method B.

![Figure 17](image.png)

**Figure 3.17.** Comparison of analysis Methods A and B. Image after Threshold. Particle analysis without circular criteria, average size of 490. Particle analysis with circular criteria, 240.

**Results and Discussion**

As described in table 3.1, each run of the furnace contained 2 samples. One charged substrate and one uncharged for comparison. This minimizes the difference between runs that occurs due to the furnace controls and conditions. From each sample, 3 images were taken from random positions on the substrate. This is to observe and mitigate the variation in catalyst formations on a single wafer. Once all the images had been processed, the data for each sample was processed to help display the results. The particle analysis processing done by ImageJ gives 5 outputs and they are Count, Total Area, Average, Area percent, and Mean. Two of these (Count and Average size) are relevant to the information that we are trying to determine. We expect, based on the SEM images above, that the particle count should
increase and the average size should decrease. It turns out that the data collected supports this hypothesis.

First, the data for count and average size was averaged for each sample. Then the data from each run was compared by subtracting the data for the uncharged substrate from the charged one. This gave a difference value that could be used to determine if the charged substrate showed higher or lower values than the uncharged. These difference values were then normalized on a scale from 0 to 1, 0 being the lowest value and 1 being the highest value. These normalized values were then plotted verses voltage to see if there were trends in the difference between the substrates that correlated to applied voltage. The plots are shown in Figure 3.18 and 3.19.

**Figure 3.18.** Shows the results of the particle analysis using method A. Number of particles counted.

**Average size of the counted particles.**
Figure 3.19. Shows the results of the particle analysis using method B. Number of particles counted.

Average size of the counted particles.

The interesting thing is that the two analysis methods led to nearly the same results. Both show an upward trend in the number of particles counted as the voltage increases. Using Method A, this means that more particles were separated from each other. The voltage might have pushed the particles slightly farther apart so that they formed few and smaller agglomerations and particle analysis was able to detect more groups and individual particles. Using Method B, this means that more particles stayed on their own instead of forming groups and had enough spacing between each other to allow the particle detection software to distinguish them.

The downward trend in the average size of the particles with respect to increasing applied voltage was also observed by both analysis methods. In the case of Method A, this meant that the catalyst particles were forming fewer or smaller agglomerations when more voltage was applied to the substrate. This is also observed in the SEM photos. This finding using Method B indicates that the particles that formed distinct, individual particles were also smaller when voltage was applied to them. This is also observed in the SEM images.
Overall the experiments were successful in providing more information in regards to how voltage applied to the substrate affects the catalyst particle formation. The initial experiments strongly suggested that applied voltage produced a larger number of small particles. The subsequent experiments do agree with the initial findings but do not definitively confirm it. The trends shown in Figures 3.20 and 3.21 are weak at best. They don’t shed a lot of light on to the mechanisms of separation or the nature of the relationships between voltage and catalyst particle size. Even more importantly, There needs to be a study to determine if the charging of the substrate affects the CNT growth rate (as suggested in figure 3.6-3.8), straightness, diameter, and the most critical property, spinability. A more detailed description of future charged CNT synthesis experiments can be found in the future work section 7.1.

### 3.1.2. Electric Field Assisted Growth

One major factor in making a CNT array spinnable is the straightness of the CNTs in the array. Arrays that have the spinnable property are often called Super Aligned CNT arrays [30,36]. In researching methods of aligning CNTs during growth we found that there has been substantial research in to using electric field to align CNTs during growth [10,11,13,14]. The theory is that the CNT becomes a dipole in the presence of a uniform electric field. This dipole creates a force that acts to align the CNTs with the electric field when the electric field is approximately 1 V/μm. This could be very useful in increasing the control over morphology of the CNT array. By inducing an external control of the straightness of the CNTs, it could allow a high success rate of spinnable CNT arrays. The morphology of the array can be controlled with the chemical feedstock ratios, growth temperature, annealing time, etc. However, growth of CNT arrays is a very small and specific subset of these parameters, and varies between different reactors and even atmospheric conditions [37]. If the subset of growth parameters that produce spinnable arrays could be increased by the addition of an electric field parameter then the production of spinnable CNT arrays would be more reliable and prone to scale up.
In PECVD the electric field is produced by a microwave or other high intensity electromagnetic wave. In order to produce a strong, aligned electric field inside of a traditional reactor means the fabrication of custom electrodes. This was not accomplished due to time and technical difficulties. The electrodes would have been similar to the substrate charging experiments with the addition of a top electrode. See Figure 3.22 for a schematic. The top electrode could either be fixed at a distance above the substrate or be movable to move as the array grows. For spinnable and other short arrays (less than 1mm) the electrode could be fixed because the voltage would not need to be dangerously high to achieve 1 V/μm, approximately 1000 V. However, the tall arrays grown at University of Cincinnati can be as tall as 3-4 cm, this would require voltages in excess of 40,000 V to achieve a 1V/μm electric field. In this case it would be advisable to have an electrode that started near the substrate to decease the required voltage needed. Then as the array grew it would push up on the moveable electrode and lift it until the array was done growing.

![Diagram of proposed CNT synthesis electric field alignment fixture](image)

**Figure 3.20.** Diagram of proposed CNT synthesis electric field alignment fixture.

### 3.2. Fiber Development

The challenges of producing CNT fibers and nano technology as a whole are vast. The CNT fiber structure is barely visible under Scanning Electron Microscope (SEM). This can make it difficult to even
characterize material made from CNTs. Individual CNTs are practically weightless and like to stick to each other in bundles and to everything else due to the Van Der Waals forces. This makes the handling of individual CNTs near impossible. CNTs are often used in mixtures after sonication in surfactants and other dispersing agents in order separate the bundles to CNTs. Using various spinning methods this can produce highly ordered structures. The limitation is that the tubes are generally short and the spinning methods leaves unwanted compounds in the fiber or they damage the tubes. These dispersion methods have been shown be practical in producing composite fibers[38] and pure CNT fibers[39].

The main method of producing fibers at the University of Cincinnati is the dry spinning method. This method relies on the production of spinnable arrays. However, it is very promising in producing high quality, pure CNT fibers. A considerable amount of research has been done to improve the strength of as spun thread [34,40]. It has been shown that this method is capable of producing pure CNT threads around 1GPa. However, this is far short of the individual tubes potential. University of Cincinnati is unique in that it holds the world record for CNT array height[8]. However, the turning of these tubes into a fiber or other usable material has been unfruitful. Below is a series of investigations into the possibility of turning these long CNT arrays into fibers.

### 3.2.1. CNT Tile mechanical Spinning

#### Introduction

The dry spinning of CNTs into thread has many advantages, including the use of clean CNTs with no anti-agglomeration compounds added as well as the process doesn’t damage the tube structure at all. However, one major disadvantage of the process is that the production of spinnable arrays is difficult and limited in height. At the University of Cincinnati there is a production of super tall CNT arrays on the order of 5-10mm. There has yet to be discovered a practical approach to turning these arrays in to a continuous fiber. This is because the CNTs in the array are highly tangled and are of low strength. In the
pursuit of reinforcing composite materials, it was found that if the catalyst was deposited in rows on the substrate that a tile like arrays was made. The Tiles were grown by Wongdong Cho as part of his PhD dissertation at the University of Cincinnati. These tiles are approximately 1cm tall, 3-4cm long, and between 10µm and 2mm wide. The Nano-Devices team was tasked with figuring out a way to turn these CNT Tiles (CNTT) into a fiber using some spinning process that took advantage of the geometry.

The major advantage of the geometry of the CNTT is that they could be considered two dimensional where the arrays are 3 dimensional. This means that a much larger percent of the CNTs have their length exposed to the side of the array. This gave an advantage in separating the array into individual tubes or small bundles. It is also an advantage that the CNTs are of considerable length. This allows them to be handled by macro size methods. First thought of by Schulz was, that a simple mechanical process, shown in Figure 3.21, which used tiny wheels and could be used to pull apart the individual CNTs and lay them into a fiber that was then twisted. The tile would be fed into a set of rollers that would control the feed rate. These rollers would then feed the CNTT at a very controlled rate into a set of smaller rollers that would pull small bundles off and move them along a conveyor style path. The CNTs on the conveyor would be aligned and stacked at random intervals. This web of CNTs could then be twisted into a fiber.
The initial concept of the mechanical tile spinning machine. The concepts was developed with Dr. Mark Schulz.

The initial design, shown in Figure 3.21, was a good concept and a start towards making a CNTT spinning machine. Next the concept needed to be turned into a design that could be built. Figure 3.22 shows a CAD model made in Solid Works of the initial concept and how it would be constructed. The rollers could be made on an Electro Discharge Machining Lathe, which allows extremely high accuracy and the ability to make high aspect ratio parts. The brackets that hold the rollers could be machined from any material using a micro milling system. There are many other difficult technical problems that would be associated with the construction and operation of this machine. The bearing surfaces of the rollers would need journal bearings because the size makes any kind of roller bearings. Getting controlled power to the wheels would be difficult. The speed of the wheels would be critical to getting a continuous output of CNT bundles. If the feed rate was too fast then the conveyor would pull clumps out of the array. If the feed rate was too slow then the machine wouldn’t pull enough bundles out to make up a continuous fiber. Another major challenge is operating the machine would be fixing the wheels relative to each other. The orientation and spacing would be very critical in producing the desired effect. It was also unknown if the CNTT would come apart like depicted in Figure 3.21.
Figure 3.2. CAD design of the mechanical tile spinning machine.

Investigation

In order determine if a CNTT spinning machine would work, the properties and morphology of the CNTT must first be understood. In order to understand the CNTT better, a hand experiment was done under a stereo microscope. First an as grown tile was used. Using a piece of double sided tape to hold the tile in place, tweezers were used to pull from the array top and middle. The observations, shown in Figure 3.23, were that the CNT have a high level of entanglement and don’t like to separate cleanly from each other. In the case where the CNTT was pulled from the top, the large bundle peeled of the array then pulled a secondary series of smaller bundles from the array that continued to peel off the array going in the other direction. When the CNTT was pulled from the middle of the tubes it had a similar result, except for that the peeling occurred in both directions and caused the secondary bundles to be very disordered and entangled. When the bundles are peeled from the top to bottom they could be peeled in a continuous manner up to 3-4 tube lengths. The bond between primary and secondary bundles is weak, therefore the tweezers must be used to pull on the secondary bundle in order to get a third bundle to peel off. It is also important to note that each bundle is progressively smaller than its predecessor.
The tiles appear to have a high level of CNT entanglement and sometimes this can prevent the bundles from separating. It also seems like the Van Der Waals forces also hold the CNTs to the tile and they don’t allow the bundles to pull continuously. It was thought that if the Van Der Waal forces were reduced it may allow the bundles to pull continuously or to separate to a greater extent. Similar experiments to the dry investigation was done while using acetone to thoroughly soak the tile. Acetone is a good CNT solvent and the tiles readily absorb it like a sponge. It was found that when the tiles are soaked in acetone they peel apart in a similar manner as when they were dry. The difference is that the bundles appear to be smaller and it takes less force to pull them out. This is shown in figure 3.26. Just like with the dry CNTTs, the bundles can be pulled in a succession where they stay interlocked at the ends. When the solvent dries up it leaves a thin and neat string of bundles. At the intersections of the bundles there is a fluffy, thicker area that appears to be a degree of high entanglement. In order to determine if this entanglement is due to the pulling process or is inherent to the CNTTs, a tile was soaked with acetone.

Figure 3.23. Pictures of CNTTs being pulled apart with tweezers to simulate a mechanical spinning process.
and allowed to dry without being agitated. The before and after pictures are shown in figure 3.24. It was found that the centers of the tiles collapse easily as the solvent dries, but the ends condense very little. This is evidence that the tiles have very high entanglement or extra mass from tube diameter or amorphous carbon at the top and bottom.

![Figure 3.24. Investigating Tile properties while in solvent.](image)

Once this continuous peeling process was realized and practiced, it was then reasonable to try to assemble a fiber from the bundles. At first the bundles had virtually no attraction to each other. If bundles were stacked they wouldn’t even stay there under their own weight or ambient air currents. It was found that the smaller the bundles that came off in a fuzzy manner were better at sticking together. It was found that rolling, smashing, roughing and other mechanical agitation in combination with repeated solvent soaking and drying allowed the bundles to become entangled enough to produce a fiber like structure, shown in Figure 3.25. After hours of placing CNT bundles on top of each other to cover the thin and weak spots, mechanical agitation with tweezers, soaking and drying in solvent and
hand twisting, this 6 cm long fiber can support its own weight but not much more. It is very vulnerable to fraying and cracking as well.

Figure 3.25. Picture of the hand made CNT fiber from 1cm long CNTTs

After the minor success in assembling the bundles pulled from the CNTTs into a fiber structure, the next step was to take what was learned and apply that to a new spinning design. It was found that the CNTs separate best when they are peeled from the tile. And the size of the peeling makes a big difference. The difficulties of building and operating the design proposed in Figure 3.24 would be tremendous. So a simple and easier method was needed to allow testing to determine if the method is feasible. It was then conceived that a traditional gear could be the mechanism that does the peeling of the CNTs from the tile. Then a second gear placed opposite the first and slightly more forward could allow the CNTs to be pulled entirely for the tile and then advanced along a path and then twisted. Both gears and the CNTT would have the same thickness and exist in the same 2-D plane, this method will be referred to the 2D gear method from here on. Based on the previous experiments this would be best accomplished submerged in solvent to allow the CNTs to slide relative to each other. A simple experiment was conducted where a gear was used to try to separate a CNTT into bundles. The test didn’t have all the
aspects of the concept but was simplified to just the top peeling gear to allow for testing with available resources. Also the gear was spinning too fast and didn’t have enough torque. However, the results, showing Figure 3.26, were encouraging. There were bundles that were peeled and laid down next to each other in a successive fashion. This seems like the most practical approach to a mechanical spinning machine for tiles.

![Image](image_url)

Figure 3.26. 2-D gear spinning concept. Picture of 2D gear test. Stereo microscope picture of a CNTT after being run through the 2D gear spinning test.

**Future Work**

Spinning of CNTT into a continuous fiber is a huge challenge. Presented above is some preliminary work that might lead to a completely novel method of producing CNT fibers. A method has not been found in
literature to date that shows an external mechanical method of assembling CNTs or CNT bundles into a macro material. The research done for this thesis is very basic groundwork in the understanding of the structure and properties of CNTTs grown at UC. A deeper understanding of the Micro and Nano structure of the tubes in the tile is need before a fully functional spinning machine can be developed to produce meaningful amounts of CNT fiber. However, the background experiments above show that there may be a possibility that such a machine could exist and be successful.

Building machines on the scale that is needed for this task is a huge undertaking on its own. The operation and tuning of such a machine is also a very ambitious goal. If such a project were to be pursued that a method of building and operating these machines is just as important to develop. This development process could easily be done in parallel with the spinning machine. It will undoubtedly take many iterations of a micro spinning machine before one optimal design is found. Also, all the machines should be developed with a large degree of adjustability. The predicted action of each machine will very likely be very different than the actual action. There is also a very large degree of variation between CNTTs. They vary widely in height, density, strength, entanglement, width, tube size, etc. All factors that that will affect the operation of any micro spinning machine. Given all the challenges associated with a micro spinning machine, it seems possible and the reward is tremendous.

### 3.2.2. Liquid Spinning

Liquid spinning methods have been around for nearly a decade [16]. One of the main advantages of the liquid spinning over dry spinning is that the CNTs used for liquid spinning don’t need to be grown in highly aligned arrays. They can be made using other bulk manufacturing processes. This is because they are dispersed in the liquid before the spinning process, so no array structure is even needed. It seems practical to develop a process that could be used at the University of Cincinnati to produce CNT thread without spinnable arrays. Typical CNTs used in liquid spinning processes are around 10-50um in
length[16,19]. The liquid spinning could be a reliable method of producing CNT fibers as well as lead to a process of turning the super long (>1cm) CNTs into a fiber.

