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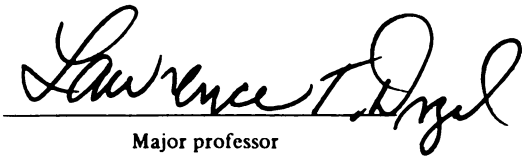
THE ALIGNED DISCONTINUOUS FIBER PROCESS: EXTENSION INTO MANUFACTURING

presented by

Scott Andrew Seymour

has been accepted towards fulfillment
of the requirements for

PhD degree in Chemical Engineering



Major professor

Date 8/25/00

THE ALIGNED DISCONTINUOUS FIBER PROCESS: EXTENSION INTO MANUFACTURING

By

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A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTERS OF SCIENCE

Department of Chemical Engineering

2000

ABSTRACT

THE ALIGNED DISCONTINUOUS FIBER PROCESS: EXTENSION INTO MANUFACTURING

By

Scott Andrew Seymour

The Aligned Discontinuous Fiber (ADF) Process, which was previously developed at Michigan State University, has been shown to work with several industrial materials. Chopped glass and carbon fibers of lengths ranging from 0.25" to 1" have been aligned in electric fields and glass fiber-nylon12/polypropylene(PP), polyethlyene terephthalate(PET), and nylon –12 powder coated ADF preforms have been manufactured using this process. Carbon fiber/nylon – 12 preforms have also been manufactured. The parameters that affect the orientation of the fibers in electric fields are investigated viz. fiber aspect ratio, dielectric nature of the fiber, and the Reynolds number of the free falling fibers. High speed motion analysis is used to record the events, and the orientations of the fibers under each condition is measured with image analysis techniques to get an orientation distribution function. Significant improvements in performance, up to 100 % in stiffness and up to 85 % in strength values have been obtained due to fiber alignment and choice of proper sizing. Unlike continuous fiber random mat reinforced composites which have poor drapeability and which have problems of delamination under compression, aligned discontinuous fiber composites are flexible and can be molded or stamped into complex parts.

ACKNOWLEDGEMENTS

This would not have been possible without the help of several people. I would like to thank Dr. Drzal for advising me and giving insight on this project. The Michigan State University Cyclotron were invaluable in the trouble-shooting of the ADF apparatus, and provided knowledge concerning electric fields. I would also like to thank all of the CMSC Staff and Chemical Engineering Office for all of their help that they have provided me with throughout my stay at MSU.

Finally, I would like to thank my fellow CMSC graduate students, Arvind Krishnaswamy, Shawn Corbin, Tammy Cummings, and the rest for making the MSU experience fun and not always serious. You all owe me an ice cream.

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1.1 Introduction

A novel process for producing composite materials was developed at Michigan State University. The Aligned Discontinuous Fiber (ADF) process was created to fill a void in composite material selection, one between aligned continuous composites and random discontinuous composites. Both will be described in further detail in this chapter.

The ADF process consists of a fiber feeder, an alignment chamber, and a collection tray. It is important to note that in most parts of this work the term 'fiber' refers to a fiber bundle that contains 200 individual fibers for glass and 3000 fibers for carbon. Fibers are fed into the alignment chamber from the feeder and a preferential alignment is imparted. The aligned fiber bundles are then collected on a tray that rests below the alignment chamber. The focus of this research was fourfold:

- To apply the ADF process to a representative number of composite material systems.
- To modify the aligned discontinuous fiber process for use with conductive carbon fibers.
- To manufacture and evaluate the mechanical properties of molded ADF parts
- To perform an economic analysis on the ADF process to determine its viability for industrial scale-up and implementation as a manufacturing process.

This chapter will give a brief overview of composites in general, elucidate the theory of discontinuous fiber composites and discuss the factors that affect their processing and properties. Finally, the research objectives will be discussed.

1.2 Composites

A composite material is defined as a solid, which is composed of two or more constituents having different physical characteristics and in which each substance retains its identity while contributing desirable properties to the whole. Examples of naturally occurring composites include bone and wood, while man-made composites such as concrete and carbon black in rubber have been in use for centuries. A composite material consists of two components: a reinforcing phase consisting of fibers or particles, and a matrix phase consisting of plastics, metals, or ceramics. Most composite materials that have been developed so far have been fabricated to improve mechanical properties such as strength, stiffness, toughness, and high-temperature performance. In this work, the main properties that will be discussed will be the composite's tensile modulus, tensile strength, and fiber orientation. The material's tensile modulus is a measure of its stiffness, tensile strength is a measure of how much force it requires to pull the material apart, and the fiber orientation is a measure the degree of fiber alignment. It is important to note that the main difference between composites and pure materials is that composite materials are on a whole anisotropic (with the exception of uniform particle reinforced composites). While composites can be engineered to have exceptional properties, they will not usually exhibit these properties in all directions. This anisotropy arises because

of the variation of microstructures that is inherent in composite materials.[1] Today these microstructures can be exploited to design high performance composites that save weight and space over conventional materials, while providing superior mechanical properties.

1.3 Particle Reinforced Composites

Composites can be divided into two main groups: fiber reinforced and particulate

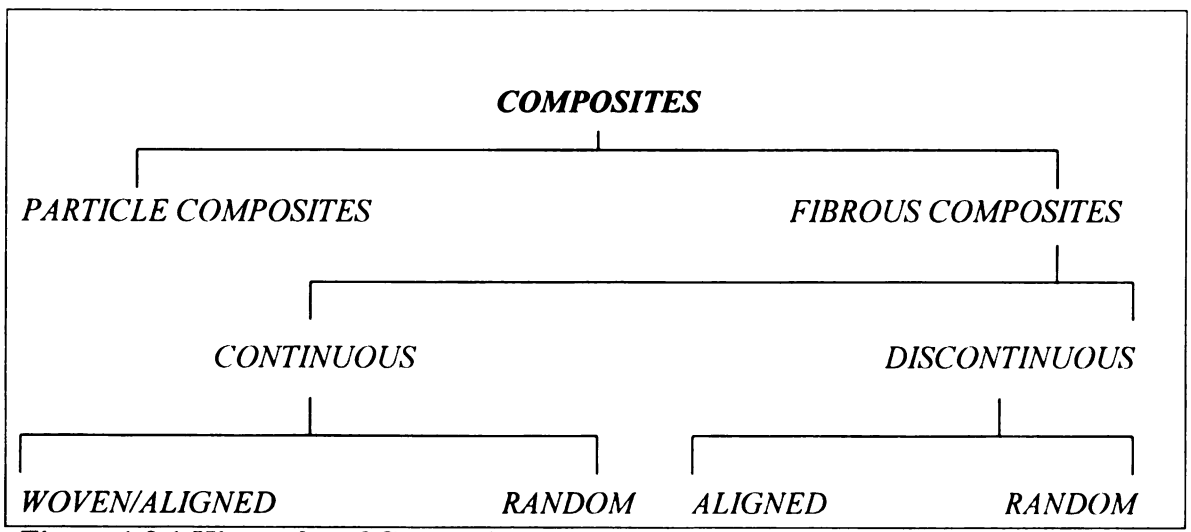


Figure 1.3.1 Hierarchy of Composites

reinforced. Figure 1.3.1 diagrams the hierarchy of composite materials. A particulate is defined as one that is non-fibrous and generally has no long dimension, with the exception of platelets. The dimensions of the reinforcement determines its ability to contribute its properties to the composite, i.e. a round particle would add properties in all directions, where a fiber would add properties along the fiber direction. Particles are

[1] Piggott, M., "*Load-Bearing Fibre Composites*", Pergamon Pres, 1987

effective in enhancing the stiffness of composites but do not offer the potential for much strengthening. For example, hard particles placed in a brittle matrix reduce the strength due to stress concentrations in the adjacent matrix material. Particles can also be used to improve the fracture resistance of brittle materials. Adding rubber-like particles to a brittle matrix improves the fracture resistance by initiating and arresting cracks.[2] Particles are also added as fillers to materials, which serve to improve various properties while reducing cost and without sacrificing the other desirable properties. Table 1.3.1 lists several reinforcing media that are widely used in industry.

Table 1.3.1 Typical Properties of Commercial Reinforcing Media

Fiber/Particle	Diameter (μm)	Specific Gravity	Tensile Modulus (10^6 PSI)	Tensile Strength (10^3 PSI)
E-Glass	10	2.54	10.5	500
S-Glass	10	2.49	12.6	625
PAN Carbon AS-4	8	1.8	35.9	590
PAN Carbon IM7	5	1.78	43.6	770
Pitch Carbon P-55	10	2.0	55	275
Kevlar 49	10	1.45	19	525
Boron	140	2.7	57	450
SiC	133	3.08	58	485
Al_2O_3	20	3.95	55	275

1.4 Fibrous Composites

Fiber-reinforced materials offer a combination of strength and modulus that are either comparable or better than traditional materials. These materials also exhibit lower

[2] Agarwal, B.D., and Broutman, L.J., *"Analysis and Performance of Fiber Composites"*, John Wiley and Sons, 1980.

specific gravities, higher strength-weight ratios, and higher modulus-weight ratios. For these reasons, fiber-reinforced materials have emerged as a fast growing class of materials used in vastly increasing areas. Fibrous composites can be subdivided into several groups, two of which will be discussed in detail in this thesis - continuous and discontinuous fiber composites. Of these two groups, they can both be further subdivided into two groups, aligned and randomly oriented composites. (Refer to Figure 1.3.1)

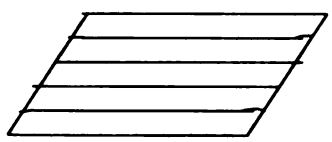
Theoretically, continuous fibers can transmit a load or stress from the application site to the reaction site via a continuous load path.[3] However, this is rarely observed in practice for two reasons: 1) manufacturing variables make it impossible to produce fibers with perfect surfaces that will fail at their ultimate tensile strength, and 2) in the ideal continuous fiber structure the fibers will be stress-free or equally stressed, but this situation rarely happens in reality. Instead, during processing some fibers will be highly stressed while others will be unstressed. As the finished composite is loaded to its ultimate strength, the pre-stressed fibers will support more of the load than the fibers with the lower built-in stresses. The pre-stressed fibers will fail before the unstressed fibers and consequentially reduce the ultimate tensile strength of the finished composite. The role of the matrix in fiber-reinforced composites is not to be a load-carrying constituent, but to simply bind the fibers together and protect them.

Fibrous composites, along with some particulate composites (whose particles contain a long axis) can be described as either random or aligned, depending on their fiber/particle orientation. There are three types of fiber orientation: one-dimensional, planar or two-dimensional, and three-dimensional reinforcement. This introduction and thesis will concentrate on one-dimensional reinforcement primarily. Although aligned

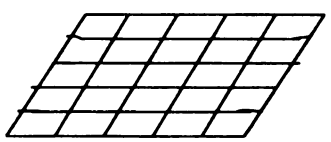
fiber composites exhibit higher mechanical properties than randomly oriented composites, aligned composites are not necessarily the best choice for every application. Aligned fiber composites are generally reserved for applications which require high mechanical properties in a single direction. Whereas randomly oriented composites are used where a low cost and uniform properties in all directions are required.

1.4.1 Continuous Fiber Composites

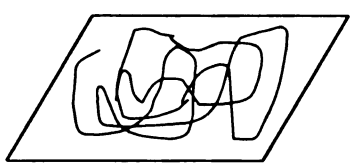
Continuous fiber composites are widely used in the aerospace, automotive, and commercial sectors. Depending upon the application, the fibers can be aligned, random, 2-dimensionally woven, knitted, braided, or woven in a 3-D pattern. In this work we will deal exclusively with random and aligned.



Unidirectional Laminate



2-D Isotropic Laminate



Random Roving

Figure 1.4.1.1 Types of Continuous Fiber Composites

[3] Piggott, M., *"Load-Bearing Fibre Composites"*, Pergamon Press, 1987

Randomly oriented continuous fiber composites are usually made from continuous fiber strands called roving or twisted yarn. These fibers are then generally laid into mats and then molded into whatever shape is required. Applications for these types of composites can be found in some papers and cardboards, along with many types of packaging materials. One advantage random continuous fiber composites have over random discontinuous fiber composites is that they do not tear as easily because of the greatly increased length of the continuous fiber.

For a one-dimensional reinforced fibrous composite the greatest strength and modulus are obtained along the fiber direction, with greatly reduced properties transverse to the fiber direction. To counteract this weakness, continuous fiber composites manufacturers have borrowed technology from the textile industry to create woven, knitted, or braided two-dimensionally reinforced preforms. These preforms can then be impregnated with matrix to form the final composite. Manufacturing of these continuous fiber composites is more difficult and expensive than discontinuous or particulate composites, but in certain applications the mechanical property requirements demand that continuous fiber materials be employed.

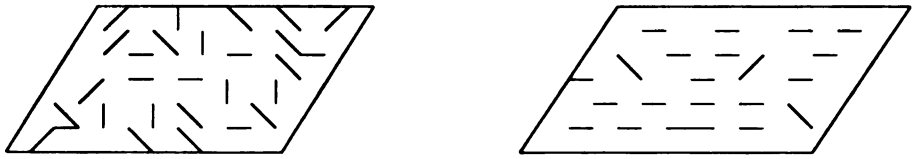
Calculating mechanical properties for aligned continuous fiber composites is quite easily accomplished by using the Rule of Mixtures:

$$E_c = E_f V_f + E_m V_m$$

Where E is the tensile modulus, V is the volume fraction and the subscripts c, f, and m stand for composite, fiber, and matrix.

1.4.2 Discontinuous Fiber Composites

Discontinuous fiber composites can be referred to by several different names, depending upon how they are used: short fiber when injection molded, discontinuous fiber when employed in reinforced metal matrix composites, chopped fibers when the fibers are secured in bundles, and long chopped fibers when the fiber bundles are longer than ½". The nature of discontinuous fibers serves as a source of discontinuity in the stress-transfer from the matrix to the fiber, which results in the reduction of composite mechanical properties. But, on the other hand, discontinuous fibers offer tremendous advantages in ease of processing, cost, and can be employed in many applications where a more isotropic material is required.



Random Layer Aligned Layer

Figure 1.4.2.1 Types of Discontinuous Fiber Composites

When designing a part that will be subjected to a complex load field, it is important to utilize a material that will have good properties in all directions – something that cannot be attained with continuous fiber composites. (Although bi-directional laminates and weaves can approach this) Fiber lengths can range anywhere from a few millimeters to upwards of 2" to 3". Because of the variety of fiber lengths and types that

can be utilized discontinuous fibers can easily be engineered and processed to have a wide range of microstructures tailored to the specific load field. An important parameter when dealing with discontinuous fibers is the critical length, l_c. When using discontinuous fibers that are shorter than this critical length, the full strength of the fiber cannot be realized in the finished composite. The following equation governs the average critical length:

$$l_c = \frac{\sigma_f d}{2\tau_c}$$

where σ_f is the fiber tensile strength, d is the fiber diameter, and τ_c is the shear stress at the boundary of the fiber and matrix. Some sample examples of critical fiber lengths are given in table 1.4.2.1. The shortest fiber used in this work was 1/8", well above the critical length for the thermoplastic polypropylene matrix listed in the table.

Table 1.4.2.1 Sample Critical Fiber Lengths for Glass and Carbon Fibers

Fiber	Matrix	L_c
E-Glass	**Polypropylene**	**0.07 inches**
E-Glass	**Epoxy**	**0.017 inches**
Carbon (AS-4)	**Polycarbonate**	**0.030 inches**
Carbon (AS-4)	**Epoxy**	**0.017 inches**

Whereas with continuous fiber composites a simple expression such as the rule of mixtures can describe the material properties of an aligned composite, the calculations become more rigorous when short discontinuous fibers are studied. A more rigorous

examination of various micro-mechanical models for predicting tensile strength and modulus in short discontinuous fiber composites will be presented in Chapter 4.

1.4.3 Discontinuous Fiber Alignment

When discussing alignment of fibers, it is important to develop a parameter to act as a measure of fiber alignment. In this work, the planar orientation parameter, f_p, will be used to describe the fiber orientation distribution. For planar orientation, f_p, is given as:

$$f_p = 2\int_0^{\pi} N(\Phi)\cos^m \Phi d\Phi - 1$$

where N is the fiber distribution function, and Φ is the angle of the fiber from the reference axis. A more rigorous derivation of f_p for the purposes of this work will be shown in Chapter 4, along with the Halpin-Tsai equations, which are used to predict mechanical properties from a micro-mechanical model.

Discontinuous fibers hold several advantages over continuous fibers, which include rapid, low-cost processibility by compression or injection molding, or by extrusion, and the ability to mold complex geometries that could not be fabricated using continuous fibers. Theoretically, discontinuous fibers can also be produced with a lower number of surface flaws, which would allow them to approach more closely their theoretical strength. Therefore, engineers and scientists have been trying to develop processes, which could align short discontinuous fibers on an industrial scale. Below is a brief summary of prior attempts to tackle this problem.

1.4.4 Flow Induced Alignment Processes

Most of these processes are dependent upon flow induced alignment. One of the first, the ERDE Glycerine Process was developed by the Explosives Research and Development Establishment (UK).[4] This process uses a dilute dispersion of fibers in glycerine passed through a tapered slit at a constant throughput onto a filter bed, which is under vacuum. Fiber alignment is achieved via the velocity gradients that are setup between the accelerated streamlined flow and the stationary boundary of the tapered slit. The viscous drag on the fibers created by the velocity gradients orients the fibers. The fibers align in the direction of the motion of the liquid and are collected on the filter bed in a reciprocating manner. The glycerine is then evacuated quickly through the filter bed which is under vacuum without disturbing the fiber orientation.

The second process, known as the Budd-Slurry process has been developed to manufacture net shape preforms for the automotive industry.[5] The composite is made from the shape preform using a conventional Resin Transfer Molding process. This process is well used in the paper manufacturing industry to make test sheets of papers. The main component of this process is a large tank that is filled with an aqueous slurry of fibers. This slurry is positioned above a filter screen that has a vacuum valve on the backside of it. The mat can be made in two different ways, either raising the filter screen up through the slurry, or using that vacuum to evacuate the liquid, leaving a fiber mat deposited on the screen. Slight fiber alignment can be achieved by using a set of baffles that the fibers pass through as the liquid is evacuated. It should be noted that this process

[4] Kacir, L., Narkis, M., and Ishai, O., "Oriented Short Glass-Fiber Composites", *Polymer Engineering and Science*, Vol 15, p 525-532, 1975.

was not developed for the manufacture of aligned preform mats, but to make high fiber volume fraction randomly oriented mats.

Whereas the above techniques incorporate the matrix in a separate step, using injection molding equipment to achieve fiber orientation removes this step by utilizing the matrix as the orientation medium. Typically, short fibers are used and are premixed in a molten polymer (typically around volume fractions between 10% to 30% with fiber lengths less than 1/8") and injected at high pressure into a mold. Orientation of the fibers is usually perpendicular to the flow in the core regions of the mold and parallel to the flow in the wall or skin regions. The phenomenon results from high shear rates near the wall and extensional flow in the core regions. Several factors can influence the amount of fiber orientation achieved, which include the design of the mold, the injection pressure, the temperature of the molten polymer mixture, and the mold temperature.

The last of the techniques to be described in this work that utilizes flow-induced alignment is extrusion. As with injection molding, the molten polymer mixture is forced through a die which the design of has a great impact upon alignment. Die geometries can be engineered to give control over fiber orientation during extrusion. The bulk of this work has been accomplished at Monsanto.[6]

1.4.5 Miscellaneous Fiber Alignment Techniques

An alternative to using flow induced fiber alignment was developed at DuPont.

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[5] Soh, S.K., "Slurry Process for Preform Manufacture", Proc. 10th Annual ASM/ESD ACCE, Dearborn, 1994.
[6] Goettler, L.A., Leib, R.I., and Lambright, A.J., *Rubber Chem. Technology.*, 52, 838, 1979.

LDF[tm] stands for long-discontinuous fiber composites, which are made by stretching the continuous fiber until it breaks.[7] These fibers are attached by an adhesive to a tape-backing which also expands but as the fiber breaks, it retains the original fiber orientation. By assembling many plies of these tapes, one can manufacture a prepreg of the desired final part shape then impregnate it with matrix. Mechanical tests have shown that these composites confirm the fundamental theory for non-continuous fiber reinforcement describing critical fiber length necessary to achieve efficient average stress transfer in a composite. (This critical fiber length will be discussed in a later section) This process has been shown to be a cost-effective solution for manufacturing complex shaped parts of aerospace structures.

1.4.5.1 Electric Field Induced Alignment of Fibers in External Fields

Studying behavior of fibers in electric fields is not a new concept. The first published work detailing the effects electric fields have on aligning fibers was published by J. O Isard in the British Journal of Applied Physics in 1954. Later, in 1958 a researcher at Stanford University named Demetriades authored the theory for ellipsoidal alignment in electrical fields for neutrally buoyant fibers with essentially zero Reynold's number.[8] Thirteen years later, Monsanto obtained the first patent for aligned particles for producing composites. The next year, the Berol Corporation produced commercial equipment for the alignment of wood fibers for the production of particle board.[9,10]

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[7] Chang, I.. and Pratte, J.F., "LDF Thermoplastic Composites Technology", *Journal of Thermoplastic Composite Materials*, Vol 4, pp 227-252, 1991.

[8] Isard, J.O., "The Orientation of Fibers in Electric Field", *British Journal of Applied Physics*, Vol 6, p 176-179, 1955.

[9] Jander, M., "Industrial RTM – new developments in molding and performing technologies", Proc. 10th annual ASM/ESD ACCE, Dearborn, 1991.

However, since that time the limitations for developing a industrially viable process were identified as the time it takes to orient the fibers in the field and the rate at which the fibers could be deposited.

