

Spray Deposition of Cork Reinforced Polyester

by

Benjamin J. Pope

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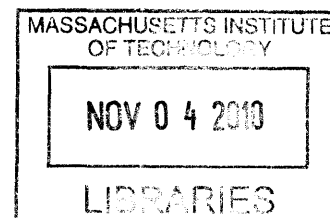
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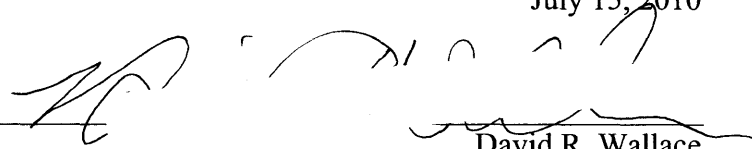
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ABSTRACT

The objective of this research is to enable large part or high volume manufacturing processes to make consumer or industrial products from a cork reinforced polymer composite, similar to current applications of glass reinforced polyester. The low initial investment and high flexibility of the spray lay-up process make it an attractive candidate to study. A spray lay-up apparatus was successfully constructed and employed in manufacturing parts from a hybrid material composed of granulated cork, chopped glass strand, and a polyester matrix. The material was tested for tensile and flexural properties following relevant ASTM standards. The material was found to have a tensile strength of 4.4 MPa and tensile modulus of 850 MPa. The flexural strength and modulus were 9 MPa and 830 MPa, respectively. Adding a fiberglass skin to the cork hybrid significantly improved its flexural strength. Additionally, a small turbine blade prototype was created as a proof of concept. It is recommended that further work focus on optimizing the hybrid material's properties, re-designing and optimizing the apparatus used for the spray-up process, and demonstrating material viability by manufacturing a cross section of a large turbine blade.

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1. Introduction

1.1 Objectives

The objective of this research is to enable large part or high volume manufacturing processes to make consumer or industrial products from a cork reinforced polymer composite. The approach was to begin by exploring existing composite manufacturing methods and applying them to manufacturing a cork polymer composite with particular emphasis on a spray lay-up technique analogous to chopped strand fiberglass production. A prototype apparatus for manufacturing will be designed and used to begin to optimize the process and manufacture material samples to test for relevant material properties (*e.g.* tensile and flexural strength and modulus). Finally, a prototype part was fabricated for demonstration purposes.

1.2 Cork: A Solution Looking for a Problem

1.2.1 Advantageous Properties of Cork

Cork is a material that has many properties that make it attractive for further study, particularly its thermal and mechanical properties. Its advantageous mechanical properties include low density, a high loss factor, resistance to surface wear and high elasticity. As a result, substantial energy absorption corresponding to relatively low stress values is possible. Furthermore, cork has low thermal conductivity. Therefore cork is capable of being an exceptional isolator from thermal, mechanical and acoustic sources. Additionally cork is a remarkably stable material and absorbs very little water even if fully immersed.¹

There are additional characteristics that highlight cork for potential use in consumer and industrial applications. In this era of “green” materials, cork is a sustainably produced and processed; it is a natural product harvested from the bark of the cork oak tree. Additionally, it is both biodegradable and recyclable.² Combining cork with other materials, as a component in a composite, offers even more potential. Cork has high wettability for non-polar fluids, including

compatibility with common resins such as polyesters, vinyl esters, and epoxies. Overall, this diverse range of properties makes cork an attractive material for many applications.

1.2.2 Existing and Potential Applications

Cork is best known for its use as a wine bottle closure, but there are many other applications. Cork's thermal and mechanical isolation properties have led to use in shoe soles and flooring materials, sealants, gaskets, automobile interiors, and coverings as mechanical and thermal insulation. Its low density has led to use as a filler material in building materials (*e.g.* gypsum board and plaster).³

Cork has high potential in other applications as well. Its damping properties and fatigue resistance make it ideal for increased use in automobile seats and door panels. The same properties could make cork attractive for use in boat hulls and other marine structures. Cork's low thermal conductivity and acoustic insulation properties indicate potential for use in consumer bathroom appliances like sinks, shower pans, and bathtubs. Industrial systems such as air handling ducts could also be composed of, or coated with, a cork-infused material, which would provide thermal and acoustic isolation.

While all industries would do well to consider sustainable product design, especially in the current consumer climate, energy technologies are under particular pressure to design for sustainability. The wind industry is one particular market segment that is striving for grid-parity and needs cost savings in all aspects of its designs. The salability of a renewable material is particularly important in the small-wind industry, which is looking for novel materials for use in turbine blades.⁴ A cork composite material is a possibility particularly for its high damping properties and may be considered because of its reasonable mechanical properties, high stability, and low density.⁵

Overall, given the availability and low cost of cork, products that are looking for a low cost solution in medium to high production are potential opportunities for use of cork composites with new manufacturing methods. Applications that can leverage cork's unique properties are uniquely suited to innovate substantially.

1.2.3 Manufacturing Process

While on its own cork makes an excellent damper, insulator and isolator, its use in consumer and industrial products is better suited to use as a component in a composite material that would contribute strength and stiffness. Therefore, this work studies existing composite manufacturing methods and explores how a cork composite could be integrated into these solutions. Particular emphasis is placed on low investment and high throughput systems, with preference given to systems that are currently used for analogous products, especially fiberglass spray-up.

2. Background Material

In order to achieve the stated objectives, it is necessary to understand the component pieces including the basics of designing composite materials, cork as a raw material, and existing manufacturing processes for glass fiber reinforced polymers.

2.1 Designing Composite Materials

2.1.1 Introduction to Composites

Composites are created by embedding fibers or particulates in a continuous matrix of polymer, ceramic, or metal. In general, composite materials are engineered for high stiffness and strength per unit weight, but they are sometimes meant to leverage certain advantageous properties of their constituent materials.

Traditional composite materials use a polymer, metal or ceramic matrix, depending on the material needs. Metal and ceramic matrices are especially useful in high temperature environments. For this application, we focus on polymers, both thermosets and thermoplastics. Thermoplastics exhibit good fracture toughness but thermosets are stiffer due to cross-linking of their polymer chains. Because of their high stiffness and lower processing temperatures, thermosets have become the predominant type in use, most notably polyesters, vinyl esters, and epoxies. Of the three, polyester is the lowest cost and has adequate strength for most applications, in addition to having excellent corrosion resistance. Epoxies have better mechanical and thermal properties, but at higher cost. Vinyl esters fall in the middle ground between the two, both in material properties and cost, but have some drawbacks such as high shrinkage during the molding process.

Reinforcing materials are plentiful and available in a wide range of strengths and costs. They can be either particulates or fibers, and the fibers can be discontinuous and randomly oriented, or continuous and woven into an engineered pattern. Commonly used materials include

glass, carbon, and aramid (*i.e.* Kevlar) fibers. Reinforcing materials are usually selected for their high stiffness and/or high strength. These properties are tunable, and can be applied to a wide range of specialized uses and applications; aeronautics, consumer goods, automotive, boats, and structural building materials all utilize composites.⁶

Some composites also incorporate natural fibers such as jute, flax, hemp, sisal, coconut fiber, and banana fiber. They are selected because they are environmentally sound and because they are inherently better suited to acoustic damping than glass or synthetic fiber composites. This makes them particularly attractive for use in thermoformed parts in automotive interiors. There have been, however, some challenges in their adoption. Without surface treatments, they often do not adhere well to the matrix in which they are enveloped. Additionally, natural fibers can char and degrade high temperatures, and so can be inappropriate for use with some thermoplastics.⁷

Engineering the directionality of the fibers and weaves allows engineers to selectively strengthen certain axes of finished parts. Many filled plastics, though not often referred to as composites, do in fact fit the definition and are strengthened by particulates. Due to their random orientation and distribution, polymers that are filled with particulate materials can be considered to be isotropic.⁸

2.1.2 Material Selection Process

Materials are a crucial part of the design process. Proper material selection enables both the form and function of a product. The material selection process requires the knowledge of the performance requirements of the component under consideration. The type of loading, mode of loading, operating conditions, desired lifetime, cost, and aesthetic concerns are common concerns.

Depending on the application, materials are chosen to optimize certain variables, stiffness per unit weight or cost per unit volume, for example. Among other requirements, a wind turbine blade needs high stiffness, low density, long fatigue life and environmental resistance. The critical structural trait is a tradeoff between stiffness and density with a merit index, M_b , based on E , the elastic modulus in tension, and ρ , the density of the material, shown in Equation 1.⁹

$$M_b = \frac{E^{1/2}}{\rho} \quad [1]$$

Ashby charts can be very helpful in illustrating the material selection process. In these charts, material traits are plotted in an x-y plane in order to compare many different materials at once. Figure 1 shows a comparison between density and the tensile elastic modulus with trend lines illustrating isoclines of the merit index established in Equation 1.

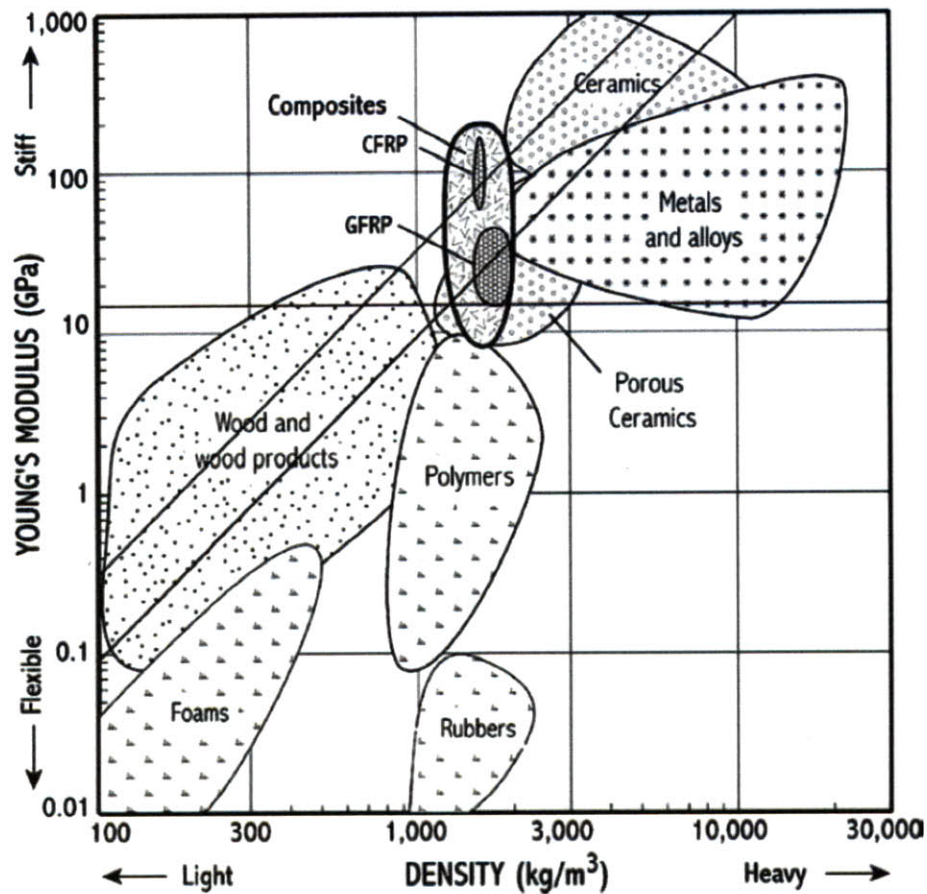


Figure 1 Ashby chart comparing density and elastic modulus with merit lines representing Equation 1.¹⁰

If, instead, the design goal is a stiff beam for minimum cost, a different merit index and Ashby chart aid the decision making process. In this case, the volume and weight are not the limiting factors, except for how they relate to price. In this case, Equation 2 shows the relevant merit index, M_b , based on the static modulus in tension, E , the density, ρ , and cost per unit volume, C_v .

$$M_b = \frac{E^{1/2}}{\rho C_v} \quad [2]$$

Just as in the previous case, an Ashby chart aids in visualizing the many material choices. An example of the above merit index is included on the chart in Figure 2. Those materials above

and to the left of the index line meet the prescribed minimum requirements for this hypothetical case.

