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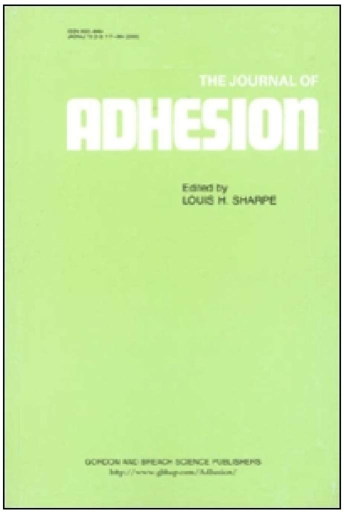

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The Effect of Surface Activation in Polymer Matrix-Carbon Fiber Interactions

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Various carbon fibers (CF) were surface modified by chemical and electrochemical treatments for the purpose of establishing organic functional groups on the fiber surface. A series of fibers, surface oxidized for various periods of time, was prepared. The amounts of surface functionalities formed were assessed by means of contact angle measurements on single fibers. A suitable set of probe liquids was used to determine the LW (Lifshitz-van der Waals) and acid-base components of carbon fiber surfaces. Similar tests were made on commercial, sized carbon fibers, polystyrene (PS) and polymethyl methacrylate (PMMA), and their surface energies determined in terms of LW, surface acidity and surface basicity components. Work of adhesion values were calculated of all combinations of CF and polymer matrix couples by using these surface energies of both constituents. The calculated work of adhesion values were correlated to the ILSS values obtained from single fiber pull out tests with PS and PMMA as matrices.

KEY WORDS: carbon fiber; surface treatment; contact angle; acid- base interactions; work of adhesion; pull out test; interfacial laminar shear strength.

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INTRODUCTION

Carbon fibers, with their exceptionally high strength and stiffness values, are good candidates for aero-space applications, where the individual filaments are bonded together by a matrix material, giving a composite structure whose properties depend on the properties of both the fiber and the matrix.[1] The matrix serves the dual purposes, among others, of protecting the fibers from damage and of transferring stress into them.

In the case of carbon fiber composites, strong and good adhesion of the matrix polymer to the surface of the reinforcing filler (carbon fiber) is required, and it is a prerequisite condition for carbon fibers to serve as reinforcements for high-performance composites.[2-5] Several processing factors and final mechanical properties of composites depend highly on the interfacial interactions and intimate contact between carbon fiber and resins. These are better penetration of the resin into the fiber bundles,[4] protection of the carbon fiber from the oxidative environments, resistance to the abrasion, and proper load transfer in composites.[6]

Carbon-fiber-based composites suffer from a lack of shear strength between fiber and matrix. The virgin carbon fibers do not demonstrate the desired level of adhesion

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with high temperature thermosetting polymers or with engineering thermoplastics.[2] In fact, due to the relative smoothness of the fiber surface and the chemical inertness of the carbon atoms of the basal planes, the interfacial interactions between fiber and matrix are very low and generally insufficient. The enhancement of the interaction between fiber and matrix is a key operation used to produce a good quality carbon fiber composite; therefore, the carbon fibers are generally given a number of surface treatments to increase their surface reactivity and surface energy.[7-9]

In this study, the fiber surfaces were modified chemically and electrochemically and their surface energies were determined in terms of Lifshitz van der Waals (LW) and acid and base components from advancing contact angle measurements. On the same set of carbon fibers, single fiber pull-out tests were carried out using polystyrene (PS) and polymethyl methacrylate (PMMA) as model matrices that have LW only and acid-base plus LW surface energies, respectively. Our purpose here is to validate the use of surface energy determination in the estimation of the adhesion strength between the fiber and the polymer matrix.