Two processes were developed that utilized electric fields to attempt alignment of short fibers. The first was by Talbot and Logan who desired to aligned wood fibers for the production of pencil stock. The wood fibers were dispersed in air and drawn down a tower under the influence of a vacuum and a parallel electric field. The fibers then were collected on a mesh weir for impregnation and consolidation. Talbot and Logan later teamed with Morrison and Knudson and extended this process for use with chopped glass fibers.[11,12] The oriented glass mats were manufactured using a complex arrangement of electrodes embedded in the bottom of the deposition tower. This process did not achieve high levels of orientation because they did not take advantage of the orientation state of the free falling fibers as they came under the influence of the electric field.

A second process for aligning fibers in electric fields was developed by Knoblach at the Massachusetts Institute of Technology.[13] His process produced uniaxially aligned mats of discontinuous graphite fibers. The fiber bundles were dispersed in a dielectric fluid and allowed to settle between a parallel plate electric field. Once the fiber bundles were collected on the deposition plate, the dielectric fluid had to be drained and filtered off of the fibers, which creates additional steps and also could leave a residue on the surface of the fibers. Because of these additional steps, this process was not received as being industrially viable.

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[10] Tucker, C.. and Advani, S.G., "Processing of Short Fiber Systems" in Flow and Rheology in Polymer Composites Manufacturing, Ed. S.G. Advani, Elsevier Science, 1994.

[11] Isard, J.O., "The Orientation of Fibers in Electric Field", *British Journal of Applied Physics*", Vol 6, p 176-179, 1955

[12] Talbot, J.W. and Logan, J.D., *U.S. Patent No. 4,113,812*, 1978.

1.5 Processing of Composites

For as many types of composites that exist, there are at least an equal amount of ways of processing them. Fibrous composites can be processed by such means as compression molding, thermoforming, injection molding, and reactive injection molding. Compression molding involves the forming of the composite under heat and pressure, where thermoforming is the shaping of complex geometries under heat and pressure. Compression molding and thermoforming will be discussed in more detail below. Injection molding can be employed for both thermosetting and thermoplastic matrices. For thermoplastics, the matrix and fiber mixture is heated up to form a liquid solution, which is then injected under pressure into a mold of which shape the final composite will have. When using thermosetting matrices, injection molding is usually referred to as reactive injection molding. Generally, the thermosetting matrix will be injected into a mold that contains a fiber 'pre-preg' (a fiber mat without matrix) along with a cross-linking agent to promote the reaction of the matrix. The finished composite is then unloaded from the mold.

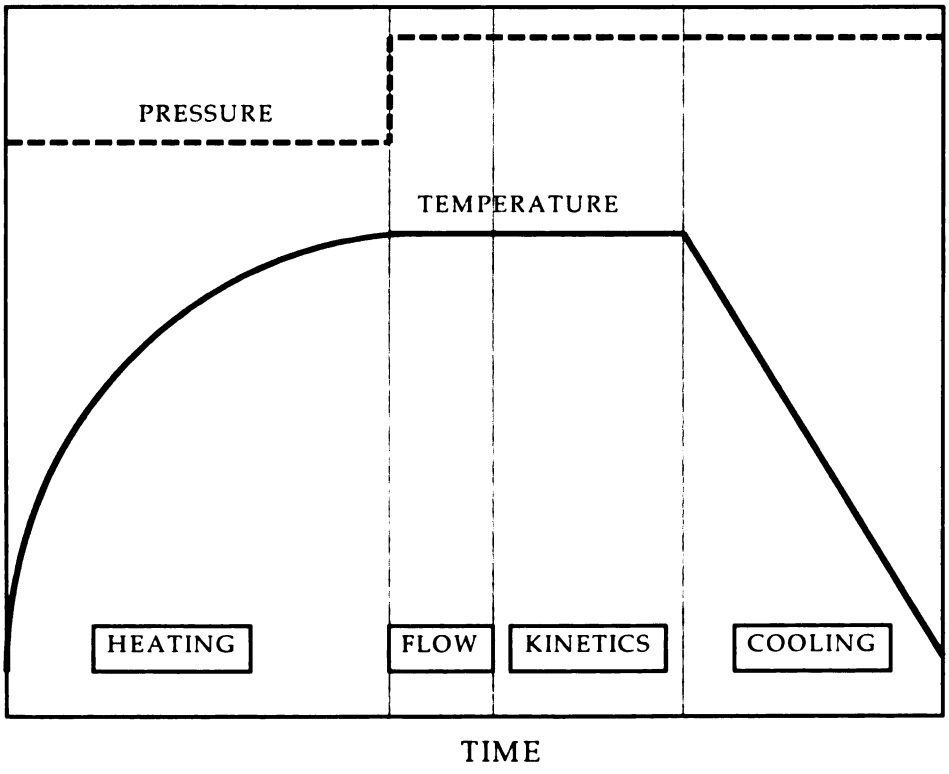
1.5.1 Compression Molding

Compression molding is presently the most technologically developed and versatile way to incorporate either continuous or random chopped fibers into a structural composite. One advantage of compression molding is that it is more rapid that labor

[13] Knoblach, G.M., "Using Electric Fields to Control Fiber Orientation During the Manufacturing of Composite Materials", *SAMPE Journal*, Vol. 25, No 6, p 9, 1989.

intensive hand lay-up techniques. However, if a random composite sheet is desired, some local orientation of fibers could occur during molding. This phenomenon arises from regions of high resin flow during the compression of the matrix at elevated temperatures. Typically, a 'charge' of material is loaded into the compression molding cavity and then the mold is closed and subjected to pressure and temperature profile similar to Figure 1.5.1.1. The finished part is then cooled well below the glass transition temperature, T_g, of the matrix and removed from the mold. It is important to note that the cooling rate of the material is an important parameter in the properties of the finished composite. For example, when compression molding polypropylene based composites, an exceptionally fast cooling rate is required in order to quench any crystallization of the matrix – if high tensile strength is desired.

Figure 1.5.1.1 Temperature and Pressure Profile of Compression Molding



1.5.2 Thermoforming

Thermoforming is a generic term encompassing many techniques for producing useful plastic shapes from a flat sheet. The first attempt at thermoforming was accomplished by the ancient Egyptians, who formed tortoise shell (keratin) in hot oil to manufacture food containers. Much progress has been made since that time, but the basic process has remained unchanged. Modern thermoforming began about 50 years ago with the developments in thermoplastic resin chemistry and the invention of the screw extruder and roll-fed sheet thermoformer. Thermoforming in the automotive industry is called 'stamping', which is why this topic was included in this research. An economic process for the forming of complex shaped parts would be a great boon to the industry.

1.6 Research Motivation

The controlled orientation of fibers in composites has been intensely studied at Michigan State University (MSU) over the past several years. *Vyakarnam, et al.*, developed a process at MSU that could control the orientation state of short discontinuous fibers.[14] This advancement opened the door for a processing technique that can be used for the large scale manufacturing of aligned discontinuous fiber composites for use in automotive, infra-structural, and durable goods applications. The ADF process served to advance the utilization of discontinuous fiber composites and bridge the property gap between continuous and discontinuous fiber composites.

[14] Vyakarnam, M.N., "Processing Discontinuous Fiber Polymer Composites: Fiber alignment using Electric Fields and Microstructure-property relationships", *PhD Dissertation*, MSU, 1996.

In order to increase the industrial acceptance of the ADF process, it was necessary to expand the number of composite systems that structural property data had been collected for. Also, it was important to expand the use of the ADF process to carbon fibers, which are used widely in structural applications already. However, the conductive nature of the carbon fiber had to be addressed when processing in an electric field – a modification of the ADF process had to be investigated.

1.7 Thesis Objectives and Outline

When the ADF process was developed, only one type of material was studied. In this work, results from three different types of reinforcing media, (polypropylene, polyethylene terephthalate, and nylon-12) and two different types of fibers (carbon and glass) were explored. The challenge of incorporating conductive (carbon) fibers was also undertaken. The ADF chamber had to be slightly modified to allow the use of the new fibers, details of the chamber modifications are addressed in chapter 3. In order to demonstrate commercial viability, a detailed economic analysis was performed and complex shaped ADF composites were manufactured. As will be discussed in chapter 4, the biggest advantage of using discontinuous fibers is the fact that they can be molded into complex shapes without micro-void formation.

This thesis is divided into six chapters. This first chapter has provided a background on composite materials and why this research was conducted. The second chapter will provide a detailed background on the different materials used in this research. Chapter 3 will delve deeply into the experimental equipment and processes used to process and

evaluate the ADF composite samples. Chapter 4 will examine the collected data and discuss the experimental results. The fifth chapter is the economic analysis report that was generated for the ADF process. Finally, the sixth chapter will be the conclusions gleaned from this research and the recommendation for future work.

2.1 Materials Introduction

This chapter will introduce all of the materials used in this research, how they are manufactured and then give a detailed description of all of the pertinent material properties. Three different polymer matrices were used, along with two different fiber types. Polyethylene terephthalate (PET), polypropylene (PP), and nylon-12 were used with carbon and glass discontinuous fibers of various lengths. These materials were each specifically chosen to represent the wide assortment of commercial matrices and fibers available in industry.

2.2 Matrices

The role of the matrix in a fiber-reinforced composite is to transfer stresses between the fibers, to provide a barrier against an adverse environment, and to protect the surface of the fibers from mechanical abrasion. The overall role of the matrix in tensile load carrying is quite small. However, the selection of the matrix has a major influence on the shear properties and compressive strength of the finished composite. The interface between the fiber and composite will also have an influence on the composite properties. Much work has been done at Michigan State University investigating the use of fiber coatings (called "sizings") that increase the adhesive coupling of matrix to fiber. Without this adhesion, the matrix and fiber are not ideally bonded and the composite properties will be starkly depressed.

There are two types of polymer matrices: thermosetting and thermoplastic. The main difference between thermosetting and thermoplastic matrices is that when heat is applied to a thermoplastic matrix, the material will soften and exhibit liquid-like behavior, ultimately becoming a liquid at temperatures above the melting point of the material. Thermosetting matrices react chemically to form cross-linked polymer chains that do not melt with the addition of heat. If enough heat is applied to a thermoset matrix, however, the material will burn instead of liquefying. The majority of commercial composites are made with thermosetting matrices, such as epoxies and polyesters, which provide superior properties at elevated operating temperatures. In the past ten years, significant progress has been made in synthesizing new thermoplastic polymers that can

Table 2.2 Properties of Polymer Matrices

Polymer	Type	T_g (°C)	T_m/HDT (°C)	Tensile Modulus (Gpa)	Tensile Strength (Mpa)
Nylon 6,6	TP-SC	78	260	1.6-3.5	82
Nylon 12	TP-SC	40	175	1.24	35
Polypropylene	TP-SC	-10	176	1.5	31
HDPE	TP-SC	-20	132	1.23	34
PET	TP-SC	73	255	2.8	81
PEEK	TP-SC	143	360-400	3.2	100
Polycarbonate	TP-A	150	130	2.4	65
Epoxy (Epon 1072)	TS	N/A	80	3.38	55-130
Polyester	TS	N/A	60-205	2.1-3.5	35-104
Vinyl Ester	TS	N/A	93-135	3-3.5	76

TP-Thermoplastic, SC – Semi-crystalline, A – Amorphous, TS – Thermoset

approach the elevated temperatures required for commercial use. That fact, coupled with the gaining public concern over the recyclability of materials, has led to greater industrial interest in the manufacturing of thermoplastic fiber reinforced plastics. Table 2.2 below lists common polymer matrices and their properties. Three thermoplastic matrices were investigated in this research: PET, polypropylene, and nylon-12. SEM's of all three matrices are shown on the following pages, Figures 2.2.1 – 2.2.3.

2.2.1 Nylon - 12

Nylon-12 is similar in structure to Nylon 6,6, but does not exhibit the same strength or stiffness as Nylon 6,6. Thus, its role in commercial composites is extremely limited. However, nylon-12 is used extensively in women's cosmetics. Because nylon-12 is easy to manufacture in small particle sizes, less than 10 microns, and is non-toxic, it is used as foundation in make-up. Because of the unique design of the Aligned Discontinuous Fiber process, the polymer matrix is required to be in powder form. The nylon-12 was supplied by Elf-Atochem under a trade-name of Orgasol 2000NX. The particle size distribution was centered around 10 microns. The density of the nylon-12 was 1.1 g/cm^3.

2.2.2 Polypropylene

Polypropylene has found many uses in the past several years. Employed in applications ranging from blow-molded plastic soft drink bottles to carpet fibers, the

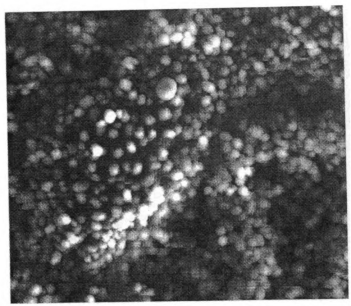


Figure 2.2.1 SEM of Nylon-12

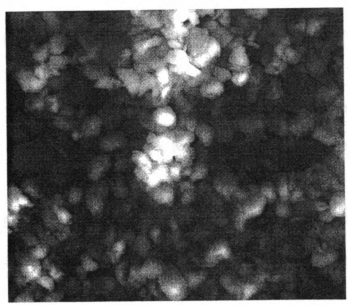


Figure 2.2.2 SEM of Polypropylene

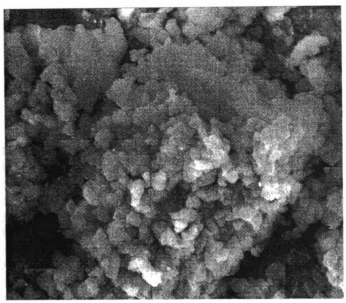


Figure 2.2.3 SEM of PET

recyclability and processing versatility of polypropylene has made it one of the most popular consumer product materials. The polypropylene (PP) used in this research was supplied by Micro Powders, Inc. (MPI) under a trade-name of Propylmatte 31. MPI sells the Propylmatte 31 as an additive to waxes for increased luster and appearance. The PP was supplied as a white powder with an average particle size of < 10 microns. As the SEM shows, the PP powder is not spherical (like the nylon-12), but cylindrical – an artifact of the grinding required to reduce the particle size. The density of the PP was 0.88 g/cm^3.

Figure 2.2.3.1 Particle analysis of ground PET

2.2.3 Polyethylene Terephthalate (PET)

PET, also called thermoplastic polyester, was supplied by Morton Thiokol under a trade name of Corvel Titanium. The PET arrived as a grayish powder (with 10% TiO_2 (by weight) added) with an average particle size of 150 microns. Because the powder was too coarse for use with the ADF process, it was necessary to have the powder further reduced in size by an outside grinding company. Union Process, based in Akron, Ohio, provided the particle reduction services produce PET particles with an average particle size of 10 microns. The PET (100 grams) was ground dry for 1 hour to develop a thin flake, then 100 mL isopropyl alcohol (IPA) and 1 mL Synthrapol KB (added to aid in the grinding) were added and processed wet for 3 hours. The wet slurry was air dried to remove the liquids added during grinding. A picture of the particle analysis is shown above in figure 2.2.3.1.

2.3 Glass Fibers

Glass fibers are unique materials that exhibit the familiar bulk glass properties of hardness, transparency, resistance to chemical attack, stability, and inertness, as well as fiber properties of strength, flexibility, lightness of weight, and processability. Glass is manufactured by fusing silicates with soda or with potash, lime, or other metallic oxides. The molten material is then quenched to prevent crystallization. Glass fibers are commercially made by melting together all of the raw materials, then formed into the glass fiber product. The molten glass is extruded through an orifice that is usually

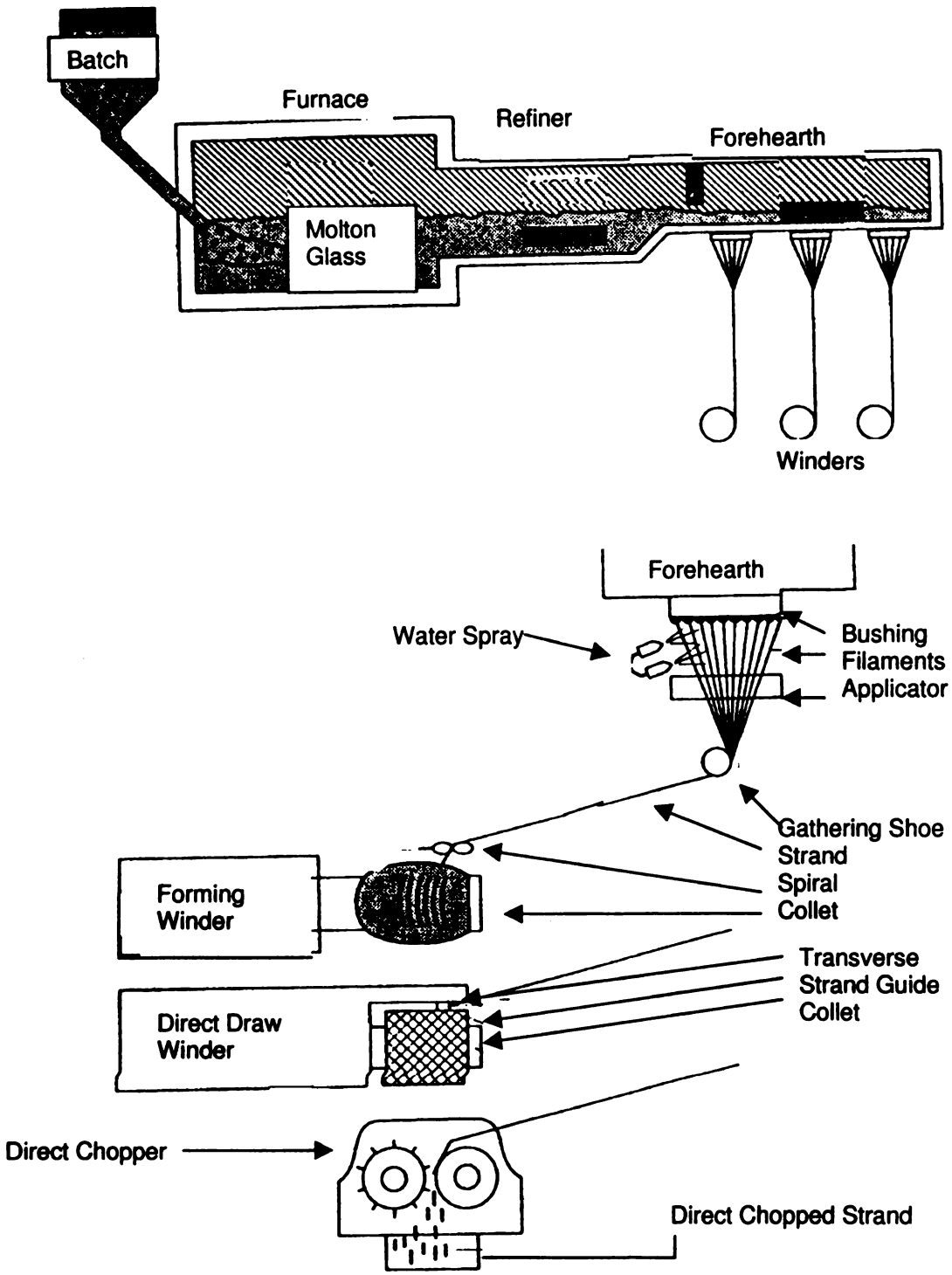


Figure 2.3.1 Manufacturing of Glass Fiber
(courtesy of Composites – Engineered Materials Handbook, ASM International, Ohio, 1987)

between 0.793 to 3.175 mm in diameter, and then rapidly drawing this glass to a fine diameter. These fine strands are then coated with a 'sizing' agent that serves to increase the adhesion of the fiber to the intended matrix, and to protect them from abrasion-related damage to the fiber surface. Finally the strands are gathered together to form the finished product. A detailed diagram of the glass fiber making process is depicted in Figure 2.3.1 above.

There are three main glass fiber types, E-Glass, S-Glass, and C-Glass. E-glass is a family of glasses with a calciumaluminoborosilicate composition and a maximum alkali content of 2.0%. E-glasses are used as a general purpose fiber when strength and high electrical resistivity are required. S-Glass has a magnesium aluminosilicate composition, which demonstrates high strength and is therefore used in applications where very high tensile strength is required. S-glass and S-2 glass fibers have the same glass composition but different coatings. C-Glass has a soda-lime-borosilicate composition that is used for its chemical stability in corrosive environments. Therefore it is often used in composites that contact or contain acidic materials.

The glass fibers used in this research were supplied by both Vetrotex-Certainteed and Owens Corning Fiberglass. All fibers were E-glass, with a density of 2.55 g/cm^3. The fibers were in bundle form, with an average fiber count of 200 fibers/bundle. They were chopped into lengths of 1/8", ¼", ½", and 1". Four different fiber sizings were investigated: polyethylene, nylon, polypropylene, and PET. The first two sized fibers were investigated by combining them with the nylon-12 to compare the effects of the sizing on the physical properties of the composite.

2.4 Carbon Fibers

Carbon fibers have been around since Thomas Edison invented the light bulb, but were not manufactured in great quantities until the late 1960's. At that time it was discovered that one could carbonize several fibrous materials into continuous fiber filaments with relatively low density and high Young's modulus of elasticity. This fiber could then be used much as glass fiber had been used: to provide a continuous reinforcement in various resin systems for the fabrication of structural components.

The two main precursors to carbon fibers are polyacrylonitrile (PAN) and pitch. PAN is the basis for the majority of carbon fibers commercially available today. These precursors can be thermally rearranged before thermal decomposition, which allows one to maintain the same filament configuration. This, along with the lack of surface defects, makes PAN based carbon fibers stronger than carbon fibers based on any other precursor. Pitch precursors are based on petroleum asphalt, coal tar, and polyvinyl chloride. Pitch based fibers are relatively low in cost. The most significant drawback of manufacturing pitch based fibers is the non-uniformity of fiber properties from batch to batch. This can be remedied somewhat by pre-mixing the pitch into a mesophase pitch.