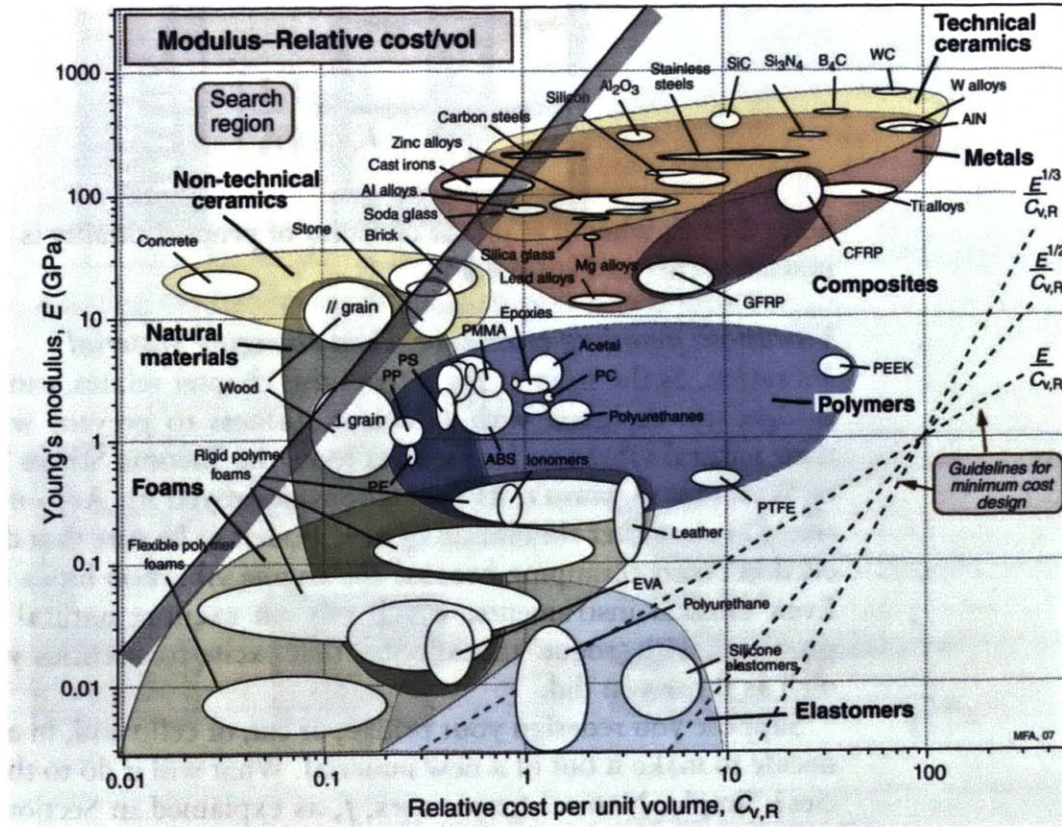


Figure 2 Ashby chart demonstrating cost vs. stiffness tradeoffs.¹¹

2.1.3 Design of a Composite Material

There are spaces on the Ashby chart that traditional materials do not cover. Creating new solutions such as sandwich structures, foamed materials and composite materials can fill these spaces. Creating a composite allows the leveraging of certain properties of each of the constituent materials. Predicting the exact properties of composite materials is difficult, and governed by many factors on both the macro- and micro-scale. Properties such as density and stiffness, which are of particular interest in designing composite materials, can be estimated or bounded.

Calculating the density of a composite material may be conducted with relative ease.

Knowing the volume fraction of the reinforcement (f) and its density (ρ_r), as well as the density

of the matrix (ρ_m), allows the composite density (ρ_c) to be calculated exactly via the rule of mixtures (an arithmetic mean, weighted by volume fraction), as show in Equation 3.

$$\rho_c = f\rho_r + (1 - f)\rho_m \quad [3]^{12}$$

The modulus is bracketed by upper bound and lower bound estimates. The upper bound is similarly predicted by the rule of mixtures. Equation 4 reflects the theory that all components strain at the same rate, like springs in parallel.

$$E_U = fE_r + (1 - f)E_m \quad [4]^{13}$$

The lower limit of the elastic modulus would occur if the constituents of the composite material act perfectly in series. This does not follow the same rule of mixtures, but is shown in Equation 5.

$$E_L = \frac{E_m E_r}{fE_m + (1 - f)E_r} \quad [5]^{14}$$

By combining these predicted traits, it is possible to more exactly engineer a material and predict its usefulness. An Ashby chart shown in Figure 3, with the same axes as shown in Figure 1, is simplified and magnified for the purpose of studying the properties of a ceramic reinforced polymer composite. The upper and lower limits of the elastic modulus are plotted for the continuum of possible composite formulations, thereby demonstrating the theoretical limitations of the material.

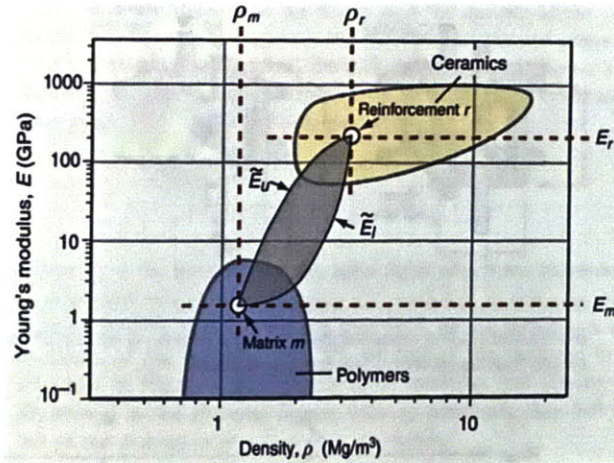


Figure 3 Ashby chart of density versus elastic modulus for a ceramic reinforced polymer.¹⁵

2.2 Raw Material: Cork

2.2.1 Manufacturing

Cork is a grown, natural product. It is harvested from the bark of *Quercus suber*, more commonly referred to as the cork oak. Cork oak is suited to a temperate semi-arid climate, so most of the world's cork comes from around the Mediterranean, especially Portugal and Northern Africa. The bark of the tree is harvested approximately once every nine years for the life of the tree, which can be up to 200 years. Following harvesting, cork is boiled in order to improve its mechanical properties. Boiling relieves internal growth stresses and decreases the corrugations in the cell walls, thereby increasing cell uniformity. Planks are then trimmed and sorted based on quality.¹⁶

Higher quality cork is cut and stamped for use in single-piece rings and cylinders to be used as gaskets and stoppers. Nearly 60% of all harvested cork is used in traditional wine stoppers. The lower quality cork, and higher quality cork that is leftover after stamping, is then granulated and sorted into several common classifications ranging from a powder to granules up to 5mm in diameter. Granulated cork is used to make agglomerated cork materials or more highly engineered materials, from linoleum to ablative coatings for space reentry vehicles.¹⁷

2.2.2 Material Properties

The cellular structure of cork has garnered it the label of “nature’s honeycomb.” The air pockets within the materials, and intervening thin walls, contribute to cork’s low thermal conductivity and high-energy absorptive capability. Both of these properties compare favorably to polyurethane and polystyrene foams currently available.

While there is significant variation in the quality and material properties of cork, which are especially dependant on density, this work has to do with granulated material. Despite being an anisotropic material, agglomerated granulated cork is assumed to be in a random orientation, which can be approximated by lumped isotropic properties. Compression is the most studied mechanical property of cork, but other mechanical properties such as tension, torsion and bending are important in wider applications. Under compression, cork does not fracture; the result of compaction is the collapse of the cellular structure and its densification. Cork fracture happens under tensile and torsional loading. Each of these modes is of potential interest, especially the tensile properties.

Under compression, cork acts linearly for the first 5-7% strain. Until about 50% strain, cork deforms elastically, beyond which densification of the material occurs. The stress-strain relationship is shown in Figure 4. The Young’s modulus in compression is 18.3 MPa in the radial direction, 16.9 MPa in the axial direction, and 12.3 MPa in the tangential direction.¹⁸

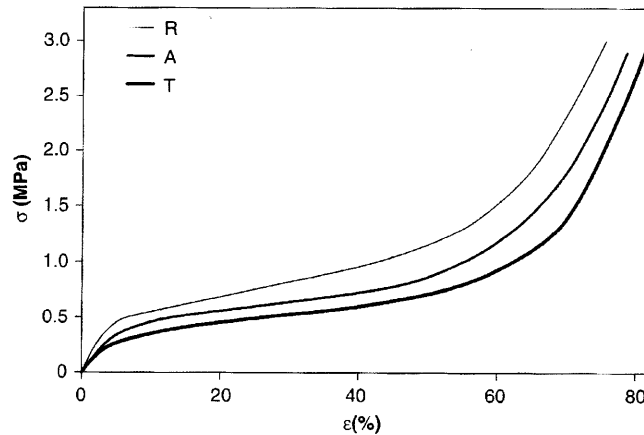


Figure 4 Stress-strain curve for cork in compression in the radial, axial, and tangential directions¹⁹

The tensile properties of cork are not as studied, and are more sensitive to factors such as strain rate and moisture content. Unlike in compression, in which the failure mode is densification, in tension the cellular walls are pulled and the material fractures under relatively small strains, as shown in Figure 5. The Young's modulus in tension is 31.7 MPa in the axial direction, 23.9 MPa in the tangential direction, 31.2 MPa in the radial direction. Its ultimate tensile strength is between 0.8 and 1.0 MPa. These properties can be highly dependent on imperfections within the cork, however.

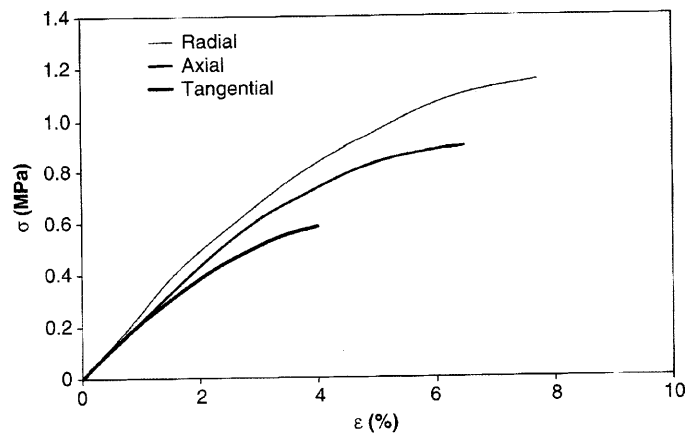


Figure 5 Stress-strain curve for cork in tension in the radial, axial, and tangential directions.²⁰

For some applications, the bending characteristics of cork will be predominant. In three point bending tests, cork ruptures at approximately 10% strain and 1.0 MPa. Consistently, the

cork ruptures on the tensile side of bending. Therefore, it is the tensile properties of cork that limit its performance, and by extension, potentially limit the resultant composite.

2.2.3 A Natural Material

Especially when compared to other composite filler materials, cork is an extremely environmentally friendly alternative. As discussed previously, cork is extracted by stripping the bark of the cork oak tree. Cork oak forests play an important role in stabilizing their local ecosystem and providing an animal habitat as well as fixing carbon dioxide. In addition to being responsibly grown (cork oak trees protect soil erosion and desertification in their habitat), cork itself is easily recycled and is biodegradable.²¹ In contrast, glass fiber is energy intensive to manufacture and polymer fibers are petroleum-based products.

2.3 Fiber Reinforced Polymer Composition and Manufacturing

In order to apply cork in a composite material, it is best to understand fiber-reinforced polymers, more specifically glass fiber reinforced polymers, since they are the most relevant type of composite in this case, especially given the envisioned applications.

2.3.1 Glass Fiber Reinforced Polymer (GFRP) Composition

GFRP is a generic term applied to any polymer, either thermoplastic or thermoset, that is strategically strengthened by the inclusion of glass fibers, thereby distinguished from polymers strengthened by organic or polymer fibrous materials. GFRPs are more commonly known as “fiberglass” which can actually refer to a range of materials including glass reinforced polyester, vinyl ester, and epoxy. Polyester is the most commonly used because of its adequate mechanical properties, excellent environmental resistance, and relatively low cost.

The strength of the final product, however, is dependent on the positioning of the fibers within the matrix. The strongest materials employ mats of woven fiber. Randomly oriented

fiber, on the other hand, produces lower strength parts. Different manufacturing methods are better suited to different types and weaves of glass fiber.

2.3.2 Manufacturing Processes

There is a range of manufacturing techniques for making products from GFRP. They can generally be classified into two areas: open mold and closed mold. Their names are relatively self-explanatory. Closed mold processes are similar to injection molding in which two halves of a mold are closed to make the part, and then opened to extract the part. Open mold processes have a one-piece mold, either male or female, that dictates that part's shape. There are many popular open mold processes that have been developed: hand lay-up, spray lay-up, CNC tape laying or filament winding, and pre-preg lay-up. These processes can all be combined with vacuum bagging in order to provide isostatic compression on parts while they cure.

In general, while many closed mold processes can be entirely automated and result in very high part throughput, open mold processes are attractive because of their relatively low cost tooling and substantial flexibility in design, especially for relatively large parts. There are drawbacks, principally that it is only possible to highly finish one surface and that part quality is dependent on the operator, assuming automation is not employed. One application of particular interest is wind turbine blades, which are made in open molds. Similarly, consumer products such as boats, bathtubs, and shower pans are all made in open mold processes. Therefore, this study will focus on open-mold procedures.

Independent of which open mold process, the first steps are the same. The mold is waxed and coated with a release agent to allow the part to de-mold. The mold is then sprayed with a gel coat resin, which will give the part an excellent surface finish. The gel coat is allowed to cure before the fiber and resin are applied to the mold.

Hand lay-up is an open mold process, as shown in Figure 6 with a female mold. Hand lay-up is the most common process for making fiberglass composite products. After a gel coat is sprayed, fabric is placed in the mold. Each ply is coated with catalyzed resin which is then worked into the fiber with brushes, rollers and squeegees to ensure complete wet-out and compact the laminate. In general, hand lay-up is suitable for low production rate operations.