THEORY

The total surface energy, γ_i^{TOT}, of a given non-metallic material (i) can be considered as composed of two parts: the Lifshitz-van der Waals (γ_i^{LW}) and the acid-base component (γ_i^{AB}).[10,11] The former represents the dispersion forces, dipole-dipole (Keesom) and induction (Debye), and the latter represents the short range H-bonding or acid-base interactions. This is written as the sum of the two components,

$$\gamma_i^{\mathrm{TOT}} = \gamma_i^{\mathrm{LW}} + \gamma_i^{\mathrm{AB}} \tag{1}$$

where the acid-base term is a property of one that depends on the mutual interaction of two unlike species, an acid and a base. γ_i^{AB} is composed of two surface parameters which are independent of the physical presence of another one: γ_i^{+}, the Lewis acid component and, γ_i^{-}, the Lewis base component of the surface free energy. These, together, yield the acid-base component of surface free energy, γ_i^{AB}.

$$\gamma_i^{\mathrm{AB}} = 2(\gamma_i^{+}\gamma_i^{-})^{1/2} \tag{2}$$

The most characteristic feature of these Lewis acid and Lewis base components is that they are not additive although the apolar ones are. It means that if phase i possesses only γ_i^{+} or γ_i^{-}, this component does not participate in the total surface free energy of the phase i. However, it will interact with the complementary component of phase j. As a result, the total surface free energy of a phase i is,

$$\gamma_i^{\mathrm{TOT}} = \gamma_i^{\mathrm{LW}} + \gamma_i^{\mathrm{AB}} = \gamma_i^{\mathrm{LW}} + 2(\gamma_i^{+}\gamma_i^{-})^{1/2} \tag{3}$$

The values of $\gamma_i^{\mathrm{AB}}, \gamma_i^{+}$ and γ_i^{-} can be determined by using the contact angle (θ) and "Complete Young Equation",[12]

$$(1+\mathrm{Cos}\theta)\gamma_i^{\mathrm{TOT}} = 2\,[(\gamma_i^{\mathrm{LW}}\gamma_J^{\mathrm{LW}})^{1/2} + (\gamma_i^{+}\gamma_J^{-})^{1/2} + (\gamma_i^{-}\gamma_J^{+})^{1/2}] \tag{4}$$

The Lifshitz-van der Waals component of a solid surface (S) can be found from the contact angle of an apolar liquid,[13] where $\gamma_{\mathrm{L}}^{\mathrm{TOT}} = \gamma_{\mathrm{L}}^{\mathrm{LW}}$, on the solid surface. In this case

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Eq. (4) reduces to,

$$(1+\mathrm{Cos}\theta_{\mathrm{LW}})\gamma_{\mathrm{L}}^{\mathrm{TOT}} = 2\,[\gamma_{\mathrm{L}}^{\mathrm{LW}}\gamma_{\mathrm{S}}^{\mathrm{LW}}]^{1/2} \tag{5}$$

or

$$\gamma_{\mathrm{S}}^{\mathrm{LW}} = [\gamma_{\mathrm{L}}^{\mathrm{LW}}(1+\mathrm{Cos}\theta_{\mathrm{LW}})^2]/4 \tag{6}$$

As a result, the LW component of a solid surface S can be calculated by applying the contact angle of an apolar liquid on the surface S from Eqn. (5) or (6).

For a bipolar liquid, with surface tension γ_1, acidic and basic surface parameters, γ_1^+ and γ_1^-, respectively, and apolar surface component, γ_1^{LW}, the equation corresponding to Eqn. (5) is,

$$(1+\mathrm{Cos}\theta_1)\gamma_1^{\mathrm{TOT}} = 2\,[(\gamma_1^{\mathrm{LW}}\gamma_{\mathrm{S}}^{\mathrm{LW}})^{1/2} + (\gamma_1^+\gamma_{\mathrm{S}}^-)^{1/2} + (\gamma_1^-\gamma_{\mathrm{S}}^+)^{1/2}] \tag{7}$$

and for a second bipolar liquid, with surface parameters γ_2^+, γ_2^- and γ_2^{LW}, the corresponding equation is,

$$(1+\mathrm{Cos}\theta_2)\gamma_2^{\mathrm{TOT}} = 2\,[(\gamma_2^{\mathrm{LW}}\gamma_{\mathrm{S}}^{\mathrm{LW}})^{1/2} + (\gamma_2^+\gamma_{\mathrm{S}}^-)^{1/2} + (\gamma_2^-\gamma_{\mathrm{S}}^+)^{1/2}] \tag{8}$$

Equations (7) and (8) constitute a set of two simultaneous equations, in terms of the parameters of the solid, γ_{S}^+, γ_{S}^- and $\gamma_{\mathrm{S}}^{\mathrm{LW}}$ and the two contact angles θ_1 and θ_2 that are measured on the solid surface. These equations are then solved for $\gamma_{\mathrm{S}}^+, \gamma_{\mathrm{S}}^-$ with the known $\gamma_{\mathrm{S}}^{\mathrm{LW}}$, provided the γ_i^+, γ_i^- and γ_i^{LW} for the probe liquids are known.