The first goal was to reproduce the results of Vigolo et. al. This was one of the original methods for producing CNT fibers. It simply involves dispersing the CNTs in water and sodium dodecyl sulfate and then injecting the mixture into a beaker of PVA solution. The biggest difference is that the CNTs used by Vigolo et. al. were around 20µm in length and the CNTs used at UC were 200-500µm in length. This provided a challenge because the longer CNTs are much more prone to entangling and making them harder to disperse. It was also thought to not use sonication to disperse the tube because it can break longer tubes. Instead lower concentrations of CNTs were used to help the CNTs to become untangled. Also, in order to try to preserve the length of the CNTs, a high shear mixing was used to disperse the tubes.

For the first attempt in conjunction with Rohit Parlapalli, short CNTs were collected from arrays that were grown to be spinnable but were not. These arrays were comprised of CNTs that were approximately 200-500 µm in length. They were collected in a small beaker and weight to be 0.635 mg. In order to follow the original paper we added 1.8 mg of SDS and approximately 50 mg of water. Then we left the mixing head on low over night. In the morning the mixture appeared homogeneous. When the mixing head was raised there was a black gel that had accumulated around the slits in the mixing head. The gel was scraped off and the mixer was turned up to a medium setting for several more hours. The dispersion was checked by placing a drop on a glass slide and examined under an Olympus microscope. The pictures, shown in figure 3.27, reveal that the CNTs were still quite entangled. This mixture was then squirted into a 5% PVA solution using a 5 ml syringe. A large petri dish was used and the syringe was dragged through it while slowly injecting the CNT. See Figure 3.27. The CNT did form continuous streaks, but they had no strength and just fell apart when they were lifted. It appears that
the mechanisms of the CNT fiber formation are intact. However, because the CNTs were not dispersed fully they were not able to for agglomerate like in the original work. This is likely do to the high level of entanglement that was still present in the CNT solution. A two-step shearing process should be used to fully disperse the Long CNTs from an array[41]. Work in this area has been passed on to other students working in Nanoworld.

![Entangled CNTs](image1.png)

**Figure 3.27.** Shows entangled CNTs at 10X and 100X magnification. CNTs after injection into PVA solution. Experiments performed with Parlapalli.

In the process of developing the liquid spinning concept for use with super long CNTs several other more advanced concepts were developed. It was thought that a similar process could be used to make a highly aligned sheet like in the dry drawing sheet making process. The Long CNTs could be dispersed at a much lower concentration then described above which should allow complete breakdown of the bundles into individual CNTs. There still may be a small level of entanglement that would allow the CNTs to form a matrix that is gel like. This would help ensure that CNTs in the solution form a continuous web. The solution could then be injected on to a conveyor style belt that would allow the CNTs to be pulled and then collected in layers that could be built up to a desired thickness.
3.3. Sheet Development

The University of Cincinnati holds a patent on a technique and method for making a sheet of aligned Carbon Nanotube. Their method is based on a conveyor belt style winding of multiple layers of CNT ribbon. The ribbon is generally pulled from as grown arrays and then spun into fibers. A single ribbon is far too thin to be handled, around 140nm. By layering the ribbons on top of one another we can create a sheet that has enough strength to be handled. The concept had been developed by Chaminda Jayasinghe, a materials engineering doctorate student at the University of Cincinnati. Jayasinghe used a sheet of Teflon and the two simple rollers to create a conveyor belt on to which he wound multiple layers of CNT ribbon. His initial design was simple and could produce CNT sheet that is approximately 1” wide and 5” long. See Figure 2.6. The next design was more robust and could create larger sheets of approximately 11” wide and 24” long. The first two sheet machines worked exactly as predicted but were slow and tedious because they had to be operated by hand.

3.3.1. Sheet Machine

In conjunction with Jayasinghe Rachit Malik, and Dr. Vesselin Shanov, a new idea was developed on how to scale up the previous machines to a small scale production level. The goal was to design a machine

![Figure 3.28. Concept for making highly aligned sheet from long CNTs in suspension.](image)
that could produce very large sheets and be reconfigured to make small sheets as well. One reason that adjustability was so important is that we are often limited in material availability. Spinnable CNT arrays are difficult to produce and are in high demand by many projects. Another aspect that was desired in a new machine is a level of automation. It may be desired to produce large sheets of many layers of CNT ribbon. This could take a long time so if a computer program could be written to take over the rotation of the belt and to move the array along the axis of rotation. By using a computer controlled system we can also precisely control and explore different patterns of wrapping.

Another aspect of the original sheet machines that was redesigned was the conveyor belt itself. The original idea was simply a Teflon sheet that was rolled over and held together with double sided tape. The ribbon is a very fragile structure and sticks to most things due to Van Der Waals forces. These forces can fray and destroy the ribbon structure. By using Teflon sheet we reduce the damage to the sheet when it is removed from the belt. Another design uses pegs to minimize the contact with the CNT material and allow for easy removal and preservation of the CNT sheet. The design resembles a hamster wheel configuration. We wanted our machine to be capable of both the conveyor belt and hamster wheel configuration. The design that we decided to pursue was a combination of the two concepts. We settled on what became known as the pizza oven concept. Advantages of the hamster wheel are the minimization of contact with the CNT sheet, and spaces between the pegs could allow for the application and drying of coating or densifying chemicals. The limitation of the hamster wheel is the length of the sheet is limited by the diameter of the wheel. The advantage of the conveyor belt is the length and width is both adjustable and less limited in size. The pizza oven concept combines the advantages of both systems. As a compliment the belt can still be covered with Teflon sheet and we have a conveyor belt configuration. In distinction, if the belt were shortened and only wrapped around one set of pulleys then we have an elongated hamster wheel.
Then next step was to take the concepts we discussed and make a CAD model to determine the parts necessary to build our machine. Because the concept for making the sheet had been well established we wanted to go as large of scale as made sense for our laboratory. The length of the sheet would simply be dictated by the length of the belt. The CNT material is virtually weightless the only forces on the belt will be its own weight. Therefore the length of the belt would be limited by the tension we can put on it so it doesn’t sag. The width would be limited by the length of linear stage we get to move the array. It was found that the longest linear stage we could get within our budget had a travel of 300mm. A Newmark Systems eTrack linear positioner with a 12mm pitch lead screw was a good choice for our application. See the specifications below in Table 3.2. In order to make the system simple and reliable a rotary stage and motor controller from Newmark systems were also chosen. This way we can control both the rotation of the belt and translation of the array with the same controller and program. The Specs for both the rotary positioner and motion controller are below in Table 3.3 and 3.4.

<table>
<thead>
<tr>
<th>Travel Range</th>
<th>50 mm, 100 mm, 150 mm, 200 mm, 250 mm, 300 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution</td>
<td>0.04 µm (2 mm pitch leadscrew @ 250 microsteps)</td>
</tr>
<tr>
<td></td>
<td>0.24 µm (12 mm pitch leadscrew @ 250 microsteps)</td>
</tr>
<tr>
<td>Accuracy</td>
<td>0.0006 mm/mm</td>
</tr>
<tr>
<td>Max. Speed</td>
<td>40 mm/sec (2 mm pitch leadscrew)</td>
</tr>
<tr>
<td></td>
<td>220 mm/sec (12 mm pitch leadscrew)</td>
</tr>
<tr>
<td>Maximum Load</td>
<td>4.5 kg (10 lb.)</td>
</tr>
<tr>
<td>Stage Weight</td>
<td>ET-50: 1.1 kg (2.5 lb.)</td>
</tr>
<tr>
<td>Material</td>
<td>Aluminum Alloy Construction</td>
</tr>
<tr>
<td>Finish</td>
<td>Black Anodize</td>
</tr>
</tbody>
</table>

**Table 3.2. eTrack Linear Positioner Specification**
The next step in the design was to setup the structure of the machine. The design that was chosen used an aluminum plate and steel shafts to create a parallel rail system for the conveyor. At one end the linear stage sits up just below the center line of the conveyor axis. At the other end another aluminum plate holds the steel shafts in place. A lab jack will be fixed to the top of the linear stage so the height of the array can be lined up with the top of the conveyor. The axles for the conveyor rollers sit inside split top pillow block bearings. The split top bearings will allow for easy, smooth rotation as well as easy removal of the entire conveyor and axles. This will also allow for easy transition between different configurations of sheet manufacturing. The bearings sit on top of the aluminum plate that has brass bushings pressed into them. The brass bushings allow the bearings to move smoothly and then they are
fixed in place with collars and set screws. The entire sheet machine in pizza oven configuration is shown in Figure 3.29.

![Pizza Oven Sheet machine configuration](image)

**Figure 3.29.** Pizza Oven Sheet machine configuration, a) eTrack linear stage, b) RT-3 rotary stage, c) axle bearing blocks, d) Pizza Oven conveyor, e) Lab lift stage, f) Precision ground steel shafts, g) Split top pillow block bearings

Many different factors affect the properties of the CNT sheet. This machine is capable of many different functions and configurations. The older CNT sheet machines that have been made are hand operated so the thickness of the sheets vary slightly along the width. This is because it is difficult to accurately move the array side to side in a pattern with turning the belt. With the computer controlled machine we will be able to have very uniform thickness. This will allow for better characterization of the material and how the number of layers affects those properties. We will also be able to compare the different configurations and see if the Teflon sheet method yields a different result than the hamster wheel or pizza oven configurations. The Hamster wheel configuration is shown in Figure 3.30.
We expect the CNT sheet material to be extremely anisotropic. The CNTs are very strong in the axial direction and the Van Der Waal forces allow for high strength in the tube direction. However in the transverse direction the tubes will pull apart very easily. The entire CNT sheet currently made sheet at the University of Cincinnati has a very uniform tube direction. Our machine will work well at producing sheet in this fashion, depicted in Figure 3.31. Another option we will have with the computer controlled sheet machine is to have a cross pattern of tubes, shown in Figure 3.32. This pattern could help increase the properties in the transverse direction. Using a similar method the ribbon or CNT thread could be wrapped around a shaft or core. This could be used to make CNT Tubing or for EM shielding. It could also be setup to wind CNT coils on a variety of cores. An example of winding is shown in Figure 3.33.
Figure 3.31. Straight sheet wrapping pattern

Figure 3.32. Cross sheet wrapping pattern
One important process regarding CNT sheet is densification. With CNT thread the properties can be increased by dipping the thread in to different organic solvents and allowing them to dry. When the solvent evaporates it reduces the space between individual CNTs and increases the Van Der Waal forces. For production purposes, it would be advantageous to have inline densification. The design of this sheet machine makes it portable so that is can be placed under a fume hood. An example setup is pictured in Figure 3.34. This way the densification of the sheet could happen during the manufacturing process.

Another process that can be applied to CNT sheet is coating with polymers of other matrixes to form composites. This process could also be done with this setup in an inline fashion. The gaps in the pizza oven conveyor allow for increased convection to decrease drying times and increase temperature control. CNT sheet can also be functionalized by different methods to increase bonding to different matrixes. This could also be done with some modification to this machine.
3.3.2. Sheet Properties

The properties of the aligned CNT Sheet have been explored extensively at UC and partners at Dalhousie University in Halifax, Canada. The cooperation of these two universities produced a paper that was published in Carbon called ‘Physical properties of carbon nanotube sheets drawn from nanotube arrays’[29]. In this paper there is data for mechanical, room temperature electrical, low temperature electrical, and thermal properties. In addition there has been electromagnetic radiation transmittance testing from the ultraviolet to the radio wave spectrum. The data included below is only the data that has been collected by, or in conjunction with, Ruff.
One of the properties of individual CNTs is that they can low resistance. Therefore it was desirable to determine the resistivity of the CNT sheet. An accurate cross section of the thread is needed to determine resistivity. The CNT sheet is very thin and it is difficult to measure the thickness. So relationship between the number of layers and the resistance was found instead. These results are shown in Figure 3.36. In addition, using a solvent to densify the sheet is a common way to increase the properties. So the as made sheet was compared to sheet that was densified. The densified sheet resistance data is shown in Figure 3.37. For each test the sheet length and width were kept constant. The thin sheets used in this test are too fragile to test on their own. The samples were tested attached to the Teflon film that was used in the manufacturing process. Teflon is an insulator so it did not contribute to the resistance values.
Figure 3.36. As made CNT sheet resistance parallel with the tube direction

Figure 3.37. Densified CNT sheet resistance parallel with the tube direction
The results were interesting in that for the as made sheet the resistance decreased overall, but the resistance per sheet increased. The resistance data is tabulated in Table 3.5. That means that the resistance of a multi-layer sheet is greater than if each layer were separated and placed in parallel. This indicates an increase in resistivity as the layers are added. This was not the case for the densified sheet. The densified sheet generally decreased as more layers were applied. The resistance per sheet was reduced to 1040 ohms compared to a single layer of as made sheet which was 1642 ohms. This can be approximated as a 35% decrease in resistivity. The trend may continue as you add layers as well. The reason it can only be approximated is that we are assuming that two layers are twice as thick as 1 layer, 4 layers is 4 times as thick, and so on. We are also not deducting any area after the densification process. This could be more accurately characterized as specific conductivity if we assume that each layer has the same mass, and that the densification doesn’t change the overall weight of the specimen.

<table>
<thead>
<tr>
<th>Layers</th>
<th>As made sheet (Ω)</th>
<th>Densified sheet (Ω)</th>
<th>As made Res per layer (Ω)</th>
<th>Densified Res per layer (Ω)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1642</td>
<td>1518</td>
<td>1642</td>
<td>1518</td>
</tr>
<tr>
<td>2</td>
<td>910</td>
<td>1016</td>
<td>1820</td>
<td>2032</td>
</tr>
<tr>
<td>4</td>
<td>510</td>
<td>303</td>
<td>2040</td>
<td>1212</td>
</tr>
<tr>
<td>10</td>
<td>191</td>
<td>104</td>
<td>1910</td>
<td>1040</td>
</tr>
</tbody>
</table>

*Table 3.5. Tabulated CNT sheet resistance data*

Although the exact resistivity of the sheet is difficult to measure, it is estimated to be pretty poor. Therefore it is desirable to develop a post treatment to decrease the resistivity of the sheet. One process that had been practiced by Rachit Malik is to electro deposit different metals on the CNT sheet. It was thought that having metals deposited on the sheet surface would increase the conductivity. The metal that was deposited on the sheet for this test was nickel. The resistance of the sheet was measured on the Soletron Impedance analyzer so that the impedance could be measured up to 10MHz. The contact was made by placing the CNT sheet between two blocks of copper. Then a vise was used to apply even pressure to the block. This method was used instead of the traditional method for DC
resistance measurements which involves using silver paste to decrease the contact resistance. The results of the experiment are shown in Figures 3.38 and 3.39.

Figure 3.38. Magnitude of CNT sheet impedance parallel with the tube direction.

Figure 3.39. Magnitude of CNT sheet impedance parallel with the tube direction.
It seems that the nickel electro deposition process actually increases the impedance in the frequency spectrum tested by the Soletron. If the Impedance were measured at higher frequencies it might be possible that the impedance of the nickel coated sheet drops below that of the pure CNT sheet. The increase in impedance might be because the process of electroplating involves submerging the sheet in an electrolyte. This electro light could have left impurities in the sheet that increased the resistivity. It is also possible that the nickel particles oxidized when exposed to air. The formation of the nano scale nickel particles means that the surface area of the metal exposed to air is very high. This makes the nickel more prone to oxidation and could increase the resistivity and impedance.

