INTERFACE OPTIMISATION OF RECYCLED CARBON FIBRE COMPOSITES

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Abstract
A range of plasma treatments of recycled carbon fibre were carried out to improve the interface in the resulting composites. Tensile tests were performed on unidirectional composites 10° off-axis to the fibre alignment to better facilitate an interfacial response. It was found that under certain treatment conditions, tensile strength in these samples was improved by over 25% when compared to the untreated controls. However some treatment conditions led to a reduction in strength of over 10%, thus demonstrating that optimal treatment parameters exist for this system with respect to the tensile strength. The modified surfaces were analysed by XPS, which showed a good correlation between surface functionality and composite performance.

1 Introduction
1.1 Composites recycling
In the current climate, as the demand for more renewable resources increases, technologies have been developed to recycle carbon fibre from end of life composites [1], so it can be re-used in other applications. The demand for these materials is driven by a greater social awareness of environmental issues. The composites industry wishes to exploit this and, potentially, appease national government and international unions in meeting "green" targets. The carbon fibre used in this work was recycled using pyrolyis. Pyrolysis is defined as "chemical decomposition occurring as a result of high temperature" [2]. More specifically it is a thermally initiated chemical process that decomposes organic matter in an inert atmosphere [3]. The recyclate is in the form of short, single filaments [Figure 1]. In its pure form it is suitable for use in bulk moulding compounds (BMCs) as the fluffy fibre can be blended with the resin in the same way that virgin fibre is used. It can also be used in compounding with thermo-plastics prior to injection moulding [4]. Non-woven veils can be produced from the recyclate in a wet processing method similar to paper making. The industrial partner in this work (Technical Fibre Products Ltd.) processed the material in such a way as to manufacture a non-woven veil that has a degree of fibre alignment. However the nature of the process means that fibre alignment decreases with increasing veil density. Such non-woven veils are applicable for SMC manufacture and also for wet lay-up (as in this work) and as a starting material for pre-impregnation with resin.

Figure 1. Form of the recyclate after pyrolysis of waste composite

1.2 Aims and objectives

The main objective of the project was to manufacture composite materials from this recycled carbon fibre in order to assess their potential as industrial materials. The secondary objective is to enhance the materials' mechanical properties by optimisaton of the interface region. The relationship between interface quality and composite performance has been demonstrated by Drzal and Madkudar 1993 [5]. The sensitivity of composite performance to interface properties lends us to the possibility of enhancing their performance by understanding the chemistry at this region, and so manipulating it for an improvement in mechanical properties.

1.3 Plasma treatment

A plasma is a partially or fully ionized gas. Plasma processes have a wide variety of applications in industry, though their use as promoting adhesion in composite interfaces is relatively new. Plasma treatment has been shown to improve the mechanical properties of carbon fibre composites previously [6]. These techniques are important, as carbon fibre surfaces are chemically inert; it is difficult to obtain a strong bond at the interface of fibre and matrix.

2. Experimental Procedure

2.1 Carbon fibre

The non-woven carbon veil used in this work has a basis weight of 10 gm^{-2}. Consequently they are very thin compared to conventional woven carbon fabrics, which are of the order of 200 gm^{-2}. However this is deliberately so, as it is possible to achieve a higher packing density with a thinner veil. Due to the nature of the manufacturing process, a thicker (higher basis weight) veil will have a lower density.

2.2 Composite manufacture and specimen preparation

Both treated and untreated samples were manufactured by wet lay-up. Two veils measuring 120 x 70 mm were cut at 0° to the fibre alignment axis prior to treatment. Gas plasma treatments were carried out at low power A, and at increasingly higher powers B, C, and D up to the highest power E. They were then laid up with an epoxy-based resin system and cured in an envelope vacuum bag. Once cured, each panel was cut at 10° to the fibre alignment axis

into tensile test strips of dimensions 100 mm x 5 mm. Fibreglass/polyester composite end tabs measuring 15 mm x 15 mm were affixed and the samples were tested in tension. 5 specimens each of untreated and plasma treated at powers A-E were tested. Measured variables were tensile strength, Young's modulus and strain to failure.

