

INTERFACIAL EFFECTS IN ULTRA-HIGH MODULUS PITCH-BASED CARBON FIBERS/POLYCARBONATE COMPOSITES

M. C. Paiva*, M. Nardin**, C. A. Bernardo* and J. Schultz**

*Departamento de Engenharia de Polímeros, Universidade do Minho
Azurém, 4810 Guimarães, Portugal

**Centre de Recherches sur la Physico-Chimie des Surfaces Solides CNRS
24, Avenue du Président Kennedy, 68200 Mulhouse, France

1. Introduction

The composite systems under study are formed by high and ultra-high modulus pitch-based carbon fibers, P75S and P120J, from Amoco Performance Products, and a polycarbonate matrix, Makrolon 2805 (Bayer). The P120 fibers, initially unsized and untreated, were submitted to different oxygen plasma treatments. The fiber surface was analysed by scanning electron microscopy (SEM), and by X-ray photoelectron spectroscopy (XPS). The mechanical characteristics were assessed by single filament tensile testing. Monofilament composites were prepared by compression moulding, and the interfacial stress transfer ability was evaluated using the fragmentation technique [1,2]. Polarised light microscopy observations of the compression moulded, monofilament composites showed the presence of a regular stress pattern along the fiber-matrix interface, bright spots being observed at fixed distances, for each type of fiber. Preliminary results indicate that there is a correlation between these distances and the measured interfacial stress transfer characteristics. The present work was undertaken to clarify this effect.

2. Experimental Part

Fiber Characterisation

P75S are commercial carbon fibers, surface treated and sized, with an elasticity modulus of about 520 GPa [3]. P120J fibers were unsized and untreated, and characterised by a modulus of about 830 GPa [3]. The latter were submitted to oxygen plasma treatments, with different powers and times of application, at 0.1 MPa of oxygen, as follows:

-75W, for 3 and 10 minutes

-100W, for 3 minutes

-150W, for 3 and 10 minutes

SEM observations showed that the surfaces submitted to power treatments higher than 75W were clearly damaged. The XPS measurements of the fibers surface were carried out on a ESCALAB 200A-VG Scientific, with a Mg/Al double X-ray source, and a vacuum limit of 10^{-8} Pa. The results obtained for the global atomic

percentage of oxygen vary with plasma treatment, from 2.7% for the untreated P120J fibers, to 8-11% for the treated fibers. The P75S fibers, treated and sized, have a higher content in oxygen, 19%, and about 3% nitrogen.

The fibers were tensile tested using a method described in the literature [4], based on the ASTM standard [5]. To account for the marked dependence of tensile strength on gauge length (g.l.), the tests were performed for at least 3 different g. l.. The variation of tensile strength (σ) with g.l., for the P120J and P75S fibers is shown in fig. 1 and 2. It is usually considered that the tensile strength data can be fitted to a single two parameter Weibull distribution for all g. l.. Although it was shown that this procedure is possibly too general [4], it has been used successfully in many cases and led to interesting conclusions. For instance, El M. Asloun et al. [6] concluded that the logarithm of σ_w (the average value of σ from the Weibull distribution), plotted against the logarithm of g.l. should be linear, providing a practical way of estimating σ at any g. l.. In the present work, it was found that the plasma treatment does not affect significantly the tensile strength (see fig. 1). In fact, to a reasonable approximation, it is possible to fit a linear regression to the overall experimental results, leading to:

$$\ln \sigma = -\frac{1}{5.40} \ln(\text{g.l.}) + 1.13 \quad (1)$$

Identically, for the P75S fibers:

$$\ln \sigma = -\frac{1}{6.30} \ln(\text{g.l.}) + 1.01 \quad (2)$$

Fragmentation Tests

The tensile testing of the monofilament composites was performed on a Instron 4505 tensile testing machine, at a speed of 0.5 mm/min. The fragment lengths, l , were measured by optical microscopy. The critical fiber length, l_c , was obtained from $l = (\frac{3}{4})l_c$ [7]. The interfacial shear strength, τ , was calculated according to the Fraser-Di Benedetto model [1,2], using equation (1) to estimate the tensile strength at l_c , for the treated and untreated P120J fibers. The results are presented in table 1.

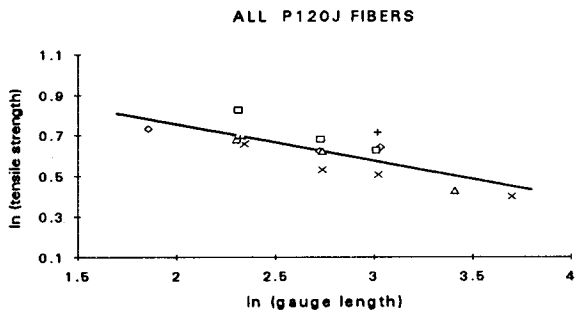


Fig. 1. $\ln(\sigma)$ vs. $\ln(g.l.)$ for P120J (treated and untreated).

