Effect of Gas Plasma Surface Treatment on Spectra 900 and Spectra 1000 Fabric Laminate Composites

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Summary

Impact and compression after impact properties of Spectra® 900 and Spectra® 1000 laminate composites were investigated in this study. The focus of this research was to determine if any improvement in impact properties existed as a result of gas plasma treating the surface of the fabric. Tests were conducted on different samples to obtain information about absorbed energy and maximum impact force at impact energies between 10J and 25J. Compression after impact tests were also performed to determine the reduction in compressive strength from impacted to non-impacted samples. The data collected helped to determine the advantages and disadvantages of surface treating Spectra fabric.

1. Introduction

Spectra fiber is one of the world's strongest and lightest fibers. It has a strength-to-weight ratio ten times higher than steel, and has a specific strength that is 40 percent greater than aramid fibers. It is an ultra lightweight, high-strength polyethylene fiber developed by Honeywell International Corporation. Some benefits of Spectra include high damage tolerance, non-conductivity and flexibility, high specific modulus and high energy-to-break, low moisture sensitivity, and good UV resistance [[1]].

Spectra fiber is made from ultra-high molecular weight polyethylene that is used in a patented gel-spinning process. The gel-spinning process and subsequent drawing steps allow Spectra® fiber to have a much higher melting temperature (150°C or 300°F) than standard polyethylene [[2]]. With outstanding toughness and extraordinary visco-elastic properties, Spectra fiber can withstand high-load strain-rate velocities.

Spectra fiber is used in numerous high-performance applications, including police and military ballistic-resistant vests, helmets and armored vehicles, as well as sailcloth, fishing lines, marine cordage, lifting slings, and cut-resistant gloves and apparel [[2]]. As can be seen, most applications are for Spectra fabrics and not for composite Spectra laminates. One reason for this is that sheets of Spectra exhibit poor wetting and a lack of chemical bonding with the resin and therefore do not adequately adhere. According to Kolluri et al [[3]], in order to improve the stress transfer at the interface and efficiently utilize the properties of the fiber, a strong interaction between the resin matrix and the fiber is crucial.

One of the best methods to achieve this is through gas plasma surface treatment. Plasma surface treatment minimizes or eliminates these problems by removing surface contaminants and weakly bound polymer layers, enhancing wettability by incorporating polar groups on the surface, and by forming functional groups on the surface, permitting covalent bonding between the fiber and the resin matrix [[3]]. Since plasma treatment is a surface modification process, the bulk properties of the fiber are maintained. This process by nature can be precisely controlled. By judiciously selecting the process gases and process parameters, the surface can be reengineered to fit specific needs, chemically or functionally [[4]]. Our fabric was plasma treated by 4th State, Incorporated in Belmont, California. They were able to adjust their process and alter the surface of our fabric to match our needs.

2. Sample Construction

A hand lay-up method shown in Figure 1 was used to construct the samples. The major components required for this method are a vacuum pump,

vacuum bagging, spiral tubing and sealant tape. The spiral tubing ensured a uniform vacuum across the sample and prevented epoxy from pooling on the side of the sample opposite the vacuum tubing, resulting in non-uniform facesheet thickness. The facesheet would be the thickest on the side away from the vacuum tubing. The carbon fiber and Spectra fabric properties are listed in Table 1. The epoxy consists of F-82 resin and TP-41 hardener, which was allowed to cure under a 600mm Hg vacuum for a minimum of 9 hours. The cured properties of the epoxy, purchased from Eastpointe Fiberglass, are listed in Table 2.

The hand lay-up method provided high quality samples with minimal defects. Special care was taken to insure the correct amount of epoxy was used in addition to being evenly spread out. After eight layers of epoxy soaked fabric were placed, the vacuum bagging was carefully spread over the sample insuring no wrinkles would form when the vacuum was applied. Any wrinkles on the vacuum bagging will affect the surface finish of the sample. A rubber squeegee was used to remove the extra epoxy and trapped air. Table 3 shows the various laminate configurations.