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EXPERIMENTAL

Materials

Three intermediate modulus carbon fibers were used in these studies: Grafil (Courtaulds) IM7 (Hercules), G40-700 (BASF). These fibers are classified as intermediate modulus grade, having tensile modulus of about 280 GPa. The former is unsized but the others are sized. The probe liquids, diiodomethane (DIM), ethylene glycol (EG) and formamide (F), were of spectroscopic grade and were used without further purification. Styrene and methyl methacrylate monomers were vacuum purified and polymerized with benzoyl peroxide as the initiator.

Experimental Procedure

I. Surface Activation

The tows of unsized Grafil fibers to be activated were cleaned with a series of treatments. First, the fibers were heated at reflux in dichloro methane for 24 hours to remove impurities, followed by boiling in deionized water for 12 hours. The samples were then dried at 120 °C for 24 hours. The sized carbon fibers were heated at reflux in boiling water and vacuum dried.

The clean Grafil fibers were treated by two methods. One of them was wet oxidation, performed by placing the samples in boiling nitric acid (70% by weight) for various periods of time (5, 30, 60, 120 minutes), after which the samples were kept in boiling water for 24 hours and dried at 100 °C for 24 hours. Grafil fibers were also anodically oxidized in a laboratory scale electrolytic cell. All the electrochemical treatments were performed using a Bank POS 73 potentiostat. Typically a 150 mm length of 12 K tow was treated. The top of the tow was wrapped tightly with aluminum foil, to ensure a good electrical conductance. The fibers were placed inside a glass tube to prevent their contact with the cathode (platinum wire). The solution was stirred to prevent concentration polarization around the anode. In addition, nitrogen gas was bubbled into the electrochemical cell. For all treatments conducted, the carbon fiber tow was the anode and 50% by weight sulfuric acid was the electrolyte solution. The oxidation potential was kept constant at 2.0 V.

II. Contact Angle Measurements

Contact angles were measured tensiometrically with a Sartorious microbalance M25D in conjunction with an Oriel motor-driven stage that has a vertical displacement capability of 10 mm. The digital signals from the microbalance were recorded as their analog counterparts with a Linseis LY 140 type, X/Y recorder. In the case of carbon fibers, a single fiber specimen was prepared first, by taping 1 cm of a 2 cm length of fiber between two pieces of adhesive tape, with about 1 cm of the fiber exposed. The specimen was then suspended from the balance through a hole drilled in the tape. In all experiments a stage velocity of 1 μm/s was used to bring the fiber into contact with the liquid. The force on the fiber was measured as the liquid container was raised to obtain an advancing contact angle. Each contact angle measurement was repeated at least three times, giving an average ± 1.8° standard deviation.

Diiodido methane (DIM) was used as the probe liquid for Lifshitz-van der Walls interactions; ethylene glycol (EG) and formamide (F) were used to probe for the acid-base interactions. After each experiment, the perimeters of the fibers were determined by clipping off the wetted tip of the fiber, using a sharp razor blade, and measuring the wetting force using n-octane, which is a completely wetting liquid that is assumed to make a zero contact angle with carbon fiber. A different fiber from the same lot number was employed for each probe liquid. The surface tensions of the liquids used are presented in Table I.

In order to determine the contact angles of probe liquids on PS and PMMA, polymer-coated thin glass rods were used. Solutions of polystyrene and polymethyl methacrylate were prepared from their respective monomers in solution at 80 °C. At a suitable viscosity, the polymerization was stopped by refrigeration. The initiator,

TABLE I
Surface tension components of probe liquids[11, 14, 15] (values are in mN/m)

Liquid	γ_L^{TOT}	γ_L^{LW}	γ_L^{AB}	γ_L^{+}	γ_L^{-}
DIM	50.8	50.8	0.0	0.0	0.0
EG	48.0	29.0	19.0	3.0	30.1
F	58.0	39.0	19.0	2.3	39.6
n-Octane	21.8	21.8	0.0	0.0	0.0

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benzoyl peroxide, was at 1% of monomer concentration. The surface of the glass rods was coated by dipping the rods vertically into the polymer solution, followed by lowering the solution container at a rate of 1 μm/s with the micromanipulator of the motor-driven stage to ensure even coating. The rods were heated for 2 hours at 60 °C to complete the polymerization. For contact angle measurements, the film-coated glass rods were then suspended from the balance by a steel wire hook by the same technique used for carbon fibers.

