INFLUENCE OF FIBER SURFACE TREATMENTS ON LOW-COST CARBON FIBER COMPOSITES

Ahmad Vakili, Zhongren Yue and Matthew P. Duran

The University of Tennessee Space Institute, 411 B.H. Goethert Pakway, Tullahoma, TN 37388

Introduction

Carbon fiber (CF) surface treatment is a key process for producing high performance composites because it is capable of improving the wetting characteristics of CFs by increasing surface functional groups and surface area [1]. The investigation of the CF surface treatments and the characterization of surface structures of CFs from microscopic to nano-scales, therefore, are very important in understanding the properties of fiber products and processing related issues to improve the fiber surface properties and the performance of their composites.

Potential low-cost carbon fiber composites (CFCs) with appropriate mechanical properties have been fabricated at the University of Tennessee Space Institute (UTSI) by using low-cost continuous roving-like mesophase pitch-based CFs. However, the efficient translation of the good mechanical properties of CFs into composites has not been achieved. The SEM fracture analysis diagram of the composites showed poor bonding between the CFs and the epoxy resin. To address these problems, fiber surface modification is required to improve mechanical properties of the resultant UTSI CFCs.

Industries and researchers have employed several CF surface treatment methods including liquid phase oxidation, electrochemical oxidation, gaseous oxidation, plasmas, whiskerization, polymer grafting, and coating to modify CF surfaces [1-2]. In this study, we will focus on gaseous oxidation, because it has several advantages over the other methods for roving-like UTSI CFs: 1)ease of operation and control in product line; 2) no liquid chemicals required; 3) no washing and drying processes; and in particular 4) potentially uniform reaction between gas and the UTSI CF due to its roving-like formation.

The goals of this work are to investigate how gaseous (air, CO_2/H_2O, O_3) oxidation and sizing strengthen the bonding between the UTSI CF and epoxy resin. Chemical absorption and single-fiber fragmentation tests are used to characterize the change in surface properties.

Experimental

Materials

UTSI mesophase pitch-based carbon fibers produced in the UTSI spin lab. Epoxy resin 105 and extra slow hardener 209 was obtained from West System.

Air and CO_2 were from compressed cylinders. O_3 was generated by an Air – Zone XT-6000 ozone air purifier. As comparison, HNO_3 with 69% assay from Fisher Scientific was also employed for fiber surface treatment.

Apparatus and Procedures

As-received UTSI CFs were oxidized in a tube furnace. The treatment temperatures and time are 400°C and 60 min for air; 150°C and 30 min for O_3; and 800°C and 30 min for CO_2/H_2O oxidation. HNO_3 oxidation was done at 100°C for 30 min, then fully washed and dried.

Sizing was carried out by impregnating CFs in a 5 wt % of epoxy/acetone solution, and then drying at 120°C.

Single-fiber diameter and tensile strength were measured with a Diastron Limited FDAS765 fiber analyzer.

Single-fiber fragmentation tests were performed by preparing and polishing "dog bond" samples, then stretching and observing under an optical microscope with a polarized light. The details can be seen from the reference [3-4].

Adsorption of methylene blue (MB) was measured with a UV-visible spectrophotometer at 660 nm. MB surface area was calculated from the adsorbed MB. NaOH uptake onto the CFs was measured with a pH meter.

Composites were fabricated from the fibers and the epoxy resin using a vacuum bagging resin infusion technique. The details are described in another paper.

The flexural properties of the CFCs were tested in a MTS machine with a 550 kN load cell and a head speed of 0.1 inch per minute. A precision extensometer was used to measure strain. ASTM D 6272 standard was used as a guide for the testing and calculations.

Apparent density and electrical resistivity of CFCs were calculated by the measurements of mass, dimensions and electrical resistance with a balance, a calibrator, and an electrical bridge, respectively.

Results and Discussion

Table 1 lists the results of fiber fragmentation tests on UTSI CFs with or without surface treatments. As expected, all the surface treatments (air, O_3, CO_2/H_2O, and HNO_3) increase the interfacial shear strength (IFSS) between carbon fiber and epoxy resin. CO_2/H_2O treated CF shows much smaller critical length and higher IFSS. The fiber fragmentation tests show an obvious difference between CO_2/H_2O treated CF with other fibers (see Fig.1). This treatment decreases the fiber tensile strength and also requires a temperature higher than 800°C. Air treated CF also shows an improvement in IFSS, but it also requires reaction temperatures higher than 400°C.

Only O_3 and HNO_3 treatments can be operated at relatively low temperatures. Table 2 shows that after surface treatments, acidic functional groups and surface area increased.