Table 1: Fabric Properties

	Spectra 900	**Spectra 1000**
Yarn Type	Spectra 900	Spectra 1000 375 denier
Weave Style	Plain	Plain
Area Density	203 g/m^2	112 g/m^2
Thickness	0.43 mm	0.18 mm
Count (Rows per Inch)	34 x 34	32 x 32

Table 2: Properties of Eastpointe Fiberglass Epoxy

Density	1095 kg/m^3
Compressive Strength	131 MPa
Tensile Strength	63.6 MPa
Cure Time	9-12 Hours
Cure Temperature	23.9°C (75°F)

Table 3: Laminate construction configuration

Sample Configuration	**Sample Abbreviation**	**Layers**
Untreated Spectra 900	US900	8 layers untreated Spectra 900
Treated Spectra 900	TS900	8 layers treated Spectra 900
Untreated Spectra 1000	US1000	8 layers untreated Spectra 1000
Treated Spectra 1000	TS1000	8 layers treated Spectra 1000

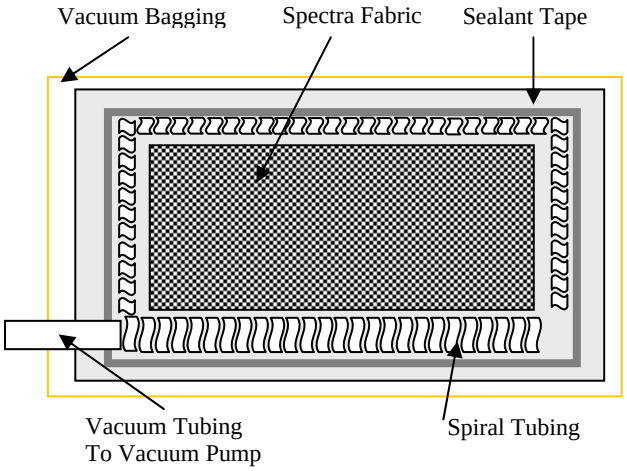

Figure 1: Sample construction setup

3. Test Method

An Instron Dynatup drop tower, Model 9250HV, was used for impact testing. This machine is capable of impacting samples at energies of up to 826 J utilizing a spring-assist. For this study, all samples were impacted with a 7.25 kg drop weight. Since the drop weight was not changed, the different impact energies were achieved by adjusting the drop height. A pneumatic clamping fixture seen in Figure 2, with a 76.2 mm diameter opening, secured each sample during impact. The samples were impacted with a 12.7 mm diameter striker with a hemispherical tip, constructed out of high strength steel. Impulse software was used to display and store the impact data.

The compression testing was conducted using a 50 kip MTS fatigue test system. The compression test fixture was designed similar to a Boeing Model

CU-CI fixture [[5]]. This fixture, seen in Figure 3, is specifically designed with side supports to prevent buckling during compression testing. For this study, the side supports were used for all compression tests.

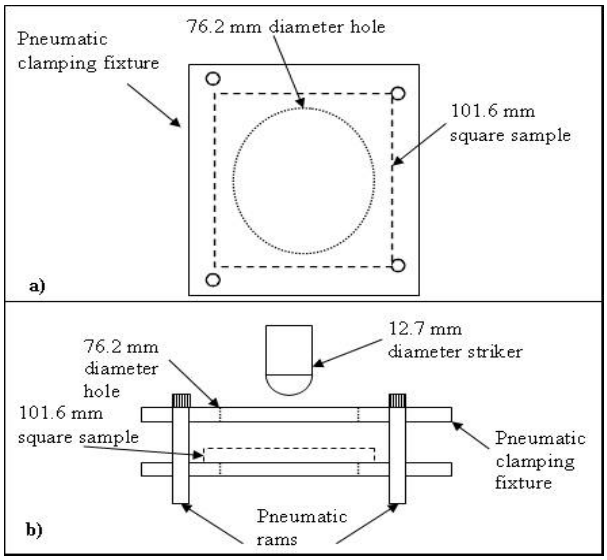

Figure 2: Pneumatic clamping fixture,
a) top view, b) side view

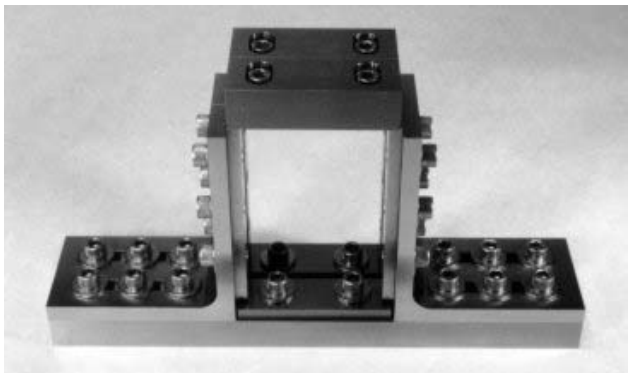

Figure 3: Boeing Model CU-CI

4. Results

The load-displacement curves for impact tests vary according to the impact energy. A typical curve is shown in Chart 1. As can be seen, the first peak in the load curve is known as the incipient damage load, Pi, which is the energy at which damage is initiated. The maximum load, Pm, is the highest peak on the load curve. Maximum energy and maximum deflection occur simultaneously as can also be seen in Chart 1. Elastic energy is defined as the difference between the maximum energy and the stabilized energy, shown by E in the figure. Total energy absorbed can be calculated as the integral of the area under the energy curve.

