Surface Treatment of TuFF Pitch-Based Carbon Fiber for Adhesion Promotion in High Tg Thermoplastic Composites

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ABSTRACT

Commercially available Pitch and PAN based carbon fibers undergo surface treatment to clean the surface of undesirable scale and to provide chemical functionality to promote resin wetting and adhesion in composites. Typically, these surface treatments are carried out in a continuous process that does not lend itself to the treatment of discontinuous fibers, like the mesophase pitch-based fibers being developed for the Tailorable Universal Feedstock for Forming (TuFF) alignment process. To address this challenge, we have developed a multiple-step batch process that oxidizes the fiber surface through ozone treatment at elevated temperature, followed by vapor deposition and/or solution coating to apply a coating package. Using this process, a series of coating formulations have been evaluated for thermal stability, coating uniformity, thickness and chemical composition on mesophase pitch-based carbon fiber. Single Fiber Fragmentation (SFF) testing of model polyether imide (PEI)/carbon fiber composites shows that the interfacial shear strength (IFSS) of as-spun carbon fibers can be increased from ~10 MPa to ~40 MPa with the right choice of coating chemistry. A discussion of the processing steps, and evaluation of the different fiber surface treatments with SFF, Energy Dispersive X-ray spectroscopy (EDAX), and X-ray Photoelectron Spectroscopy (XPS) measurements, as well as the potential for process scale up will be presented.

1. INTRODUCTION

Fiber reinforced plastics have become a popular choice of material for high-strength low-weight applications. In the last few decades, military and civil aviation aggressively adopted carbon fiber reinforced plastics (CFRP) for its low-weight, high-strength properties[1]. Boeing's 787 Dreamliner and Airbus A350 XWB are examples of successful deployment of CFRP in commercial aviation platforms[2]. However, adoption of CFRP outside of aerospace and high-end sport equipment has been slow due to the prohibitively high cost of these materials. Much of the cost is due to the material cost of the carbon fiber. Polyacrylonitrile (PAN)-based carbon fiber is currently the state-of-the-art reinforcement material for demanding aerospace composite applications due to its combination of high specific strength and stiffness. This material is generally used in the form of a woven or uni-directional fabric composed of continuous fiber yarns, or tows. Much of the cost of this material is associated with the production of the PAN precursor fiber. Additionally, the continuous nature of the carbon fiber textiles and prepreg limits the use of high-volume processing techniques like stamping for composite fabrication.

To address this issue, the TuFF alignment process, described in the study by Yarlagadda et al, allows for the use of short fibers to produce composites with continuous fiber properties, while greatly improving formability and processability[3]. The process, as designed, is material agnostic and allows for the use of any type of discontinuous fiber, including those produced from low cost mesophase pitch precursor like those used in the current study. Critical to the success of this process is a high degree of fiber alignment and the development of a strong fiber resin interface that promotes transfer of load between the filaments. The current work looks at the effect of various modifications to fiber surface chemistry have on interface properties between resin and fiber.

For continuous carbon fiber, manufacturers use a proprietary surface treatment process to clean and functionalize the carbon fiber surface. The most commonly reported approach is an electrolytic process using either an acid or base solution, where the continuous fiber acts as an anode. This surface treatment process promotes wetting and creates reactive functional groups that can react with either the resin directly or secondary adhesion promoters. To protect the fiber and further promote the adhesion between the fiber and matrix, sizing packages are used. The sizing packages often consist of a film former, a surfactant, and chemical coupling agent as well as some other components. The state-of-the-art aerospace carbon fiber reinforced thermoplastics use a PAN-based carbon fiber with an electrolytic treatment only since the adhesion promotor must be thermally stable at the high processing temperatures (>300°C). Furthermore, the surface treatment and sizing application traditionally are conducted in continuous bath processes, an approach which does not lend itself to the use of the discontinuous reinforcements used in the TuFF process.

In the current work, a multiple-step batch process to first oxidize the surface of Mesophase pitch-based fibers and coat the fibers with an adhesion promoter was developed and validated. Mesophase pitch-based carbon fibers have undergone surface oxidation and subsequent coating with high temperature polyamic acid (PAA) and silane-based adhesion promoters. This paper will focus on the influence of surface chemistry and coupling agent chemistry on the interfacial shear strength (IFSS) between a low-cost pitch-based carbon fiber, developed for the TuFF process, and polyetherimide (PEI). A discussion of the surface treatment process and analysis of the interface properties as determined by single fiber fragmentation (SFF) test follows.

2. EXPERIMENTATION

2.1 Materials

2.1.1 Carbon Fiber

Mesophase pitch (MP) derived from petroleum has the potential to be a very cheap precursor when produced from innovative production routes[4]. The specific MP used for the current study was also derived from petroleum and was converted into pitch fibers by the use of a high-volume and inexpensive melt-spinning process[5]. The pitch fibers were thermo-oxidatively stabilized in air and then carbonized in an inert gas environment. The carbonization temperature was limited to 2100°C (rather than 3000°C) in an attempt to make the resulting carbon fibers cost-competitive.