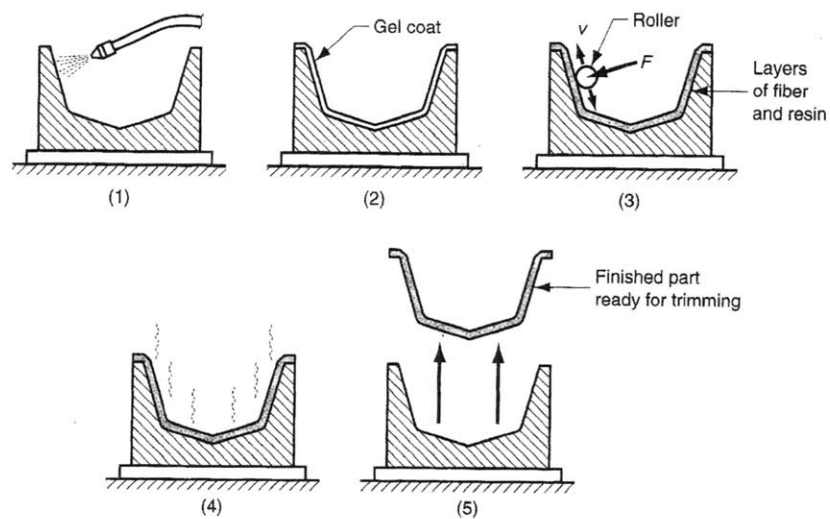


Figure 6 Hand layup procedure.²²

In a spray lay-up, or spray-up, operation, following gel coat application, resin and chopped strand glass fiber are simultaneously sprayed into the mold. Typically, a chopper gun chops continuous strand roving and sprays it directly into the resin stream, as shown in Figure 7 Spray-up method for GFRPs. Alternatively, a lower cost chopper-dedicated gun can be used to spray the glass fiber while a cup-gun is used to deposit the resin separately. In both cases, workers use rollers to compact the material and ensure complete wet-out. A core material can be added with a secondary fiberglass layer to embed the core between the laminates. The part is then left to fully cure before being removed from the mold.

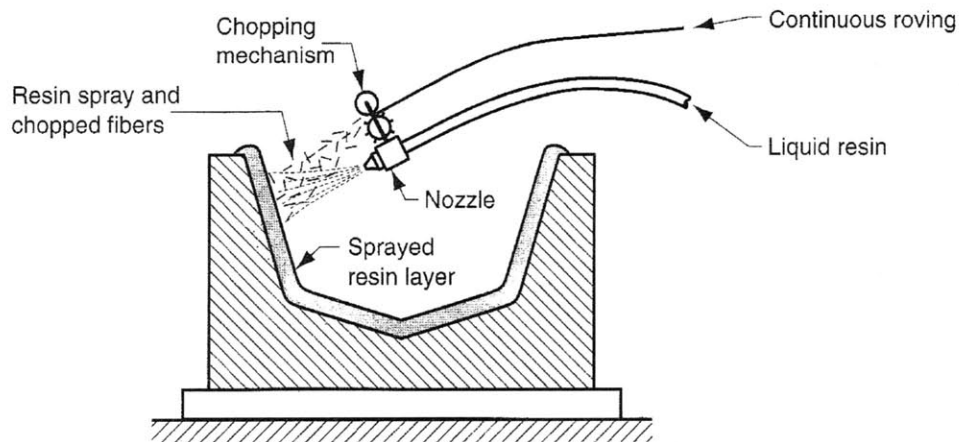


Figure 7 Spray-up method for GFRPs²³

The spray-up process is capable of a greater production rate, more isotropic parts (assuming highly skilled operators), and can often use more complex molds than hand lay-up processes. Furthermore, spray-up processes are less labor-intensive and even potentially portable. There are drawbacks to the process, however. From an environmental point of view, styrene and other volatile organic compound (VOC) emissions are troubling. Emissions can be mitigated using low-styrene resins or high-volume low-pressure (HVLP) spray guns. HVLP guns also limit overspray. This can be a problem for small parts in particular, in which the amount of material wasted in overspray can be significant compared to the material required to make the part. Finally, it is important to note that because the reinforcing fibers are short and in random orientation, spray up is often not suitable for products with high structural requirements.²⁴ Some parts that are commonly made via spray-up are boat hulls, bathtubs, sinks, and shower pans.

2.4 Cork Reinforced Polymers

2.4.1 Previous Work on Cork Reinforced Polymers

Cork reinforced polymers have seen use in a wide range of applications. Agglomerated cork with a urethane binder is in use in applications like bulletin boards and shoe soles.

Recently, cork has also become popular as a principle component in flooring material, instead of a minority component like it is in linoleum.²⁵

Given the growing popularity in alternatives to traditional corks for wine closures, there has been increasing research and development of alternative uses for cork. Amorim Cork has been marketing an agglomerated cork material for use in hand lay-up manufacturing named Corecork. They have developed prototypes of a vertical axis wind turbine, a competition kayak, light aircraft components, aluminum sandwich panels and ceramic floor tiles.²⁶

Scholarly work on fiber reinforced polymers including cork has also been published in recent years. There has been some success with a granulated cork and epoxy composite within carbon fiber skins to make a sandwich construction, which showed excellent structural properties and controllability. These samples were manufactured by compression molding in a hot press.²⁷ At MIT, granulated cork, epoxy, and glass microfibers were combined within woven glass fiber skins. Emphasis in the studies was focused on exploring the cork and reinforcing fiber mix ratios. These samples were made via hand lay-up and vacuum bagged during curing.^{28 29}

2.4.2 Selecting a Manufacturing Process

As discussed previously, cork is attractive for use in a composite material for many applications. Of particular interest in this research are those products that can be made at low cost in medium to high volume while accommodating large parts in potentially complex geometries. Previous work has focused on hand lay-up and compression molded techniques. Therefore, this work focuses on the spray lay-up manufacturing process. This process can be used to make some of the products discussed earlier including wind turbine blades, air handling systems, automotive door panels, and bathroom components.

2.4.3 Predicting Properties of a Cork and Glass Fiber Reinforced Polymer

Previously, the rule of mixtures and a variation thereof were applied to predicting the properties of a composite material. The same reasoning can be applied to a hybrid material that is composed of three or more parts. As before, the rule of mixtures yields the density and upper limit of the tensile elastic modulus as shown in Equations 6 and 7, respectively. As before, f designates the fiber volume fraction, ρ designates a density and E designates the elastic modulus in tension. Subscripts are chosen for this particular application: c represents cork, g represents glass fiber, m represents the matrix material, and h represents the complete hybrid material.

$$\rho_h = f_c \rho_c + f_g \rho_g + (1 - f_c - f_g) \rho_m \quad [6]$$

$$E_U = f_c E_c + f_g E_g + (1 - f_c - f_g) E_m \quad [7]$$

The lower limit of the elastic modulus represents the case in which the constituent materials act like springs in series. Therefore, the reciprocal of the resultant stiffness is the sum of the reciprocals of the components. Weighting the components by their respective volume fractions yields Equation 8, below.

$$E_L = \frac{E_c E_g E_m}{f_g E_c E_m + f_c E_g E_m + (1 - f_c - f_g) E_c E_g} \quad [8]$$

Because there are now three components, the Ashby chart is not as simple to graph fully. Rather, the envelopes for different solid volume fractions are shown. Figure 8, below, shows the upper and lower limits of a hybrid material's tensile modulus at three levels of total solids content by volume. (NB In this case, the cork and glass fiber comprise the "solids.") The 100% solids content level is physically impossible, but is demonstrative of the absolute limits of any such material. 50% solids volume fraction is reasonable for hand lay-up applications while 30% is closer to the fiber volume fraction of fiberglass parts created via spray-up manufacturing. Material properties for cork³⁰, E-glass fiber³¹, and polyester³² were taken from the cited sources.

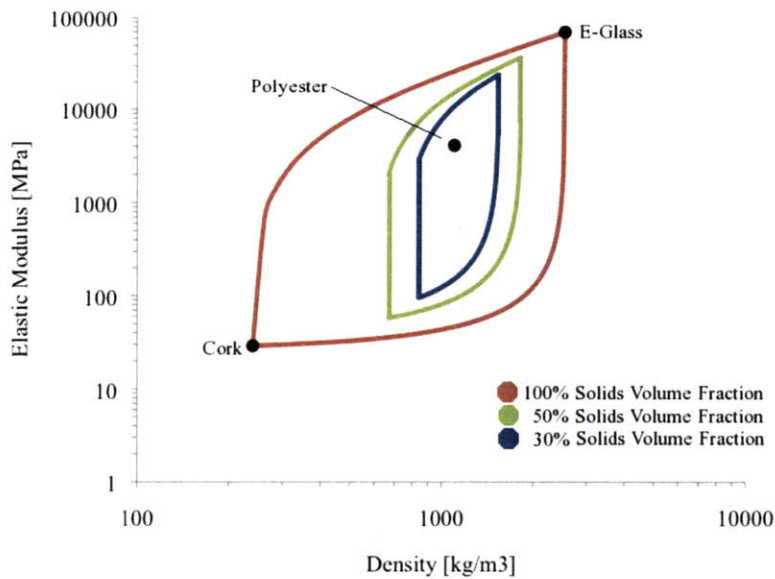


Figure 8 Predicted density versus tensile elastic modulus of a cork/E-glass/polyester hybrid material.

A similar process can be carried out to show data like that shown in Figure 2 (an Ashby chart showing the relationship between specific cost and stiffness). This predicted relationship is shown in Figure 9, below. Price data for cork (\$2.38/kg)³³ and E-glass fiber (\$1.11/kg)³⁴ are taken from correspondence with corporate representatives and polyester price information (\$5.92/kg)³⁵ is taken from online sales material.

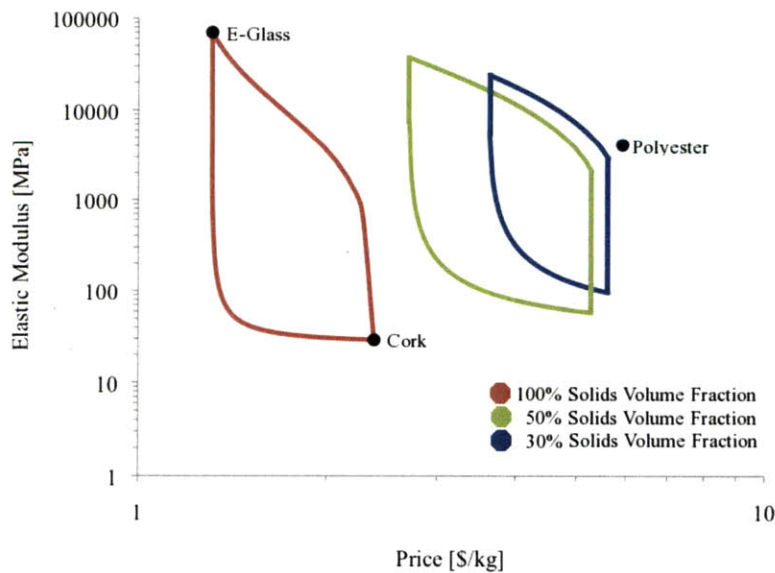


Figure 9 Predicted cost per kg versus tensile elastic modulus of a cork/E-glass/polyester hybrid material.

3. Sample Preparation and Evaluation

3.1 Sample Preparation

3.1.1 Spray-up Apparatus

The inspiration for the apparatus to spray-up cork composites comes from existing fiberglass chopper guns used for spray-up manufacturing. More specifically, the selected strategy is to propel the solid materials into the atomized resin stream in order to simultaneously wet and deposit the solids. For a refresher, refer to Figure 7, a schematic of the fiberglass spray-up process. Like existing spray-up operations, after spraying, the composite is then rolled or otherwise compacted in order to eliminate voids and ensure even wet-out.

For this proof of concept, the gun and resin deposition were selected to minimize cost while retaining necessary functionality. Therefore, catalyzed resin was propelled from a pressurized container and sprayed with an externally air atomized spray gun (*i.e.* Binks model 80-295 “Steady-Grip” pressure container and 2001 gun with 68SS nozzle). This apparatus did have drawbacks (including overspray and the short pot-life of catalyzed resin) but was easily available and adequate for the proof of concept phase.

In fiberglass spray-up, continuous roving is chopped as it is sprayed. For a cork composite using granulated cork, an analogous solution is impossible. Therefore, an air operated venturi conveyor (*i.e.* Exair Corporation Light Duty Line Vac) is used to propel the solids from a hopper to the gun nozzle. A photo of the entire setup used to create all samples is shown in Figure 10, below.

