FUNCTIONALIZATION AND METALLIZATION OF CARBON NANOTUBE MATS

by

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DEPARTMENT APPROVAL

of a senior thesis submitted by

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This thesis has been reviewed by the research advisor, research coordinator, and department chair and has been found to be satisfactory.

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ABSTRACT

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An intriguing mechanical material would be an aluminum / carbon nanotube composite. It could combine the ultra high strength of carbon nanotubes with the ductility and manufacturability of aluminum. We are studying the formation of this metal matrix composite by electroplating aluminum on preformed carbon nanotube structures. In order to induce aluminum growth on the nanotubes, chemical modification of the nanotube surface is required. Surface chemical functionalization was performed by suspension and immersion in a succinic acid bath for the loose nanotubes and nanotube mats respectively. The active surfaces consisting of carboxyl groups should form a stable chemical bonds with the aluminum. Characterization of the chemically functionalized buckypaper by water contact angle and x-ray photo electron spectroscopy (XPS) measurements will be presented. Initial metallization studies will also be presented.

ACKNOWLEDGMENTS

I would like to acknowledge the help of my research advisor Dr Robert Davis. Without his support, this project would not have left the ground.

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Chapter 1

Introduction

1.1 Overview

Since their discovery and identification in the early 1990's [1], carbon nanotubes (CNTs) have intrigued investigators. It has been shown that CNTs have many unique properties. Depending on their structural form, both conducting and semi-conducting carbon nanotubes exist. More relevant to this project is the exceptionally high tensile strength of CNTs. Single-walled carbon nanotubes can have a tensile strength up to 1 TPa. However, these unique properties of CNTs have yet to be harnessed in a bulk material. In particular, the tensile strength of a bundle of 10-15 nanotubes may fall off to about 100 GPa [2]. In this project, we attempt to combine the strength of carbon nanotubes and the manufacturability of aluminum in a macroscopic material. This is done by forming a metal matrix using carbon nanotube mats as a substrate, then depositing aluminum around the substrate. It is hoped that such a material would retain the strength of carbon nanotubes, while the aluminum would form a sort of glue that would keep the CNTs from separating. Such a material would be both lightweight and strong. When manufactured in bulk, it could be used in the construction of vehicles where lighter materials would result in greater fuel efficiency while stronger materials would result in greater passenger safety.

1.2 Metal Matrices

Metal matrices are a specialized form of material typically constructed for their inflexibility, high tensile strength, or other unique properties [3]. A metal matrix consists of two parts: a metal and the reinforcement material. The reinforcement material can be any one of several materials. Common substrates include polymer chains, carbon fibers, and other non-metallic substances. In many instances, the reinforcement material is embedded into or around the metal matrix. The matrices are completely continuous, meaning that there is a path through the metal from any one point to any other point. Sandwiching or layering materials does not constitute a metal matrix. A familiar example of a matrix is tungsten carbide, which is used in many tools. Amorphous carbon in powdered form is compressed into tungsten power at high temperature and pressure to form this type of matrix. In this examples, the carbon substrate provides added strength and durability to the metal tungsten. Research is also being done with carbon fibers as a substrate in aluminum matrices. Matrices formed by injecting molten aluminum into preformed fiber arrangements have been made with reported ultimate strengths on the order of 10 GPa [5]. These matrices must be created at high pressures to insure the adhesion of the aluminum to the carbon, which leads to the quick degradation of the carbon fibers, thus lowering the bulk strength of the material. Additionally, other matrices may be constructed for their high resistance to wear, or a lower coefficient of friction. Though many different methods for joining metals with substrates exist, in this project we have used electrodeposition. Carbon nanotube mats act as a reinforcing material to which aluminum is adhered.