There are five main process steps to making a carbon fiber: stabilization, carbonization, graphitization, surface treatment, and application of surface sizing. Stabilization is carried out at temperatures below 400 ^{o}C. The fibers are stressed to improve the orientation of the carbon chains. This pre-stress also improves the strength and modulus of the finished fibers. Carbonization is done at intermediate temperatures, from 800 – 1200 ^{o}C. The fibers are pyrolyzed in inert environments to reduce the

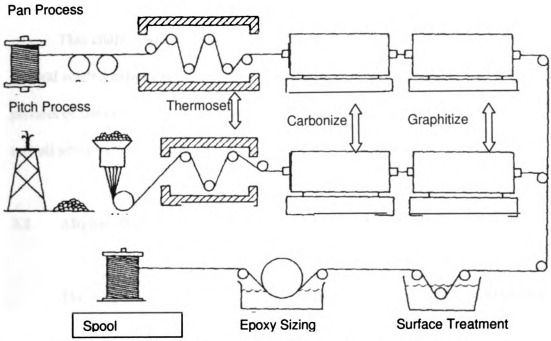


Figure 2.4.1 Schematic of Carbon Fiber Manufacturing Process
(courtesy of Composites – Engineered Materials Handbook, ASM International, Ohio, 1987)

impurity levels and increase crystallinity. Graphitization then heats the fibers to temperatures in excess of 2000 °C in an inert environment. This step also reduces the level of impurities and further increases the crystallinity. It is important to note that the higher the temperature, the higher the modulus of the fibers. Processing of the fiber is now complete, and the only remaining steps are treatment of the surface to increase adhesion, add surface groups to aid in bonding with the matrix that is to be reinforced, and protect the fiber surfaces during shipment and handling.

The carbon fibers used in this research are Hercules-Magnamite 3000 strand tow (3K Tow) PAN based AS-4 fibers. These fibers were not chopped and sized. Advanced Composites, L.L.C. of Mamoroneck, NY chopped and sized these fibers to lengths of ¼", ½", and 1". The carbon fiber sizings included nylon, polypropylene, and PET.

3.1 Experimental Equipment

This chapter will describe each piece of the experimental equipment in detail. Several related experiments used during this research will also be described, such as the process of the composite, the cutting of specimens, fiber and void volume calculations, and all other various sample preparations.

3.2 Aligned Discontinuous Fiber (ADF) Process Equipment

The Aligned Discontinuous Fiber Process (ADF) consists of four main pieces of equipment: the high voltage transformer, the alignment chamber, the vibratory feeder,

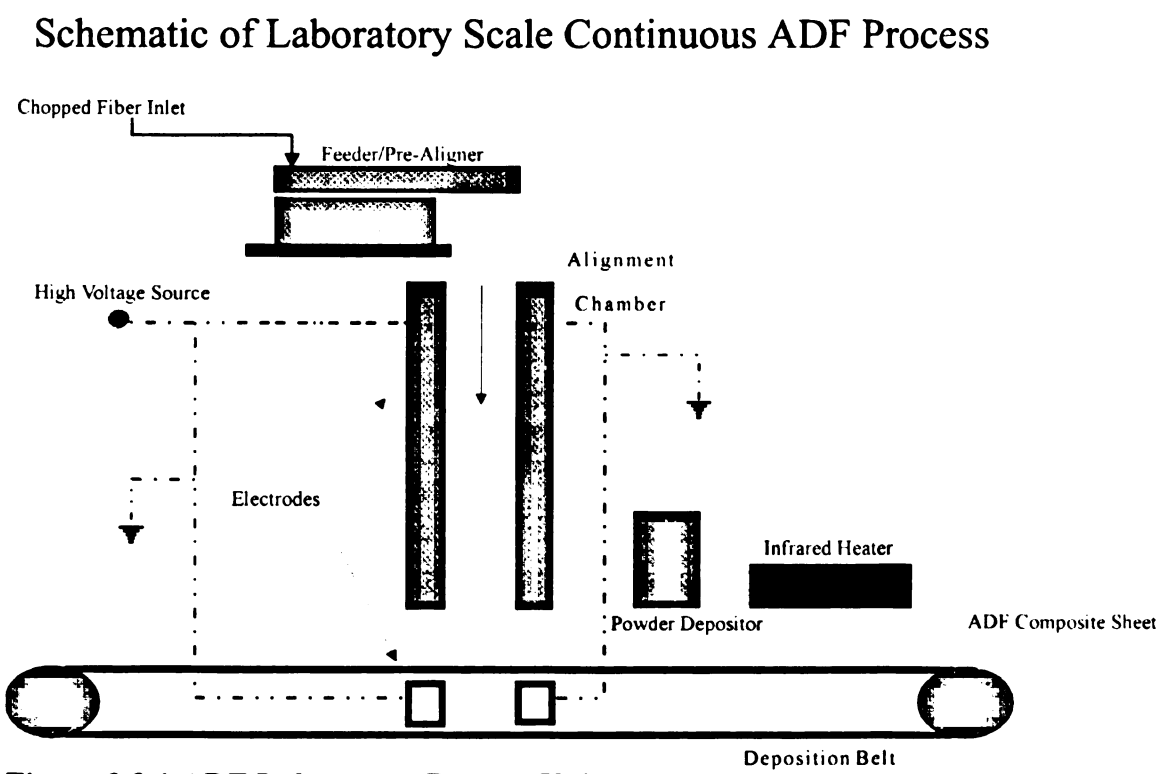


Figure 3.2.1 ADF Laboratory Process Unit

and the deposition platform. Several other minor pieces of equipment are also employed, such as the infrared heater, the multimeters, the variac, and the high voltage probe.

3.2.1 ADF Transformer

The key component to the ADF process is the Hipatronics RM25 transformer, which is rated at 25 kV (A.C.) at an input voltage of 110 V. There are two 'coils' of wiring inside the transformer, the first is a dense coil in the very center of the transformer which initiates the stepping down of the current, and the second much larger coil which surrounds the center coil and amplifies the voltage. The transformer is housed in a

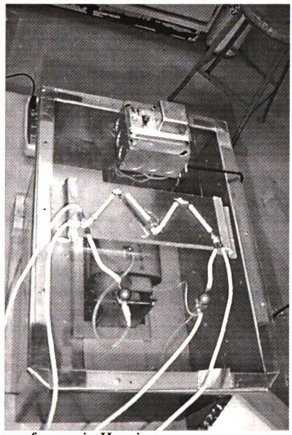


Figure 3.2.1.1 ADF Transformer in Housing

silicone oil bath to reduce arcing from the transformer to the surrounding air. Arcing would occur if the voltage within the transformer exceeded the breakdown voltage of the surrounding medium, in this case it would indeed exceed the breakdown voltage of air, but not of the silicone oil, which has a larger breakdown voltage. The output is fed to the two high voltage electrodes, which can be controlled two ways, first a variac is used to feed a controllable voltage to the transformer (maximum output of 30 KV), and second, the high voltage can be taken off of the transformer at 100%, 75%, 50%, and 25% of the output. This can also be used to vary the voltage of the upper and lower electrodes. It is important to note that this process is not inherently dangerous, the voltage is stepped up to a maximum of 30 KV, but the current is stepped down to ~15 mA.

3.2.2 Vibratory Feeder

The second piece of equipment in the ADF process is the vibratory feeder. The vibratory feeder sits atop the alignment chamber and serves two purposes in the ADF process, first it will feed the material to the alignment chamber at a constant rate, and second, it induces a pre-alignment to the fibers. The design of feeder chosen consisted of a modified vibratory FMC Syntron Magnetic feeder. The slight angle of inclination of the feeder, coupled with the vibratory motion of the chute, propelled the fibers towards the opening. The vibration also reduced entanglements and clumping of the fibers. It is important to note that the feeder did not align the fibers in such a way that the electrode only preserves the initial alignment. On the contrary, the feeder conveyed the fibers into the alignment chamber perpendicularly to the electric field.

It was found by Vyakarnam, *et al.*, that fibers having a random orientation state before entering the orientation chamber cannot be easily aligned by the electric field over small distances due to the tumbling nature of the fiber motion. This tumbling motion and random orientation state is also detrimental to the final degree of alignment because once the fiber hits the moving platform at the bottom of the orientation chamber, the fibers will rebound causing misalignment and disturbing the orientation of the neighboring fibers.

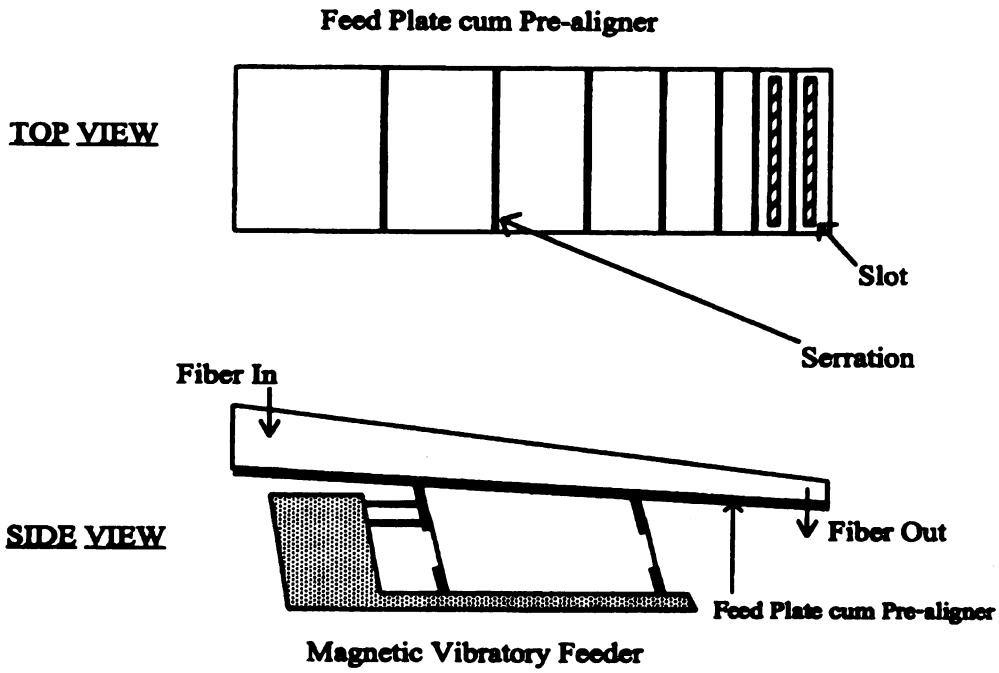


Figure 3.2.2.1 Vibratory Feeder Diagram
(reprinted with permission from Vyakarnam)

3.2.3 Alignment Chamber

The alignment chamber consists of two sets of planar copper electrodes housed between two plexi-glass supports. As the fibers fall into the chamber, they come under the influence of an aligning torque resulting from the electrical polarization. This torque induces a charge complex on the ends of the fiber, which serves the purpose of aligning the fiber with respect to the electrodes and keeping the fiber in the desired alignment until it reaches the deposition plate.

The upper set of electrodes is used to align the fibers, while the bottom set of electrodes is used to counteract the resulting electric field generated by the bottom edge of the electrode. Without this bottom set of electrodes, the electric field is disturbed resulting in fiber misalignment if operating in a continuous mode. These 'edge effects' occur at every boundary of an electrode, where the electric field is greatly concentrated, so much so that the bottom of the electrodes had to be fit with cylindrical caps to help dissipate the electric field. By analyzing the strength of the electric field in the main chamber, and at the uncapped edges, when 25 KV is applied to the high voltage electrode, the electric field intensity would be 500 KV/m (assuming a 5 cm gap width). At the edge, which has a thickness of ~0.0625 inches, the electrical field strength would be ~ 15,000 KV/m, well above the breakdown voltage in air. (3000 KV/m) Without these caps, the electric field would redirect itself to the closest grounding point (the grounded electrode) and cause arcing between the plates, or between the plates and a metal object nearby – which is an unsafe operating condition.

The minimum electrode height required for fiber alignment was discovered by Vyakarnam to be dependent upon two parameters: fiber alignment time, and fiber settling velocity. A simple correlation was determined:

Minimum Electrode Height = Alignment Time x Settling Velocity

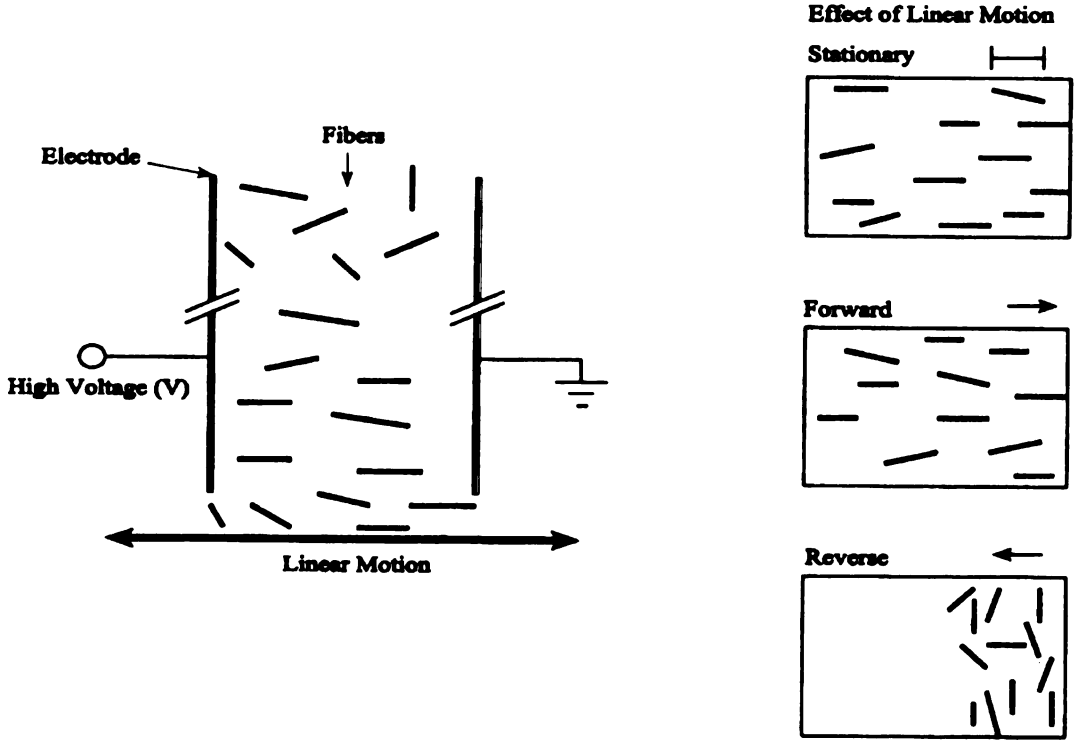


Figure 3.2.3.1 Effect of Non-Neutralizing Electrode
(reprinted with permission from Vyakarnam)

Based upon Vyakarnam's calculations, a height of 50 cm was determined adequate to align fibers varying in lengths from 1/8" to 1.25". The width of the alignment chamber

was designed to be 25 cm. Therefore, a composite part that was 20 cm in width could be made with the deposition plate that was chosen.

By changing the spacing of the electrodes, one can effectively control the strength of the electric field when keeping the input voltage constant. On the laboratory unit, the

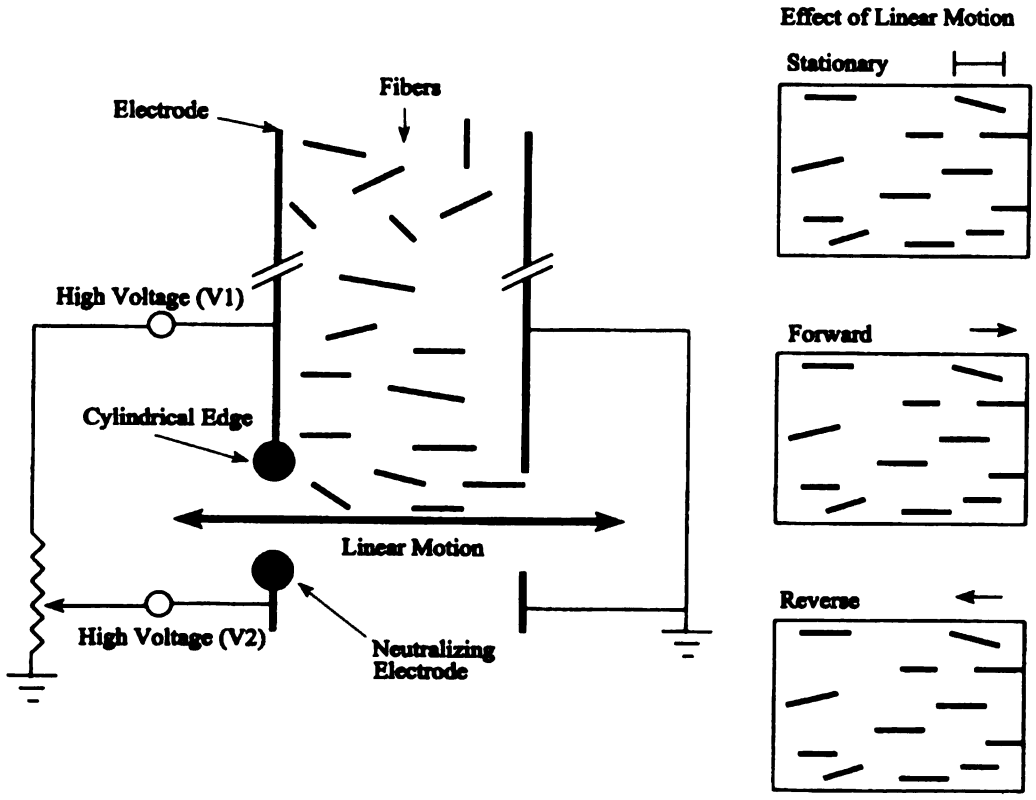


Figure 3.2.3.2 Addition of a Neutralizing Electrode
(reprinted with permission from Vyakarnam)

gap width can vary between 3 - 15 cm, which resulted in electric field strengths of ~800 KV/m to ~150 KV/m. The strength of the electric field was controlled by a variac unit, which allowed the operator to adjust the amount of electricity flowing into the

transformer. The scale on the unit was 0 – 125 % of the incoming current. A high voltage probe was used to measure the actual electric field strength. A Fluke 80K-40 probe was employed and connected to a Fluke 45 multi-meter to provide continuous monitoring of the electric field strength. Normal operating parameters for E-glass fibers is between 400-500 KV/m, and 60-80 KV/m for carbon fibers, depending on fiber length used.

3.2.4 Miscellaneous Equipment

Once the fibers have been aligned in the alignment chamber, they deposit onto a movable platform that is covered with a teflon release cloth and connected to a motorized slide. The linear slide was driven by a D.C. motor which was, in turn, connected to a speed regulator. The largest uncut ADF preform that could be made was 20 x 35 cm. The total distance that could be traversed by the slide was 30 inches, at speeds that could

Figure 3.2.4.1 Linear Slide motor and Speed Regulator

vary between 10 cm/min and 180 cm/min in both a forward and reverse direction. The platform can be used for both batch (by remaining stationary) and continuous operation (by moving back-and-forth under the alignment chamber). The aligned fibers then are coated with the polymer matrix in powder form, and sintered under an infrared heater. The particles partially melt and solidify bridging between adjacent fibers and then holding the fibers in place. As a consequence, the part can be handled without losing the desired alignment. A common kitchen strainer was used to distribute the matrix (in powder form) over the aligned fibers. The infrared heater consisted of two infrared heating elements nested into a metal support frame, which the polymer containing fiber mat passed underneath at a distance of < 1 inch. Because of the variation of melting points of the three polymers investigated in

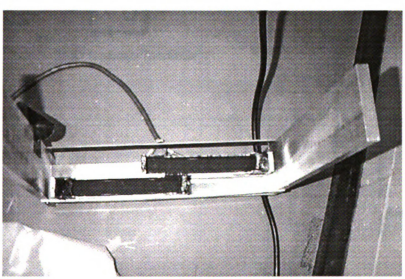


Figure 3.2.4.2 Infrared Heater

this work, PET, PP, and nylon-12, different sintering conditions were required. The nylon-12 and PP were the easiest to melt. Care was required in order not to burn the

polymer, which would have an adverse effect on the material properties. The PET was the highest melting point material and the infrared heater had to be placed very close to the material to start the melting process.

