Mechanical Properties of Buckypaper Laminate Composites and Buckypaper Subjected to Microwave Irradiation

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Abstract

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Hydrogen has proven itself to be of particular interest as a clean renewable energy storage system. However, modern pressure tanks cannot handle the high pressures required to store the large quantities needed. Carbon nanotubes have already been shown as an effective mechanical reinforcement for composite materials. This research furthered this idea and studied if nanotube composites could be used as an outer reinforcement layer on pressure tanks. Laminate buckypaper composites and buckypaper subjected to microwave irradiation were tested. Single-wall carbon nanotubes were formed into buckypaper by vacuum filtration. Strips were cut from this paper and irradiated with microwaves in an effort to weld the nanotubes. Single and four ply laminate composites consisted of non-irradiated buckypaper strips and were layered with one of two epoxies. Tensile testing was conducted on samples press cut with a microtensile die conforming to ASTM D1708 standards. Samples were strained at 0.5 mm per minute until failure while recording the extension applied and resulting force. Laminate samples were also tested using field emission scanning electron microscopy to determine failure type and buckypaper epoxy impregnation. Microwave powers above 130 W were found to cause excessive damage to the buckypaper strips while 120 W formed only minimal damage. Samples microwaved at 120 W gave a 19 percent increase in average modulus of elasticity and a decrease of 31 percent in average ultimate tensile strength (UTS) in comparison to raw buckypaper. Both single and four ply laminates caused a decrease in both average modulus of elasticity and UTS, with the exception of four ply laminates with a 50 percent weight loading of epoxy where the UTS increased. Samples were determined to fail in a brittle manner and epoxy impregnation was found to be very low. With the low resulting average modulus of elasticities and UTSs, these buckypapers and composites were determined to not have the mechanical properties necessary for pressure tank reinforcement. Further research with larger sample sets is needed to determine this conclusively.

Introduction

Renewable energy technologies have increasingly become important as the world's population continues to expand and current energy resources continue to decline. Of these energy technologies, hydrogen has been of particular interest as a replacement for non-renewable energy resources, leading to the idea of the hydrogen economy [1]-[3]. While promising in theory, hydrogen is severely limited in practice by its current storage capabilities [4]. Current storage methods consist of either chemical absorption/adsorption, bonding into a material, as a chemical constituent in a hydride, as a cryogenic liquid, or as a compressed gas [5]. Due to their mechanical simplicity and long history of use, compressed gas tanks are the most mature storage method. However, to store reasonable quantities of hydrogen pressures in excess of 700 bar are required. Pressure tanks have been successfully designed and tested that can safely withstand 700 bar. These tanks are constructed with carbon composites utilizing a high molecular weight polymer liner to prevent gas diffusion [6]. These composites require a large quantity of high-quality carbon fibers and supporting materials. Because of this, their manufacture has a high cost per unit and produces a tank that doesn't meet the Department of Energy (DOE) standards for

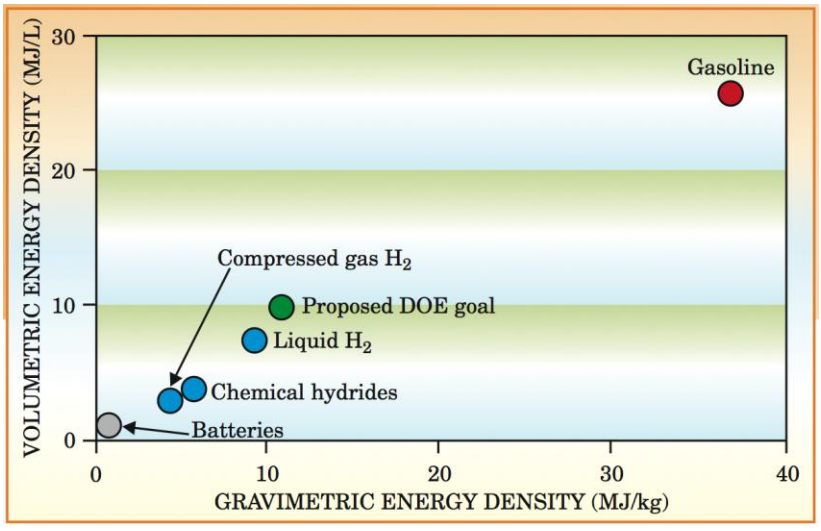

Figure 1: Energy densities of hydrogen fuels stored in various phases and materials [2].

gravimetric energy density of 3 kWh per system kilogram (volumetric energy density 0.09 kg-H_2 per system liter) [7]-[8]. Figure 1 displays volumetric and gravimetric energy densities of recent hydrogen storage maximums and DOE goals [2].

Since the discovery of nanotubes in 1952 [9], extensive research into their properties has been conducted [10]. Many of these studies have focused on the nanotubes as a reinforcement for composite materials [11]-[15]. Regarding hydrogen storage, there has been research into utilizing nanotubes for hydrogen absorption/adsorption as a storage technique [16]-[18]. However, there has been no work on using the mechanical strengthening properties of the carbon nanotubes as a reinforcement device for high-pressure tanks. The objective of this research was to mechanically test and analyze various nanocomposites that could be applied as an over-wrap for these reinforcement purposes.

Single-wall carbon nanotube (SWCNT) soot produced via the laser ablation method [10] was purified and functionalized with carboxyl groups to improve the composite interfacial strength [12],[14]. The purified nanotubes were then formed into buckypaper using a vacuum filtration technique [19]-[20]. The buckypaper was formed into a composite by cutting it into equal sized strips, laminating with an epoxy resin, and impregnating in a vacuum bag with cold pressing until cured. Both single ply and four ply samples were created. Raw buckypaper strips were exposed to microwave irradiation [21] in efforts to weld the nanotubes together [22] and enhance the mechanical properties. Testing was conducted on raw buckypaper, irradiated buckypaper, single and four ply buckypaper composites, and raw epoxy resin samples. All samples were tested in tension to obtain the modulus of elasticity and ultimate tensile strength (UTS).

