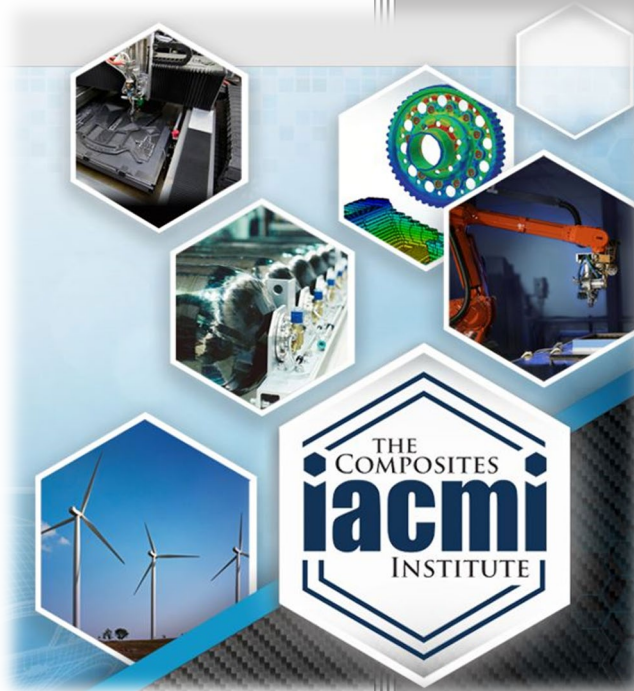


6.26 Low Cost Basalt Fiber for Automotive Applications



Author: Jared Stonecash
Date: January 2022

Final Technical Report
PA16-0349-6.26-01

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The information, data, or work presented herein was funded in part by the Office of Energy Efficiency and Renewable Energy (EERE), U.S. Department of Energy, under Award DE-EE0006926

Low Cost Basalt Fiber for Automotive Applications

Principal Investigator: Jared Stonecash

Organization: Michelman

Address: 9080 Shell Road Cincinnati, OH 45236

Phone: 513-794-4247

Email: jaredstonecash@michelman.com

Co-authors:

University of Tennessee-Knoxville: Stephen Young, Josh Crabtree

Mafic USA: Lee Berry

Volkswagen Group of America, Inc.: William Henken

The authors would like to acknowledge the work of many team members that made this work possible, from project guidance, to administration, and to execution. This extended and talented team included:

Michelman, Inc.: Steve Bassetti

University of Tennessee: Dayakar Penumadu, Uday Vaidya

Mafic USA: Jeffrey Thompson, Mike Cretella

IACMI: Erin Brophy, Catherine Ross, and John Unser

Department of Energy: Chad Schell

Oak Ridge National Laboratories: John Lindahl

Volkswagen: Hendrik Mainka

The team would also like to thank material suppliers and processors for their advice, donation of materials, and willingness to support the project:

Owens-Corning, IDI Composites International, INEOS, Omya, Superior Oil

Date Published: (January, 2022)

Prepared by:
Institute for Advanced Composites Manufacturing Innovation
Knoxville, TN 37932
Managed by Collaborative Composite Solutions, Inc.
For the
U.S. DEPARTMENT OF ENERGY
Under contract DE- EE0006926

Project Period:
(July 2019 – December 2020)

Approved For Public Release

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1. LIST

1.1 List of Acronyms

APE = alkylphenol ethoxylates
AR = Assembled Roving
DOE = Department of Energy
DR = Direct Roving
E-CR = Electrical/Chemical Resistance
EV = electric vehicle
GPa = Giga Pascals
IACMI = Institute for Advanced Composites Manufacturing Innovation
ICE = internal combustion engine
INEOS = INSpec Ethylene OxideSpecialities
IDI = IDI Composites International
kN = kilo Newton
LOI = Loss on Ignition
MEB = German: **Modularer E-Antriebs-Baukasten**; English: modular electric-drive toolkit)
MPa = Mega Pascals
NCF = non-crimped fabric
OC = Owens Corning
OEM = Original Equipment Manufacturer
ORNL = Oak Ridge National Laboratories
PUD = polyurethane dispersion
REDOX = reduction-oxidation
SMC = sheet molding compound
SUV = sport utility vehicle
U.S. = United States
UTK = University of Tennessee-Knoxville
UTS = ultimate tensile strength
VOC = volatile organic compounds
TFP = tailored fiber placement
VE = vinyl-ester
VW = Volkswagen

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Appendix B. Technical Data Sheet for IDI’s STC 2400 Series SMC

Appendix C: Results of SMC Panel Fabrication and Mechanical Testing. (to be added at

undetermined date in future)

2. EXECUTIVE SUMMARY

Vehicle lightweighting is an essential component to the automotive industry to improve fuel economy of internal combustion engine (ICE) vehicles to meet ever improving emission standards and to improve the range of electric vehicles (EV). Composite materials offer high specific modulus and specific strength, which makes them appealing for these light weighting efforts. Sheet molding compounds (SMC) are particularly interesting from an automotive perspective because of the relatively low cost and high volume of producing SMC parts. Traditionally, composite materials for automotive application are glass fiber reinforced because of the attractive price - performance ratio, but basalt fibers are a cost and recycling competitive reinforcement alternative in this market.

The aim of this project was to examine the feasibility of utilizing basalt fiber for automotive applications. More specifically, an effort was made to examine different fiber sizings on basalt fiber combined with vinyl-ester (VE) resin, and their performance as part of an SMC process. In addition to offering vehicle lightweighting with fiber reinforced polymer composites, basalt fiber is a fully recyclable material and thus supports the IACMI technical goal of: **Demonstrate that the technology is capable at a sufficient scale for >80% recyclability or reuse of fiber reinforced polymer composites in five years into useful components with projected cost and quality at commercial scale competitive with virgin materials on a pathway to 95% recyclability or reuse starting in ten years.**

Three different fiber sizings were applied to a continuous roving of basalt fiber and compared to a traditional Electrical/Chemical Resistance (E-CR) glass fiber that is typically used in these types of applications. Fiber tows were examined for Loss on Ignition percentage (LOI%), Tex, and tow strength. Some sizings clearly outperformed others, and the ability to process these fibers on a pilot scale SMC line was demonstrated. A test plan for the manufacturing and mechanical testing of SMC panels was developed. This work continues outside the time frame allocated for this project. When this work is completed, it will be added to this report and posted as **Appendix C**.

Glass fiber reinforced SMC materials have already proven feasible as a light weighting method for traditionally steel parts like the Volkswagen (VW) Atlas lift-gate (**Figure 1**); this project team is seeking the feasibility of basalt fiber as a drop-in replacement for glass fiber reinforced SMC. Sizing development for basalt fibers has proven that the mechanical properties are better than E-glass and closer to S-glass, which makes it an interesting material for SMC applications. Better mechanical properties translate to less material needed to achieve load case requirement for target applications.

The business case has already been demonstrated for 100,000 parts per year of glass fiber reinforced SMC Atlas lift-gates compared to traditional steel manufacturing processes. Reduced overhead and assembly costs are offset by glass fiber SMC higher cost per kg beyond 100,000 parts per year, which is still a relatively low volume for the automotive industry. For basalt fiber reinforced SMC to become feasible for automotive applications, the price-performance ratio has to be precisely determined. Based on the mechanical performance it is possible to establish a range

of applications and technical solutions in which the potential of basalt SMC can be utilized, while the price of the material can be used to compile the business case for such applications. Based on these business cases and the sustainability indicators, glass fiber reinforcement (or other) materials can be directly substituted.

Volkswagen's commitment to reducing carbon emissions cannot be understated. Basalt fiber shows promise of reducing the carbon footprint in SMC materials, especially if sizing optimizations can be made with thermoplastic based SMC. To fully realize the value of basalt fiber reinforced materials, a lifecycle cost analysis should be performed on basalt's production and recycling, and then compared against E-glass. From this assessment, a true judgement can be made on the commercialization potential of this material.



Figure 1. Example of Fiber Reinforced Polymer Composite Liftgate

As a conclusion, we can state that Mafic basalt fiber is not a direct replacement for E-glass or E-CR glass based on price, but should be considered a technical solution when E-glass does not provide adequate performance in a composite design and S-glass, aramid and carbon fibers are too costly. Mafic basalt fiber can be placed on the high-performance fiber spectrum next to S-glass for performance but at one third the price. It should be considered for more technically challenging structural designs wherein the performance can demonstrate 20-25% performance enhancement over E-glass to elicit more strength or a weight reduction. Both Michelman and Mafic produce thermoplastic sizings which, in combination with Nylon and polypropylene resin and fibers, can further advance high speed composite implementations while maintaining an eco-friendly manufacturing process.

3. INTRODUCTION

Composites continue to find application and growth in various markets, including aerospace, automotive, infrastructure and others. Several reasons for this growth are inherent in the ability to consolidate functionality through efficient design, allow for the design flexibility that composite processes utilize and lastly, to leverage the cost/performance benefits that composites offer. However, when considering the use of composites, there has long been a significant cost-performance gap in the market between carbon fiber and fiberglass composites. Basalt fiber has the potential to fill this gap in the market as a high-quality material that performs mechanically significantly above E-glass (**Figure 2**) and at a price that mitigates the premium that carbon fiber typically carries. The chart is a reasonable comparison of many materials for modulus, tensile strength and strain. **Figure 2** shows an approximate scale but lacks detail due to the scale of the Y-axis. Tensile testing is a delicate process and dissimilar materials are tested with variation in test methods. Due to the scale of the Y-axis, it is difficult to identify a small increase of 20% Tensile strength compared between E-glass and Basalt. Arguably the modulus for S-glass could be higher than E-glass based on changes in the sizing chemistry. From standardized lab tensile testing of continuous fibers, Mafic finds 20-25% more modulus and tensile strength of its Basalt fiber compared to standard E-glass formulation. Mafic's basalt performance is comparable to that of S-glass. The data represented in **Figure 2** is a strong and legitimate visual to compare various materials on one scale.

In this project and through various discussions, several OEM's (including Volkswagen as a core team member) have expressed interest in understanding the value-in-use of basalt fiber composites. This interest spans multiple polymer systems – both thermoplastic and thermoset. Light-weighting continues to drive the automotive and aerospace industries and composites provide potential solutions to achieve the targets that have been defined by both government regulations and industry desires. This provides strong motivation for developing an optimized interface for both thermoset and thermoplastic resin systems to meet the anticipated demands from automotive industry.

