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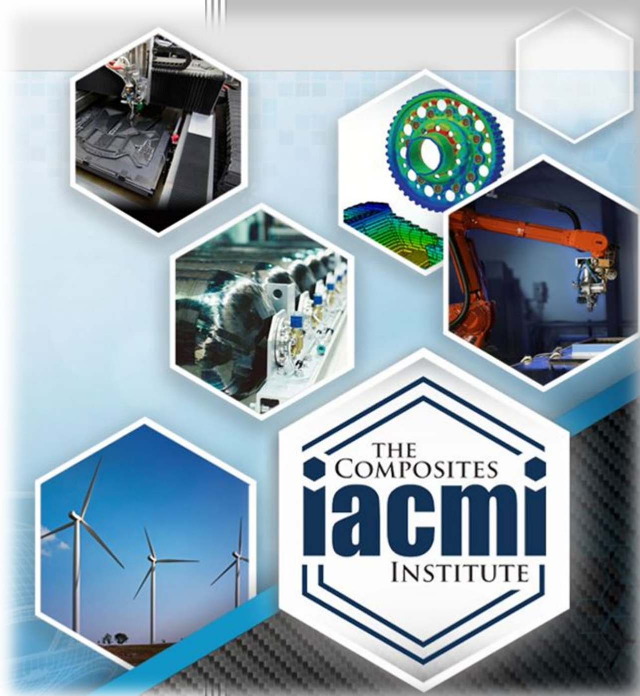
# Multiple Stream Low-Cost Recycling Method

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The Institute for iacmi Advanced Composites Manufacturing Innovation THE COMPOSITES INSTITUTE

U.S. DEPARTMENT OF ENERGY

# Multiple Stream Low-Cost Recycling Method

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# 1. LISTS

## 1.1 LIST OF ACRONYMS

| | |
|---|---|
| CF | Carbon Fiber |
| BMW | Bayerische Motoren Werke GmbH |
| EOL | End of Life |
| FCMF | Fibers and Composites Manufacturing Facility |
| GT | GreenTex |
| EPRI | Electric Power Research Institute |
| MSC | Mediterranean Shipping Containers |
| GPa | Giga Pascal |
| GF | Glass Fiber |
| IACMI | Institute for Advanced Composites Manufacturing Innovation |
| ILS | Interlaminar Shear |
| kJ/m2 | Kilo Joules per meter squared |
| MPa | Mega Pascal |
| ORNL | Oak Ridge National Laboratory |
| UTK | University of Tennessee Knoxville |

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## 1.3 Acknowledgements

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Project acknowledgement: University of Tennessee, Oak Ridge National Laboratories, and Snider Mold

# 2. EXECUTIVE SUMMARY

The global composite industry generates large quantities of waste and which mostly end as landfill due to lack of meaningful end-use applications for the multiple waste streams. In a recent report by the Electric Power Research Institute (EPRI), waste generated by the wind industry could reach 370,000 tons a year of composite wind energy blades being decommissioned and scrapped. Wind energy is just one major industry utilizing composite materials. The waste generated by industry includes End-of-Life (EoL) materials and manufacturing process scrap. GreenTex Solutions has developed a unique and innovative technology to recycle the composite waste streams in a range of forms made from production waste and EoL materials. This includes manufacturing waste materials such as dry chopped fiber tow, loose fibers, shredded fibers from reinforcement fabrics, cured/semi-cured prepregs, and it also includes fully-cured composite structure waste (such as edge trims from cured parts) from manufacturing aircraft, automobiles, wind blades, boats, and composite cylinders (tanks). Current recycling methods involve recovering the structural fiber by removing the matrix resin through methods such as pyrolysis. The resulting fibers are used in injection molding or wet laid nonwoven mats and other usable forms. The GreenTex technology bypasses these intermediate steps to create the lowest possible recycling processing costs and the lowest embodied energy/$CO_2$ emissions. The end-product from the GreenTex technology is a finished industrial composite part/application versus intermediate fibers or fabrics.

The GreenTex manufacturing process enables cross-industry reuse of recycled feedstock by taking waste from multiple industries (wind energy, aerospace, marine, etc.) and recycles the waste into a product used in other industries. The initial target market application is structural flooring for intermodal shipping containers and truck bodies. One of the team's key partners is Wabash National which produced 29,000 truck bodies in 2019. The current flooring system is comprised of solid oak "butcher board" laminated panels. Additionally, Mediterranean Shipping Containers (MSC) transports over 1.8 million twenty-foot equivalent units per year in intermodal shipping containers. The current container floor is laminated hardwood that is harvested from the rain forests of Central and South America. The project is to develop a flooring system made from recycled composites that can be qualified for both companies (Wabash and MSC). Initial prototypes validated that the recycled composites panels are lighter and thinner with much higher mechanical strength. These results suggest a typical truck trailer would have 20% lower tare weight. The GreenTex technology is not limited to flooring and is widely applicable to other transportation elements such as walls, roof elements, cab areas and related structural components.

Under this project different composite waste streams were evaluated and then combined to develop a formulation that would meet the targeted performance criteria for a flooring system. Wet compression molding was used to fabricate plaques at different tonnage using various composite waste streams. The plaques were tested for flexure and impact.