The lower frequency impedance data lead us to believe that there would be interesting properties at very high frequencies. In order to test the electrical properties of the sheet at high frequency a Vector Network Analyzer (VNA) was used in conjunction with and under the direction of Dr. David Mast. The VNA produces a polarized electromagnetic wave that travels through wave guides and then the wave is fed back into the VNA. The signal strength of the transmitted and reflected signal is measured and can be used to determine the relative impedance. Two wave guides were placed very close to each other with a small gap between them. In order to protect the sheet from damage during the test two pieces of Poly(methyl methacrylate) (PMMA) plastic was used to sandwich the CNT sheet that was still on the Teflon backing that was used to manufacture it.

First, the test was run with just air in between the wave guides to get a base line measurement. In order to determine the effect of the PMMA and Teflon on the impedance, the test was run with just the PMMA and then with the PMMA and Teflon film. This lead to little change in the frequency response of the VNA meaning that the PMMA and Teflon don’t absorb much if any of the electromagnetic wave and have very high impedance. The EM wave travels in one plane and that is the plane in which the impedance is measured. The plastics have isotropic electrical properties unlike the CNT sheet which has
the CNTs all aligned in one direction. The CNT sheet on the Teflon backing was placed between the PMMA and in the wave guide. First the CNT sheet was tested with the EM wave parallel to the direction of the CNTs, and then with the CNTs perpendicular to the EM wave. It was found that the impedance in the direction of the tubes is much lower than the impedance perpendicular to the CNTs. This is what was expected. Absolute resistance values are possible to attain with this method but require more testing and calculations.

![VNA Transmitence Data](image)

**Figure 3.40.** High frequency transmittance data for CNT sheet collected in collaboration with Dr. Mast.
3.3.3. Twisted Sheet

The CNT Sheet material doesn’t have very high strength or electrical properties as we can measure them. There may be some error because we don’t have an accurate way of measuring thickness which is critical for calculating material properties. There also might be some difficulty in the testing of the sheet. It is highly anisotropic in strength and tears along the tube direction very easily. This is compounded by the tendency of the sheet to stick to everything. So it is often that the material is visibly damaged in the process of handling it. It is also likely that is often damaged in a manner that can’t be seen visually. Even still the properties that have been measured are on the same order as the properties measured for the thread.

In an attempt to increase the properties of CNT materials made a UC a small study was done on taking the sheet and twisting it into a fiber. There are many advantages to this. One is that the traditional spinning process doesn’t have enough control. The ribbon is pulled from the array and twisted. But the rate at which the ribbon is pulled and the thread is collected is not controllable, or even measureable. This means that the only diameter control is by the width of the array, and that there is no ability to induce strain in the thread during the spinning process. Stretching allows the fibers in the thread to align and increased the strength considerably. The intermediary step of making sheet before spinning also allows the functionalization of the CNTs or the infiltration of a matrix material while there are more of the CNTs exposed.

The first step towards making twisted sheet is a mechanism to do the twisting. As mentioned above, handling of the sheet is very difficult. If the twisting mechanism doesn’t have enough control over the sheet then the twisting will not work as desired. The process that was developed used a custom made linear actuator that was fitted with set of clamps and a rotation axis on one side. This allowed a sheet to be placed between the two clamps and then tension taken out of it using the linear stage. Then, once
the sheet was fixed in the machine solvent was added to the sheet to allow the sheet to conform to its new shape. The sheet had a tendency to elongate once the solvent was added and the linear stage was used to keep the sheet taught. Then the rotation axis was used to twist the sheet. It was apparent that the tension increased as twist was added. However, the linear stage was not adjusted during the twisting process. This further induced tension and strain in the sheet during the twisting process. The process was crude but repeatable and a number of samples were created. Picture of the sheet twisting fixture is shown in Figure 3.4.1. The one major downside is that neither the linear or rotation axis could be measured. So the twist per millimeter and percent elongation couldn’t be measured. Images of twisted sheet samples were taken with the SEM, and are shown in Figure 3.4.2.

![Sheet twisting fixture](image-url)

**Figure 3.4.1.** Sheet twisting setup.
3.42. SEM images of the twisted sheet. Shows the structure

There were 3 specimens that were made using the process described above. Each of the three specimens was divided into 4 samples. Two samples were used for electrical resistance testing and two of them were to be used for mechanical testing. The mechanical testing was performed by Bolaji Suberu. The tensile tester is fitted with a 5N load cell and all the samples maxed out that load cell without failing. The stress of each of the samples at the max load of 5N is shown in Tables 3.6. It is encouraging that the smaller diameter samples reached 500 MPa in strength. It is not possible to determine what the actual maximum strength would have been. More samples would have to be made and tested on a more powerful tensile test machine. Or the process could be modified to produce smaller diameter thread. In previous experiments at UC it has been found that smaller diameter thread generally has
better properties.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Specimen</th>
<th>Resistance</th>
<th>Diameter (um)</th>
<th>Length (mm)</th>
<th>Resistivity (ohms*m)</th>
<th>Strength @5N (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>18</td>
<td>111.3</td>
<td>4.16</td>
<td>4.212E-05</td>
<td>513.6</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>17</td>
<td>111.3</td>
<td>4.36</td>
<td>3.795E-05</td>
<td>513.6</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>6</td>
<td>157.2</td>
<td>4.19</td>
<td>2.778E-05</td>
<td>257.7</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>6.5</td>
<td>160.8</td>
<td>4.43</td>
<td>2.984E-05</td>
<td>246.1</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>5.5</td>
<td>175.0</td>
<td>3.79</td>
<td>3.491E-05</td>
<td>207.9</td>
</tr>
</tbody>
</table>

Table 3.6. Electrical and mechanical properties of the twisted sheet samples.

The electrical resistance data was measured using a Keithley current source and Nano-voltmeter. The sample fixture and user interface is a custom setup and program that was created by Brad Ruff. The fixture starts by placing the CNT fiber across 4 copper prongs and electrically connecting the thread to the prongs with silver paste. The prongs are placed in a socket that connects the outside prongs to the current source and the inside two to the nano-volt meter. A diagram of the testing configuration is shown in Figure 3.43. This 4 wire resistance measurement technique is used to reduce the contact resistance during the test. The resistivity values were calculated and shown in Table 3.6.

![Figure 3.43. Electrical Testing diagram. The outside prongs are connected to a current source. The inside prongs are connected to a voltmeter.](image-url)
Twisted sheet shows a lot of promise in creating a new classification of CNT thread. The process of stretching and twisting while simultaneously densifying and/or functionalizing opens a lot of doors for continuous processing that would not be possible with traditional direct spinning with arrays. It is also possible to more directly control the diameter by using a specific number of layers and then cutting the sheet to specific widths. This a more accurate way to control the mass of CNTs that in used to make a given length of fiber. More investigation in to the properties of twisted thread is needed before investment in new equipment is justified. This investigation could easily be performed using the equipment available at UC.

### 3.4. Post Treatments

One important aspect of CNT material production is the treatments that can be done to the thread after the spinning has taken place. The thread and sheet materials produced at University of Cincinnati generally have very low properties in the as-made state. Therefore, there is a need for processes that either occur inline or during the spinning process or are done separately after the spinning process. Most of these processes can be divided into two different categories, addition of polymer material to make a composite, and other process that change the structure of the tubes in the thread or the structure of the tubes themselves.

Processes that are aimed at modifying the structures of CNTs or CNT materials come in a large variety. The one primarily discussed below has to do with densification using organic solvents. Densification generally occurs when the CNT thread or sheet is soaked with a solvent that is absorbed into the material. As the solvent evaporates the surface tension pulls the CNTs into closer contact and eliminates the air voids. This increase in tube to tube contact allows the CNTs to have greater Van Der Waals interaction as well as increases in conductivity. To this date a detailed investigation into the methods and mechanisms of chemical densification has not been found in a publication.
Composites are used today in everything from phones to airplanes. Specifically, carbon fiber composites can have truly exceptional strength. The addition of powder CNTs to polymers can marginally increase the strength of polymer fiber[38,42]. There have also been many attempts at infiltrating CNT thread and sheet with a polymer matrix to increase the load sharing between the CNTs[23,33]. These highly aligned, high volume fraction composites have tensile strengths up to 2 GPa. The electrical conductivity of the threads tends to decrease because the polymers generally have high dielectric properties. If the CNT threads were to be used as electrical conductors then they would need a coating that electrically insulated the CNTs from outside contact but not from each other.

3.4.1. Densification Machines

Densifier 1.0

At UC the densification of thread was done by taking a segment of thread and dropping it into a petri dish with a solvent in it. The thread would then wiggle around as it swelled with solvent and untwisted slightly. Then the fiber was removed from the dish with tweezers. The process was difficult, uncontrolled, and was limited to short segments or the thread would twist on top of itself and be unusable. Based upon a request by Mark Hasse, it was desirable to make a machine that could be used to density a long section of CNT thread in a controlled and repeatable manner. Towards this end, a machine was devised that became known as the Densifier 1.0.

The main purposes of the Densifier 1.0 were to dip CNT thread into a solvent, let it dry and then collect it. This was accomplished with a simply unreeling the thread from one bobbin and collecting it on second bobbin. There was a set of belts that transfer the reel/unreeling torque from one bobbin to other. There was a second purpose in that there was a desire to try to do some stretching of the thread while it was in the solvent. Stretching is a common practice in everything from cotton to polymer fibers. In order to induce linear stretch in the thread a gear box was placed between the reeling and unreeling
bobbins. This gear box increased the reeling speed by 1-5% depending on which gear box was chosen.

The Densifier 1.0 was never extensively used for densification experiments. Instead it ended up being modified many times to fit different roles in various experiments.

![Diagram of Densifier 1.0 and Finished Densifier 1.0](image)

**Figure 3.46.** Densifier 1.0 concept. Finished Densifier 1.0

**Densifier 2.0**

The original Densifier 1.0 worked as designed ended up being modified for use with other processes in coating and doping processes. At the request of another colleague Dr. Noe Alvarez, a second version of the Densifier was made, known as the Densifier 2.0. He was interested in performing densification on threads while they were entirely submerged in solvent. This allowed the thread to pick up different dopants while it was being densified. However, the solutions that were used to densify and dope were expensive so it was desirable to make the Densifier 2.0 as compact as possible to save solution. This lead to a machine that could be placed on top of a beaker and would have two shafts that this with the unreeling and collecting bobbins on them. Then in order to have the stretching feature in the densifier2 a gear box was placed on top of the two shafts and allowed the differential speed of the two shafts like in the Densifier 1.0. This was used extensively in the doping experiments.
3.4.2. Densification Investigation

During the use of the Densifier 2, it was observed that sometimes when the bobbins are first placed in the solvent the thread appears to fall down the bobbin like rings on a shaft. This would suggest that there is slack in the thread while it is soaked in solvent that isn’t present while the thread is dry. After some literature searching it was found that there was little reported research on the chemical densification process, and none that had reported a length change in the thread after densification.

After some planning and discussion with Dr. Noe Alvarez and Mark Hasse, a quick study was planned to investigate this phenomenon. It would be necessary to measure the length and diameter of a short piece of thread, density with a solvent and then measure the diameter and length afterward. This data would allow a Poisson ratio to be calculated for the densification process. It is likely that the reason that this has never been directly investigated is because of the technical difficulty in both measuring the diameter and length but also the handling of a short piece of thread is difficult. After much trial and error, a setup was conceived that would allow simple measurement of the thread diameter and repeatable densification process.

The process involves gluing a Teflon coated quartz string to each end of 2-3 inches of CNT thread using a two part epoxy. The thread is then anchored to one end of a board that has a Teflon film on top of it.
There is a solid Teflon bath that has a slit cut at each end of it. The thread is placed so that the CNT thread is suspended inside the bath and the quartz string passes through the slot on each end. The slot is small enough that the surface tension doesn’t allow the solvents to leak out of the bath. Then the other end of the thread is left to hang over the end of the board and a weight is attached to it. This weight allows the thread to be taught and level during the measurements. The thread being level and straight is very important because the measurements were done using an optical microscope. The diameter would be measured using an optical Olympus Microscope fitted with a Pixelfly digital camera. The camera images were captured using ImageJ open source image processing software. The length of the thread was found by marking the end coordinates on a ProScan III digital x-y stage.

The experimental procedure consisted of first placing the thread in the fixture without any solvent. Then the diameter and length were measured. Next, a solvent was added to allow the thread to soak. The solvent was then allowed to evaporate or removed with an absorbent material. Once the thread was completely dry the diameter and length was measured again. The length and diameter were not feasible to measure while the thread was soaked because of the optical distortions from the solvent surface. Also the surface tension of the solvent caused the thread to move around, making consistent measurements impossible. The length measurements proved repeatable within a micron. However the diameter measurements needed further investigation.
The diameter measurements were traditionally taken from a straight line measurement perpendicular to the thread’s length axis. For this experiment the exact diameter is critical for accurately measuring the change in cross section. This is also a critical measurement in determining the material properties. Often in publication they don’t mention how they determine the cross section of the fibers they test. For example, if there is a 1um error on a 15 um diameter thread that can change the strength measurements by up to 15%. So, one of the first challenges was to establish a repeatable means of measuring diameter and diameter change. The first method of diameter measurement was to take multiple pictures along the length of the thread and then make several straight line measurements from each picture. The results of three sections of the same thread that were measured 21 times along the length before and after densification are shown in Figure 3.49. The averages of the thread diameter show a clear change (19.7 before, 16.3 after) but the individual measurements have a huge range. But this is evidence that if only a few measurements were taken then there is a possibility of a significant error. In response to this a new method of diameter measurements was developed that consisted of taking a series of images along the length of the thread. Then instead of using straight line measurements, ImageJ was used to separate the black thread from the white background and then the area of the thread in that picture was calculated. A step by step comparison of the measurement methods is shown in Figure 3.51. This approach leads to a much more consistent diameter measurement. An example is shown in Figure 3.50.
Figure 3.49. Diameter measurements using straight line distances of three sections of the same thread before and after densification.

Figure 3.50. Diameter measurements using the cross section method of a thread that was densified 4 times.
Figure 3.51. Microscope Image of the thread for diameter measurement. Threshold image that is used to determine the area of the axial cross section of the thread. Using straight line measurements the diameter varies from 15.7 to 19.7um. The area equals 16303 um^2 if divided by the length of 912 um this gives an average diameter of 17.9um for that given image.

3.4.3. Initial densification experiments

Once a repeatable method of measuring diameter and length change and was established, there were several general investigations into the mechanisms of the densification process. It needed to be discovered which factors were the most important to investigate and to what depth they were to be explored. In Table 3.7 shows a series of the first experiments. Acetone was used often because it is easily available and has been used in the lab before for densification. The acetone series of experiments seemed to point to an increase in densification with an increase of tension during the solvent soaking and drying. It also suggested that repeated densification doesn’t decrease diameter significantly but the length will continue to increase slightly with repeated densifications of the same specimen. Using DMSO as the solvent lead to the better volume reduction than any of the acetone tests. Its important to mention that during these tests there were still some experimental uncertainties due to incremental improvements in the processing and measuring of the thread.
With the DMSO outperforming the acetone, it was then set out to discover what makes a solvent good for densification. It was thought that the better the solvent was at dissolving CNTs the better it would be at densifying the thread. The solubility of CNTs in different solvents has been explored in previous literature[43,44]. A large variety of commonly available solvents were then tried to see if there was a trend between solubility and densification. The results are shown in Figure 3.52 and there is a greater explanation of each experiment is Table 3.7. The order of results is in the reverse from the order that they were performed.