1.3 Electrodeposition

Electrodeposition, also known as electroplating, is a process by which any electrically conductive substance can be plated with a layer of metal. A common example of electrodeposition is the plating of many surfaces, especially in automobiles, with nickel and chromium. Materials can be electroplated for cosmetic effect, as well as to discourage oxidation of the underlying metal. Conventional electrodeposition methods include creating a salt bath in which free metal ions are made. A current is run through the salt bath solution from some cathode to the item to be plated, which acts as the anode. The ions present in the solution are electrically attracted to the target item. As they come in contact with the surface, they form bonds to the item. Throughout the plating process the anode slowly dissolves, replenishing the ion content of the bath [4]. This project is unique for its use of aluminum as the metal to be electroplated. Aluminum is not usually electrodeposited. Aluminum salts, such as aluminum chloride are unstable and require special caution in their use. In spite of these difficulties, the high corrosion resistance and thermal conductivity of aluminum make it an attractive material for study. The mechanical properties of the electrodeposited aluminum layer are similar to those found in annealed commercially pure aluminum [6, 7].

1.4 CNT Modification

An additional problem inherent in the electrodeposition of aluminum is that aluminum does not wet carbon nanotubes. That is, aluminum does not readily adhere to the surface of the carbon nanotube surface. In the electrodeposition process, the aluminum ions need to bind to the carbon nanotubes in order for the plating to be successful. In order to facilitate this bonding, we modify the sidewalls of the nanotubes in such a way to form ready binding sites for the aluminum. The sidewalls of carbon nanotubes are structurally similar to graphene sheets. Graphite is the most stable form of carbon at standard temperature and pressure, and the bonding structure of nanotubes in particular is such that nanotubes are not reactive as molecules. In modifying the sidewalls of the nanotubes, we destabilize small sections of the tube and add in a functional group that is more readily reactive [8]. The functional groups that we use are carboxyls. Free carboxyl groups are easily formed from a number of different chemical reactions. In addition, they react comparatively easily with both the existing carbon bonds of carbon nanotubes as well as with aluminum ions.

1.5 Buckypaper

Carbon nanotube mats, the substrate for our electrodeposition, are thin sheets of non-aligned carbon nanotubes. These mats are made by suspending CNTs in solution and then filtering them. This results in a layer of nanotubes spread across the filter, which can then be separated from the tubes resulting in a sheet of CNTs commonly called buckypaper. In comparison to the strength of the individual tubes, the nanotube mats are extremely delicate and fragile. The tubes are held together in a bulk structure only by intermolecular van der Waal's forces. These forces are very weak as compared to the intramolecular carbon bonds. Due to their small dimensions, small forces, when applied to the buckypaper, can cause the nanotubes to slip and separate. Because of this, buckypaper by itself is not a useful bulk material. Standing alone, buckypaper has a tensile strength that can range from approximately 6-9 MPa up to 30 MPa. The

Young's Modulus of buckypaper has been measured to be from 1.1 to 6.9 GPa [9,10].

1.6 Project Scope

Over the past several years, many attempts have been made to incorporate carbon nanotubes into a bulk material. These materials would, in theory, have very high strength to density ratios. The majority of current research focuses on various methods for combining CNTs and polymer chains. One such method involves the simple mixing of unpurified nanotubes with polyacrylonitrile (PAN), resulting in a composite structure with a tensile strength of approximately 100 MPa [9]. Another commonly used method involves intercalating polymers such as poly(vinylpyrrolidone) into buckypaper from solution. At certain solution densities, the polymer will diffuse into the buckypaper structure. These composites increase the strength of the polymer by up to six times [11]. A third popular method involves layered deposition of CNTs and polymer solutions. These composite stacks may be up to 60 % nanotubes by weight, and have tensile strengths near 100 MPa [12, 13]. In each of the above methods, as well as with many methods involving polymer/CNT composites, surfactants were used to lower the interfacial tension between the two substances. The degree to which the tensile strength of these materials is improved depends directly on the effectiveness of these surfactants. Stronger polymer/nanotube bonds result in stronger bulk materials.

