SURFACE TREATMENTS FOR IMPROVING THE PROPERTIES OF VAPOR-GROWN CARBON FIBER/POLYPROPYLENE COMPOSITES

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Introduction

Vapor-grown carbon fibers (VGCFs) are grown catalytically from gaseous hydrocarbons using metallic (typically iron) particles [1]. Good adhesion between these fibers and the matrix is essential for the performance of any composite material. Thus the mechanical properties of composites are strongly influenced by fiber surface morphology and chemistry [2]. In this report the fiber-matrix adhesion problem was addressed by surface treating our VGCFs using a variety of methods.

Experimental

The fibers used in this study were produced at the pilot plant of Applied Science, Inc., (ASI), in Cedarville, OH. They were approximately 200 nm in diameter and were formed by iron-based catalyst particles in a methane atmosphere [3]. The following fiber designations refer to differing reactor gas mixtures used in production:

- In increasing order of gas space velocity: Clean, PR-18, PR-5, Best Shot.
- Coal-based fibers utilizing a mixture of ground coal to supplement the natural gas and hydrogen sulfide.
- Acetylene-based fibers.

Other surface treatments used heat and oxidation:

- Graphitized fibers heated to 3000°C for 1 hour.
- Air-Etched fibers oxidized in air at 450°C .
- CO_2 oxidized fibers oxidized with CO_2 in a tube furnace at temperatures from 850 to 950°C for from 15 minutes to 2 hours.
- Chemical treatments with epoxy (EL, EH) and a diamine salt (DL, DH).

Each of these materials was ball milled to improve resin permeation for 2 minutes using a Spex 8000 mixer mill and injection molded in a benchtop CSI MiniMAX Molder using a cup temperature of 230°C. The mold was held at room temperature (23°C) for polypropylene (PP) resin to inhibit crystallization. The resulting specimens were mounted in the grips of an MTI tensile testing machine and were stretched at 1 mm/min until failure occurred.

Results and Discussion

Figure 1 shows the tensile strength and stiffness for PYROGRAF/polypropylene composites using reinforcing fibers produced by several methods and having the various surface treatments described in the previous section. These data show that it is possible to triple both the modulus and strength of the polypropylene resin (open circle) by adding only 15 volume % VGCF. They also underscore the fact that some fiber production methods and surface treatments produce composites with much better properties than others.

Figure 2 shows a relationship between the gas phase residence time of the feedstock mixture used to grow several varieties of fibers and the tensile strength (top graph) and modulus (bottom graph) of a 15 vol% composite fabricated from the fibers. Fibers grown under higher flow conditions tend to be more graphitic; with their shorter residence times they produce composites with poorer mechanical properties.

A systematic examination of the tensile strengths of composites made from fibers with differing surface oxidizing treatments is presented in Figure 3. The abscissa in Figure 3 represents the total surface energy (mJ/g) of each type of fiber; it is determined by independent measurements of the external surface area of the treated fibers (m^2/g) times the surface energy of the fibers (mJ/m^2). It is evident from Figure 3 that a modest amount of oxidation of the surface increases the tensile strength of the composite, while too much etching can decrease it. The optimum etch was carried out in a tube furnace using CO_2 at 850°C for 15 min at a 9.2 l/min flow rate.

Conclusions

- Less highly graphitized fibers adhere better to the polypropylene matrix than more highly graphitized fibers.
- Fiber/matrix adhesion may be improved by moderately oxidizing the fibers either in air or CO_2. This oxidation seems to become more effective as it increases the product of the external surface area and the surface energy of the fibers; however, excessive etching can be destructive.

Acknowledgment

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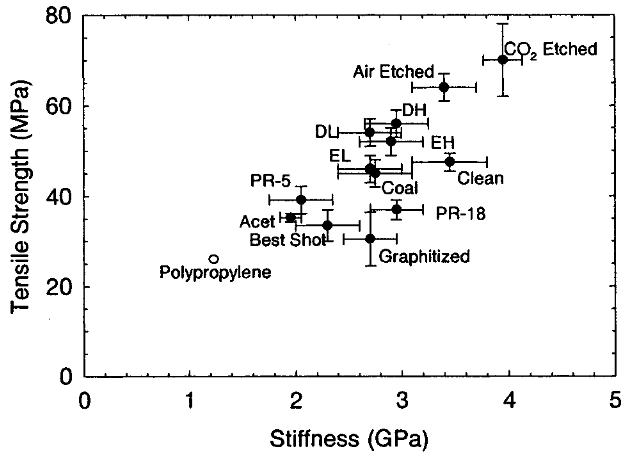

Figure 1. Tensile strength versus modulus for 15 vol% composites using different types of VGCF in polypropylene. The open circle shows the properties of PP.

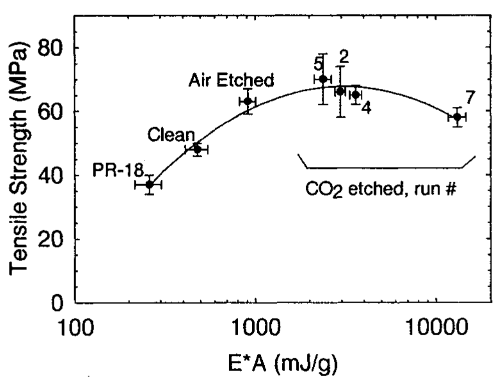

Figure 3. .Tensile strengths of various 15 vol% VGCF composites as a function of total surface energy x external surface area.

References

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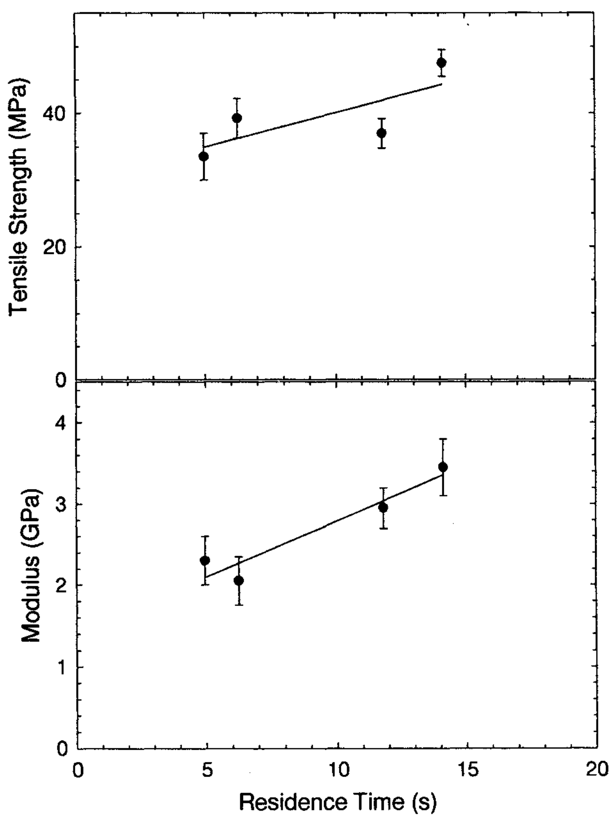

Figure 2. Tensile strength and modulus of various 15 vol% VGCF composites produced with different gaseous feedstock residence times.