Laminate samples were also characterized using field emission scanning electron microscopy (FE-SEM) to obtain cross-sectional data including failure mode and epoxy impregnation.

Materials & Methods

SWCNT Purification, Functionalization, and Buckypaper Formation

SWCNT soot was weighed out using an aluminum foil weight boat in a Sartorius 1601 MP8/8-1 scale in quantities between 130 mg and 160 mg. This soot was then transferred into a 500 mL Pyrex® flat-bottomed boiling flask with the aid of an aluminum foil funnel. Into this flask 200 mL of deionized water and 60 mL of concentrated (70%) electronic grade nitric acid were added, making sure to add the nitric acid after the first 100 mL of deionized water. A 1.5 inch Scienceware octagonal magnetic stir bar was also added into the solution. The flask was connected to either a Friedrichs or an Allihn condenser, then refluxed at 240 °C with a stir rate of 400 rpm for 16 hours. Once the solution was allowed to cool to room temperature, the solution was then vacuum filtered using a 90 mm polytetrafluoroethylene (PTFE) Advantec® filter membrane with a 0.5 μm pore size. The vacuum pump utilized was a Thermo Scientific® 420-1901, providing approximately 11 psi of vacuum. After initial filtration, the paper/filter was vacuum rinsed with 30 mL quantities of acetone, deionized water, and 1M sodium hydroxide in the following order; deionized water, acetone, deionized water, acetone, deionized water, acetone, deionized water, 1M sodium hydroxide, deionized water, 1M sodium hydroxide, deionized water, acetone, deionized water, acetone, deionized water, acetone, deionized water, 1M sodium hydroxide, deionized water, acetone, deionized water, acetone. The paper/filter was left to air dry for 15 minutes in the filtration assembly, then removed and sandwiched between a paper towel on the bottom of the filter, and a 0.015 inch thick PTFE film on top. A 3.5 lb flat

plate aluminum weight was placed on top, and the paper/filter was left to dry for four hours. Once air-dried, the paper was carefully removed from the filter and dried further in an oven at 80 °C for 15 minutes.

Single and Four Ply Composite Formation

A fully dried and cooled buckypaper was laid flat on top of a standard piece of 20 lb copy paper. The buckypaper was then cut into 12 mm wide strips using a single edge blade, starting from the middle and working outwards. These strips were then cut to a length of 56 mm. Epoxy was prepared by mixing 5.98 parts of EPON 828 resin with 1 part of EPIKURE 3245 hardener (100 parts by weight resin to 14.3 parts by weight hardener). For some of the composites prepared, a pre-measured Cotronics® EE 4525 epoxy was used instead. The epoxy was applied in 0.1 mL quantities to both sides of one buckypaper strip, and spread evenly using the tip of a tightly wound cotton swab. This strip was immediately pressed when forming a single ply composite, or layered with three more coated buckypaper strips before pressing when forming a four ply composite. Pressing consisted of placing the composite between two 0.015 inch thick PTFE films, then cold pressing in a vacuum bag between two polycarbonate plates in a vice for 18 hours to cure. After curing, the vacuum bag was removed and the PTFE film gently peeled away from the composite. Excess epoxy was trimmed from the composite using scissors.

Buckypaper Microwave Treatment

One buckypaper strip of the same dimensions as used for the laminate composites was inserted into a 10 mL glass vial. The vial was closed with a snap-on plastic open top cap with silicon septa. The vial was then simultaneously evacuated of air and replaced with argon. Microwaving

was conducted using a CEM Discover S-Class microwave system controlled remotely via CEM Synergy™ software. The sample was microwaved using a dynamic method, with the temperature maximum set to the system's maximum of 300 °C and the pressure maximum set to 150 psi. Power and time were varied on sacrificial samples until a suitable set was developed that would not cause damage to the buckypaper. Samples for testing were irradiated at this power and time, with the first two minutes allowing the Synergy™ software to vary the power to ensure the sample would not overheat during initial exposure. Irradiated samples were air cooled to 60 °C in the CEM device, then removed and allowed to cool to room temperature.

Testing: Tensile (Mechanical Properties)

Each sample to be tested was first press cut using a microtensile die conforming to ASTM D1708 standards. All samples were press cut to one metric ton, with the exception of the raw epoxy samples, which were pressed to half of a metric ton to prevent cracking. This yielded consistently sized samples with overall dimensions of 38 mm by 12 mm, and a gauge dimension of 22 mm by 5 mm. Samples were then loaded into an Instron 5500R tensile testing machine. The Instron was equipped with a 50 kg load cell and Instron 2712-018 pneumatic side action grips supplied with 50 psi of compressed air controlled by a universal pneumatic footswitch. Each sample was strained at a rate of 0.5 mm per minute until failure while recording the force and crosshead extension data.

Testing: Field Emission Scanning Electron Microscopy (Failure Type and Impregnation)

Representative samples broken during tensile testing were analyzed along the fracture cross-section. Samples were exclusively from the single or four ply laminate sample sets. The

microscope used was a JEOL JSM-7000F field emission scanning electron microscope (FE-SEM) with an accelerating voltage of 2 kV. Magnifications used ranged from 100x to 15000x depending on the sample and area of focus.

Results

Buckypaper Microwave Treatment

The microwave power used on the sacrificial samples was started at 40 W and incrementally worked up to 300 W. It was found that as the power increased, so did the damage to the buckypaper. Significant paper damage was noticed after 130 W of power applied for any amount of time. Photographs of papers damaged by excess power application are presented in Figure 2.