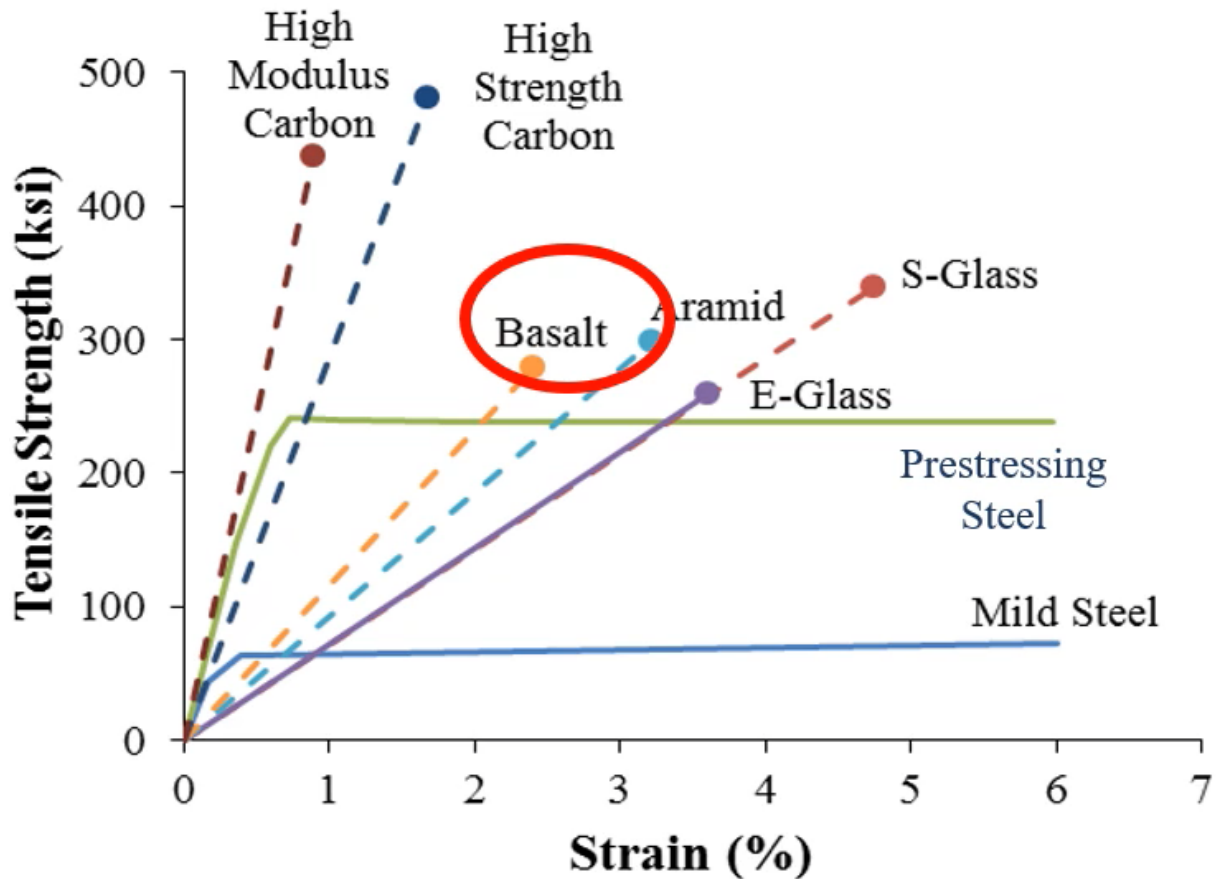


Figure 2. Basalt Fiber Tensile Strength Compared to Alternative Materials.

Mafic is a market leader in the development of basalt fiber and its technology can be an enabler in cost effectively light-weighting automotive parts. Vinyl Ester (VE) SMC's have been identified as initial resin system of high interest. Thermoplastics were also identified as an area of interest, but due to time and budget constraints, this project focused on VE thermoset technologies. By way of example, Mafic has shown performance in epoxy composites demonstrating low cost weight reductions over glass fiber composites and without the energy intensive procedure required for carbon fiber. However, Mafic has not yet demonstrated these characteristics in VE systems. Sizing chemistry appears to be a key barrier to achieving similar results with vinyl ester, as has been shown in epoxy systems. As Michelman is a global leader in the development of fiber sizings, we believe we are in a unique position to help improve the performance of basalt fiber in these polymeric systems. Additionally, automotive OEMs have stated their interest to use basalt fiber materials in VE systems to further their light-weighting and performance improvement objectives.

The project considered several critical aspects associated with interfacial performance and accordingly the mechanical properties by considering a targeted and promising list of sizings for basalt fiber in a thermoset vinyl ester resin. Vinyl esters are an important group of thermosetting

plastics, and are widely used for their toughness and resistance to a broad range of chemical environments. They have mechanical properties similar to those of epoxies but can be handled easily at room temperature offering greater control over cure rate and reaction conditions. Therefore, as one looks at the potential commercialization of basalt fiber, it is important to note that the ultimate performance of the composite is influenced by the efficiency of the sizing to translate mechanical load/energy thru the polymeric matrix to the basalt fiber.

4. BACKGROUND

In this project, Mafic supplied suitable basalt fiber types, optimized for low cost applications associated with targeted VE-based SMC compounding material. Michelman identified high prospect sizings for the targeted vinyl-ester system, and worked closely with the University of Tennessee-Knoxville (UTK) team on detailed characterization-based studies. These included the use of highly precise nano-tensile testing for single fiber strength and modulus, and properties at the fiber tow level were also determined using tensioned strands with optimized fiber surface for VE. Initial trials were performed at IDI Composites International. Despite several attempts, the fiber simply would not run in their SMC machine dedicated to research and development trials. IDI's system has options to pull fiber from creel or feed through tubes, but this fiber was simply "de-bundling" too easily to feed. Trials performed at Oak Ridge National Laboratories were more promising as the fiber is gravity fed from above on their SMC line. Section 5 of this report contains more details.

Future work is planned to fabricate compression molded, flat, SMC test panels, and test for properties such as tensile, flex, and in-plane shear. Detailed characterization including relative performance will be quantified considering fiber orientation and matrix rich regions as a function of targeted charge placement. Failure zones will be carefully studied using optical and electron microscopy to qualitatively evaluate the fiber interface and translations observed. In summary, for the first time, this project will lead to a low-cost, basalt-based SMC product for the potential molding of a large automotive component, such as a VW liftgate. This project has shed additional light on the critical role of sizing chemistry for improved mechanical performance of reinforced composites. Although the importance of sizing is often acknowledged, a study such as this did not exist (for basalt fibers) in the literature and forms an important first step in developing next generation thermoplastic and thermoset basalt fiber-based composites in a rigorous and comprehensive fashion.

5. RESULTS AND DISCUSSION

5.1 Sized Basalt Fiber Selection

The original plan was to have Mafic provide unsized fiber to Michelman, and Michelman would size the fiber in a secondary process on a sizing line. This did not prove to be feasible as the unsized fiber was very difficult to handle and the forming cake tended to collapse on itself (**Figure 3**). The plan was quickly revamped to have the aqueous phase samples sent to Mafic USA in Shelby, North Carolina and then repackaged for safe delivery to Mafic Ireland where direct sized fiber was produced.



Figure 3. Unsized Basalt fiber from Mafic.

Michelman down-selected sizing candidates based on company expertise and research, including a previous IACMI project that focused on improving interfacial properties of carbon fiber/vinyl-ester composites. (1). As part of this collaborative project, Michelman submitted two aqueous sizing candidate systems for basalt fiber/vinyl-ester composites. These sizings were shipped to Mafic in Shelby, North Carolina. Subsequently, basalt fiber was produced by Mafic in Ireland with three candidate sizing systems:

- “E” sizing: Michelman experimental sizing product; which is a modified polyurethane dispersion (PUD), functionalized for vinyl-ester thermoset resins. (58% solids content)
- Hydrosize HP302: Michelman commercial product based on phenoxy dispersion. This product offers a high glass transition temperature and is APE free. (30-33% Solids content)
- 5X1: Mafic’s own basalt fiber sizing product.

For this project, Mafic produced:

1. 24 Spools (3 kg each (6.6 lbs.)) of the “E sizing” component from Michelman – MultiEnd Roving 2400 Tex
2. 24 Spools (3kg each (6.6 lbs.)) of the “HP-302” component from Michelman – MultiEnd Roving 2400 Tex

3. 24 Spools (3 kg each (6.6 lbs.)) of the 5X1 sizing from Mafic – MultiEnd Roving 2400 Tex

Total weight was approximately 476 lbs. (216 kg) of sized basalt fiber. A bobbin of each sized fiber was sent to UTK for evaluation. Mafic used the same rock source, furnace conditions and melting parameters to produce the precursor fiber as Direct Roving 600 Tex, 17-micron filaments. The various chemistries (sizes) were applied with the same coating parameters for speed of applicator roller, angle of applicator and the usual contact points. The goal was to standardize the fiber process and only vary the sizing chemistry. Quality control testing was performed on the basalt fiber material with three different sizings at Mafic, and those results are detailed in **Table 1**.

Table 1. Comparison of 3 Basalt Fiber Sizings

Product Type	Basalt Fiber	Basalt Fiber	Basalt Fiber
Sizing	5X-1	E-Sizing	HP302
Product Name:	Assembled Roving 2400-17	Assembled Roving 2400-17	Assembled Roving 2400-17
Production Start Date	3/25/2020	3/24/2020	3/25/2020
Production Finish Date	3/25/2020	3/25/2020	3/25/2020
Batch No:	20034-084	20032-084	20033-084
Linear Density (Tex) (g/km)	2301	2308	2236
Moisture Content % Weight of Fiber (%)	0.03	0.01	0.02
Loss on Ignition (LOI%) on weight of fiber (%)	0.82	0.51	0.69
Tenacity (Breaking Strength) (cN/tex)	64.85	61.06	48.67

For comparative purposes, a corrosion resistant, E-glass fiber (Advantex E-CR, ME1960) fiber from Owens Corning was chosen. ME1960 is a multi-end roving recommended for SMC (**Appendix A**). It has a TEX of 2400, and is designed for the manufacture of SMC used in general purpose and transportation applications such as sanitary products, heavy truck/bus/train interior and exterior parts. The LOI% was listed as 1.30%. **(2)**. It was also selected as this was a material evaluated on a previous IACMI project that VW and UTK participated in, and it is the same fiber utilized by IDI Composites International in their STC 2400 Series SMC **(3)** (**Appendix B**). The mechanical properties of basalt single fiber and tows were evaluated for SMC applications using the three fiber sizing types at UTK (**Table 2**). Fiber diameters for all four fibers evaluated appeared very consistent (**Figure 4**).