Carbon fiber diameters as small as 6-8 μm were successfully produced. These fibers possessed a d_{002} spacing of approximately 0.34 nm as measured by wide-angle x-ray diffraction. The average tensile strength was 2-2.3 GPa; the tensile modulus exceeded 350 GPa, which is almost twice that of PAN-based carbon fibers. The electrical resistivity was measured at about 8 micro-ohm-meters. Izzi-Lavin correlation then indicates a thermal conductivity upwards of 120 W/m·K, which is almost 8 times larger than that of PAN-based carbon fibers.

2.1.2 PEI

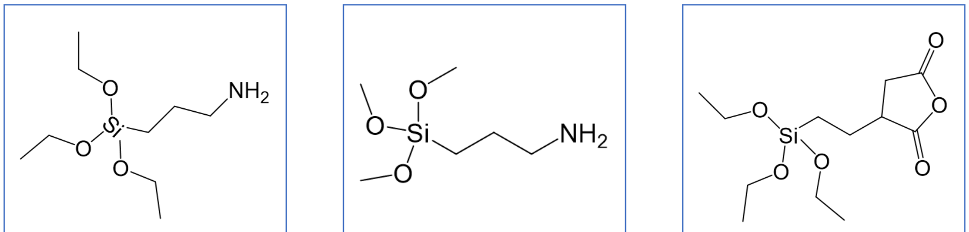
The polymer matrix used for this study was PEI. The PEI used in this study was 0.127 mm thick Ultem 1000 PEI film, purchased from Professional Plastics.

2.1.3 Polyamic Acid Salt

Two types of PAA salts were tested in this study. The first was a commercially available low molecular weight PAA salt system, HP-1632 produced by Michelman Inc. The second experimental system was synthesized and provided for the study by the authors from Virginia Institute of Technology. The procedure for synthesizing a 20,000 g/mole M_n poly(amic acid) is outlined in a publication by Zhang, et al[6], and is described as follows. Ultem dianhydride (11.3804 g, 0.0224 mol) and phthalic anhydride (0.10925 g, 0.0007 mol) were charged to a 250-mL, two-neck, round bottom flask equipped with a magnetic stir bar and a N_2 purge inlet. Anhydrous DMAc (70 mL) was added to the flask. The solution was stirred at room temperature and *m*-AP (2.5 g, 0.0231 mol) was added once a homogeneous solution was obtained. The solution was stirred for at least 12 hours at room temperature. A stoichiometric amount of dimethylethanolamine (5.519 g, 0.0582 mol) was added to the flask and reacted for 1 hour. The solution of the dimethylethanolammonium PAA Salt was stored under N_2 in the refrigerator.

2.1.4 High Temperature Silanes

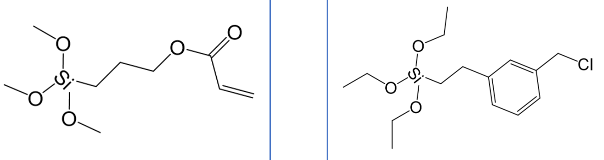
Five commercially available silanes were chosen for this study, 3-aminopropyltriethoxysilane, 3-aminopropyltrimethoxysilane, (3-triethoxysilyl)propylsuccinic anhydride, (3-acryloxypropyl)trimethoxysilane, and ((chloromethyl)phenylethyl)triethoxysilane. All five silanes, purchased from Gelest, were selected for its thermal stability. Figure 1 shows the chemical structure of these five silanes.



3-aminopropyl ethoxysilane (APES)

3-aminopropyl methoxysilane (APMS)

(3-triethoxysilyl) propylsuccinic anhydride



(3-acryloxypropyl) trimethoxysilane

((chloromethyl)phenylethyl) triethoxysilane

Figure 1: Chemistry of silanes used in this study

2.2 Thermally Activated Ozone Oxidation

In this study, the as-received experimental pitch-based carbon fiber, does not have any chemical functional groups on its surface. For this reason, it was necessary to functionalize the carbon fiber surface to promote wetting and adhesion[6,7]. Recent studies have shown success oxidizing carbon fiber using an ozone-based approach[8–12]. Unlike the standard electrolytic approach, the use of a reactive gas does not require the carbon fiber to be continuous. Therefore, an ozone-based thermal oxidation technique was chosen to oxidize the carbon fiber surface. This is a reactive gas-based process which allows for even treatment of chopped or discontinuous fiber. Utilization of ozone as a reactive gas enables oxidation of the carbon fiber surface at very moderate temperatures, result in lower energy expenditure and less damage to the fiber, compared to higher energy corona/plasma treatments which are often used to functionalize particle fillers.

