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Cast Forming of Carbon Nanotube Networks Using Paraffin

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Abstract

Carbon nanotube thin film, or buckypaper, is one of the most revolutionary materials in the 21st century. Mechanical, electrical, and thermodynamic properties that can only be dreamed of in science fiction novels are now within reach in the science and technology field. As amazing as this material is, there exist problems within the manufacturability of buckypaper. Problems such as process time, scalability, and cost effectiveness to produce a sample hinder the ability to produce buckypaper to the commercial market.

This research effort is to study, through experimentation, a new approach to create buckypaper using cast formation of a carbon nanotube network while in a paraffin suspension. Because current nanotube dispersion and filtration methods, such as sonication can produce high costs and slow processing times, the need for new buckypaper manufacturing method is evident. During this experiment, buckypaper was created using two methods for dispersion, the first was a mechanic mixing method and the second was the traditional method of sonication.

The study proves that the use of paraffin as the dispersion and flow medium does not provide ideal results to eliminate steps such as sonication and filtration. The resultant buckypaper through mixing did not yield good results due to the nature of carbon nanotube's tendency to agglomerate while heat is applied during the dispersion process. Poor dispersion leads to a decrease in functional properties such as mechanical, electrical or thermodynamic. It is conclusive that further investigation into this method is necessary.

THE FLORIDA STATE UNIVERSITY

College of Engineering

CAST FORMING OF CARBON NANOTUBE NETWORKS USING PARAFFIN

By

KENNETH BLAKE VELIKY

A Thesis submitted to the

Department of Industrial and Manufacturing Engineering

in partial fulfillment of the requirements for graduation with

Honors in the Major

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The members of the Defense Committee approve the thesis of Kenneth Veliky defended on April 18th, 2014.

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1.0 Introduction

Carbon nanotubes (CNTs) are cylindrical molecular structures with a diameter in the nanometer scale and length ranging from 1 micron to a few millimeters.[1] Carbon nanotubes can be distinguished by two types: single-walled carbon nanotubes (SWCNT) or multi-walled carbon nanotubes (MWCNT). SWCNTs consist of a single graphene layer rolled up into a cylinder, whereas MWCNTs consist of two or more concentric cylindrical shells of graphene sheets arranged around a central hollow core held together by van der Waals forces between adjacent layers.[2] Figure 1 shows typical structures of SWCNTs and MWCNT. CNT networks are formed by adjacent nanotubes bundling together through van der Waals forces. The CNT network is also called buckypaper as shown in Figure 2. Buckypaper is valued for its potentially high mechanical strength and high electrical and thermal conductivity as well as easy to handle and use.[4] The theoretical values for SWCNT tensile strength is ~200 GPa and modulus is about~1400 GPa whereas MWNT strength is 11-63 GPa and modulus is about 70-100 GPa.[5,6] An example of buckypaper potential applications is lightweight aerospace composites structures due to CNT extraordinary properties. Buckypaper can be applied as a layer on the skin of an aircraft for electromagnetic interference (EMI) shielding and lightning strike protection (LSP).[4,7]
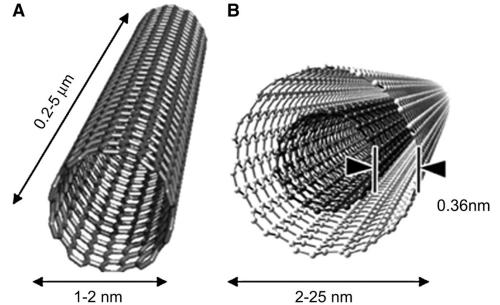
Figure 1: Conceptual image of a SWCNT (A) is displayed in reference to a MWCNT (B) [3]
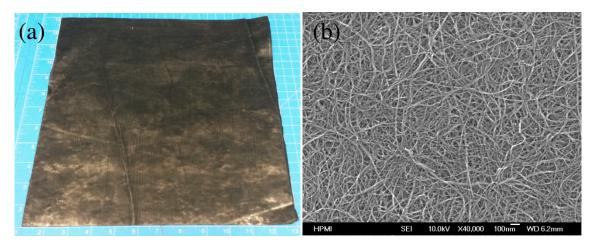
Figure 2: A sample of MWCNT buckypaper from the High-Performance Materials Institute (a)

and a scanning electron microscope (SEM) image of the sample showing the dense CNT network

(b)

1.1 Carbon Nanotube Buckypaper

Carbon nanotube networks or thin films, also called buckypaper, are used in a wide range

of applications from composite development to electrical property optimization. Buckypaper is

generated from a method that begins with dispersion and leads to filtration of the dispersed CNT

suspension. The nanotubes are dispersed in a suspension then filtered through a porous membrane, where the nanotubes are held together using van der Waals forces which creates a thin film called buckypaper.[4] Buckypaper hosts advantages such as it being highly porous, self-supporting, flexible, and thermally and electrically conductive.[8]

1.2 Suspension Selection

The selection of proper suspension is important when attempting to achieve a high level of CNT dispersion. High levels of dispersion allow for the separation of individual CNTs which increases the density of the CNT network. The higher of the density in a CNT network will lead to an increase in high performance thermal, electrical, and mechanical properties due to better tube-to-tube contact. Carbon nanotubes yield problems during dispersion due to their morphology. Their chemical makeup allows them to attract to one another, thus creating bundles. Additionally, their high aspect ratios create more surface area to attract and create bundles once dispersed.[9] Suspension material is used to create a matrix for the dispersion of carbon nanotubes. The suspension's goal must be to inhibit dispersion as well as separate and maintain separation of individual nanotubes.