Table 2. Summary of Sized Basalt Fiber and Glass Fiber

Fiber ID	Fiber Type	Sizing	Mean Fiber Diameter (μm)	Manufacturer
BF7	Basalt	Michelman Hydrosize® HP302	16.7 (1.5)	Mafic/Michelman
BF8	Basalt	Mafic 5X1	16.6(1.3)	Mafic
BF9	Basalt	Michelman E-Sizing	16.2 (1.1)	Mafic/Michelman
GF	Glass Fiber	-	15.9(1.6)	Owens Corning

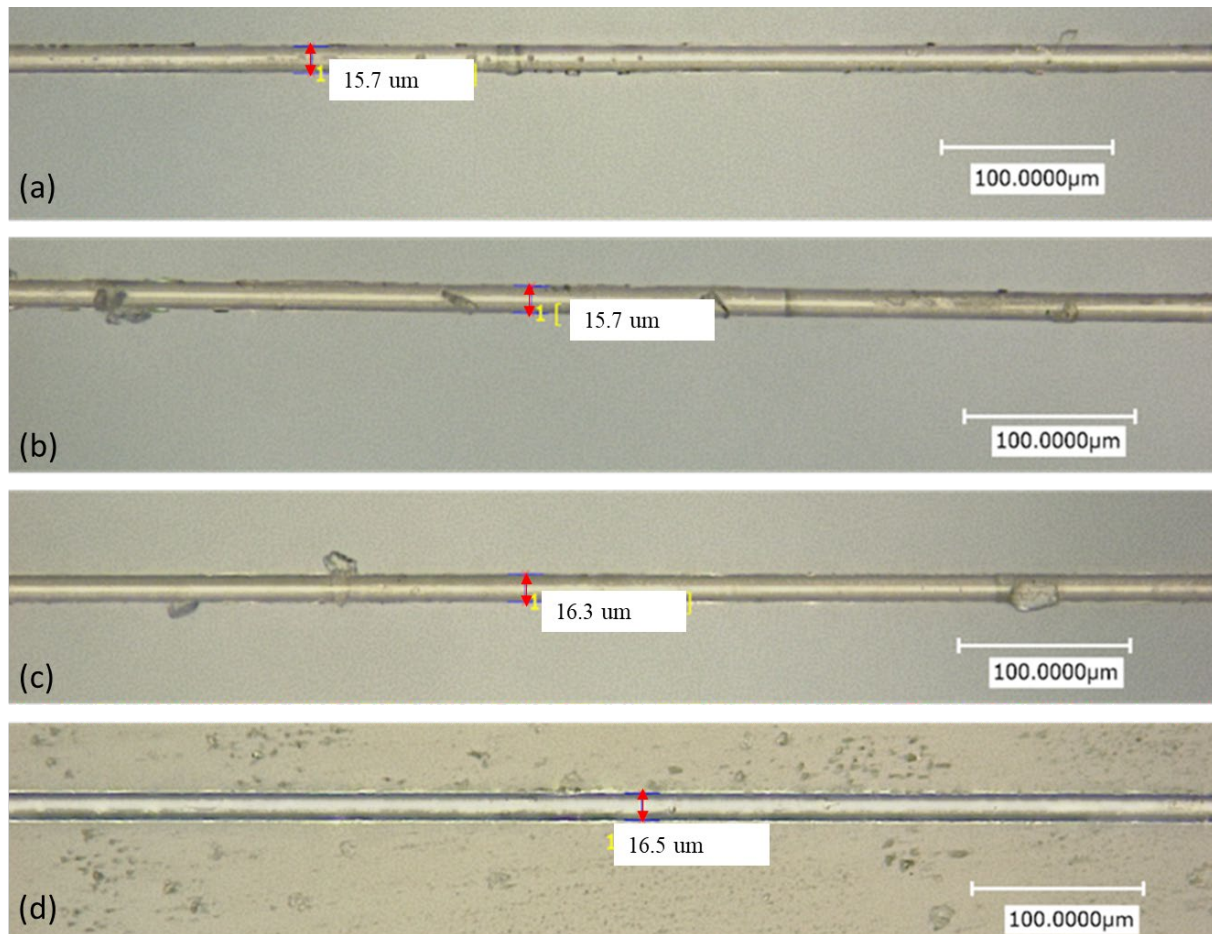


Figure 4. Optical micrographs of single basalt fibers sized with (a) Michelman Hydrosize® HP302, (b) Mafic 5X1, (c) Michelman E-Sizing and (d) single ME1960 glass fiber.

5.2 Tensile Behavior of Single Fiber Basalt Fiber

Figure 5 illustrates the details of mechanical testing setup for single fibers, where each fiber was carefully mounted onto a metal template using an adhesive and allowed to cure overnight prior to testing. The fibers were tested in accordance with ASTM C 1557-03 and ISO 11566 standards

using a Nano UTM load frame instrument. A total of eleven single fibers were tested for each fiber type. As shown in **Table 3**, all three basalt single fiber types exhibited higher failure stress, strain to failure %, and modulus values compared to the glass fiber.

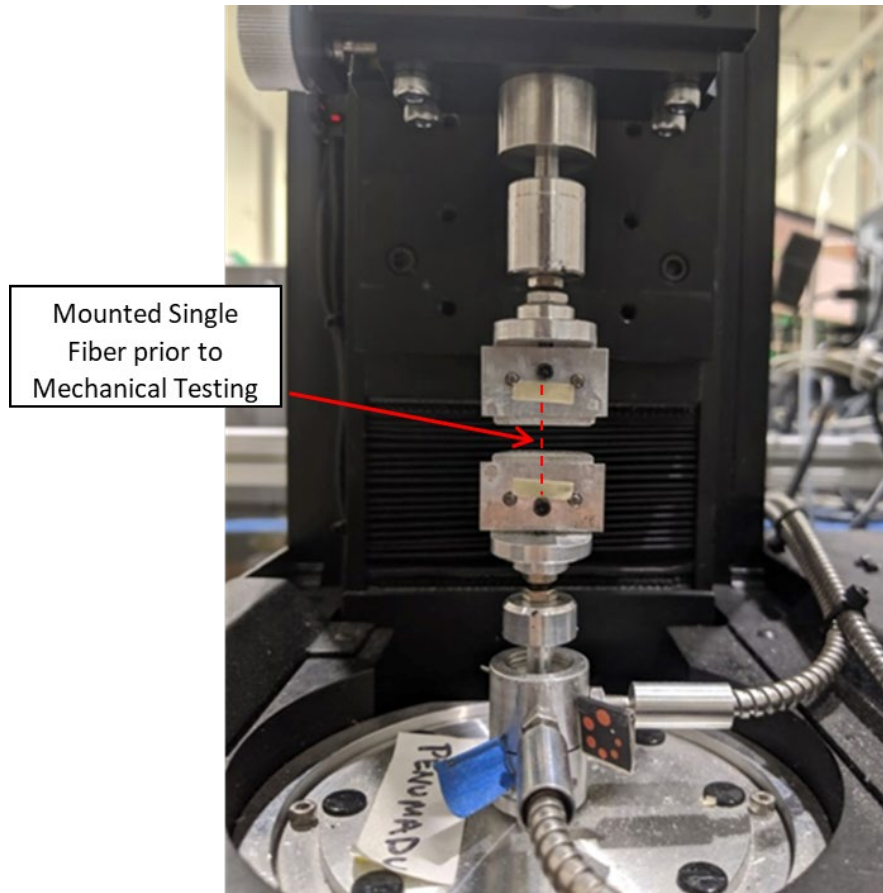


Figure 5. Tensile testing of single fibers in accordance with ASTM C 1557-03 and ISO 11566.

Table 3. Summary of Single Fiber Failure Stress, Failure Strain, and Modulus comparison of Basalt and Glass Fiber

Fiber ID	Fiber Type	Sizing	Failure Stress (MPa)	Engineering Failure (%)	Modulus (GPa)
BF7	Basalt	Michelman Hydrosize® HP302	2068(454)	2.69(0.69)	79(6)
BF8	Basalt	Mafic 5X1	2477(614)	3.10(0.71)	82(7)
BF9	Basalt	Michelman E-Sizing	2201(330)	3.07(0.47)	74(3)
GF	Glass Fiber	-	1500(432)	2.11(0.62)	73(2)

Note: The standard deviation values are in the parentheses.

The failure stress of BF7 (2068 MPa; +32%), BF8 (2477 MPa; +49%), and BF9 (2201 MPa;

+38%) were higher than GF (1500 MPa). The modulus of BF7 (79 GPa; +8%), BF8 (82 GPa; +12%), and BF9 (74 GPa; +1%) were higher than GF (73 GPa). The scatter on strength for reinforcing fibers depends on the type of reinforcement and its manufacturing conditions. As an example, T700 Standard Modulus carbon fiber has lot less scatter (5 to 10 %) when measured using identical sample and testing conditions as was used in this study. we have seen more scatter with Glass and Basalt than that of Commercial Carbon fiber (typically around 5 to 8 %). Also, Carbon Fiber (single tow) is not as prone to twisting as the Glass and Basalt (multi-end roving) which lead to this larger scatter along with inherent material defects.