# 3. INTRODUCTION

The growing global composites industry lacks meaningful demand and market applications for composite waste [1-3]. The composites industry has developed over many decades from a niche material to current major industry applications. Composite materials are unique as they provide end-users the ability to custom design materials tailored for an application. Industries consume various structural fibers (carbon fiber, E-glass, aramid) combined with many matrix resins options (polyester, vinyl ester and epoxy) in many forms (dry, unidirectional and woven). Composite's customization features provide awesome structural performance to design large aircraft wings, massive wind turbine blades, and large volume automotive structure. However, this customization feature presents a recycling problem – how to capture, reprocess and repurpose the many material types into meaningful products that a customer will purchase. The massive recent growth of wind energy and large commercial composite aircraft (Boeing 787 and Airbus A350) demonstrate the need for recycling solutions. These programs use world scale volumes of materials – all unique to the programs [4-13].

Composite materials work in structural applications because differing materials (fibers and resins) combine to deliver superior mechanical properties. By themselves, the fiber and resin offer no structural benefit. Fiber structural properties and surface bonding characteristics are selected and combined with proper liquid resins – then cured to solid form. The cured resin to fiber surface interface is why composites work and is what translates winning structural properties. Most resins used in structural applications are thermoset vs. thermoplastic resins. Thermoset resins can deliver fabulous process and structural benefits, but these resins are notably difficult to recycle and are usually disposed of through landfilling.

Metals (example steels, aluminum, titanium) have long been used in many industrial applications and provide a model for composites to study. Metal processors once had the same industry recycling problem as composites. With time and maturation, the industry developed robust and efficient methods to capture and reuse metals within the value streams (example aluminum beverage can recycle, titanium CNC machining reuse, large ship scrapping and recycle). Metal industry recycle and reuse infrastructure developed over time – and it works. The composites industry now requires similar reuse solutions to avoid the current landfill practice. GreenTex technology provides an efficient process to convert all major forms of composite materials and provides an initial major market application for the repurposed materials.

GreenTex (GT) has developed a patent pending approach to recycling high performance fibers. The GT method allows for any type of composite waste in any type of form to be reprocessed to make a high-performance composite. The GT process takes cured composite scrap and combines that with scrap fibers or fabrics from glass and carbon fibers, and with fresh resin that is then cured to form panels or shapes. GT feels that it has developed the cheapest possible manufacturing method to recycle high performance fibers. To date, GT has made prototypes in its own shop as well as at the McNair Center at The University of South Carolina and The South Dakota School of Mines and Technology (SDSMT). Initial testing at Clemson University, McNair, and SDSMT has concluded that the GT panels have significant strength and impact properties. The panels also have a distinctive aesthetic look. GT has introduced its panels to a variety of potential customers and has received positive feedback. GT is at the point in which it needs to go commercial but before doing so it needs to finalize some essential points.

For the last several years GT has been working with Mediterranean Shipping Company (MSC) to come up with a new flooring system for their ocean containers. Today they use 1 1/8” Luan plywood which is super heavy and absorbent. MSC, along with being one of the largest shipping companies in the world also owns a huge cruise ship line in Europe. MSC builds all their own equipment (containers and ships). Developing container flooring using recycled composite materials is the focus of this project.

The focus of this effort was to optimize the curing parameters to minimize cycle time and provide performance equal to existing plywood-based flooring panels used in shipping containers, with targeted lower weight at the same thickness. This required process optimization in terms of temperatures, curing times and molding pressures. The resulting work establishes a product pathway for the shipping container applications, among others. The target was to get to panels that can be installed for testing in Mediterranean Shipping Company (MSC) containers in Charleston, SC in consideration of lower cost and improved recyclability.

# 4. BACKGROUND

The composite industry generates a lot of waste. It is commonly estimated that around 30% of carbon fiber production ends up in waste streams. The average estimated global carbon fiber demand is around 71,650 – 93,696 tons per year so that means around 21,495 - 28,109 tons per year of carbon fiber waste is generated [11]. Another striking example is wind blades. In a recent report generated by the Electric Power Research Institute (EPRI), waste generation could reach 370,000 tons per year from wind blade decommissioning and disposal [12, 13]. These decommissioned blades are currently landfilled.

Several of the partners in this proposal (Wabash National, Hexcel, Zoltek and NEG) are composite waste generators and produce enough waste to support GreenTex’s production line. These companies are in close proximity to GreenTex supporting simple logistics. The annual production of the flooring panels for the truck bodies would eliminate 1.5 million pounds of Wabash trailer waste, 2.0 million pounds of fiberglass waste and 1.5 million pounds of carbon fiber waste. Shipping container flooring represents a market potential many times this amount. GreenTex’s technology will significantly reduce the composite waste going to landfill. This includes, but is not limited to, manufacturing waste materials such as dry chopped fiber tow, loose fibers, shredded fibers from reinforcement fabrics, cured/semi-cured prepregs, and it also includes fully-cured composite structure waste from manufacturing aircraft, automobiles, wind blades, boats, and composite cylinders (tanks). Current recycling methods are energy intensive, i.e. recovering the structural fiber by removing the matrix resin through methods such as pyrolysis. The resulting fibers could then be used in injection molding or wet laid to form nonwoven mats. The GreenTex technology bypasses these intermediate steps and enables cross-industry reuse of recycled feedstock by taking waste from multiple industries (wind energy, aerospace, marine, etc.) and recycles the waste into a product used in other industries. The current flooring system comprised of solid oak “butcher board” laminated panels are used in shipping containers and truck bodies. The project objective was to develop a flooring system made from recycled composites that can be qualified for both truck bodies and shipping containers. Initial prototypes validate that the recycled composites panels are lighter and thinner with much higher mechanical strength. These results suggest a typical truck trailer would have 20% lower tare weight. The GreenTex technology is not limited to flooring and is widely applicable to other transportation elements such as walls, roof elements, cab areas and related structural components. Various recycled fibers were evaluated and are shown in Figure 1.