2.0 Problem Statement and Research Objectives

2.1 Problem Statement

Currently there are various methods to create buckypaper including filtration, die cast batch sampling, and continuous floating catalyst. These methods host many benefits, but also pose disadvantages such as small sample size, high cost and long production time that hinder potential commercialization applications. For example, the filtration process of buckypaper

fabrication is typically several hours, which is considered a very slow process for industrially scaled parts.

The current High-Performance Materials Institute (HPMI) method to manufacture buckypaper is through CNT suspension and filtration. Filtration creates many disadvantages to the overall process costs. Filtration materials include chemicals needed for dispersion, disposable filter membranes, and the filtration apparatus and sonicator. Figure 3 demonstrates the flow chart of current buckypaper filtration process.   Together, these materials and equipment create issues for production efficiency and costs. New methods for fast and low-cost production of buckypaper materials are highly desired.
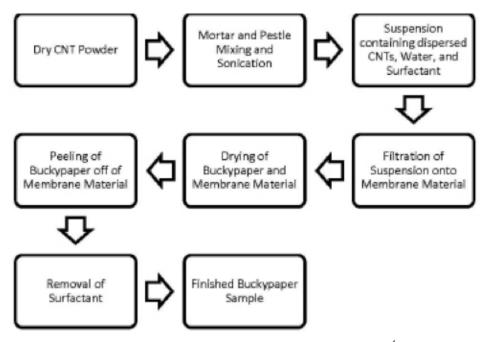

Figure 3: Buckypaper filtration process flow chart [4]

2.2 Research Objectives

The objective of this study is to setup and test a prototype for scalable buckypaper production by suspending CNTs in wax and using a simple die casting approach. This new process will potentially optimize the manufacturing process by eliminating high cost materials

and sonication processes. The research will study the processing variables, including apparatus temperature, sample time spent under heat and pressure, proper amount of material, required force, and optimal heating element. The quality of the resultant buckypaper will be tested and analyzed, including electrical conductivity, dispersion evaluation, permeability, mechanical testing of tensile strength and Young's modulus, and thermal conductivity. The detail goals include:

- Develop the Die Cast Method

- Produce Buckypaper Samples Formed Using Die Cast Method

- Quick Batch Fabrication for High Throughput

- Process Without Filtration and Sonication

3.0 Literature Review

There are different methods for manufacturing buckypaper, such as die cast halogen and evaporation[10], filtration of CNTs in suspension[4], and spray method[11]. The two most relevant methods are discussed below.

3.1 Die Cast Halogen Evaporation

This method of die-casting begins by dispersing the carbon nanotubes into a liquid halogen, and substantially removing the liquid halogen, whereby the cohesive assembly of CNT networks is formed, as shown in Figure 4 .[10] This method relies on the evaporation of halogen. The material is cost efficient, but production time is very long because the user must remain idle for the halogen to evaporate off. The material produced creates a density level of 0.5 – 1.0 g/cm^3.[10] and has a very poor surface quality as shown in Figure 4.

Figure 4: Sample from die casting by halogen evaporatio.[10]

3.2 HPMI Filtration Method

The filtration method is a batch production style method that uses CNTs that have been sonicated in a solvent to achieve even dispersion, injects this pressurized CNT/surfactant/solvent solution through a nylon membrane for filtration. The suspension is filtered through the membrane and the residual carbon nanotube forms a buckypaper sample. This method is optimal in creating thin sheets with high electrical properties due to good dispersion of CNTs and filtration to densely pack the CNTs. In addition to the high cost and long processing time, another problem that occurs is during the separation of buckypaper and membrane peeling phase. Since the buckypaper sample is very thin (5-10 microns) and does not simply detach from the nylon membrane, a series of manual removal steps must occur. Figure 5 shows a fractured buckypaper from the peeling process. The operator must perform a manual process of peeling the buckypaper sample from the membrane. It is not reasonable for a commercially available product to require such operator experience and skill for each unit. This process is tedious, delicate, can introduce defects due to user error, and slows down the entire production process.[4]

Figure 5: A sheet of buckypaper that was fractured while separating from the filtration membrane

4.0 Technical Approach

4.1 Parameter Selection

The objective of this research is to study a new method of CNT dispersion and formation of CNT network using a flow medium in order to more efficiently create buckypaper. The parameter selection is an important part of this research because it is the initial stage of experimentation. To create a buckypaper sample, it is necessary to start at CNT dispersion. The parameters are broken down into two types based on the method of dispersion. The two techniques researched are dispersing dry carbon nanotubes in paraffin wax through mixing or sonication. Prior to this procedure, it is important to understand that the boiling point of paraffin is achieved at 370°C. The following discussion is to identify the parameters selected in order to perform the dispersion techniques.