Table 1 Effects of surface treatments on the IFSS

Fiber Type	Fiber Dia. (μm)	Tensile Strength (Mpa)	Average length (μm)	Critical Length (μm)	IFSS (Mpa)
UTSI CF1	12.46	465	491	655	4.4
Air treated CF1	12.63	649	446	595	7.0
UTSI CF2	19.95	723	724	965	7.4
O_3 treated CF2	19.15	706	450	600	11.3
CO_2/H_2O treated CF2	20.65	641	<200	<267	>24.8
HNO_3 treated CF2	19.05	884	596	795	10.6

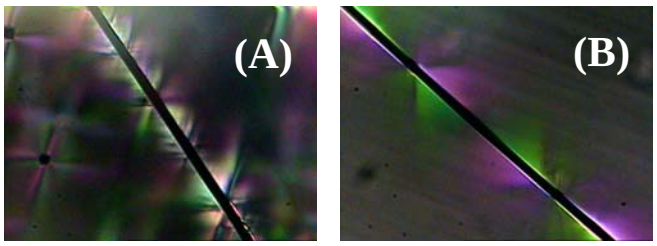

Fig.1 Typical fragmental images of CFs. (A): CO_2/H_2O treated CF; (B): Other treated or untreated CFs.

Table 2 Adsorption of MB and NaOH uptake on the CFs

Sample	MB adsorption		NaOH uptake (mmol/g)
	Adsorbed MB (mg/g)	Surface area (m^2/g)	
UTSI CF2	0.29	0.61	0.037
O_3 treated CF2	0.85	1.77	0.062
CO_2/H_2O treated CF2	1.20	2.51	0.038
HNO_3 treated CF2	0.74	1.55	0.055

Fig.2 compares the physical properties of CFCs fabricated with or without surface treated CFs. Surface treated CF composites (B and C) show higher flexural strength, modulus, and E-resistivity than the as-received CF composite (A). O_3 treated CF (C) is better than HNO_3 treated CF (B) for making high performance CFCs.

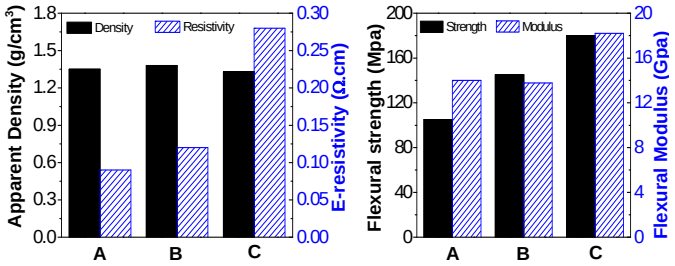

Fig. 2 Physical properties of CFCs. A: UTSI CF; B: HNO_3 treated CF; C: O_3 treated CF.

Fig. 3 shows more fiber fragments in sized CF (top) than in unsized CF (bottom) indicating a good bonding between sized fiber surface and matrix. Less fiber fragments and appearance of debonding zone at the fiber break (bottom) indicate a weak bonding between unsized CF and matrix. These results combining with the property testing of the CFCs in Table 3 suggest that the increase in wettability of the sized CF surface results in a good bonding and an improvement in mechanical properties of the CFC.

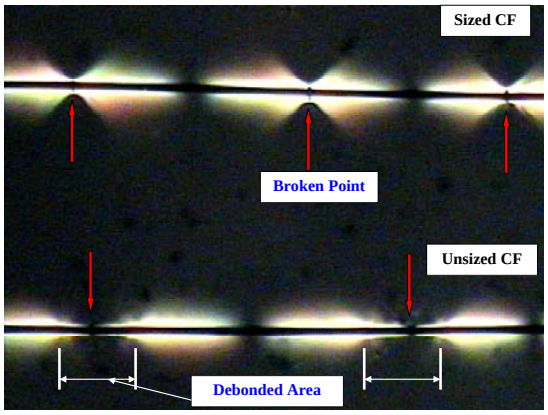

Fig. 3 Fragmentation tests comparing sized CF with un-sized CF in epoxy resin.

Table 3 Effect of sizing on physical properties of CFCs

	Apparent density (g/cm^3)	E-resistivity (Ω*cm)	Flexural Strength (Mpa)	Flexural Modulus (Gpa)
Un-sized CF	1.313	0.050	108	14.7
Sized CF	1.352	0.051	158	17.9

Conclusion

Surface treatments with gases (air, CO_2/H_2O and O_3) can improve the surface properties of the UTSI low-cost CFs by introducing more functional groups and increasing surface area. O_3 oxidation was proven to be an optimal process for such CFs. Sizing with 5 wt% of epoxy/acetone solution also greatly improves the wettability of CF surface, thus increases the mechanical properties of the CFCs.

References

1. Morgan,P. Carbon Fibers and their Composites. CRC Press Taylor & Francis Group, 2005
2. Chung, D.L. Carbon Fiber Composites. Butterworth-Heinemann, Newton, MA, 1994
3. Feih, S., Wonsyld, K. Minzari, D., Westermann, P., and Lilholt, H. Testing Procedure for the Single Fiber Fragmentation Test. Rise National Laboratory, December 2004.
4. Hatta, H., Goto, K., Aoki, T. Strengths of c/c composites under tensile, shear, and compressive loading: Role of interfacial shear strength. *Composites Science and Technology*. **65** (2005) 2550-2562.