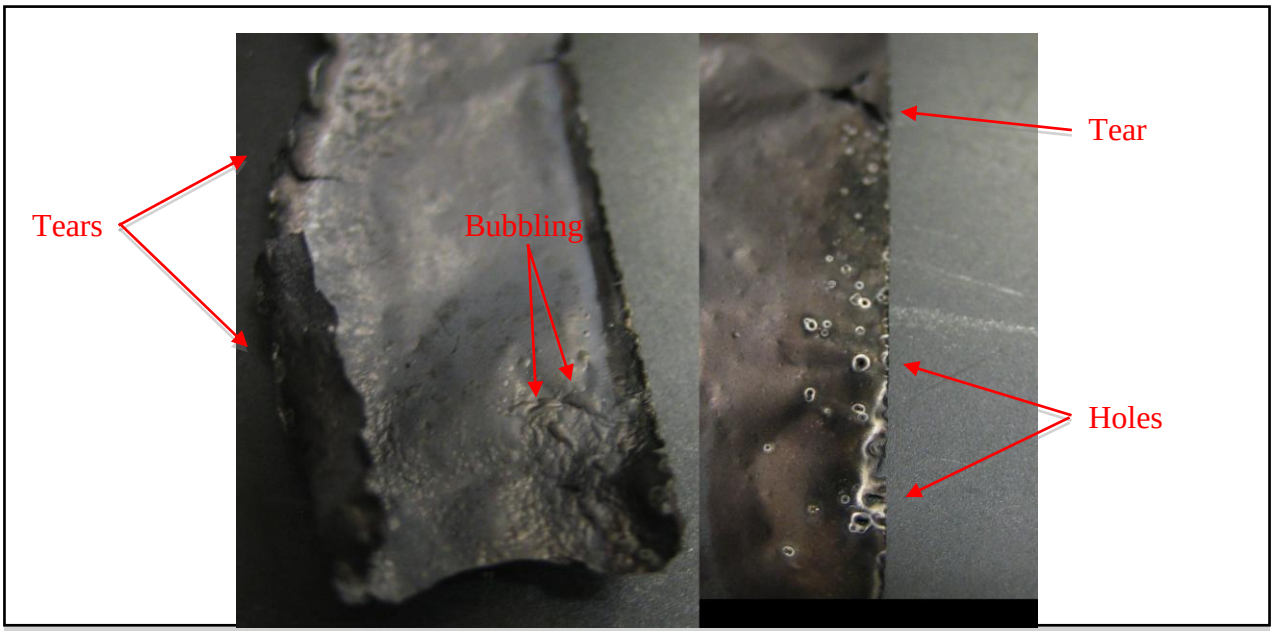

Figure 2: Buckypaper damage from microwave irradiation at higher power (150 W).

Rapid temperature and pressure increases within the first few minutes were also noticed with powers higher than 130 W, often surpassing the CEM device's 300 °C temperature maximum within one minute of irradiation. Papers microwaved at 120 W showed only minimal damage on the edges at any time tested up to 30 minutes, with enough of the paper remaining undamaged

that a tensile sample could be successfully created. Samples for tensile testing were thus irradiated at 120 W for 30 minutes.

Testing: Tensile (Mechanical Properties)

A total of 43 samples were tested until failure. This included 10 samples of each raw epoxy (20 in total), five samples of raw buckypaper, five samples of microwaved buckypaper, five single ply composites with the EPON 828 and Epikure 3245 epoxy, five four ply composites with the EPON 828 and Epikure 3245 epoxy, and three four ply composites with the Cotronics® EE 4525 epoxy. The primary output of the Merlin software was the maximum load and maximum extension, while the full load and extension data was recorded for analysis. A representative sample of the load and extension output data is shown in Figure 3. Sample mass, thickness, and

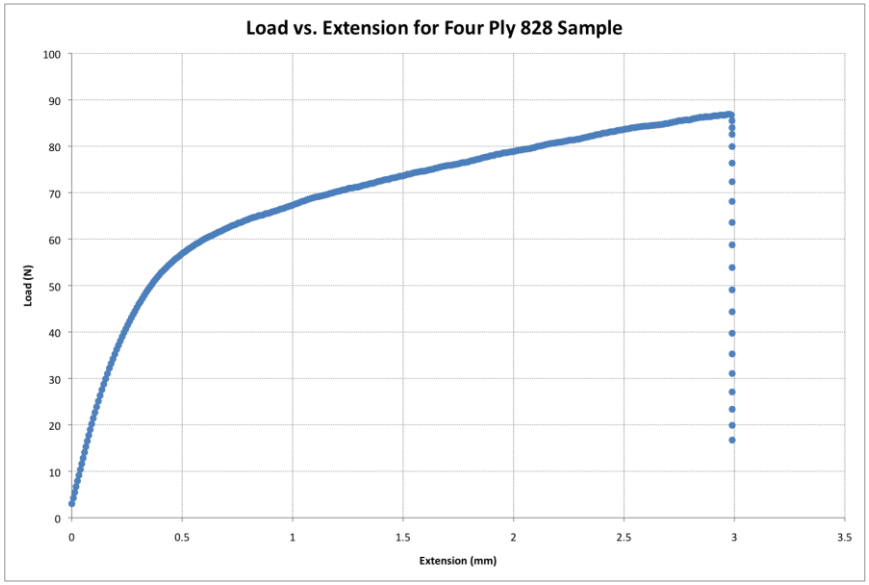

Figure 3: Representative graph of load vs. extension data output by Instron 5500R (Sample 38).

percent by weight of epoxy were also measured prior to testing. The elastic modulus and UTS of each sample was determined by applying the simple engineering stress and strain equations to the load and extension data. The results when applied to the data in Figure 3 are presented in

Figure 4. Where the thickness of a sample was not accurately known, a thickness to mass ratio was used and assumed to be equal between samples of the same composition. In this manner, a