4.1.1 Mechanical Mixing Dispersion

The mechanical mixing method begins with measuring out 200 mL of paraffin wax into a 600 mL beaker. The 600 mL beaker is placed on a hot plate set at 300°C, in order to melt the solid paraffin wax into a viscous mixture with a lower viscosity. Once the wax is completely melted and has reached 200 mL, the melted wax container is removed to a designated nanotube measuring station. Dry carbon nanotubes must be measured and placed directly into the melted paraffin and covered. This experiment used 600 mg of vertically aligned multi-walled carbon nanotubes roughly 1-2 mm in length. Once the CNTs are in the wax suspension, a medium sized magnetic stir bar is placed within the beaker. The beaker is placed over a hot plate and the magnetic stirring is applied while covered for 20 minutes at 600 RPMs and 250°C. At the end of 20 minutes, the mixture becomes very viscous and the stir bar will struggle to rotate about the hot plate; this is mixing maximum and buckypaper sample preparation stage follows.

4.1.2 Sonication Dispersion

The sonication dispersion method begins similarly to that of the mixing type dispersion method. Paraffin wax is melted over a hot plate at 300°C in a 600 mL beaker until 200 mL of melted paraffin is reached. The melted paraffin wax is removed from the hot plate and placed in a designated nanotube measuring station where 600 mg of vertically aligned multiwall carbon nanotubes, with 1-2 mm in length, are poured into the viscous solution. The newly mixed solution is covered and transported back to the hot plate and mixing station.

Before applying to the sonicator, the nanotube agglomerates must be broken down into smaller pieces. Using the hot plate and medium stir, the covered nanotube and paraffin mixture is stirred at 250°C and 600 RPMs for 10 minutes. Again, this step is to break down any large bundles that may slow down the sonication process. Once the 10-minute stir is complete, the

mixture can be transferred to the sonication stage. The paraffin and CNT solution is sonicated

using a 3/4" Qsonica Solid Tip Sonicator Model 334 while being supported by a hot plate set to

100°C. The sonication process is applied in two phases:

1. 75% Amplitude for 30 minutes with hot plate

2. 50% Amplitude for 15 minutes without hot plate

The change in amplitude, time and heat is due to the nature of sonication. Sonication uses

sound vibrations at a high frequency. This, in turn, produces heat within the mixture. It is crucial

to not allow the mixture to get above 370°C due to the nature of paraffin vaporizing. This would

change the amount of paraffin to CNTs, thus producing a different wax to CNT ratio than that of

the mixed sample. Overall that would lead to variability in a comparative analysis, which is

undesirable. Once the sonication process is complete, it is transported to the sample preparation

phase.

4.2 Buckypaper Sample Preparation

Both the mixed dispersion and sonicated dispersion methods undergo the same sample

preparation. Polyamide is used as the mold to pour the hot paraffin and CNT solution. The

polyamide mold is within a lab tray to avoid any runoff that may occur during the pouring stage.

Figure 6 provides a top view of the mass prior to molding to shape. At this point, the wax is still

a viscous stage and ranges from 280°C to 330°C, so it must cool before being able to move to the

next stage.

Figure 5: CNT and wax sonicated mixture prior to molding

At 100 °C, the wax viscosity is lowered, allowing for the user to manually press the dispersed nanotube and paraffin solution. Between 80 - 60 °C, the paraffin is at its optimal point to retain shape. One at a time, the samples are kneaded and rolled flat until they create a user specified shape. Below in Figures 6A and 6B, the two different dispersion techniques are compared visually. In Figure 61, it can be seen that the CNT network did not completely infiltrate the paraffin suspension. This can be identified through the light patches as located in the Figure. Figure 6B hosts a sonicated sample where there are no wax pouches and the CNT network is dispersed through the paraffin suspension.
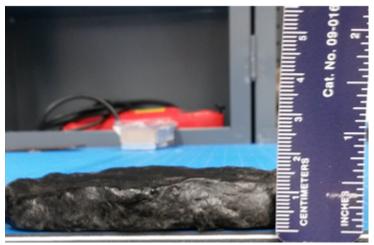
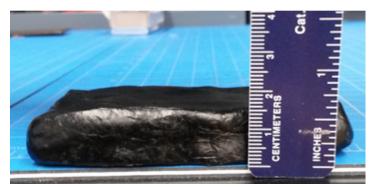*Figure 6A: Post molding of mixed dispersion*

Figure 6B: Post molding of sonicated dispersion

4.3 Wax Removal

When removing such a long molecular chain like that of Paraffin, it is optimal to process the sample through a reduction phase. Once reduced, then the sample must undergo further processing eliminate the remaining paraffin in a refinement phase. The reduction phase used in this study is vacuum bagging. This process reduces excess wax content to ensure a shorter refinement phase. The vacuum bagging process also reduces the width of the sample. The vacuum oven process is referred to as a refinement phase because of its ability to eliminate the remaining wax from the CNT network. This stage provides high temperature without oxygen, creating the desired environment for removing paraffin. The next stage explored in detail below

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is the process to reduce the wax content using vacuum bagging   with a vacuum oven to remove the wax.