5.3 Manufacturing of Basalt Fiber Tows using a Manual Method

Two types of resin systems (epoxy and vinyl ester) were considered to evaluate the sizing effects on mechanical properties of the basalt fibers and glass fiber types. The epoxy system consisted of Epon 862 (Diglycidyl Ether of Bisphenol F), Cardura E10P (Glycidyl Ester), and EpikureW (Aromatic Amine Curing Agent) using a mix ratio recommended by manufacturers. Similarly, the vinyl ester system consisted of Derakane 780 (vinyl ester epoxy) and Trigonox C (tert-Butyl peroxybenzoate) using a mix ratio recommended by manufacturers. The tow infusion was conducted manually based on the ASTM D4018 standard. As depicted in **Figure 6**, tows for each fiber type were cut to approximately 1 m in length. Each tow was submerged into a resin bath at room temperature and manually fed through a 1.55 mm diameter Wilton # 2 cake tip, for collimation and to remove excess resin.



Figure 6. Example basalt fiber sized with (a) Michelman Hydrosized® HP302 (b) Mafic 5X1, (c) Michelman E-Sizing and (d) ME1960 glass fiber tow strands before infusion with epoxy and vinyl ester resin systems.

The tow strands were mechanically mounted onto a metal rack (**Figure 7**). For the epoxy infused tows, the tows were cured in an oven using a temperature profile of 93 °C for 1 hour and increased to 180 °C for 1.5 hours. For the vinyl ester infused tows, the tows were cured in an oven using a temperature profile of 100 °C for 30 minutes, immediately followed by an increase to 125 °C for 30 minutes, and finally increased to 150 °C for 1.5 hours. A portion of the cured tow strands were then cut to approximately 228.6 mm in length as shown in preparation for tensile specimens. The resulting tow strands were tabbed with \pm 45-degree glass fiber composite tabs (38 mm length x 12.7 mm width x 3.175 mm thickness) using an epoxy resin (West System 105) and epoxy curing agent hardener (West System 206) with colloidal silica adhesive filler (West System 406) as illustrated in **Figure 8**.

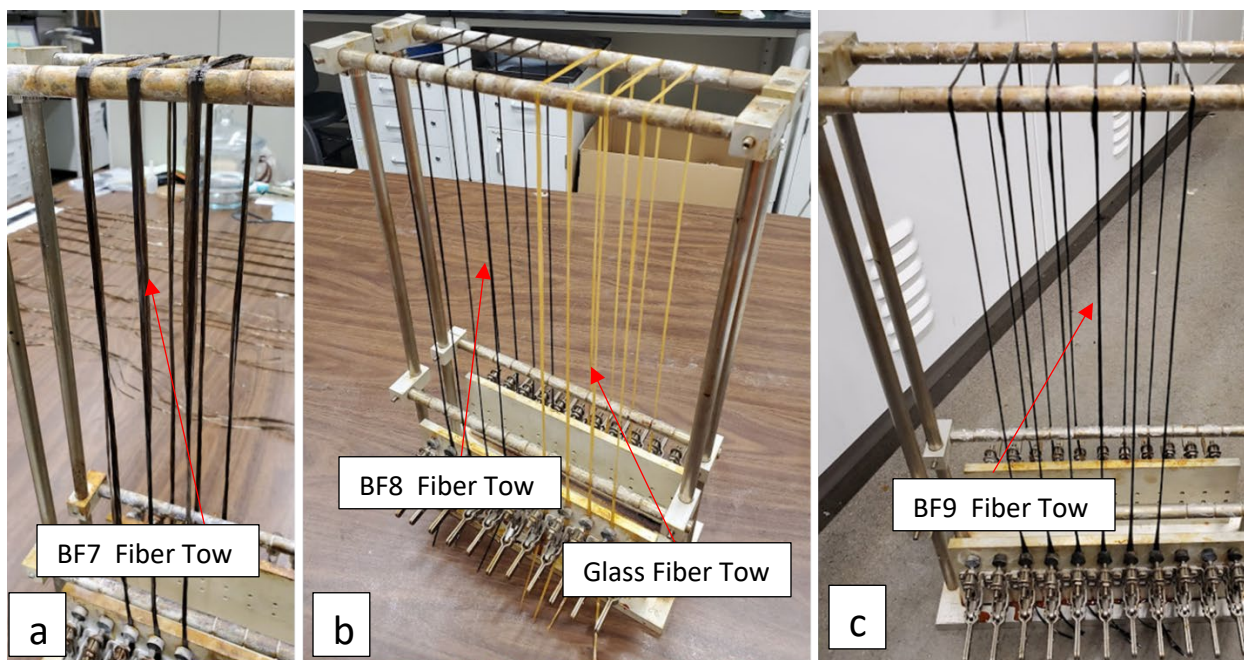


Figure 7. Example basalt fiber with (a) Michelman Hydrosize® HP302 (b) Mafic 5X1 and (c) Michelman E-Sizing tow strands after infusion with epoxy and vinyl ester resin systems. Example infused glass fiber tow strand is shown in (b).

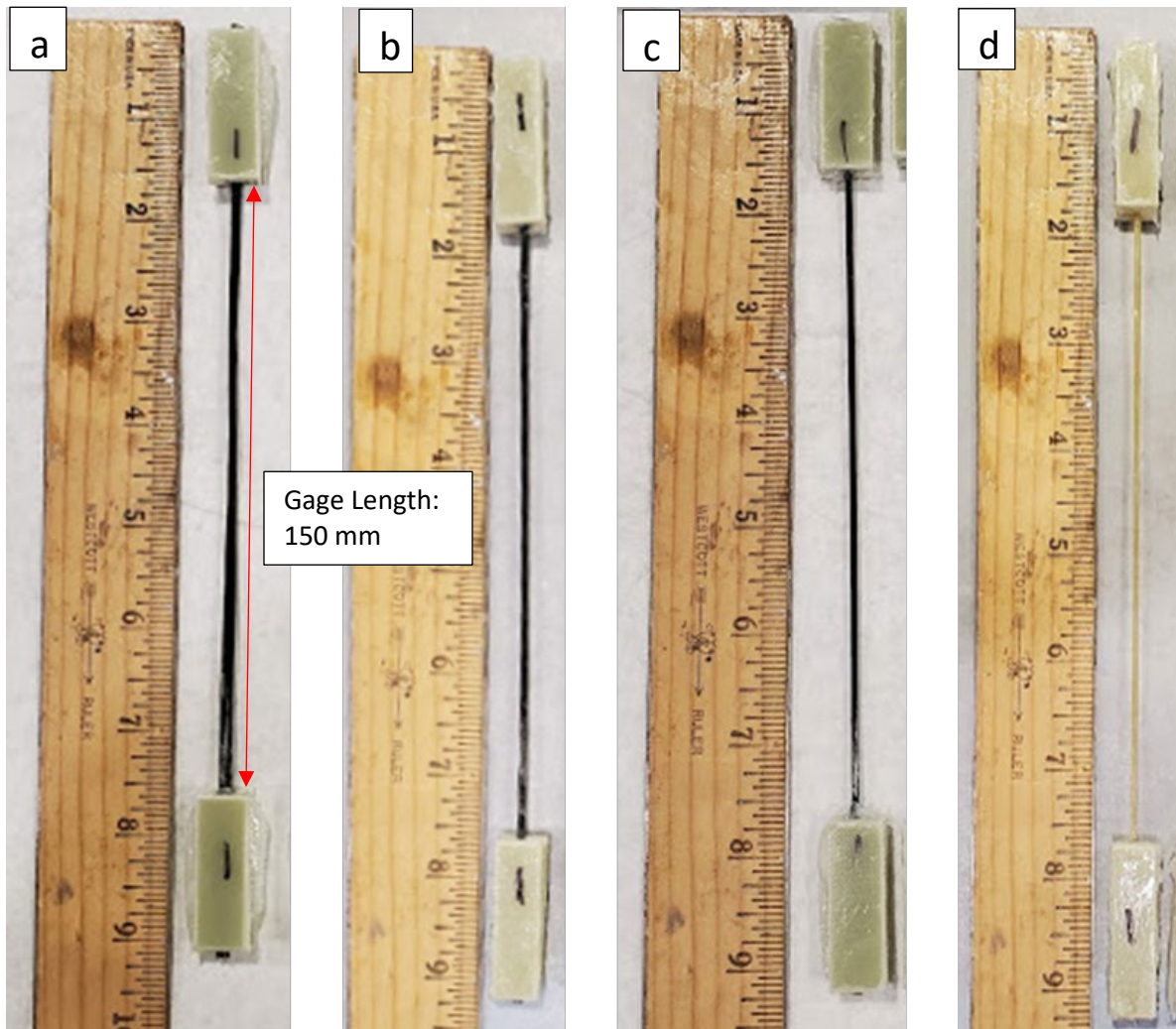


Figure 8. Example basalt fiber tow strands of (a) Michelman Hydrosize® HP302 (b) Mafic 5X1 and (c) Michelman E-Sizing. (d) Example glass fiber tow strand prior to mechanical testing.

Figure 9 details the mechanical testing of tow strands tested in accordance with ASTM D4018. The tows are loaded monotonically in tension on a servo-hydraulic load frame (MTS 858) with 24.5 kN load cell capacity at a crosshead rate of 30 mm/min. An extensometer (MTS 634.12E-4) was mounted on each tow strand prior to tensile loading to monitor the modulus and strain behavior until mechanical failure.



Figure 9: Example mechanical testing setup of tow strand.

5.4 Tensile behavior of Basalt Fiber Tow Strands Infused with Epoxy Resin

Table 4 lists the three sized basalt fiber tow mechanical properties compared to the glass fiber tow infused with epoxy resin. A total of ten infused tow strands were tested for each fiber type.

Table 4. Summary of Failure Stress, Failure Strain, and Modulus comparison of Epoxy Resin reinforced Basalt and Glass Fiber Tows

Sample	Fiber Type	Resin	Sizing	Failure Stress (MPa)	True Failure Strain (%)	Modulus (GPa)
TE-BF7	Basalt	Epoxy	Michelman Hydrosize® HP302	1915 (198)	2.58 (0.36)	77 (3)
TE-BF8	Basalt	Epoxy	Mafic 5X1	2634 (166)	3.73 (0.32)	81 (1)
TE-BF9	Basalt	Epoxy	Michelman E-Sizing	2879 (76)	4.12 (0.41)	82 (1)
TE-GF	Glass Fiber	Epoxy	-	2447 (180)	3.52 (0.42)	79 (1)

Note: The standard deviation values are in the parentheses.