In this project, we attempt to negate the necessity for any type of surfactant by directly modifying the nanotubes in the buckypaper. In theory, this will create additional covalent bonding sites between the tubes, as well as providing bonding sites for aluminum atoms. We then electrodeposit aluminum onto the modified buckypaper. The aluminum is expected to bond with the functional groups on the nanotubes, as well as to already bonded aluminum ions. We allow the aluminum to 'grow' on the CNT substrate and hold the nanotubes together. The expected result is a composite material with physical properties similar to aluminum, but with a greatly increased tensile strength. The modification of the nanotubes is described in chapter two. Additional information regarding the electrodeposition process is also provided in chapter two, as well as a short summary of the future work needed. Chapter three will focus on the equipment developed and built to take mechanical measurements on the buckypaper samples. We will also include preliminary results and discuss the current direction and goals of the project.

Chapter 2

Metallization and Functionalization

2.1 Functionalizing Tubes

In order to facilitate strong carbon-aluminum bonds in our material, we modify the sidewalls of the carbon nanotubes. Nanotubes come together with hybridized sp^2 carbon-carbon bonds. To functionalize the nanotubes we break a small number of these carbon-carbon bonds. Because these bonds are the fundamental structure of the nanotube, the broken bonds weaken the overall structure of the tube. In order to maintain the structural properties of nanotubes, the number of broken bonds must be small compared to the overall size of the nanotube structure. The broken bonds 'dangle' off the sides of the nanotube and are ready binding sites for the free carboxyl radicals. These free radicals are very reactive, and easily bond at any point possible. The resulting product consists of mostly intact carbon nanotubes with exposed carboxyl groups scattered throughout the length of the tube. The bonded carboxyl groups have oxygen atoms at the end pointing away from the CNTs. These oxygen atoms act as bonding sites for the free aluminum ions in the electrodeposition solution. In addition, carboxyl groups from neighboring CNTs in nanotube mats increase the intermolecular bonding strength and provide greater rigidity to the mats.

Currently we have investigated two processes for accomplishing this reaction. The first uses peroxide composed of succinic acid. In this process, succinic anhydride powder is mixed with hydrogen peroxide at zero degrees Celsius. The powder/peroxide mixture is stirred and heated until a thick gel is formed. This gel was then filtered, washed, and vacuum dried overnight. The resulting peroxide is a mixture of two similar peroxide radicals.

When the peroxide has been formed and dried, carbon nanotubes are added to a solvent, such as o-dichlorobenzene, and sonicated until they become suspended in solution. The CNT solution is then heated to approximately 80 °C, and a small amount of the dried peroxide is added. The reaction rate for the CNT-peroxide reaction is very slow. The small diameter of the nanotubes forces the carbon-carbon bonds into a very tight conformation. As a result, the attacking carboxyl radicals do not easily react with the sidewalls. To ensure sufficient reactivity, the solution is heated and stirred continuously for up to ten days. Each day more dried peroxide is added to the solution. At the end of this reaction period, the nanotubes are retrieved from solution and rinsed with excess amounts of ethanol. The solution is also filtered. The ethanol breaks down unreacted peroxides and cleans the modified nanotubes of other reaction by-products. The modified buckypaper can then be obtained from the filter.

The second functionalization process involves a similar chemical ($C_{12}N_6Cl_2H_{20}$) commercially available as Vazo 68. When heated above 68 °C, the Vazo will spontaneously decompose into free carboxyl radicals. As with the succinic peroxide, Vazo is added to a nanotube suspension over the course of several days. Unfortunately, at the point of writing, the Vazo process has not been successful.