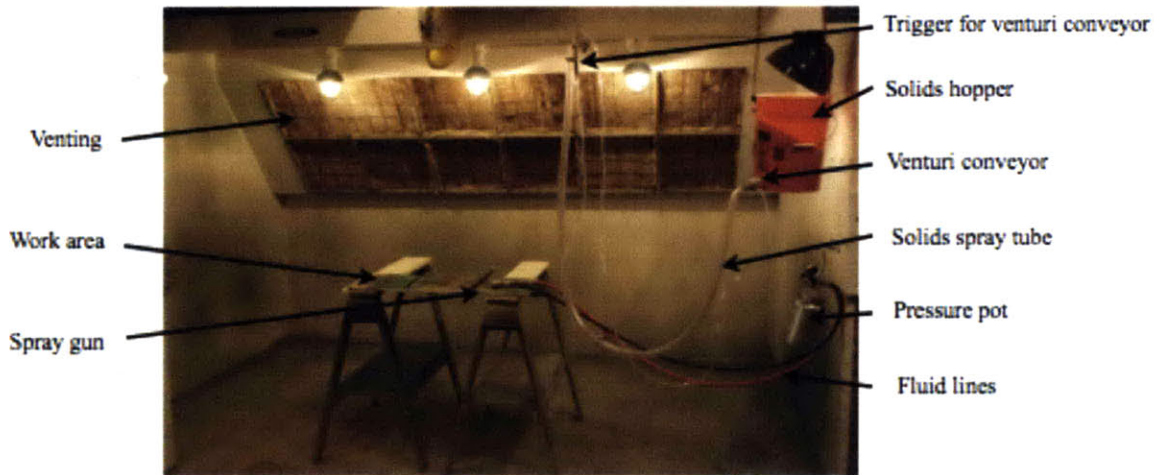


Figure 10 Annotated picture of the work area. Each spray line is independently operated.

Solids are mixed and loaded into the hopper. In this case, the hopper is an orange bucket with a cardboard cone on the interior in order to direct the flow of material. A small motor with an eccentric mass is attached to the outside of the hopper in order to vibrate it. The solids stream is sprayed and controlled entirely independently of the resin stream. The solids stream is controlled by a valve that opens or closes an airline to the venturi conveyor. The pressure in this line is controlled by a regulator with +/- 0.5 psi resolution. The resin flow is controlled by two separate pressure inputs. One adjustment controls the atomization of the resin; this is generally a regulator from the line-pressure source. This pressure should be set as low as possible while still achieving adequate coverage and atomization. A regulator on the pressure pot, which is downstream from the atomization regulator, controls the rate at which the resin is dispensed.

In actual operation, one of the primary challenges is to prevent clogging of the system, which was found to happen both before and after the Line Vac. Paying special attention to keeping the line kink-free was particularly important. Any necking, including a nozzle to direct the cork while it exits the line, causes clogging. A tube 1 inch in diameter was found adequate for the setup. In industry, solutions to similar problems rely on air fluidization and mechanical vibration. To prevent plugging at the Line-Vac input, it was found that mechanical vibration was

easier to implement and proved to be reasonably effective in this case. Building static electricity is also a concern, so the system should be grounded if possible. (This is recommended for all standard spray equipment.)

During manufacturing, the material proportions need to be mixed at a controllable and tunable ratio. In this case, this is carried out by individually controlling the amount of resin and solids being sprayed; the solids are premixed before being loaded into the hopper. As stated previously, the resin spray rate is controlled by adjusting the pressure of the pre-mixed pot. There is a linear relationship ($r = 0.99$) between the pressure in the pot and the polyester spray rate, as shown in Figure 11.

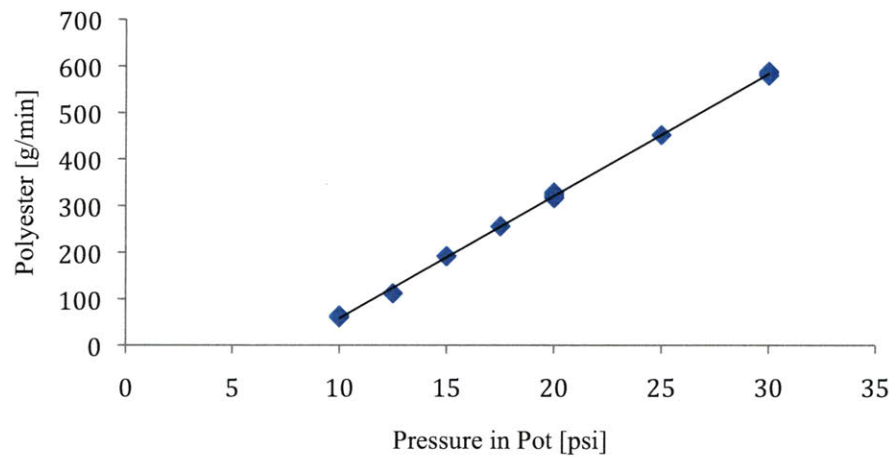


Figure 11 Resin sprayed versus pressure in pot, with linear fit ($r = 0.99$).

Solids are similarly controllable by adjusting the pressure powering the venturi conveyor. However, following preliminary testing, it became clear that the lower limit of the solids spray-rate was still too high compared to the upper limit of the resin spray rate (in order to achieve a reasonable solid to resin mix ratio). Therefore, in this implementation, an approximation was used such that one pass of the resin and solids would be sprayed together and then subsequent coats of resin were applied in order to achieve the proper mix ratio.

It is important to mention operator safety in this process. The materials involved can irritate both the skin and the lungs. At the very least, gloves, safety glasses and an organic vapor respirator are required. Full body covering personal protective equipment is recommended.

3.1.2 Sample Composition

For the exploration of this spray-up process, polyester was selected as the resin component. Initially, the decision was guided by the prevalent use of polyester in industrial spray-up processes and other GFRP products. High performance epoxies have been used in cork composites both at MIT and elsewhere, but difficulty in spraying due to its viscosity made its selection less attractive. A polyester capable of long shelf life and room temperature curing, Fiberglast Isophthalic Polyester Resin, was eventually selected.

The mixture of cork granules and glass fibers was guided by previous work.³⁶ A combination of two sizes of cork granules (bulk diameters of 1 mm and between 4-5 mm were mixed in equal parts by weight. 1/4" chopped glass strand fibers were then added to the mixture until the ratio, by weight becomes 1.5:1.5:2. The appropriate resin content was determined by experimentation. It was assumed that a material with the smallest amount of resin possible, while still eliminating voids in the manufacturing process, would lead to the strongest and stiffest material per unit weight. In traditional fiberglass hand lay-up methods, fibers can reach up to 70% by volume, while in spray-up fiber is limited to 30-35% of the total volume of the composite.³⁷ This was the starting place for experimentation, with a final selection of approximately 50% solids by volume. For repeatability, metering is carried out by mass, translating to a ratio of 6:1 polyester to solids.

4. Prototype Manufacturing

4.1 Mechanical Testing

While normally an open-mold process, for consistency and controllability, specimens for testing were made with two flat sides. The sides of the “molds” were left open so that no compression would be applied to the curing composite. The cork composite material had a density of 732.25 kg/m^3 . All panels were made to satisfy the referenced ASTM standards (see below). Samples referred to as “cork hybrid” measured 6.5 mm thick. Samples referred to as “cork hybrid with single fiberglass skin” contain the same thickness of cork with an additional layer of 4 oz. woven fiberglass cloth³⁸ wetted through with additional polyester resin adding approximately 0.2 mm in thickness. The weave is aligned with the axis in which the load is applied in testing.

4.1.1 Tensile Test: Cork Hybrid

Tensile testing was carried out according to ASTM D-638 “Standard Test Method for Tensile Properties of Plastics” since it applies to both filled and reinforced plastic materials. For background material, please refer to the documentation in the standard.³⁹ The goal of the testing was to measure the tensile strength and modulus of the cork hybrid material.

Testing was carried out on an Instron 5567A load frame outfitted with a 5kN load cell and 50kN wedge grips outfitted with finely gnurled faces. The machine was controlled with Instron’s Bluehill 3 software package. Specimens, in the shape prescribed by the standard, were tested to rupture at a rate of 5 mm/min. Images of the setup, and raw data are shown in Figure 12 and Figure 13, respectively.

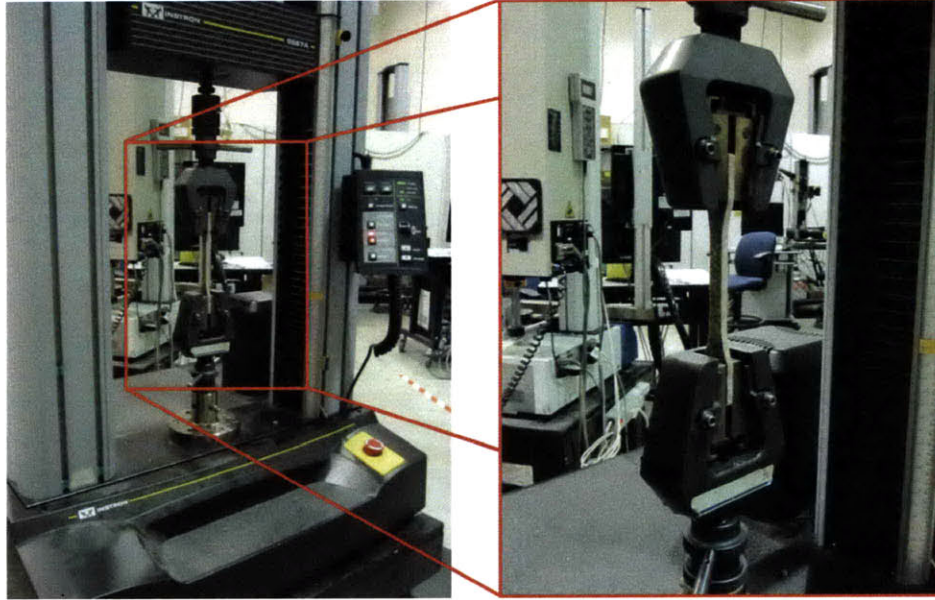


Figure 12 Setup for tensile testing on Instron 5567A. Detail view is on the right.

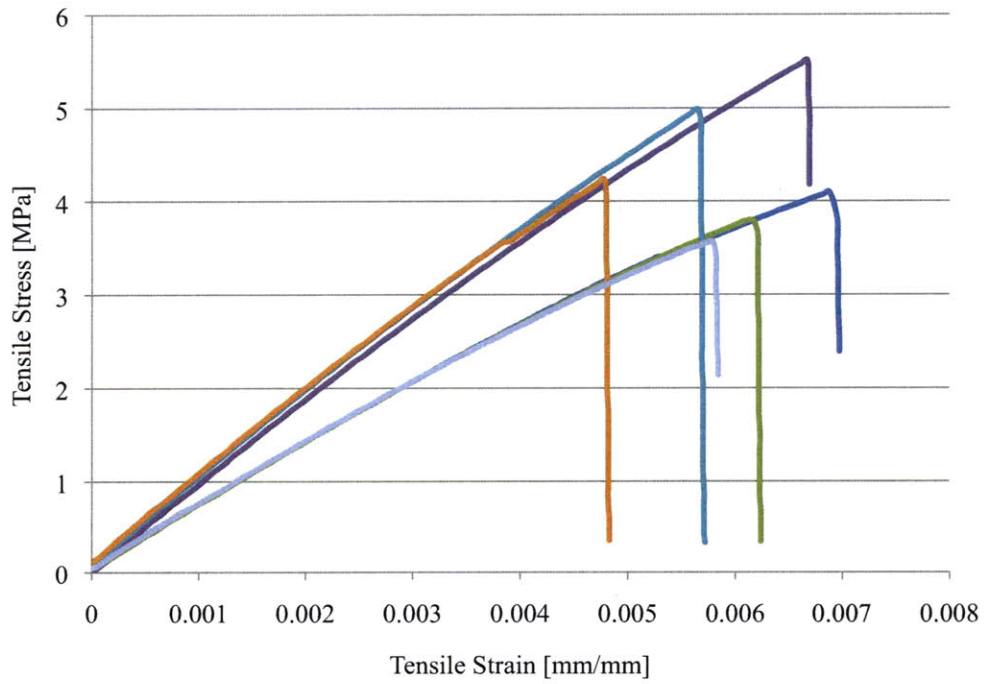


Figure 13 Results of tensile test of cork hybrid material according to ASTM D-638.

These raw data are converted into tensile strength and modulus measurements, as calculated by the Bluehill software. These data are available in Table 1, below.

Table 1 Tensile strength and modulus of cork hybrid material based on tensile testing.

Specimen	Tensile Strength [MPa]	Tensile Modulus [MPa]
1	4.09	730.00
2	3.79	718.21
3	5.51	955.10
4	4.98	991.39
5	4.24	991.43
6	3.56	713.10
Std. Deviation:	0.74	142.51
Mean:	4.36	849.87

From these raw data, it can be seen that there is an evident bi-modality. Examination of the specimens indicates that this is due to an imperfect mixing of the material. The stronger specimens tended to rupture at a cross-section that was composed of more polyester than the weaker specimens, which tended to have more cork in the cross section. Detailed photos are shown in Figure 14, below.