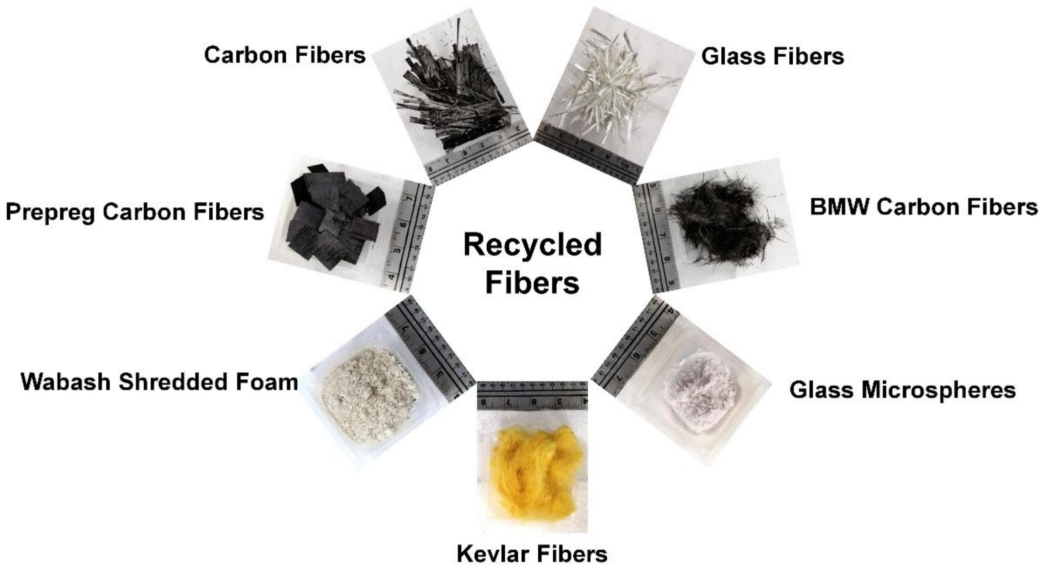



*Figure 1. Various recycled fibers evaluated for composites processing*

## 5. GREENTEX PROCESS

The GreenTex process is simple and cost effective. Cured fiberglass composite parts from Wabash National, wind energy blades, cars/trucks and boats and cured carbon composite parts (airplanes, bikes, golf club shafts) are crushed to fine "dust-like" material. To prepare a sandwich construction (which comprises a core and face sheets), this material is mixed with a minimal amount of resin to form the core of the part. Fiberglass, carbon, aramid fibers edge trimmings or offcuts are used to form the top and bottom laminates (face sheets). The material is placed in a close cavity mold and resin is poured to saturate the scrap/recycled fibers under transverse compression. The panels can be extended to a sandwich construction by using ground up material from wind blades and boats which can be saturated with foam recycled scrap to produce lightweight core. Collectively the core and facesheets can form fully recycled sandwich composites. All these forms are used in this project.

## 6. RESULTS AND DISCUSSION

Recycled fiber reinforced composite panels for the shipping container flooring application were manufactured using a wet-compression molding process. In wet-compression the chopped carbon, glass, aramid fibers in tows or flake form are laid inside a close cavity mold and vinyl ester resin is poured to saturate the fibers through transverse impregnation during compression molding. The produced composites were evaluated for fundamental mechanical properties namely, flexure (three-point bending) and impact. Recycled fiber composites were processed at three pressures-low psi (< 20 psi), medium psi (< 100 psi) and high psi (<200 psi). Repurposing the recycled fibers for various applications can be achieved by wet compression molding process that avoids post processing of fibers such as pyrolysis and solvolysis, which reduces the processing cost of overall recycling of composite waste streams.

To evaluate the mechanical performance of the recycled fiber composite panels manufactured via wet-compression molding, coupon level testing was conducted by extracting test specimens from the panels of all the variants, as shown in Table 1. Table 1 also shows various processing pressures evaluated for certain recycled raw materials. Foam/core panels were not evaluated for mechanical properties.

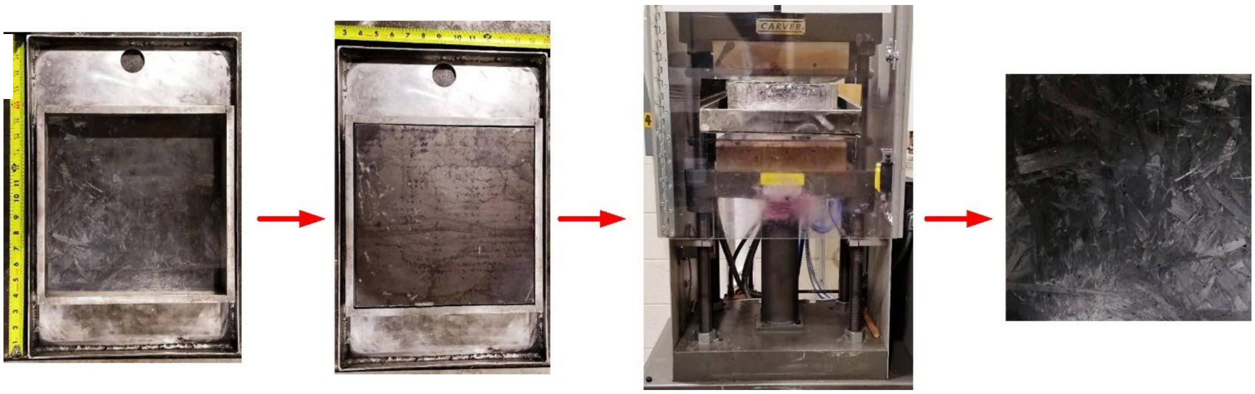
*Table 1. Various material systems and respective processing conditions for recycled composites preparation and mechanical testing.*