3.3 Electrical Wiring and Diagram of the ADF Process

The ADF Alignment chamber could be described as one giant capacitor, which is never allowed to discharge. One electrode is fully grounded, where the other electrode is

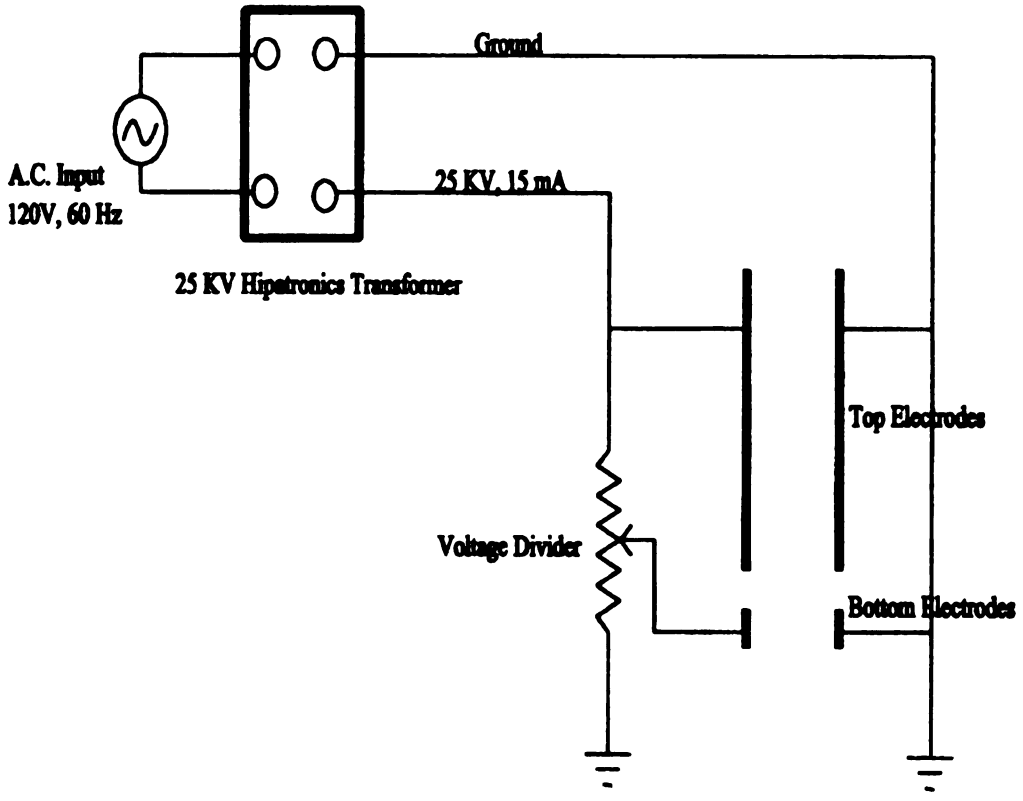


Figure 3.3.1 Electrical Wiring Diagram of the ADF Process
(reprinted with permission from Vyakarnam)

attached to the high voltage transformer. In the electrical diagram shown above in Figure 3.3.1, the electricity flow, once turned on and fully charged, theoretically is zero, since there is no direct flow from the high voltage plate to the ground. But in actuality, there is a small amount of current loss due to corona discharge from the entire surface of the high voltage plate. The total electrical duty is quite small, making this process quite attractive to industrial scale-up, which will be discussed in greater detail in Chapter 5.

3.4 ADF Preform Processing

The laboratory scale ADF Process Unit was not capable of manufacturing a full composite preform at once. Because the matrix was not deposited at the same time as the fibers, only a finite amount of fibers were able to be deposited before the bottom layer of aligned fibers would not be incorporated into the perform post sintering. Also, when a thick layer of fibers were deposited at once, the matrix powder was not able to penetrate the upper layers and fully secure the fiber alignment of the bottom most fibers. To alleviate this problem, several ADF 'layers' would be deposited and sintered separately. A finished composite would then be made by assembling a pre-determined amount of layers and molding them into the final composite shape. The molds used in this research were 5 inch x 7 inch rectangular "picture frame" molds that were 1 mm (0.03937 in) and 2 mm (0.07874 in) in thickness. To aid in the manufacturing of these 'layers', a simple spreadsheet program was written, called the ***ADF Composite Tool.*** One would input the desired fiber volume fraction, the matrix and fiber densities, the mold volume or dimensions, and the number of layers desired. The spreadsheet program would then calculate the amount of fiber and matrix required to make each layer, along with the total

weight of the finished composite. After sintering, the preform would then be trimmed on a paper cutter to roughly 5 x 7 inches. An additional 20 % of material was added to compensate for the amount of fiber and matrix that was deposited outside of the 5 x 7 inch preform area. An example of the ADF Composite Tool is shown below: (the

ADF Composite Tool

	Length	by	Width	by	Thickness	
Finished Composite Size	5	inches	7	inches	0.07874	inches
Composite Volume	2.7559	cubic inches				
# of Layers Desired	8					
Density of Matrix	1.1	g/cm3	PP			
Density of Fiber	2.55	g/cm3	Glass			
Fiber Volume Fraction	0.5					
Weight of Matrix	1.515745	grams				
Weight of Fiber	3.513773	grams				
Weight of Layer	5.029518	grams				
Total Weight of Composite	40.23614	grams				

example shown below was for a polypropylene and glass fiber composite) All composites manufactured in this research were calculated to be 50% (by volume) fiber.

3.4.1 ADF Preform Consolidation

Once all of the preform layers were manufactured, subsequent processing was required to form the finished composite. A Tetrahedron Compression Molding Press was employed in this research to accomplish this.

Using heat and pressure, compression molding heats the polymer matrix until it is in a liquid state, then the pressure is applied, which forces the molten polymer into the

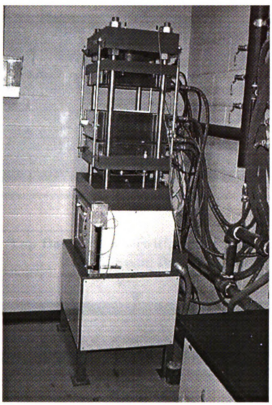


Figure 3.4.1 Tetrahedron Press

5" x 7" mold. The mold is then cooled while still under pressure.

All of this molding was done while a vacuum was drawn on the mold assembly. The top and bottom platens were lined with Teflon release cloth. The mold rested on the bottom platen and the preforms were stacked inside of the mold. A Teflon release cloth 'channel' was made from the bottom of the vacuum hose port that traveled under the top platen and rested against the mold. This channel allowed for air to be pumped out of the mold cavity once the pressure was applied to the platens. Below (in Figure 3.4.1.1) is a diagram of the mold assembly:

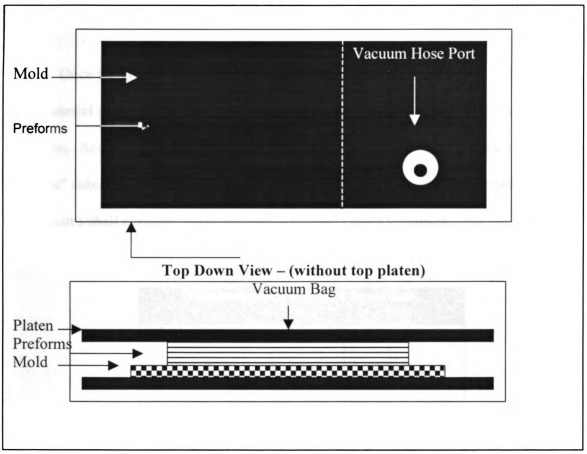


Figure 3.4.1.1 Vacuum Bag Assembly

Because of the pressure gradient emanating from the mold to the vacuum hose port, and the porous nature of the Teflon release cloth, a finite amount of molten matrix was 'pulled' out of the mold. To counteract this, an additional 20% of polymer powder was added to the preform stacks. This addition also drove out any remaining air pockets within the mold when pressure was applied, which would have led to voids in the finished product, which plagued this research until it was incorporated into the processing.

3.4.2 ADF Composite Sample Preparation

Once the ADF Composite was manufactured, it needed to be prepared for testing. The material properties that were of interest were the tensile strength and tensile modulus. According to ASTM D638-96 "Standard Test Method for Tensile Properties of Plastics" subsection 6.1.3 Reinforced Composites, "the test specimen for reinforced composites shall conform to the dimensions of the Type I specimen."

Dimensions of a Type I Specimen	
Width of Narrow Section	0.50 inches
Length of Narrow Section	2.25 inches
Width Overall	0.75 inches
Gage Length	2.0 inches

Table 3.4.2.1 Dimensions of a Type I Specimen

The 5" x 7" x 1 mm sample was cut into six 'slices' along the long axis of the composite, (which was along the aligned fiber direction) using the diamond tipped Felker saw. The Felker saw kept the sample cool during the cutting with a water spray, but this exposure to water did not have any detrimental effects on the final properties of the specimens because the samples were allowed to dry thoroughly before testing was done. These six slices then had to be shaped into the "dog bone" structure. A tool called the Tensilkut

was employed to accomplish this. The Tensilkut was quite similar to a carpenter's router, it was mounted upside down on a metal deck, and the abrasive die poked through a hole in the deck. A prior researcher in the group, Ed Drown, had several steel "molds" made for use with the Tensilkut. One of them was designed to the Type I specifications, which was then used for this research. These molds would hold one of the ADF slices and protect the interior section of the piece, while grinding away the unwanted center sections. After this mechanical grinding away of the slice to make the narrow section, the microstructure of the edges was quite disturbed. Various grades of sand paper were used to smoothen the sidewalls of the dog bone. An added benefit of the sand paper was that the frictional heat that was generated also helped anneal the damaged matrix. The sanded dog bone structures were then stored at room temperature for 48 hours prior to testing.

One important difference between the technique used in this research and that used prior by Murty Vyakarnam, was that his samples were cut using a high-speed laser on the MSU campus. Due to lack of availability of the equipment, it was decided to use the Tensilkut for the processing of the samples in this research. It is important to note that the microstructure that is created on the sidewalls of the samples is quite different when these techniques are compared. Figure 3.4.2.1 contains six SEM's of different sample preparations. The first row of two show the "as cut" by the Tensilkut, these show extensive surface damage from the mechanical abrasion. The second row of two are Vyakarnam's samples that were cut using the MSU High-Speed Laser. These samples

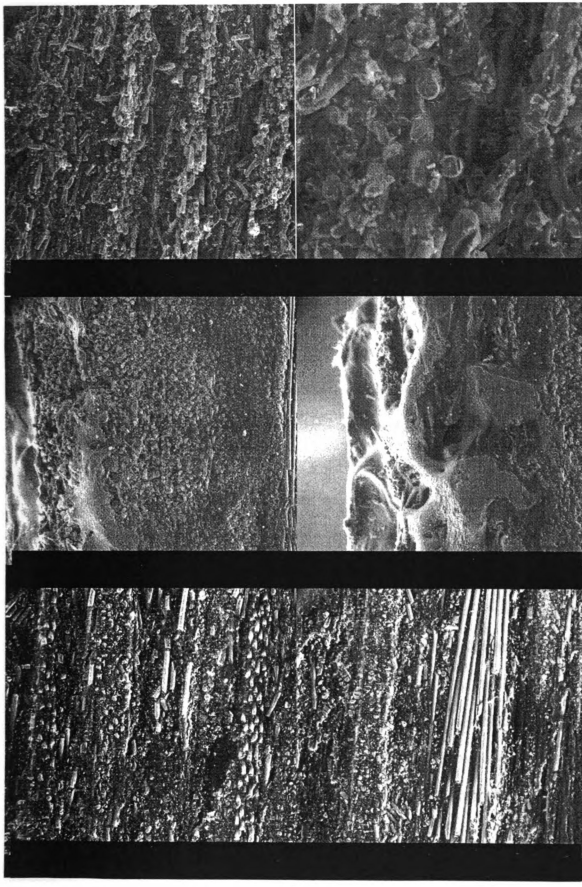


Figure 3.4.2.1 SEM Micrographs of the Sample Specimens

show considerable annealing of the matrix surface, with small amount of fibrous residue with sand paper. Some of the surface damage was annealed by the frictional heat, and there is less fibrous residue. The strength difference between the composites made by Vyakarnam and those made in this research can be explained by this coupon preparation difference.

3.4.3 Mechanical Testing of ADF Composite Samples

Testing of the properly conditioned samples was accomplished using an MTS in the Composite Materials and Structures Center Laboratory. The MTS was employed in tension mode using the laser extensometer to measure the strain on the sample. The laser extensometer used reflective tape placed on the gage section of the sample coupon to reflect the laser signal back into the machine to measure how far the sample had been 'stretched' before failure. This tape was applied to the sample using a special 'mold' that held the sample coupon, while tape was applied at specific locations on the gage area of the coupon. A straining rate of 0.02" per minute was used for sample testing, and the 1000 lb load cell was used for this testing. Plots of the stress versus strain were made by the MTS computer. A representative sample of this plot will be presented in Chapter 5. The tensile strength was computed by dividing the load at failure by the cross sectional area of the sample. Tensile modulus was computed by the MTS computer by measuring

the slope of the stress/strain curve generated by the machine. The slope up until failure was used for this calculation.

For mechanical property comparison purposes, the testing technique used in this research was copied directly from the prior researcher.

3.4.4 Void and Fiber Fraction Analysis of ADF Composites

Two of the critical parameters when manufacturing composites are void fraction and fiber volume fraction. Anything over 3-4% void in a composite will cause poor mechanical performance, as the stress will not be uniformly transferred across the composite. In the case of fiber volume fraction, it is important to know the value of this parameter if any accurate prediction of the mechanical properties is required. All composites used in this research were targeted to be ~50 % +/- 2% fiber by volume.

The methods used to compute these values were similar. In the case of fiber volume fraction, the sample was weighed and placed in a furnace to burn the matrix off. The glass fiber composites were burned off at a temperature of 600 ^{o}C for 4 hours. The fibers were weighed and the void fraction was calculated by determining how much of the sample (by volume) was matrix. Of course, the sizing of the fiber bundles was also burned off, but for the purposes of this research, the weight/volume of the sizing was assumed to be negligible. The carbon based composites had to be handled with more care than the glass – because the carbon fibers would burn off with the matrix if the furnace temperature was too high. Since glass fibers are thermally stable in an oxygen containing atmosphere to temperatures in excess of 1500 ^{o}C, this was not a problem. The

oxidation of the carbon fiber could have been avoided by using an inert atmosphere, such as N_2, but matrix combustion rate would have been affected as well. A furnace temperature of 400 °C was then used for 6 hours to completely remove the matrix, while keeping the carbon fibers intact.

The void fraction was computed similarly to the fiber volume fraction, since the fiber volume fraction was needed to compute the void fraction. The void fraction samples were carefully cut to be 0.50" by 0.50" squares with sanded edges to make the square as uniform as possible. The squares were then measured and weighed. The volume of the square was then calculated. An additional volume measurement was conducted in which the sample was immersed in a 100 mL graduated cylinder. This measurement was not as accurate as the volume calculated by the square's geometric measurements, but it served as a 'sanity' check on the volume. The samples were then loaded into the oven and the matrix was baked off just as with the fiber volume fraction. Once the fiber volume fraction was known, the computed composite volume was measured and compared to the composite volume by the geometric measurements. The

Void Fraction Analysis	
Aligned 0.25"	6%
Random 0.25"	11%
Aligned 0.50"	10%
Random 0.50"	9%

Table 3.4.4.1 Void Fraction of Early ADF Composite Samples

difference in these two measurements was the void volume fraction of the composite. Voids were a large problem in the beginning of this research. The early ADF composites that were made contained an unacceptable amount of voids.

It was determined that the voids were being formed during the compression molding step, specifically when the matrix was molten and pressure applied. Under a microscope, it was determined that the void fraction was weighted more heavily on the top surface of the composite. It was theorized that since the Teflon release cloth was porous, the molten matrix would be pushed out of the bottom of the mold when the pressure was applied. To counteract this effect, an additional 20 % (by weight) of matrix was added to the top of the preform stack in the mold prior to vacuum bag assembly. This additional matrix would compensate for the loss through the Teflon release cloth, and also mechanically force the voids out of the composite during consolidation.

3.4.5 Manufacturing of the Radii of Curvature ADF Composites

The third goal of this research was to manufacture ADF composites to test the 'drapeability' of the ADF composites. These curved composite were made using special molds that were manufactured by the Michigan State University Metal Shop. Three different radii of curvature molds were constructed, 0. 125", 0.25", and 0.50". A diagram of the molds is shown below:

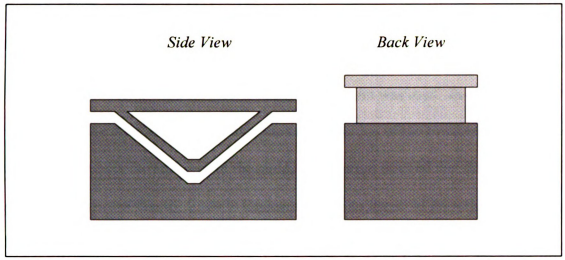


Figure 3.4.5.1 Radii of Curvature Molds

The ADF preforms would be hand placed on the bottom piece of the mold, centered with respect to the radius of curvature. The top piece would then be placed on top of the preforms and inserted in the vacuum bag. Careful sample preparation was required to minimize shifting of the mold pieces during vacuum bag assembly and positioning of the sample inside of the tetrahedral press. A Teflon release cloth sheet was placed between the metal parts and ADF preforms.

The radii of curvature of the molds were calculated by using 25% of the circumference of the appropriately sized circle. Additional length was added to each half of the composite to make the sides 7". This method is illustrated below:

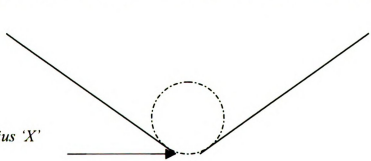


Figure 3.4.5.2 Design of Radii of Curvature Molds

For design consideration, a composite thickness of 1 mm was used. In order to keep the top piece of the mold from compacting the curved portion of the composite, metal 'stops' were placed under the top piece of the mold. These stops ensured that the very tip of the curved portion was exactly 1 mm in thickness. Besides the mold preparation difference, the processing of the radii of curvature composites was identical to that of the flat composites. Sample coupon preparation was slightly different, since these samples were simply cut with the Felker saw into 1" strips, as there is no ASTM method for testing of these type of coupons. Testing of the coupons was conducted on the MTS machine in compression. Instead of grips, the flat disks were used top and bottom, with the curved coupon centered between the two plates. These plates were then compressed until failure of the sample.

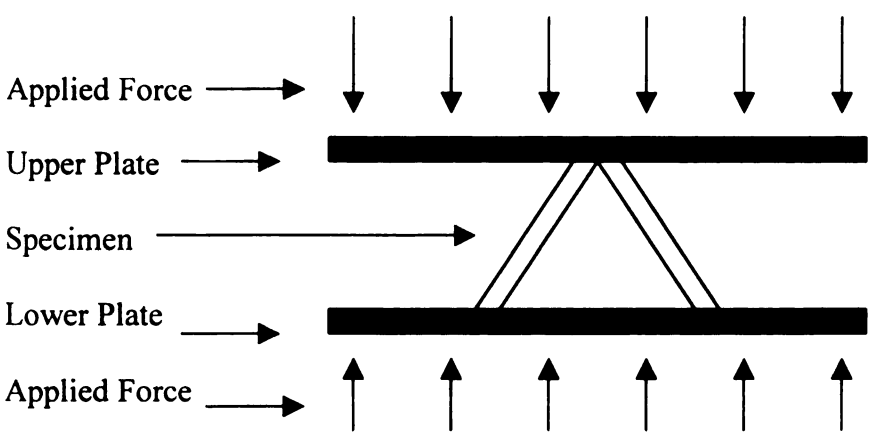


Figure 3.4.5.3 Curved Specimen Compression Testing

4.1 Results and Discussion

This chapter will delve into the results obtained during this research, touching on four main areas: glass fiber, carbon fiber, drapeability, and the corresponding Halpin-Tsai predictions.

$$\frac{P}{P_m} = \frac{1 + \varsigma \eta V_f}{1 - \eta V_f}$$

where:

$$\eta = \frac{\frac{P_f}{P_m} - 1}{\frac{P_f}{P_m} + \varsigma}$$

$$\zeta = 2(a/b)$$

a and b are defined as the two dimensional parameters associated with a reinforcement, most commonly length and width. The other parameters include the fiber volume fraction (V_f) and the material and fiber property being estimated. (P_f and P_m) The key parameters that will be presented and discussed are tensile strength, tensile modulus, and fiber orientation.

The first section will describe the results from the three glass fiber systems studied, polypropylene, nylon-12, and polyester terephthalate. Nylon-12 was evaluated as an ADF matrix based on prior research. The nylon systems served as a direct comparison to the prior results (i.e. a sanity check that the ADF was functioning properly) and also as a baseline to compare properties with the carbon fiber based systems. The other two matrices used in this research were chosen for

several factors, which included: high mechanical properties and widespread commercial availability.

The second section will show the results from the corresponding carbon fiber systems, the third section will detail the amount of orientation that was imparted to the fibers during ADF processing, the fourth section will describe the Halpin-Tsai relationships and how well the equations predicted the properties of the ADF composites, and the final section will talk about the drapeability results from the glass fiber system.

Below is a table listing the physical parameters of interest of the pure materials used in this research. The first three listings are the matrices, the final two are the fibers.

Polymer/ Fiber	Type	T_g (°C)	T_m/HDT (°C)	Tensile Modulus (Mpsi)	Tensile Strength (Psi)
Nylon 12	TP-SC	40	175	.179	5100
Polypropylene	TP-SC	-10	176	.217	4500
PET	TP-SC	73	255	.406	11750
E-Glass	N/A	N/A	N/A	10.5	500000
Carbon	N/A	N/A	N/A	30	480000

Table 4.1.1 Physical Properties of Fibers/Matrices Used

4.2 Glass Fiber Systems

A total of four glass fiber systems were investigated – two nylon-12, PP, and PET. The two nylon-12 systems were nylon sized fibers and polyester (PE) sized

fibers. For the PP and PET systems, they were sized with PP and PET compatible materials. It is important to note that the different sizings for the nylon-12 systems were investigated to determine how much of an influence on mechanical properties the sizing agent had. Below are the results of the two nylon systems.

In the beginning of this research, the amount of alignment imparted to the fibers was minimal. In order to determine what process parameter had changed since the prior researcher had worked at Michigan State, several detailed equipment checks were conducted. Most of the initial checks revolved around the transformer and voltage meters. However, one process variable that was thought to be insignificant was the location of the apparatus. When the CMSC moved from the Research Complex to it's new home in the Engineering Building, the experimental conditions changed. The new building's air conditioning facilities maintained the ambient air at a much drier level. This lack of humidity was found to have a large processing effect. In order to combat the lack of humidity, all of the glass fibers were kept in a humidity chamber were the relative humidity was kept at a level of around ~89-92%. It was determined that the sizing agent on the fibers absorbed the water vapor, which greatly increased the electrical polarization and aided alignment in the electric field. However, it was still quite difficult to maintain the electric field at a strength of >450 KV/m for sustained periods due to the dryness of the laboratory air. In a production facility, it is imperative that the raw materials and electrodes be housed in a controlled environment to provide process stability.

