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Optimization of plasma treatment variables for the improvement of carbon fibres/epoxy composite performance by response surface methodology

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ABSTRACT

The purpose of the current work was to investigate the effect of oxygen plasma treatment of carbon fibre on the interfacial adhesion in carbon fibre/epoxy composites. The adhesion between the carbon fibre and matrix was examined by interlaminar shear strength (ILSS). It was observed that the composites prepared from plasma treated carbon fibre had higher ILSS compared with untreated fibres. Atomic force microscopy (AFM) and scanning electron microscopy (SEM) were employed to study the changes in the surface morphology of carbon fibres and the fracture of carbon fibre/epoxy composite before and after plasma treatment. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) analysis indicated that the plasma process created hydroxyl and carboxyl groups at the surface of carbon fibres. An experimental design with the identified process parameters namely, treatment time, power and flow rate of oxygen gas was followed. Experiments were performed according to the design in order to optimize the plasma variables by response surface methodology (RSM) using Box Behnken design. A second-order polynomial model was developed to predict the ILSS of carbon fibre composites in relation with the changes in plasma parameters. The process conditions were optimized with the quadratic model and the most efficient conditions were confirmed by the empirical results.

1. Introduction

Carbon fibres reinforced polymer composites have been widely utilized and studied due to their excellent properties, including high specific strength, modules, thermal and electrical conduction, corrosion resistance, good dimension and stability, etc. [1–3]. However, carbon fibres have certain drawbacks such as smooth and inert surface; low surface free energy and poor wettability. Therefore, carbon fibres have weak adhesion between their surface and the resin, which is an important factor for composite performance [2,4]. Thus, surface treatment of carbon fibres is decisively required to increase the surface functional groups and surface area for improved chemical bonding and physical interaction between the fibre and matrix, all leading to good stress transfer from the resin to the carbon fibres [5,6]. Various surface modification techniques have been developed such as electrochemical oxidation [7], wet chemical oxidation [8], ozone (O_3) treatment [9], γ-ray treatment [10], sizing [11], nanoparticle modification [12] and plasma treatment. Among these methods, treatment of carbon fibres by plasma has drawn increasing attention to itself, since it is environmentally friendly, economic, fast and adaptable, which does not significantly alter the bulk characteristics of materials [13,14]. Numerous works have been done to study the effect of plasma modification on carbon fibres [14–17]. Plasma modification effects are dependent on several variables such as treatment time, power, type and flow rate of gas, etc. So studying the impact of process parameters on the surface characteristics of carbon fibres is essential. In most studies, the effect of a single factor is examined while other factors are constant. This method may cause errors in result, because the interactions of the factors are not taken into account. This problem is solved by using response surface methodology (RSM). This method is a collection of statistical and mathematical techniques to design experiments, evaluate the effects of variables, develop models and

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finally finding the optimum parameters for the ideal response [18].

In this study, carbon fibres were treated by oxygen plasma and the effects of different variables of plasma modification on the mechanical properties of composite were investigated. Box-Behnken design was used to study the effect of the levels of the variables namely, treatment time, power and oxygen gas flow rate on interlaminar shear strength (ILSS) of the carbon fibre composite. An experimental model was developed for the plasma treatment process in order to obtain the exact evaluation of the optimum conditions for the output factors. The effect of plasma modification on the carbon fibre was evaluated by scanning electron microscopy (SEM), atomic force microscopy (AFM) and attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy.

2. Experimental

2.1. Materials

Carbon fibres used in the experiments were unsized and untreated polyacrylonitrile (PAN)-based type, with 3000 single filament per tow supplied by advanced fibre research center (Tehran, Iran).

EPON resin 828 (an undiluted clear bifunctional bisphenol A/epichlorohydrin derived liquid epoxy resin) and EPIKURE 3200 were obtained from Hexion, USA, and used as matrix and curing agent respectively.