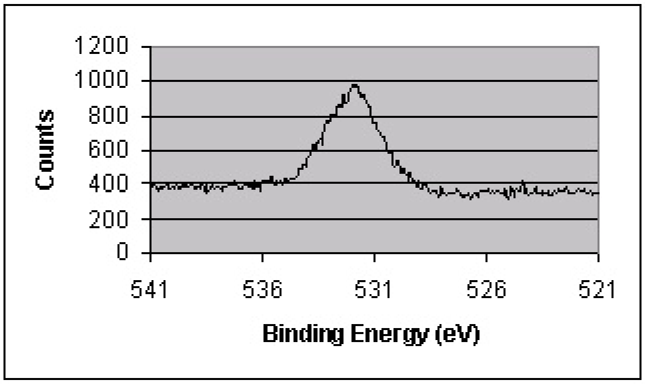

Figure 2.1 Spectrum of Unmodified Buckypaper

2.2 Characterizing Functionalization

Upon completion of the functionalization process, the modified nanotubes were tested to verify the presence of the desired carboxyl groups. Measurements were taken from samples of buckypaper composed of nanotubes modified with the succinic acid process. The nanotubes were thoroughly rinsed with excess amounts of ethanol, washing away any unreacted anhydrides or peroxides. The nanotubes were then filtered and vacuum dried overnight at 80 °C, resulting in buckypaper composed of modified nanotubes.

Using X-ray photoelectron spectroscopy (XPS), we analyzed both regular buckypaper and buckypaper made from modified nanotubes. Figure 2.1 shows the spectrum of typical buckypaper. The single peak is representative of a single type of carbon present in the structure. The two peaks present in Figure 2.2 show that two distinct types of carbons are present in the sample. The shift of the smaller peaks leads us to believe that carbon-oxygen bonds are present in the nanotube structure.

Additionally, qualitative measurements were made using water droplets and contact angle measurements. A small droplet of water was dropped onto an untreated buckypaper sample. The contact angle between the droplet and nanotube was ap-

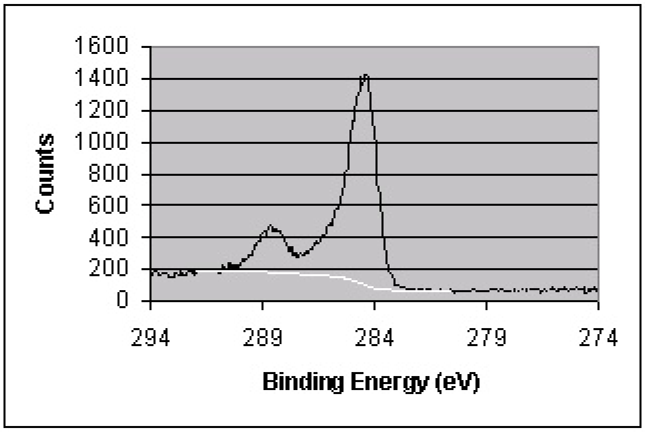

Figure 2.2 Spectrum of Modified Buckypaper

proximately 70°. The droplet remained beaded up on the surface and was easily blown off. This signifies that the water did not wet the surface of the mat. When water was dropped onto the surface of a CNT mat composed of modified nanotubes, the water contact angle reduced to approximately 25° to 30°. Additionally, the water was not easily removed from the surface, signifying the water wetted the surface of the modified paper. Such visual measurements can be done quickly in the lab and allowed us to test modified buckypaper samples with reasonable confidence that the modification was successful.

2.3 Electrodeposition

The electrodeposition solution uses diethyl ether as a solvent. Aluminum chloride is dissolved in the ether. This reaction is highly exothermic, and must be cooled constantly while being mixed. We found this was best accomplished by keeping liquid nitrogen in close thermal contact with the solution. A small amount of lithium aluminum hydride is also dissolved into the ether. The addition of the lithium will react readily with any moisture in the solution or in the environment. The solution needs to be stirred well in order to ensure that the aluminum chloride is fully mixed. The final electroplating solution is very anhydrous and the whole process must be carried out in an inert environment. The solution composition is detailed in table 1.

Solution Components	Quantity
Aluminum Chloride: $AlCl_3$	400 g/L
Lithium Aluminum Hydride: $LiAlH_4$	15 g/L

In order to test the usefulness of this solution, we first electroplated pennies. We found that currents between 10-15 mA resulted in steady plating rate of a few nanometers per minute.