A schematic depicting the laboratory scale reactor set up is shown in Figure 2. Carbon fiber is placed into a one-liter glass reactor residing in a convection oven. Ozone is pumped into the reactor within the oven through ozone resistant silicone tubing. The ozone was produced using the Enaly 5000BF Industrial Ozone Generator, which uses a corona discharge technique to convert the oxygen in the dry air feed stock gas into ozone. The ozone concentration is monitored using the 2B Technology UV-106-MH. The reactor is capable of treating up to 100 grams of chopped carbon fiber per run. Time, temperature, and ozone concentration can be changed in this setup. For the purposes of the current discussion the temperature and ozone concentration are held constant while the process residence time has been varied. The ozone-base thermal oxidation was conducted on the pitch-based carbon fiber using the equipment described in Figure 2. The ozone generator is set to 2 lpm at full power using dry air as the feed stock gas. This allows for a steady state ozone concertation of ~3500 ppm. The ozone generator and the detector were warmed up for 20 minutes on a bypass. For a typical experiment using as-received pitch fiber, approximately 27 g of chopped carbon fiber is placed into the reactor and the oven is set to 70°C. The inlet for the ozone gas is positioned at the bottom of the reactor and the reactive gas is allowed to percolate up through the carbon fiber. The carbon fiber reacted with the ozone for 30, 90, and 180 minutes has been evaluated in order to monitor the effect of oxidation time on contact angle. Times beyond 180 minutes was determined to be impractical for the TuFF project and not considered in this study. Upon completion of the run the oven and the ozone generator are turned off and the dry air is left on to purge the reactor until the ozone detector reads <1ppm.

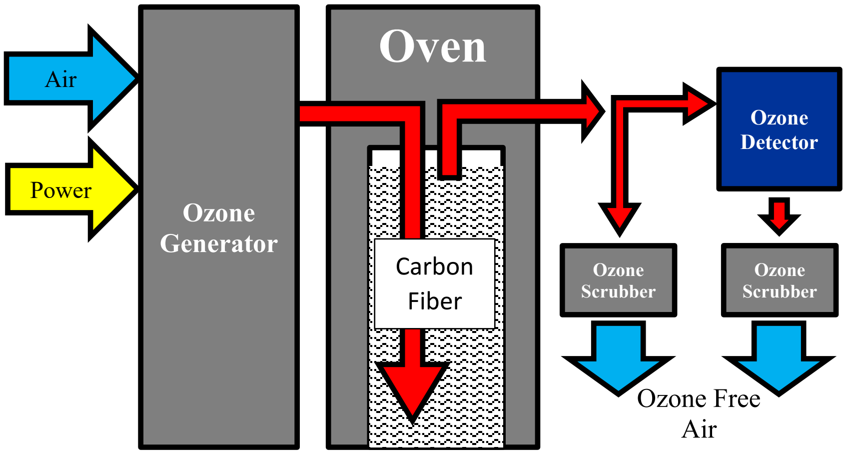


Figure 2: Thermal oxidation reactor setup

2.3 Surface Characterization

To monitor the degree of surface modification, a simple dynamic contact angle measurement was used. Measurements were carried out using the Cahn Dynamic Contact Angle (DCA) Analyzer seen in Figure 3. The contact angle between a single carbon fiber and water was measured. This DCA analyzer measures the load as a function of displacement into the liquid. By adopting a modified Wilhelmy plate technique for a cylindrical shape, the contact angle can be determined[13]. These results are correlated with results X-ray Photoelectron Spectroscopy (XPS) experiments that have been carried out to monitor changes in the ratio of Oxygen to Carbon after Ozone treatment.

Figure 3: Cahn DCA analyzer used to measure contact angle

2.4 Application of Adhesion Promoters

For this study, two classes of coupling agent chemistries were investigated. The first class was the PAA salts. This choice was made with the matrix in mind since PAA has been reported in the literature to improve interface adhesion in PEI and polyetheretherketone (PEEK) thermoplastic composites[14,15]. The second class of coupling agent chosen for this study was the silane-based coupling agent. A third iteration looking at the combination of the two chemistries was also examined.

2.4.1 Polyamic acid Salts

Two types of PAA salt were tested in this study. The first was a commercially available low MW PAA-based sizing system, HP-1632. The other system was a 20,000 g/mole synthesized at Virginia Tech. Both systems were provided in solution form for fiber coating. For each material, a simple dipping method was used to coat the PAA salt onto the thermally oxidized carbon filaments. Each individual fiber was dipped into the PAA solution for 30 seconds, and a pipet was used to ensure the fiber would be thoroughly coated. The coated fibers were hung and allowed to dry overnight.

2.4.2 High Temperature Silanes

The thermal stability of each candidate silane at the process temperature of PEI (~350°C) was confirmed by Thermal Gravimetric Analysis (TGA). Organosilanes were deposited onto the carbon fiber surface using a vapor phase deposition technique, outlined elsewhere[16]. Using this technique, silane molecules are both covalently bonded to the fiber surface via hydroxyl moieties created during oxidation as well as deposited in very small droplets on the fiber surface. After crosslinking the silanes at elevated temperatures, a monolayer of polyorganosilane is present on the fiber surface with highly tuned functional groups as determined by silane chemistry. These groups provide favorable wetting characteristics as well as increased physical attractions with the matrix polymer increasing interfacial shear strength (IFSS).

