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The Effect of Plasma Treatment on Interfacial Bond Characteristics of Kevlar/Epoxy Laminates as Measured by T-Peel Tests

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13. ABSTRACT (Maximum 200 words) Composite materials are used in a wide variety of applications where their anisotropic properties allow the material to be tailored for each application. Unfortunately, aramid fiber composites may have limited applications due to poor transverse properties. These poor transverse properties are the result of weak interfacial bonding. Improvement of the interfacial bond strength has been an area of active research. Plasma treatment is one method that can be used to modify the surface of a fiber in an effort to improve the fiber-matrix adhesion. In this study, Kevlar/epoxy laminates were peel-tested (T-peel) to study the effect of plasma treatment on the interfacial properties. The T-peel results indicate that ammonia plasma treatment improves the interfacial bond strength of Kevlar/epoxy composites, increasing the T-peel strengths by approximately 30%.					
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Introduction

Polymer composite materials possess high specific strength and stiffness properties. These advantageous features, combined with competitive cost compared with metals, are driving composites into widespread application. They are commonly used in aerospace, military, sporting goods, and transportation to name a few major markets.

The interfacial bond that exists between the fiber and matrix of a composite is an important component of composite materials as it facilitates load transfer between fibers. Composites having weak interfacial bonds have low interlaminar and intralaminar shear strengths. Allred, et al. [1] have shown that moisture can weaken the fiber-matrix interface in aramid/epoxy composites, leading to poor transverse properties in wet environments. Improvement in fiber-matrix bonding should produce composites with greater resistance to property degradation by moisture.

Aramid fiber composites are especially hindered by poor interfacial bonds, and improvement of the interfacial bond strength has been an area of active research. Creation of covalent bonding sites on the fiber surface is expected to increase the interfacial strength between the fiber and resin. Stoller, et al. [2] treated Kevlar fibers to modify their surface chemistry by an ammonia plasma process. This process introduces reactive amine groups to the surface of the fiber that are potential covalent bonding sites between the fiber and resin through participation of the amine group in the epoxy curing reaction.

To evaluate qualitatively the interfacial bond strength of fibrous composite Kevlar/epoxy laminates a peel test (T-peel) was performed using ASTM D1876, "Standard Test Method for Peel Resistance of Adhesives (T-peel Test)" as a reference. In this study, the laminates were T-peel tested in order to study the effect of plasma treatment on the interfacial properties. The T-peel results indicate that ammonia plasma treatment improves the interfacial bond strength of Kevlar/epoxy composites, increasing the T-peel strengths by approximately 30%.

Materials

The Kevlar/epoxy specimens studied were two-ply laminates obtained from PDA Engineering (now TPL, Incorporated) under a Phase II Small Business Innovative Research (SBIR) contract. Two different types of specimens were evaluated. One type of laminate was prepared from plain weave fabric and the other was prepared from unidirectional tape. In the unidirectional material, the fiber was aligned parallel to the long axis of the specimen. Ten control and ten plasma treated specimens were studied from each group.

The fabric specimens were prepared using Kevlar 49 (style 281) treated in an ammonia plasma batch process. Unidirectional Kevlar/epoxy prepreg was prepared by treating 7100 denier roving in a continuous ammonia plasma treatment-resin

1. ALLRED, R. E., and ROYLANCE, D. K. *Transverse Moisture Sensitivity of Aramid/Epoxy Composites*. Journal of Materials Science, v. 18, 1983, p. 652-656.
2. ALLRED, R. E., HARRAH, L. A., SALAS, R. M., and GORDON, B. W. *Plasma Treatment Processes for Improved Interfacial Adhesion in Kevlar/Epoxy Composites*. SBIR Phase II Final Report, MTL TR 90-60, November 1990.

impregnation facility developed under the SBIR Phase II program [2]. The epoxy resin used was Epon 826, a diglycidyl ether of bis-phenol A epoxy, cured with Epon curing agent Z (both products of Shell Chemical Company). For a detailed description of the processing and preparation of these specimens refer to Reference 2.

Experimental

All specimens in the study were rectangular with a 1 in. width. The fabric specimens were 6 in. long, and the unidirectional specimens were 12 in. long. One inch of the laminate length was not bonded to allow the specimen to be clamped into the grips of the testing machine, as shown in Figure 1.



Figure 1. T-peel test geometry.