| Materials | Processing pressure | Fiber length, cm | Polymer matrix | Testing |
|---|---|---|---|---|
| Chopped Carbon Fibers | low, medium, high | 7.6 | Huntsman Epoxy | Flex, ILSS, IZOD |
| Chopped Glass Fibers | low, medium, high | 7.6 to 15.2 | Huntsman Epoxy | Flex, ILSS, IZOD |
| Aerospace grade CF prepreg | low, medium, high | 2.5 | Huntsman Epoxy | Flex, ILSS, IZOD |
| Kevlar | high | 7.6 to 20.3 | Huntsman Epoxy | Flex, IZOD |
| BMW RCF | high | 7.6 | Huntsman Epoxy | Flex, IZOD |
| Wabash Foam characterization | low | Shredded | Huntsman Epoxy | Impact |
| Glass microsphere-Wabash Foam core | low | - | Huntsman Epoxy | Batch process evaluation |

**Materials and Methods:**

Various recycled fibers and prepreg material (glass fibers, carbon fibers, Kevlar fibers and carbon prepreg material) were all procured from various sources and were carefully sorted and stored in dry atmosphere. Epoxy resin used in this work was Araldite 3492 and catalyst Aradur 1568 from Huntsman Resin systems. As mentioned in table 1, carbon (CF) and glass fibers (GF) were chopped to 76.2 mm in length while CF prepreg material was chopped to 25 mm. Rest of the material waste streams were chopped to various length scales as mentioned in the table 1. A steel tool was designed and built as shown in the Figure 2. After the recycled material was mixed with epoxy, caul plates were placed on the material mix and wet- compression molded in the Carver press as shown in the Figure 2. Test specimen extraction was carried out at University of Tennessee (UTK), Fibers and Composites Manufacturing Facility (FCMF), per ASTM requirements. Flexure (ASTM D790), ILSS (ASTM D2344) and Izod (ASTM D256) specimens were extracted in 0 and 90 directions as shown in Figure 3. Mechanical characterization was carried out on a Test Resources load frame with 50 kN loadcell at FCMF. Mechanical testing of the specimens was performed on the face sheets alone and not on the sandwich structure.

**1**




Representative image of recycled panels processing on Carver press. Panels were processed at three pressures low, medium and high psi.

**2**

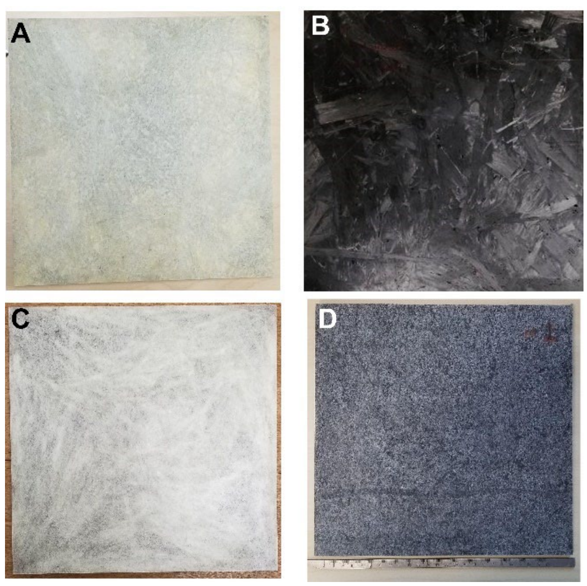



*Figure 2. (1) Processing of the panels was carried out on a tool compression molded on Carver press. (2) Representative panels of (A) GF face sheets with Kevlar core, (B) CF face sheets (C) GF face sheets with Wabash Foam core (D) GF mixed with Wabash Foam core*

**Results and Discussion:**

Various compositions were manufactured by wet-compression molding technique and mechanical testing was carried out for all the variants as shown in Table 1. Specimens were extracted out of the panels along (0°) and (90°) direction as shown in the Figure 3.

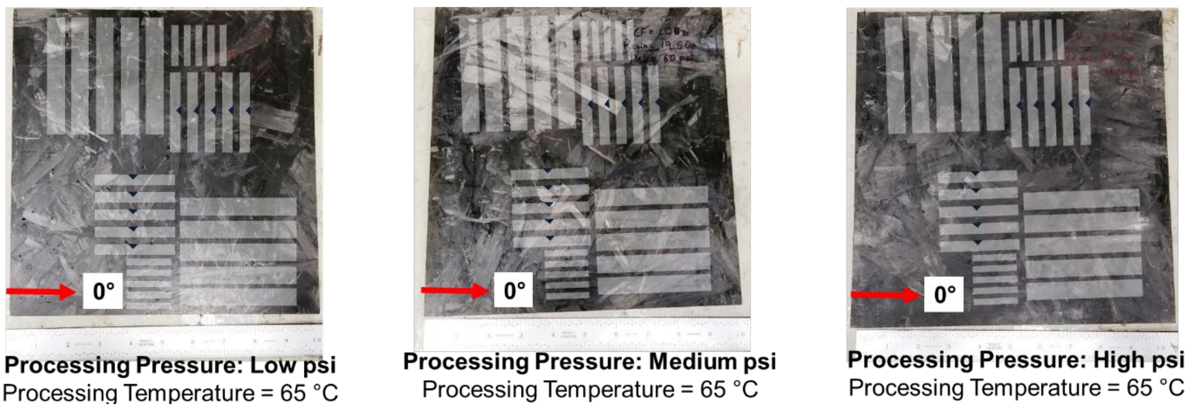



*Figure 3. Test specimens extraction from representative CF panels processed at 16, 60 and 160 psi respectively*