4.3.1 Vacuum Bagging

The vacuum bagging process begins with the initial stages of setup. The typical vacuum bag process is used to infuse resin in   composite fabrication. For this application of vacuum bagging, we are solely focused on the application of pressure and heat to remove the excess paraffin. Figure 7 displays a sample prior to entering the vacuum bagging process. This process begins with the base plate. Metal plates are ideal due to their thermal conductivity. If the base plate contains any contaminants, it must be cleaned immediately. Contaminated surfaces can lead to a poor interface with the tape and base, ultimately creating leaks in the vacuum system.
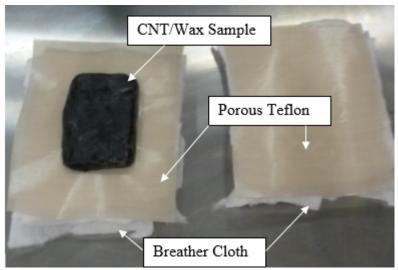
Figure 7: Inside the vacuum bag

Both the mixed and sonicated sample will undergo identical vacuum bagging procedures. The CNT and Wax mixture lies on top of porous Teflon sheet, as shown in Figure 8, which is placed on top of 5 layers of breather cloth. The sample is then covered directly with another porous Teflon sheet and 5 layers of additional breather cloth are placed on top of that Teflon. The CNT and wax sample will only interact with the Teflon. The Teflon acts as a membrane

between the sample and the breather cloth. Since the breather cloth is being used as a sponge to absorb the fluids, it will inadvertently soak up nanotubes if direct contact occurs. Once the parts are laid-up, the sample area is traced with two layers of double sided tape in order to hold the bag to the metal workstation. On one side of the bag, a gate must be placed on top of the tape in order to allow air to be removed from the bag. The sample is then covered with the bag and held down by tape. Figure 8 displays the cross sectional layup of the vacuum bagging process.
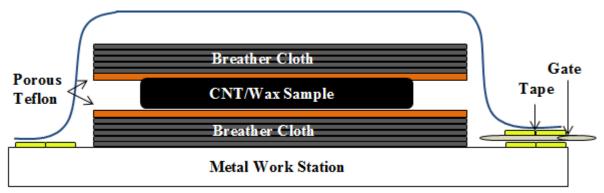

Figure 8: Cross section view of the vacuum bagging process

The prepared sample is then placed within a heating chamber in order to provide a constant temperature during the process. The humidity chamber set to 100°C was chosen to create the heat source necessary to complete the project. The sample was placed inside the pre-heated chamber, the vacuum is applied immediately. The application of immediate removal of air is ideal in order to provide a constant pressure to create self-sustaining form while the paraffin is melted down. The sample is placed under a constant atmospheric pressure for 1 hour and then removed from the heat. Figure 9 visually represents the buckypaper sample during the heating process. It can be seen that the wax is used as a flow media to spread the CNT network while the pressure creates form and constrains the CNTs within an area relative to the sample's original size.
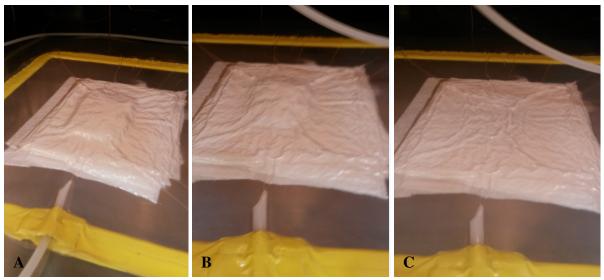
Figure 9: Sample under heat and pressure at A) 15 minutes B) 30 minutes C) 45 minutes

Once the sample has reached 1 hour of heated pressure, it is removed from the oven. All components from the vacuum bagging process are scrapped, while the workstation is cleaned for reuse. The sample is now reduced from the bulk wax and is removed from the porous Teflon layer. Figure 10 is an example of an ideal sample at this point in the process. The ideal sample is reduced thickness by 1/20th the original thickness. The sample in Figure 10 is a sonicated sample with original thickness of 15 mm which has been reduced to 0.5 mm, thus achieving thinner results being labeled an optimal sample.

Figure 10: Sonicated sample after the vacuum bagging process

4.3.2 Vacuum Oven Evaporation

Once the vacuum bagging process is complete, the sample is cut into smaller rectangular samples of 25 mm x 13 mm. The smaller samples are placed on a glass petri dish with a layer of porous Teflon separating the sample from the glass. An example of this set up can be located in Figure 11. The samples are then placed into a vacuum oven at 280°C for 4 hours. Similarly to the vacuum bag process, the air is removed from the chamber and the sample is exposed to atmospheric pressure. The sample is burned under a vacuum for the safety of the user due to the flash point of paraffin wax vapor.
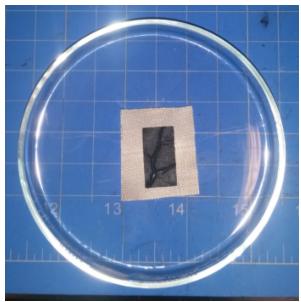
Figure 11: Sonication sample prior to entering the vacuum oven