It was thought that the fiber sizing may have an influence on the mechanical properties of a finished composite. A hydrophilic sizing may aid fiber alignment more than a hydrophobic one, and the effect of humidity on the ADF process was studied. To quantify the effect the sizing had on the final properties, the two nylon-12 systems were directly compared. For the random fiber systems, the effect was much greater on the tensile strength, as a ~30% tensile strength improvement (averaged over the three fiber lengths) was noticed, where the tensile modulus only increased ~4%. (the small increase for the modulus is most likely attributed to the conflict of results between the random 0.25" fiber PE sized system – and the random 0.5" PE sized system) On the aligned systems, a 14% increase was seen for the tensile strength and a ~7% increase was observed for the tensile modulus. The results are briefly summarized in Table 4.2.1. The results suggest that the fiber sizing does not have a large impact on the mechanical properties of the composite. Although the random results show a large increase, due to the inherent nature of the random composite, the result most likely is not significant. However, the 14% increase in tensile strength and the 7% increase in tensile modulus for the aligned sample may be significant.

Fiber Length	Strength		Modulus	
	Aligned	Random	Aligned	Random
0.25"(*)	21%	37%	3.3%	-14%
0.5"	7.6%	38%	6.4%	17%
1"	14 %	13%	10%	9.3%
Average	***14%***	***29%***	***6.7%***	***4.1%***

Table 4.2.1 Percentage Improvement in Mechanical Properties due to Fiber Sizing

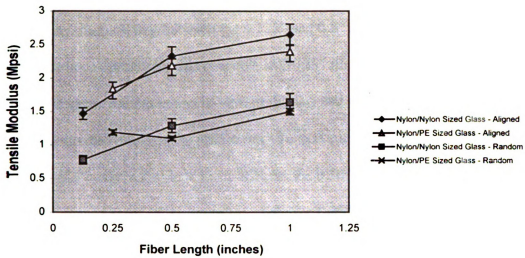


Figure 4.2.1 Effect of Fiber Sizing on Tensile Modulus

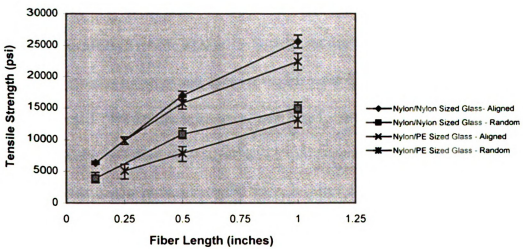


Figure 4.2.2 Effect of Fiber Sizing on Tensile Strength

For direct comparison purposes, the 0.25" data points for the nylon sized fibers were estimated by interpolating between the 0.125" and 0.5" properties. (0.25" nylon sized fibers were not available for this research, so 0.125" fibers were substituted.)

The improvement in mechanical properties that was due to the fiber alignment was similar to the results of the prior work. Tensile strength for the nylon-sized fibers was increased by 64% for the 0.125", 56% for the 0.50", and 70% for the 1" fibers. For the PE-sized fibers, 94% improvement for the 0.25", 100% for the 0.50", and 70% for the 1" fibers. The tensile modulus was increased 88% for the 0.125" nylon sized system, 80% for the 0.50", and 61% for the 1" system. For the PE sized system, the increase was 53% for the 0.25", 99% for the 0.5", and 60% for the 1" system. These results indicate that a high degree of alignment was achieved, which will be described in more detail in section three.

The average tensile strength improvement due to ADF processing was 63% for the nylon sized system and 88% for the PE sized system. The tensile modulus was increased 76% for the nylon sized and 70% for the PE sized system. Unfortunately, no statistically significant improvement due to fiber sizing compatibility can be proven from the above results. Although it can be shown that the overall mechanical properties are increased for the nylon compatible system, the average tensile strength improvement for the PE sized system is 25% more than the nylon-sized system. This result indicates that the greatest effect on mechanical properties is the degree of alignment. Fiber sizing compatibility, although desirable when designing a composite part for optimum performance, has a secondary effect on

the overall mechanical properties. Thus, a small increase in fiber alignment would have more of an effect on the mechanical properties compared to a large increase in compatibility between fiber and matrix.

When comparing results from this research to the work of Vyakarnam, *et al.*, it is important to note that there is a 10% different in fiber volume fraction. This research used 50% fiber, where 40% fiber was used for the prior work. Even with less fiber, Vyakarnam was reporting mechanical properties that exceeded the results in this research. Of the two mechanical properties, the largest difference was observed between the tensile strength of Vyakarnam's samples and those used in this research, the difference between the tensile moduli was markedly less. As was discussed in Chapter 3, the reason for this disparity was determined to be the microstructure on the edge of the finished test coupons – laser cut vs. mechanically grinded.

The polypropylene sized fibers used in this research were supplied by Vetrotex Certainteed (who supplied all of the E-glass fibers). One of the problems encountered working with these fibers was that the sizing agent was not as structurally sound as the nylon and PET compatible sizings. Below are two optical micrographs comparing nylon-sized fiber bundles with representative polypropylene fiber bundles. (the nylon sized fiber bundle is on top, the PP sized bundle is on the bottom) One can immediately notice that some of the bundles have broken down into individual filaments. This made the processing of these fibers through the vibratory feeder quite tedious and time consuming. Another problem encountered with the PP

system was the matrix. The PP supplied by Micron Powders, Inc., had a molecular weight (weight average) of ~3,000 g/mol. Because of this low molecular weight (and

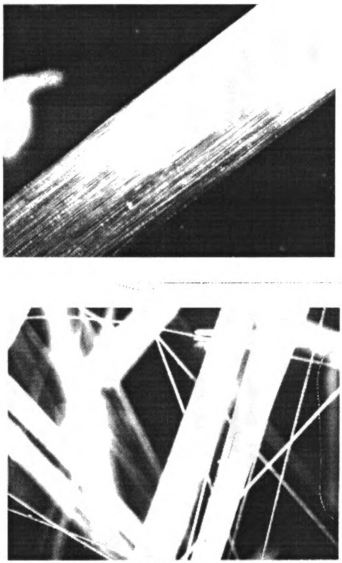


Figure 4.2.3 Optical Micrograph of Polypropylene and Nylon Sized Fibers

Nylon/Nylon E Glass
Nylon-Sized

	Aligned		Random	
	Modulus (Mpsi)	*Strength (psi)*	*Modulus (Mpsi)*	*Strength (psi)*
0.125"	1.47	6400	0.78	3900
0.25"				
0.50"	2.33	17000	1.29	10900
1"	2.65	25600	1.64	15000

Nylon/Nylon E-Glass
PE-Sized

	Aligned		Random	
	Modulus (Mpsi)	*Strength (psi)*	*Modulus (Mpsi)*	*Strength (psi)*
0.125"				
0.25"	1.84	9900	1.2	5100
0.50"	2.19	15800	1.1	7900
1"	2.4	22400	1.5	13200

PET/Glass

	Aligned		Random	
	Modulus	*Strength*	*Modulus*	*Strength*
0.25"	2.83	16600	1.84	11900
0.50"	3.27	22500	2.00	13000
1"	4.33	30100	2.35	16000

Polypropylene/Glass

	Aligned		Random	
	Modulus	*Strength*	*Modulus*	*Strength*
0.25"	1.65	2100	0.76	1200
0.50"	2.48	3900	0.95	2000
1"	3.4	5500	1.41	3700

Note: PET samples have 10% Titanium Dioxide by weight as filler

All samples at 50% volume fiber +/- 3%

Table 4.2.2 Glass Fiber System Results

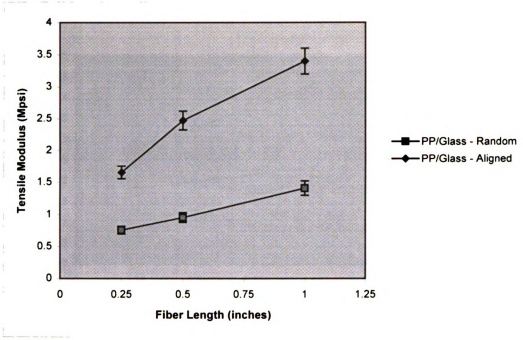


Figure 4.2.4 Tensile Modulus Results from the PP/Glass Systems

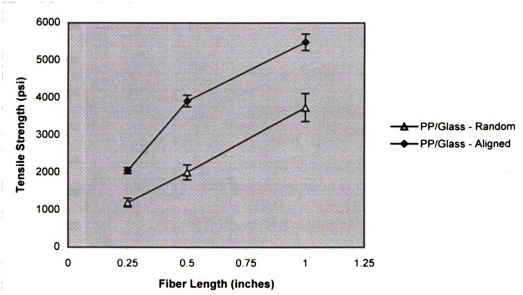


Figure 4.2.5 Tensile Strength Results from the PP/Glass Systems

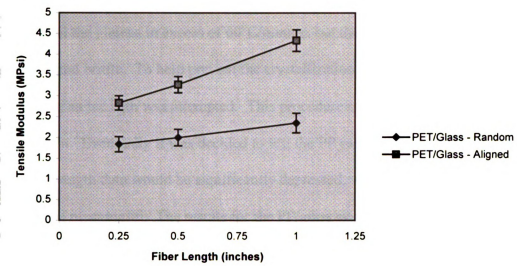


Figure 4.2.6 Tensile Modulus Results from the PET/Glass Systems

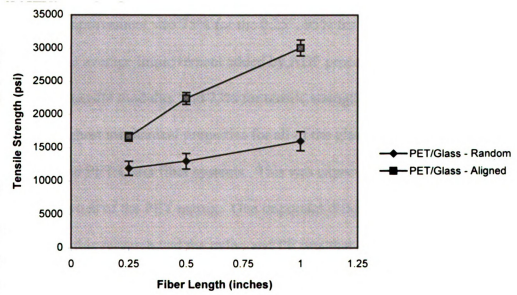


Figure 4.2.7 Tensile Strength Results from the PET/Glass Systems

therefore short polymer chain length), the final consolidation of the composite part without large-scale crystallization was impossible. The Tetrahedral press had the ability to cool the platens in excess of 60°C/minute, but the PP would still become crystallized and brittle. To help prevent the crystallization from occurring, quenching the sample in an ice bath was attempted. This procedure still did not prevent the crystallization. Eventually it was decided to test the PP samples as is, realizing that all tensile strength data would be significantly depressed, while tensile modulus data would still be meaningful. The results for the PP/glass system are presented in Figures 4.2.4, 4.2.5 and Table 4.2.2, along with the results for the rest of the glass systems.

When comparing the alignment effect on the tensile modulus, the 0.25" system showed a 117% improvement, the 0.50" system showed a 161% improvement, and the 1" system showed a 141% improvement. For the tensile strength, the improvement was 75% for the 0.25", 95% for the 0.50", and 49% for the 1" system. The average improvement added by ADF processing for the PP system was 140% for tensile modulus, and 73% for tensile strength.

The highest mechanical properties for all of the glass fiber systems was exhibited by the PET/Glass fiber systems. This was expected, due to the higher intrinsic properties of the PET matrix. One important difference between the PET matrix used in this research and the nylon and PP was that the PET contained 10% Titanium Dioxide (by weight) as a filler. Normally the role of fillers in matrices is to reduce the amount of matrix in a given sample, therefore reducing the raw material cost to the manufacturer. They also are used as a plasticizing material for

exceptionally rigid materials. Typically the mechanical properties of the filled matrix will not be as large as that of the unfilled matrix. However, the mechanical properties of the PET system used in this research do not appear to be affected in that fashion, as they were higher than what was expected. A possible explanation might be that the compression molding and/or the sample preparation caused a beneficial alteration of the microstructure. The titanium dioxide might also have concentrated around the fiber and acted as a stress/load conduit that more efficiently transmitted the load. Either way, the mechanical properties were higher than expected but were consistent across the sample set and no obvious explanation exists at this time.

When comparing the alignment effect on the tensile modulus, the 0.25" system showed a 54% improvement, the 0.5" system showed a 64% improvement, and the 1" system showed an 84% improvement. For the tensile strength, the improvement was 39% for the 0.25", 73% for the 0.50", and 88% for the 1" system. The average improvement added by ADF processing to the PET system was 67% for tensile modulus, and 67% for tensile strength.

For all of the glass fiber systems, the ADF processing added at least a 50% improvement in mechanical properties. The results also indicate that this improvement increases with increasing fiber length. There are several possible reasons for this. First, as will be shown in section 4.4, the longer fibers were aligned to a greater degree. When processing the smaller fibers, they would be aligned as they fall, but when they hit the deposition platform there would be some bouncing of fibers, thus causing disruption of the alignment. The longer fibers would also exhibit this bouncing effect, but to a lesser degree). As the fiber length increased, the

individual fiber itself became more ellipsoidal than cylindrical. This change in cross-sectional geometry had a profound impact on the fluid mechanics. As a result, the longer fibers were more 'buoyant' during the fall through the alignment chamber and thus hit the deposition platform with less momentum per unit weight, causing less bouncing and 'dis'-alignment. The other possible explanation would be an argument for the minimum effective fiber length – the longer fibers would theoretically act more like continuous fibers (on a micro-mechanical scale) than the 0.25" and 0.5" fibers.

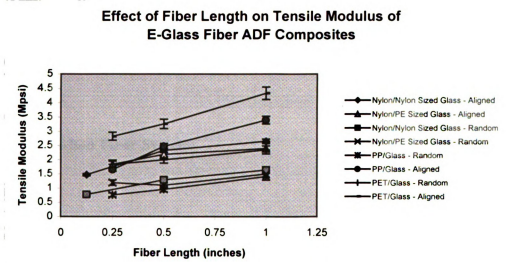


Figure 4.2.8 Glass Fiber Systems Tensile Modulus Comparison

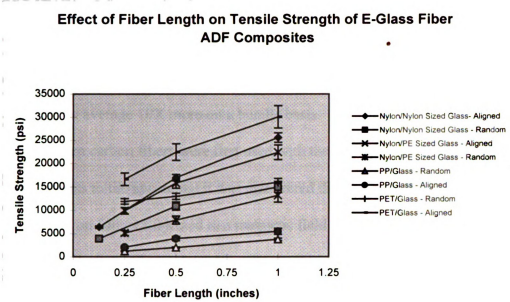


Figure 4.2.9 Glass Fiber Systems Tensile Strength Comparison

4.3 Carbon Fiber Systems

The primary motivation of this research was to develop a process in which to align electrically conductive fibers, such as carbon fibers. In theory, the conductive fiber would act as an electrical 'bridge' between the electrodes, thus shortening the effective electrode spacing. Since the effective distance between the electrodes was shortened, the localized electric field strength would be increased, thus causing more electrical discharges (arcing) between the electrodes.

The carbon fibers used in this research were obtained from Hexcel. These fibers were PAN based-AS4 supplied in a 3K tow (continuous). The fibers were sent out for sizing and chopping to Composite Materials, LLC, based in Mamoraneck,

NY. The fibers were coated with a nylon-12 compatible sizing. It is important to note that where the glass fibers were ellipsoidal in cross-section, the carbon fibers were perfectly circular. The carbon fibers were also much more dense as a bundle – weighing on average 10X more on a bundle basis.

When carbon fibers were first used with the ADF process (before any modifications to the apparatus) it was discovered that the carbon fibers would line up much like iron filings subjected to a magnetic field when they were landed on the deposition platform. There was also considerable arcing between the plates as the fibers fell. However, when arcing occurred during processing of the glass fibers, the fuse in the variac power supply would be blown. With the carbon fibers, the fuse did not blow but instead the electrical field strength was decreased. This drainage of the electrical field continued until the local instability corrected itself, i.e. the fiber fell onto the deposition plate. Another phenomenon observed was that when the carbon fibers passed underneath the electrodes, (when processing in continuous mode) the field had a strong enough 'pull' on the fibers to lift one of the ends up to the electrode. This effect, combined with the fact that the fibers would form 'chains' lining up between the electrodes, proved to be a difficult problem. Once the chain was completed, with fibers spanning from the bottom of one electrode, across the deposition platform, and up to the opposite electrode, the field would arc and actually burn the Teflon release cloth underneath. These initial experiments indicated that to be successful:

1) The electric field strength had to be < 100 KV/m (4 – 5X less than for glass fibers)

2) The electrode spacing had to be increased.

3) The spacing between the deposition plate and the upper electrode had to be increased.

4) A padded layer had to be added under the Teflon release cloth on the deposition platform.

The padded layer had to be added to account for the increased mass of the carbon fiber bundles. Without the pad, the carbon fibers would bounce upon impact and all imparted alignment would be lost. One added benefit of processing the carbon fibers was that the heat capacity of the carbon was higher than the glass, therefore the sintering of the matrix powder onto the deposited mat was accomplished with much less time.

When looking at the results of the carbon fiber system as shown in Figures 4.3.2 – 4.3.5, it is easy to see that the ADF processing had a significant improvement on the mechanical properties. When comparing the alignment effect on the tensile modulus, the 0.25" system showed a 175% improvement, the 0.50" system showed a 149% improvement, and the 1" system showed a 60% improvement. For the tensile strength, the improvement was 35% for the 0.25", 100% for the 0.50", and 15% for the 1" system. The average improvement added by ADF processing to the carbon/nylon system was 128% for tensile modulus, and 50% for tensile strength. When analyzing these results, there appears to be a diminishing return on mechanical

property improvement as the fibers became longer – which is opposite of the glass fiber system trends. However, there is a logical explanation for this. As was discussed prior in this section, the cross-sectional geometry difference between the glass and carbon fiber tows played a large role in the behavior when processed with the ADF apparatus. Below, in Figure 4.3.1 is a diagram of the cross-sections of the glass and carbon fiber tows:

Figure 4.3.1 Cross Sections of Glass and Carbon Fiber Bundles

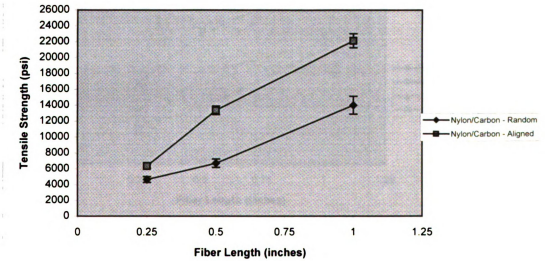


Figure 4.3.2 Tensile Strength of Nylon-12/Carbon Fiber

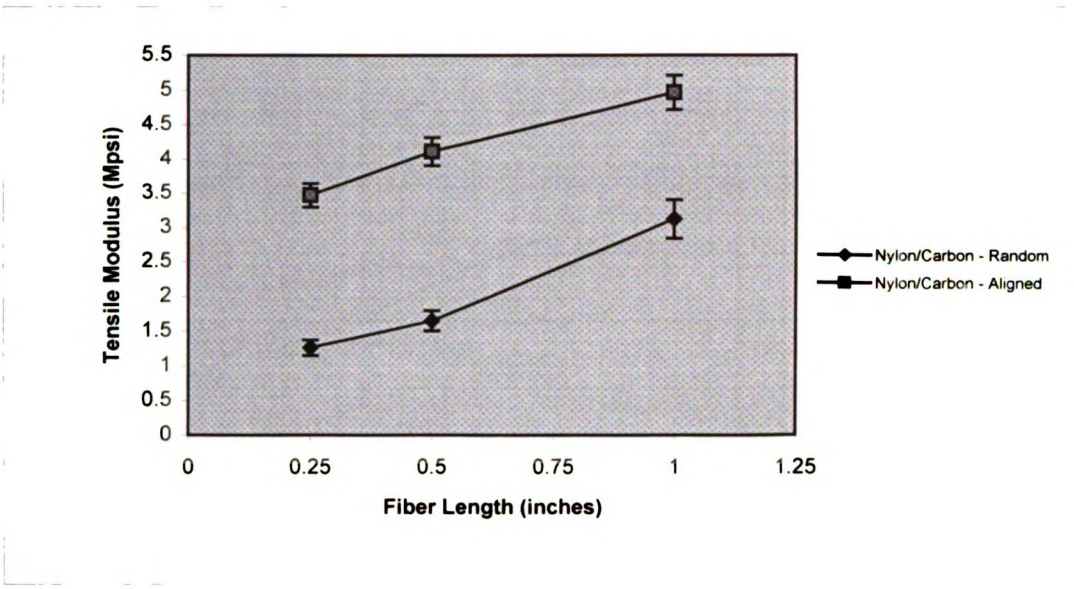


Figure 4.3.3 Tensile Modulus of Nylon-12/Carbon Fiber

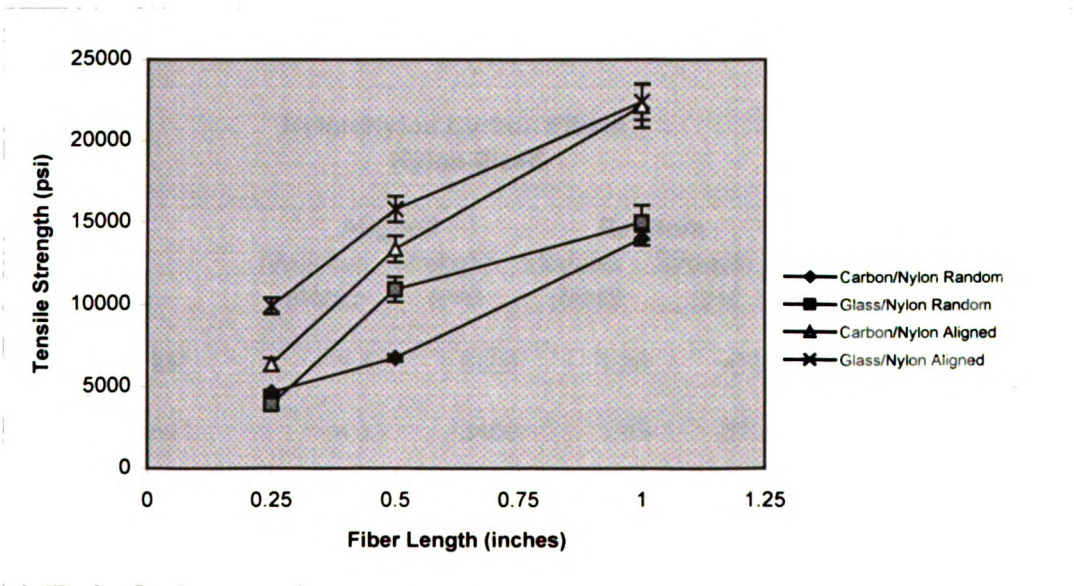


Figure 4.3.4 Comparison of Tensile Strength for Glass and Carbon Fiber ADF Composites

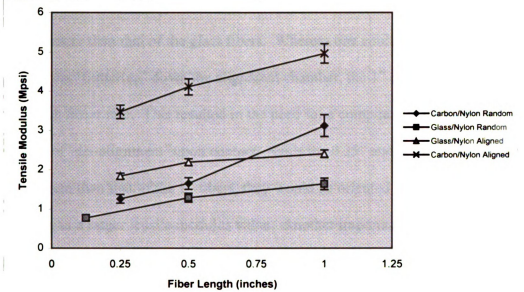


Figure 4.3.5 Comparison of Tensile Modulus for Glass and Carbon Fiber ADF Composites

Nylon/Nylon Carbon Fibers
Nylon-Sized

	Aligned		**Random**	
	Modulus (Mpsi)	*Strength (psi)*	*Modulus (Mpsi)*	*Strength (psi)*
0.25"	3.47	6300	1.26	4700
0.50"	4.11	13400	1.65	6700
1"	4.97	25600	3.11	22200

All samples at 50% volume fiber +/- 3%

Table 4.3.1 Results for Carbon Fiber ADF Composites

The combination of the increased mass of the carbon fiber bundles and the more aerodynamic shape of them resulted in the velocity at impact with the deposition plate being greater than that of the glass fibers. Whereas one could visually see the 1" glass fibers 'fluttering' down the alignment chamber, the 1" carbon fibers would fall at a much faster rate. This resulted in the need for a compliant plate to reduce the amount of 'dis-alignment' upon contact. Since the 0.25" and 0.50" fibers had less momentum than that of the 1" fibers, they would be oriented to a larger extent – resulting in a larger tensile modulus value. Another important difference between the carbon and glass fibers was that the sizing material applied to the bundles appeared to be thicker or of a stronger material. While processing, some glass fibers would appear 'fuzzy', which under an optical microscope would appear as individual filaments that had penetrated the sizing material. One could release the contents of the glass fiber bundles by rolling them between ones fingers. The carbon fibers, on the other hand, had a much greater structural integrity of the sizing agent. This lack of 'fuzziness' allowed the carbon fibers to pack more efficiently overall than the glass fibers, although the bouncing problem was still an issue. Also, the shorter carbon fibers would make more efficient use of the space on the deposition platform as compared to the longer carbon fibers. As is true in real life, the smaller the object, the more efficiently the object can be packed. Since all of the ADF composites processed in this research were made from several ADF mats, e.g., eight mats were used for each composite, the weight of fibers going into the manufacture of each mat was constant. The only variables were then the length and number of fibers. The longer fiber ADF mats were made with less fibers than the shorter fiber ADF mats. Since

the carbon fibers were stiff, the 1" ADF mats were much thicker than those of the 0.25" and 0.50" fibers. This increased thickness was a direct result of the inefficient packing of the fibers on the deposition plate Due to less parallel alignment for the longer fibers and more crossing of the fibers.