The laminates were T-peel tested in a floor model Instron screw-type test machine to determine interlaminar shear strength. ASTM D1876 was used as a reference for the procedure, but the crosshead speed was changed to 2 in./minute. The unbonded end of the specimen was bent at a 90° angle to the bonded end of the specimen and clamped into the test grips. The test load data was collected with an IBM personal computer using a Keithley data acquisition system. The Instron chart recorder was used as a backup for the computer. The raw data was reduced separately in a later analysis.

During testing the computer sampled voltage signals from the Instron at a rate of 10 points per second. The raw data was later converted to load values and averaged using another program. All valid data points from the initial peak to the end of the test were used to calculate the average load value. For the fabric specimens, the end of the test was the point when the specimen was completely peeled apart. For the unidirectional specimens, the test was ended after the first 9 in. were peeled. The T-peel strength was calculated by dividing the average load by the specimen width. After testing, the specimen failure surfaces were studied under an optical microscope to compare differences between the control and treated specimens.

Results and Discussions

T-Peel Tests

The T-peel test is generally used to study the effectiveness of an adhesive bond to increase the peel resistance between two adherends. During a T-peel test the force builds up until fracture initiates and the energy is released. The force then builds up again and the fracture cycle is repeated. This test geometry favors failure between the plies; therefore, the load needed to initiate the fracture cycle should be

related to interlaminar bond strength [3]. Figure 2 compares the typical continuous load per unit width versus crosshead displacement curve obtained for representative fabric control and treated specimens. Figure 3 shows the same curve generated for typical unidirectional specimens.

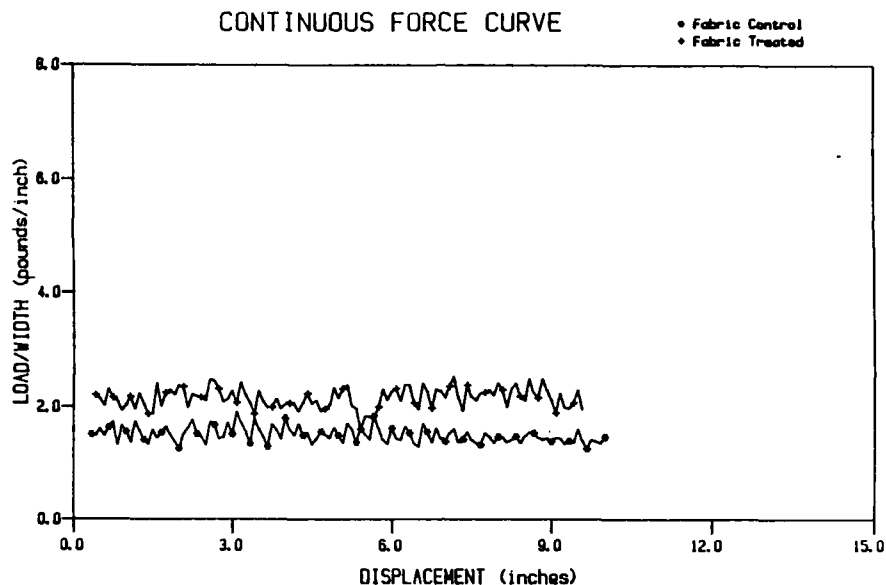


Figure 2. T-peel curve for fabric specimen group.

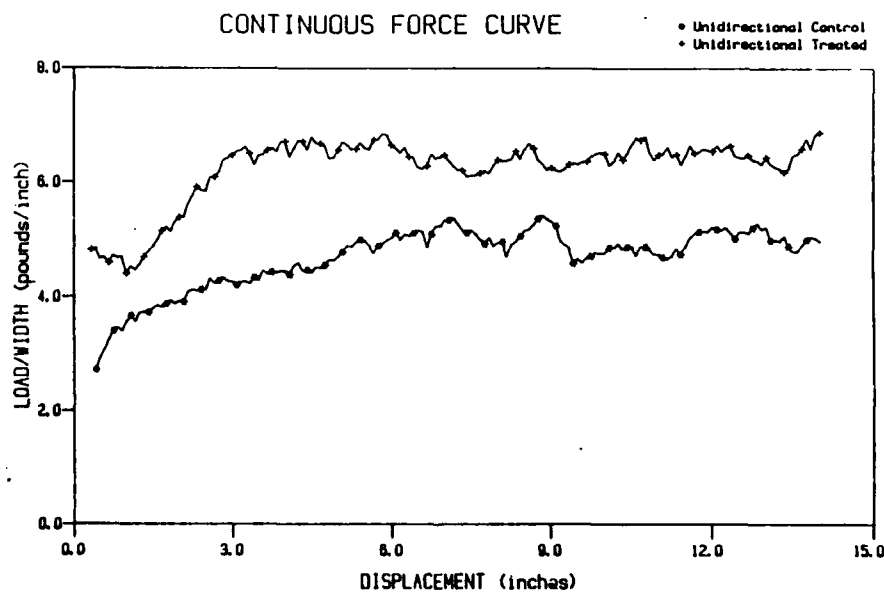


Figure 3. T-peel curve for unidirectional fiber group.