Once the sample has undergone the vacuum oven process, it is removed from the porous Teflon and tracked for the use of testing. In Figure 12, a comparison of a sonicated sample before and after the vacuum oven process has undergone. As shown in exhibit A of Figure 12, there is a shine to the sample, but compared to exhibit B, the sample is lacking shine. This is due to the removal of layers of paraffin wax. The wrinkles also remain in the sample after the treatment. These wrinkles in the sample are created while using the vacuum bagging process. Since the sample with components are thick prior to entering the vacuum bag chamber, the excess bag wrinkles while flat.
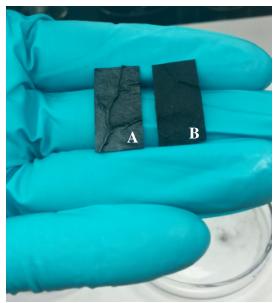
Figure 12: A sonicated sample (A) prior to the vacuum oven is compared to another sample (B) after the vacuum oven process

5.0 Characterization Setups

Morphology of the CNT/Paraffin thin films were viewed using a field emission scanning electron microscope (JSM-7401F, JEOL Co.). Images were taken at 10 kV and a working distance of 7 mm.

TGA measurements were performed using a TA instruments Q50 thermogravimetric analyzer. The sample was heated to 1000°C with a 10°C/min ramp in an Oxygen atmosphere. The mass of the sample is recorded over time as chemical species evaporate or sublimate.

Mechanical property tests were conducted using the DMA Q800 machine (TA Instruments Inc.) using the film mode with a constant frequency of 1 Hz at room temperature. Specimen gauge length for testing was controlled at approximately 10 mm. Tensile properties were estimated from the stress-strain curves with preloaded force of 0.01 N and force ramp of 1 N/min.

The electrical conductivity was measured using the four-probe method. A 4-probe resistivity tester and a nano-voltmeter were used to measure voltage changes and resistivity, while a DC/AC current source was used to supply current to the samples.

6.0 Problems Encountered

While performing this research in the experiment stage, several obstacles were uncovered. Due to the properties of paraffin, the amount of time and energy to remove the molecule lead to challenges and opportunities. Below is a few of the obstacles that were encountered throughout this experiment:

1. <u>Flash Point</u> – Paraffin vaporizes at 370 °C, its boiling point. Not only are the fumes noxious, but also when in the presence of high heat and dense paraffin vapor, these fumes become combustible. Early experimentation using open flame had proven this property and is visually represented in Figure 13. Performing tests upon a hot plate created a safe alternative to open flame testing.

Figure 13: Paraffin reaching its flash point

2. <u>Minimizing Wax Content</u>– Throughout this project, the objective of minimizing the total wax content at the beginning of CNT/wax mixing lead to the discovery of both the positive aspects and negative of residual wax content. In positive effects, low wax content led to greater properties of both mechanical and electrical aspects. Low content also produced shorter sample creation times. Yet in a negative effect, if the wax content was too low, then the CNT network would fail to spread evenly throughout and the resultant sample was thick and poorly dispersed. Figure 14 provides an example of a mixed sample that was reduced in wax content prior to the vacuum bagging process. The wax acts as a flow media and provides beneficial support needed to create a thin buckypaper sample. Figure 14 supports evidence that the paraffin wax acts a flow media. It can be seen that pressure was applied, but the CNT network was unable to move

beyond the extents of its form. This problem occurred when filtering the sample prior to vacuum bagging was set in place, in order to cut down on post processing time. Removing the filter stage, when molding the sample, will provide the proper amount of paraffin necessary for the CNT network to expand within the vacuum bag.

Figure 14: Post vacuum bag process with low wax content

7.0 Results and Discussion

7.1 Scanning Electron Microscope (SEM) Analysis

In order to understand the resultant buckypaper that was produced, the samples must be viewed using scanning electron microscope (SEM) to identify the microstructure. The SEM allows the user to see up to 150,000 times the normal observation. The following samples were gold coated to enhance surface conductivity to produce a clearer picture. Figure 15 identifies a mixed dispersion sample at the surface of x200 magnification. It can be observed at the top layer of the buckypaper still has a layer of paraffin wax that did not be removed during the vacuum

oven process. The surface is extremely uneven, which can happen due to failure to disperse the CNT network which leads to agglomerations during the vacuum bagging phase.
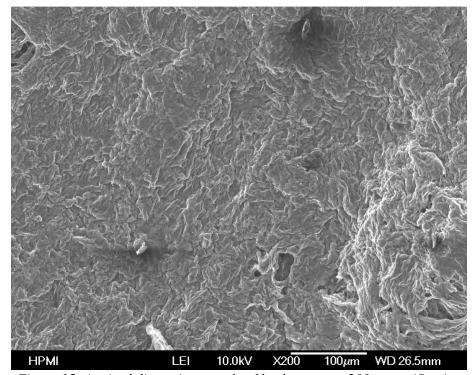