3. Results and Discussion

3.1 Tensile testing

The data is summarised in Table 1 below. Firstly, the mean and standard deviation in each data set was calculated. The mean values were first normalised for fibre volume fraction (V_f). For example, mean tensile strength values in MPa were divided by V_f in % to give a normalised value. V_f of the samples ranged from 10-13%. It must be noted that due to the potential Intellectual Property (I.P.) associated with this system, all figures are quoted relative to the untreated control data, which are assigned a value of 100. This was done by scaling the normalised data up to a value of 100 for the untreated samples, and scaling the treated sample data accordingly so that the relationship between the data is the same as for the raw data normalised for V_f. The same process was applied to the standard deviation values.

Sample	Tensile Strength	Young's Modulus	Strain
Untreated	100 +/- 7.3	100 +/- 10.6	100 +/- 9.7
Power A	110 +/- 10.1	106 +/- 11.8	106 +/- 12.8
Power B	128 +/- 11.1	113 +/- 7.8	111 +/- 6.4
Power C	95 +/- 7.6	88 +/- 7.2	89 +/- 8.9
Power D	87 +/- 10.9	94 +/- 16	94 +/- 10.5
Power E	88 +/- 7.7	116 +/- 11.9	75 +/- 4.7

Table 1. Normalised 10° off-axis tensile test data N.B. (+/-) figures indicate one standard deviation from the mean

It can be seen from Table 1 that there is a gradual improvement in each mechanical property with treatment up to Power B. However from Power C onwards there is a steady decline, with a notable exception that the highest value of Young's modulus is after treatment at the highest power E. The data for tensile strength is presented graphically in Figure 2. Error bars indicate one standard deviation either side of the mean, representing the (+/-) values in Table 1.

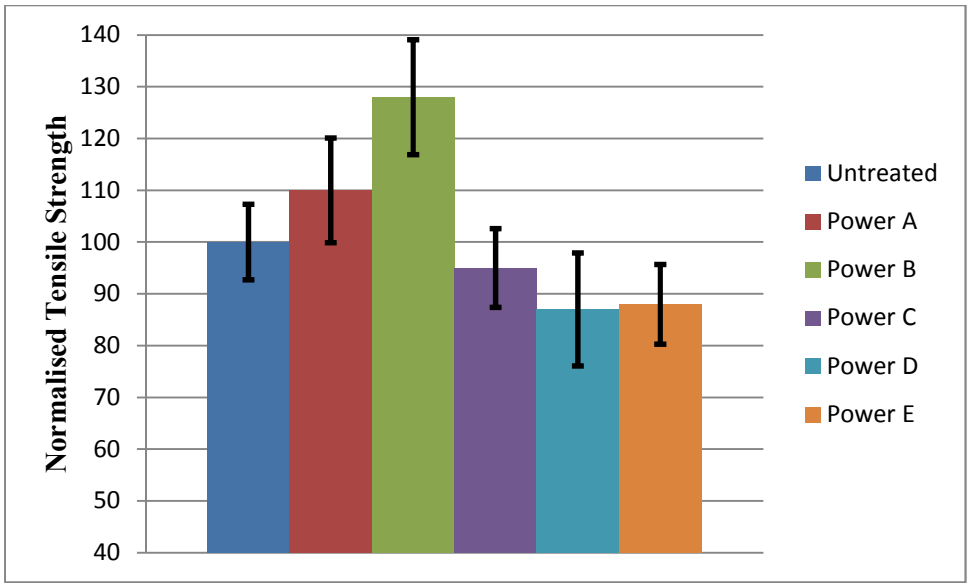

Figure 2. Normalised 10° off-axis tensile strength results comparing untreated controls to samples gas plasma treated at various powers.