The total energy can be broken into sections and used to calculate the ductility index, DI. The DI is a convenient, dimensionless parameter relating how much of the total energy is used towards damage initiation and damage propagation. The energy applied prior to the incipient damage load is called the incipient energy, Ui; the energy applied prior to the maximum load is called the initiation energy, Um. The energy applied after the maximum load is known as the propagation energy, Up. The ration Up/Um is the ductility index. Brittle materials, such as cooled laminates, have lower ductility indices due to their low propagation energy. Likewise, ductile materials, such as heated laminates, have higher ductility indices due to their high propagation energy.

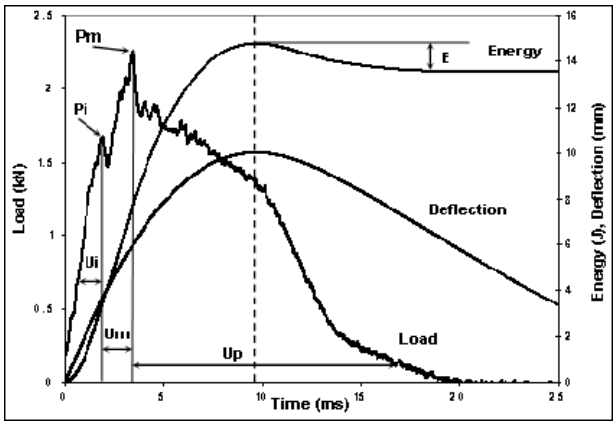

Chart 1: Sample load-energy-deflection-time curve.

Table 4: 10 joule impact properties

10J Samples	US1000	TS1000
Modulus, E (GPa)	11.542	15.879
Maximum Load (kN)	2.054	2.438
Incipient Load (kN)	1.923	2.327
Elastic Energy (J)	0.841	1.298
Maximum Energy (J)	10.195	10.016
Incipient Energy (J)	7.860	7.157
Ductility Index	-0.022	0.038

Table 5: 15 joule impact properties

15J Samples	US1000	TS1000
Modulus, E (GPa)	12.414	16.602
Maximum Load (kN)	2.569	2.848
Incipient Load (kN)	2.435	2.184
Elastic Energy (J)	0.973	1.538
Maximum Energy (J)	14.871	14.759
Incipient Energy (J)	11.682	6.061
Ductility Index	-0.002	-0.043

Table 6: 20 joule impact properties

20J Samples	US1000	TS1000
Modulus, E (GPa)	12.794	15.561
Maximum Load (kN)	2.898	2.961
Incipient Load (kN)	2.629	2.045
Elastic Energy (J)	0.437	1.870
Maximum Energy (J)	19.318	19.186
Incipient Energy (J)	13.474	5.556
Ductility Index	0.041	0.084

Table 7: 25 joule impact properties

25J Samples	US1000	TS1000
Modulus, E (GPa)	10.777	16.019
Maximum Load (kN)	1.870	3.181
Incipient Load (kN)	1.622	2.269
Elastic Energy (J)	0.234	2.070
Maximum Energy (J)	23.609	23.076
Incipient Energy (J)	6.253	6.541
Ductility Index	0.535	0.077

Characteristic Loads

Chart 2 shows the incipient load, Pi, and maximum load, Pm, for both treated and untreated samples as a function of impact energy. For the treated samples PI appears to be nearly the same, roughly 2.2kN, regardless of the impact energy; this phenomenon is also noted by Hirai et al [[6]] and Cartie [[7]]. This suggests that damage initiation is independent the impact energy.

This is beneficial since internal, non-visible damage can cause catastrophic failure of composite structures. By knowing Pi for a given composite, internal damage can be assessed by simply knowing the impact load. Chart 2 also shows that Pm increases with increasing impact energy.

The untreated samples exhibited completely different behavior. Chart 2 shows that both Pi and Pm followed similar trends across the range of impact energies. They increase with increasing impact until the 25J impact where there is a sudden decrease in both characteristic loads. The large drop at the 25J impact level was caused by the samples deforming and being pressed into the hole in the center of the clamping fixture (Figure 2) which can be seen in Figure 4. An improperly bonded sample will not exhibit the same response to impact ad a properly bonded sample. This was a major factor leading to Pi shadowing PM. As can be seen in Figure 5, the untreated samples showed complete internal delamination after impact whereas the treated samples only exhibited local delamination.