Figure 15: A mixed dispersion sample of buckypaper at 200x magnification

Zooming into a section of this sample, at x3700 magnification, it can be seen that the wax holds bundles of nanotubes together. Figure 16 demonstrates the ability wax has to hold bundles if not properly dispersed. The large bundle located in the center of the image in Figure 16 is roughly 4.5 µm (4500 nm) wide. This is not optimal due to the objective of thoroughly dispersing a CNT network to inhibit mechanical, electrical, and thermodynamic properties.
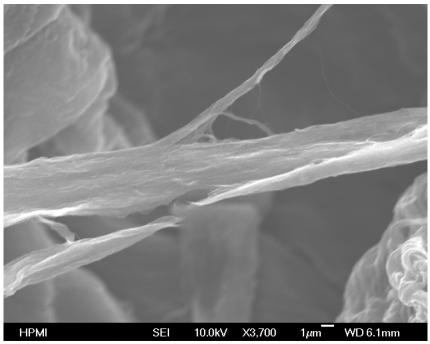
Figure 15: A mixed dispersion sample of buckypaper at 3700x magnification

An example of a sonicated dispersion method sample is shown in Figure 16. The sample's surface is identified in this example. Similarly to the mixed sample, the sonicated sample did not completely remove off the top layer of paraffin wax. The surface is also uneven, but due to the Teflon weave, which can be identified by its rectangular pattern.
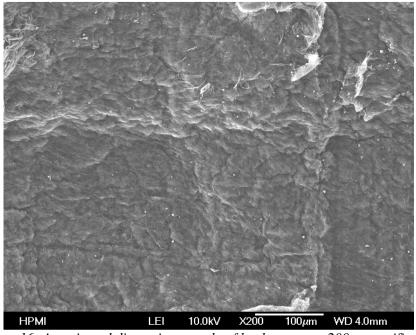

Figure 16: A sonicated dispersion sample of buckypaper at 200x magnification

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Upon further observation, the sonicated dispersion sample provides evidence of a higher level of dispersion than that of the mixed dispersion sample. Figure 17 provides a sonicated sample that shows dispersed bundles of nanotubes. Although bundles of nanotubes remain, when compared to Figure 15, it can be seen that more wax has been removed from the top layer due to the visibility of the nanotube bundles. This reduction in wax is due to the higher degree of sonication allowing for fewer areas of bulk wax, making it easier to remove the paraffin away.

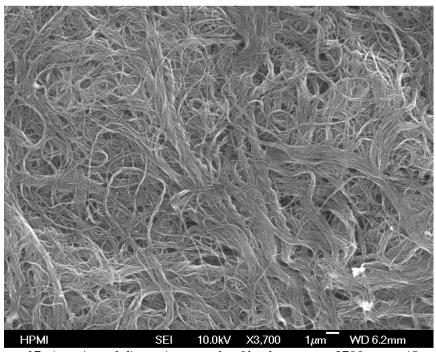*Figure 17: A sonicated dispersion sample of buckypaper at 3700x magnification*

In conclusion to studying the levels of dispersion through scanning electron microscopy, the level of dispersion is higher in the sonicated dispersion method than that of the mixed dispersion method. Good dispersion can lead to higher multifunctional properties; therefore, it is desired to achieve a high level of dispersion when producing a buckypaper.

7.2 Thermogravimetric Analyzer (TGA) Analysis

Thermogravimetric analyzer (TGA) allows the user to identify the mass percentage of weight in a sample through burning. Samples of both mixed and sonicated dispersion where burned and quantified using TGA outputs as shown in Figures 18 and 19Each figure hosts two plots with respect to time, the percentage weight in green and the derivative of percentage weight in blue. The derivative graph allows the user to identify individual instances of mass reduction by heat necessary to burn off.

A sample of mixed dispersion can be identified in Figure 18. The figure hosts three areas of local maximums on the derivative graph. Reading from left to right on the graph, these three areas have the peaks at 363.78°C, 539.88°C, and 738.22°C. Knowing that paraffin has a boiling point of 360°C, it can be assumed that the first peak is the bulk paraffin burning away. The bulk is the primary top layer of the paraffin on the CNT network. When looking at the end of this cycle, it can be seen that the bulk mass of paraffin in the mixed sample is a little less than 12% of the buckypaper's total mass. The second local maximum occurs at 529.88°C, knowing this is too low to be CNTs and a few hundred degrees higher than the previous maximum; it can be identified as the residual paraffin between the nanotubes. This requires a greater heat to release from the CNT network. The residual paraffin accounts for 24% of the buckypaper's weight. The final and largest local maximum occurs at 738.22°C, this is the CNTs that remain after all paraffin is burned away. In this mixed sample, the CNT network accounts for 62% of the buckypaper's total weight. At 789.04°C, the percent weight increases. This is due to oxidation of the catalyst involved in producing the CNTs, creating rust.
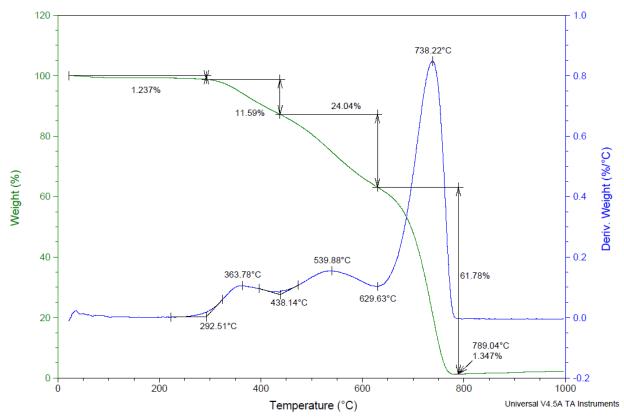
Figure 18: TGA results of a mixed dispersion sample