The failure stress of TE-BF8 (2634 MPa; +7%) and TE-BF9 (2879 MPa; +16%) was significantly higher than TE-GF (2447 MPa). However, the failure stress of TE-BF7 (1915 MPa) was significantly lower (-24%) than TE-GF failure stress. Similarly, the modulus of TE-BF8 (81 GPa; +3%) and TE-BF9 (82 GPa; +4%) was higher than TE-GF modulus (79 GPa). However, the modulus of TE-BF7 (77 GPa) was lower (-3%) than TE-GF modulus.

5.5 Tensile behavior of Basalt Fiber Tow Strands Infused with Vinyl Ester Resin

Table 5 shows the three sized basalt fiber tow mechanical properties compared to the glass fiber infused with vinyl ester resin. A total of ten infused tow strands were tested for each fiber type. Unsurprisingly, the HP302-sized basalt fiber performed the worst. Unlike the E-sizing, this sizing is a commercial product that was not formulated specifically for vinyl ester resin systems.

Table 5. Summary of Failure Stress, Failure Strain, and Modulus comparison of Vinyl Ester Resin Basalt and Glass Fiber reinforced Tows

Tow Sample ID	Fiber Type	Resin	Sizing	Failure Stress (MPa)	True Failure Strain (%)	Modulus (GPa)
TV-BF7	Basalt(BF7)	Vinyl Ester	Michelman Hydrosize® HP302	1831 (120)	2.75 (0.36)	74(6)
TV-BF8	Basalt(BF8)	Vinyl Ester	Mafic 5X1	2618 (103)	3.72 (0.20)	81(3)
TV-BF9	Basalt(BF9)	Vinyl Ester	Michelman E-Sizing	2682 (55)	3.82 (0.18)	83(1)
TV-GF	Glass Fiber(GF)	Vinyl Ester	-	2595 (131)	3.92 (0.38)	82(3)

Note: The standard deviation values are in the parentheses.

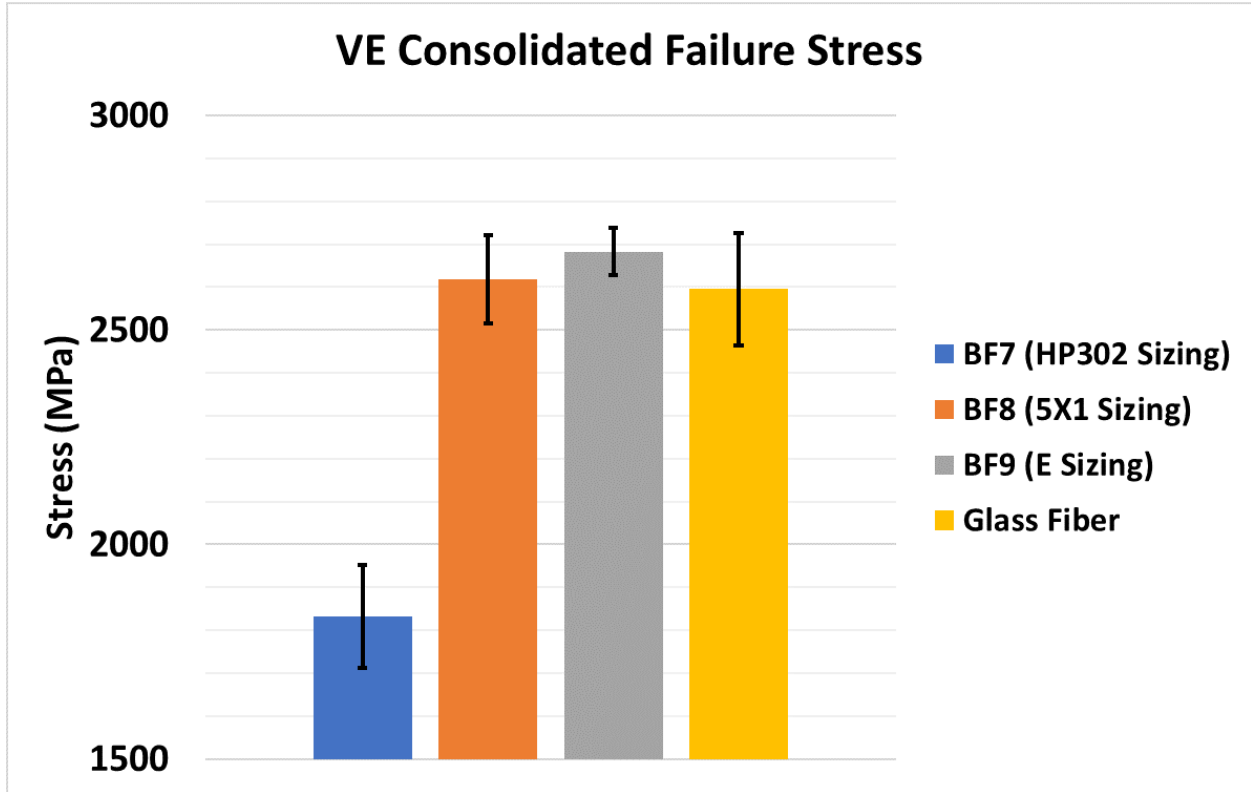


Figure 10. Tensile Stress Comparison.

The failure stress of TV-BF8 (2618 MPa; +0.9%) and TV-BF9 (2682 MPa; +3%) was slightly higher than TV-GF (2595 MPa). However, the failure stress of TV-BF7 (1831 MPa) was significantly lower (-35%) than TV-GF failure stress. The modulus of TV-BF7 (74 GPa; -10%) and TV-BF8 (81 GPa; -1%) was lower than TV-GF modulus (82 GPa). However, the modulus of TV-BF9 (83 GPa) was slightly higher (+1%) than TV-GF modulus.

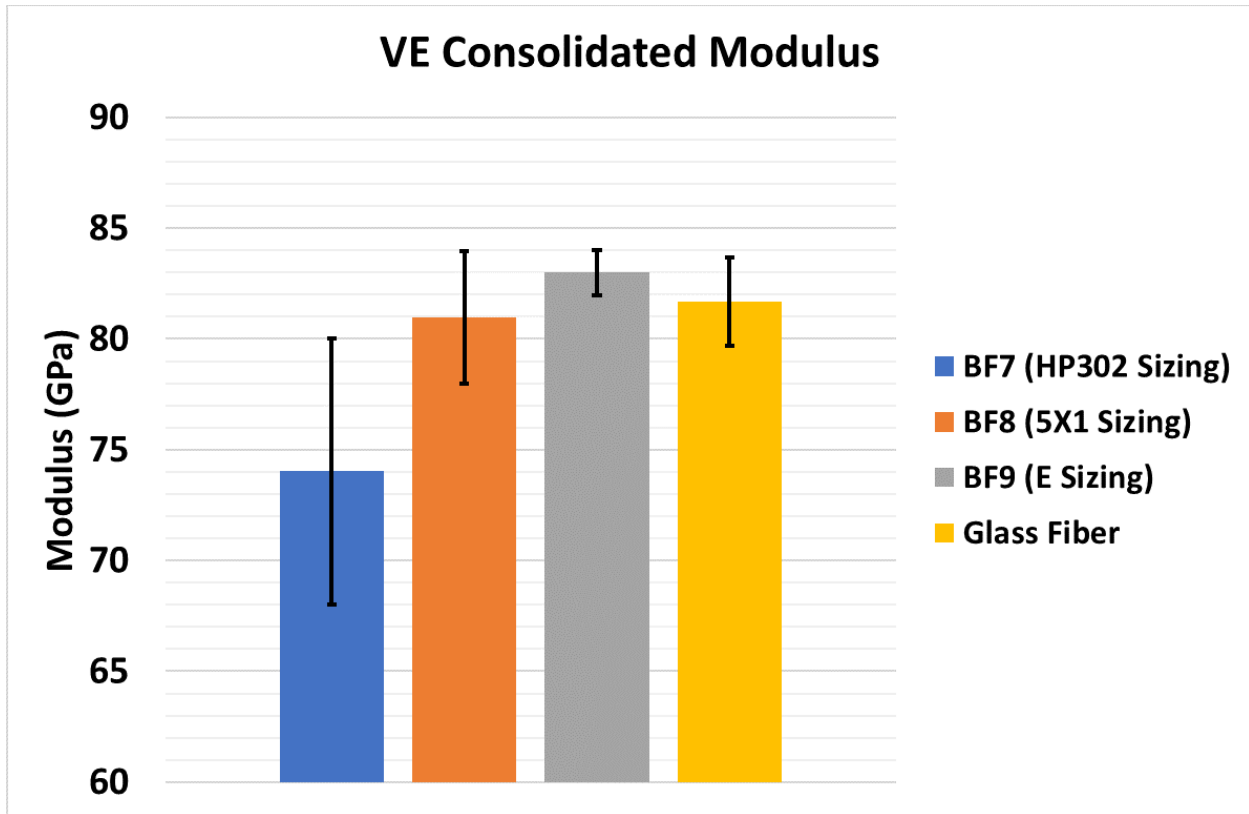


Figure 11. Tensile Modulus Comparison.

The next step was to plan the fabrication of SMC test panels and subsequent evaluation. Early on, continuous fiber panels were considered in order to evaluate the resin/fiber interface more readily. However, the nature of SMC paste resin systems dictated that we find a partner that could help with both the SMC resin ingredients and the fabrication of the SMC charges for subsequent compression molding. INEOS Composites was contacted about helping put together an ingredient list for the SMC resin formulation, and they generously supplied **Table 6** as well as the base resin ingredients Arotran 805 (**Figure 12**) and thickening agent Arotran 241. These ingredients were either sourced by Michelman or UTK, and they are currently on-hand at Oak Ridge National Laboratories (ORNL) as part of panel fabrication trials. For the basalt fiber panels, the ME1960-2400 glass will be replaced with basalt fiber with the same 50% weight fraction. IDI was also consulted and recruited to help with panel fabrication, and agreed with the selection of Arotran 805. It was determined that Derakane 780 is similar, but an older product offering, and IDI does use the Arotran 805 in newer automotive formulas so this made sense from a manufacturer’s perspective. The applications that use composites with Arotran 805 tend to be focused on structural performance (not aesthetics or lowest density) and that makes sense for potential replacement of glass with basalt. INEOS’s Arotran 805 polyester in weatherable SMC resin can be pigmented to white or black, offering the environmental plus of eliminating painting, which helps to reduce volatile organic compounds (VOCs) and waste products. (5)

Table 6. SMC Panel Ingredient Recipe

Component	Parts By Weight	Wt%	Function	Supplier
AROTRAN 805	104	30.60	Resin	Ineos
Trigonox 29C75	1.5	0.44	Initiator	Akzo Nobel
POWER BLOC 10	0.5	0.15	Stabilizer	Chempak Intl
OMYACARB 5	45	13.24	Filler	Omya
CF 02811	12.4	3.65	black color carbon black pigment	Chromaflo Technologies
AROTRAN 241	6.5	1.91	MgO based thickening agent for use	Ineos
ME1960-2400	170	50.01	Glass Fiber	Owens-Corning
Total	339.9			

AROTRAN™ 805

UV-STABLE SMC RESIN SYSTEM

At INEOS Composites, we strive to be good partners by meeting the expectations that are important to our customers. Today, that means we're focused on reducing our environmental footprint, while continuing to provide the innovative products and sustainable solutions our customers require.