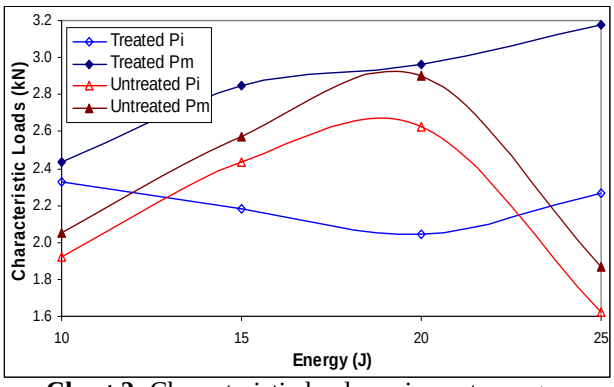

Chart 2: Characteristic loads vs. impact energy.

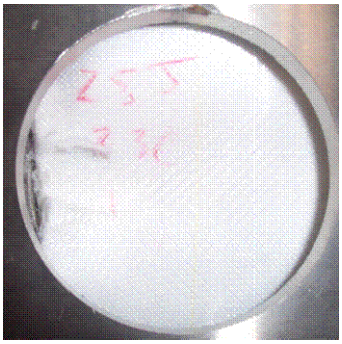

Figure 4: Untreated sample pressed into the clamping fixture.

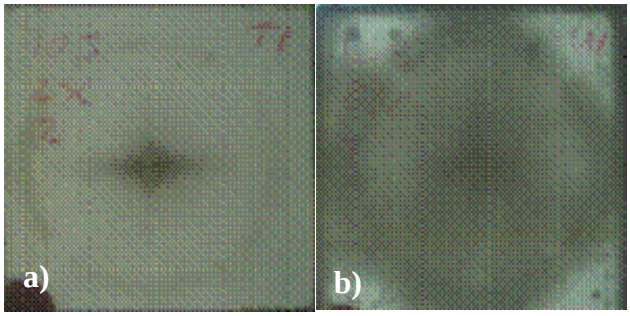

Figure 5: a) Treated 10J impact sample, **b)** Untreated 10J impact sample

Maximum Energy

Chart 3 shows the maximum energy applied at the various impact levels. As can be seen, the applied energies were very close to the desired impact energies with the largest variance of 7.6% occurring at the 25J impact level. Likewise, the energies applied to both the treated and untreated samples were nearly identical across all impacts with the largest variance of 2.3% occurring once again at the 25J impact level.

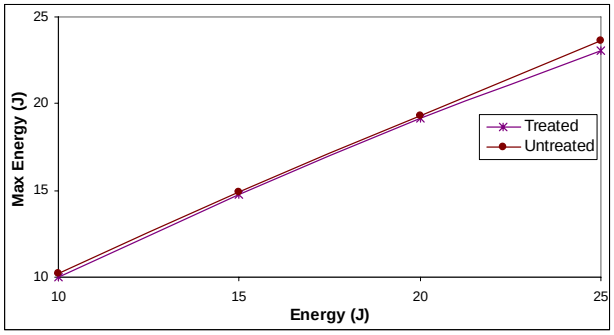

Chart 3: Maximum energy applied vs. impact energy.

Maximum Deflection

Maximum deflection of the sample is affected by impact energy as can be seen in Chart 4; the amount of deflection increases as impact energy increase. At lower energy impacts, the untreated samples exhibited more deflection than the treated ones. At higher impact energies, the treated samples deflected more than those that were untreated. The difference in deflection is nearly negligible at the 10, 15, and 20-joule impact energies. There is a large difference at the 25-joule impact energy. More deflection is desired in order to withstand larger impacts. Under higher impact energies, the untreated samples deflected enough to press them into the 76.2 mm hole in the center of the clamping fixture, see Figure 4. The samples never failed they simply deformed enough to allow the test apparatus to reach its maximum travel.

Maximum Load

Chart 5 shows the variation of maximum load versus impact energy levels. At all impact levels the treated samples withstood a higher load that those left untreated. This is even more noticeable at the 25J impact energy. In the treated samples, as impact energy increased so did the maximum load. The untreated samples followed the same trend until the 25-joule impact where the maximum load decreased drastically. As mentioned previously, the untreated samples deformed to a point of being pressed into the hole in the center of the clamp fixture, see Figure 4, causing a reduction in maximum load.