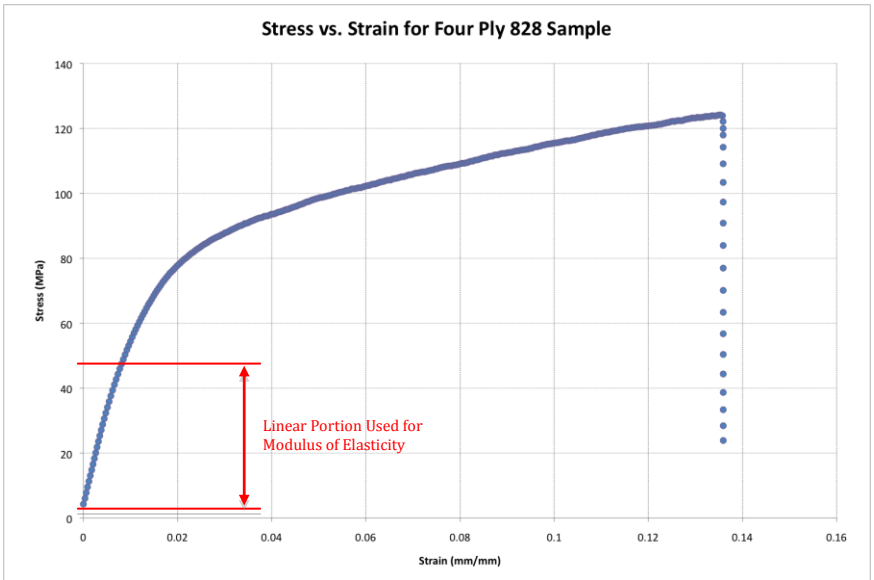

Figure 4: Representative graph of stress vs. strain data (Sample 38).

sample with known thickness and mass could provide a reasonable estimate of the thickness of another sample with smaller known mass and unknown thickness. The relevant data for each sample is presented in Table 1, and a comparison of sample UTS against modulus of elasticity is graphed in Figure 5.

Table 1. Data and results for all samples tensile tested on the Instron 5500R.

Sample Type	Sample Number	Maximum Load (N)	Maximum Extension (mm)	Mass (mg)	Thickness (mm)	Percent by Weight Epoxy	Modulus of Elasticity (GPa)	Ultimate Tensile Strength (MPa)
EPON 828 and Epikure 3245 Raw Epoxy	1	37.43	0.63	42.9	0.11	100.0	2.65	68.1
	2	48.58	0.60	55.4	0.14	100.0	2.75	69.4
	3	37.03	0.51	55.2	0.14	100.0	2.33	52.9
	4	40.74	0.63	47.3	0.12	100.0	2.62	67.9
	5	43.86	0.68	46.8	0.12	100.0	2.78	73.1
	6	32.91	0.61	38.6	0.10	100.0	2.61	65.8
	7	33.89	0.53	43.4	0.11	100.0	2.69	61.6
	8	42.90	0.65	47.2	0.12	100.0	2.71	71.5
	9	37.94	0.57	41.7	0.11	100.0	2.86	69.0
	10	35.65	0.62	43.6	0.11	100.0	2.47	64.8
Average		39.1	0.60	46.2	0.12	100.0	2.65	66.4
Standard Deviation		4.89	0.05	5.49	0.01	0.00	0.15	5.5
Cotronics® EE 4525 Raw Epoxy	11	22.09	0.18	115.0	0.20	100.0	3.37	22.1
	12	18.12	0.18	89.1	0.15	100.0	3.45	24.2
	13	21.79	0.27	85.3	0.15	100.0	3.93	29.1
	14	18.48	0.17	101.0	0.18	100.0	3.24	20.5
	15	28.52	0.24	124.0	0.22	100.0	3.28	25.9
	16	14.22	0.18	80.2	0.14	100.0	3.48	20.3
	17	18.26	0.15	99.3	0.17	100.0	3.71	21.5
	18	24.52	0.17	132.0	0.23	100.0	3.48	21.3
	19	14.71	0.12	86.0	0.15	100.0	3.71	19.6
	20	18.96	0.18	84.7	0.15	100.0	3.99	25.3
Average		20.0	0.18	99.7	0.17	100.0	3.56	23.0
Standard Deviation		4.4	0.04	18.2	0.03	0.0	0.26	3.0
Raw Buckypaper	21	4.11	0.94	4.2	0.01	0.0	5.23	82.2
	22	2.25	0.46	3.3	< 0.01	0.0	5.14	57.6
	23	4.57	1.23	4.4	0.01	0.0	5.24	91.4
	24	5.30	1.72	4.2	0.01	0.0	5.70	105.9
	25	4.97	1.65	4.1	0.01	0.0	5.47	99.4
Average		4.2	1.20	4.0	0.01	0.0	5.36	87.3
Standard Deviation		1.2	0.52	0.4	0.00	0.0	0.23	18.8
Microwaved Buckypaper	26	1.27	0.23	2.9	< 0.01	0.0	5.72	37.1
	27	1.34	0.27	2.2	< 0.01	0.0	6.09	51.5
	28	1.95	0.35	2.8	< 0.01	0.0	6.05	58.9
	29	2.38	0.38	2.9	< 0.01	0.0	6.27	69.4
	30	2.19	0.34	2.3	< 0.01	0.0	7.87	80.5
Average		1.8	0.31	2.6	< 0.01	0.0	6.40	59.5
Standard Deviation		0.5	0.06	0.3	0.00	0.0	0.85	16.6
Single Ply Composite (828 & 3245)	31	9.81	0.47	11.4	0.04	74.2	4.10	49.1
	32	8.58	0.39	11.2	0.04	73.5	3.82	42.9
	33	9.69	0.47	12.4	0.04	75.6	4.15	48.4
	34	7.37	0.31	12.4	0.04	74.2	3.97	36.9
	35	9.88	0.43	14.1	0.04	74.6	4.14	49.4
Average		9.1	0.41	12.3	0.04	74.4	4.03	45.3
Standard Deviation		1.1	0.07	1.1	0.00	0.8	0.14	5.4
Four Ply Composite (828 & 3245)	36	20.24	1.12	24.7	0.08	34.0	3.29	50.6
	37	13.72	0.52	30.3	0.09	31.1	2.71	30.5
	38	86.89	2.98	47.4	0.14	50.8	5.10	118.7
	39	73.39	2.33	42.5	0.13	50.4	4.98	112.9
	40	62.73	2.01	49.7	0.14	59.8	4.92	89.6
Average		51.4	1.79	38.9	0.12	45.2	4.20	80.5
Standard Deviation		32.6	0.98	10.9	0.03	12.2	1.12	38.7
Four Ply Composite (Cotronics® EE 4525)	41	39.97	0.44	88.3	0.22	75.2	3.71	36.3
	42	39.16	0.46	64.4	0.12	72.5	5.74	65.3
	43	19.50	0.17	45.6	0.12	83.8	4.98	32.5
Average		32.9	0.36	66.1	0.15	77.2	4.81	44.7
Standard Deviation		11.6	0.16	21.4	0.06	5.9	1.02	17.9