Flexure data of CF, GF, and CF prepreg epoxy variants processed by wet compression are presented in Figure 4. Test specimens were extracted per ASTM D790, in 0 and 90 directions and subjected to mechanical testing. Panels processed at high pressure clearly showed higher flexure strength and modulus, respectively. As CF and GF have longer fibers the flexure strength is higher compared to CF prepreg specimens. Here the dry chopped CF and GF were longer ~1.5-2" length while the CF prepreg were chopped to 0.5" length. Within the dry chopped CF and GF, the GF had longer length ~2" compared to CF ~1.5". Also vinyl ester resin was used to produce the final panels with the CF and GF waste. It is known that the GF has superior compatibility with VE resin compared to CF. This could be another reason for the higher strength with GF.

As these are not vacuum processed there will be lot of macro pores which will also result in lower mechanical properties. Furthermore, in GF and CF prepreg, low psi 0° specimens show slightly higher flex strength, however, the standard deviation (n=5) is also high. For the face sheets used to make the final sandwich structure panels, high pressure was finalized as the processing pressure as it reduces the voids by coalescing macro voids. Flexure stiffness for CF dry fiber and CF prepreg are higher compared to GF epoxy as CF has higher stiffness than GF.

The data in these panels has larger scatter since the method to introduce the resin causes transverse impregnation which varies based on fiber filaments versus bundles and aspect ratio. The key to the process is low cost where these variations even out due to the large panel size. This is an acknowledged aspect of this process.

Interlaminar shear (ILS) strength data of CF, GF, and CF prepreg epoxy variants processed by wet compression are presented in Figure 5. Test specimens extracted, per ASTM D2344, in 0 and 90 directions were tested uniformly. CF panels processed at medium psi showed higher ILS strength compared to the panels processed at low psi and high psi, respectively. At high psi, the fibers in the panel appeared to be dry which is why there was lower ILS strength. However, GF panels showed similar ILS across all the processing pressures. This could be due to sizing compatibility as well, as GF are generally sized for epoxy resin. For CF prepreg material, ILS strength was higher for high psi specimens compared to CF epoxy. This reduction in ILS in CF specimens could be due to weaker interface bonding due to excess resin squeeze out.

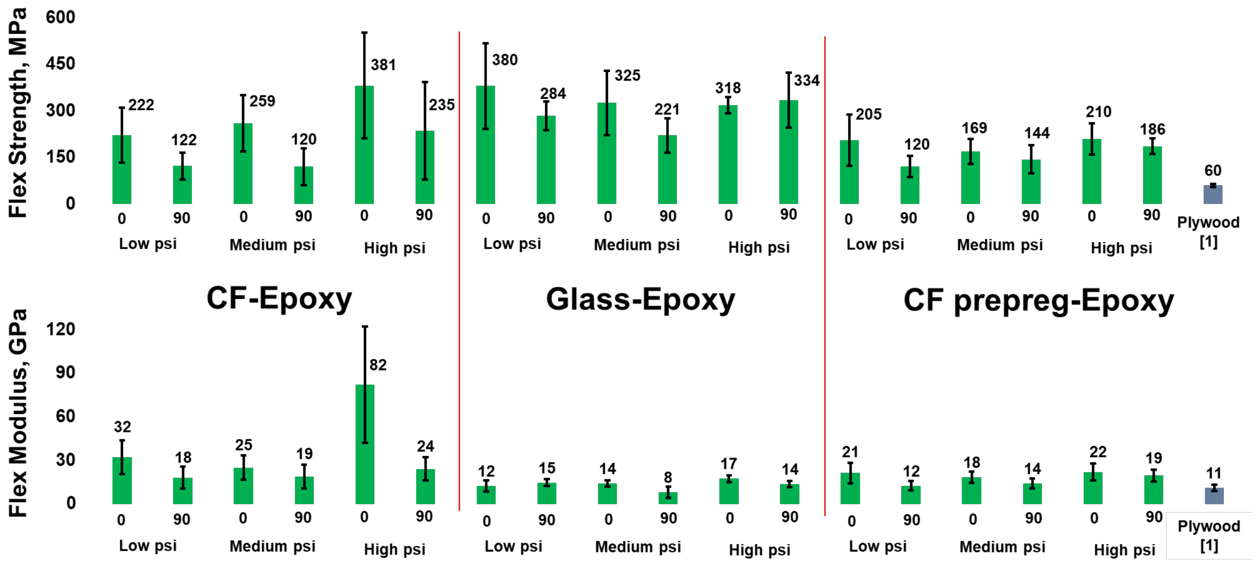



Figure 4. Flexure strength and modulus of CF, GF and CF prepreg epoxy variants processed at low, medium and high psi are presented.