Working with you to find solutions that meet your performance standards as well as end user expectations – that's where INEOS excels. Look no further than our work advancing truck bed performance as an example. Truck beds are exposed to long hours of sunlight which can result in undesirable gloss and color loss. In addition, painting and finishing parts after production can add countless work hours and substantial cost to an original equipment manufacturer's (OEM) operation. While in the field, truck beds see the kind of heavy duty use that can scratch away the paint and finish, resulting in exposed materials. The Arotran™ 805 resin system was designed to address these unique issues.

The Arotran™ 805 resin system provides a molded-in black color that eliminates the need for paint. The through-black color means that scratches and dings don't show in heavy duty applications. This material package is formulated using UV-stable technology providing excellent color stability and gloss retention even after extensive weather exposure.

Parts made from the Arotran™ 805 resin system will maintain gloss retention with minimal color change, even after 3,500 hours of UV exposure.

Applications

- Truck box
- Tailgate
- Rear hatch
- Tire cover

Benefits

- Produces large part with very complex design
- Tougher than thermoplastics
- Lowers emissions by eliminating the need for paint
- Provides good shrink control

Figure 12. INEOS Description of Arotran 805 (4).

Trials at IDI Composites International:

IDI was recruited as a project partner to help with the actual SMC processing and panel production. Initially, there were some environmental concerns around handling chopped basalt fiber. Mafic addressed these concerns as follows:

- Basalt is a naturally occurring inert rock; it is non-toxic and does not contain any toxic impurities.
- The surface coating is considered as non-hazardous.
- The basalt fiber provided by Mafic was a continuous basalt roving with a nominal filament diameter of 9-18 μm , well above the respirable size of 3 μm , and a moisture content of below 0.1% (6).

The basalt fiber was shipped to IDI's facility in Noblesville, Indiana. Despite several attempts, the fiber simply would not run in their SMC machine dedicated to research and development trials. IDI's system has options to pull fiber from creel or feed through tubes, but this fiber was simply "de-bundling" too easily to feed. It is very clearly 4 separate bundles in each roving, and they separate very easily (**Figure 13**). Previous work with some similar "split-tow" fibers has been trialed at IDI, but they are often sized such that they stay bundled in the roving until the chopper blades hit the fiber.

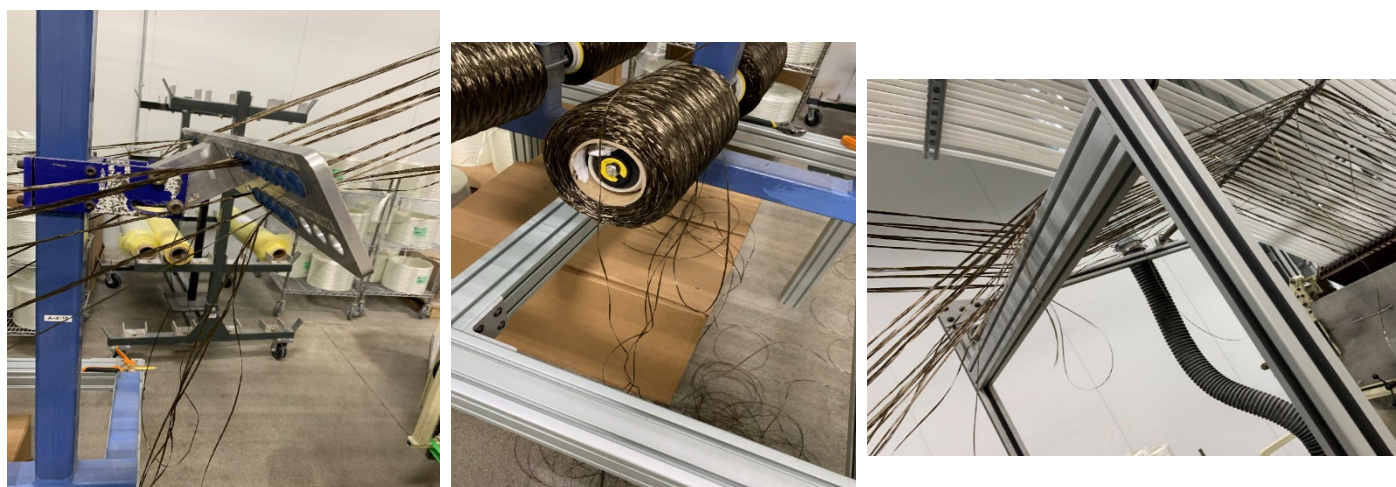


Figure 13. Unsuccessful Basalt Fiber Trials at IDI.

Further feedback on the fiber samples was as follows:

- HP302 fiber was trialed.
- The other 2 fibers did not look promising enough to put on the creel. 5X1 appeared to be the softest and HP302 was the stiffest.
- Each tow appears to consist of 4 separate tows that are very loose. They lose tension on the fiber.
- IDI's fiber creel has manual tensioners and nothing automatic, active, or self-

- compensating.
- A system with the spool feed from the top would allow gravity to assist and should reduce the problem we see at IDI. A system with an active tensioning system might also help. (The IDI system “pulls” the fiber from the chopper/cot and a system that also pushed the fiber might prevent some of the de-bundling.)

This last observation led the project team to consider the SMC line owned by UTK and recently re-installed at ORNL. UTK had also recently installed a large hydraulic press that could be used to compression mold the SMC test panels. A test panel matrix (**Table 7**) and a specimen test matrix (**Table 8**) were assembled with the goal of testing the best performing sizing systems from tow results and comparing them with traditionally utilized glass fiber. Obviously, time and budget drove the scale of these matrices.

Table 7. Panel Fabrication Matrix

Panel I.D.	Fiber	Resin	Sizing	Panel Size	QTY.
Baseline	ME1960 Fiberglass	Arotran 805 SMC		12" x 14" x .125"	2
E Panel	Mafic Basalt	Arotran 805 SMC	Michelman E sizing	12" x 14" x .125"	2
5X1	Mafic Basalt	Arotran 805 SMC	Mafic 5x1	12" x 14" x .125"	2

Table 8. Test Specimen Matrix

Panel I.D.	Fiber	Resin	Sizing	Panel Size	ASTM Method	Property	# of Specimens
Baseline	O.C. 1960 Fiberglass	Ineos Recommended SMC		12" x 14" x .125"	ASTM D7264	0 Flex	5
					ASTM D7264	90 Flex	5
					ASTM D3846	In-Plane Shear	5
					ASTM D638	Tension	5
					ASTM D3171	Physical Properties	3
						Microscopy	1
E Panel	Mafic Basalt	Ineos Recommended SMC	Michelman E sizing	12" x 14" x .125"	ASTM D7264	0 Flex	5
					ASTM D7264	90 Flex	5
					ASTM D3846	In-Plane Shear	5
					ASTM D638	Tension	5
					ASTM D3171	Physical Properties	3
						Microscopy	1
5X1	Mafic Basalt	Ineos Recommended SMC	5X1	12" x 14" x .125"	ASTM D7264	0 Flex	5
					ASTM D7264	90 Flex	5
					ASTM D3846	In-Plane Shear	5
					ASTM D638	Tension	5
					ASTM D3171	Physical Properties	3
						Microscopy	1

All the fiber materials were sent to UTK and subsequently to ORNL. All the resin ingredients were also sourced, and UTK performed some preliminary work on mixing procedure and viscosity as a function of out time. Initial feeding and chopping studies were performed by representatives from UTK and ORNL. This SMC line feeds the fiber in from above (**Figure 14**) and can feed up to 14 tows concurrently (**Figure 15**).