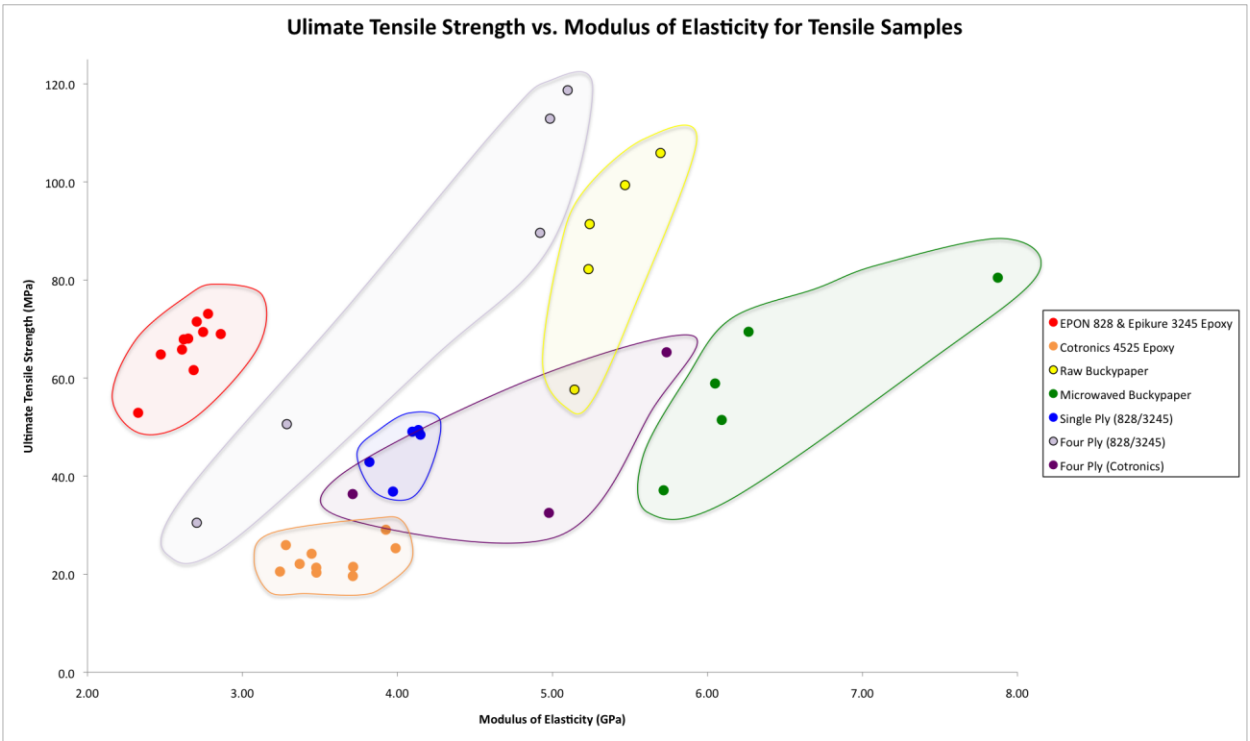

Figure 5: Plot of sample ultimate tensile strength against modulus of elasticity with sample envelopes.

Testing: Field Emission Scanning Electron Microscopy (Failure Type and Impregnation)

Four of the laminate composites were examined under FE-SEM. The first sample examined was a four ply laminate with the Cotronics® EE 4525 epoxy (Sample 41). The cross-section is presented in Figure 6 and in Figure 7. Both the second and third samples examined were four ply laminate with the EPON 828 and Epikure 3245 combination epoxy. The second sample had a lower percentage by weight of epoxy (Sample 37), while the third had a higher percentage (Sample 38). These cross-sections are presented in Figures 8-11. The last sample examined was a single ply laminate (Sample 31). This cross-section is presented in Figure 12.