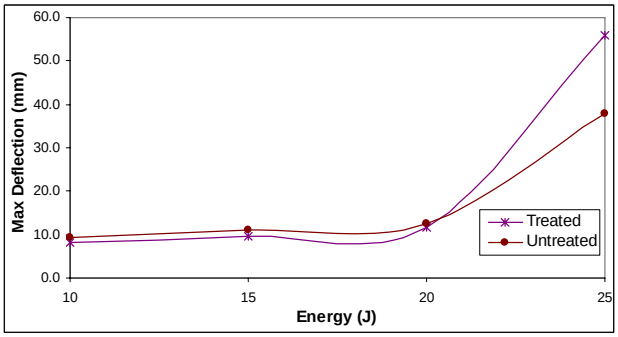

Chart 4: Maximum deflection vs. impact energy.

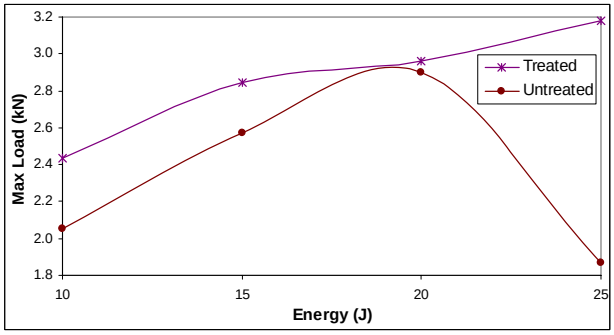

Chart 5: Maximum load vs. impact energy.

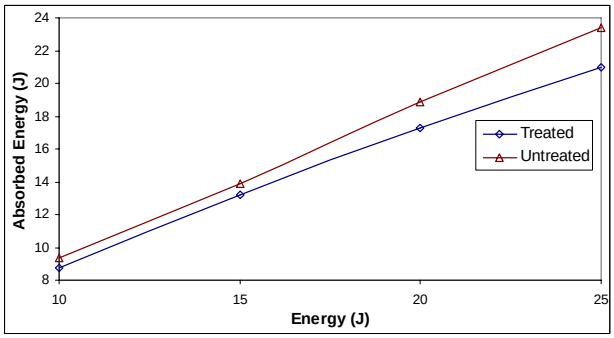

Chart 6: Absorbed energy vs. impact energy.

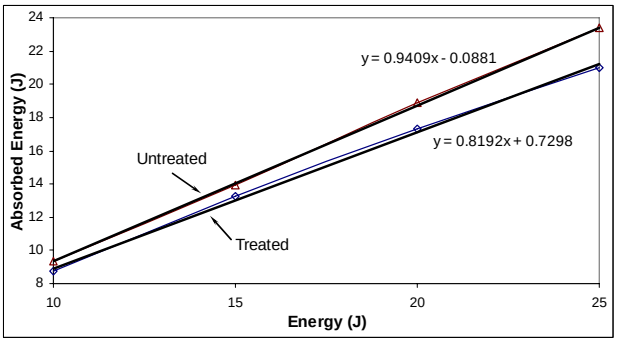

Chart 7: Slopes of absorbed energy vs. impact energy.

Absorbed Energy

The trends of absorbed energy versus impact energy can be seen in Chart 6. As the impact energy increases, so does the absorbed energy. Both the treated and untreated samples increased in a nearly linear manner. The untreated samples exhibited slightly higher absorbed energies at all impact energies. Chart 7 shows the slopes for each set of samples. The slope of the trend line for the untreated sample is slightly higher that that of the treated samples. A higher absorbed energy level leads to more permanent damage to the samples as well as higher residual stresses. Higher residual stresses reduce the energy necessary to cause further damage during subsequent impacts.

Elastic Energy

Elastic energy is the amount of energy not converted to permanent damage, denoted by E in Chart 1. Most elastic energy recovered from impact tests is in the form of striker rebound. As can be seen in Chart 8, increasing impact energy causes the amount of elastic energy to increases in the treated samples. Conversely the elastic energy of the untreated samples decreased with increasing impact energy. At lower impact energies both sets of samples absorbed nearly the same energy, which can be seen in Chart 6, and produced roughly the same amount of elastic energy. At higher impact energies the treated samples absorbed less energy than the untreated ones and therefore produced more elastic energy. At the highest impact energy there was virtually no elastic energy in the untreated samples due to the fact that almost all the energy is transferred to the part in the form of permanent deformation and internal delamination which can be seen in Figure 5. Another contributing factor is the samples being pressed into the center of the clamping fixture.