When comparing the mechanical results for the carbon and glass fiber nylon systems, they agree well with the physical property data. The carbon fibers have a tensile modulus that is roughly 3X higher than that of glass, and the glass fibers have a slightly larger tensile strength. In figures 4.3.4 – 4.3.5, one can see that the results track these trends. An interesting result was observed that as the fiber length increased, the amount of improvement in material property decreased compared to the shorter fiber lengths. Whereas the 0.25" and 0.5" systems increased 175% and 149%, respectively, for tensile modulus, the 1" fibers only increased 60% compared to the random sample. When analyzing the fiber orientation diagrams in the next section, they show that the longer carbon fibers also were aligned to a greater degree. The only plausible explanation was that the random 1" carbon/nylon fiber system had an usually large tensile modulus. Overall, the carbon fiber systems increased 128% for tensile modulus and 50% for tensile strength, which shows that the ADF process adds value to the raw material.

4.4 Measurement of Fiber Orientation

The key parameter of any ADF composite is how aligned the fibers are. Without knowing how well the fibers are aligned, it would be impossible to predict

the mechanical properties using micromechanical models and determine if the ADF process equipment was functioning at its optimal conditions. A measurement of the alignment is known as the fiber orientation distribution (FOD). Typically, the fiber orientation distribution is obtained by a measurement of the orientation of a statistically significant number of fibers by cross sectioning the composite. The cross section is then analyzed and all non-circular fiber cross sections are reconstructed using the aspect ratio of the ellipse that is formed. This technique can be very tedious and inaccurate, so a new technique developed earlier by a previous researcher was used.. The technique measures the fiber orientation in an indirect way. A series of images of fibers being deposited on the deposition plate were collected using a Panasonic CCTV (Model WV 1410) camera, which was connected to a PC outfitted with a frame-grabber board. Because glass fibers will reflect light preferentially when it is incident at an angle, utmost care was taken to ensure that the lighting in the room was uniform across the deposition plate. A statistically significant number of fibers, 1000, were recorded for each FOD. On average, a larger number of images were required for the larger fibers. The Global Lab Image Analysis software package was used to digitally filter and sharpen the images of the fibers to enhance the edge detection and improve the contrast of the fibers compared to the background (Teflon release cloth). The "particle classify" routine in the software was used to identify the fibers and their orientation with respect to a pre-entered guide. It is important to note that fibers that were touching each other or otherwise connected in some fashion were unable to be classified by the analysis package, therefore all fiber orientations are for

less than monolayer coverage of fibers. This means that any 'dis-alignment' that occurs in normal processing of fibers

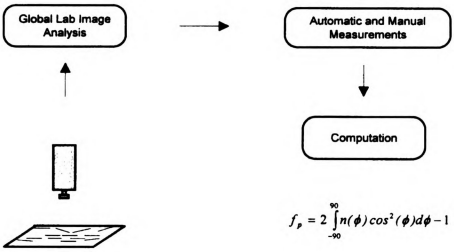


Figure 4.4.1 FOD Image Collection
(reprinted with permission from Vyakarnam, *et al.)*

falling on top of other fibers is not included in the FOD. These FOD's can then be described as 'best' case. All of the FOD data for all six of the systems studied is reported and a summary of the alignment is presented above in a table. Table 4.4.1

Angle	1" Glass	0.50" Glass	0.25" Glass	1" Carbon	0.50" Carbon	0.25" Carbon
+/- 10^0	59%	56%	54%	51%	46%	44%
+/- 20^0	74%	72%	71%	68%	66%	65%
+/- 30^0	82%	80%	79%	77%	76%	78%

All results +/- 3%

Table 4.4.1 Results of Alignment for ADF Systems

above lists the percentages of fibers oriented within a specified angle. For the glass fibers, the trend is clear that increasing fiber length slightly increases the amount of orientation that one can impart. The carbon fibers also exhibit this trend except for the 1" and ¼" cases when looking at all fibers within +/- 30°. The carbon fibers also appear to not be aligned as well, which makes sense. The process for aligning the carbon fibers has not been fully optimized. A careful investigation into a pad material which absorbs the most energy from the fibers upon impact could increase the fiber alignment percentage. A clear indicator that the carbon fibers are still bouncing when they hit the deposition plate is that while the data points for the+/- 10° is around 10 % lower for carbon, the total amount within +/- 30° is roughly the same. This same total within 30° suggests that an equivalent amount of alignment is imparted to the carbon fibers, but the bouncing disrupts the alignment slightly.

One can describe the orientation state of a short fiber by two angles, ϕ and θ. The transformation matrix from the original (composite) coordination system of the fiber to that of the three principle axes can be obtained by T below:

$$T(3D) = \begin{vmatrix} \sin\theta\cos\phi & \sin\theta\sin\phi & \cos\theta \\ \cos\theta\cos\phi & \cos\theta\sin\phi & -\sin\theta \\ -\sin\phi & \cos\phi & 0 \end{vmatrix}$$

For planar orientation, the transformation tensor simplifies to the following form:

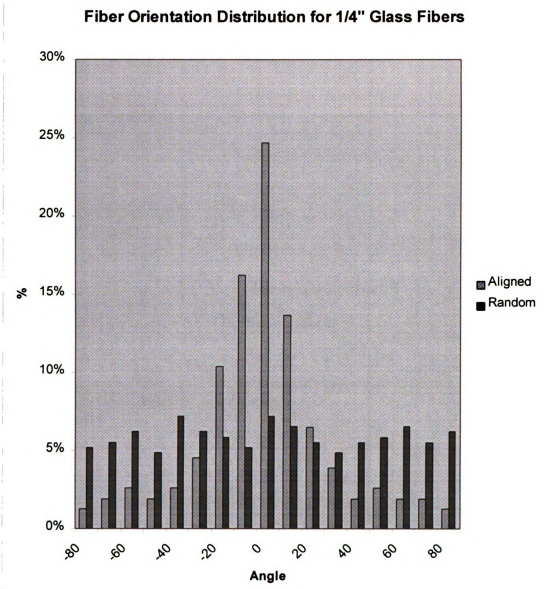


Figure 4.4.2 FOD for 0.25" Glass Fibers
(+/- 2 %)

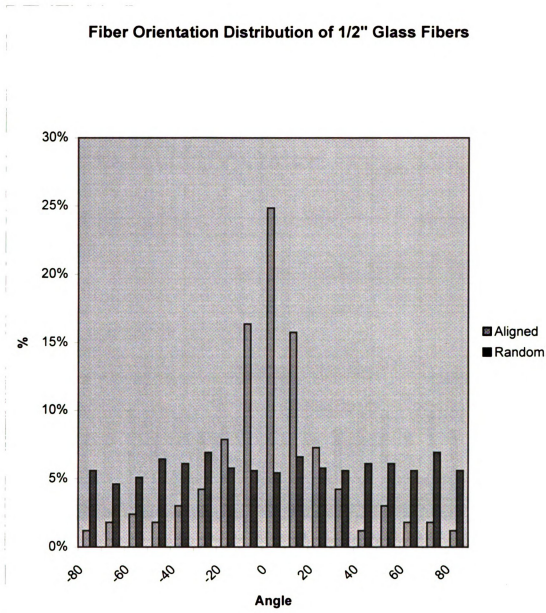


Figure 4.4.3 FOD for 0.50" Glass Fibers
(+/- 2%)

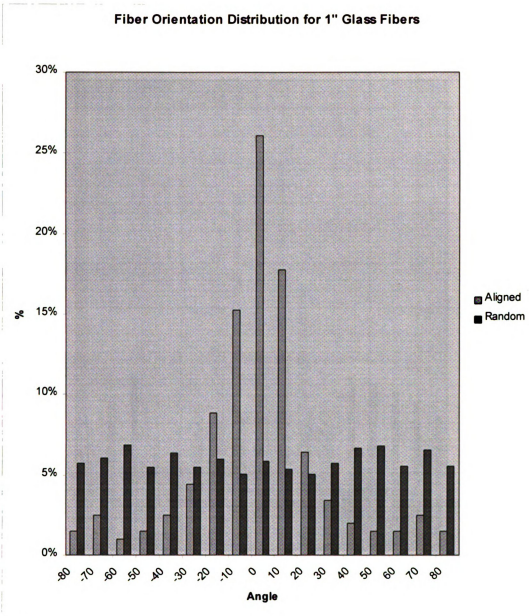


Figure 4.4.4 FOD for 1" Glass Fibers
(+/- 2%)

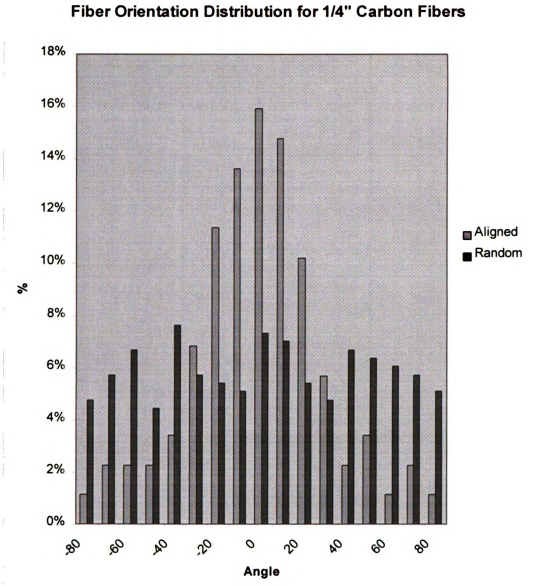


Figure 4.4.5 FOD for 0.25" Carbon Fibers
(+/- 2%)

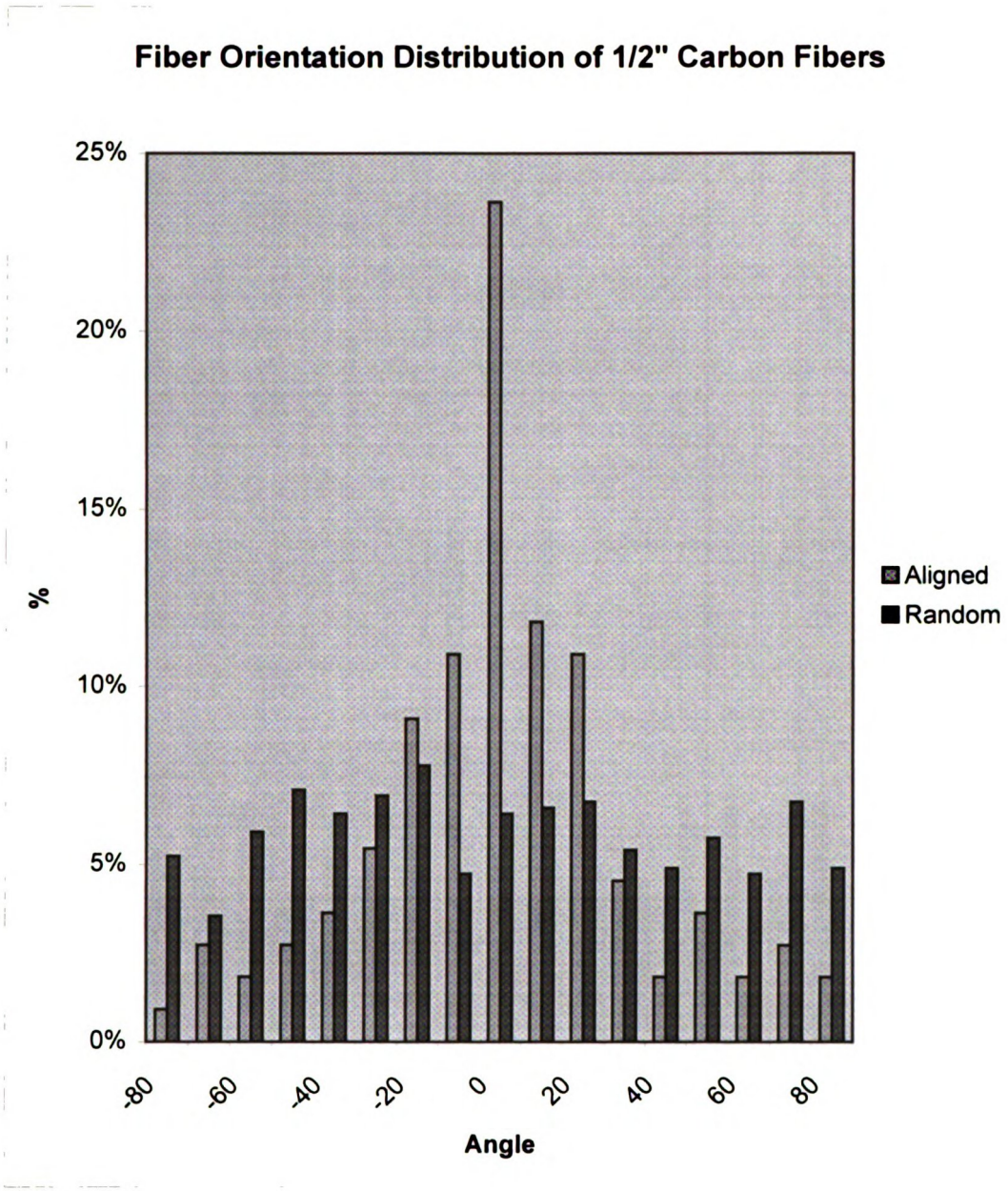


Figure 4.4.6 FOD for 0.50" Carbon Fibers
(+/- 2%)

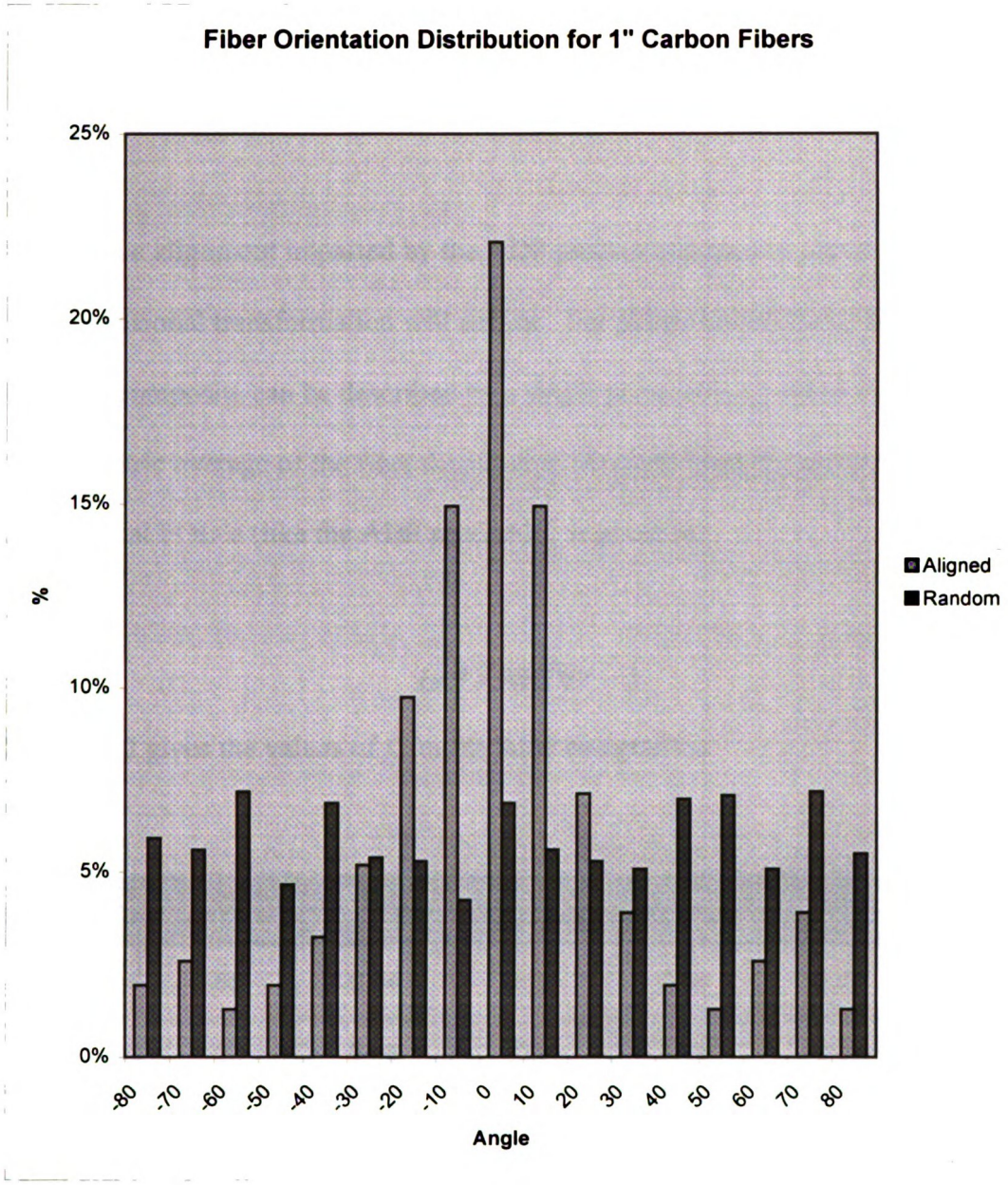


Figure 4.4.7 FOD for 1" Carbon Fibers
(+/- 2%)

$$T(2D) = \begin{vmatrix} \cos\phi & \sin\phi & 0 \\ -\sin\phi & \cos\phi & 0 \\ 0 & 0 & 1 \end{vmatrix}$$

Because the alignment imparted by the ADF process completely planar in nature, the two-dimensional transformation will suffice. For planar orientations, the orientation state of a composite can be described by a single parameter, f_p, which is the trigonometric average of the fiber distribution obtained from the above tensor. For symmetrical FOD's (like the ADF process) f_p is given as:

$$f_p = 2\langle\cos^2\phi\rangle - 1$$

Table 4.4.2 gives the values of f_p for the ADF composites:

	f_p					
	1" Glass	½" Glass	¼" Glass	1" Carbon	½" Carbon	¼" Carbon
Aligned	0.65	0.64	0.62	0.58	0.57	0.58
Random	0.03	0.08	0.07	0.04	0.11	0.09

Table 4.4.2 f_p values for the ADF composites
(all results +/- 3%)

The results gathered in this work agree well with the results from the prior researcher. In the case of fiber mats, Vyakarnam found that for all the four fiber lengths, the orientations of about 70% of the fibers laid between +/- 20° and nearly 80-90% of the fibers between +/- 30°. The results from this work were roughly 70% between 20° and slightly over 80% between 30°. Fiber orientation parameter results

also agree with the prior work – for 0.25", 0.50" and 1" aligned Vyakarnam recorded values of 0.63, 0.63, and 0.73, respectively.