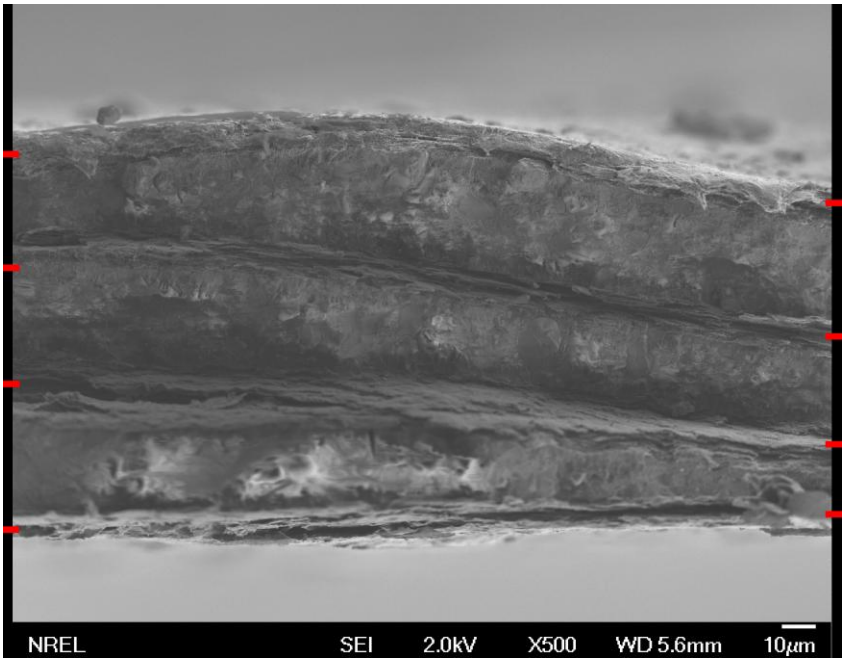

Figure 6: FE-SEM of four ply Cotronics® 4525 sample 41 at 500x. The red dashes indicate the start and end points of the buckypaper layers.

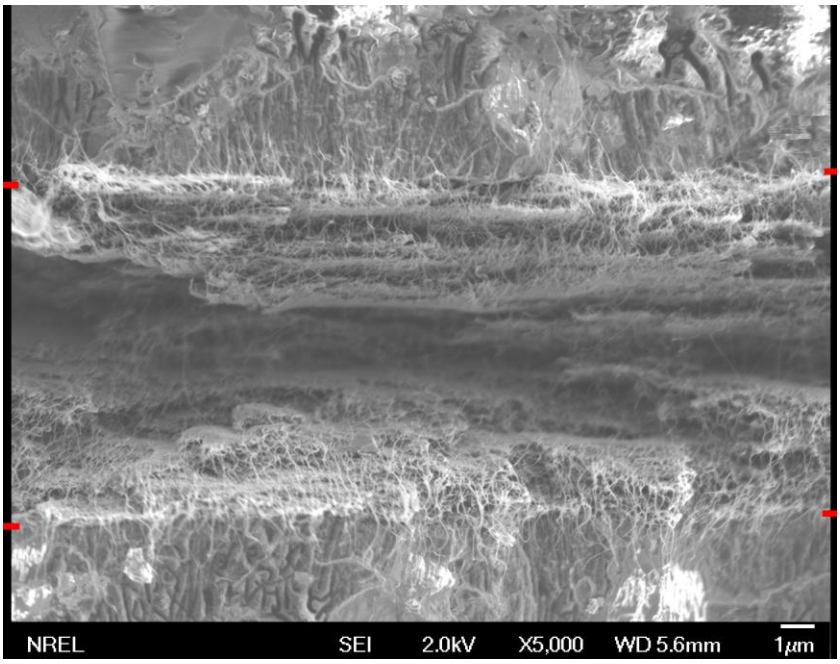

Figure 7: FE-SEM of four ply Cotronics® 4525 sample 41 at 5000x. The red dashes indicate the boundary layers between buckypaper layer in the middle and the epoxy layer above and below. Note the layered structure of the buckypaper created during filtration.

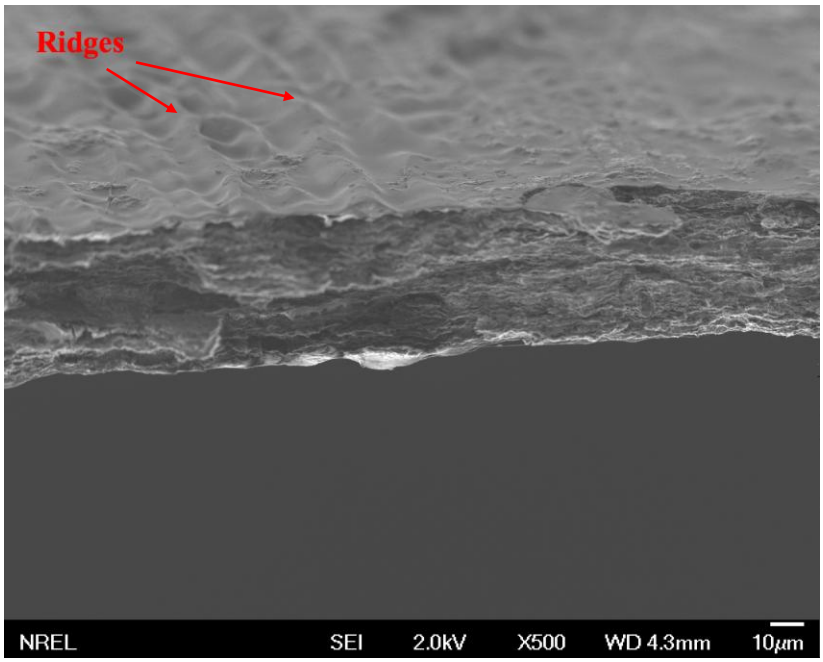

Figure 8: FE-SEM of four ply EPON 828 & Epikure 3245 sample 37 at 500x. Ridges caused by grooves in the filter membrane are clearly visible along the surface of the paper.