<table>
<thead>
<tr>
<th></th>
<th></th>
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<th></th>
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<tr>
<td>Test</td>
<td>Flat surface no tension using direct diameter measurements</td>
<td>Acetone densification under small tension</td>
<td>Acetone densification under tension</td>
<td>Acetone densification 4x</td>
<td>DMSO densification</td>
<td>Acetone With No Weight</td>
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<td>length Before (mm)</td>
<td>83.00</td>
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<td>84.34</td>
<td>45.78</td>
<td>40.03</td>
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<td>19.70</td>
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<td>Length After (mm)</td>
<td>83.21</td>
<td>84.72</td>
<td>84.46</td>
<td>46.23</td>
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<td>29.27</td>
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<tr>
<td>Diameter after</td>
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<td>16.49</td>
<td>16.01</td>
<td>16.66</td>
<td>17.38</td>
<td>15.66</td>
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<td>0</td>
<td>0.55 g</td>
<td>2.2 g</td>
<td>2.2 g</td>
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<td>Length Change</td>
<td>0.3%</td>
<td>0.5%</td>
<td>0.2%</td>
<td>1.0%</td>
<td>0.4%</td>
<td>1.2%</td>
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<tr>
<td>Diameter Change</td>
<td>-14.1%</td>
<td>-21.5%</td>
<td>-16.4%</td>
<td>-25.0%</td>
<td>-11.8%</td>
<td>-22.5%</td>
</tr>
<tr>
<td>Poisson ratio</td>
<td>-1.8%</td>
<td>-2.5%</td>
<td>-0.9%</td>
<td>-4.0%</td>
<td>-3.4%</td>
<td>-5.4%</td>
</tr>
<tr>
<td>Volume change</td>
<td>-26.0%</td>
<td>-38.1%</td>
<td>-30.1%</td>
<td>-43.2%</td>
<td>-21.9%</td>
<td>-39.3%</td>
</tr>
</tbody>
</table>

**Table 3.7.** Shows data from first set of densification experiments.
Figure 3.52. Normalized diameter and length change for all the experiments performed in the investigation
Table 3.8. Descriptions of the densification tests

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Test Name</th>
<th>Test description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl-Pyrrolidinone/1.2g</td>
<td>Single densification with 1.2g of tension using Methyl-Pyrrolidinone</td>
</tr>
<tr>
<td>2</td>
<td>4:1 Acetone-H2O/1.2g</td>
<td>Single densification with 1.2g of tension using a 4:1 ratio of acetone and water</td>
</tr>
<tr>
<td>3</td>
<td>1:1 Acetone-H2O/1.2g</td>
<td>Single densification with 1.2g of tension using a 1:1 ratio of acetone and water</td>
</tr>
<tr>
<td>4</td>
<td>1:4 Acetone-H2O/1.2g</td>
<td>Single densification with 1.2g of tension using a 1:4 ratio of acetone and water</td>
</tr>
<tr>
<td>5</td>
<td>Toluene/1.2g</td>
<td>Single densification with 1.2g of tension using toluene</td>
</tr>
<tr>
<td>6</td>
<td>Ethanol/1.2g</td>
<td>Single densification with 1.2g of tension using ethanol</td>
</tr>
<tr>
<td>7</td>
<td>W,A,DMSO/1.2</td>
<td>3 rounds of densification using water then acetone then DMSO with 1.2g of tension</td>
</tr>
<tr>
<td>8</td>
<td>H2O,Ace/1.2g</td>
<td>2 rounds of densification using water then acetone with 1.2g of tension</td>
</tr>
<tr>
<td>9</td>
<td>H2O/1.2g</td>
<td>Single densification with 1.2g of tension using water</td>
</tr>
<tr>
<td>10</td>
<td>Acetone/0g</td>
<td>Single densification with no tension using acetone</td>
</tr>
<tr>
<td>11</td>
<td>DMSO/1.2g</td>
<td>Single densification with 1.2g of tension using DMSO</td>
</tr>
<tr>
<td>12</td>
<td>Acetone x4/2.2g</td>
<td>4 sequential densifications with 2.2g of tension using acetone</td>
</tr>
<tr>
<td>13</td>
<td>Acetone x3/2.2g</td>
<td>3 sequential densifications with 2.2g of tension using acetone</td>
</tr>
<tr>
<td>14</td>
<td>Acetone x2/2.2g</td>
<td>2 sequential densifications with 2.2g of tension using acetone</td>
</tr>
<tr>
<td>15</td>
<td>Acetone x1/2.2g</td>
<td>1 densification with 2.2g of tension using acetone</td>
</tr>
</tbody>
</table>

Conclusions

The initial densification experiments gave some good data as to which factors are the most important in reducing diameter and increasing length. Initially it was seen that repeated rounds of densification can continue to increase the length and decrease diameter. This can be seen in tests 12-15. The diameter levels off after a few rounds but the length continues to experience small increases. Also, repeated densification seems to make the thread a more even diameter along its length. This seems to indicate that there is uneven internal stress in the thread from the spinning process. This is also supported in the
observation that while the solvent is evaporating the thread rotates back and forth in the middle while the ends are stationary. The repeated densification allows the thread to shift its structure to more evenly distribute the internal stresses.

One other observation is that the length and diameter reductions are better while under weight. Test 15 used a 2.2 g weight and after one densification had a diameter reduction and length increase of 20.7% and 1% respectively. This is in stark contrast to the test in which the weight was removed during the densification process as in test 10. In that test the thread was only under tension during the measuring process. This resulted in a 12% diameter reduction and 0% length change. In terms of increasing the density of the thread, having the thread under tension during the soaking and drying process seems to produce better results. There is one other noteworthy observation in regards to tension. In test 5, the weight was reduced from 2.2g to 1.2 g. The reason for this is that the DMSO thread repeatedly broke as soon as the solvent was added. This could be because DMSO is a better solvent for CNTs than acetone is. This could cancel the Van Der Waals and let the CNT thread slip apart. The weight was reduced to 1.2g and the breaking problem was reduced. It was still found that when the thread was soaked in the stronger solvents that it took very little force to break the thread. However, once the thread dried in returned at minimum to its original strength, but it’s likely that it was much stronger.

Over all it appeared that the solvent seemed to have the biggest impact. Water did very little to densify the thread, while solvents like DMSO and N-Methyl-Pyrrolidinone did a much better job at increasing density. Trying different solvents is also a very easy thing to test. Just repeat the procedure with different solvents to investigate which solvents produce the best densification. There is also plenty of literature on which solvents work the best to dissolve CNTs. By choosing a range of solvents we can see what the effect of each solvent is on the change in density.
There was one other interesting discovery in this series of experiments. There is a dip coating process that is carried out by Dr. Alvarez in which the polymer is suspended in a 1:4 mixture of acetone and water. This combination was tried to see how it compared to other solvents. It was found that a mixture of acetone and water out performed both pure water and pure acetone. This is quite interesting because that means that there must be some non-linearity. In order to investigate this farther a series of mixtures were tested. The results are shown in Figure 3.53 and Figure 3.54. The results were not clear but what was consistent was that a combination of water and acetone was better that water or acetone. The top performer over all was 80:20, water to acetone. This is very interesting because pure water was the worst and by just adding 20% acetone it became the best. This may suggest that the water which is not a good CNT solvent may apply pressure to the thread while the acetone infiltrates the fiber and allows the CNTs to move relative each other.

![% Acetone in Water Solution Densification](image)

**Figure 3.53.** Acetone-water mixture verses diameter change.
3.4.4. Densification Experiments 2

Procedure

The initial densification experiments showed several correlations between the method of densification and the resulting dimensional changes in the thread. The two factors that were chosen for immediate further investigation were the solvent and the amount of tension on the thread during the process. These factors were chosen because they were the most consistent and the most basic of the trends seen. It was decided to only look at one solvent at a time and not investigate mixtures of solvents. In order to understand the effect of solvent mixtures, the individual effects of each solvent must be understood first. No mechanical or electrical property measurements were taken and only single densifications were done on each sample in order to allow a more extensive experiment with the available time. Once the basic parameters were better understood, the other secondary factors will be
investigated to fully understand and optimize the densification process. Dylan Van Dyne was enlisted to help with the tremendous amount of work that was need to complete this experiment.

A short list of common lab solvents was chosen for the tests. Some of the solvents were taken from the available CNT literature on the dispersion of single wall CNTs in solvents (N-Methyl-Pyrrolidinone, Chloroform) [43,44]. These are considered solvents that are very good and dissolving the CNTs. Some of the solvents (Toluene, DMSO, Acetone, and Ethanol) effectiveness was based on cursory work by Rachit Malik in the densification of the CNT sheet. Toluene and DMSO are considered good solvents for densification of CNT sheet. Ethanol and acetone are considered moderately good at densification of the CNT sheet. This trend is confirmed by the literature on solvation of SWCNT [43,44]. Then, Water was chosen because the CNTs are known to be hydrophobic and the CNTs in the thread shouldn’t be very soluble in water. The tension of the thread was initially tested at 0g and 1.2g of force. This was to hopefully show the importance of tension on the densification process. After many rounds of the testing it became apparent that the procedure worked well for the weaker solvents but produced some difficulties with the stronger solvents. So the tension values were changed from 0 and 1.2g to 0.55g, 0.8g, and 2.2g for some of the later tests.

The same testing fixture that is pictured in Figure 3.48 with a 1.2g weight suspended from the end of the thread was used to complete these experiments. First the thread was suspended in the boat and the end points were mapped using the ProScan III computerized x-y microscope stage. Once the end points were mapped the length was determined and ten points evenly spaced along the length were defined and images were taken at those points. The images were used to determine the average diameter of the section as shown in Figure 3.51. Once the length and diameter measurements were taken, the chemical densification process was performed. For the tests that called for tension during the densification process the 1.2g weight was left hanging during the entire soaking and drying process. For the
densifications that were performed under no tension, the weight was removed before the solvent was added and not re-attached until the solvent had dried completely. After the densification process, the length and diameter measurements were taken exactly as before.

The problem that was encountered with the test procedure was that the stronger solvents is that the while the thread was immersed in solvent the strength was decreased to the point that the 1.2g weight would cause it to fail. Even the when the thread was densified under no tension, often the process of drying and surface tension could cause the thread to fail. After many trials it was decided that there need to be a small amount of tension always applied to the thread to keep the surface tension from breaking the thread, but not as much as before to keep the tension from pulling the thread apart. Three different weights were used that would allow the thread to be tested at increasing loads instead of tension and no tension. This would hopefully allow for a more consistent test procedure.

Results

The results of the second densification experiments show a lot of variation in both length and diameter change. The trends with are not obvious and the data must be processes for some subtle trends to show up. The over noisiness of the data is believed to be cause by the random structure of the thread. The densification process is essentially am internal restructuring of the CNT thread. If the initial structure of the thread has very high fluctuation then the resulting structure change will also vary. The eliminating the variations in CNT thread structure and producing a structure with the highest density and CNT contact is the ultimate goal of densification. Therefore, understanding the effect of the initial structure on the densification is also important. The data from the second densification experiments is shown in Table 3.9 and Figure 3.55, shows some trends based on more than just the tension and solvent used. The Data is normalized based upon the highest value of the test. This comparison is so that trends can be more easily spotted. The diameter reduction of the thread is compared in the cross sectional area
format. This is because the density and other material properties (strength, resistivity, etc) are more directly related to the cross section area than the diameter.

<table>
<thead>
<tr>
<th>Test 1</th>
<th>Tension (g)</th>
<th>Avg dia (um)</th>
<th>Length (mm)</th>
<th>Long dia (um)</th>
<th>Length (mm)</th>
<th>Length Change %</th>
<th>Dia % Change</th>
<th>CS Area % Change</th>
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<td>Water</td>
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<td>27.35</td>
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<td>27.4</td>
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<td>23.1</td>
<td>33.19</td>
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<th>Length (mm)</th>
<th>Long dia (um)</th>
<th>Length (mm)</th>
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<th>Test 3</th>
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<th>Avg dia (um)</th>
<th>Length (mm)</th>
<th>Long dia (um)</th>
<th>Length (mm)</th>
<th>Length Change %</th>
<th>Dia % Change</th>
<th>Area % Change</th>
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<td>39.74</td>
<td>21.5</td>
<td>40.1</td>
<td>1.024</td>
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<td>-9.498</td>
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<td>Acetone</td>
<td>0.88</td>
<td>19.8</td>
<td>39.95</td>
<td>18.8</td>
<td>39.9</td>
<td>0.063</td>
<td>-5.051</td>
<td>-9.846</td>
</tr>
<tr>
<td>Acetone</td>
<td>2.2</td>
<td>20.5</td>
<td>42.76</td>
<td>17.6</td>
<td>42.8</td>
<td>0.115</td>
<td>-14.146</td>
<td>-26.291</td>
</tr>
<tr>
<td>N-Methyl P</td>
<td>0.55</td>
<td>23.6</td>
<td>30.05</td>
<td>14.8</td>
<td>30.5</td>
<td>1.571</td>
<td>-37.288</td>
<td>-60.672</td>
</tr>
<tr>
<td>N-Methyl P</td>
<td>0.88</td>
<td>17.6</td>
<td>31.22</td>
<td>15.5</td>
<td>31.2</td>
<td>0.016</td>
<td>-11.932</td>
<td>-22.440</td>
</tr>
<tr>
<td>Chloroform</td>
<td>0.55</td>
<td>23.9</td>
<td>42.30</td>
<td>22.2</td>
<td>42.6</td>
<td>0.702</td>
<td>-7.113</td>
<td>-13.720</td>
</tr>
<tr>
<td>Chloroform</td>
<td>0.88</td>
<td>24.4</td>
<td>39.34</td>
<td>17.6</td>
<td>40.0</td>
<td>1.667</td>
<td>-27.869</td>
<td>-47.971</td>
</tr>
</tbody>
</table>

Table 3.9. Full results from Densification Experiment 2
**Figure 3.55.** Normalized results of the Densification Experiment 2.
The most pronounced trend that comes from Figure 3.55 is that the classes of solvents that were established earlier tend to group together in their densification abilities. The solvents that were considered weak, water and Ethanol, both had a maximum normalized effects near 0.5. This means that they were half as effective at reducing the cross section and increasing the length as the best values in the test. The solvents that were considered good for densification, DMSO and Toluene, had maximum normalized values around 0.75. Then both the maximum values for diameter and length change occurred with the solvents that were identified in the literature as very strong CNT solvents. This is good support for the hypothesis that solvents that have a stronger ability to dissolve CNTs can produce the highest densification results. It is also important to note that the strong solvents were used with a lower tension than the other solvents because the CNT thread consistently broke once it was exposed to the solvents under higher tension. The tension didn’t seem to have as well defined trend as the solvents did. See Figure 3.56. The increasing tension didn’t seem to directly affect the diameter of the length change as originally thought. This could be simple masked by other factors in this experiment.
In order to produce more useable trends and to better understand the densification process. The initial thread cross section was considered as a possible variable that could affect the densification process. All the threads used for the entirety of the densification experiments were spun at one time from the same array. This means that the threads used for the second densification experiments were more towards the inside of the bobbin. It also means that they have a higher likelihood for damage that incurred from normal handling of the thread and bobbin. It was observed that the threads had a higher likelihood to be none circular in cross section. This could be because the inner layers of threads were compressed by the outer layers wound on top of it. It could also be from coincidental touching of the CNT thread by the experimenter. This compressing would lead to areas of increased density and possible contamination that would inhibit the infiltration of solvent into the thread.