4.5 Halpin-Tsai/Micromechanics

The three most important parameters that affect the mechanical properties of discontinuous fiber composite systems (for a constant fiber volume fraction) are 1) the fiber orientation distribution (FOD) 2) fiber aspect ratio and 3) fiber-matrix interaction. Fiber-matrix interaction can be best described as the amount of matrix impregnation of the fiber bundle. This parameter depends greatly on the fiber sizing and the interfacial adhesion between the matrix and the fiber sizing. While the first two parameters play a pivotal role in the modulus predictions, the last parameter is critical for the strength and fracture behavior. The role of the fiber volume fraction is well understood and has a linear effect relationship to the mechanical properties as expected from the Rule of Mixtures.

All ADF samples produced during this research were targeted for 50% fiber volume fraction (+/- 3 %). Experimental matrix burn-off analysis of test coupons verified this. Void content was comparable to samples made by the prior researcher. Whereas commercially available continuous fiber composites are usually < 1% void, the average void content in these ADF composites was ~ 4%. Higher void content could influence tensile strength values, but tensile modulus should not be affected.

The degree of impregnation of the matrix into the chopped fiber bundle plays an important role in determining whether the fiber or bundle will be the dominant

factor. Vyakarnam investigated whether the reinforcement aspect ratio was more bundle or fibrous in nature. When using compatible fiber sizings with the matrix, good impregnation can be achieved, as seen in micrographs shown in Chapter 3. Incompatible fiber sizings, such as the PE-sized glass fibers, result in lower impregnation of the fiber bundle and therefore lower mechanical properties.

Fiber aspect ratio is calculated by dividing the length of the fiber by the diameter. Bundle aspect ratio is slightly more complicated to calculate:

$$a_b = \frac{L_b}{d_b}$$

where b indicates the dimensions of the bundle. The following table lists all of the aspect ratio for glass and carbon fibers.

Nominal Length(mm)	Glass Fibers		Carbon Fibers	
	Filament Aspect Ratio	Effective Bundle Ratio	Filament Aspect Ratio	Effective Bundle Ratio
6.3	485	28.5	870	7.9
12.7	977	57.5	1750	16.0
25.4	1990	115	3500	32.0

Table 4.5.1 Fiber Bundle Length and Aspect Ratios

From the table above, one can see how the carbon fibers have a higher filament aspect ratio and a smaller bundle ratio, due to the smaller fiber and the larger number of filaments (i.e. 3000) that make up each bundle. The glass fibers, on the other hand, had on average ~ 200 filaments per bundle.

Fiber Length	f_p Exp. Result		Halpin	Tsai	Model	Predictions
			Aligned	Aligned	Random	Random
(inches)	Aligned/ Random	Aligned/ Random	*'Filament'*	*'Bundle'*	*'Filament'*	*'Bundle'*
0.25"	**0.62**/0.03	**1.84**/1.20	**2.15**	**1.63**	1.07	0.85
0.50"	**0.64**/0.08	**2.33**/1.29	**2.40**	**2.05**	1.10	0.99
1"	**0.65**/0.07	**2.65**/1.64	**2.71**	**2.19**	1.08	1.01

Table 4.5.2 Halpin-Tsai Model Predictions for the Glass Fiber/Nylon –12 System

Fiber Length	f_p Exp. Result		Halpin	Tsai	Model	Predictions
			Aligned	Aligned	Random	Random
(inches)	Aligned/ Random	Aligned/ Random	*'Filament'*	*'Bundle'*	*'Filament'*	*'Bundle'*
0.25"	**0.62**/0.03	**2.83**/1.84	**3.37**	**2.89**	1.79	1.65
0.50"	**0.64**/0.08	**3.27**/2.00	**3.84**	**3.16**	2.34	1.92
1"	**0.65**/0.07	**4.33**/3.4	**4.46**	**3.67**	2.84	2.33

Table 4.5.3 Halpin-Tsai Model Predictions for the Glass Fiber/PET System

Fiber Length	f_p Exp. Result		Halpin	Tsai	Model	Predictions
			Aligned	Aligned	Random	Random
(inches)	Aligned/ Random	Aligned/ Random	*'Filament'*	*'Bundle'*	*'Filament'*	*'Bundle'*
0.25"	**0.62**/0.03	**1.65**/0.76	**1.86**	**1.71**	0.86	0.81
0.50"	**0.64**/0.08	**2.48**/0.95	**2.57**	**1.85**	0.94	0.87
1"	**0.65**/0.07	**3.4**/1.41	**3.22**	**2.28**	1.11	0.99

Table 4.5.4 Halpin-Tsai Model Predictions for the Glass Fiber/PP System

Fiber Length	f_p	Exp. Result	Halpin	Tsai	Model	Predictions
			Aligned	Aligned	Random	Random
(inches)	**Aligned**/ Random	**Aligned**/ Random	*'Filament'*	*'Bundle'*	*'Filament'*	*'Bundle'*
0.25"	**0.58**/0.04	**3.47**/1.26	**3.91**	**3.10**	1.35	1.15
0.50"	**0.57**/0.11	**4.11**/1.65	**4.35**	**3.76**	1.55	1.42
1"	**0.58**/0.09	**4.97**/3.11	**4.63**	**4.32**	2.18	1.89

Table 4.5.5 Halpin-Tsai Model Predictions for the Carbon Fiber/Nylon-12 System

The Halpin-Tsai equation is given below:

$$\frac{P}{P_m} = \frac{1 + \varsigma\eta V_f}{1 - \eta V_f}$$

where:

$$\eta = \frac{\frac{P_f}{P_m} - 1}{\frac{P_f}{P_m} + \varsigma}$$

$$\zeta = 2(a/b)$$

P are the property to be calculated, subscripts f and m are fiber and matrix, and a, b are geometric constants of the reinforcement.

The Halpin-Tsai equations were used to calculate two extremes of aspect ratios. The 'filament reinforcement' was defined as compatible fiber sizing with the matrix, and the 'bundle reinforcement' was defined as incompatible fiber sizing with

the matrix. The tensile modulus was calculated with the Halpin-Tsai equations for both cases. Two fiber orientation states were also measured, varying from random (f_p values ~ 0.10) to 'aligned' (f_p values ~ 0.6) The calculated modulus values were used to obtain the modulus of the actual ADF composite by integrating the effect of fiber orientation using the FOD. From the results on the two tables above, it appears that the filament reinforcement approach predicts the values far better than the bundle approach, which is contrary to the results in the prior work.

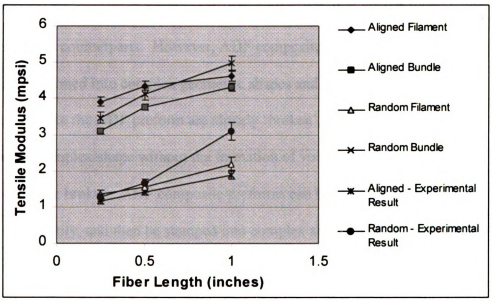


Figure 4.5.1 Experimental vs. Model Predictions for Nylon/Glass Fiber System

The bundle approach underestimates the modulus for all of the glass and carbon fiber systems. The filament approach tends to overestimate the modulus for most cases, except for the 1" carbon fiber results, which it underestimates. The results make sense from the standpoint that all of the fiber systems studied in this work, except for the PE-sized glass fibers, had compatible sizings with the matrices.

4.6 Drapeability Results

In industrial use, aligned discontinuous fiber composites compete against continuous fiber composites. In order to be commercially competitive, there must be some advantage that attracts interest, such as ease of processing, cost, or some type of improved mechanical/physical property. ADF composites, when made from the same type of fiber as continuous fiber composites, cannot match the strength of their continuous fiber counterparts. However, ADF composites do possess two advantages – they can be formed into complex geometric shapes and they are easier to process. Since the fibers in the ADF preform are already 'broken', they have the mobility to conform to a complex shape without the formation of voids where the continuous fiber would have broken. ADF composite preforms can be manufactured in large volume quite easily, and then be stamped into complex shapes with the same ease – something that continuous fiber composites cannot do. Thus, it was decided that these attributes would be experimentally verified for the ADF process. The work conducted was the first of it's kind and several processing metrics were invented to deal with this new property called 'drapeability'. The most important was how to test this parameter, which will be described later in this section.

The processing of the samples for the drapeability work was the same as for the regular ADF systems. Eight ADF mats were processed for each composite sample under identical processing conditions. The molds for this work were described in Chapter 3. Three molds were made with a 0.125" radius of curvature,

one with 0.25", and one with 0.50". The composites mats were hand-laid into the mold, with an additional amount of matrix added to the top to counter the losses of matrix through the sides of the mold. The entire assembly was then vacuum bagged and processed with the Tetrahedron press. The only processing difference between the shaped and flat ADF composites was that additional heating time was required for the shaped composites – due to the differences in the size of the mold.

A sample preparation technique had to be developed for this new test, as ASTM did not have a standard method for testing the curved specimens. The samples were first cut with the Felker saw so that the length of the sample was 2 inches from the middle of the curved section. Once the 2-inch gauge length was cut, the piece was then sectioned into 6 samples, which then had the sides sanded with consecutively finer grits of sandpaper. Once the samples had been polished and allowed to dry at room conditions overnight, they were tested using the same equipment as the tensile specimens. However, the equipment was modified to use compression instead of tension. Large cylindrical load plates were attached to the load points, with the curved sample resting in the center of the lower disc.

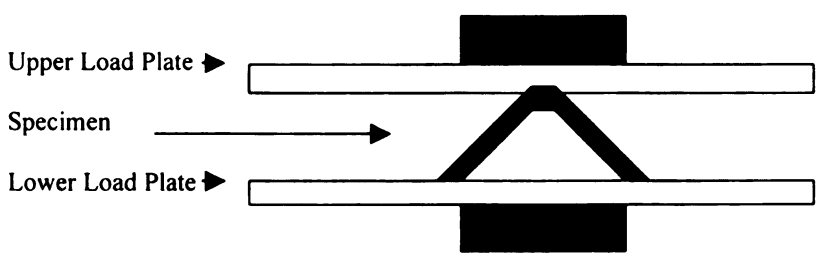


Figure 4.6.1 Compression Testing Apparatus

Compression was then applied at a rate of ½"/min.

ROC	Fiber Length (all values lbs/in²)		
	0.25" Aligned	0.50" Aligned	1" Aligned
0.125"	44	23	54
0.25"	33	49	60
0.50"	20	57	82

Table 4.6.1 Radius of Curvature Results
(all results +/- 4 %)

The data above is considered to be the strength of the curved 'joint'. The same equation for tensile strength computation was used for the compression data. Where the strength was equivalent to the compressive force divided by the cross sectional area of the joint As one can see from the table, the 0.25" fiber exhibited the largest compressive strength at the shortest radius of curvature. As the ROC increased, the same length of fiber had a more difficult 'bridging' the ROC gap. For example, at 0.25" and 0.50", the 0.25" fiber was not able to fully 'bridge' the gap and thus exhibited lower strength numbers. The same phenomenon is true for the 0.5" and 1" fibers. From this preliminary research, if the ROC study was expanded to 0.75", 1", and 1.5", we would observe a 'peaking' of compressive strength for the

0.50" and 1" fibers, much like what was seen for the 0.25" fibers in the 0.125" to 0.25" progression. The 0.25" fiber 'peaked' at the 0.125" ROC, where the 0.50" and 1" fibers both became stronger as the ROC increased.

The drapeability testing done in this research was quite basic. The data suggested several trends discussed above, but the magnitudes of the mechanical properties are not indicative of the potential strengths of the ADF composites when stamped into complex shapes. More work is needed in this area, such as comparisons of randomly oriented samples with the aligned samples and the measurement of the properties of a neat resin coupon of the same configuration.

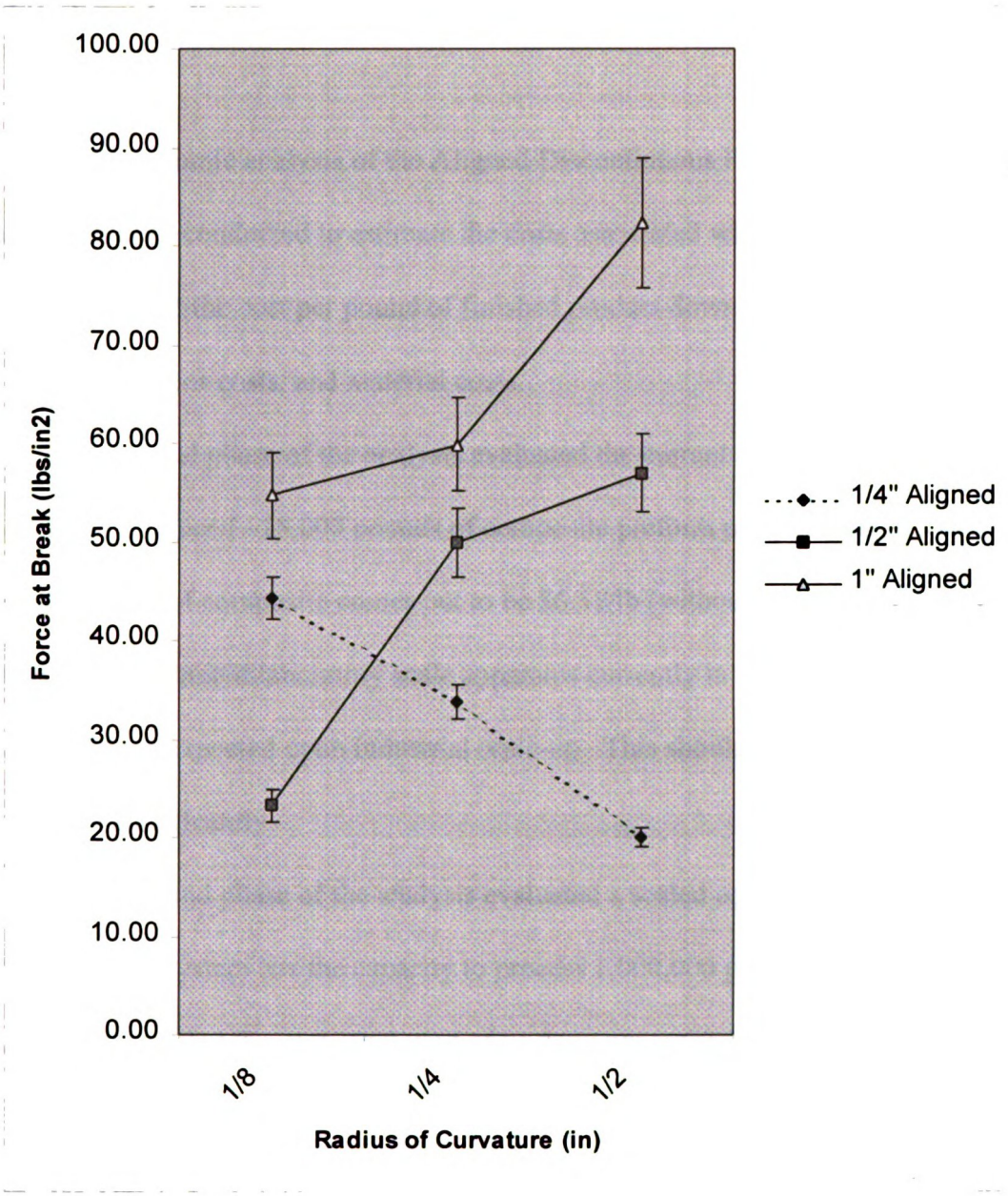


Figure 4.6.2 Radius of Curvature Results

5.1 Introduction

An economic analysis of the Aligned Discontinuous Fiber Composites Process (ADF) has been conducted to estimate the costs associated with this process. This evaluation broke the cost per pound of finished product down into three main categories, capital costs, labor costs, and material costs.

The initial phase of the analysis evaluated the current system. This system has the capacity to produced ~18,000 pounds of composite preform per year. At this rate, the cost per pound of composite comes out to be $6.51/lb (without material costs). However, because of the limited laboratory scale apparatus currently in use, greater speeds and throughput are expected upon industrial scale-up. This should reduce the cost of composite significantly.

The second phase of the analysis evaluated a scaled up production ADF process. This scaled up system has the capacity to process 1,000,000 pounds of preform per year. Analysis shows that the cost per pound of preform approaches ~$0.35/lb.

The ADF process has the potential to be cost effective composite production process. The process requires state-of-the-art off-the-shelf equipment with low power requirements, a minimum of moving parts and operates under principles, which can easily be scaled up without new machine design requirements. The ADF process appears to have the speed and reliability, which make the process desirable for large-scale industrial production.

5.2 Process Description

The process being evaluated produces aligned discontinuous fiber (ADF) composites. The initial step of the process uses a vibratory feeder to feed discontinuous bundles of reinforcing fibers (e.g. glass or carbon) into the alignment chamber. The feeder also "pre-aligns" the bundles into a planar orientation perpendicular to the electric field and also controls the rate at which the fiber bundles are fed to the alignment chamber. The fiber bundles then pass through the high voltage alignment chamber where they come under the influence of an electrical field, which produces a polarization torque on fibers oriented at an angle to the field, which causes them to align. This alignment chamber consists of two parallel plate copper electrodes housed within a plexi-glass support structure. An important feature of this process is that since the electrodes are parallel in alignment, one can scale up the process according to theory in both the x- and y-directions. The aligned bundles then deposit onto a moving veil, which runs at a controlled speed predetermined by the thickness required. This veil then passes under a powder dispersion apparatus, which coats the fibers with the desired amount of thermoplastic matrix in powder form. Alternatively, the powder can be coated on individual fibers with the MSU high-speed polymer powder process and then chopped into short bundles. After being coated with the powder, the preform sheet passes under an infrared heater, which sinters the polymer particles to the fiber bundles and ensures that the desired alignment is not disturbed during subsequent processing/handling. The sintered preform sheet can then be carried by the veil to a take-off apparatus which can roll the preform into drums for packaging and delivery.

The electric field strength is monitored via a high voltage probe that is connected to the high voltage plate. The high voltage probe is in turn connected to a multimeter voltage readout. Control of the electric field strength can be accomplished two ways, by altering the setting on the variac unit (which controls the input voltage to the transformer), or by changing the separation distance of the electrodes. The parallel plate geometry simplifies the mathematics, yielding a straightforward relationship between the electric field strength and the electrode gap.

$$\mathbf{E = V/d}$$

Where E is the electric field strength, V is the input voltage, and d is the electrode separation. Control of the fiber orientation can be maintained by periodic examination of the fiber orientation diagram (FOD), which is measured by a video camera/computer tool.

When working with E-glass fiber composites it is important to have control over the environmental conditions. Earlier in this research it was noticed that the humidity of the workspace played an important role. Humidity of ~50-60% is sufficient to reduce the breakdown voltage of the equipment sufficiently to prevent excess discharges. On a worker's safety note – the electric field utilizes a high voltage, however the amperage is stepped down to the ~15 milliamp range which is well under the heart stopping threshold. Although one would not want to "bridge" the electrodes, it would not do any serious damage to the individual.

5.3 Process Economic Model

In order to effectively estimate the costs associated with different levels of production, a cost and economic model was developed using a spreadsheet running under Microsoft Excel. The main elements of this model are available in Tables 5.4.2 and 5.5.1. The model takes into account all of the usual costs associated with operating a process, i.e. real estate, HVAC, utilities, materials, etc. However, since the cost of producing the preform is going to depend to a large extent on the cost of the composite fiber and matrix, it was decided to compute the cost per pound of the material, independent of the material costs so that the evaluation would only take into account the process.

The costs were broken down into two main categories, capital and variable costs. The capital costs were broken down into the major components of the system. The estimates used for these major components were derived based upon costs incurred in building the current bench top system or through quotes from industrial vendors. The useful life of the process was assumed to be three calendar years. This was assumed because of the nature of the equipment. The capacitor is operating at constant voltage; the conveyor and its components are off-the-shelf items, and the electrodes are static and are not degraded by the process.

1,000,000 lbs/year Example

1) 1,000,000 lbs /(80% of 365 days/year (using 80% uptime) * 24 hours/day) =

143 lbs/hour preform

2) Next, we chose 50% fiber volume fraction:

Checking densities: $1.94 * 10^{-3}$ lb/cm^3 Polypropylene

$5.62 * 10^{-3}$ lb/cm^3 E-Glass Fiber

The ratio of densities = 5.62/1.94 => 2.89

Now it is a simple algebraic equation:

$$2.89X + X = 143 \text{ lbs/hour preform}$$

$$3.89X = 143 \text{ lbs/hour}$$

$$X = 36.8 \text{ lbs/hour}$$

3) Therefore:

Flowrate of fiber = 2.89X => 106 lbs/hour E-Glass

Flowrate of matrix = X => + 37 lbs/hour Polypropylene

For a total of 143 lbs/hour preform

Figure 5.3.1 Equipment Sizing Calculations

The variable costs included the utilities, matrix, fibers, and labor. A labor rate of $15/hr was assumed for this process. This rate was applied to production by dividing the $15 figure by the pounds produced per hour by one operator. Utilities were computed by measuring the amount of electrical usage in an hour and multiplying by the cost of electricity per hour. Finally, the material costs were computed by first determining the desired volume fraction, 50% fiber (by volume) in this work, and then back-calculating the weight per hour from the total production for one year. An example of this calculation can be found in Figure 5.3.1.