III. Single Fiber Pull Out Test

Glass frames were constructed first, by bringing two borosilicate flat glass slides within 1 cm of each other. The slides were then secured from the two sides with clips. A 50 mm length of carbon fiber tow was laid out on a piece of white card and a single fiber was separated from the tow. The perimeter of the fiber was determined with wetting experiments by using a completely wetting liquid. This fiber was then mounted onto the frame using an adhesive tape such that the fiber transversed vertically the interval between the plates. One end of the fiber was bonded with a drop of cyanoacrylate adhesive onto the glass. To the other end on the second plate, a drop of the resin to be tested was added. Then, with the aid of a microscope, the end of the fiber in the resin was cut with a sharp razor blade to produce an immersion depth of around 100 μm. Finally, this assembly was placed in an oven to carry out the polymerization of the resin. The final form of the specimen is shown in Figure 1. The machine used for the pull-out tests was an Instron, type TM 1102. The test rate was controlled externally by using the Oriel motor-driven stage at very low velocity (~ 1 μm). One end of the specimen was gripped by the lower jaw of the tensile testing machine, which, in turn, was fixed on the vertical mobile stage attached to the base of the iron barrel of the testing machine. The top jaw, which is connected to the stress transducer, was fixed to the other end of the specimen. The clips were then carefully removed without breaking the fiber. The fiber was pulled out of the resin and the load recorded. Assuming that the shear stress is uniformly distributed along the immersion length, the mean value of the shear strength, τ, of the

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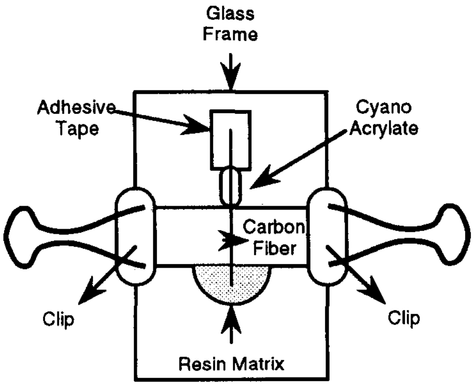

FIGURE 1 Diagram of a single fiber pull out specimen.

fiber-matrix interphase is, therefore, given by

$$\tau = F_{\max}/(\pi d l) \tag{9}$$

where d and l are the diameter and the embedded length of the fiber, respectively, and $F_{\max}$ is the maximum force corresponding to the pull-out process.[16,17]

RESULTS AND DISCUSSION

The thermodynamic model of adhesion is the most widely used approach in adhesion. This theory considers that the adhesive will adhere to the substrate because of interatomic and intermolecular forces established at the interphase, provided that intimate contact between both materials is achieved. The most common interfacial forces result from van der Waals (London, Debye and Keesom) and Lewis acid-base interactions. The magnitude of these forces is generally related to the surface free energies of both materials in contact.[18] Therefore, these surface free energies should be identified first. In order to determine the surface energies of carbon fibers, characterization was done by wetting experiments in which the Wilhelmy Balance Method was used. In this method, a solid that has vertical surfaces is partially immersed into a liquid; due to capillary effects, the liquid will either rise or be depressed along the vertical wall, thus exerting a force on the solid.[19]

The surface energy components of CF and the solid polymers were then calculated from Equations (6), (7) and (8). The calculated surface energy values are given in Table II. Untreated surfaces were found to have minimal levels of Lewis acidity. However, the surface acidity was increased progressively and significantly by the HNO_3 treatments. For instance, the first treatment in five minutes caused a rise from 0.7 mJ/m^2 to 2.3 mJ/m^2. Then it became 2.6 mJ/m^2 after 30 minutes of oxidation, and finally

TABLE II

Surface energy components of the carbon fibers and the polymer matrices (values are in mJ/m^2)