3. STOLLER, H. M., DELOLLIS, N. J., and RODACY, C. *Plasma Treatment Optimization of Polyaramid Filaments to Improve Kevlar/Epoxy Composites*. SBIR Phase I Final Report, PIDA Engineering, May 1986.

The fabric specimens show a jagged curve indicating crack propagation-crack arrest cycles, while the unidirectional specimens exhibit a smoother fracture curve more representative of continuous crack propagation. The T-peel test results for each specimen are listed in Table 1.

Table 1. T-peel test results

T-peel tests			
Fabric control		Fabric treated	
Specimen (number)	Avg load (lb/in.)	Specimen (number)	Avg load (lb/in.)
1	2.07	11	2.35
2	2.05	12	2.16
3	1.85	13	2.62
4	2.29	14	2.53
5	2.45	15	2.46
6	2.06	16	2.38
7	2.06	17	3.01
8	2.01	18	3.12
9	1.51	19	3.05
10	1.68		
	Avg		Avg
	2.00		2.63
	Std		Std
	0.26		0.33

Unidirectional control		Unidirectional treated	
Specimen (number)	Avg load (lb/in.)	Specimen (number)	Avg load (lb/in.)
1	4.45	11	5.33
2	4.55	12	6.61
3	4.92	13	7.02
4	4.35	14	6.53
5	5.27	15	6.22
6	6.37	16	5.57
7	5.11	17	5.72
8	4.29	18	7.14
9	4.31	19	6.64
10	5.39		
	Avg		Avg
	4.90		6.31
	Std		Std
	0.63		0.61

Figure 4 compares all the specimens with one standard deviation range. These data show that higher values of T-peel strength are obtained for the plasma treated specimens. The fabric specimens show an increase of 32% in the T-peel strength with ammonia plasma treatment. The unidirectional specimens show an increase of

29% in T-peel strength with continuous plasma treatment. This indicates the plasma treatment is effective in promoting improved adhesion between the fiber and matrix. Modification of the Kevlar surface with an ammonia plasma offers a method for improving the mechanical properties of Kevlar/epoxy composites as evidenced by the higher peel strengths obtained for plasma treated specimens. The values obtained for the fabric controls in this study are in line with those obtained in the Phase II SBIR report (the untreated specimens had values of 1.9 lb/in. in the Phase II study) [2], but the values found in this study for the treated fabric specimens are lower (treated specimens had values of about 4 lb/in. in the Phase II study) [2].