5.4 Current System Evaluation

The current bench-top system deposits chopped fiber bundles (200-400 filaments/bundle for E-glass) onto a moving deposition plate at a rate of 2 lb/hr, resulting in a yearly production value of ~18,000 lbs/yr.(schematic of process in shown in Figure 5.4.2) The preform is 8 inches wide and would be wound up onto a roll for shipping and handling. The system also has a relatively small footprint, it only would take up ~200 ft^2 of floor space. One operator can easily supervise this process and possibly more, which would reduce the overall cost of the process. The ability to use one operator to run multiple systems has a major effect on the costs as seen in Table 5.4.2. Results from the economic model are listed in Table 5.4.1

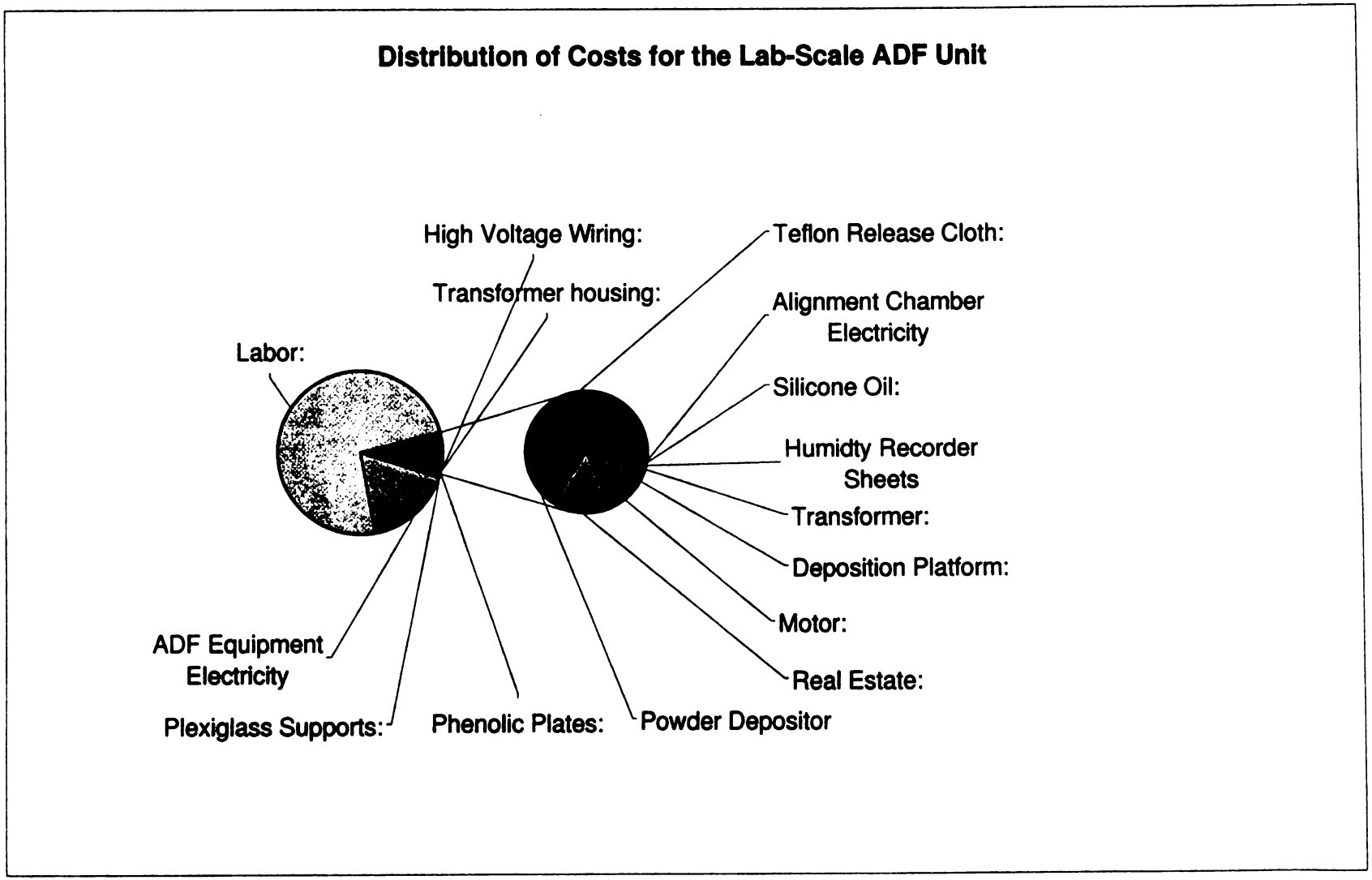


Figure 5.4.1 Cost Distribution for Lab Scale ADF Process

Aligned Discontinuous Fiber Process Economics - Lab Scale
Glass Fiber/Polypropylene Composites

			Capital Costs			Variable Costs
Size of Preform:	8 in wide		Price	3-Year Amortized		
		Transformer:	$600.00	$200.00	Teflon Release Cloth:	$10.27/yard
		High Voltage Probe:	$250	$83.33	Powdered Matrix:	$7.60/pound
Fiber Volume		Multimeter:	$150	$50.00	Chopped Fibers:	$0.65/pound
		Transformer				
Fraction:	50% Fiber	housing:	$500	$166.67		
		Phenolic Plates:	$50	$16.67	Electricity: (@ $0.07/kw)	
Fiber Flow Rate:	2 lb/hr	Deposition Platform:	$1,500	$500.00	Alignment Chamber	$0.01/hour
Matrix Flow Rate:	0.687273 lb/hr	Motor:	$2,750	$916.67	ADF Equipment	$0.70/hour
Density of Fiber	0.0055 lb/cm3	Plexiglass Supports:	$50	$16.67		
Density of PP	0.00189 lb/cm3	High Voltage Wiring:	$50	$16.67	Labor:	$15.00/hour
		misc hardware:	$100	$33.33	Silicone Oil:	$10.00 liter
		Humidity Monitor:	$250	$83.33	Humidity Recorder	
		Real Estate:	$5,000	$1,666.67	sheets:	$1.00/sheet
		Vibratory Feeder:	$3,400	$1,133.33		
		IR Heater:	$300	$100		
		Powder Depositor	$3,400	$1,133		
Production Rate:	2.687273 lbs/hr preform	Capital Costs:	$18,350	$6,116.67		
	18832.41 lbs/yr	Total Costs:	$257,696			

Total Variable Costs:	$239,346.14
Total Costs w/o Materials:	$116,497.21
Total Costs w/o Materials + Labor:	$11,377.21

Cost per Pound:	14 $/lb	w/material costs
1 System	6.51 $/lb	w/o material costs

Table 5.4.1 Laboratory Scale Economic Results

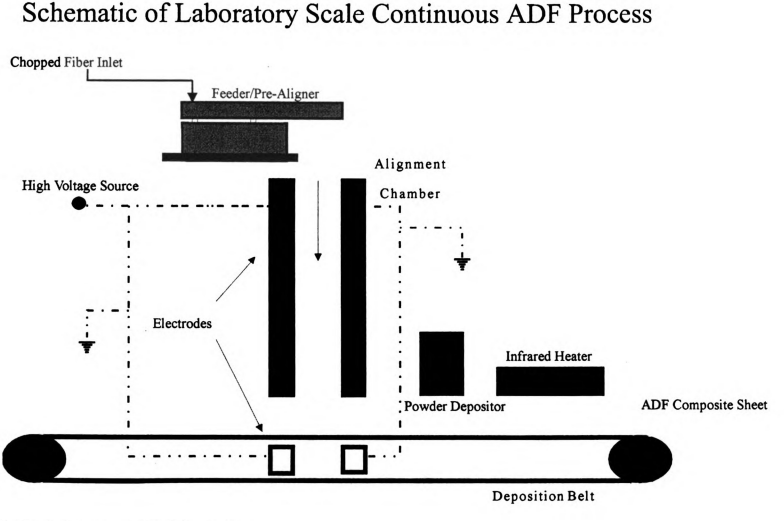


Figure 5.4.2 Laboratory ADF Schematic

Table 5.4.2 - Effect of Labor Cost on Laboratory Scale ADF Process Economics

Systems/ Operator	1	2	3	4	5
Added Cost/lb.	$6.51	$3.72	$2.79	$2.32	$2.05

After reviewing the analysis developed for the current system, it is apparent that it is highly cost prohibitive. At ~$6/lb added to the material cost during processing, the price of the ADF composite preform product would be very high compared to other processes and therefore noncompetitive. On the positive side, the total capital investment for setting up a pilot-scale line is relatively low. It is important to note that the main factor contributing to the high costs in the lab scale is the capital costs. The low-volume of material produced coupled with the expensive pieces of equipment combine to push the $/lb upwards. Figure 5.4.1 shows the distribution of the various costs in the laboratory scale process.

5.5 Scale-up Evaluation

The industrial scale ADF model is based on 1,000,000 lbs of preform per year. The preform would measure 36" across, which would allow for the stamping of relatively large, complex parts. Based on simple capacitor theory, scale-up of the ADF process would be relatively simple. A schematic diagram of the industrial process is shown in Figure 5.5.1. Since there is negligible charge flow between the electrodes, the

transformer is used only to sustain the electric field. Based on this fact, one large transformer could power an entire process line. Scale-up of the electrodes is accomplished quite easily in the x- and y-directions. Although in this analysis a 36" size preform was chosen, one can easily fabricate a much larger preform for the stamping of very large, complex parts, e.g. car doors, undercarriage battery holders for electric vehicles, etc. The thickness of the preform mat can easily be controlled by the spacing and number of ADF Alignment chambers. For example, using a wider spacing between the electrodes will increase the amount of fiber that can be processed in that chamber. The reason for this is that there is a critical fiber density, which when exceeded causes misalignment of fibers. This misalignment happens because a large amount of fibers come into contact and 'clump' together, becoming harder to align as they fall. When the electrodes are moved farther apart, this fiber density is decreased, allowing for larger fiber flows. However, increasing the electrode gap will require a larger input current because of the electric field strength relation discussed in Section 5.2.

The economic results for the 1,000,000 lb/yr process are listed in Table 5.5.2. Having one operator operate the industrial scale ADF Process adds $0.35 to each pound of material processed. Adding multiple ADF lines under the control of single operator greatly improves the economics, as seen in Table 5.5.1. As one can see, the labor cost.

Table 5.5.1 Effect of Labor Cost on Industrial Scale ADF Process Economics

Systems/ Operator	1	2	3	4	5
Added Cost/lb.	$0.35	$0.30	$0.28	$0.28	$0.27

greatly affects the economics, which means that the equipment used in the ADF process is relatively inexpensive

Compared to the laboratory scale, the industrial scale price is fairly independent of the capital costs. As Table 5.5.1 shows, the labor cost has an affect to a certain degree, but does not in any way drive the economics. The relationship between the various costs and comparison with the bench scale model can be seen in Figure 5.5.2.

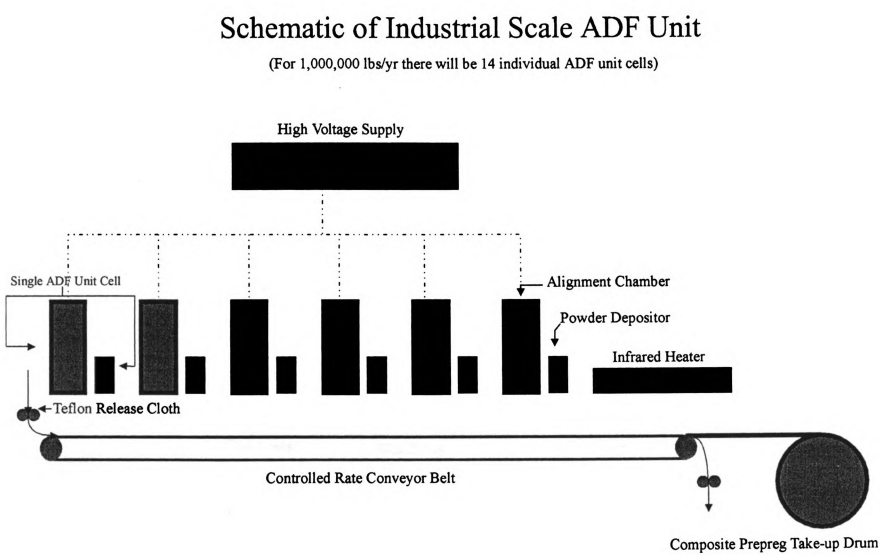


Figure 5.5.1 ADF Industrial Schematic

Aligned Discontinuous Fiber Process Economics – Industrial Scale
Glass Fiber/Polypropylene Composites

			Capital Costs			
36in wide					Variable Costs	
	|	Transformer:	$600.00	$200.00	Teflon Release Cloth:	$10.27/yard
	|	High Voltage Probe:	$250.00	$83.33	Powdered Matrix:	$7.60/pound
	|	Multimeter:	$150.00	$50.00	Chopped Fibers:	$0.65/pound
50% Fiber	|	Transformer housing:	$500.00	$166.67		
	|	Phenolic Plates:	$100.00	$33.33	Electricity:(@$0.07/kw hour)	
106.2lb/hr	14X	Plexiglass Supports:	$200.00	$66.67	ADF Equipment	$14.70/hour
36.49416lb/hr	|	High Voltage Wiring:	$50.00	$16.67	Alignment Chamber	$0.45/hour
0.0055lb/cm3	|	Vibratory Feeder:	$3,400.00	$1,133.33	Labor:	$15.00/hour
0.00189lb/cm3	|	Powder Depositor:	$3,400.00	$1,133.33	Silicone Oil:	$10.00/liter
	|	misc hardware:	$100.00	$33.33	Humidity Recorder	
		Real Estate:	$20,000.00	$6,666.67	sheets:	$1.00/sheet
		HVAC:	$10,000.00	$3,333.33		
		Humidity Monitor:	$250.00	$83.33		
		IR Heater:	$17,500.00	$5,833.33		
		Motor&Conveyor	$50,000.00	$16,666.67		
142.6941lbs/hr preform		For Line: (14 individual)	$220,250.00	$73,416.67		
		Total Costs:	$2,928,348			
1000000lbs/yr					Total Variable Costs:	$2,708,098.32
					Total Costs w/o Materials:	$280,627.90
					Total Costs w/o Materials + Labor:	$175,507.90

3$/lb w/material costs
0.35$/lb w/o material costs

Table 5.5.2 Economic Analysis Results for Industrial Scale-up

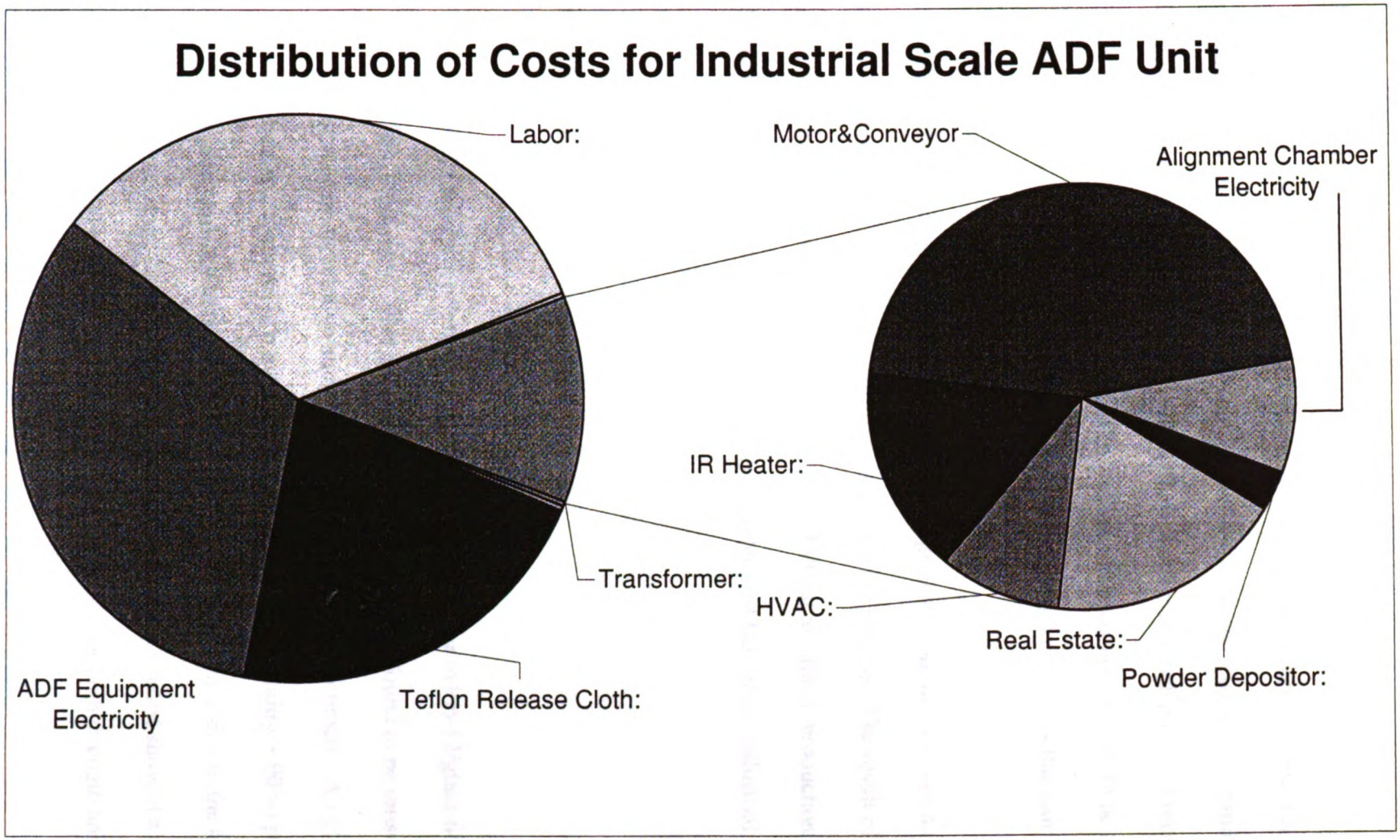


Figure 5.5.2 Cost Distribution of the Industrial ADF Process

6.1 Conclusions

The ADF process was successfully modified to accommodate alignment and processing of conductive fibers. ADF composites have superior properties to random orientation composites. An investigation into the thermoforming behavior and properties of ADF composites was also conducted. The ADF process has been shown to be capable of being utilized with any discontinuous fiber to produce rapid pre-forms that can be shaped into complex geometries.

An additional question is the economics of large-scale commercialization of the process. An economic analysis was performed on the ADF process. The result of the analysis was that ADF processing adds a cost of $0.35/lb at an annual production of 1,000,000 lb/year. With the economic analysis complete, all barriers to industrial scale-up for manufacturing have been removed.

Conclusions of this work are summarized below:

1) Results from the prior researcher were duplicated for nylon-12/glass fiber ADF composites. The alignment of sized fibers was found to be sensitive to changes in the relative humidity of the processing environment. All glass fibers were housed in a humidity chamber (relative humidity > 90%) prior to alignment. Carbon fibers did not exhibit this phenomenon due to the large intrinsic conductivity of the fiber. All fiber systems studied showed a mechanical property improvement of at least 70% for both strength and modulus.

2) The ADF process was modified to process conductive (i.e. carbon) fibers. The key modifications were to further separate the electrodes, add a compliant pad to the deposition plate to reduce fiber bouncing, and increase the distance between the upper and lower set of electrodes.

3) Data was collected for alternate polymer matrix ADF composites. PET and PP/E-Glass ADF composites were manufactured and tested.

4) The fiber orientation distribution of the ADF composites was shown to be, ~ 70% of fibers were aligned +/- 20° and nearly 80% within +/- 30°.

5) E-glass fibers were oriented to a higher degree than the carbon fibers. The fiber orientation parameter, f_p, of the glass fiber ADF composites was between 0.62 – 0.65, while the carbon fiber ADF composite, f_p, was between 0.52 – 0.58. This indicates that the carbon fiber process has not been fully optimized and further work needs to be done in this area to address this.

6) A study of the drapeability of the ADF composites was conducted. Three molds of various radii of curvature were constructed to investigate how well ADF composites can be formed into complex shapes. Molds with radii of 1/8", ¼", and ½" were made for use with nylon/glass fiber ADF composites. Results indicate that fiber length should be longer than the radius of curvature

to get the strength benefit of ADF composites. Based on this result, commercial use for complex shaped parts should use a distribution of fiber lengths to maximize part strength.

7) Micromechanical models were employed to predict the mechanical properties of finished composites. The Halpin-Tsai equations were used with two different reinforcement types, filament and bundle. It was found that 'filament' reinforcement predicted the composite properties better than that of bundle reinforcement.

8) A detailed economic analysis was completed on the ADF process, for both the laboratory scale (18,000 lbs/year ADF composite), and industrial (1,000,000 lbs/year). Cost added per pound for laboratory scale processing was $6.51/lb, while the industrial scale reduced the cost to $0.35/lb.

6.2 Future Work

During this work, several off-shoots were discovered that were worthy of investigation at a future date. Below are summary of these ideas:

1) Examine the conductivity of fibers sizings and how they affect alignment of fibers. Of the three different sizings on the fibers used in this research,

it appeared that the most hydrophilic sizing was the easiest to align in the process chamber.

2) Further work needs to be done on the drapeability of ADF composites. The results here should be compared to random composites to see the benefit of ADF processing. Also, carbon fibers should be investigated to see whether their increased stiffness will allow them to be processed in this fashion. More mold geometries should be investigated beyond the single 'L' shape.

3) Process limitations, such as fiber flow through the chamber needs to be studied to see where the drop-off in alignment occurs. There will be a finite density of fibers in the chamber that when exceeded results in a degradation of orientation.

4) Construct an industrial proto-type, which would consist of several alignment chambers in series to build-up the preform thickness. What affect would this have upon the final orientation?

5) Investigate alternative strategies of aligning fibers in the z-axis for use in adhesives. If fibers could be aligned perpendicular to the adhesive joint, a higher bonding strength and rigidity would be observed. One could take advantage of the phenomenon where the fiber was pulled upwards to stand

on end by the electrode passing over it to make the fibers 'stand' up. An adhesive sheet passing below the electrode could then 'capture' this alignment for future consolidation in an adhesive tape.

6) Create a laboratory unit with more than one pair of electrodes in the processing chamber. By alternating power through the various sets of electrodes, a multi-directional laminate structure could be manufactured with discontinuous fibers.

7) Investigate advanced fiber/matrix systems used in the aerospace industry with the ADF process. While the critical parts of the aircraft will still need to constructed of the continuous fiber version of the advanced system, various non-critical parts could be fashioned from ADF versions to save weight and cost.

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