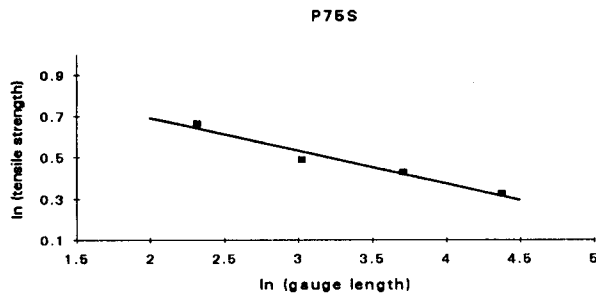


Fig. 2. $\ln(\sigma)$ vs. $\ln(g.l.)$ for P75S fibers.

Interfacial Stress Concentration Pattern

The monofilament composites were observed under the polarised light microscope, before the fragmentation tests were performed. A regular stress pattern along the fiber-matrix interface was observed, as shown in figure 3. This pattern was quantified by the fixed distance between the bright spots, l_b , also indicated in table 1. l_b is clearly dependent on the fiber surface treatment, and correlates well with the critical fiber length, l_c , as shown in table 1.

Table 1. Interfacial shear strength results from the fragmentation test and polarised light observations.

Sample	l_c (μm)	τ (MPa)	l_b (μm)	l_c/l_b
P120J	1353	10.3	130	10
75W/3min	509	33.0	65	8
75W/10min	652	23.2	95	7
100W/3min	712	21.6	68	10
150W/3min	652	22.9	57	11
150W/10m	605	22.8	83	7
P75S	361	48.8	33	11

To clarify this effect further, the composites were tensile tested under the polarised light microscope, and the evolution of colour was monitored. When the direction of the applied force was perpendicular to the fiber axis, the colour change at the stress concentration points indicated an increase of the optical path difference (OPD), according to the Michel Lévy Colour Chart. The OPD monitored in the remaining zones of the interface also

increased, but starting from a much lower (near zero) value. This was taken as evidence for the non-homogeneity of the stress distribution along the interfacial region. It can also be concluded that the forces acting there are mainly directed towards the matrix and away from the fiber (positive tension). This is confirmed by the disappearance of the localised bright spots when the tensile test is performed in the direction of the fiber axis.

3. Conclusions

In the present work evidence has been found to support the view that, in pitch-based carbon fibers/polycarbonate monofilament composites, the stress distribution at the fiber-matrix interface is non-homogeneous. Stress concentration points are observed at regular intervals, whose distance correlates well with the interfacial parameters l_c and τ .

4. Acknowledgements

The authors are indebted to Dr. Ana Cuesta, from Instituto Nacional del Carbon (CSIC), Oviedo, for performing the plasma treatments, and to Dr. Jovita Oliveira, of the Dep. Polymer Eng., Universidade do Minho, for the support on the optical microscopy work.

References

1. W.A. Fraser, F.H. Ancker, A.T. Di Benedetto, 30th Ann. Techn. Conf., Reinf. Plastic/Composites Struct., Soc. Plastic Ind., 22-A, 1 (1975).
2. A.T. Di Benedetto and L. Nicolais, *Plast.* 10, 83(1979).
3. Amoco Product Information.
4. E.G. Stoner, *The Effect of Shape on the Tensile Strength of Pitch-Based Carbon Fibers*, PhD. Thesis, Clemson University, December 1991.
5. ASTM Standard, D3379-75 (Reapproved 1989).
6. El M. Asloun, J.B. Donnet, G. Guilpain, M. Nardin, J. Schultz, *J. Mater. Sci.*, 24(1989) 3504.
7. A. Kelly, W.R. Tyson, *J. Mech. Phys. Solids*, 13, 329 (1965)

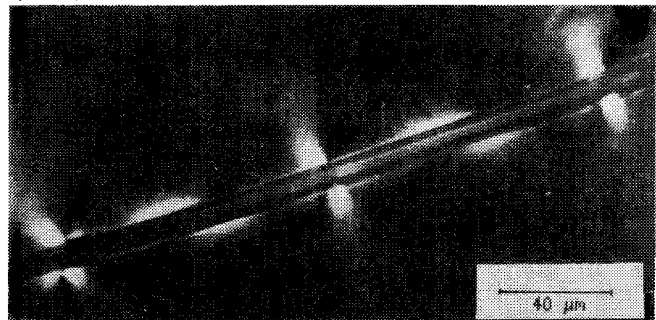


Fig. 3. Stress pattern along the fiber-matrix interface (observed by polarised light microscopy).