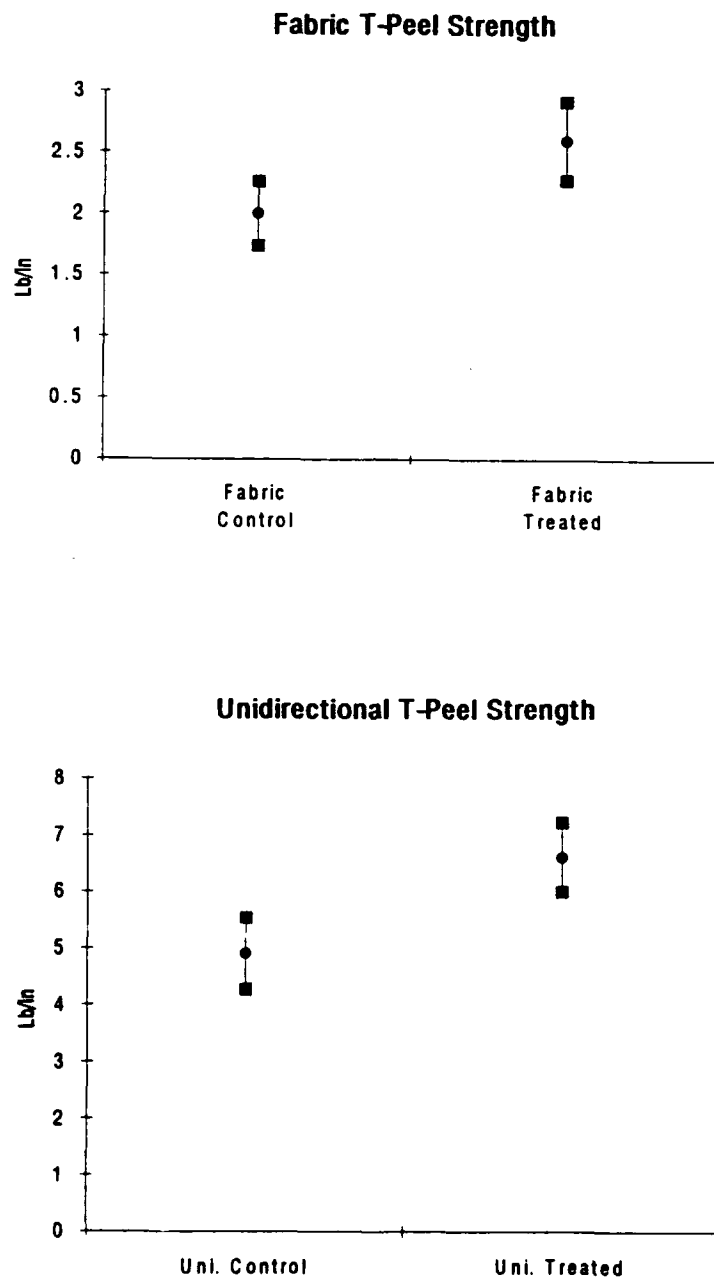


Figure 4. T-peel strength data.

Microscopy

Figures 5 and 6 compare the failure surfaces of the fabric and unidirectional specimens, respectively. There are no discernible differences in the failure surfaces of the treated and untreated specimens. However, further microscopy study, particularly scanning electron microscopy, should be performed to further explore the effects of plasma treatment.



Figure 5. Failure surfaces of fabric specimens.

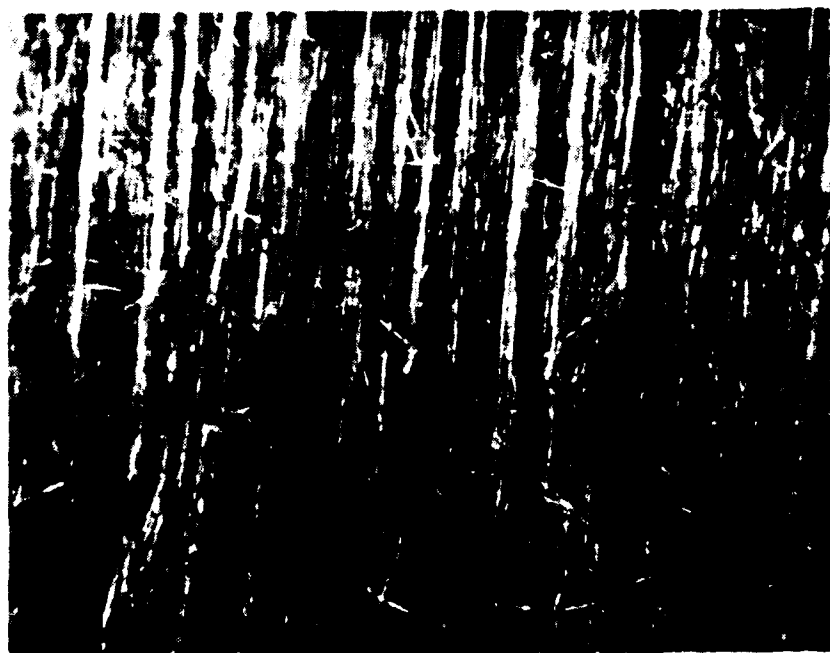


Figure 6. Failure surfaces of unidirectional materials.

Summary

The results of the T-peel tests indicate that ammonia plasma treatment is effective in improving the fiber-matrix adhesion and the mechanical properties of Kevlar/epoxy composites. The treated fabrics show an increase of 32% in the T-peel strength when compared to the control. The treated unidirectional material shows an increase of 29% when compared to the control data set. These data are in agreement with the results obtained by Allred, et al. [2] that report ammonia plasma treatment increases the interfacial bond strength leading to improved mechanical properties for the treated materials.

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