**Figure 3.56.** Normalized Diameter and length Change compared with applied tension.
In order to visualize the effect of initial cross section on densification the total data was accumulated and plotted. The diameter and normalized length change were compared to the initial cross section for every test performed in the second densification experiment. See Figure 3.57 and 3.58. The experimentally observed tendency was that the threads that had a smaller and less uniform cross section didn’t show as much diameter and length change as the threads that had larger initial cross sections. When the data was plotted there didn’t seem to be an obvious trend. However, if we assume that a number of the samples had some contamination or even just a certain internal structure that prevented them from being densified there is a trend that immerses. If we assume that a number of the samples were not going to show significant dimensional changes no matter what densification process they were subjected to then we exclude all the data points that showed less than about 20% and 0.1 d diameter and normalize length change respectively. Once these data points are eliminated then an upward trend immerses that suggest that the larger the initial cross section the larger the dimensional changes.
**Figure 3.57.** Normalized Diameter change compared to Initial cross section. The red oval is the data points that are excluded from the trend and black line is the resulting trend.

**Figure 3.58.** Normalized length change compared to Initial cross section. The red oval is the data points that are excluded from the trend and black line is the resulting trend.
For the diameter change verses initial cross section, the most important characteristic is that the resulting trend has a slope that shows a greater factor for decrease in cross section than the increase in initial cross section. The trend shown by the black line in Figure 3.57 gives a 20% area decrease for a 300 um^2 intial cross section. This gives a final cross section of 240 um^2. Then, if the data is slightly extrapolated, a 500 um^2 initial cross section gives a 60% decrease in diameter to 200 um^2. This suggests that the larger the initial cross section the small the final cross section would be.

The length change shows a similar trend to the diameter change. When the data below 0.1 normalized change was removed a clear trend in produced. The surprising thing about the trend is that the largest length change was only measured to be 1.9%. So the data excluded was length changes below 0.19%. In many cases, this change is less than 100um. Even though thought the changes were very small it still appeared to depend greatly on the initial diameter, even more so than the solvent used or the amount of tension. The grouping of the data into a linear trend seems to be tighter than the diameter change. It also doesn’t appear that, based on Figure 3.55, the length change and diameter changes are as closely related as previously thought. This means that the length change can’t be directly attributed to a Poisson ratio effect. The correlation of initial diameter to length change is a suggestion that the inter spacing of the CNTs is very important to allow the solvent to penetrate the thread to allow the CNTs to slip relative to each other.

The results presented above are based on the data derived from the experiments as a whole. There are also some important results that can be determined by looking at the data for each individual experiment. In particular the difference in thread structure after the densification process is consistently different based on the solvent used. The phenomenon that is the distribution of individual CNT thread can vary greatly even in short sample lengths. It has been observed that the as spun thread can vary in diameter as much as 6 um in just a few centimeters, as shown in Figure 3.49. Also, it is important to
keep in mind that these diameter measurements are based on the image averaging technique shown in Figure 3.51. The densification process helps to reduce the amount of diameter variance. What is discovered by comparing Figure 3.59 with Figure 3.60 is that the stronger the solvent the better the densification process is at equalizing the diameter along the length. It is also of tremendous interest that the two densification tests using N-Methyl-Pyrolidinone, shown in Figure 3.60, started with very different diameters and the resulting diameter turn out to be very close. This is likely due to the solvent being able to easily penetrate the thread even though it might have had areas with that were compressed or contaminated.

\[ \text{Figure 3.59. Diameter Changes for weak CNT solvents} \]
Figure 3.59. Diameter Changes for a strong CNT solvents

The overall conclusion based upon all the trend, observation, details and experience gained is that the chemical densification process is more than just a simple increase in density. Some of the sample underwent a very significant dimensional change and increased in density. Perhaps more important than the averaged dimensional changes, the as spun threads seem to have a very large range of micro and possibly nano structures, even over very short distances. This is likely due to the randomness of the CNT arrays and ribbons from which the threads are produced. This random structure of the ribbon directly translates to a random distribution of stress and mass in the thread. The densification process, if performed under certain conditions, seems to allow for a re-distribution and equalization of the internal stresses and linear mass of the thread.

With respect to glass, annealing can be defined as “a process of slowly cooling glass to relieve internal stresses after it was formed.”[45] For metals, “Annealing is used to induce ductility, soften material, relieve internal stresses, refine the structure by making it homogeneous”[46]. It may be more accurate to describe densification as chemical annealing. This is very important to the continued improvement of
the CNT thread and there should be much more extensive and controlled experiments that build upon the lessons learned and data gathered so far.
4. Electromagnetics Background

4.1. Electromagnetic Theories

In standard model of particle physics, there are four fundamental forces that govern how elementary particles interact with each other. Those forces are the strong nuclear force, the weak nuclear force, gravity, and electromagnetism[47]. A magnetic field is the result of electrons spinning and when groups of atoms have electrons that are spinning along parallel axes they are considered a magnetic domain in the direction of the spin axis. When magnetic domains are all aligned and fixed in that orientation they can become a permanent magnet[48]. Also, the movement of a charge though a space can cause the direction of the magnetic domains to align. We represent this alignment of domains with lines of flux that form closed loops. The strength of the magnetic field is represented by the spacing of the flux lines, also known as flux density[49]. This chapter will provide a background for electromagnetic laws as they pertain to electric motors and devices.

4.1.1. Electrostatics and Electric Fields

The most basic manifestation of electric charge is the point charge. A point charge has no mass or volume and can represent an atom, a molecule, or a particle. Point charges can have varying strength positive or negative charge value. Coulombs law, shown in Equation 4.1, relates the forces that stationary point charges apply on each other based on the value of the charge and the distance between them. It states that the force between particles varies linearly with the charge of the particles and by the distance squared. It also states that the force is always along an axis that goes though both points and like charged particles repel and opposite charges attract. A diagram is shown in Figure 4.1 of the forces between charged particles.

\[
F = \frac{1}{4\pi\varepsilon_0} \frac{q_1q_2r_{21}^2}{r_{21}^2}
\]
Equation 4.1. Force between two charged particles

![Diagram of two charged particles with electric field lines]

**Figure 4.1.** Forces of point charges. Representation of the electric field between two particles.

If we have a series of particles in a space and they form a system where, if an additional charged particle were placed in the system then it would have a resulting net force. We can define the force on the new particle in the space by applying Coulomb's law to each particle in the system. If the force on an unknown particle were calculated and plotted for each point in the space you would be able to visualize a field that would act on any particle in the space. This field is called an electric field. The electric field lines are always tangent to the resulting force on a particle placed at that point. An example of the electric field between two oppositely charged particles is shown in Figure 4.1. The density of the electric field lines also represents the strength of the electric field in Newton per Coulomb. The unit of Newtons per coulomb means that if the charge of the said particle is doubled then the force acting on it also doubles.

If you consider an infinite flat plate of constant charge density instead of a point charge then the resulting electric field lines form a different pattern. If the plate is considered to be infinite in its plane then the resultant field lines will all be parallel to each other and perpendicular to the plate. They also extend in both directions away from the sheet. If two plates are sufficiently large and close together
then the field lines can be approximated to be all parallel with each other as shown in Figure 4.2 [49].

This configuration with a dielectric material place between the two plates is used in capacitors.

Using the idea of point charges, we imagine a rigid object with a positive point charge attached to one end of it and a negative point charge attached to the other end. This type of structure can be considered a dipole. If the material is placed in a uniform parallel electric field it will have a force that will try to align it with the field lines[49]. An example is shown in Figure 4.2. If a material is placed in the electric field and the charges in the material are free to move then the material can become a dipole.

**Figure 4.2.** Electric Field between two parallel plates of opposite charge. Forces on a dipole in an electric field.

### 4.1.2. Magnetic Field from Conductors

In 1819 Hans Christian Oersted discovered that there is a relationship between moving charge or current and the needle on a compass. He placed a compass horizontally above a wire with current passing through it. The need was placed parallel to the wire and then when the current was turned on the needle turned perpendicular to the wire. When the current was reversed the need switched directions. This was the first discovery of the interaction between moving charge and magnetism. This lead to the relationship described in Equation 4.2, which states that a force moving in a magnetic field experiences
a force that is perpendicular to both the magnetic field and its own velocity. This force can be observed in an experiment that observes the forces on parallel wires carrying current. See Figure 4.3. Wires with current in the same direction attract, and wires with opposing currents repel.

\[
F = qE + \frac{q}{c} v \times B
\]

**Equation 4.2.** Force acting on a charging moving in a Magnetic Field

**Figure 4.3.** Interaction between wires carrying current [49]. Purcell E., 2011, Electricity and Magnetism, Cambridge University Press.

The interaction between two wires with parallel current shows how the forces act on moving charges effect one another. We can apply Equation 4.2 to a single point charge moving parallel to a conductor, see Figure 4.4. This shows that the magnetic field lines form circles around a straight conductor. If we take the conductor and wrap it to form a loop or coil then the magnetic field lines are condensed in the center of the coil and then travel back around to the other side. This forms the same pattern similar to a permanent magnet. This comparison is shown in Figure 4.2.
Figure 4.4. Shows the force on a moving charge in and the magnetic field [49]. Purcell E., 2011, Electricity and Magnetism, Cambridge University Press. The magnetic field associated with a conductor, a coil, and a permanent magnet [48]. Hubert C. I., 1991, Electric Machines, Theory, Operation, Application, Adjustment and Control, Macmillan Publishing Company.

Magnetic fields are represented as lines of flux. These lines of flux have a direction and the strength of the magnetic field is determined by the density of the flux lines. The closer the flux lines are together, the higher the flux density and the stronger the magnetic field. In addition, the flux lines must always make a full, closed loop. Magnetic flux can be analogous to electric circuit in that the flux lines will always take the path of least resistance. The magnetic equivalent to electrical resistance is reluctance. The higher the reluctance the hard it is for magnetic flux to form in the material. If a material with low reluctance in placed in proximity of a magnetic circuit, the flux will prefer to travel along the material instead of through the air or other paths of higher reluctance. The parameter that is equivalent to the voltage in a magnetic circuit is the magnetomotive force (abbreviated by mmf or H). If either the mmf is increased or the reluctance is decreased then the flux in the circuit is increased. Therefore the flux can be thought of as the current in a magnetic circuit[48].
4.1.3. Faraday’s Law of Induction

In 1831, after Oersted discovered the interaction between current and magnetic fields, Michael Faraday was performing experiments to better understand the phenomenon. He was trying to understand how currents affect magnetic fields. He was disappointed that current in wire passing near a second wire didn’t produce current in the second wire. He tested this by wrapping a coil with two wires. One of the coils was connected to a battery and the other was connected a galvanometer, which was a device to detect current. An artist’s rendering of the experiment is shown in Figure 4.5. The direct current didn’t induce current in the second coil. However, he observed that the when the current was turned on and off that there was a small twitch in the galvanometer. This was the discovery that changing magnetic flux produces an electro motive force. He published his work under the title “Experimental research in Electricity”.

![Figure 4.5](image)

**Figure 4.5.** Experiments done by Faraday. The experiment where the galvanometer jumped when the current was stopped and started. Shows an example of a dc generator using magnetic induction that was performed by Faraday [49]. Purcell E., 2011, Electricity and Magnetism, Cambridge University Press.
Faraday's discovery shows that changing magnetic field can induce current in a conductor. This can be modified for use in electric machines as equation 4.3. This states that the voltage generated in a coil of wire is equal to the number of turns time the rate of flux change. This means that an increase in the frequency of AC current or an increase in the number of turns in a coil will produce a higher voltage in the system.

\[ e = N \frac{d\Phi}{dt} \]

**Equation 4.3.** Equation for induced voltage in a coil. \( e \) – Induced Voltage (V), \( N \) = number of turn is the coil, \( \frac{d\Phi}{dt} \) – change in magnetic flux (Wb/s)

Electromagnetic induction is used in nearly all of modern electronics and power equipment. The AC transformer, shown in Figure 4.6, is a very basic example of Faraday's discovery. A transformer has one coil, also known as the primary coil, wrapped around a magnetic core. A second coil, also known as a secondary coil, is also wrapped around the core. An alternating current is passed through the primary coil and this produces an alternating flux in the core. The alternating flux in turn produces an alternating voltage in the secondary windings. By varying the number of turns in the coil we can adjust the voltage in the secondary. This is useful in power transmission because the electrical power is transmitted long distances as very high voltage in order to minimize losses. Then the voltage is reduced with a series of transformers to a safe level for household use. Another use of electromagnetic induction is the generation of electrical power from mechanical power. An example of a generator is shown in Figure 4.6. When a coil is rotated in a constant magnetic field it experiences a changing flux. The flux passing through the coils is highest when the coil is perpendicular to the flux lines and zero when the coil is parallel to the flux lines. If the coil is rotated at constant angular velocity then the flux experienced by the coil and the voltage produced will be sinusoidal. This can be modified to produce DC current by
using commentators that switch the contacts of the coil or rectifiers to produce a voltage with constant polarity.

**Figure 4.6.** A schematic of a transformer. A simple schematic of an electric generator.

Electromagnetic induction can be incredibly useful and powerful, but it can also have some undesired consequences as well. The most common side effect is the formation of eddy currents. Eddy currents are formed when a conductor is in the presence of an alternating magnetic field. The magnetic field induces voltage and therefore currents in the conductor similar to those produced in a coil or transformer. This alternating current will be circular and concentric with the coil producing it. A diagram is shown in Figure 4.7. The eddy currents then produce a magnetic field that is opposite to the field that produced it. This can contribute to power loss due to the eddy current flowing in the conductor and decreases in the magnetic field strength produced by the primary coil.
Figure 4.7. Shows the formation of eddy currents. a) Shows the magnetic flux produced by a coil. b) The formation of eddy currents produced when the coil is placed near the surface of a conductor. c) The resulting magnetic flux produced by the eddy currents that opposes the original flux from the coil. “Eddy current image” [Online]. Available: http://static2.olympus-ims.com/data/Image/eca-tutorial/Principe_CF_04.gif?rev=6B67.

There is one type of transformer that is important for use with electric motors. That is a rotational transformer. The rotational transformer is a variation of the basic transformer but it has an air gap between the rotor and stator that allows them to rotate relative to each other. Some example rotational transformers are shown in Figure 4.8. The axis of rotation is through the axis of the coils so that the mutual magnetic flux isn’t disturbed by the rotation[50].
4.1.4. Magnetic Materials and Cores

When designing a magnetic core for an electric machine there are many different considerations that must be taken into account. One of the most basic ones is the core geometry. There are many different types of magnetic losses that can occur in electric machines if core geometry is not considered. Flux leakage, shown in Figure 4.9, is a common occurrence in transformers. This is where some of the flux doesn’t travel in the core as desired but instead may jump or leak through the air \[48\]. In a transformer, as shown in equation 4.3, the voltage generated in a coil is proportional to the amount of flux that passes through it. If some of the flux is leaking then the voltage developed in the secondary coil will be decreased\[49\]. This effect is minimized by winding the primary and secondary coils concentrically on top of each other. Another form of magnetic loss called fringing flux is shown in figure 4.9 \[48\]. This happens at an air gap or a point of higher reluctance (magnetic resistance) in a magnetic circuit. Sometimes air gaps are intentionally used in transformers to regulate the amount of flux. Other times there may be an unintentional restriction in the cross section of the core perpendicular to the flux lines.
This restriction will cause the flux density to increase and may cause some of the flux to fringe around the restriction. Fringing is also common at sharp corners in a core [48].


Eddy currents as discussed above can contribute to massive losses in a core if they are not considered in a design. All commercial magnetic material used in power electronics and electric machines are made of bulk metals. Iron and different steel alloys are the most common. The problem with Iron and steel is that they are also very conductive. So if they were used in a solid form the eddy current losses would be very large even at low frequency. What is done commercially is the Iron or steel is cut or stamped from thin sheets. Then these thin sheets are stacked with a thin film of insulating glue between them. This prevents current from passing from one sheet to another. The currents must flow in a circle according to Faraday’s law of induction, the current in any given area is reduced. A comparison is shown in Figure 4.10.
In order to determine what magnetic material should be used for an application the properties of magnetic materials should be understood. For magnetic materials, there is very extensive scientific research into the atomic and quantum phenomenon that lead to the macroscopic magnetic effects. For the purpose of this document, the background of magnetic materials will be limited to information necessary to make engineering design considerations and what is important to support the magnetic developments presented herein.