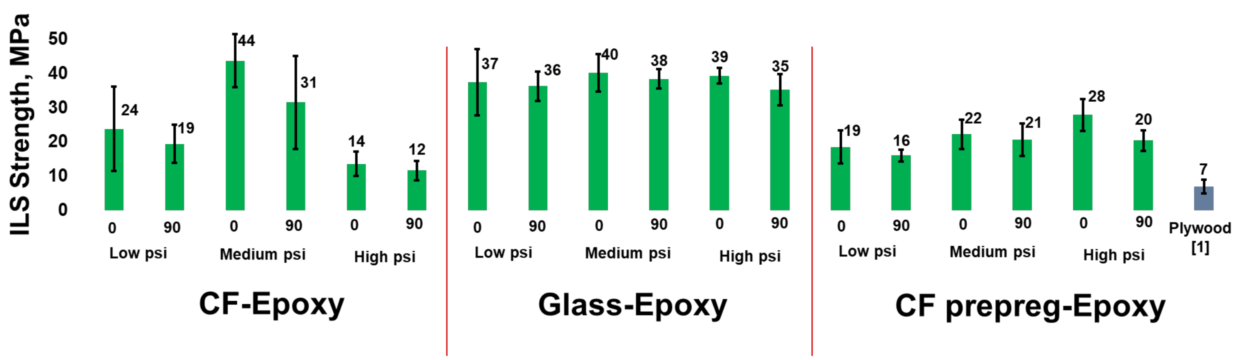



Figure 5 Interlaminar shear strength of CF, GF and CF prepreg epoxy variants processed at low, medium and high psi are presented.

Izod impact strength data of CF, GF, and CF prepreg epoxy variants processed by wet compression are presented in Figure 6. Test specimens extracted, per ASTM D256, in 0 and 90 directions were tested uniformly. In CF epoxy, high psi processed specimens showed highest impact strength compared to low psi and medium psi specimens, respectively. However, the standard deviation is also too high to be called viable data. GF epoxy specimens showed higher impact resistance, which is evident because glass fibers absorb more energy than carbon fibers. Strain to failure of GF is 4 to 5%, however strain to failure of CF is 1 to 1.5%. Hence GF epoxy composite specimens showed higher impact resistance than CF and CF-prepreg specimens.

Flexure data of dry CF, GF source, CF prepreg, BMW CF material, and Kevlar epoxy variants processed by wet compression are presented in Figure 7 and 8. All the panels were processed at high psi because face sheets of the sandwich structure need to be stronger and stiffer than marine grade plywood. Test specimens extracted in 0 and 90 directions were tested carefully and uniformly. CF and GF panels processed at high psi clearly showed higher flexure strength respectively, compared to CF prepreg, BMW CF and Kevlar variants. The marine grade plywood flexure strength is added from the literature for comparison; however, it has much lower flex strength compared to other composite options. As CF and GF have longer fibers the flexure strength is higher compared to CF prepreg specimens. As these are wet compression molded without vacuum, there are macro pores which will also result in lower mechanical properties. The baseline is ~ 60 MPa. All the variants can be used for ship flooring applications.

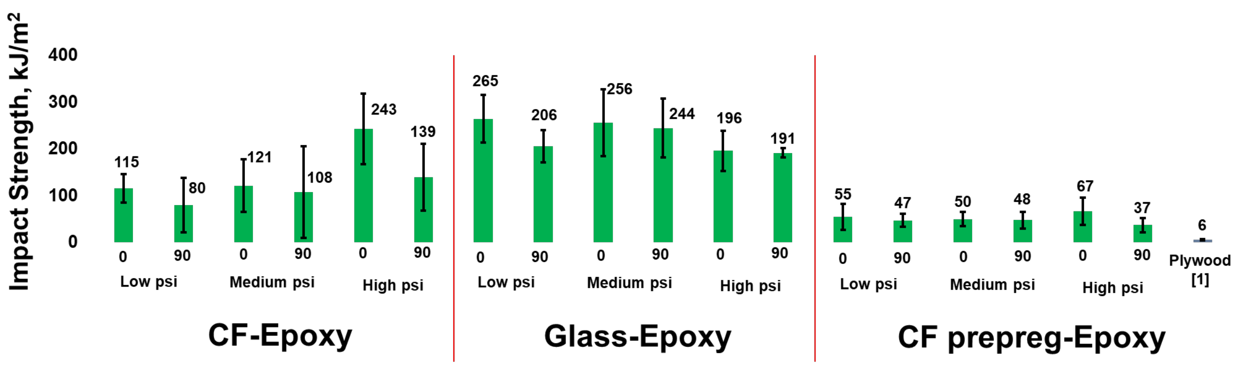



*Figure 6. Impact strength of CF, GF and CF prepreg epoxy variants processed at 16, 60 and 160 psi are presented*

Bending stiffnesses of CF, GF, CF prepreg and BMW variants are higher than plywood data as shown in Figure 7. Bending stiffness of CF and GF epoxy variants are almost double compared to plywood. However, Kevlar epoxy showed poor stiffness as the polymeric fiber Poly-paraphenylene terephthalamide is primarily known for high strength and high elongation to break. Hence Kevlar showed poor stiffness compared to marine grade plywood as mentioned in baseline properties. However, CF, GF, CF prepreg and BMW CF material are all well above the baseline proving the use of composites for shipping container flooring applications.