2.2. Plasma treatment

The non-sized carbon fibres were treated in a low-pressure radio frequency plasma equipment (model: junior advanced, 13.56 MHz) manufactured by Europlasma, Belgium. The system was depleted to 100 mTorr, then oxygen was injected into the chamber subsequently; the chamber pressure was fixed at 100 mTorr and plasma was generated at different amounts of process variables, i.e. Treatment time, power and flow rate for each sample according to Table 1. Finally, air was injected into the chamber, and the modified carbon fibres were removed.

2.3. Design of experiments

The effect of different combinations of input variables namely, treatment time, power and flow rate of oxygen gas on ILSS of modified carbon fibre composites were investigated at three levels (low, medium and high) using Box Behnken design. The actual values of the input parameters corresponding to the coded levels are indicated in Table 1. The design includes 17 runs of different conditions. The test on the center point is repeated five times to evaluate the pure error. The runs were carried out randomly in order to reduce the effect of irrelevant factors on the response. The variables and their levels were chosen by the preliminary screening test, the restrictions of the plasma reactor and according to previous reports [4,13,19].

Table 1
Variables and levels used in the experiments.

	Levels		
	Low	Medium	High
Factors	−1	0	+1
Treatment time, min (X_1)	1	3	5
Power, W (X_2)	50	125	200
Flow rate, $\frac{cm^3}{min}$ (X_3)	20	60	100

2.4. Statistical analyses

The following second-order polynomial model was employed to describe the relationship between the response values (ILSS) and the independent process factors:

$$\widehat{Y} = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{11}X_1^2 + B_{22}X_2^2 + B_{33}X_3^2 + B_{12}X_1X_2 + B_{23}X_2X_3 + B_{13}X_1X_3\varepsilon + \varepsilon \quad (1)$$

Where $\widehat{Y}$ is the predicted response; X_1, X_2 and X_3 are plasma process variables; B_0 is a constant coefficient; B_1, B_2 and B_3 are linear coefficients; B_{11}, B_{22} and B_{33} are quadratic coefficients; B_{12}, B_{23} and B_{13} are interaction coefficients and ε is the error of the model. Analysis of variance (ANOVA) was employed for the statistical analysis of the results. In addition, the coefficient of regression (R^2) was determined to check the model adequacy. Statistical significance was judged by F-test and if the probability (P) value less than 0.05 were taken as the levels of significance. The design of experiments and data analysis was performed using Design Expert software (version 8.0.7.1).

2.5. Composite preparation

The matrix resin was prepared by mixing epon828 epoxy resin and curing agent with the weight ratio of 10:2. Untreated and plasma treated carbon fibres tows were impregnated in the matrix and subsequently were placed into a special mold. Finally, the samples were solidified for 16 h at 25 °C then cured for 2 h at 100 °C.

2.6. Evaluation of interlaminar shear strength (ILSS)

To evaluate the interlaminar adhesion between carbon fibres and matrix resin the ILSS of composite was tested. To measure ILSS, Short beam bending test was conducted using Instron 5566 (USA) according to ASTM D2344. Briefly, the cross head speed was 1 mm/min; the span-to-depth ratio was 5:1, and the width and the thickness of the specimens were 6 mm and 2 mm, respectively. At least five samples were measured successfully to obtain the average values. ILSS amount, Γ, was calculated according to the following equation:

$$\mathrm{ILSS} = \frac{3\ P_b}{4b \times d} \quad (2)$$

where, P_b is the maximum force during test, b and d are the width and thickness amounts of the specimen.

2.7. ATR-FTIR analysis

In order to study the possible changes in functional groups of the surface of carbon fibres by plasma modification, ATR-FTIR analysis was done on a Nicolet 670 spectrometer. An average of 40 scans was recorded for each sample with a resolution of 4 cm^{-1}.

2.8. Scanning electron microscopy (SEM)

The surface of carbon fibres and fracture morphology of carbon fibre/epoxy composite were studied using an AIS 2100 SEM (Seron technology, South Korea).

2.9. Atomic force microscopy (AFM)

AFM (Dimension-3100, Veeco instruments, USA) was adopted for the analysis of the surface topology of untreated and plasma