Similarly to the mixed sample, the sonicated sample, in Figure 19, reacts in the same manner. There are three local maximums, reading from left to right are at 365.60°C, 547.36°C, and 726.77°C, respectively. The first local max at 365.60°C is as well the bulk paraffin, which accounts for 10% of the total mass. The second local max occurring at 547.36°C is the paraffin between nanotubes. This portion of paraffin accounts for 18% of the buckypaper's total weight. The final and absolute maximum on the derivative graph occurs at 726.77°C, this is the CNT network. The CNT network in the sonicated sample accounts for 69% of its total mass. Similarly to the sample in Figure 18, the percentage weight increases after the CNTs burn off, this is the oxidation of the ferrous metal catalyst. This sonicated sample is preferred because total paraffin removal is the most optimal.
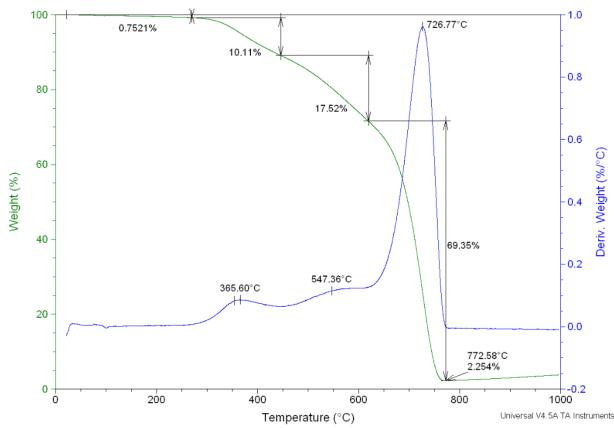
Figure19: TGA results of a sonicated dispersion sample

7.3 Dynamic Mechanical Analyzer (DMA) Analysis

DMA tests will reveal mechanical testing for small samples with a high load resolution. Of the mixed and sonicated samples, 5 of each type were tested. Due to outliers within the results that fractured at the sample tab, 3 samples were chosen to represent the mechanical properties of each dispersion method. Samples of both mixed and sonicated were cut to have a length of 12.7 mm and a width of 6.35 mm.

The first test was of the mixed dispersion method samples, located in Table 1. The table identifies each sample's Young's modulus, tensile strength, and strain-to-failure. The average of each of those properties among the sample population were found to be 5.54 MPa, 0.254 MPa, and 6.23% respectively. The standard deviation was found to be 4.78 MPa, 0.107 MPa, and 1.85% respectively. Analyzing the standard deviation of each sample's property generates a

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consistency test. With a standard deviation of 4.78 MPa, it can be hypothesized that there is little

consistency among the Young's modulus results. The least standard deviation of 0.107 occurs

with the tensile strength. Figure 20 compares the DMA results from each test that best represents

the sample data. The mixed sample requires low force to elongate, but trades off with a low

tensile strength. The sonicated sample generates a higher strength as well as a high elongation.
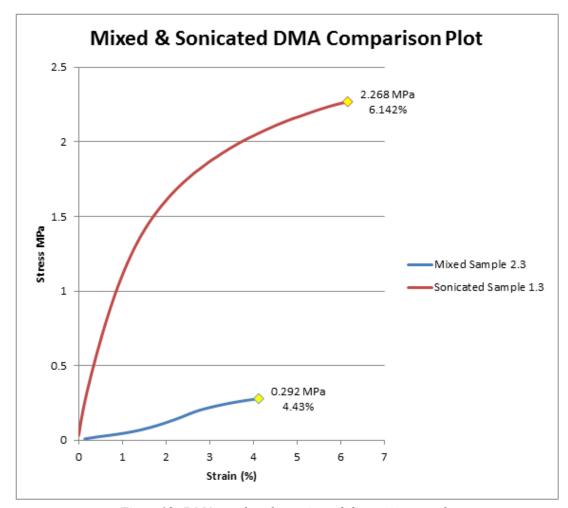

Figure19: DMA results of a sonicated dispersion sample

Table 1: DMA testing resultant buckypaper samples using mixing as dispersion

Name	Young's Modulus (MPa)	Tensile Strength (MPa)	Strain-to-Failure (%)
Mixed Sample 1.3	1.35	0.133	6.114
Mixed Sample 2.3	4.51	0.292	4.431
Mixed Sample 3.3	10.75	0.336	8.132
Standard Deviation	4.78	0.107	1.853

Equally, the sonicated samples were measured. . Each sample, like the mixed samples, was measured to quantify Young's modulus, tensile strength, and strain-to-failure, as shown in Table 2. The averages of each property was found to be 135.61 MPa, 1.754 MPa, and 4.924% respectively. The standard deviations of sample results were 7.506 MPa, 0.490 MPa, and 1.067% respectively. The largest standard deviation comes from the Young's modulus results and the lowest standard deviation occurring within the tensile strength.