Figures 4 and 5 show typical results from both EDS and XPS analysis of the fiber surfaces that have undergone treatment with 3-Aminopropyltriethoxy silane (APES) adhesion promoter. Inspection of the EDS scans depicted in Figure 4, shows the uniformity of coverage of the silane over the surface of a typical carbon fiber that has undergone the vapor deposition process with APES silane.

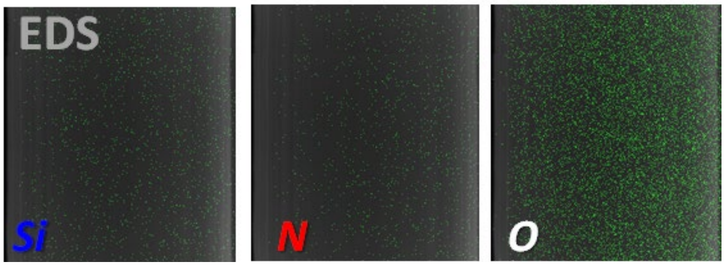


Figure 4:SEM/EDS surface scans of APES coated mesophase pitch-based precursor fibers for silicon, nitrogen and oxygen

Figure 5a shows a comparison of the atomic concentrations, of Si, N, and O as determined by EDS (multiple filaments) and XPS (single filament). The results from both techniques are comparable, indicating the uniformity of coverage is consistent from fiber to fiber. Figure 5b shows plots of that atomic concentrations for Si, N, and O measured (as determined by XPS) for carbon fibers processed in different batches. Little or no variation is seen in the atomic concentration for each element from batch to batch.

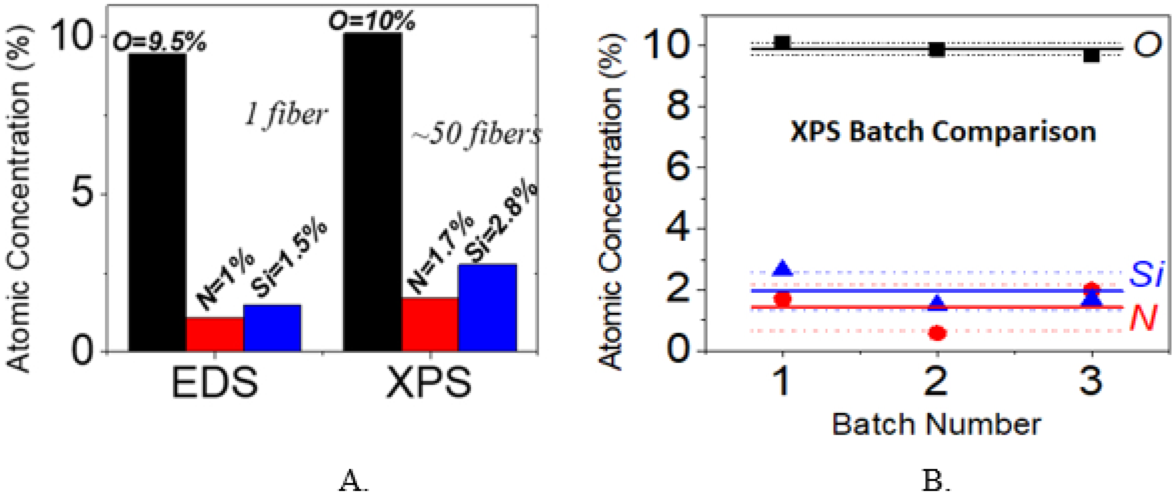


Figure 5: A. Comparison of atomic concentrations of Si, N, and O as determined by EDS and XPS techniques, B. Atomic concentrations of Si, N, and O as determined by XPS measurements for different treatment batches

2.5 Filament Tensile Testing

The aggressive thermal oxidation process could potentially degrade the mechanical properties of the carbon fiber. Since the thermal oxidation is not only cleaning the surface, but also reacting and etching the carbon fiber surface, the mechanical strength of treated filaments was closely monitored to identify any negative effects. The pitch-based carbon fiber was tested using a single fiber tensile testing approach. The equipment used was an automated tensile tester developed by Diastron shown in Figure 6. Using the Diastron proprietary sample holder and cassettes, the tensile samples could be prepared and tested rapidly. The fibers were manually separated and placed into the sample holders with a 12 mm gauge length. Using a UV curable epoxy Loctite AA3201, the fibers were adhered into the sample holder. The epoxy was cured using a spot UV light and finished in a UV flood lamp chamber. The fiber diameter is measured using the laser confocal microscope to determine the cross-sectional area of each individual fiber sample. The samples extension rate was 0.01 mm/sec.