Sample	γ_i^+		γ_i^-		γ_i^{LW}	
			polymer matrices			
PS	0.2		0.0		41.4	
PMMA	0.0		5.8		40.7	
			commercial, sized carbon fibers			
IM7	6.2		0.0		31.0	
G40-700	4.6		0.0		31.4	
			activated Grafil carbon fibers			
Treat. time (mins.)	HNO_3	Anodic	HNO_3	Anodic	HNO_3	Anodic
0	0.7	0.7	19.9	19.9	35.4	35.4
5	2.3	3.9	0.0	0.0	39.9	39.7
30	2.6	5.0	0.0	0.0	40.4	40.2
60	3.6	4.1	0.0	0.0	42.2	41.3
120	2.4	2.3	0.0	0.0	43.5	43.3

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3.6 mJ/m^2 after one hour. The Lewis base character of the untreated fiber was reduced to nil by even the shortest treatment time with HNO_3. Untreated carbon fiber had a considerable LW component of 35.4 mJ/m^2, and the size of this constituent increased to 39.9 mJ/m^2 in five minutes, and then a gradual increase was observed up to 2 hours.

A similar tendency was noted for the anodically-oxidized fibers. The Lewis acid and LW nature of fibers were notably improved, while the Lewis base nature was reduced by even the shortest of the treatment times. The extremely large treatment durations of 60 and 120 minutes do not have practical use. At these excessive conditions, the deterioration and the etching of the carbon fiber surface is indicated by the drop of acidic character of surface energy components.

In the cases of both HNO_3 and electrochemical treatments, when the acid components were enlarged, the basic components were reduced. The decrease in basic condition is not so desirable because both factors are effective in enhancing the interaction between the matrix and fiber. On the other hand, resins such as bismaleimides have pre-dominantly basic surface.[20] The decrease in Lewis base character of the fiber is of very little consequence in composite formation with these resins. When both treatments are compared, the anodic oxidation is found to be the most effective in developing acidic surface character. In addition to the unsized carbon fibers investigated here, commercial sized carbon fibers were also tested. The tested commercial carbon fibers, IM7 and G40-700, were found to have a primarily acidic surface and a moderate amount of LW component as seen in Table II. The surface energies of PS and PMMA resins are also given in Table II where it can be observed that PS has a non-polar character and PMMA has a basic character. Thus, the latter will interact best with acidic CF surfaces.

When phases 1 and 2 contact each other to form an interphase, the following relationship exists with respect to the energy required to separate them (work of adhesion), W_a;

$$W_a = \gamma_1 + \gamma_2 - \gamma_{12} \tag{10}$$

According to van Oss and co-workers,[21-24] this work of adhesion of phase 1 and phase 2 is calculated as,

$$W_a = 2((\gamma_1^{LW}\gamma_2^{LW})^{1/2} + (\gamma_1^{+}\gamma_2^{-})^{1/2} + (\gamma_1^{-}\gamma_2^{+})^{1/2}) \tag{11}$$

The works of adhesion between carbon fibers and the matrix resins PS and PMMA were calculated by using Eq. (11) and are presented in Table III. Although these thermoplastic polymers are not very suitable for advanced composite manufacture, they symbolize two general classes of polymers. One is essentially non-polar and the other is basic in its surface behaviour.

When Table III is examined, it is observed that the work of adhesion for the CF/PS interface slowly increases from 80.6 mJ/m^2 to about 85 mJ/m^2 with the wet oxidation and this increase primarily is due to LW interactions. On the other hand, the adhesion of PMMA to CF was increased from 79.9 mJ/m^2 to 91.6 mJ/m^2, a large increase due to increasing acid-base interactions.

The work of adhesion values calculated do not take into account the effect of mechanical interlocking or the effect of dissipation during mechanical loading, therefore the work of adhesion can not be compared directly with ILSS values measured in a

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TABLE III
Work of adhesion values between CF and polymer matrices (values are in mJ/m^2)

Carbon Fiber	PS		PMMA	
	commercial, sized carbon fibers			
IM7	71.6		83.0	
G40-700	72.1		81.8	
	activated Grafil carbon fibers			
Treat. time (mins.)	HNO_3	Anodic	HNO_3	Anodic
0	80.6	80.6	79.9	79.9
5	81.3	81.1	87.9	89.9
30	81.8	81.6	88.9	91.7
60	83.6	82.7	92.0	91.8
120	84.9	84.7	91.6	91.3

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pull-out experiment. It is, nevertheless, possible to see the effects of activation on both work of adhesion values calculated from contact angle data and the measured ILSS values. They both display an increasing tendency with activation intensity.