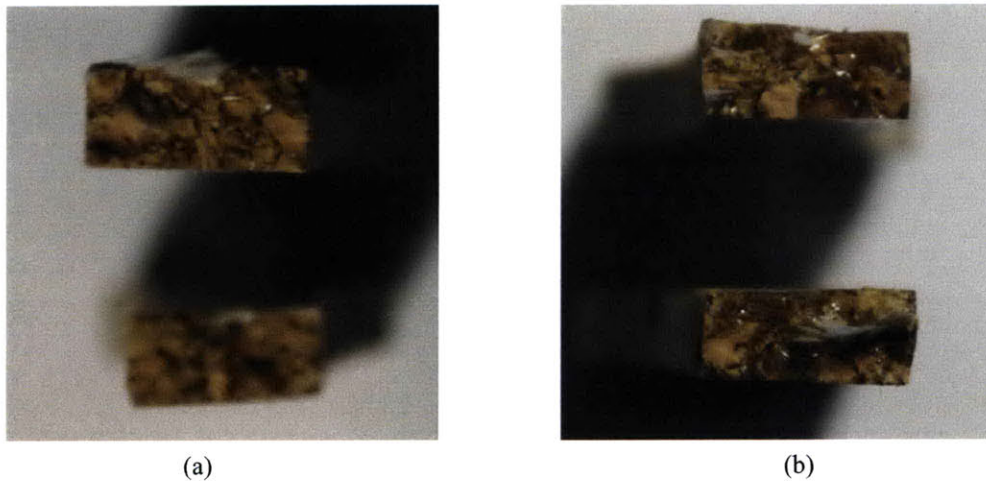


Figure 14 Failed specimens from tensile testing. (a) Specimen 6 (b) Specimen 3.

The root cause for the bimodality in stiffness is less evident. The stronger specimens tend to be stiffer and the weaker ones tend to be less stiff, so it possible that the cork fraction at the weak point in the sample dominates the performance of the specimen as a whole. Specimen 5 is an interesting case, however, because it falls in the middle ground of strength but is among the stiffer specimens. This contradicts the previous explanation. Another potential cause is the

orientation and bonding strength of the glass fibers in the failure region. If the fibers were aligned in the direction of pull they could contribute more substantially to stiffening the material.

4.1.2 Tensile Test: Cork Hybrid with Single Fiberglass Skin

Tensile testing of the cork hybrid with a fiberglass skin was carried out using the same equipment and test method as discussed previously. Raw data are available in Figure 15, below.

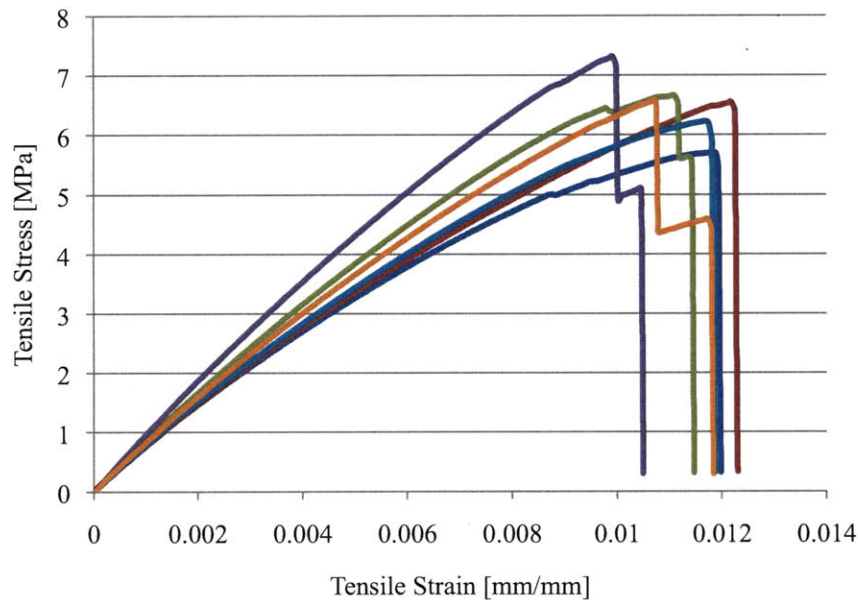


Figure 15 Results of tensile test of cork hybrid material with a single fiberglass skin.

One interesting component of the stress vs. strain curves is that in several cases there is an initial failure followed by a second, subsidiary failure. From visual observation of the tests it was evident that this phenomenon is caused by the bulk cork hybrid and the polyester in the fiberglass skin failing at different times. The glass fibers themselves did not rupture. Instead, the polyester failed, allowing the woven fiber to expand. The cork composite failed secondarily. The raw data were converted into tensile strength and modulus measurements, as calculated by the Bluehill software. These data are available in Table 2, below.

Table 2 Tensile strength and modulus of cork hybrid material with a single fiberglass skin.

Specimen	Tensile Strength [MPa]	Tensile Modulus [MPa]
1	5.70	705.75
2	6.55	717.67
3	6.66	826.03
4	7.31	936.08
5	6.23	741.81
6	6.57	787.12
Std. Deviation:	0.53	86.24
Mean:	6.50	785.75

In comparing these results to those found previously for the cork hybrid specimens, the pattern of stronger specimens having a higher proportion of fiberglass at the breaking point holds true. The fiberglass cloth apparently added strength to the overall material. However, it is unclear why these specimens were found to be less stiff. It is possible that this is a function of the bimodality of the previous specimens because the majority of the skinned specimens are approximately equal to the lower of the two modes found in previous testing. It is also possible that there was some delamination between the layers, as exhibited by the failure mechanism discussed above.

4.1.3 Three Point Bending Test: Cork Hybrid

Flexural testing was carried out according to ASTM D-790 “Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials.” For background material, please refer to the documentation in the standard.⁴⁰ The goal of the testing was to measure the flexural strength and modulus of the cork hybrid material.

Testing was carried out on an Instron 5567A load frame outfitted with a 5kN load cell and a 5kN Instron Flexure Fixture with 10mm anvils. The machine was controlled with Instron’s Bluehill 3 software package. Specimens tested were cut to 12.6 mm wide and were placed on a span of 104 mm. As prescribed by the standard, they were tested until rupture at a rate of 2.77 mm/min. Images of the setup, and raw data are shown in Figure 16 and Figure 17, below.



Figure 16 Setup for three-point bend testing.

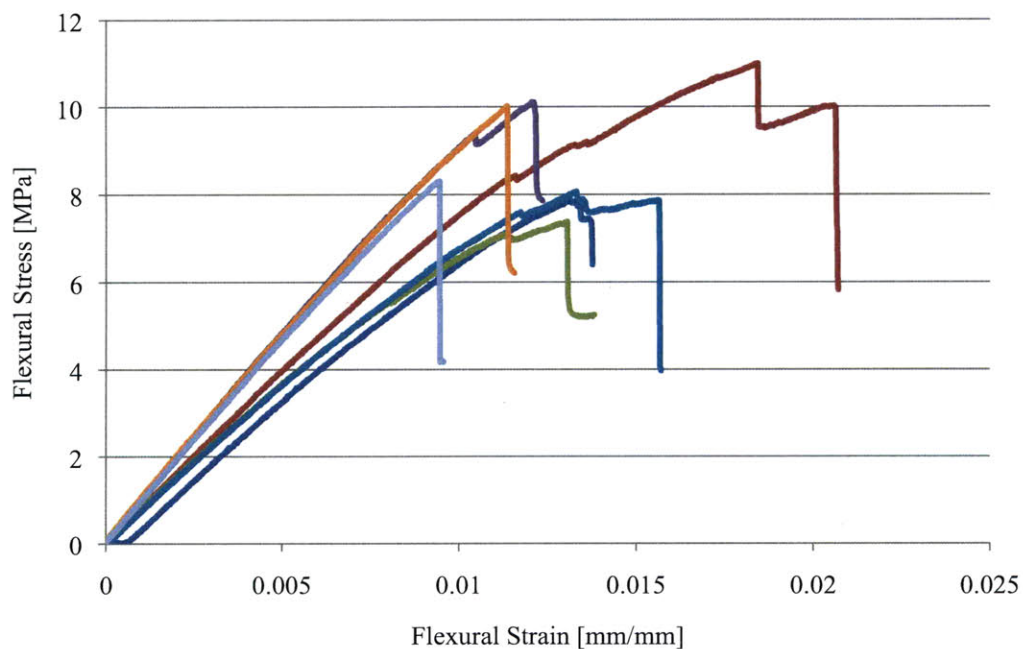


Figure 17 Results of three-point bend testing of cork hybrid material, according to ASTM D-790.

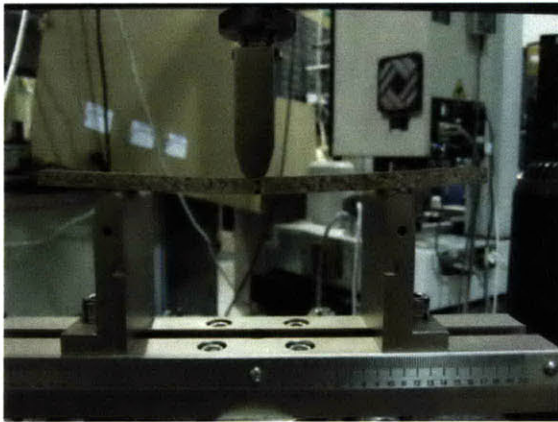
The raw data show a similar bimodality in stiffness and rupture strength to that seen in the tensile testing, and examination of the specimens show that it is for the same reasons. Also like the tensile testing, all specimens failed in brittle fracture. In some cases a ‘jog’ in the data can be seen. This was observed at the same time a ‘ping’ was heard from the test piece. It seems

that this was from pullout or failure of a glass fiber. Also, the calculated flexural strength and modulus are recorded in Table 3, below.

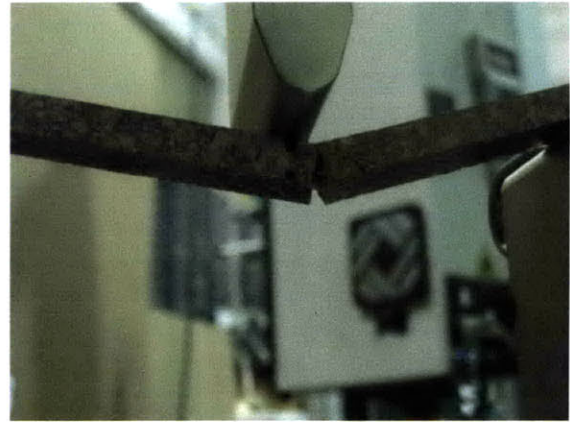
Table 3 Flexural strength and modulus of cork hybrid material based on three-point bend testing.

Specimen	Flexural Strength [MPa]	Flexural Modulus [MPa]
1	7.87	762.19
2	10.99	848.78
3	7.37	756.94
4	10.12	950.53
5	8.06	779.44
6	10.02	901.84
7	8.31	831.80
Std. Deviation:	1.39	73.56
Mean:	8.96	833.07

Photos of the tested specimens can be seen in Figure 18, below. All of the failures occurred at or near the center of the testing span and all initiated on the bottom of the specimen, as expected.



(a)



(b)

Figure 18 (a) Flexural test specimen immediately after the beginning of a test. (b) The same flexural testing specimen following brittle fracture.

4.1.4 Three Point Bending Test: Cork Hybrid with Single Fiberglass Skin

The three point bending testing of the cork hybrid with a single fiberglass skin is carried out using the same test setup, method, and parameters as the sample discussed previously. It is important to note that because there is only one skin, its orientation is an important factor in this

test. Therefore, both orientations were examined. The stress vs. strain curve for each sample is shown for both orientations in Figure 19 and Figure 20, below.

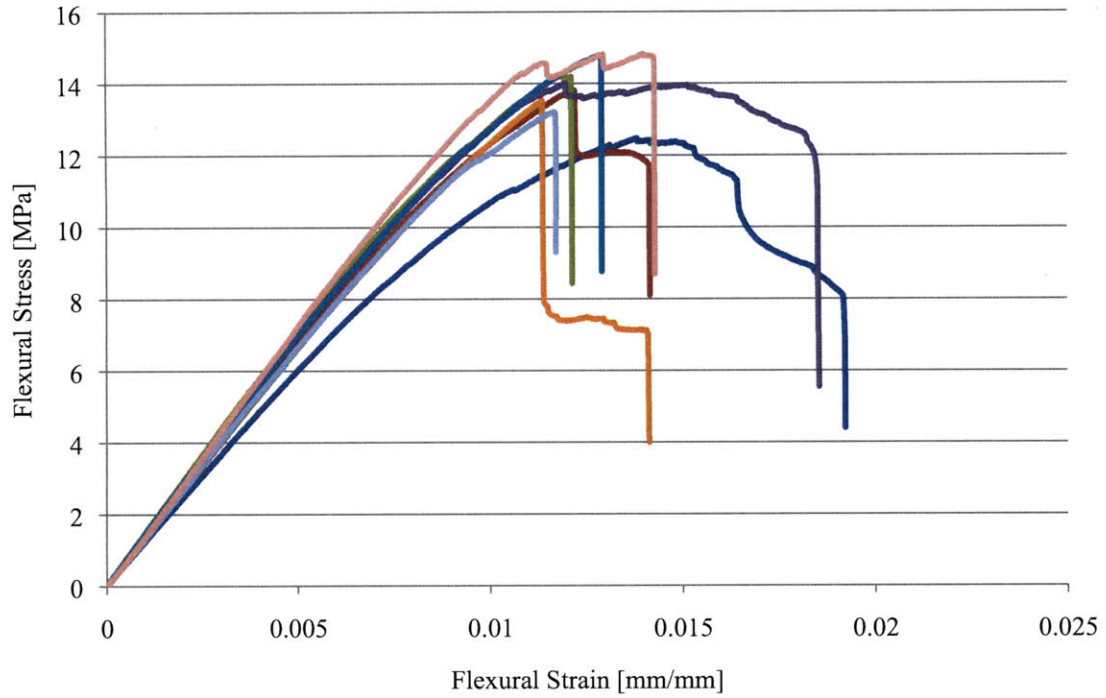


Figure 19 Results of three-point bend testing of cork hybrid with a fiberglass skin on its upper surface.