There are several different magnetic material properties that must be understood when choosing a material for a certain application. The first discussed here, but not necessarily the most important, is permeability or susceptibility. In general terms this is the ratio of the magnetomotive force (mmf or H field) and the resulting magnetic field. It is often measured relative to air, call relative permeability. For example if a material has a relative permeability of 100 then for a given applied H field, the resulting magnetic flux density (B field) will be 100 times greater than if the same H field is applied to air. This value is also dependent on the value of the applied H field and the previous magnetic state of the material. Permeability can also be determined from the slope of B-H curve seen in Figure 4.11.

Other properties that can be determined from the example plots shown in Figure 4.11 are saturation magnetization, residual magnetization and magnetic coercive force. Magnetic saturation is a property that all materials have. It occurs when all of the magnetic domains of a material align and they stop contributing to the flux density. It can also be defined as the H field value when the relative permeability drops to 1. So only free space in contributing to the increasing magnetic flux. Once a material is saturated and then the applied mmf is reduced to zero, there may still be a magnetic field present in the material. The strength of this field is known as the residual flux density. For a permanent magnet, this is the maximum magnetic field that can be maintained. If the strength of the H field were to be increased but in the opposite direction of residual magnetization, then the material would eventually reach a zero magnetization state. The strength of this opposing field is known as the magnetic coercive force. As the strength of the applied H field is increased the material reaches another saturation state. If the strength of the H field is oscillated between the saturation states of a material then an ‘S’ shaped curve is created. This is known as the B-H curve, or Magnetization curve, examples of both are shown in Figure 4.11. The area inside of this loop is known as hysteresis and is equivalent to energy during the cycle. The larger that area inside the curve the more energy is lost on every cycle.
Based upon the magnetic properties of a material we can classify materials into different categories. In order to keep the distinctions clear only three types of magnetic materials will be discussed here, ferromagnetic, paramagnetic, and superparamagnetic. Most magnetic materials used in electric machines and all materials used in permanent magnetics are considered ferromagnetic. Ferromagnetic materials are generally characterized by high saturation and remnant magnetizations. They also have a magnetization curve with large hysteresis. This is good for making permanent magnets where the remnant magnetization determines the strength of the magnet. But in machines like electric motors, the large hysteresis means that a lot of energy is lost in the form of heat during every cycle of the current. Paramagnetic materials don’t have the hysteresis that ferromagnetic materials do. They can go through cycles in magnetization without generating heat in the core. The down fall of paramagnetic materials is that they have low saturation magnetization and permeability in comparison to ferromagnetic materials.
There is a phenomenon known as superparamagnetism that occurs when a ferromagnetic materials is made in very small particles and placed very close to each other. The particle size and spacing needed to achieve superparamagnetism is different for each material. The characteristics of superparamagnetic materials are magnetic saturation and permeability like ferromagnetic materials and zero hysteresis like paramagnetic materials. This means that they can achieve high magnetic flux densities and still have zero or near zero remnant magnetization and coercive force. A comparison or ferro-, para- and superparamagnetic magnetization curves is shown in Figure 4.11.

4.1.5. Permanent magnets

In general, Permanent magnets are made from materials that are ferromagnetic. The best materials are those that have a large coercive force so that it is difficult for them to loss their magnetization. Another term used to describe these materials is ‘magnetically hard’. One of the most common materials that is used for making permanent magnet is an alloy made from Iron, Aluminum, Nickel, and cobalt, abbreviated Alnico. Examples of commercially available magnets are shown in Figure 4.12. These metals are sintered together and casted under the application of a strong magnetic field. The heat treatment and magnetic alignment procedure leave the material with a very high coercive force at room temperature. They can have a magnetic flux density of up to 1500 gauss [3]. Another type of permanent magnet based on hematite (Fe$_2$O$_3$) and magnetite (Fe$_3$O$_4$) are known as ferrite or ceramic magnets. They are made into ceramics with different elements like Strontium, Barium and Cobalt to make materials that have very high coercive forces. Examples of commercially available Ceramic magnets are shown in Figure 4.12. Ceramic magnets can hold up to 700 gauss [3]. The raw materials used to make these magnets are widely available and the process is well established so the price of Alnico magnet is relatively low.
There is another class of magnets known as rare earth magnets because of the use of elements like Samarium and Neodymium. Commercially available rare earth magnets are shown in Figure 4.13. These elements are classified as rare earth materials even though they are about as common as Tin in the earth’s crust [3]. These types of magnets can have residual magnetizations as high as 2800 gauss. The strength of these magnets has made them extremely popular for use in everything from electric motors to toys. Neodymium magnets are prone to oxidation so they are usually covered is a zinc coating to make them shiny, shown in Figure 4.13. One problem with them is that they are very brittle and chip easily. They can also cause injury if a person’s hand is caught between two magnets that snap together [3]. The biggest issue associated with rare earth elements is the geopolitical situations surrounding the locations for these materials. The largest production of rare earth elements is in China and because of this they are strategically controlling the proliferation of these elements [51].
4.2. Modern Electromagnetic Devices

Ever sense electricity was discovered it has been an important goal to figure out how to use the electrical power to do work for people. There are many different types of electric machines that can be made. Some of the basic types of electric machines are described below. Solenoids are simple and many other machines use some form of solenoid coils in them. Therefore it is important to understand the principles associated with solenoids. Electrical transformers are used throughout our power grid and are important for the distribution of electricity. Their role is to increase voltage for transmission and decrease voltage for safe usage. They are also a great example of electromagnetic principles.

Electric Motors and generators are used with more and more frequency in the modern world. Electricity is the universal power medium and can be generated with zero impact to the environment. So the development of new and better electric motors can have a wide reaching effect in everything from power plants to electric cars and airplanes. Every motor has two basic components, a rotor and a stator.
The stator is the frame or the stationary part of the motor. It is generally on the outside. The rotor is the part that spins inside of the stator. It is connected to the shaft or object that is to be turned. There are many different designs of motors for different applications. These motors can be classified in many different ways. Below, they will be divided into two categories based on the whether the input power is Alternating Current (AC) or Direct Current (DC).

4.2.1. Solenoids and transformers

A solenoid is a coil of wire around an axis and is one of the simplest magnetic machines. When current is passed through the coil it generates a magnetic field similar to a bar permanent magnet. The major difference is that the solenoid can be turned off and can vary in strength depending on how much current is passed through it. Solenoid coils are generally used for linear actuators. They consist of a magnetic core material that is able to slide inside the coil. When current passes through the solenoid it pulls the magnetic material towards the center of the coil. Once the current is released the core is generally moved back to its initial position by a spring. A schematic and pictures of industrial solenoids are shown in Figure 4.14. Solenoids are important because most electric machines use solenoids as their method to generate magnetic fields.
Figure 4.14. Schematic of flux in a solenoid. Examples of commercially available solenoid actuators.


A transformer is two or more solenoid coils that share a flux path. When alternating current is passed through one coil then the resulting alternating flux induces voltage in the second coil. The ratio of the number of turns between the primary and secondary coils determines the ratio of the voltages between the two coils. The inverse of the turn ratio determines the maximum current that can be induced in the second coil. Generally, power transformers use steel laminate cores to reduce eddy current. The steel is a silicon steel alloy that features low coercive force to reduce hysteresis loss. The laminates are stacked on top of each other to add thickness. Many times there are two pieces so that concentric coils can be slid on to one section of the core and then the two core pieces are fused together. This forms a closed loop for the magnetic field. Another core configuration is a toroid transformer core. The problem of the toroid is that the wire must wound around the toroid which is more difficult than winding a solenoid on a straight core. Examples of commercial transformers are shown in Figure 4.15.
4.2.2. Direct Current Motors

DC Commutated Motor

One of the most basic types of electric motors is the DC commutated motor. It uses a one or more permanent magnet for the stator poles. The rotor consists of one or more coils of wire. The wires can be wrapped around a magnetic core in order to increase the flux. The key to a DC commutated motor is a set of brushes that change the direction of the current in the rotor. They rub against a set of
commutators or metal contacts that are connected to the rotor coils. As mentioned above, a solenoid or coil of wire can be thought of as a magnet with switchable polarity. So the brushes are configured so that once the magnetic flux in the rotor becomes aligned with the stator it switches polarity. This switching produces continual torque on the rotor and produces rotational motion.

![Typical Brushed Motor in Cross-section](image)


DC commutated motors were very popular in the past for many reasons. One of their common characteristics is high starting torque. The torque curves of DC commutated motors are generally flatter than other motors. Using the brushes to switch the polarity of the rotor means that the switching happens as fast as the rotor spins. This leads to some motors having a run-away speed problem when they don’t have any load on the rotor. This can lead to catastrophic mechanical failure due to centripetal force. The brushes used to transfer power to the rotor (usually made of graphite) can be another source of failure. The brushes generate sparks and friction that heat up and wear away the brush material. So they require periodic maintenance to change the brushes. DC commutated motors are inherently simple because they don’t have any controls other than input power. This made them popular in the
past when control electronics were not available. However, they have become less popular as electronics have become cheaper and more powerful.

**DC Brushless Motor**

A variation of the DC commutated motor, the brushless DC motor uses permanent magnets for the rotor and coils of wire for the stator. See Figure 4.17. The difference is that the commutating or stator coil polarity switching is done by solid-state electronics instead of brushes. The lack of brushes means that Brushless DC motors require less maintenance than brushed commutated DC motors. The switching instead must be done by electronics, generally called a motor controller. The motor controllers add cost to the system but offer a much greater level of control. DC brushless motors with controllers can offer precise speed and position control. Having speed control increases the number of applications over the brushed DC motor. As electronics become cheaper and increasingly integrated into everything that we do Brushless DC motors increase in popularity.

![Figure 4.17. Brushless DC motor diagram and examples. “DC brushless 1” [Online]. Available:](http://experimentalev.wordpress.com/2011/03/22/motor-brush-replacment-how-to/).

http://www.phonature.com/education_DIYMotor.htm.; https://encrypted-tbn1.gstatic.com/images?q=tbn:ANd9GcTT8NnmyeXWiNT-jw1k_xJSv1xYmJbS01__V2ZsItNDVcJLqK_
4.2.3. Alternating Current Motors

Induction Motor

Likely the simplest of all electric motor designs, an AC induction motor has no moving parts other than the rotor. The stator consists of a set of coils that face inward to the rotor. The coils are powered by alternating current that is generally multiphase. The multiphase current going through the coils produces a magnetic flux with constantly rotating orientation. A diagram of the magnetic flux in a 3 phase, 6 pole stator is shown in Figure 4.18. The rotating flux induces current in the rotor instead of powering the rotor with external power. The rotor can have several different configurations. One type is a wound rotor induction motor. This has coils of wire that interact with rotating field, inducing current in them. Another type of rotor is known as a squire cage induction motor. It was bars of conducting metals that act like the windings in a coil. Some examples of squire cage rotors are shown in Figure 4.18. This type of motor is less variable than a wound rotor because in a wound rotor resistances can be placed in line with the coil to modify the speed torque characteristics. However, it is simpler and can produce a stronger interaction with the field than a wound rotor.

AC induction motors are based on the rotating magnetic field produced by the stator. This field rotates at a constant speed known as the synchronous speed. It is defined by Equation 4.3. The ratio of the difference of the two speeds to the synchronous speed is known as the slip. Induction motors are known as asynchronous machines because the current is produce in the rotor due to the difference in speed between the flux and the rotor. Therefore the Induction motor can never reach synchronous speed under its own power. The simplicity of the induction motor is a big advantage in cost and longevity. There is no need for any control electronics or regular maintenance. The down side to Induction motors is they have little torque at low speeds. An example of an Induction motor torque curve is shown in Figure 4.19.

\[ n_s = \frac{60 \times f_s}{p}, \quad s = \frac{n_s - n_r}{n_s} \]

Equations 4.3, 4.4. \( n_s \) – synchronous speed; \( f_s \) – frequency of input power; \( P \) – number of pole pairs; \( s \) – slip; \( n_r \) – rotor speed. [48]

![Induction motor torque curve](http://cnx.org/content/m28334/latest/)


**AC synchronous Motor**

Like the Induction motor, the AC synchronous motor uses a series of coils in the stator and multiphase AC to create a rotating flux. The difference is that the field generated by the rotor is not due to an
induced current in the rotor but by other means. Permanent magnet, a magnetically hard material, through reluctance, or coils fed through slip rings are common methods of producing rotor flux. The major difference is that the rotor then rotates at the synchronous speed of the rotor flux. Due to the synchronous nature of the machine they only operate at synchronous speed as long as enough power is supplied. Some of the motors employ an induction aspect in order to get the machine to synchronous speed. AC synchronous motors are used extensively in industrial applications because of their simplicity and constant speed.

Induction and synchronous motors have a fixed synchronous speed based on the number of poles, the input frequency. Equation 4.3, shows how the synchronous speed is found. One method of speed control is to use a variable frequency drive to adjust the frequency of the input voltage. The frequency of the input voltage is directly proportional to the synchronous speed of the motor. For permanent magnet synchronous machines a variable frequency drive is often required for operation as they do not have conductors that can be utilized for an induction start.

**Universal Motor**

The universal motor is a unique motor in that it operates as a commutated DC motor but uses AC voltage supply. A diagram and example motor is shown in Figure 4.21. By using a wound coil stator and wiring it in series with the rotor, the motor will operated on any frequency AC or DC voltage. This has an advantage of operating at house hold outlet frequency and still maintains the high starting torque and high speed operation of a DC motor. For those reasons, universal motors are often used in blenders, vacuums, and other household consumer products.
**Figure 4.21.** Universal motor wiring diagram[48]. Picture of a commercial universal motor with labels for the different parts of the motor. Hubert C. I., 1991, Electric Machines, Theory, Operation, Application, Adjustment and Control, Macmillian Publishing Company; “Universal Motor” [Online].

5. Electromagnetic Developments

5.1. Electromagnetic Tests

5.1.1. Magnetic materials

The magnetic material research that is presented below was either done in collaboration with Rajiv Venkatasubraman or builds upon his work[52]. Venkatasubraman tried many different methods of producing a superparamagnetic material using a variety of purchased magnetic nano-particles. The method that is covered in this section is the method of mixing in the Nanoparticles into a polymer matrix.

The Sensorcyte

The type polymer matrixes chosen for this set of experiments were elastomers because the goal was to create a flexible magnetic material. The flexible magnetic material was desired for an application where a very small coil made around a flexible core could be placed in the blood stream. See Figure 5.1 for a diagram of the concept. This artificial blood cell, known as the Sensorcyte, was conceived by Dr. Mark Schulz.

![Figure 5.1. Sensorcyte Prototype designed by Dr. Mark Schulz. Image from “Bionic Artificial Cells for Diagnostics and Therapy” Presentation [53]. Schulz M. J., Li W., Mast D., Ruff B., Yin J., and Shanov V., 2012, “Bionic Artificial Cells for Diagnostics and Therapy.”](image-url)
The first version of a prototype Sensorcyte was built, in collaboration with Venkatasubraman, using a core material that used a Nitrile Rubber Elastomer that was synthesized by Dr. Noe Alvarez. The Rubber is synthesized in a highly diluted Acetone solution. The Acetone solution was then mixed with Iron Oxide nanoparticles that were purchased from www.us-nano.com. The polymer and nano particles were mix in approximately a 1:1 by weight ratio. The solution of elastomer and nanoparticles in acetone was then left in a vacuum chamber for 3 days until the acetone had fully evaporated. After the acetone had been evaporated the resulting Composite material was pressed into a mold with 0.1” diameter holes and heated to cross link the polymer.