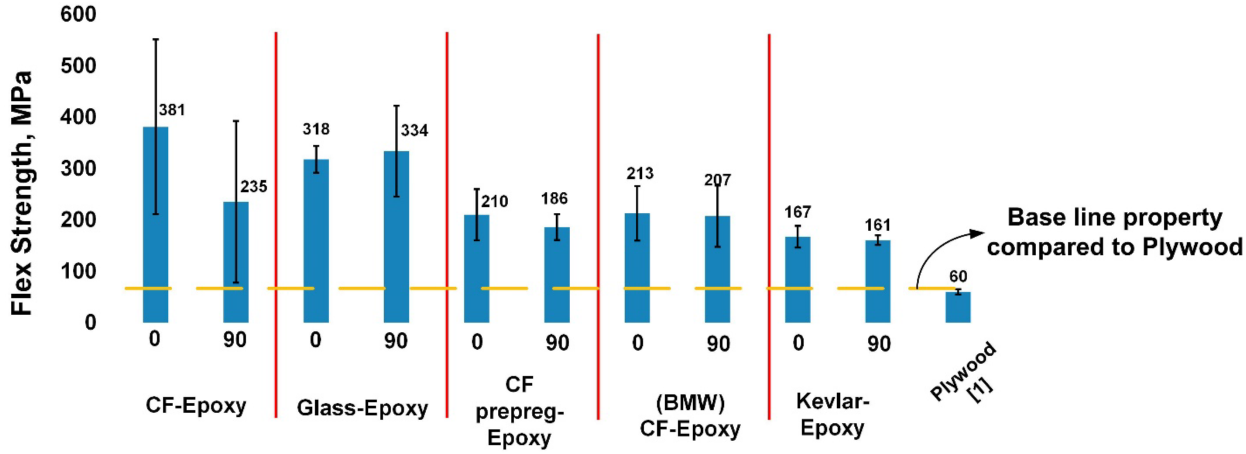



*Figure 7. Flexure strength of CF, GF, CF prepreg, BMW CF and Kevlar epoxy specimens all processed at high psi are presented. Marine grade plywood is presented for comparison and to establish base line.*

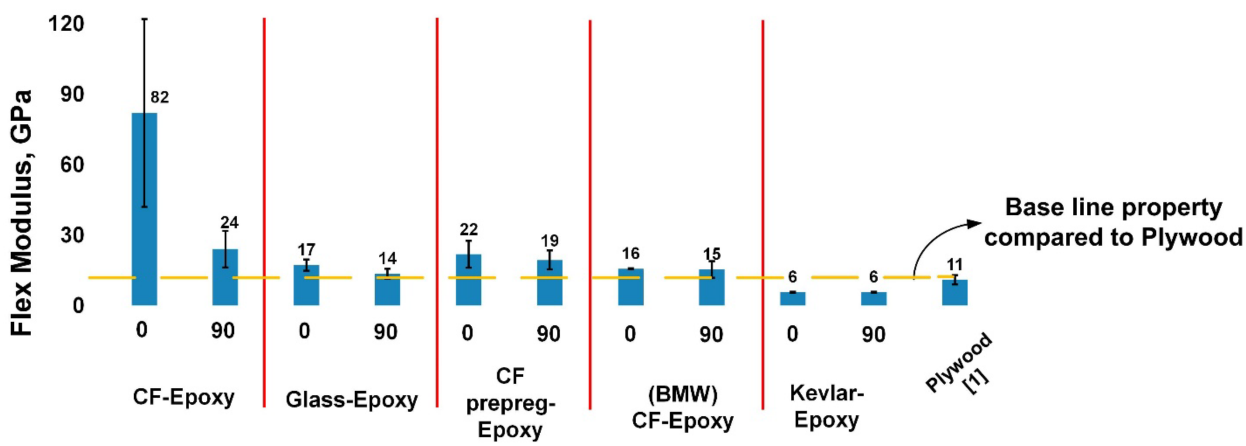



*Figure 8. Flexure modulus of CF, GF, CF prepreg, BMW CF and Kevlar epoxy specimens all processed at high psi are presented. Marine grade plywood is presented for comparison and to establish base line.*

Izod impact strength data of dry CF & GF sources and CF prepreg, BMW CF material and Kevlar epoxy variants processed by wet compression are presented in Figure 9. All the panels were processed at 160 psi compression since these variants were evaluated for face sheets of the sandwich structure as they need to be more impact resistant than marine grade plywood. Test specimens extracted in 0 and 90 directions were tested carefully and uniformly. We observed for the Kevlar fibers that the transverse wet out during compression molding still rendered some dry fibers through the thickness. The resin coated the Kevlar fibers but did not have intimate wet out. It is well acknowledged that Kevlar is generally challenging to wet out but used for its fibrillation properties in ballistic applications. The marine grade plywood impact strength is added from the literature for comparison. All the specimens outperform the baseline value. Marine grade plywood has impact strength 6 $kJ/m^2$ whereas dry CF & GF source, CF prepreg, BMW CF and Kevlar are all at least more than 4 times higher. Impact resistance is a very important property for ship flooring application as in a shipping container there are impacts multiple times a day and the floor should be able to withstand this abuse without failing catastrophically.