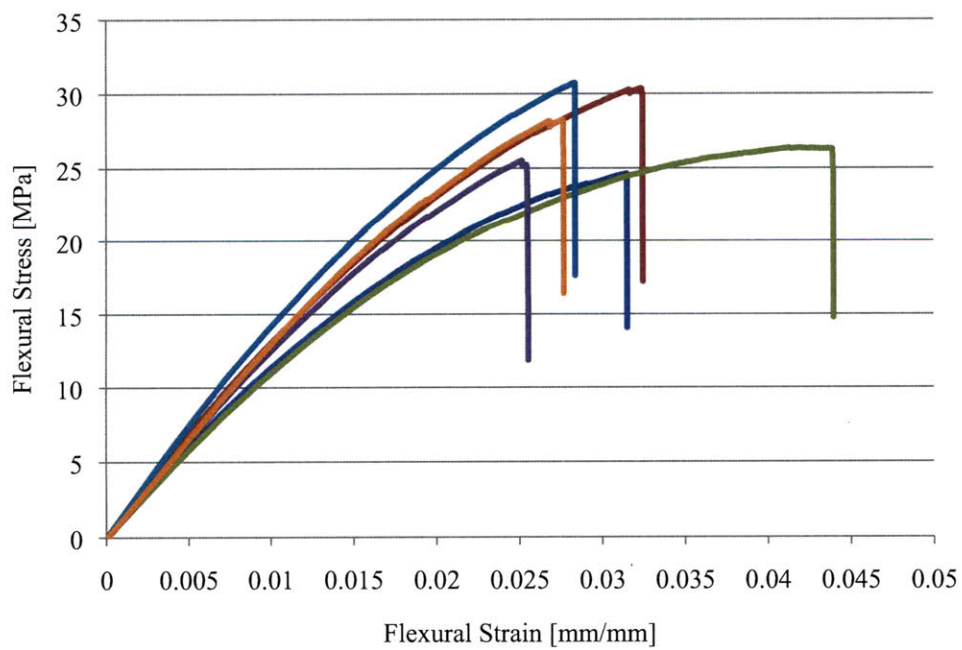


Figure 20 Results of three-point bend testing of cork hybrid with a fiberglass skin on its lower surface.

Interestingly, the curves from the sample with the fiberglass on the lower surface are much smoother and fail in a more repeatable manner than the specimens with the fiberglass on the upper surface. One potential cause of this is that the failure of the skin in tension is more repeatable than the compressive buckling of the fiberglass skin. The raw data are processed by Bluehill to calculate the flexural strength and modulus of each specimen. These values are available for both the sample with a fiberglass skin on its upper surface and lower surface in Table 4 and Table 5, respectively, below.

Table 4 Flexural strength and modulus of cork hybrid material with a fiberglass skin on its upper surface.

Specimen	Flexural Strength [MPa]	Flexural Modulus [MPa]
1	12.46	1233.78
2	13.81	1388.61
3	14.24	1468.77
4	14.00	1427.55
5	14.79	1430.73
6	13.52	1334.09
7	13.21	1362.98
8	14.81	1483.68
Std. Deviation:	0.80	81.33
Mean:	13.85	1391.27

Table 5 Flexural strength and modulus of cork hybrid material with a fiberglass skin on its bottom surface.

Specimen	Flexural Strength [MPa]	Flexural Modulus [MPa]
1	24.63	1205.14
2	30.35	1370.61
3	26.37	1133.45
4	25.53	1311.34
5	30.73	1492.07
6	28.27	1314.05
Std. Deviation:	2.55	125.55
Mean:	27.65	1304.44

As can be seen in the data, the flexural modulus is similar for both orientations of the specimens. However, the strength of the material is significantly improved when the fiberglass skin is oriented downwards in the fixture. This makes sense because that is the mode in which the skin, which is much stronger than the cork hybrid material, can absorb most of the tensile

loading, which is the failure mechanism of the cork. In all cases, however, introduction of the fiberglass skin improved both the flexural strength and stiffness of the specimen.

4.2 Turbine Blade

The purpose of manufacturing a small wind turbine blade is as a visual proof of concept and example of parts that could be manufactured from cork composite by spray-up.

While utility scale turbine blades exceed 50m in length, small-scale wind turbines can have blades even shorter than a meter in length. The small wind industry in particular is looking into new composite materials, controlling cost, design-for-manufacturing techniques⁴¹ making it an area ripe for innovation. Therefore, a common airfoil was selected for prototyping. The blade prototype is a GOE422 profile that has a 7-inch chord length and is 48 inches long.

In order to make the blade, molds were manufacturing from high-density foam by CNC milling. as seen in Figure 21, below. Models of the mold were created in SolidWorks and converted to a toolpath using MasterCam. After machining, the foam molds were coated with several coats of epoxy to seal and strengthen the molding surface. Following a coat of paste wax and PVA release liner, a clear gelcoat was applied before finally spraying the cork composite. Since spray-up is an open mold process, the open surface of each half of the blade must be smoothed before the two can be joined.

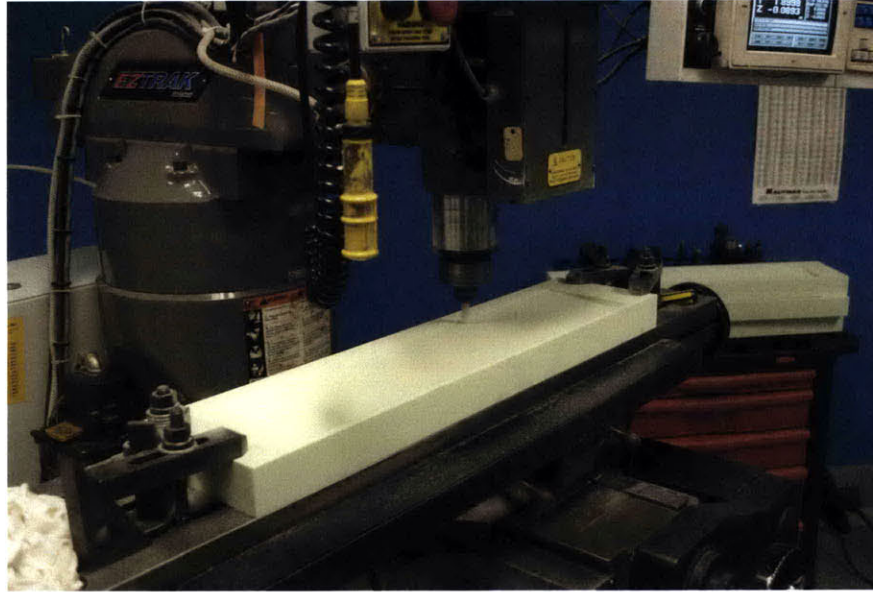


Figure 21 CNC machining of high density foam for a turbine blade mold.

From Figure 22 and Figure 23 it can be seen that, during the curing process, the thicker blade section bowed considerably. It is thought that this occurred because the gel coat is applied to only one side of the work piece. Therefore, as the cork swells due to changes in temperature or humidity, the piece warps. This explanation makes sense given the direction of curvature of the part. Overall, the process was successful for a first attempt at a prototype, but it is recommended to find an external coating with the same expansion characteristics as the cork hybrid or to apply gelcoat to both sides of the part.



Figure 22 Two pieces of a cork composite turbine blade manufactured by spray-up. The pieces have already been demolded and bowing of the upper part is evident.



Figure 23 Two pieces of the turbine blade are joined. The curve of the stiffer piece dominates the shape.

5. Analysis and Discussion

5.1 Performance versus Predicted Performance

Earlier in this document, a modification of the rule of mixtures was used to predict the performance of potential cork/glass-fiber/polyester composites. After testing, it is shown that the spray-up process produces material that falls within the predicted envelopes. These graphs are shown in Figure 24 and Figure 25, below. From the location on the curves it appears that the material acts roughly midway between the two extremes of its constituents acting perfectly in parallel and perfectly in series.

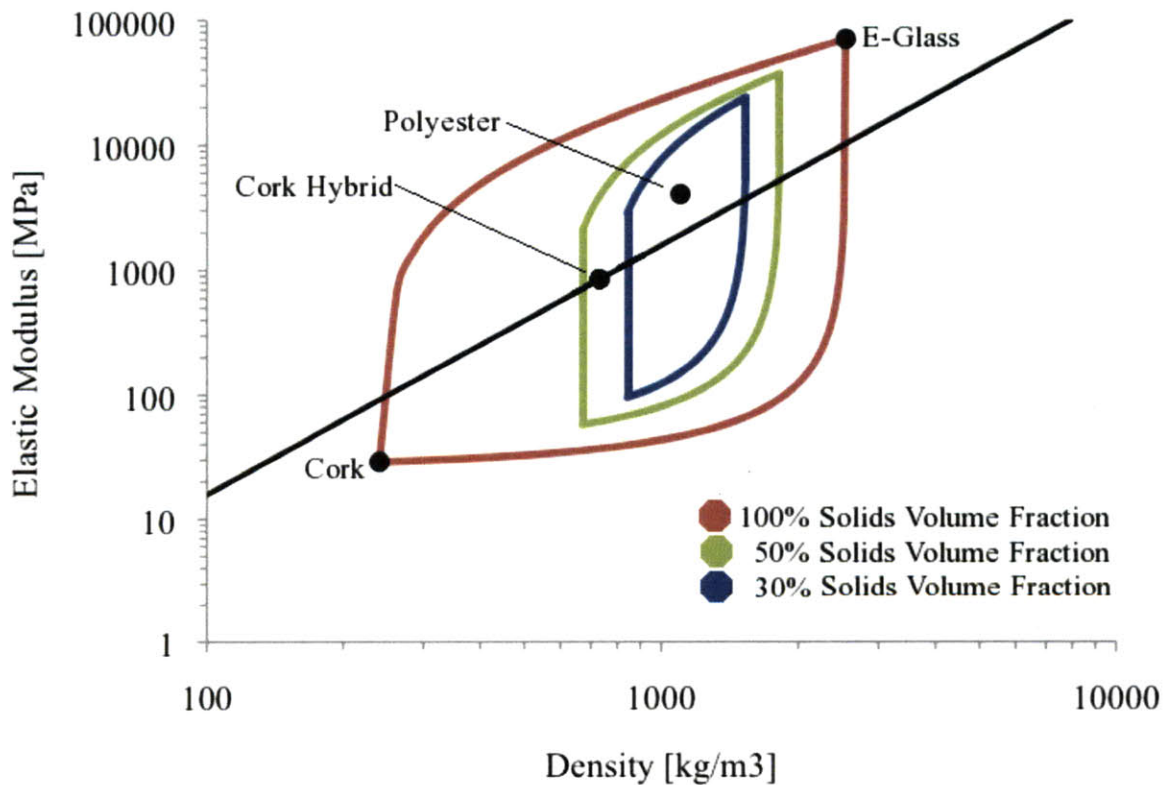


Figure 24 Predicted density vs. elastic modulus with measured elastic modulus and merit index overlaid.

From Figure 24, the merit index tells about the potential usefulness of the material. In this particular formulation, the cork hybrid is better suited for use as a beam than pure cork, but falls short of either pure polyester or glass fiber. Given the strength and stiffness of cork, and its

high volume fraction, this is relatively unsurprising since the failure mode has been noted as brittle failure.

From the measured density, it is possible to calculate the predicted cost of the material.

This is shown in Figure 25.