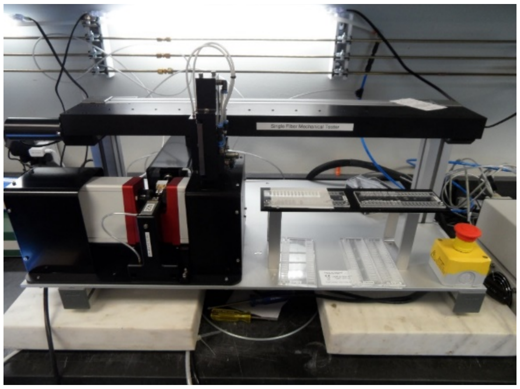


Figure 6: Diastron automated single filament tensile tester

2.6 Single Fiber Fragmentation Testing

To determine the effectiveness of the surface functionalization, it was necessary to quantify the interface properties. IFSS between the carbon fiber to the PEI matrix is critical for the load transfer in the composite. Evaluation of the fiber resin interface properties was carried out via SFF tests. The SFF test is a micro-composite test which can determine the average IFSS by straining a sample with a single fiber imbedded in a matrix[17–21]. Samples were tested in the MTI Instruments SEMTester 1000, which symmetrically moves both crossheads of the tester. The experiment was conducted using a Nikon compound microscope with cross-polarized light. The SEMTester in the microscope can be seen in Figure 7.

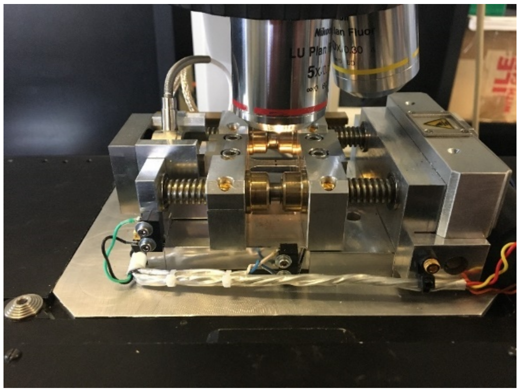


Figure 7: MTI Instruments SEMTest 1000 Tester in the Nikon compound microscope

3. RESULTS

3.1 Thermal-Ozone Oxidation of the Fiber Surface

The effect of thermal-ozone oxidation was monitored by evaluating the wettability of the fiber with water. This technique enables quick monitoring to confirm the changes to the surface wetting behavior as a function of treatment time. Since the thermal oxidation would deposit oxide groups to the surface of the fiber, the contact angle was expected to reduce as a function of processing time. A modified Wilhelmy plate technique was used to determine the contact angle[13]. A sample was made by adhering a short fiber onto a metal rod. This rod was hung on the balance in the Cahn DCA analyzer, and the motor slowly moved the fiber into the liquid. The DCA analyzer recorded the weight change due to the liquid contact. To determine the fiber diameter, the fiber sample was tested first using toluene as the liquid and assuming perfect wetting i.e. θ=0°,

Equation 1: Modified Wilhelmy equation for a rod geometry

$$\theta = cos^{-1}(\frac{m \cdot g}{\pi d_f \gamma})$$

d_f: fiber diameter
m: mass
g: gravitational constant
θ: contact angle
γ: surface tension

and the surface tension of toluene is known, the diameter of the fiber could be determined using the modified Wilhelmy equation shown in Equation 1.

The same sample was tested using water to determine the contact angle between the fiber and water. Using Equation 1 and inserting the fiber diameter determined from the toluene experiment, as well as the surface tension of water, one can calculate the contact angle between the carbon fiber and water. Figure 8 shows the contact angle of the carbon fiber with various degrees of surface modification through thermal oxidation. This data shows a clear decrease in contact angle as a function of thermal oxidation time, suggesting the increase in oxide groups is a function of thermal oxidation time.

Evaluation of the surface chemistry of the carbon fiber were conducted by XPS in order to monitor the change in the O:C ratio, seen in Table 1. The ozone treated fiber shows a clear increase in the oxygen content, which is consistent with the contact angle data.

Table 1: Oxygen to Carbon ratio from XPS measurements

Conditions	*O:C Ratio*
Baseline	0.09
Thermal Oxidized at 70°C for 180 minutes	0.24