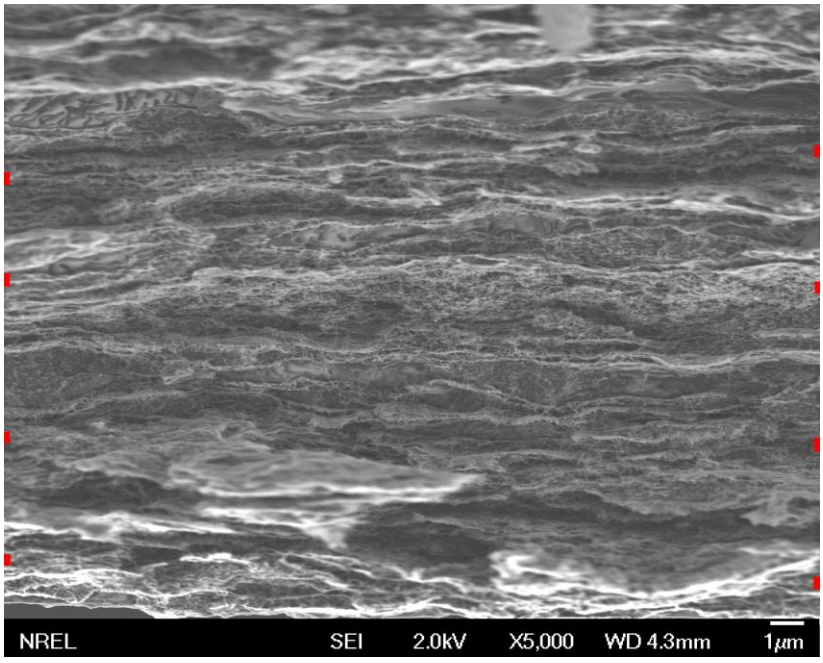

Figure 9: FE-SEM of four ply EPON 828 & Epikure 3245 sample 37 at 5000x. The red dashes indicate start and end of buckypaper layers. Note the drastically decreased epoxy layers due to slightly increased pressing force.

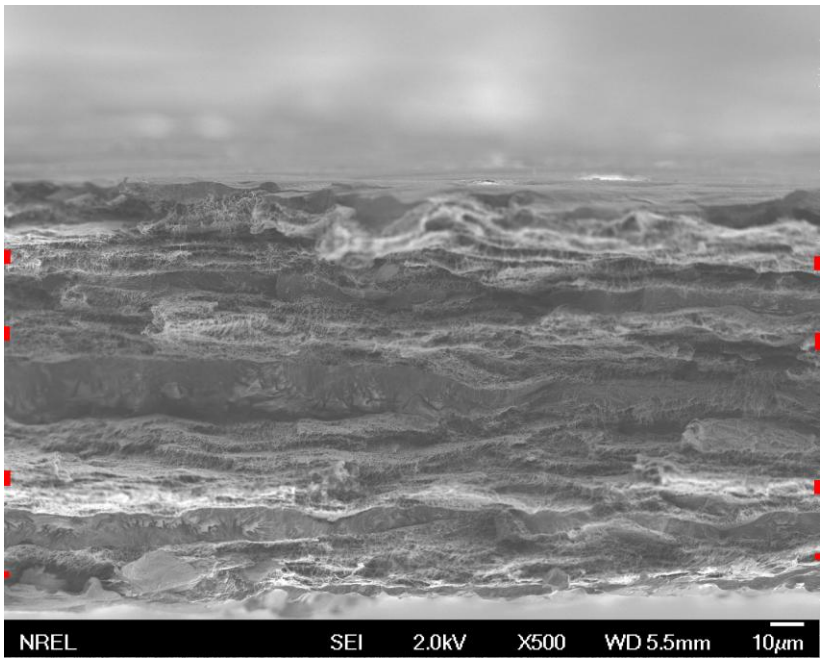

Figure 10: FE-SEM of four ply EPON 828 & Epikure 3245 sample 38 at 500x. The red dashes indicate start and end of buckypaper layers. Note the epoxy layer thickness in comparison to Figure 9.

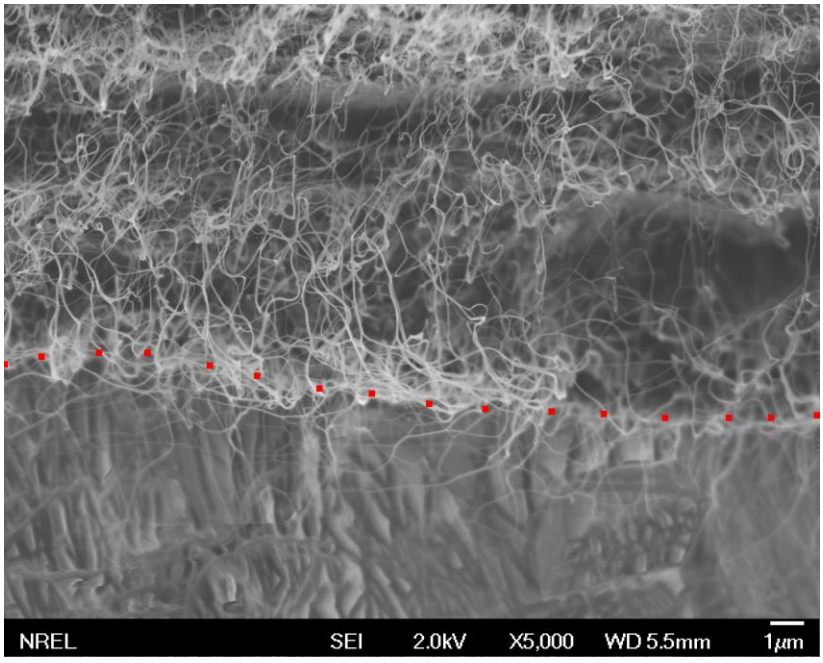

Figure 11: FE-SEM of four ply EPON 828 & Epikure 3245 sample 38 at 5000x. The red line highlights the distinct boundary between the upper buckypaper and lower epoxy layers.