Figure 14. Basalt Fiber Trial on UTK’s SMC Equipment

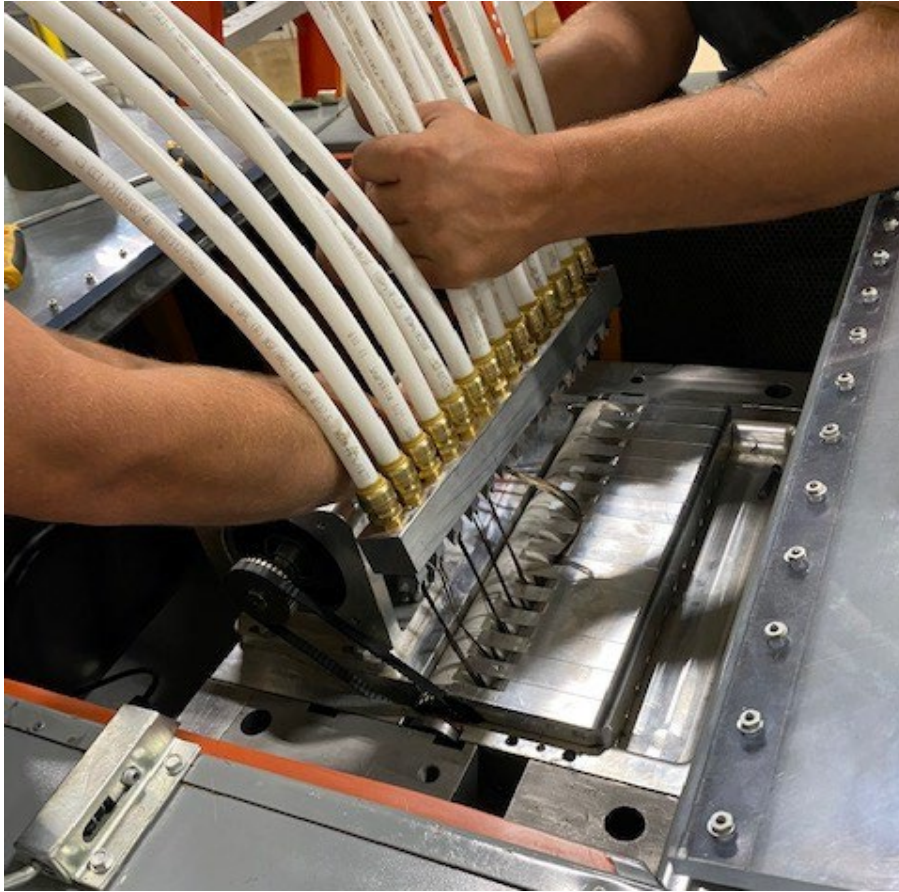


Figure 15. 14 Tows of Basalt Fiber Feeding into UTK's SMC Line



Figure 16. Chopped Basalt Fiber from UTK's SMC Line

Success was achieved in uniformly cutting basalt fiber to 1", 2", and ½" cut lengths (**Figure 16**). The team was also successful in setting the conveyor and chopping speed to accurately pay out 20 weight % fiber content. The work ahead will involve dialing this up to the 50 weight % required for SMC test panels. It was observed that a stiffer sizing and/or heavier coat weight of sizing has a direct effect on the cutting speed required. It was also observed that the 5X1 and HP302-sized fibers did not filamentize well; the E-sized fibers have not been trialed to date. In

general, the basalt fiber has a soft feel to it, and there is often a tradeoff between the ability to chop the fiber and the tendency for the fiber tow to debundle or filamentize after chopping. Further research is needed to fine tune the coat weight and sizing content specifically for the dual need in SMC processing for both choppability and filamentization of the fiber.

6. BENEFITS ASSESSMENT

Vehicle light weighting is an essential component to the automotive industry to improve ICE fuel economy to meet ever improving emission standards and to improve EV range. Composite materials offer high specific modulus and specific strength which makes it appealing for these light weighting efforts. Sheet molding compounds are particularly interesting from an automotive perspective because of the relatively low cost and high volume of producing SMC parts. Traditionally, composite materials for automotive application are glass fiber reinforced because of the attractive price - performance ratio, but basalt fiber has emerged relatively recently to compete in this market.

Basalt fiber offers some material properties between E-glass and S-glass; it has also been identified as being capable of mechanical property retention at higher operating temperatures, highly resistant to chemically corrosive environments as well as offering better acoustic damping than E-glass and S-glass. Basalt fiber also has the inherent benefit of being a naturally occurring mineral with virtually 100% yield on basalt fiber from the fiberized basalt rock. This material also benefits from easier recycling compared to glass fibers because basalt does not require a secondary process before melting again. Where basalt fiber lacks, in comparison of glass, is a well refined sizing system to accentuate the material's beneficial characteristics. Furthermore, variation in Basalt mineral content can decrease or increase the tensile, modulus, thermal and corrosive properties. Mafic is working with ASTM and other Basalt fiber producers to establish specification ranges for each component. The ASTM specification for E-glass formulation is the blueprint for the Basalt ASTM standardization.

This project offers the benefit of optimizing basalt fiber to perform better as a material individually and as a constituent of SMC materials. The chopping of basalt fiber does not diminish or damage the physical properties, merely adjusting fiber/filament length. Data on tow fibers is mirrored by data for chopped fiber. SMC have already proven to be reliable lightweight materials which reduce the use-phase energy consumption of automotive parts compared to traditionally metallic parts. SMC parts also provide reduced tooling costs because of the ability to consolidate parts through a single-step manufacturing process compared to steel or aluminum processes. SMC materials do not interfere with electromagnetic radiation which facilitates the integration of antenna, radars and sensors into SMC parts. Thermoplastic based molding compounds, in combination with basalt fibers offers an interesting recycling option in the near future as sizing development for thermoplastic resins continues to be part of ongoing research at Michelman.

7. COMMERCIALIZATION

Volkswagen Group of America, Inc., was founded in 1955, and is a fully owned subsidiary of

Volkswagen AG, the largest carmaker in Europe and one of the largest automobile manufacturers in the world. Across the 12 brands under the Volkswagen AG umbrella is a broad range of products from motorcycles to commercial vehicle products, and also mobility as a service. Volkswagen Group of America, Inc. opened its Chattanooga assembly plant in 2011 which assembles the Volkswagen Passat, Atlas and Atlas Cross Sport. This Chattanooga assembly plant will soon feature the manufacturing line of Volkswagen's first all-electric SUV built on the MEB platform, the ID.4. The MEB platform (German: **M**odularer **E**-Antriebs-**B**aukasten; English: modular electric-drive toolkit) is a modular car platform for electric cars developed by the Volkswagen Group and its subsidiaries. The MEB platform is applicable for several brands (Audi, Porsche, Seat, Skoda, even truck brands like Man or Scania) in the Volkswagen group. To aid the development efforts of the current and the future line of Volkswagen vehicles, the Knoxville Innovation Hub was established in East Tennessee marking the first North American innovation hub. The partnership between the University of Tennessee Knoxville and Volkswagen has become an innovation hub for vehicle light weighting through composite materials and electrification of vehicles and other automotive platforms.

Glass fiber reinforced SMC materials have already proven feasible as a light weighting method for traditionally steel parts like the VW Atlas lift-gate; this project team is seeking the feasibility of basalt fiber as a drop-in replacement for glass fiber reinforced SMC. Sizing development for basalt fibers has proven that the mechanical properties are better than E-glass and closer to S-glass, which makes it an interesting material for SMC applications. Better mechanical properties translate to less material needed to achieve load case requirement for target applications.

The business case has already been demonstrated for 100,000 parts per year of glass fiber reinforced SMC Atlas lift-gates compared to traditional steel manufacturing processes. Reduced overhead and assembly costs are offset by glass fiber SMC higher cost per kg beyond 100,000 parts per year, which is still a relatively low volume for the automotive industry. For basalt fiber reinforced SMC to become feasible for automotive applications, the price-performance ratio has to be precisely determined. Based on the mechanical performance, it is possible to establish a range of applications and technical solutions in which the potential of basalt SMC can be utilized; while the price of the material can be used to compile the business case for such applications. Based on these business cases and the sustainability indicators, glass fiber reinforcement (or other) materials can be directly substituted.

Volkswagen's commitment to reducing carbon emissions cannot be understated. Basalt fiber shows promise of reducing the carbon footprint in SMC materials, especially if sizing optimizations can be made with thermoplastic based SMC. To fully realize the value of basalt fiber reinforced materials, a lifecycle cost analysis should be performed on basalt's production and recycling, and then compared against E-glass. From this assessment, a true judgement can be made on the commercialization potential of this material.

Basalt fiber has been available on the world market for decades with primary supply streams originating from Russia, Ukraine, Uzbekistan, and China. This supply stream has limited the development of basalt fiber in the USA. Mafic started development of basalt fiber in 2013 based in the Republic of Ireland near Dublin. This start has grown to 3 pilot furnaces with monthly capacity of 50 tons/month. To penetrate strategic markets in the U.S., a regional furnace was

needed for commercial volumes. In 2020, Mafic USA completed a >\$70M investment for the first ever domestic basalt furnace whose capacity at 500 tons/Month makes it the single largest furnace in the world. Based on commercial demand, this first furnace will be followed by an additional 6 furnaces on the same campus in Shelby, North Carolina. The objective of Mafic is to provide a “green”, sustainable, recyclable fiber as a technical solution to composite designs. The goal is to provide 20-25% performance enhancement over E-glass, properties similar to S-glass at a third the price and a low-cost alternative to carbon and aramid.

The original project purpose was to evaluate the performance of Mafic basalt fiber in SMC composites compared against Owens Corning Advantex E-CR glass. The SMC was considered by Volkswagen as a performance enhancement in a lift gate design. Properties of Mafic continuous basalt fiber exhibit a 20-25% increase in tensile strength and modulus compared to standard E-glass fibers. Advantex E-CR glass (no Boron) also has increased tensile strength compared to standard E-glass.

The conclusion of the tow tests showed equal performance comparing the Advantex ME1960 and Mafic basalt fiber. The basalt test fiber was provided from the pilot furnaces in Ireland with standard and experimental sizing systems. Mafic could improve fiber quality from the new large commercial furnace due to increased dwell time of molten materials in the furnace. Considering more consistent flow properties in the large furnace coupled with advanced sizing technologies, Mafic foresees an additional 5-7% increase in performance if the project were executed today. Furthermore, other downstream processes for drying and combining fiber from Direct Roving (DR) to Assembled Roving (AR) were not optimized for the application. Both these processes can influence stiffness, chop ability, strength and wet out characteristics.

To truly exploit the performance capabilities of Mafic basalt fiber, the best mechanism is in a continuous fiber format using composite matrixes via woven or multi-Axial Non-Crimp Fabrics (NCF), Tailored Fiber Placement (TFP) or braided structures. Chopping Mafic basalt fiber diminishes the fiber property and performance. Mafic knows the basalt fiber has parallel properties as S-glass. Thus, it is not surprising that S-glass is largely marketed and consumed as a continuous fiber and not as a chopped fiber.