2.4 Characterizing Electrodeposition

To ensure that the end result of the electrodeposition was aluminum, we prepared a small sample that could be viewed on a Scanning Electron Microscope. The grain size of the deposited aluminum was fairly uniform, on the order of several hundred nanometers. In many areas of the sample residual aluminum chloride deposits could be found. In order to clean the samples of any remaining salts, the samples were then washed with ether, which reacts easily with aluminum chloride while not reacting with the aluminum or nanotubes. When plating unmodified buckypaper, the aluminum did not adhere uniformly across the surface. Aluminum streaks began from different points along the edge of the sample and spread vertically up the paper. The resulting paper was extremely brittle, and unusable for taking any strength measurements. In the future, testing longer plating times may increase the coverage of aluminum on the sample. Further testing to see what structural changes occur in the buckypaper will also be necessary. Modified buckypaper, made from CNTs put through the succinic acid process, accepted the plating much more readily than the regular samples. The coverage appeared uniform across the plated surface. Additionally the sample was not brittle and could be used in mechanical measurements.

Chapter 3

Mechanical Measurements

3.1 Strain Testing

The chief measurements describing the different types of CNT mats are the Young's modulus and the ultimate strength. The Young's modulus is a measure of tensile strength, and the ultimate strength is the maximum stress that can be applied to the sample. The ultimate strength is also known as the tensile strength. To characterize our various sample materials, we applied a small horizontal force on our sample. The force on the sample was gradually increased, and we measured the increase in length due to the applied force. We then generated graphs showing applied force versus horizontal strain. The slope of the curve during the initial stage of the run is the Young's modulus, and the maximum stress reached during the pulling is the ultimate strength.

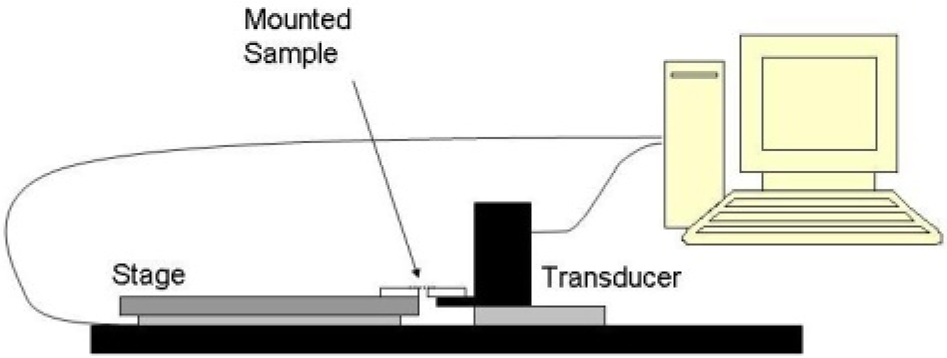

Figure 3.1 Setup of apparatus used to take mechanical measurements.

3.2 Stage and Transducer

We measured the Young's modulus of our various types of treated, untreated, plated and non-plated CNT mats. To facilitate these measurements we built a two-part apparatus (see figure 3.1). The first part consisted of a movable stage on which we mounted one side of the sample. The other side of the sample was mounted on the end of a force transducer. This left a small area of the sample suspended between the two halves of the apparatus. The stage was controlled by a piezoelectric crystal. The stage was set up so that over a ten volt range, it would move by approximately 116 microns. Using LabView, I designed a small virtual instrument to control the apparatus. (see Appendix A for details) The program had both automatic and manual control over the output voltage to the stage. On most runs, the program automatically moved the stage over its complete range of movement in three minutes. The program was also set to record the force exerted on the sample resulting from that movement. Samples were taken at a rate of 100,000 Hz. Each second, the samples were averaged and the average voltage was recorded. The force transducer was found to have a voltage response of approximately 0.18 mV/gram. This calibration was obtained by orienting the transducer vertically and hanging weights of known mass from its end. The voltage readings were recorded for each weight and a linear curve fit was found from the resulting voltages.

The force transducer used in this apparatus is the Honeywell FSG15N1A. The transducer was constructed to measure compressive force with a maximum measurable force of 14.7 N. In order to use this transducer to measure tensile force, we set a spring against the transducer pushing with a force of 1.5 N. As the stage was moved, the samples would pull on that spring, reducing the force measured by the transducer. Additionally, the force transducer was mounted on a movable base so that we could increase the tension on the sample independently of the stage movement. In the event that the sample did not break after an increase in length of 116 microns, the force transducer could be moved back by approximately 100 microns and then the program could be rerun. In this way, any sample could be stretched to its ultimate breaking limit in 100 micron increments. The force exerted on the sample was monitored continuously throughout this movement.