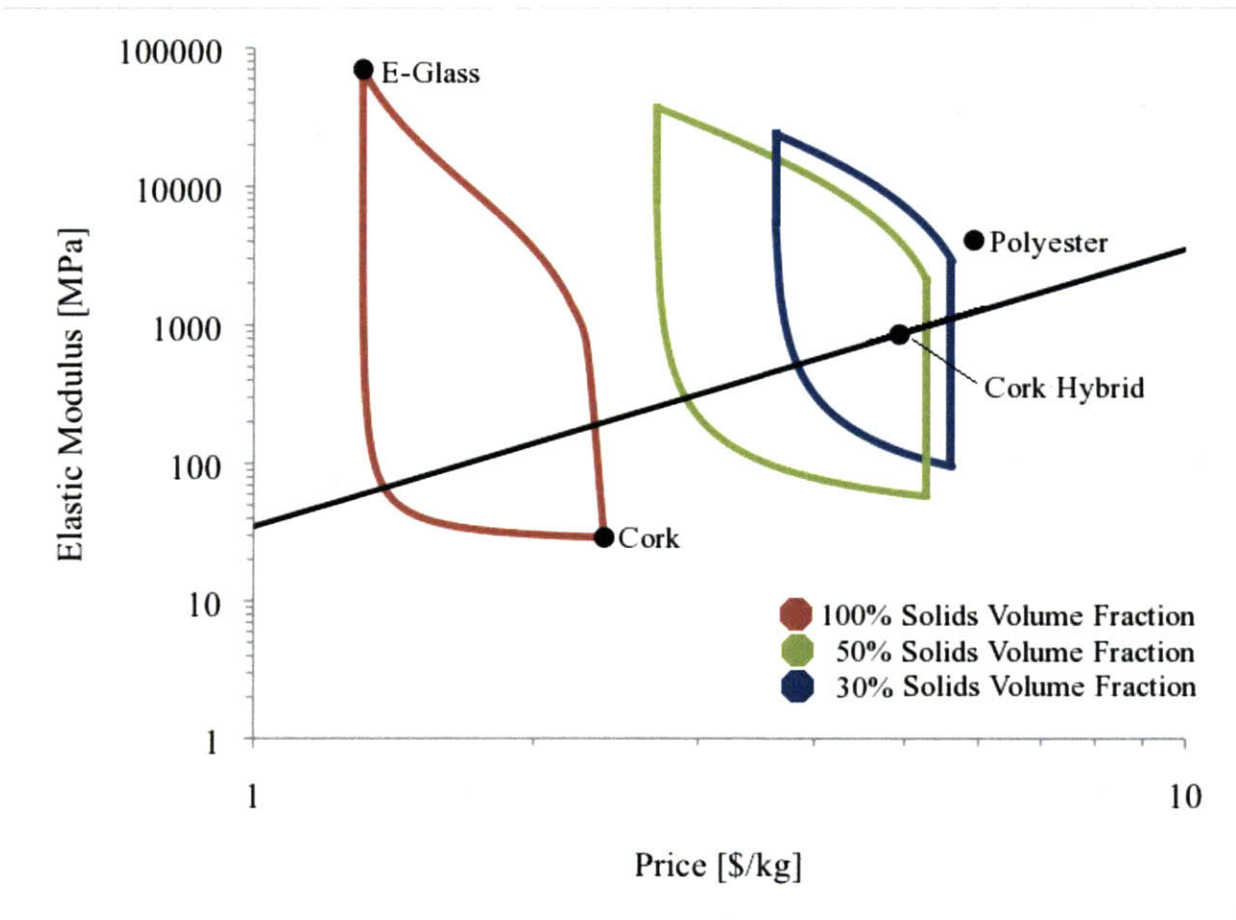


Figure 25 Predicted price vs. elastic modulus with measured elastic modulus and merit index overlaid.

Similar to the above results, when designing for a specified stiffness at minimum cost, the cork hybrid performs better than pure cork, but lags when compared to pure polyester or pure glass fiber.

5.2 Performance versus Competing Materials

It is also instructive to understand how the spray-up produced cork composite performs when compared with other cork composites. A summary of the data collected from a parallel study, also supervised by Professor David Wallace⁴² is presented in Table 6, below. These samples differ from the spray-up produced samples in three substantial ways: the matrix is epoxy rather than polyester, the glass fiber used is microfiber rather than ¼” chopped strand, and the samples are made by vacuum bagging rather than spray-up.

Table 6 Comparison of material properties of vacuum bagged composites using an epoxy matrix.⁴³

	Cork Composite	Cork Composite + Fiberglass Skin	Cork Composite + Fiberglass Skin, Sandwich	GFRP
Density [kg/m ³]	690	690	730	1320
Flexural Modulus [MPa]	930	1160	4690	8170
Flexural Strength [MPa]	15.02	40.71	48.69	201.82
Tensile Modulus [MPa]	480	-	1380	5010
Tensile Strength [MPa]	10.9	-	57.38	259.79

A similar summary, and comparison to spray-up glass fiber reinforced polyester, for the data from this study are shown in

Table 7, below. Because of the lower fiber volume fraction, the material is denser than the vacuum bagged samples. Also, it is important to remember that polyester is both weaker and less stiff than epoxy, which has a strong effect on the strength of the resultant composite.

Table 7 Comparison of material properties of spray-up manufactured composites using a polyester matrix.

	Cork Composite	Cork Composite + Fiberglass Skin, Upper Surface	Cork Composite + Fiberglass Skin, Lower Surface	GFRP ⁴⁴
Density [kg/m ³]	732	732	732	1200
Flexural Modulus [MPa]	833.07	1391.27	1304.44	4136
Flexural Strength [MPa]	8.96	13.85	27.65	117
Tensile Modulus [MPa]	849.87	785.75	785.75	-
Tensile Strength [MPa]	4.36	6.5	6.5	70

Ashby charts are particularly helpful when comparing a wide range of materials including fiberglass, balsa wood, existing cellular foams, and a number of composites. The following pages show these charts in Figure 26, Figure 27, Figure 28 and Figure 29. The former two figures show comparisons of density vs. elastic modulus while the latter two figures show comparisons of density vs. yield strength, all with data from both cork composites added.

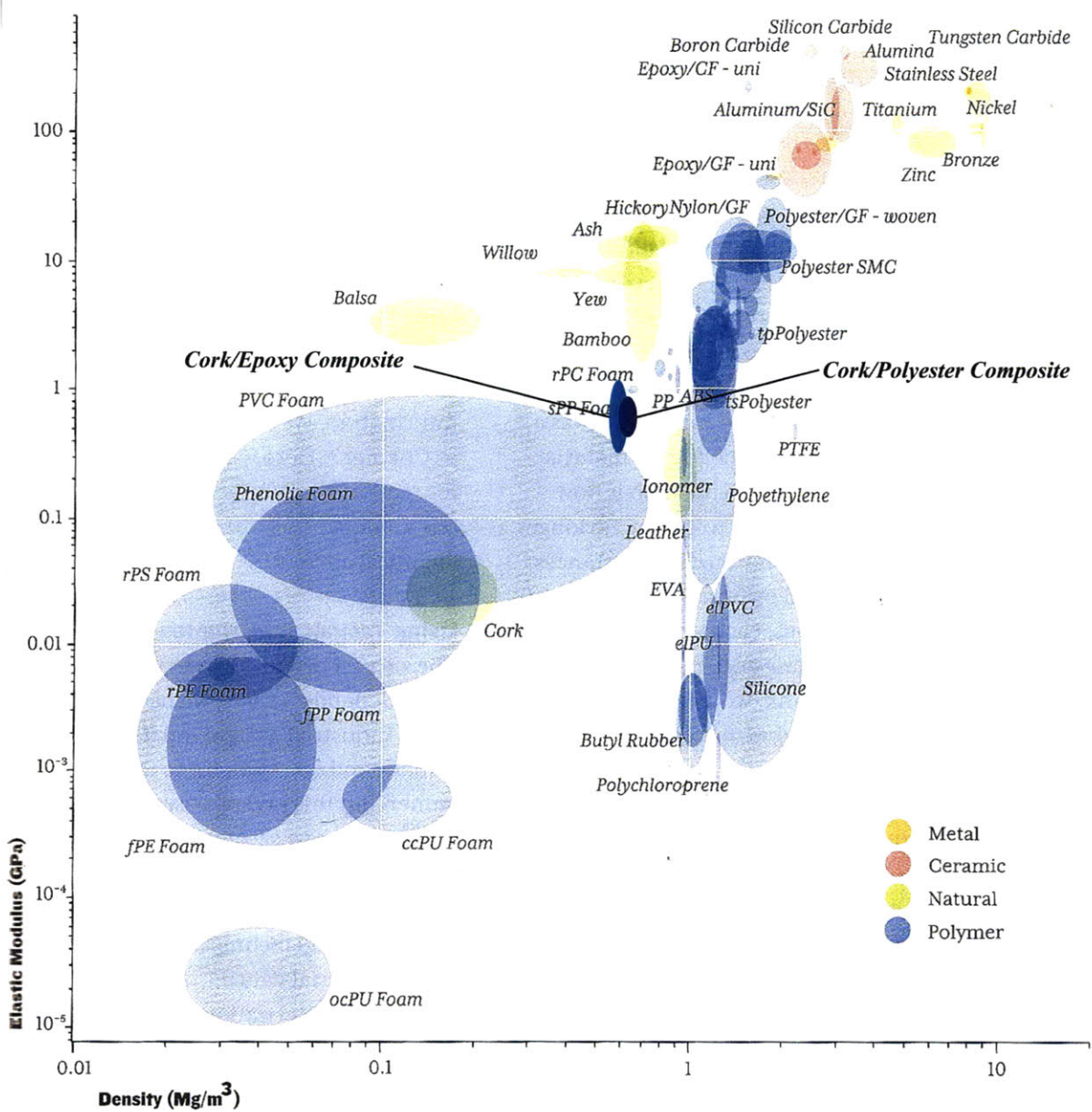


Figure 26 Ashby chart of density vs. elastic modulus with cork composite properties overlaid.⁴⁵

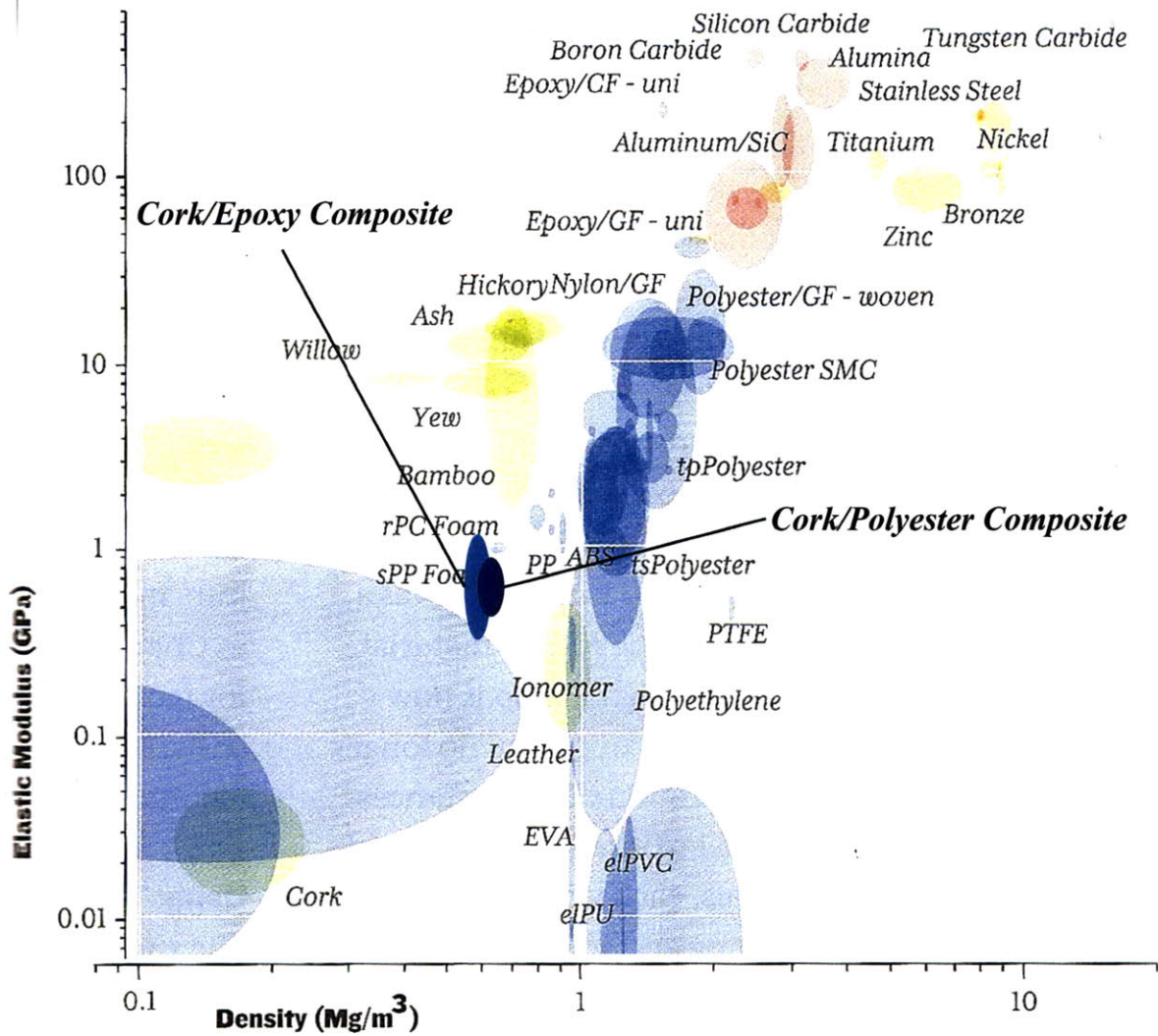


Figure 27 The same data as the previous figure, concentrated on the section where most GFRPs lie.⁴⁶