It can be seen from Figure 2 that the optimal plasma treatment with respect to tensile strength is Power B. From Power C upwards the fibres have been over-treated resulting in strength less than that of the untreated control. Strength changes range from an increase of 28% for treatment at Power B to a reduction of 13% for Power D. This trend can partly be explained by looking at the failure strain data [Figure 3].

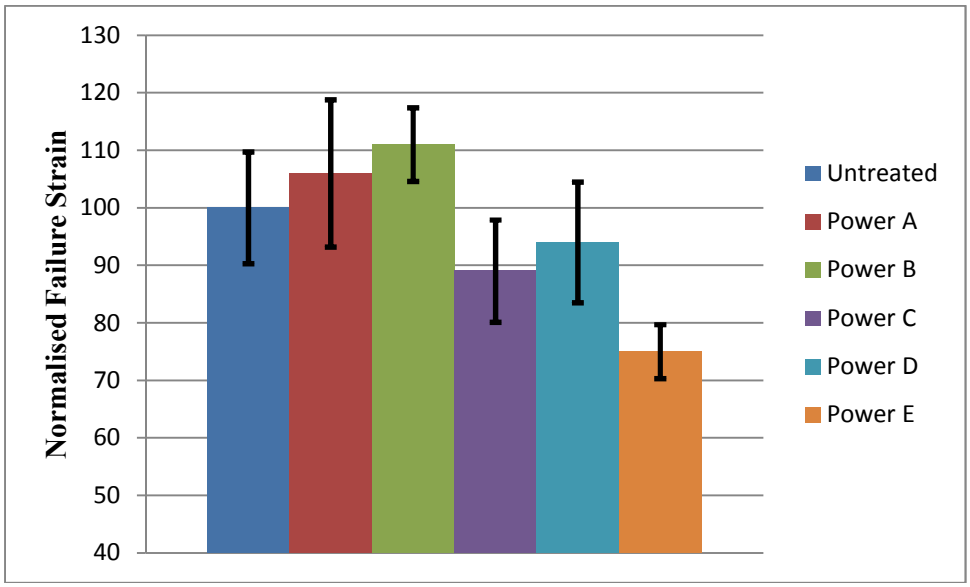

Figure 3. Normalised 10° off-axis failure strain results comparing untreated controls to samples gas plasma treated at various powers.

Although the pattern in the data is less obvious than that for strength, it can still be seen that samples treated at Powers C, D and E have a significantly lower failure strain than those treated at Powers A and B and the untreated control. This correlates well their lower tensile strength values and is indicative of an overly strong interface.

3.2 Surface analysis

Treated and untreated surfaces were analysed by XPS but due to I.P. the speciated narrow scan data cannot be disclosed. The oxygen:carbon ratios from the survey scans are detailed in Table 2 below.

Sample/Composition	O/C ratio
Untreated	0.32
Power A	0.41
Power B	0.41
Power C/D	0.31

Table 2. O/C ratios on the surface of untreated and treated samples analysed by XPS

The results of this analysis showed higher O/C ratios in the samples treated at Powers A and B. Also at higher powers this ratio was the same as for the untreated controls. This correlates well with the mechanical test data. Yet the strength and failure strain of the over-treated samples was much lower than that of the untreated controls. The surface chemistry can be explained by a complex mechanism of desorption of process aids and oxidation.

4. Conclusions

Improvements in 10° off-axis tensile strength were achieved in the material using plasma treatment. An increase in O/C ratio (oxygen functionality) was responsible for this, though other mechanisms such as surface roughening may have played a role. The highest improvement in strength was achieved at an intermediate plasma power, possibly indicating an optimal level of interfacial adhesion for tensile strength. Over-treatment at higher powers led to a reduction in tensile strength, probably due to fibre damage as the O/C ratio was the same as for the untreated controls.

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