3.3 Results and Discussion

The initial tensile strength tests were taken on non-plated buckypaper made from unmodified tubes. These samples yielded a Young's modulus on the order of 650 MPa, with high repeatability. A Young's modulus of 650 MPa is 50% lower than expected for buckypaper, and comparable to materials such as rubber, low density polyethylene, and other polymers. However, the ultimate strength of these buckypaper samples averaged 30 MPa. This ultimate strength matched values for similarly prepared buckypaper samples as reported by Berhan, Smalley and others [10, 14]. A high ultimate strength in combination with a low Young's modulus leads us to believe

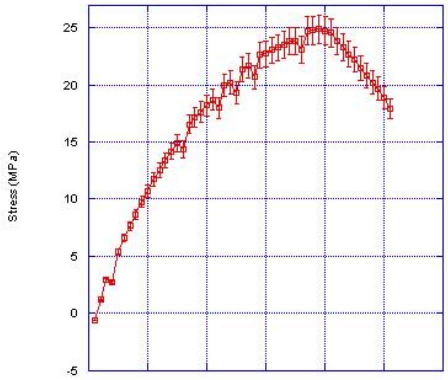

Figure 3.2 Stress/Strain graph for unmodified buckypaper. The sample was 1.22mm X 1.44mm with a thickness of 10 microns.

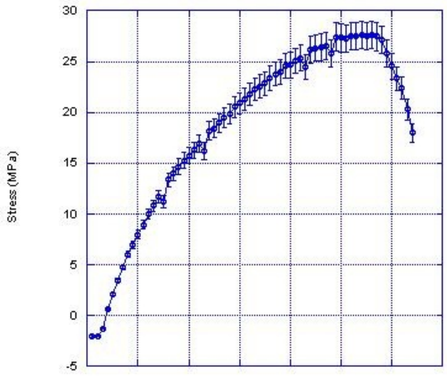

Figure 3.3 Stress/Strain Graph for unmodified buckypaper. The sample was 1.16mm X 1.26mm with a thickness of 10 microns.

that untreated buckypaper is a fairly elastic material. This theory is supported by observations while taking these measurements. Some buckling in the buckypaper was observed when a stretched sample was relaxed. It is probable that the individual nanotube bundles and ropes that compose the sample are somewhat free to slip past each other as tension is applied. As the tension increases, the tubes become increasingly aligned along the direction of the applied force. This alignment lengthens the sample along the direction of the applied force. At some limit, the increased tension overpowers the intermolecular forces and causes the nanotubes to slip apart from each other, breaking the sample. Figures 3.2 and 3.3 show typical results obtained from buckypaper samples. The samples used in taking this data were approximately 1.2 mm wide with a suspended length of 1.5 mm. The average thickness of the buckypaper produced for this study was 10 microns. During these runs, the stage moved 140 microns per minute. The maximum force reached during this time was approximately 0.026 N.

Initial results for buckypaper made from CNTs modified with succinic acid show a substantial increase in ultimate strength but only limited increase in Young's modulus.

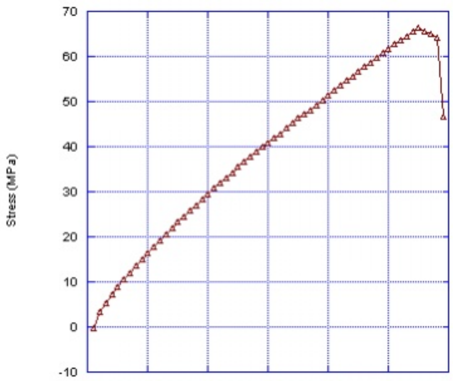

Figure 3.4 Stress/Strain graph for buckypaper composed of CNTs modified with succinic acid. The sample was 3.1mm X 1.45mm with a thickness of 10 microns.