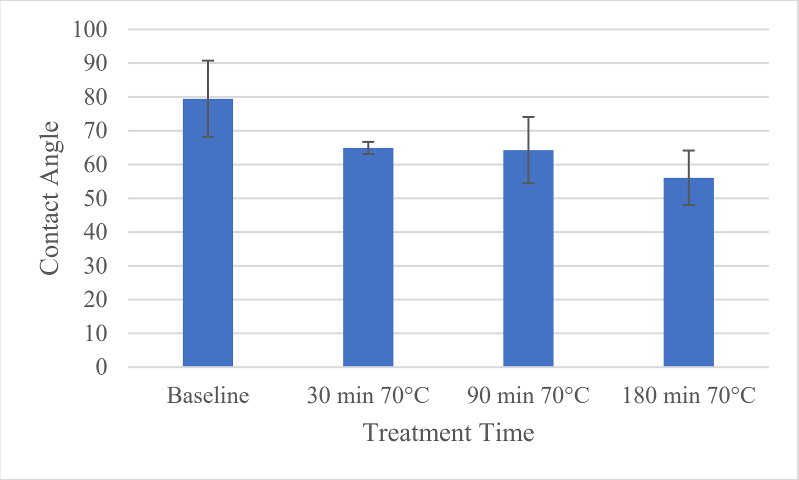


Figure 8: Contact angle measurement for the various thermal oxidation conditions

3.2 Tensile Testing

Tensile tests for as-received fibers and fibers with various degrees of ozone treatment were conducted to determine the effect of the treatment on the fiber performance. As described in the previous experiment section, individual fibers were tested in a single filament tensile test using the Diastron automated single fiber tensile tester. Figure 9 shows the tensile data summary for the strengths of the pitch-based carbon fiber. Each data point represents > 20 replicate tests. Strength data was evaluated using a student T-test, the results of which suggest little significant difference between the baseline and treated specimens. From the minimum contact angle achieved and tensile strength retention, the 180 minutes at 70°C oxidation process was chosen for the interface study to follow.

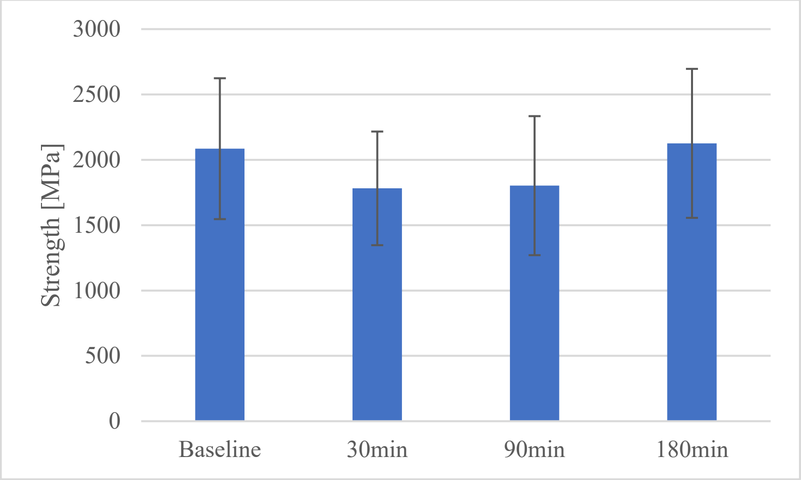


Figure 9: Single Fiber Tensile Test Results for the Various Thermal Oxidation Conditions

3.3 Interface Shear Strength

To make the SFF test samples, two aluminum plates with a high-temperature mold release were used. A mold release treated Kapton film was placed onto the aluminum plate for a smooth surface finish of the sample, which was critical since the sample needed to be free of surface defects to observe the fiber while testing. The one piece of carefully cleaned (with acetone) PEI film was placed onto the Kapton film with great caution being taken to only expose one side to the mold release treated surface. An array of individual fibers, spaced ~20 mm apart were placed across the PEI film with ~25mm of fiber exposed past the Kapton on each side. The fiber ends were taped to the aluminum using a high-temperature aluminum tape. This was to prevent the fibers from moving during the sample processing. A second PEI film was placed onto the fiber, followed by another piece of Kapton film and the aluminum plate. The entirety of this setup was vacuum bagged using Kapton film and high-temperature vacuum sealing tape. This package was then heated to 335°C from room temperature over 45 minutes, then pressed under ~0.7MPa pressure for 45 minutes at temperature. After this step, the press was cooled under pressure to room temperature in about 30-45 minutes. The sample was removed from the plates carefully and cut into 6.6 mm x 40.6 mm (0.26”x1.6”) strips. This was done in such a manner as to keep 1 single filament centered and straight in the test replicate, keeping a 20.3 mm (0.8”) gauge length. These samples were tabbed using card stock and superglue to prevent damage to the replicate from the serrated grips of the tester. During testing, the sample was strained at a constant 1% strain/minute.

During the testing the sample was using imaged using polarized light at 0.25% strain increments for analysis. From the images captured, the cross polarization will highlight the strain fields around the fiber breakage, seen as bright white regions in Figure 10. As the sample was strained, the number of fragments increase. By monitoring the number of fiber fragments, one can identify when the number of fragments saturate, which corresponds to the point where the load from the interface is smaller than the load required to break the fiber. By measuring the number of fragments per unit length after saturation and the average fragment length, the critical fiber length, $\boldsymbol{l_c}$, can be determined. With that information, the Kelly-Tyson equation (Eq. 2), can be used to determine fiber/resin interfacial shear strength.

The effects of thermal ozone treatment and the various silane and PAA salt on the IFSS was evaluated using this SFF testing technique. Using the Kelly-Tyson, equation seen in Equation 2, one can determine the IFSS[21–23].