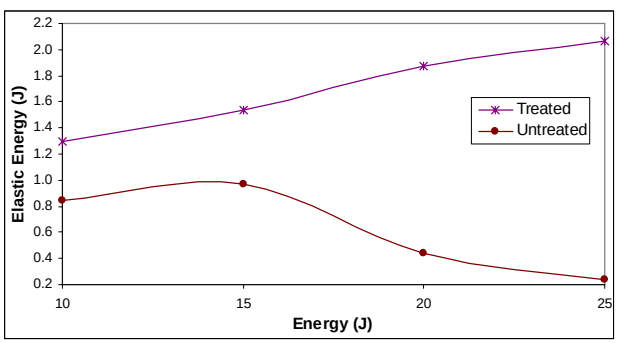

Chart 8: Elastic energy vs. impact energy.

Initiation Energy

Chart 9 shows the initiation energy, Um, which is also known as the energy required to reach maximum load. As can be seen, the initiation energy chart and the maximum load chart, Chart 5, look very similar. The initiation energy increased with increasing impact energy for the treated samples; likewise it increased for the untreated samples until the 25J impact. The drop in initiation energy at this impact level was probably caused, once again, by the untreated samples being pressed into the clamping fixture. Overall, the untreated samples required more energy to achieve the maximum load than the treated samples.

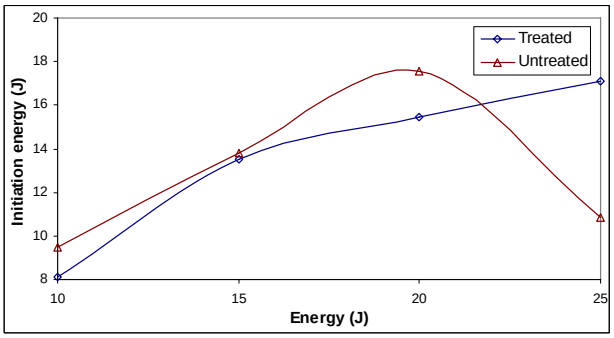

Chart 9: Initiation Energy vs. impact energy.

Propagation energy

The propagation energy, Up for the different impact energies and samples can be seen in Chart 10. Lower impact energies resulted in low, even negative, propagation energy. Both the treated and untreated samples had a noticeable increase in propagation energy at the 25-joule impact level. Higher propagation energy means more energy is required to continue damage growth after it has been initiated. Samples with higher propagation energy are more stable after damage has been initiated than those with lower propagation energy.

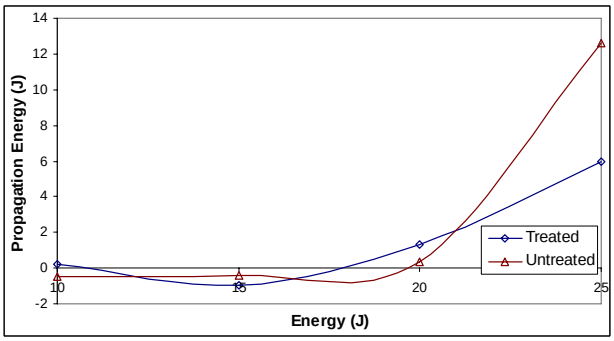

Chart 10: Propagation energy vs. impact energy.

Ductility Index

The ductility index, DI, is the ratio of propagation energy to initiation energy (Up/Um). Brittle materials tend to exhibit lower DI's than do ductile materials. As can be seen in Chart 11, the treated samples have a lower ductility index at higher impact energies, and have a more constant DI across all energy levels. The DI's of the untreated samples is extremely low at lower impact energies, but at the 25-joule impact the ductility index increases greatly. This sharp increase comes from the samples being pressed into the camping fixture in a ductile manner. Low ductility indexes are preferred for objects subjected to impacts since a low DI indicates a lager amount of energy is required to initiate any damage. Unfortunately, a low DI also indicates that if damage does occur very little additional energy is required to cause total failure.

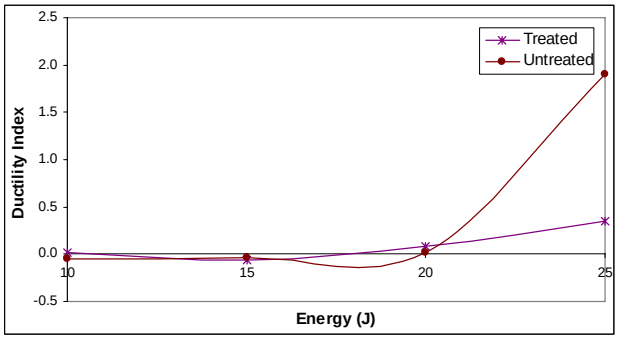

Chart 11: Ductility index vs. impact energy.