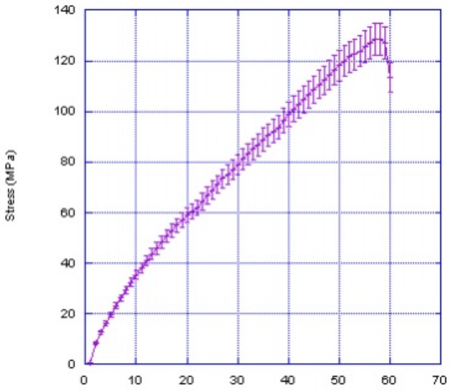

Figure 3.5 Stress/Strain graph for modified buckypaper, plated with aluminum for 12 hours. The sample was 1.66mm X 1.1mm with a total thickness of 20 microns.

Figure 3.4 shows the stress/strain curve for a sample of buckypaper made from CNTs modified with succinic acid as described in chapter 2. The ultimate strength of this sample was more than double that of regular buckypaper, averaging approximately 60 MPa. Though the ultimate strength of the material increased as compared to regular buckypaper, the Young's modulus remained around 650 MPa. The samples used to obtain this data were approximately 10 microns thick, 2.5 mm wide and had a suspended length of 1.5 mm.

Figure 3.5 is from a sample of modified buckypaper that was plated with aluminum using a 9 mA current for 12 hours. The original sample was 10 microns thick, and the estimated thickness after electrodeposition is 20 microns, with the aluminum distributed on both sides of the sample as well as interspersed throughout the original sample. The ultimate strength of this sample was 128.6 MPa, and the Young's Modulus remained approximately 650 MPa. Pure aluminum, on the other hand, has an ultimate strength of approximately 40-50 MPa. This sample was 1.6 mm wide and had a suspended length of 1.1 mm.

3.4 Future Work

In the future, additional tests will be run on both plated and unplated buckypaper made from both carbon nanotubes modified with both the succinic peroxide as well as unmodified nanotubes. We hope to be able to test the mechanical measurements of plated, unmodified buckypaper. We also plan to plate buckypaper samples with several different thicknesses of aluminum so that we can find a minimum thickness for maximum strength. Moreover, it may be helpful to test the Young's modulus of aluminum plated onto a thin layer of gold. In this way we could test the strength of the aluminum structure as manufactured through electrodeposition. Further studies are also needed in determining the nanoscale structure of the different samples. Specifically, we hope to discover the effect of succinic acid modification on the structure of the nanotubes, as well as its effect on intertubular bonds in buckypaper. More study is needed on discovering how the succinic acid modification aids in the uniform aluminum deposition as well. Through these measurements the feasibility of a carbon nanotube based, bulk quantity aluminum matrix material can be determined.

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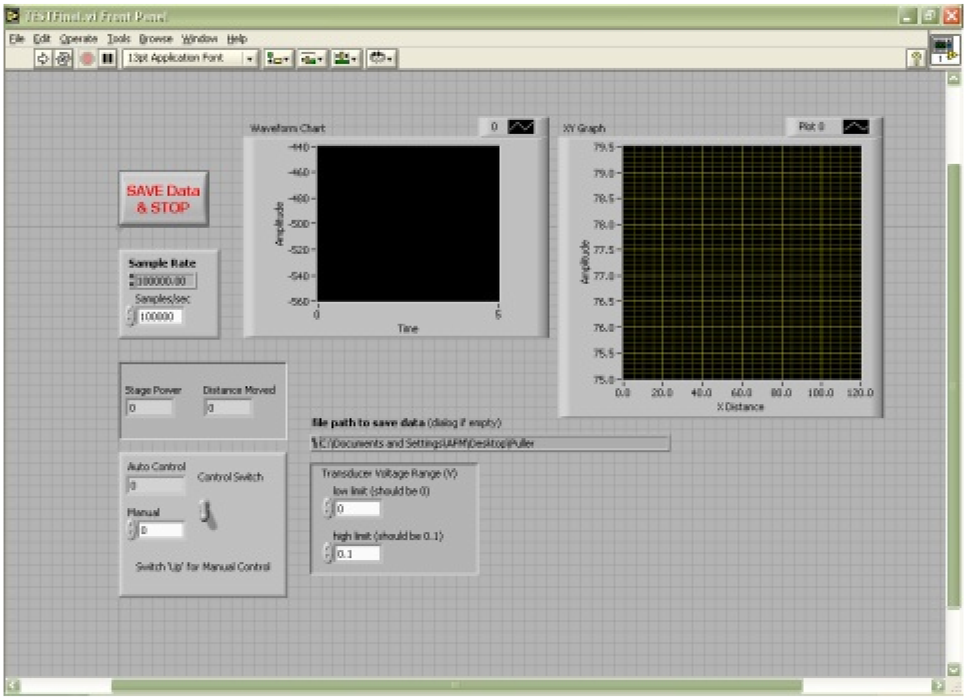
Appendix A