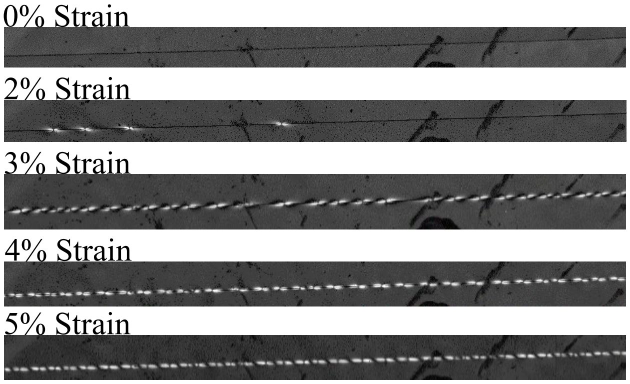


Figure 10: Example of SFF test sample showing the progression of fragment formation

Equation 2: The Kelly-Tyson equation used to calculate the IFSS

$$\tau = \frac{\sigma_f d_f}{2 l_c}$$

$$\tau\text{: } Interfacial\ Shear\ Strength$$

$$\sigma_f\text{: } Fiber\ Strength$$

$$d_f\text{: } Fiber\ Diameter$$

$$l_c\text{: } Critical\ Fragement\ Length$$

$$l_c = \frac{4}{3}\bar{l}$$

$$\bar{l}\text{: } Average\ Fragement\ Length$$

Figure 11 shows a comparison of the measured IFSS for baseline as-received Mesophase Pitch-based carbon fiber to fibers that have undergone both thermally activated ozone oxidation and subsequent dip coating with commercial and high MW PAA salt. Each data point represents an average of 5 measurements. In Figure 11, we see that the baseline IFSS obtained for as-received, untreated mesophase pitch precursor-based carbon fibers and PEI resin is measured to be 10 MPa. This compares to an IFSS value of 40 MPa obtained for commercial unsized IM7

PAN-based carbon fiber and PEI obtained in our lab as part of a separate study, using the same approach to testing outlined above. Ozone oxidation of the Pitch-based carbon fiber does not significantly increase the IFSS, but it does markedly reduce the magnitude of the standard deviation. This is likely due to the removal of larger surface defects during the oxidation process.

Two cases were evaluated for the commercial PAA-salt, where fibers were treated with a high concentration PAA solution (~16% by wt.) and a low concentration solution (~0.16% by wt.). Figure 11 clearly shows that the addition of the PAA salt to the oxidized fiber surface significantly enhances the IFSS reaching a value of 25 MPa for the high loading condition. The highest IFSS value reached with the PAA chemistry was achieved with the experimental 20,000 g/mole MW PAA salt (~0.017% by wt.), which was 42 MPa. Because PAA-salts also provide a potential benefit as a surfactant that can aid in dispersion of discontinuous fibers in the TuFF process, IFSS was measured for fibers that had been first coated with 3-aminopropyltrimethoxysilane (APMS-Silane 2 in Table 2) and then dip coated with the high molecular wt. PAA salt. The result was an IFSS value of 40 MPa, comparable to what was achieved with the High MW PAA alone, but higher than the IFSS of 30 MPa measured for fibers treated with APMS alone. It is thought that the PAA salt forms a complex with the amine terminal-groups of the silane, resulting in a synergistic enhancement of IFSS, which will be the subject of further investigation.