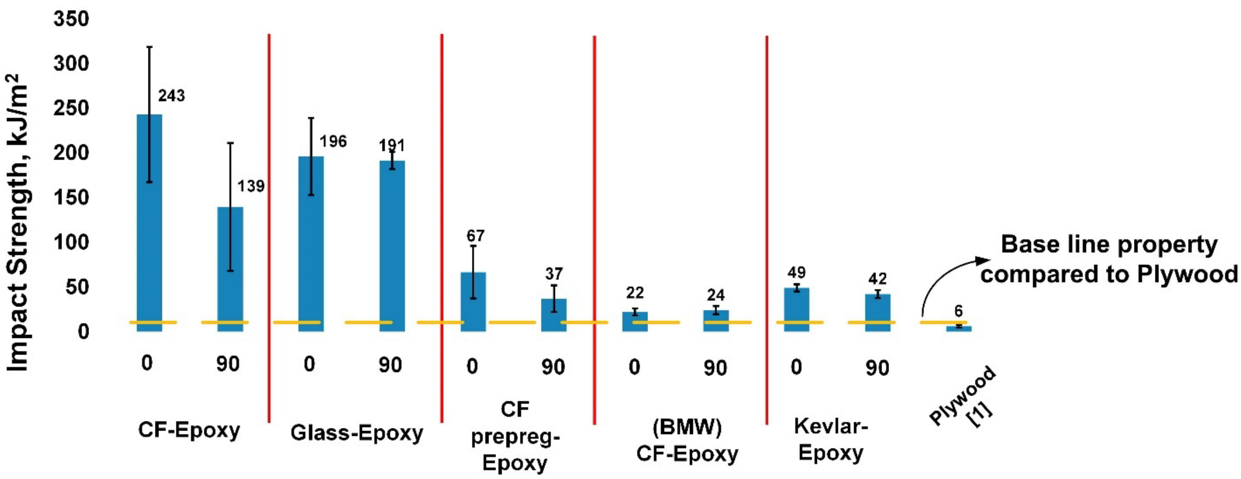



*Figure 9. Impact strength of CF, GF, CF prepreg, BMW CF and Kevlar epoxy specimens all processed at high psi are presented. Marine grade plywood is presented for comparison and to establish base line.*

**Change in Scope**

The original plan for the project was to manufacture 4 ft x 8 ft panels to be evaluated at Wabash National. The large panels were to be produced at GreenTex Solutions. However, the press at GreenTex was not capable of handling the tonnage needed to produce high psi pressure on such a large area. GreenTex made arrangements to produce the panels at Clemson University. This is when Covid 19 hit and Clemson facilities were not available. Where to produce the larger panels was still being decided when this report was completed.

This project was very aggressive for a short program. The project was originally only meant to be a six-month study and it was extended several times. A significant amount of data was generated on formulations for GreenTex to use to make the flooring in production. However, the final two milestones and the Go/No Go associated with large panels were not achieved. The team decided to close out this project since the large panels could not be produced at UTK. GreenTex considers the project successful and will continue to pursue making flooring panels.

# 7. BENEFITS ASSESSMENT

There are several key innovations that are part of the GreenTex Solutions technology. These include but are not limited to: (a) We have described the shortcomings of the incumbent materials used in transport trailers and shipping containers. The current project advances the state of the art by providing a moisture-resistant, corrosion-free, lower weight (by 30% compared to wood), lower thickness panel manufactured from fully recycled composite waste streams that would otherwise go into landfill. Preliminary work has shown strong indications of the success and scalability of the proposed recycled materials and configuration; (b) We also provide ‘materials by design’ and ‘design for manufacturing’ innovation in the choice, conversion and utilization of the waste stream from a variety of sources such as truck trailer, aerospace, wind blade waste, raw materials production waste etc. This provides a high degree of flexibility of tailoring the design by hybridization on demand. Our designs for the proposed floor component will comprise strategic material placement by design, such as carbon fiber waste streams for stiffness dominated regions, and glass fiber waste streams in impact dominated sections as examples; (c) The ability to incorporate generative designs is another notable innovation of the work. For example, assuming ribs geometries in the floor structure, we will be able to conduct generative design where ribs thicknesses, shape, geometry and transitions will be enabled through integrated process and intelligent processing.

# 8. COMMERCIALIZATION

The GreenTex process is simple and cost effective. Cured fiberglass composite parts from Wabash National, wind energy blades, cars/trucks and boats and cured carbon composite parts (airplanes, bikes, golf club shafts) are crushed to fine “dust-like” material. To prepare a sandwich construction (which comprises a core and face sheets), this material is mixed with a minimal amount of resin to form the core of the part. Fiberglass edge trimming is used to form the top and bottom laminates (face sheets). The sandwich layup is consolidated in a press. GreenTex is currently evaluating various commercial opportunities.

# 9. ACCOMPLISHMENTS

GreenTex technology is not only proving the recycling solutions, but also represents a cost effective solution that cuts down the lead time to introduce composite materials in the transportation and shipping industries. This process also further reduces the energy consumption for processing recycled composites.

# 10. CONCLUSIONS

The work provided the pathway to utilize scrap from edge trimmings, offcuts, manufacturing waste in dry carbon, glass and Kevlar fibers and carbon fiber prepregs. The work demonstrated simple conversion of these scrap fibers along with vinylester resin into useful composite laminates and sandwich constructions with recycled cores. The results suggested that laminate compaction had significant influence on the resulting flexure, interlaminar shear and impact properties. Average flexure strength of 300 MPa was attained across the CF & GF dry fiber sources and CF pre-preg and average flexural modulus of ~25 GPa. The interlaminar shear strength averaged ~30 MPa. The impact strength average was 100 kJ/m$^2$. Baseline marine grade plywood in contrast has flexural strength of 60 MPa, flexural modulus of 11 MPa and impact strength of 6 kJ/m$^2$. Every recycled composite variant regardless of the source of the scrap had order of magnitude or more higher values of all mechanical properties. The end eventual goal of this panel development is to complete the qualification/certification testing of the flooring panels and provide the panels for commercialization. The ultimate objective is to transform the recycling process technology to commercial scale producing full-size transportation flooring panels.

# 11. RECOMMENDATIONS

The best recommendation is if the industries see the value and ease of employing recycled composites for various applications, there will be confidence to use recycled composites with unbiased view towards cost. A rapid means of qualification of the panels for shipping containers needs to be established.

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