One problem with this method was that it took so long to evaporate the acetone that a large portion of the magnetic nanoparticles settled to the bottom and precipitated out of the composite. It made it impossible to determine the final volume fraction of the composite. Despite the difficulty in mixing the composite, a handful of flexible magnetic core were made. They appeared to display paramagnetic behavior when an external magnetic field was applied. See Figure 5.2 for series of pictures showing the particles grouping together when a permanent magnet is placed nearby and then removed.

![Figure 5.2](image-url) Shows prototype Sensorcyte cores in the Presence of a magnetic field and the after the magnetic field is removed.
In order to test if the Sensorcyte concept would work the best core that was made was wrapped with a CNT thread to make a solenoid. This solenoid is the first prototype of the Sensorcyte design. The prototype is then placed in side of a set of Helmholtz coils that were driven with alternating current. The resulting voltage in the Sensorcyte prototype was measured. The setup and results are shown in Figure 5.3. This configuration was basically using the Sensorcyte as the secondary of a transformer and the Helmholtz coils as the primary. If the design could be scaled down this is evidence that it could be used to power a variety of devices that could measure velocity or pressure in a vein or artery.

![Prototype Sensorcyte Test. Optical microscope image of the Sensorcyte.](image)

**Figure 5.3.** Prototype Sensorcyte Test. Optical microscope image of the Sensorcyte. [53] Schulz M. J., Li W., Mast D., Ruff B., Yin J., and Shanov V., 2012, “Bionic Artificial Cells for Diagnostics and Therapy.”

**Freeman Elastomer Magnetic Nano Composite**

The difficulty that was encountered in the fabrication of the flexible magnetic cores using the nitrile rubber solution gave motivation to find a new method of fabrication. Dylan Van Dyne assisted in using a two part urethane elastomer mas purchased from freemansupply.com. The Freeman1035 Mold Making Polyurethane Elastomer was used and is shown in Figure 5.4. To make a magnetic nano composite, the same magnetic nanoparticles that were used to make the first prototype Sensorcyte were used. Nano particles were added to each part of Freeman1035 independently. Part A of the system is thicker than Part B so more particles were able to be added. Then each part was mixed with the high shear mixer to
disperse the particles. Once each part was mixed separately, part A and B were mixed together and then poured into various molds. The advantage of the Freeman 1035 is that it doesn't rely on any external heating or evaporation to cure. After 12 hours the parts can be removed from the molds. Pictured in Figure 5.4 are the first magnetic composites made with the Freeman 1035 flexible elastomer. This method of making the composite was much easier. It also can easily be made with a rigid urethane to obtain a rigid magnetic nano composite for use in an electric motor.

![Image](image_url)

**Figure 5.4.** The two part Freeman 1035 elastomer. The Silverson High Shear mixer used to mix particles into the matrix. Various cured flexible magnetic nano composites made with help from Van Dyne.

### 5.1.2. CNT/Copper Solenoid Transformer

One of the most basic electric machines that can be tested is the solenoid. It is also critical to be able to make and operate a solenoid if more complicated machines are to be built. The first tests of making a CNT coil were performed by wrapping a CNT thread around a quartz tube. The wrapping was done by hand using a reeling stage that was hand operated. Then the thread position was adjusted so that the CNT thread didn’t overlap itself. This thread was coated but the coating was unreliable so a small gap was left between each turn in the coil. The coil was then tested using a DC power supply and a Hall...
Effect sensor. The resistance of the coil was found to be 9.37 kΩ and the highest available power supply was 61.5 V. This only allowed a current of 6 mA which isn’t enough H field for the Hall Effect sensor to pick up. See Figure 5.5 for a picture of the first CNT coil test.

![Image](image1.png)

**Figure 5.5.** Picture of the first hand made CNT coil at UC.

The first CNT coil had a resistance of 9.7 kΩ which was too high for the available power supply to pass enough current through. It was made using a quartz tube that was approximately 10mm in diameter. The equations that define what the flux density is inside a solenoid are not affected by the diameter of the coil. Therefore, in order to reduce the resistance without decreasing the magnetic flux, the diameter of the coil was reduced to decrease the total length of the wire in the coil. This time a copper wire was used to make a second coil that was on top of the CNT coil. The diameter of the thread was small enough that it fit underneath and between the copper wire turns as shown in Figure 5.6. Then the coil was held in place with clear nail polish to prevent the coil from unraveling. One side effect of the nail polish was that it started to melt and oxidize when the coil heated up from the current. The coil after testing is shown in Figure 5.6. Both the CNT and copper coils were tested using a DC power supply and a Hall Effect sensor. The data was transformed into the magnetomotive force (H) and the magnetic flux density (B) using equations 5.1 and 5.2 below. The results of the DC magnetic field tests are shown in Figure 5.7.
Figure 5.6. Diagram of the cross section of the CNT/copper coils. Picture of the CNT/copper coil after testing.

<table>
<thead>
<tr>
<th></th>
<th>copper</th>
<th>CNT</th>
</tr>
</thead>
<tbody>
<tr>
<td># of turns</td>
<td>23</td>
<td>23</td>
</tr>
<tr>
<td>R (Ω)</td>
<td>1217</td>
<td>3470</td>
</tr>
<tr>
<td>Dcoil (mm)</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Lcoil (mm)</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Lcoil (m)</td>
<td>0.004</td>
<td>0.004</td>
</tr>
<tr>
<td>Dwire (mm)</td>
<td>0.175</td>
<td>0.04</td>
</tr>
</tbody>
</table>

Table 5.1. properties of CNT/Copper coil.

\[ H = \frac{Ni}{l} , \quad B = \mu H \]

Equation 5.1 and 5.2
The results from the DC magnetic field test of the CNT/copper coil. The magnetic flux density $B$, was measured using a Hall Effect sensor made by Dr. Mast.

The DC test of the CNT/copper coil was successful and confirmed that the CNT thread can be used to generate a magnetic field. There was some deviation from the theoretical linear line that should have been produced on the $B$-$H$ curve. There were some experimental errors that could have accounted for deviation, flux leakage, movement from the sensor, variations in temperature, etc. These errors could have been investigated further but because they would have only proved existing magnetic theory it was determined to move forward instead.

The next test to be performed was an Alternating Current test. The Hall Effect sensor that was used in the DC test isn’t designed for higher frequency use. Instead, the best way to measure an alternating magnetic field is with a second coil of wire. Faraday’s law of Induction can be used to determine the magnetic flux change in a coil of wire. The equipment and setup was done with supervision from Dr. mast. First the Copper coil was driven with AC current and the voltage was measure at different

**Figure 5.7.** The results from the DC magnetic field test of the CNT/copper coil. The magnetic flux density $B$, was measured using a Hall Effect sensor made by Dr. Mast.
frequencies up to 50 MHz. Then the CNT coil was driven and the voltage in the second coil was measured. The results are shown in Figure 5.8 and 5.9.

**Figure 5.8.** Air core transformer with copper primary and CNT secondary

**Figure 5.9.** Air core transformer with CNT primary and copper secondary.
A test like this between two coils of different materials and resistances is not common. Many of the
equations that are used to define transformers make assumptions that can’t be used to determine why
there is such a difference in the response of the transformer when the different coils are driven. There
could be a physics explanation or there could be a resonance of the circuit that could cause the different
peaks. There was one additional phenomenon that was discovered with respect to the use of CNT
thread in power electronics like transformers. It was found that when the copper coil was driven and the
CNT coil was used as secondary winding, there was a nice clean sign wave with a 90 degrees phase shift
of the secondary voltage. This is as expected from a transformer. However, when the CNT coil was
driven at certain frequencies there was a significant sine on sine wave form generated in the secondary
copper coil. To date this has not been fully explained. It was determined that the minor sine wave
frequency was approximately 13 MHz. It is interesting that the frequency response of the coil also had a
resonance just above 10 MHz. The different sine wave outputs are shown in Figure 5.10.
5.1.3. Halbach Array

A Halbach Array is a configuration of permanent magnets named for one of its discoverers Klaus Halbach [54]. It was first discovered by John C. Mallinson in 1973[55]. Then Halbach made an independent discovery in 1980 when trying to focus particle beams. It is generally described as an arrangement of permanent magnets that leads to a one-sided flux density. Examples of Halbach arrays are shown in Figure 5.11. This flux density is stronger and extends farther from the surface of the magnets compared to a single magnet [54]. The Halbach magnet array is used for various applications including electric motors. One example is the launch point motor, shown in Figure 5.11, designed for use with electric
UAVs [56,57]. The Halbach arrays allows the motor to be very efficient (95%) and have a high power density of 5 HP/lb [57].


The Halbach array is of interest in designing a new motor. The flux concentrations on one side mean that less flux is lost to the surrounding. The CNT thread is very small in diameter and this might allow us to use smaller coils in a motor. The downside to the decreased coil diameter is that the smaller the coil the shorter the flux lines extend from its surface. If a Halbach array could be used to help extend the length of the field to cross the air gap in between the motor it would be very advantageous. The one thing that hasn’t been found is whether the phenomenon is also true for wound coils. So an experiment was created to test the properties of electromagnetic coils in a Halbach configuration.

The concept of using small coils of wire arranged in a Halbach array is a novel idea. In order to provide some data to support the idea five small coils were made. The core material was machined in to “I” shapes from part of a laminate silicon steel transformer core. Each core is approximately a 0.4375” cube with a 0.125” wide and 0.125” deep slot cut in two opposing sides. Each coil was then wound with 100
turns of copper magnet wire. The coils were then wired in series and tested in two configurations. See Figure 5.12.

Figure 5.12. (a) Small magnetic coil used for testing different configurations; (b) Standard series of coils all oriented in the same direction; (c) Halbach array of electromagnetic coils

The test involved placing a Hall sensor with approximately 0.1” above the coils. Magnetic flux readings were taken every 0.05” across the centerline of the coils. In order to make the testing consistent a two axis stage was used to move the Hall Effect sensor along the axis of the array. The height of the platform that the arrays were set on was adjusted with a stack of shims. The platform remained at a constant level to keep the same geometry of the test. The hall sensor was moved along the length of the array and magnetic flux readings were taken at each position. Pictures from the test are shown in Figure 5.13.
Figure 5.13. The x-y stage that was used to move the hall sensor along the magnetic arrays. Picture down the axis shows the spacing of the sensor from the array.

First, the coils were arranged all in the same direction and placed in a series next to each other. This is a simple design that could be used in any electric motor. The other configuration that was tested is a Halbach array. The coils were oriented in a rotating pattern with the goal of increase magnetic flux on one side of the coils and near zero fields on the other. The same coils were used in each configuration. There was some difference in the performance of each coil so they were kept in the same order as well for a better comparison. In each test the current was kept constant at 0.5 Amps which is well below the maximum current density of the copper wire. The hall sensor can only measure magnetic flux in one direction. So for simplicity we only measured the vertical component of the field. Each coil was tested on the top and then flipped over to measure the field on the bottom. See Figure 5.14 and Figure 5.15.
Figure 5.14. Vertical component of the magnetic flux density along the center line on the top and bottom of a series of similarly oriented coils under constant current.

Figure 5.15. Vertical component of the magnetic flux density along the center line on the top and bottom of a series of coils arranged in a Halbach array configuration.
The series of similarly oriented coils produced a series of plateaus that were larger in the middle. This magnitude varies slightly between coils which could easily be due to variations in the steel core material or slight air gaps between the coils. The field lines all come out of the top of the coils then reenter on the bottom. The polarity of the magnetic field is opposite between the top and bottom. This is due to the rotation, relative to the hall sensor, of the coils to measure the field on the bottom. Relative to the coils themselves the field is in a constant direction through the core

The Halbach array of coils had more complicated results. The top showed the desired effect of increased flux density. As predicted the field is in a different direction in the middle than on the end. This is because the magnetic field lines come out of the array in the middle and then re-enter the array on the ends. On the bottom of the Halbach array the field is near zero except for the large positive and negative spikes that appear to happen right over the center of the 2nd and 4th coils. It’s difficult to fully understand the magnetic field without doing a finite element model but it does suggest that there is flux leakage around the 2nd and 4th coils. The most likely situation is shown in Figure 5.16. The pathways between around the edges have very high flux density and there is flux leakage to the surrounding air from the copper wire. This would lead to the large spikes in magnetic flux density in these spots.
Figure 5.16. Likely magnetic flux paths inside the Halbach array of electromagnetic coils. The white X and O denote current flowing in and out of the page respectively. This is a representation based on the experimental results, not a simulation result.

5.2. Electric Motor Designs

In order to make an electric motor without using copper or iron would only require a recreation of an existing motor. This wouldn’t be as effective as understanding all the properties of the materials and redesigning a motor to take advantage of all these properties. The correct motor design is critical in order to take advantage of the unique nano material properties. There is a large variety of electric motors that use very different methods of generating rotational power. Choosing the correct motor action and the correct geometry can help make the carbon motor the motor of choice for future applications.

5.2.1. Induction Motor

The CNT thread drops in impedance as the frequency of current increased into the megahertz range. So an induction motor was first investigated as a possible configuration for a carbon motor. The induction motor uses a series of poles and two or three phase input. The poles are configured so that the magnetic flux in the rotor is rotating. This rotating induces current in the rotor. The rotor is generally
one of two configurations, wound rotor, or squirrel cage. See Section 4.2.3 for more information on induction motors.

Early in the investigation of electric motors it was decided that making a squirrel cage rotor from the long CNTs grown at UC by Wongdong Cho would make a good application of CNTs in an electric motor. This was determined without very much understanding of how the squirrel cage rotor operated and was implemented. A commercially available electric induction motor was purchased and the rotor was removed. In its place a solid polystyrene rod of the same diameter was cut to the length of some of the long CNTs. This way the CNTs would span the entire height of the rotor and no electrical junctions needed to be made between the individual CNTs. Then slots were cut in the rod to place the long CNTs inside. The tops of the CNTs were connected with silver paste to complete the rotor circuit.

In order to test the new rotor a test stand was made. The purchased squirrel cage rotor was first tried in the test stand to verify that the stator was correctly established. The purchased motor worked and spun in the test stand. Then the long CNT squirrel cage rotor was tried in test stand and it didn’t move at all. In retrospect it seems like the iron laminates that are around the aluminum bars in the purchased rotor increase the magnetic flux in the rotor and in turn the current. The plastic material used to make the CNT rotor didn’t do the same. Some of the magnetic polymer might have been a much wiser choice for the supporting material. Also the CNTs were taken from an array and force into the slots. Only mechanical force was used to compress the bundles together. A solvent could have been used to help densify the CNTs so that a larger mass could have been used. A denser CNT material leads to a decrease in resistance and an increase in current.
Figure 5.17. The handmade long CNT rotor. The motor test stand with the purchased squirrel cage induction rotor.