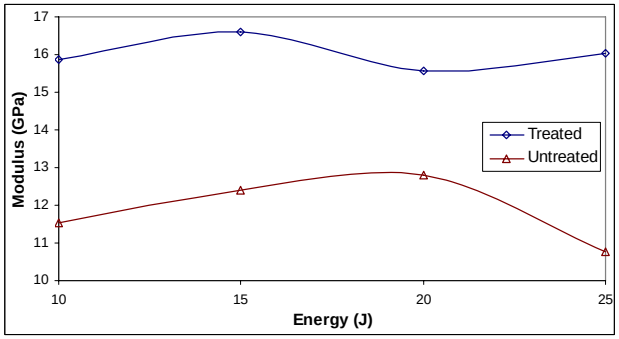

Chart 12: Modulus of elasticity vs. impact energy.

Modulus

The modulus of elasticity is a measure of the amount of deformation a part can undergo before permanent damage (deformation) occurs. Chart 12 graphically shows the treated samples had a higher modulus than the untreated ones across all impact energies. The treated samples exhibited a relatively constant modulus, roughly 16 GPa, while the modulus of the untreated samples increased slightly as the impact energy increased, with a high of nearly 12.5 GPa, until the 25-joule impact where it drastically decreased to around 11 GPa. **Figure 6.**

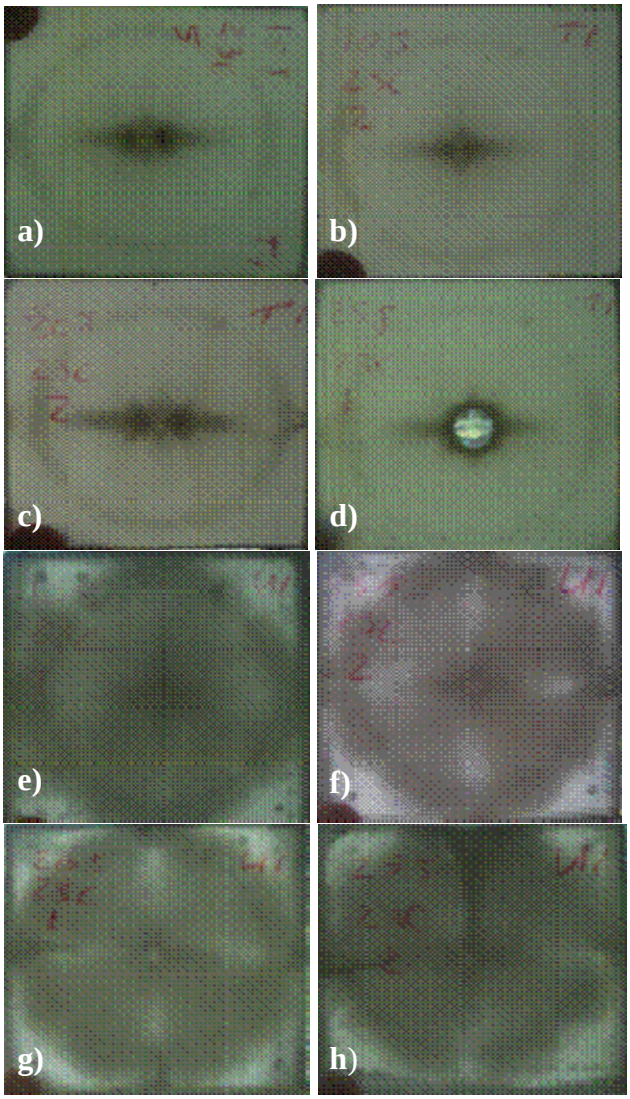

Figure 6: Delamination and damage of **a)** 10J treated, **b)** 15J treated, **c)** 20J treated, **d)** 25J treated, **e)** 10J untreated, **f)** 15J untreated, **g)** 20J untreated, and **h)** 25J untreated

Spectra 1000 Conclusions

By visual inspection the treated samples appeared to have wetted and absorbed the epoxy better than the untreated samples. Figure 7 shows the difference in color between the two sets of samples. As can be seen, the untreated samples appear to have absorbed very little epoxy. Also to be noted, when the untreated samples were cut, a large amount of edge delamination occurred. Internal delamination could be noticed by simply applying a slight bending

pressure to the samples by hand. The untreated samples were very flexible at room temperature while the treated samples were far more rigid. The treated samples showed no permanent delamination when bent by hand. The response to impact of the treated samples was more what was expected while the untreated samples did not have the rigidity to perform as expected.
Picture of sample folded into clamping fixture…the pneumatic clamp could not hold the sample from shifting and pressing down in to the hole. (13) A large buckle can be seen in the untreated samples impacted at 25J in Figure 8. This was a result of being pressed into the fixture.