The ILSS values observed for untreated fiber are 13.2 MPa for PS and 11.2 MPa for PMMA (Table IV). PS, having a non-polar surface, and PMMA, having a basic surface, interact with the untreated fiber through LW interactions only. The ILSS value stays constant within experimental accuracy which signifies the lack of acid-base contribution in the case of PS. On the other hand, by HNO_3 treatment, the acid-base interactions improve the adhesive strength of the PMMA-CF pair from 11.2 MPa to 14 MPa in five minutes and a twofold increase is observed in 60 mins treatment time. It reaches a three-fold increase in 120 mins where the failure occurs in the fiber itself. When compared qualitatively with the work of adhesion results (Table III), W_a also shows a similar trend. The failure at 120 mins can be explained by the damage to the fiber structure itself by the long oxidation period.

The ILSS of anodic oxidized carbon fibers are given in Table IV. An improvement in the ILSS values for both PS and PMMA is observed. This effect may be attributed to surface pitting which becomes dominant in the adhesion mechanisms by causing mechanical interlocking. Anodic oxidation may cause a more disordered surface than wet oxidation and can result in more powerful interlockings.

The effect of anodic oxidation is more significant than that of HNO_3 oxidation. The interlaminar shear strength was almost doubled for both CF/resin interfaces with 5 minutes of treatment. However, the severe etching of the fibers is suggested for long treatment times by the decreased tensile strength of the fibers. The fibers with long treatment periods failed during the pull-out measurements. When the adhesional strength of fiber to matrix is greater than the fiber's tensile strength the fiber breaks before the pull-out. These types of experiments are designated as "failure" in Table IV.

Sized carbon fibers, according to their surface behaviours, showed similar performance. IM7, having the most acidic surface, gives the largest ILSS value. What is

TABLE IV
ILSS values of carbon fiber-polymer matrix combinations (values are in MPa)

Carbon Fiber	PS		PMMA	
	commercial, sized carbon fibers			
IM7	12.9		22.0	
G40-700	26.5		15.0	
	activated Grafil carbon fibers			
Treat. time (mins.)	HNO_3	Anodic	HNO_3	Anodic
0	13.2	13.2	11.2	11.2
5	11.7	19.4	14.0	28.8
30	12.4	18.5	16.3	27.2 (failure)
60	14.5	22.0	22.9	29.5 (failure)
120	14.0	8.3 (failure)	37.4 (failure)	11.9 (failure)

surprising is the large ILSS for the G40-700-PS (26.5 MPa) couple. This result cannot be explained by surface interactions; therefore, physical interlocking is probably the cause of it.

In general, there is a good qualitative correlation between the calculated W_a values (from contact angle data) and the experimental ILSS results. An exact quantitative comparison is not possible because ILSS is a stress term with other physical effects that influence its value and W_a is an energy term.

CONCLUSIONS

Composite matrix resins are in general weak bases.[2] As such, acidic fiber surfaces are required to optimize this bonding mechanism. It is shown that the chemical and electrochemical treatments of carbon fibers create acidic surfaces that contribute significantly to the adhesion mechanism. Determination of LW, acid and base components of the carbon fiber surfaces and the matrix resin surfaces make possible the judicious choice of fiber-matrix combinations. The efficiency of surface activation techniques can be evaluated by the same approach. In this study, we have shown that there is a good correlation between the individual surface energy components and the adhesional strength of the CF to the matrix.

From the pull-out test results, it is understood that the fiber-matrix adhesion was made stronger in two ways. These are the development of intermolecular and interatomic interactions and of mechanical interlocking due to surface pitting that results from oxidation.

It is possible to investigate systematically the effect of other surface activation techniques on the fiber-matrix adhesion. Such studies can aim at specific purposes such as creating acidic and basic moities on fiber and matrix combinations through different techniques such as plasma coating. Ultimately the usefulness of the methodology developed here should be tested with macroscopic composite samples.

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