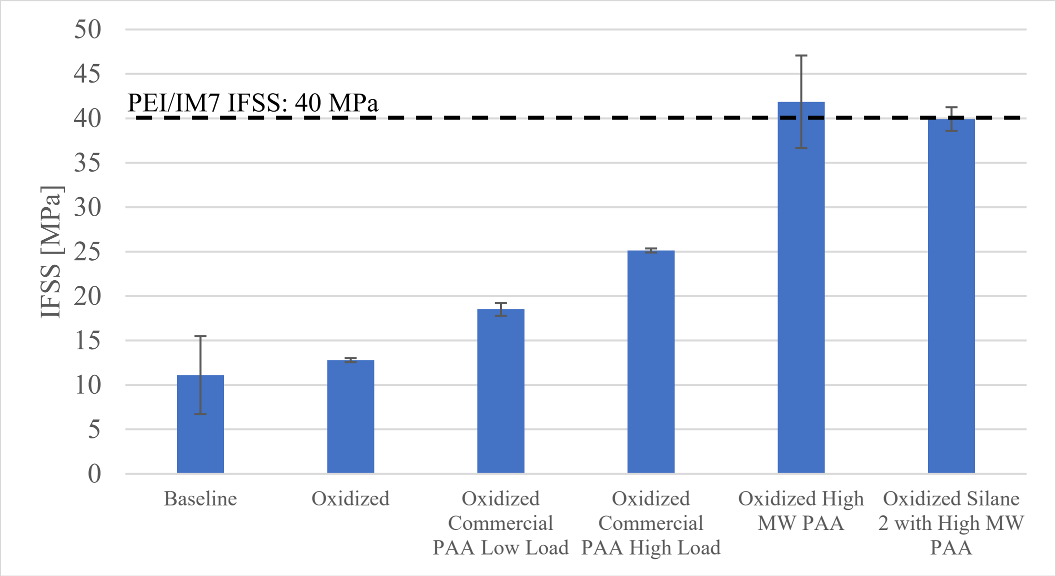


Figure 11: Effect of PAA on the IFSS of carbon fiber with PEI

Figure 12 shows the IFSS for the different silanes chosen for this study. In all cases, the addition of the silane coupling agent improved the IFSS over the baseline by at least a factor of 2. The greatest improvement was achieved by the 3-aminopropyltriethoxysilane (APES), with an IFSS value of 42 MPa, which as discussed earlier compares favorably with what has been measured for model unsized IM7/PEI systems. The APES molecule is an amine terminated silane with three ethoxide branches. It is interesting to note that 3-aminopropyltrimethoxysilane (APMS), which is also an amine terminated silane with three methoxide groups attached to the silicon, has a noticeably lower IFSS of 30 MPa. It is unclear exactly what mechanisms are at work, but it is reasonable to intuit that the longer ethoxy branches of the APES lead to a network with a slightly lower crosslink density, compared to the network formed by the APMS. Understanding how the molecular architecture of the silane influences both the kinetics of network formation and the final network architecture on the fiber surface is the subject of ongoing research.

Table 2: Silane Chemistry Legend

	Silane Chemistry
Silane 1	3-aminopropyltriethoxysilane (APES)
Silane 2	3-aminopropyltrimethoxysilane (APMS)
Silane 3	(3-triethoxysilyl)propylsuccinic anhydride
Silane 4	(3-acryloxypropyl)trimethoxysilane
Silane 5	((Chloromethyl)phenylethyl)triethoxysilane

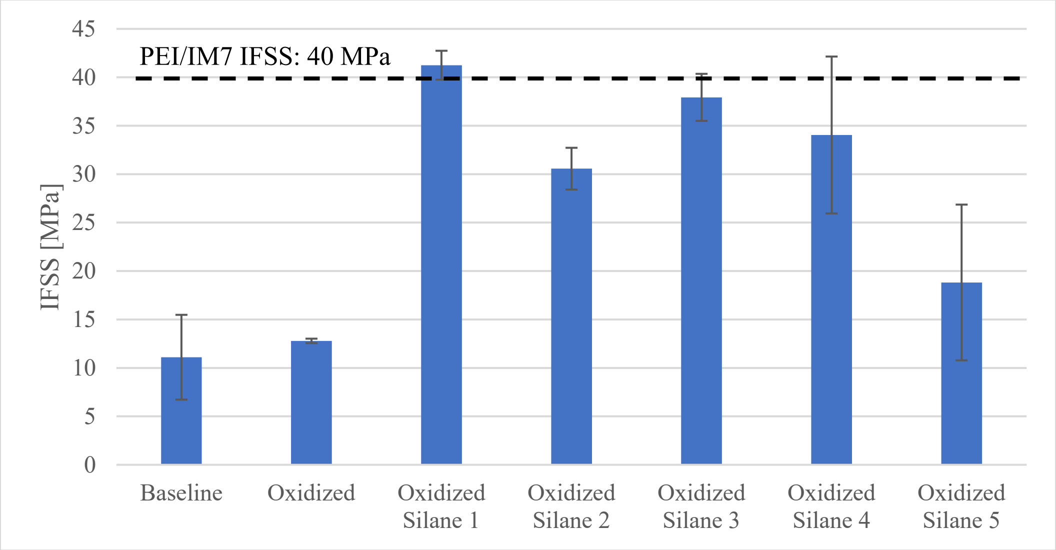


Figure 12: Effect of silane on the IFSS of carbon fiber with PEI

4. CONCLUSIONS

This study has successfully demonstrated a scalable batch process for surface treatment discontinuous pitch-based carbon fiber to tailor the IFSS. It has been demonstrated that discontinuous mesophase pitch-based carbon fibers can be modified through exposure to ozone at elevated temperature. Evaluation of the treated fibers have shown improvement in both the wetting behavior of the fiber and increase the oxygen to carbon ratio by >100 %, with no significant change in tensile properties. PAA and silane-based chemistries were investigated for their potential to promote resin compatibility and adhesion to the oxidized fiber surface in model single filament/PEI resin composites. Fibers coated with these adhesion promoters were evaluated using the SFF test. An increase from an IFSS of 10 MPa for as-received mesophase pitch-based carbon fiber to ~40 MPa IFSS was achieved using both chemistry approaches. This process is easily scalable, material agnostic, and has the potential to be used treat a wide variety of materials, including recycled carbon fibers, for use with the TuFF alignment process developed at the University of Delaware.

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