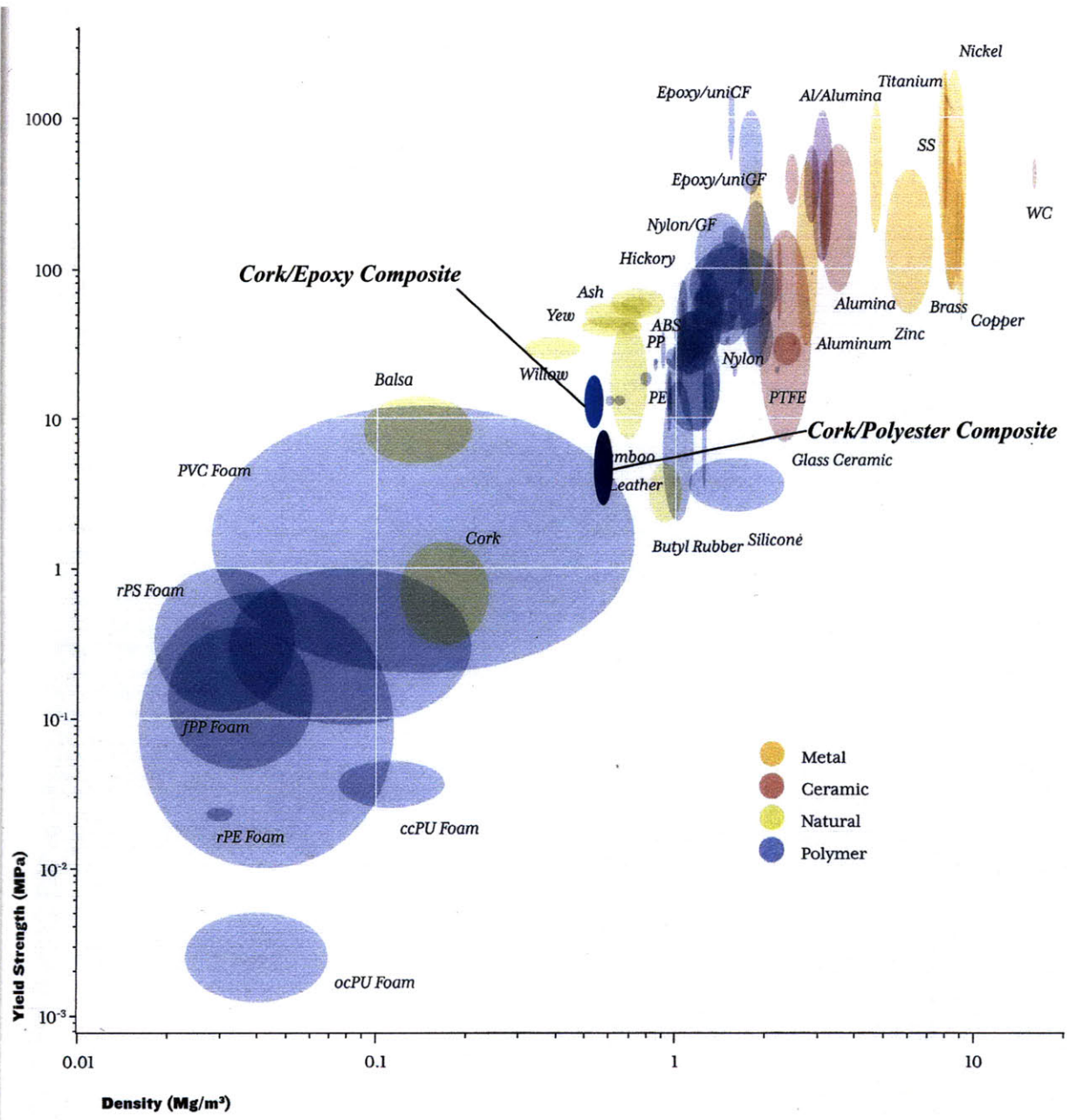


Figure 28 Ashby chart of density vs. yield strength with cork composite properties overlaid.⁴⁷

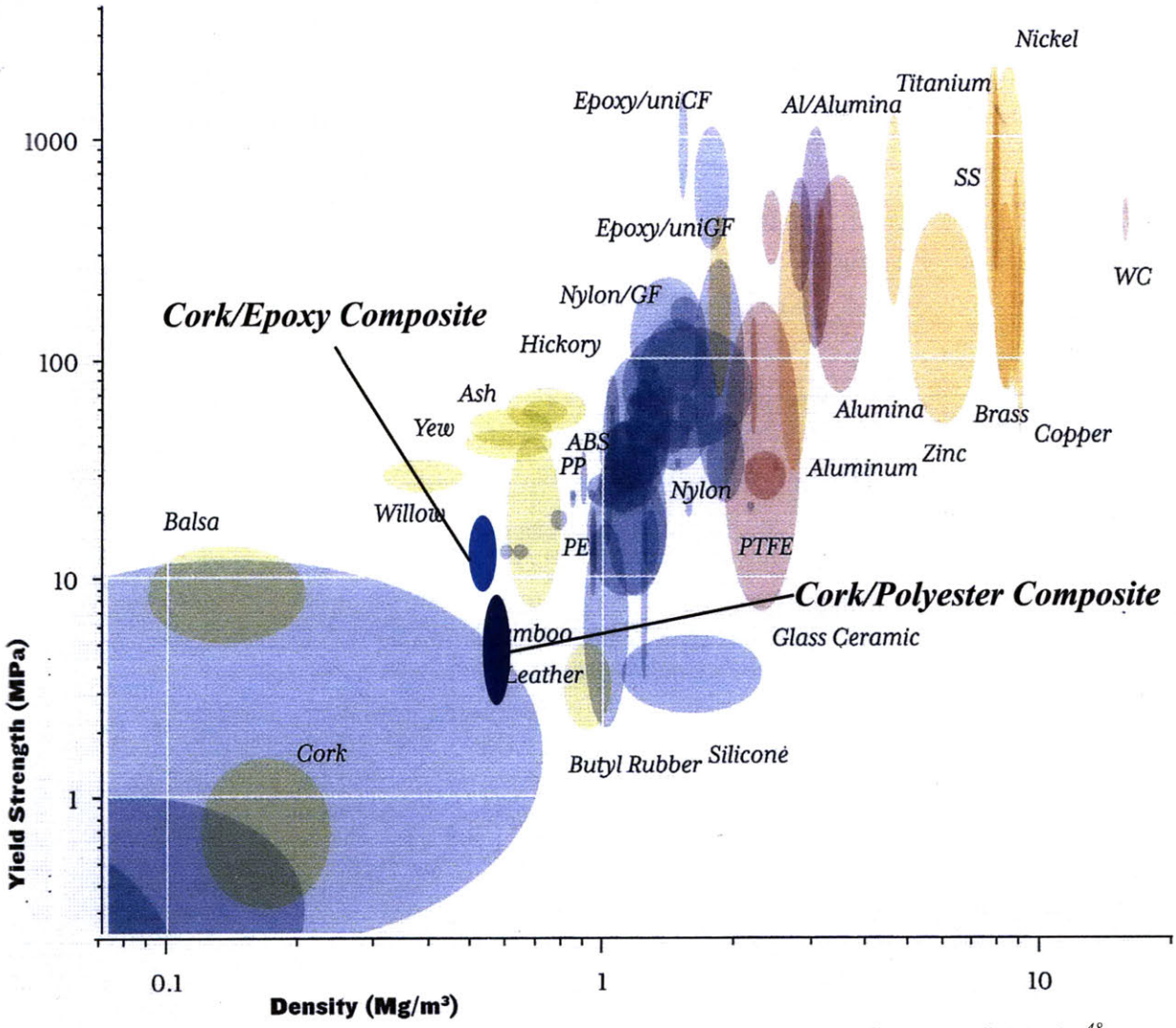


Figure 29 The same data as the previous figure, concentrated on the section where most GFRPs lie.⁴⁸

6. Conclusions

6.1 Synopsis

Overall, this work showed the spray deposition of cork reinforced polymer to be a viable process. A spray lay-up apparatus was successfully constructed and employed in manufacturing parts from a hybrid material composed of granulated cork, chopped glass strand, and a polyester matrix. After developing a model to predict the properties of the hybrid material, the model was validated by mechanically testing specimens of the material made by the spray deposition process. Mechanically, the tested composite was found to have a tensile strength of 4.4 MPa and tensile modulus of 850 MPa. The flexural strength and modulus were 9 MPa and 830 MPa, respectively. These mechanical properties were substantially improved by adding a woven fiberglass skin. Adding a fiberglass skin to the cork hybrid significantly improved its flexural strength. As expected, comparing these results to previous work showed that using an epoxy matrix in place of a polyester matrix creates a substantially stronger material. Therefore, while polyester was chosen as the matrix material for this study, materials should be engineered specifically for each application.

Additionally, a four-foot long wind turbine blade prototype was manufactured to test the spray-up process on a larger workpiece. While there was warping of the cured part, likely due to the fact that gelcoat was on only one side of the part, this is a manageable issue. It is recommended that further work focus on optimizing the hybrid material's properties, re-designing and optimizing the apparatus used for the spray-up process, and demonstrating material viability by manufacturing a cross section of a large turbine blade.

6.2 Future Work

6.2.1 The Material

In making the material, the optimal composition was not studied exhaustively. Rather, the focus of the experimentation was to create a proof of concept cork composite spray-up apparatus. In future work, the material will need to be optimized for different applications. Some applications, such as wind turbine blades, require a higher stiffness per unit weight than the current recipe. Applications like shower pans or bathtubs would focus on aesthetics and cost for a specified strength.

While the current composition was based on educated guesses of macro-mechanical properties, further detailed study of the failure mechanisms would allow the material to approach its theoretical limits. One suggested area of study is investigating the strength of the bond between the matrix and solid constituents. Another suggested area of study is longer glass fibers. Traditional spray-up operations utilize chopped roving in excess of 1 inch in length. While microfibers and ¼” chopped strand have been explored, it is possible that longer strands could better tie the material together. It might also be of interest to attempt a stratified structure rather than mixing the cork and the fiberglass. Overall, further detailed study of failure and micromechanical studies could yield further improvement in material properties depending on the failure mechanisms discovered.

6.2.2 The Apparatus

The apparatus is very clearly in its infancy. There are numerous suggestions for improvement in future iteration. From a basic technology standpoint, the matrix should be sprayed using an airless atomization spray or a high volume low pressure (HVLP) gun. In airless atomizing spraying, resin is atomized by being forced through specially designed nozzles at high pressure (e.g. 1000-2000 psi). HVLP involves a very high volume but at pressure less than 7 psi,

giving greater control of the spray, with less over spray because of the absence of a blasting effect that is common with higher pressure systems. Additionally, airless spray and HVLP solutions offer a reduction in VOCs released.⁴⁹

Furthermore, using an external mix spray gun, rather than premixing small batches, will allow more large scale, continuous processing instead of small batch operations. While spray-up is attractive for small batches because of its low cost tooling, in order to be viable for large parts, externally mixing the resin and catalyst will be essential to allow for greater flexibility and time in making parts.

Additionally, it would make sense to separate the cork and glass fibers instead of premixing them. For one, spraying glass fiber by chopping roving is an established process, and is highly reliable. Also, the chopped glass strand is the largest contributor to clogging in the spray line. Furthermore, since static electricity is a concern, better consistency may be achieved by separating the materials to prevent localized clumping.

Perhaps most glaringly, the apparatus, while functional, is not well designed. Because the ease of use is an integral part of the operator's ability to create a reliable spray pattern, it is a significant concern. As a start, all of the controls and outputs should be integrated onto one gun instead of separating the solids and liquid dispensing systems.

6.2.3 Applications

From the Ashby charts, it is evident that, at least in its current composition, the cork hybrid is not ideal as the principle strength element in any large product. However, when compared to foams and other materials at the center of sandwich, the cork hybrid compares much more favorably. Therefore, it is suggested the next prototype constructed should be a cross section of a large wind turbine panel. Figure 30 shown below offers an example geometry.

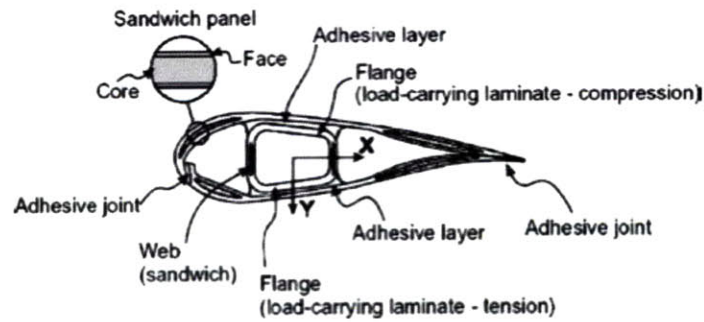


Figure 30 Cross-section of a large rotor blade giving the nomenclature of different blade construction elements.⁵⁰

Spray-up manufacturing is also known for allowing more flexibility in mold design.

Therefore, another potential innovation that could be enabled by spray-depositing cork composite is more complex blade geometries, such as those designed by WhalePower.⁵¹

WhalePower designs turbine and fan blades that use leading edge tubercles, such as those found on the leading fins of humpback whales, to improve efficiency and stall performance.



Figure 31 Turbine blade designed with leading edge tubercles.⁵²

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