LabView

A.1 LabView VI

Figures A.1 and A.2 show the front panel and block diagram of the Virtual Instrument used in taking data. This program was set to slowly increment the voltage controlling stage movement to its maximum. Upon reaching the maximum, the voltage is decreased, relaxing the buckypaper back to its original length. Throughout this process, the force as measured by the transducer is taken as described in chapter 3. The data is plotted on a voltage vs. distance graph and saved to a spreadsheet for further analysis.



Sample Rate 100000.00

Samples/sec 100000

Stage Power 0

Distance Moved 0

file path to save data (dialog if empty) C:\Documents and Settings\AFM\Desktop\Puller

Auto Control 0

Manual 0

low limit (should be 0) 0

high limit (should be 0.1) 0.1

Figure A.1 VI Front Panel

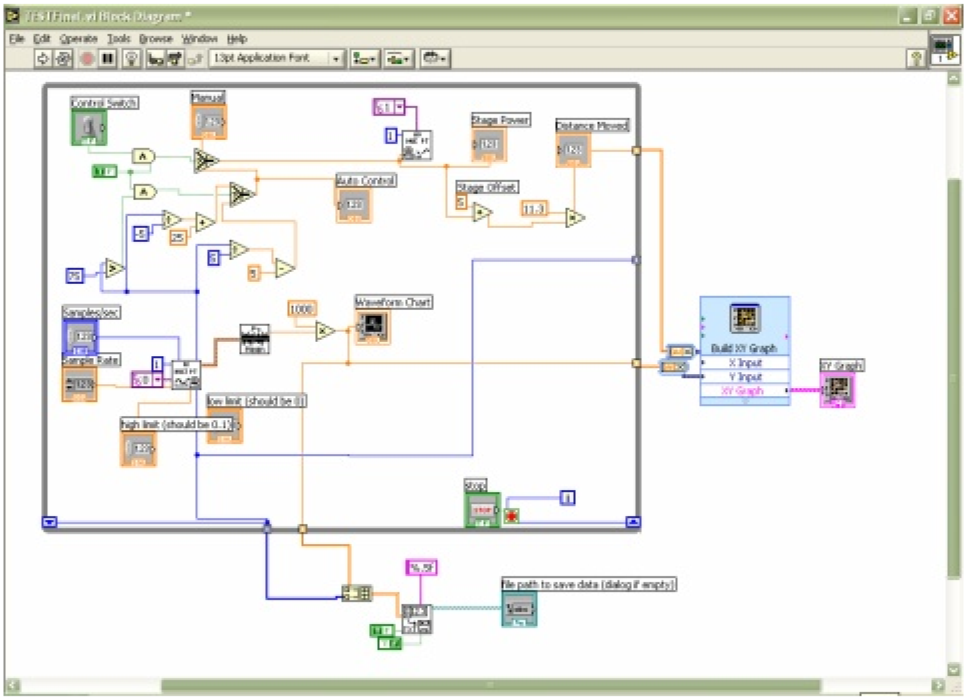


Figure A.2 VI Block Diagram

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