To address the price of Basalt fiber for feasibility of commercial business practices, we standardize the fiber weight to length ratio (Tex, Denier or Yield) and summarize as:

E-glass	\$ 1X
E-CR glass	\$ 1.2X
Basalt fiber	\$ 2.2X
S-glass	\$ 8X
P-Aramid	\$ 16X
Carbon	\$ 20X

Corporations are moving towards lower-carbon, clean manufacturing and a story line that Eco-consumers demand. We must take time to educate, promote, market, and place a value of the Eco-solutions for new technologies. **The important environmental considerations of Basalt fiber can be highlighted as:**

- Basalt fiber is made by melting Basalt rock wherein no chemical reaction occurs and bubbles trapped within the rock are the only “off gassing”. The melting of rock is merely a change of state to allow fiberization to specified tolerances.
- Melting E-glass, E-CR glass and S-glass constituents in a furnace initiates a chemical reaction liberating gases through Reduction-Oxidation (REDOX). These gases require scrubbing and permits. These gases reduce the “chemical yield” of tons in and tons out. Similarly, production of carbon fiber creates huge loss of yield from the base raw materials. This comparison can be summarized as follows:
 - Basalt 100 Tons raw material = 100 Tons fiber
 - E-glass 100 Tons raw material = 93-94 Tons fiber
 - E-CR glass 100 Tons raw material = 93-94 Tons fiber
 - S-glass 100 Tons raw material = 93-94 Tons fiber
 - Carbon 100 Tons raw material = 50 Tons fiber
- Since the melting of basalt rock is a change of state with 100% yield, nonconforming fiber can be reprocessed (washed and agglomerated) to allow recharging back to the original “virgin” furnace. This is not possible for E-glass, E-CR glass and S-glass which have to be recycled to a secondary dedicated recycle furnace. Carbon fiber cannot be recycled and is usually repurposed in other carbon conversion programs.
- Alternative sizing chemistries focused on thermoplastic composite structures could be examined to accelerate cycle times in automotive production. Thermoplastic techniques would increase composite production, lower costs, and achieve light weight initiatives while utilizing recyclable materials.
- Michelman’s basalt fiber sizings are water-based, environmentally friendly solutions for protecting the fiber during processing and optimizing the interfacial adhesion between the polymers and reinforcing fiber.

As a conclusion, we can state that Mafic basalt fiber is not a direct replacement for E-glass or E-CR glass based on price, but should be considered a technical solution when E-glass does not provide adequate performance in a composite design and S-glass, aramid and carbon fibers are too costly. Mafic Basalt fiber can be placed on the high-performance fiber spectrum next to S-glass for performance but at one third the price. It should be considered for more technically challenging structural designs wherein the performance can demonstrate 20-25% performance enhancement over E-glass to elicit more strength or a weight reduction. As a non-regulated fiber, Mafic basalt products can service S-glass type applications internationally without penalty. Both Michelman and Mafic produce thermoplastic sizings which, in combination with Nylon and polypropylene fibers, can further advance high speed composite implementations while maintaining a cleaner, low emission, Eco-friendly, recyclable manufacturing process.

8. ACCOMPLISHMENTS

Several contributing factors led to fewer accomplishments than originally planned for the project. Among these were a global pandemic, delays in equipment installation, and sourcing of

raw materials. However, the project did achieve the following significant achievements:

- Mafic demonstrated consistent fiber diameter and fiber strength properties of sized basalt fiber.
- Michelman's E-sizing demonstrated promise in increasing the tensile strength and modulus at the tow level for basalt fiber reinforced vinyl-ester composites.
- Demonstrated consistent feeding of basalt fiber into an SMC line and consistent chopping to three different fiber lengths.
- Succeeded in setting the conveyor and chopping speed on SMC line to accurately pay out 20% weight fiber content for sized basalt fiber.

9. CONCLUSIONS

The project purpose was to evaluate the performance of Mafic basalt fiber in SMC composites compared against Owens Corning Advantex E-CR glass. The SMC was considered by Volkswagen as a performance enhancement in a lift gate design. Properties of Mafic continuous basalt fiber exhibit a 20-25% increase in tensile strength and modulus compared to standard E-glass fibers. Advantex E-CR glass (no Boron) also has increased tensile strength compared to standard E-glass.

The conclusion of the testing at the reinforced tow level showed equal performance comparing the OC-Advantex and Mafic basalt fiber. The basalt test fiber was provided from the pilot furnaces in Ireland with standard and experimental sizing systems. Mafic could improve fiber quality from the new large commercial furnace due to increased dwell time of molten materials in the furnace. The process of extruding molten basalt is analogous to E-glass fiberization and formation. Over decades, E-glass producers established trends for fiber optimization wherein formation of small fibers (4.3 micron to 6 micron) are best from small batch furnaces and larger fibers (>13 micron) are better from larger batch furnaces. Due to timing and the delayed startup of the USA operations, the fibers produced in this project were larger 600 Tex, 17-micron but made in Ireland from small "pilot" furnaces. Since the startup of the furnace at Mafic USA in July 2020, there are corollaries to show the importance of larger volume and optimization of heat and mass transfer towards the improvement of physical properties. Considering more consistent flow properties in the large furnace, coupled with advanced sizing technologies, Mafic foresees an additional 5-7% increase in performance if the project were executed today.

The cost-performance valuation of basalt fiber positions it as a lower cost alternative to S-2 glass and other high-performance fibers, and not in the category of E-glass or E-CR glass. The best application areas are most likely where structural, impact durability, or high strain rate performance are required from a continuous fiber. A continuous basalt fiber reinforced thermoplastic composite part would maximize the performance benefits of the fiber and provide a nearly 100% recyclable part.

10. RECOMMENDATIONS

The work in progress at ORNL should be completed to determine if the tow results are repeated at the test panel level. Special attention should also be paid to the failure modes of the specimens to gain a better understanding of the interfacial properties and the effect of the different sizings.

Further work is needed to optimize the basalt fiber sizings for the ideal LOI% for chopping and filamentizing the fiber. This would be beneficial research for SMC or for any spray-up composite processes. It would be interesting to see if a basalt fiber with a higher LOI% would behave better for feeding, chopping, and filamentizing in the SMC line. Existing commercial products have relatively high sizing content for example (1.30% LOI) on the Owens-Corning ME 1960 glass product. The sizing is applied to fiber to assist in downstream process allowing lubrication, cohesion of filaments, reducing filamentation and increasing fiber protection. Mafic typical sizing systems are optimized at a lower %LOI of 0.4 – 0.8 % dependent on the application. For this series of tests, the %LOI sizing content of Mafic samples were 0.82%, 0.51% and 0.69% for the -5X1, E-sizing and HP302, respectively. The OC sizing was significantly higher at 1.30%. It questions what advantages the Mafic fiber would have produced if coated to 1.30 %LOI.

Any further work should also utilize basalt fiber produced from the new furnace in Shelby, North Carolina to realize the optimum fiber properties from a U.S. manufacturing source.

It is recommended that Mafic and Michelman continue to work together to advance the state of art for basalt fiber sizings, and help provide alternative cost competitive solutions that accelerate the utilization of composites for a sustainable and eco-friendly transportation and infrastructure marketplace.

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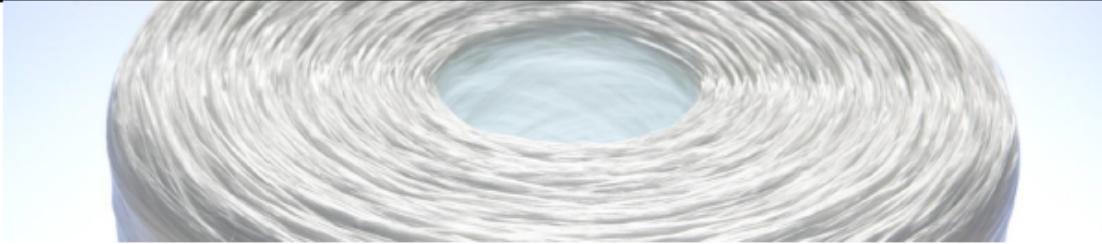
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6. Mafic Safety Data Sheet to ECHA-13-G-11-EN for Continuous Basalt Roving with
Filament Diameter of 9µm to 18µm

12. APPENDICES

Appendix A. Technical Data Sheet for Owens-Corning ME 1960 E-CR Glass Roving (2)

ME 1960 – MULTI-END ROVING FOR SMC



DESCRIPTION

- Produced using Advantex® corrosion resistant glass fibers which combines the electrical and mechanical properties of traditional E-glass with the acid corrosion resistance of E-CR glass
- Manufactured from a collection of continuous glass fibers which are gathered, without mechanical twist, into a single strand or roving
- This Advantex® glass roving has a sizing system with a silane coupling agent. It has been designed to provide excellent process-ability and wetting properties, It is compatible with Polyester, Vinylester unsaturated resins and some Polyurethane resins

BENEFITS

- Excellent process-ability such as easy unwinding and chopping, flat lay-down & uniform dispersion with low fuzz and static.
- Excellent wet-through and impregnation
- White color in finished part
- Good mechanical properties
- Good flow in mold



APPLICATIONS

Designed for the manufacture of Sheet Molding Compound used in general purpose and transportation applications such as sanitary products, heavy truck/bus/train interior and exterior parts

TECHNICAL CHARACTERISTICS (NOMINAL VALUES)

Linear weight of roving (TEX) (g/km)	Loss on Ignition (%)	Moisture (%)
ISO 1889 : 2009	ISO 1887:2014	ISO 3344 : 1997
2400	1.30%	< 0.20%

Appendix B. Technical Data Sheet for IDI's STC 2400 Series SMC (3)

PRODUCT SERIES: STC 2400 SERIES SMC
PRODUCT DESCRIPTION: HIGH STRENGTH VINYL ESTER HYBRID COMPOSITES

Properties Units	STC-2450	
	Imperial	SI
Impact Strength-Izod Notched Test Method: ASTM D-256	38 ft.lb./in.	1,330 J/m
Flexural Strength Test Method: ASTM D-790	48,000 psi	193 MPa
Flexural Modulus Test Method: ASTM D-790	1,800 ksi	14 GPa
Tensile Strength Test Method: ASTM D-638	30,000 psi	138 MPa
Tensile Modulus Test Method: ASTM D-638	2,700 ksi	12 GPa
Water Absorption (24 Hrs @ 23C) Test Method: ASTM D-570	0.1 to 0.3%	0.1 to 0.3%
Specific Gravity (+/- .03) Test Method: ASTM D-792	1.5 to 1.8	1.5 to 1.8
Shrinkage Test Method: ASTM D-955	0.0 to 0.2%	0.0 to 0.2%
Barcol Hardness Test Method: ASTM D-2583	30 to 60	30 to 60

Appendix C: Results of SMC Panel Fabrication and Mechanical Testing. (to be added at undetermined date in future)