The induction motor was chosen initially because it uses alternating current to drive the stator coils. This was thought to have an advantage for use with CNT thread because the thread resistivity drops at very high frequency. Based on the synchronous speed equation for a 3 phase induction motor with 3 pole pairs was operated at 10MHz the synchronous speed would be 200,000,000 rpm. This is about 10,000 times faster than commonly available high speed electric motors. The amount of centripetal force on the rotor at those speeds would destroy any motor. If we desired to have a 20,000 rpm synchronous speed when operated at 10MHz, which is a common speed in small high speed DC motors then we could decrease the number phases to 2. This means that we could increase the number of pole pairs to 30,000 to achieve a 20,000 rpm synchronous speed. This isn’t feasible for a relatively small electric motor. That means that each pole would only occupy 0.012 degrees of the stator. It is possible to design what is known as a ‘high slip’ induction motor. However, the high slip versions of the induction motor are not as efficient or powerful as those meant to operate near synchronous speed [48].
5.2.2. AC Commutated Motor

DC commutated motors use a set of brushes that slide on the commutators. The commutators are arranged so that stator coil switches polarity at the right point in the rotation in order to continue to rotate. There is a special type of commutated motor called a series connected or universal motor that has the ability to run on any type of current (DC, any frequency AC, square, etc) supplied to it. This is because the rotor and field windings are in series so the polarity of the stator and rotor oscillates exactly in phase with each other. See Figure 4.21 for a diagram of an AC commutated motor.

The main advantage of the universal motor is that it can operate at any frequency AC current. This means that, in principle, the motor operates similarly at DC as it does at 10MHz. Universal motors are generally used in household appliances because they run on the 120V, 60 Hz residential power outlets. After investigation into series wound DC motors it was discovered that there is no commercially available motor meant to operate at anything higher than 60 Hz. In order to prove that the universal motor would work at frequencies much higher than 60 Hz a test motor was built from a small DC commutated motor. A small permanent magnet DC motor was used as a base motor. The motor was out of a track car so it was high speed and powerful for its size. The permanent magnets were removed and wound field coils were used instead. The same steel laminate transformer core that was used to make the cores for the Halbach array test was used to make cores for the universal motor. The original motor and the custom universal motor are shown in Figure 5.18.
Once the custom made series wound motor was built it was then needed to test the motor. A dynamometer could have been built using a second DC machine setup as a generator. The load on the generator could have been varied to provide an applied torque to the motor. Then the torque and speed could have been measure to determine the motor performance. This wasn’t necessary because the goal of the test was simply to determine if a commutated motor could operate at high frequencies. Based on qualitative observations the motor still worked well with a DC power supply. This verified that the motor was constructed and connected right. Once it was determined that the motor still operated well after its modifications it was needed to test the motor at variable frequencies. Then to test the AC performance of the motor a Titian AC power supply was used. It has variable voltage from 0 to 120 VAC and variable frequency from 20 Hz to 20 kHz. It was found that the universal motor worked well, very similar to the DC test, at 20 Hz. The frequency was turned up until the motor began to slow in speed. The voltage was increased to maintain the same speed. As the frequency of the motor increased the voltage also needed to be increased to maintain the same speed. This was continued until the frequency maxed out at 20 kHz. The voltage on the power supply had begun to reach its max also. Once the test
ended, the motor was felt to see if it had heated up. The field windings and cores were still close to room temperature. However, the rotor was extremely hot.

The temperature increase in the rotor suggests very high losses in the rotor core material. By observation it looked similar to the stator core that was taken from a commercial transformer. After investigation it was determined that the steel laminates that were used for the motor core weren’t insulated from each other. This was probably fine for the DC motor operation but once the frequency of the current was increased the eddy current force also increased. This could have led to very high currents in the rotor core and high magnetic losses that would have caused the rotor to heat up. If the motor were left running then it would have likely failed due to excess heat. By determining that motor operated at 20 kHz, and that the loss of performance was likely due to the rotor core material, it suggested that the universal motor concept could operate at any frequency if the right core material and the right design were chosen. This meant that a new motor design had to be created.

5.2.3. New Series Wound Carbon Motor Designs

After the universal motor tests it was discovered that the core material for a high frequency motor needed to be able to have very little loss at high frequency. The Magnetic Nano particle Composite (MNC) was an excellent option for the core material. In discussion with a representative from the electric motor design division of Parker Hannifin about this material he mentioned that the rapid switching of the MNC could allow a motor to have more poles. He said that the more poles a motor had the more torque it would have. The downside is that at higher speed operations the smaller pole have to switch faster and the hysteresis of the steel laminates didn’t allow the pole to switch faster enough. So the Carbon motor design should use the MNC and have the largest number of pole possible.

The downside of having more poles in a motor is that the coils must be smaller. The distance that the magnetic field of a coil projects out from its surface is in relation to its diameter [49]. The smaller
diameter of the coil means that the less the field projects from its surface. This means that the air gap between the rotor and the stator must also be smaller. In order to increase the distance that the field projects a Halbach coil arrangement, like in Figure 5.19, could be used. This would allow the flux to extend further than if a single coil was used. Then these coils would be arranged in a design that could be used in a series wound universal motor configuration.

Figure 5.19. New design for an electric motor pole. (a) Magnetic Polymer Nano composite core. (b) Shows one core with the CNT thread wrapped round it. (c) An assembled electromagnetic Halbach motor pole. (d) The white arrows show the direction of the current and the red arrows show the direction of magnetic flux at one instance. (e) shows the magnetic interaction between two Halbach motor poles. [58] Schulz M. J., Li W., Ruff B., Mast D., and Shanov V. N., 2011, “UC Invention Disclosure No. 112-016 Carbon Electromagnetic Materials to Replace Copper, Iron and Rare Earth Metals in Electric Motors by,” Carbon, (112), pp. 1–30.
Building on all the experience gained from the electric motor investigation and testing a new motor design was conceived. A CAD model is pictured in figure 5.20 and an exploded view of the components in figure 5.21. To this date no motor design has been discovered that is similar to the design proposed. The main feature is the use of electromagnetic Halbach arrays for both the rotor and stator. They are arranged in a radial flux configuration. This means that the flux between the rotor and stator travel in the direction of the radius and perpendicular to the rotation axis. It is depicted with CNT based rails that travel around either end of the rotor and stator. These are envisioned to act both as structure and as power rails to deliver current to each stator. Just as the polarity of the rotor is commutated the polarity of the rotor or the stator could be switched at specific points in the rotation. Instead of brushes, this motor would use rotatory transformers to transfer power to the rotor. Then the commutation or switching would be done using solid state electronics like in a brushless DC motor.

![CAD Model of the new AC commutated Halbach array Carbon motor design.](image)

**Figure 5.20.** CAD Model of the new AC commutated Halbach array Carbon motor design. [58] Schulz M. J., Li W., Ruff B., Mast D., and Shanov V. N., 2011, “UC Invention Disclosure No. 112-016 Carbon Electromagnetic Materials to Replace Copper, Iron and Rare Earth Metals in Electric Motors by,” Carbon, (112), pp. 1–30.

There wasn’t enough progress made in the synthesis, processing and characterization of the materials need to build the novel carbon motor design. Although many of the concepts were explored in regards to the operating principles they were never combined into a single machine. So it is possible that the design wouldn’t work as predicted. It is possible in the near future to build a motor that follows the design presented above. However, the performance of the motor is based on many assumptions in material properties that may or may not be true. The reduction in resistivity of the CNT thread is critical for operating an efficient and powerful motor. Most motors are limited in operation by the buildup of heat. So having high resistivity conductors will drastically increase the heat produced and limit performance. Also the processing of CNT thread needs to be streamlined and reliable in order to produce consistent and specific diameter thread. In order to design a motor, the resistance of the winding must be adjustable. This is done by varying the diameter of the conductors used. The magnetic
properties of the magnetic nano composites must also be increased in order to provide the high flux densities need in electric motors.
6. Conclusion and Future/On Going Work

6.1. Further Charged Substrate Investigation

The substrate charging experiments that were performed above are good indicators that substrate charging has an effect on the catalyst particle formation. However, there is still a decent amount of effort required to determine the relationship between voltage and particle formation. There are several factors that are not explored in the later charging experiments. One is the amount of time that the substrate spends at growth temperature. The substrates that were used in the SEM photographs were run with a slightly different recipe than the substrates that were used in the larger study. They were left at the growth temperature of 750°C for an extra minute. The extra minute was removed in order to try to capture the catalyst at the exact moment the CNT start growth. This extra minute may have allowed the catalyst to disperse more or reduce slightly more. This would account for the difference in apparent catalyst formation between the initial experiments and the larger one.

The really important discovery would be if substrate charging has an end effect on the produced CNT arrays. In order to determine this effect a larger study would need to be performed with the more accurate larger furnace that accounted for all the variations associated with CNT growth. Similar recipes grown with different voltage could be measured for height to see if the voltage affects the growth rate. Transmission Electron Microscopic images would be need to determine the average diameter of the CNTs. The SEM could be used to determine the straightness and tube spacing/density of the arrays which could also be affected by the substrate charging.

6.2. Electric Field CNT Synthesis

The phenomenon that CNTs align with an electric field is well established. The affect that an electric field can have on CNT growth has also been established through the use of PECVD. However, the direct application of static electric field on the growth of a CNT array has not been explored to date. This is
likely due to the difficulty in creating a static electric field in the conditions of the reactor without affecting the growth. The investigations into the best ways to construct a charging base provide a invaluable insight into how such an electric field growth fixture could be constructed. Using the gold and chrome deposition on to quartz could provide the needed fixture construction to investigate CNT electric field growth.

6.3. Sheet Spinning

It is believed that the sheet spinning alternative to CNT thread production has many benefits. The biggest advantage with sheet spinning is an increased in production control. The regular dry spinning method has very few controllable parameters. The width of the array is decided before the synthesis. The pull and twist rates are adjustable without feedback. These are the only controllable parameters with traditional dry spinning. In a sheet spinning machines a sheet could be made in a continuous process with adjustable thicknesses based on the number of layers. Then the sheet could be slit into different widths. Then the different widths could be pulled and twisted into fibers. By layering the ribbons into a sheet the diameter of the as made treads could be better controlled. Dry spinning produces a non-uniform diameter fiber, likely because the ribbon has a high degree of randomization in it. This doesn’t allow the ribbon to collapse into a fiber at the same rate along the length. A sheet spinning method could allow the resulting fiber to be more uniform and have higher properties.

Another advantage of sheet spinning is the ability to process the CNTs while in a stable sheet form. The sheet is surprisingly robust while it is attached to the Teflon backing. It also offers a much higher surface area than a spun fiber does. The fibers could be too dense for dopants or functionalizations to reach the inside of the thread uniformly. By introducing the processing while the CNTs are in sheet for you allow much greater and evenly distributed access to the tubes.
6.4. Densification Testing Machine

The densification under tension, as it is investigated above, is an area of CNT research that hasn’t been found in literature to this date. Arguably, the most important step in making cotton threads is the last step before spinning. This is where the cotton web is stretched many times its original length to orient and straighten the cotton fibers [59]. This is so that the density of the fiber is increased to its max and the contact between fibers is maximized. This can be done easily in cotton because the cotton fibers don’t have an intrinsic attraction between them, so they easily slide. CNTs, in contrast, have Van Der Waals forces that make them stick together. It is also very difficult to untangle the CNTs in the bundles. This makes it not possible to stretch the CNTs like in cotton processing. However, it may be possible to temporally negate the VDW by partially dissolving the CNTs in an organic solvent. This would allow the CNT thread to be stretched and align the CNTs in the fiber.

Based on the data presented here I think that it calls for a machine that can perform densification is a much more controlled way. If a small tensile tester was tuned on its back so that the sample was horizontal then the sample could be lowered into a bath as done in the densification experiments. The tensile tester could be used to stretch the thread while it is being soaked. This would provide important information about how the solvent was affecting the thread. Once the thread was stretched in the bath, the solvent could be removed and the thread left to dry. The axial stress in the thread could be measure as the thread dried. This would provide even more information about how the thread structure was changing. A diagram is shown is figure 6.1. The data gained from this experiment could be directly used to produce a continuous spinning/densification machine.
Figure 6.1. A diagram of a proposed densification test bench. The first picture is with the solvent bath lowered. The second picture the solvent bath is raised to soak the sample.

6.5. Build Carbon Motor

It is needed to build a version of the motor designed in Figure 5.20. This is critical for generating funding for further exploration of the CNT and MNC based electric machines. These machines could have higher efficiencies than iron and copper based devices. They could also have potential to achieve higher power to weight ratios than traditional materials. Basic power electronics (solenoids, transformers, etc) could first be built to prove the function of the materials. This is also an opportunity to practice many of the details that will be critical to building and operating these devices. The electrical interfaces, winding procedures, etc will likely be a challenge in building these machines.

If a process is developed that produces materials with the desired properties than there will be potential for these materials to be used for a variety of motor and electronic designs. The development of these materials should be made as the top priority because if the material properties are not improved then the applications will be very limited or nonexistent. If the desired material properties are realized then there is huge commercial potential for the materials in nearly unlimited applications.
6.6. Flexible Magnetic sensor actuators

The rigid MNC that will be for use to replace iron cores in devices are one type of magnetic nano composite. Another type of magnetic composite uses a flexible elastomer that yields a flexible magnetic material. This is more novel than a rigid iron core replacement because there isn't a flexible paramagnetic material that is available today. There are flexible ferrite permanent magnets that are common refrigerator magnets but they have limited uses. The flexible magnetic material could have many uses in sensor, actuator, and combination sensor actuators. An unsuccessful example of a flexible magnetic actuator is shown in figure 6.2. This design is based on the principle shown in Figure 4.3. Wires with current running the same direction attract and if the current is opposing then the wires repeal each other. It was thought that if the wires were placed in a flexible magnetic material then the forces between the wires would increase and then they would actuate. It is believed that the wires need to be closer together for the force to be strong enough to produce significant actuation. Using the Freeman elastomer described in Section 5.1.1 allowed for easy fabrication of the device so further investigations could be done easily.

Figure 6.2. The mold used to make and a picture of the unsuccessful prototype of a flexible magnetic actuator.
6.7. Sensorcyte and other Biomedical Devices

One application of the flexible magnetic material is devices that could be placed inside the body. One could be an artificial device that is the size of a red blood cell and could be placed inside the circulator system. The flexible magnetic material would be useful because red blood cells must fold in order to make it through some of the small veins in the body. So a rigid device wouldn’t be able to move about the body as easily.

Another possible use for a flexible magnetic material would be as a magnetic pipe to transport energy into the body without the possibility of shocking the patient. This could also be useful for sensors in environments where there is danger of ignition or other chain reaction. The magnetic flux can be used to generate voltage in a coil of wire if the flux passes through it. It would have no effect on materials that were non-magnetic or very weakly magnetic. However, it could generate current in a conductor. The flux density outside of the magnetic material would be low and the resulting current induced in a nearby conductor would also be low.

6.8. Conclusion

In conclusion, many of the materials investigated in above show potential in commercial electric motor applications. If the materials are look at as an alternative to the traditional materials then they could have a place in the short term for being used where weight saving is very important. There is still a lot of work to be done to improve the properties to a point where they have an advantage over traditional materials. The work here is a wide variety of investigations into which factors may have the biggest impact on the properties that are important for this application.

The sheet twisting in line with a densification process could be in the answer to commercialization to the dry drawing process. It also may just be a fluke in the data that makes it seem like it has potential. After the entirety of the work done for the completion of this thesis, it is recommended that this is the
best short to medium term path to producing CNT fibers with the desired properties. At least there should be further investigation and equipment made towards this end. The weakness of the entire dry drawing process is the synthesis of spinnable arrays. It may be desirable to construct a custom furnace for the specific and dedicated growth of spinnable CNT arrays. The furnace should have a detailed and simple control of the parameters and also be constructed in a manner that allows a high throughput of arrays. This furnace may also include the electro static charging and fields that were investigated here.

The magnetic nano composite materials that were fabricated above were interesting and useful for laboratory experiments. They still need extensive testing and characterization to determine the properties. There needs to be additional factors introduced into the fabrication process that can increase the magnetic properties. Also the use of the correct particles will be critical. It is believed that these technical challenges are possible to overcome with extensive and intelligent experimentation.

7. References