Table 2: DMA testing resultant buckypaper samples using sonication as dispersion

Name	Young's Modulus (MPa)	Tensile Strength (MPa)	Strain-to-Failure (%)
Sonicated Sample 1.3	138.00	2.268	6.142
Sonicated Sample 2.3	141.63	1.702	4.472
Sonicated Sample 3.3	127.20	1.292	4.158
Standard Deviation	7.51	0.490	1.066

Analyzing both results, it can be inferred that the sonicated samples produce higher mechanical properties than the mixed match. The sonicated batch produces an average Young's modulus just below 10 times the amount of the mixed batch. This means that plastic deformation will occur at a much higher stress in the sonicated samples than that of the mixed. The tensile strength average is also greater in the sonicated samples. In a mixed sample, the highest tensile

strength was found to be 0.336 MPa. In a sonicated sample, the lowest sample was found to be 1.292 MPa. This as well shows the extent of stress required to create failure within the sample. Sonicated samples prove to generate the higher stress required to fracture a sample. Although the tensile strength has proven to be much higher in sonicated samples, the strain-to-failure in both was relatively equal. The sonicated samples hosted a strain-to-failure of 4.9% and the mixed samples held an elastic modulus of 6.23%. This information yields the hypothesis that the methods of dispersion with paraffin does not change the amount of strain capable for buckypaper to elongate, but more tests must be performed in future work to test this hypothesis.

7.4 Electrical Conductivity Testing

Electrical testing examines the conductivity of a buckypaper sample. Naturally, CNT networks are found to be electrically conductive due to their carbon bonds; however, the conductivity is undermined by resistive residual materials, such as paraffin. The following was a measurement of 3 samples, quantifying the effects of dispersion on the electrical properties of the resultant buckypaper. Sample sizes were cut to be a length of 25.4 mm by a width of 12.7 mm.

The mixed samples were shown to host electrically conductive properties. These properties can be located below in Table 3. The average value for the conductivity and resistivity are 58.08 S/cm and 0.0198 Ω/cm respectively. The standard deviations among samples were found to be 27.16 S/cm and 0.0085 Ω/cm respectively. Sample standard deviations can be high due to the variance in thickness, samples were found to have a thickness ranging from 0.5 mm to 0.1 mm along the surface of a single sample. The resistivity is relatively low which is consistent with the nature of CNT networks. Resistivity is the nature in which a material can resist the flow of electrical current.

Table 3: Electrical testing resultant buckypaper samples using mixing as dispersion

Name	Conductivity (S/cm)	Resistivity (Ω/cm)
Mixed Sample 1.4	88.09	0.0114
Mixed Sample 2.4	35.20	0.0284
Mixed Sample 3.4	50.94	0.0197

The second batch comprised of the samples dispersed using sonication. The following table, Table 4, identifies the measured values that were found when characterizing the material. The average conductivity among the samples is 172.44 S/cm. As for the average resistivity is 0.0058 Ω/cm. The standard deviation among the conductivity and resistivity samples were found to be 7.742 S/cm and 0.000252 Ω/cm. The standard deviations among the two electrical properties are rather low, giving confidence to the application of producing consistent samples.

Table 4: Electrical testing resultant buckypaper samples using mixing as dispersion

Name	Conductivity (S/cm)	Resistivity (Ω/cm)
Sonicated Sample 1.4	179.43	0.0056
Sonicated Sample 2.4	164.12	0.0061
Sonicated Sample 3.4	173.78	0.0058

In comparison of the methods for dispersion, it is evident that the sonication method produced higher electron carrying results. With averages that differed in conductivity of 172 S/cm to 58 S/cm, it can state with confidence that the sonicating method will produce higher conductivity than the mixing dispersion method. This is due to the CNT network being more dispersed in the sonication process which leads to better nanotube to nanotube contact. The more dispersion that occurs, the greater the bond density there will be.

8.0 Conclusion

       The objective of this research was to analyze a method of creating a buckypaper that would maintain its functionality while eliminating expensive and time consuming manufacturing components such as sonication and filtration. The overall analysis was a not fully successful in producing a highly structural buckypaper, but proved that simply mechanical mixing the CNTs with the suspension material is not as effective as the sonicating procedure. The mixed dispersion buckypaper, that was produced, held low conductivity and mechanical strength. These low properties were due to the low level of dispersion prior to the vacuum bagging process. Good dispersion will lead to great surface contacts, improving the properties of buckypaper Optimally, to produce a buckypaper using this method, further research must be explored in generating high heat through a means of low energy consumption as well as mixing must be further investigated in order to disperse the CNT network to a higher extent.

9.0 Future Work

1. Investigate into high speed thermo-conductive chambers with the absence of oxygen.

2. Test the alignment of CNT networks while stretching in temperature range of 40-50 °C.

3. Quantify the differences in strain among dispersion methods.

4. Generate a scalable plan of action required to implement the process.

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