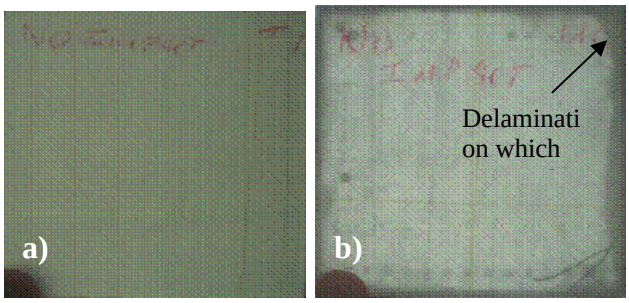

Figure 7: Non-impacted **a)** treated and **b)** untreated samples

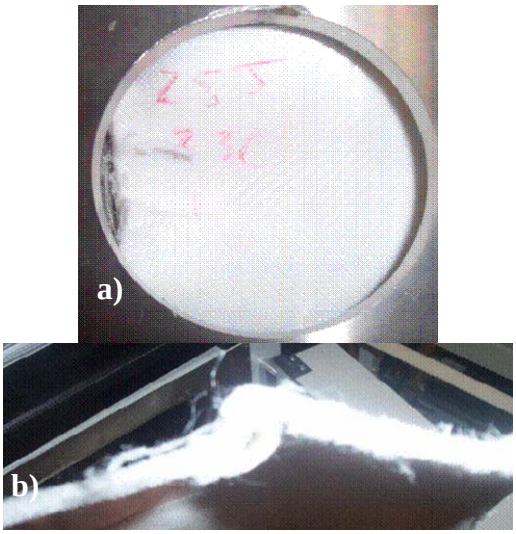

Figure 8: Bending caused by 25J impact on untreated samples; **a)** top view, **b)** side view

Damage Types (Modes)

Figures 9. a-d show the different damage modes. At low energy levels, back surface cracking, bending, and plastic deformation of the laminates are the most common types of damage. At intermediate energy levels the most common damage is delamination and back surface cracking. At high energy levels fiber breakage leading to total penetration is the most common form of damage. At low temperatures the laminates are rigid and primarily only back surface and matrix cracking occur with a small amount of bending. At higher temperatures the laminates are more fluid and elastic and undergo more bending. Inter-laminar bonding decreases with increasing temperature causing increased delamination. Delamination leads to weaker laminates and eventually causes total penetration. Damage type and failure should be considered when designing the use environment for these laminates.

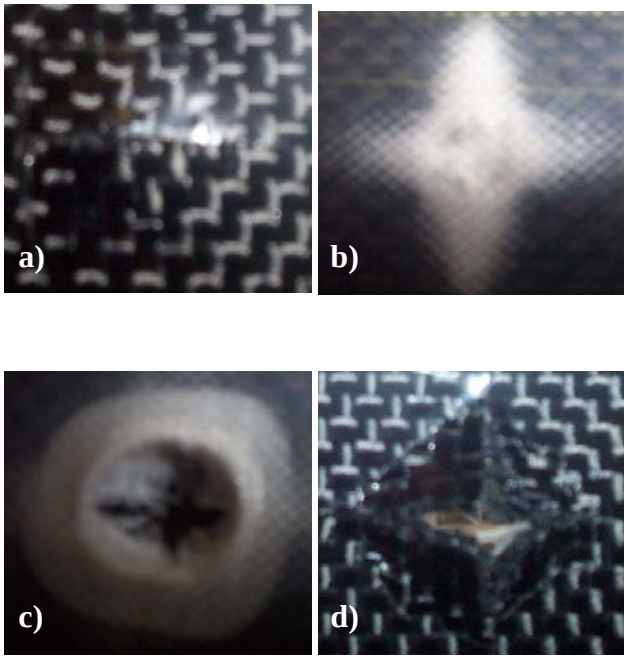

Figure 9: Damage types: **a)** back surface cracking, **b)** bending/plastic deformation, **c)** delamination, **d)** total penetration.

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