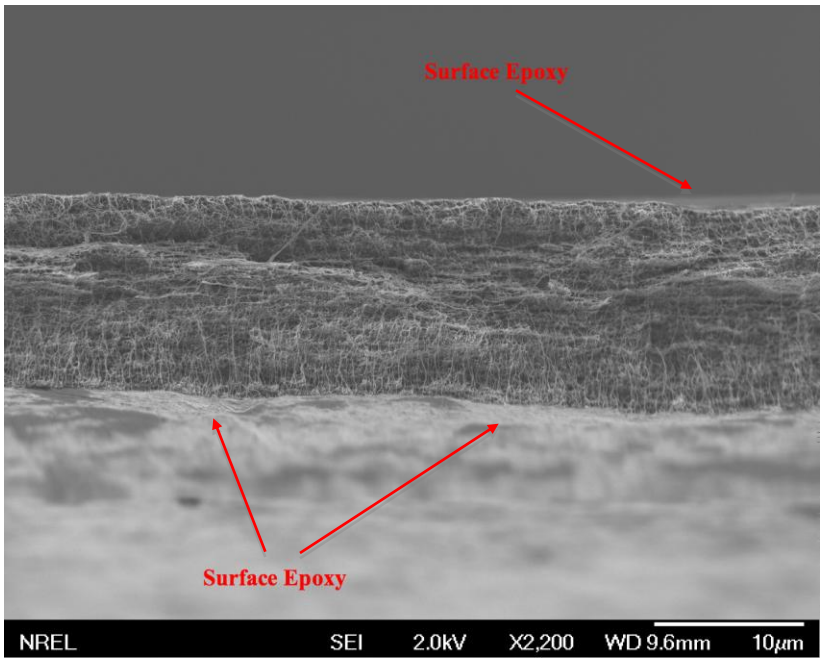

Figure 12: FE-SEM of single ply EPON 828 & Epikure 3245 sample 31 at 2200x. Note the even fracture surface and lack of nanotube wetting and infiltration by the epoxy.

Discussion & Conclusions

Damage caused to the buckypaper during microwave irradiation was a result of arcing on the paper at higher power levels. Larger arcs would cause tears and holes in the paper while smaller arcs rendered areas extremely brittle and caused bubbling, both which can be seen in Figure 2. This proved a problem in the effort to weld the nanotubes since higher powers are desired so that the nanotubes are provided sufficient energy to fuse with each other. The 120 W power used for the samples was chosen to minimize paper damage, not as an ideal power for welding nanotubes. While not best suited to welding, the microwave irradiation did cause a change in the tensile data. The average modulus of elasticity increased by 19 percent in comparison to the raw buckypaper while decreasing the average UTS by 31 percent. Due to the macroscopic study of the irradiation effects, it is not known if the tensile data changed due to nanotube welding or

another chemical reaction occurring in the paper. The change was also not significant or definitive due to the small sample size and fairly large range of values.

The data also shows that the lamination of the buckypaper decreased both the average modulus of elasticity and the average UTS in comparison with the raw buckypaper. Some of the specific samples displayed UTS improvement over the raw buckypaper, as seen in Samples 38 and 39. These samples show that a 50/50 combination by weight of the EPON 828 and Epikure 3245 epoxy and buckypaper may provide a stronger and more elastic material than raw buckypaper. However, Samples 36, 37, and 40 show that a weight balance deviating from 50/50 creates a more flexible material, but at the cost of a relatively unchanged or reduced UTS.

The FE-SEM images provide a microscopic insight into testing that was otherwise macroscopic. Figures 6-12 all show that the samples failed in a brittle manner. This is seen by the very even fracture planes, rather than a ductile failure that would have shown an uneven and ragged fracture surface. The single ply image, Figure 12, makes this particularly easy to see. Figures 7, 11, and 12 also show that the epoxy had a very limited impregnation. The boundary layer between the epoxy and the buckypaper is clearly visible, showing very little wetting and infiltration of the SWCNTs by the epoxy. This is not a surprising result since while highly porous, the buckypaper is also very tightly woven. Variations in epoxy viscosity and pressing force are visible between Figures 6, 9, and 10. The Cotronics® EE 4525 epoxy was considerably more viscous when applied and thus more difficult to press out, as visible by the thick epoxy layers in Figure 6. This is also seen to a lesser extent with the lower viscosity EPON 828 and Epikure 3245 epoxy in Figure 10. Because of the lower viscosity, a similar pressing force was

able to displace more of the epoxy from in between the layers. Figure 9 displays how a slightly increased pressing force can significantly reduce this epoxy layer, as could reduction of the applied epoxy. Figure 8 displays an unexpected consequence of the particular filter membrane used in the vacuum filtration process. The ridges located along the surface of the buckypaper are due to the grooves in the filter membrane. While not likely a major contributing factor in tensile studies, this adds a highly uncontrolled aspect to the uniformity of the papers, and suggests that a new filter membrane be used to decrease the paper thickness variations.

While specific buckypaper/epoxy composites show improvement in flexibility and UTS over raw buckypaper, none of the samples displayed mechanical properties that can compare to current technologies used in pressurized hydrogen storage. Carbon fiber composites often have a UTS 10 times greater than the best produced in any of these samples [23], and are currently much easier to manufacture. More importantly, the potential strength of the nanotubes is not being utilized in any of the samples. Research has indicated that individual nanotubes have a modulus of elasticity above 1 TPa and a UTS up to 200 MPa [12], both magnitudes larger than the properties of these samples. Microwave irradiation may provide a means for increasing the modulus of elasticity of buckypaper, but the powers required to do so may destroy the paper before any welding can occur. With the low resulting average modulus of elasticities and ultimate tensile strengths, these buckypapers and composites were determined to not have the mechanical properties necessary for pressure tank reinforcement. However, further research is needed to conclusively determine the mechanical properties. This research needs to include much larger sample sets than those used in this study, and should explore higher purity buckypapers and alternative techniques of epoxy impregnation such as vacuum pass-through infiltration. Alignment techniques for the

nanotubes in the buckypaper should also be developed and tested so that the properties of the individual nanotubes may be more closely harnessed.

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