MECHANICAL PROPERTIES / FIBER SURFACE CHEMISTRY AND MORPHOLOGY RELATIONSHIP IN OXYGEN PLASMA TREATED UHMW POLYETHYLENE / EPOXY COMPOSITES

Moutushi Dey,[a] Denis Kissounko, [a] Joseph M. Deitzel, Johw. W. Gillespie, Jr. [a] Jacqueline H. Yim,[b] Guiseppe R. Palmese,[b] D. Daphne Pappas[c]

University of Delaware . Center for Composite Materials . b Drexel University . c ARL

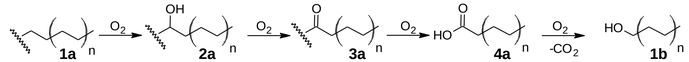
Introduction and Objectives

UHMW polyethylene was oxygen plasma treated under ambient pressure and temperature. Variations in the plasma exposure time at a constant flow rate of 1LPM lead to the surface morphology change, which reflects on mechanical properties of a corresponding composite.

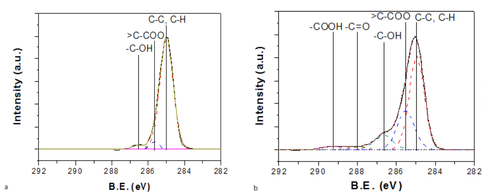
Objectives:

- Correlate the Interfacial Shear Strength (IFSS) and energy absorption of polyethylene-epoxy microcomposite with plasma exposure time.
- Investigate the surface morphology of untreated and plasma treated polyethylene fibers by AFM technique and correlate surface morphology change to interfacial properties of the polyethylene-epoxy composite.

Surface XPS studies



Surface roughening occurs

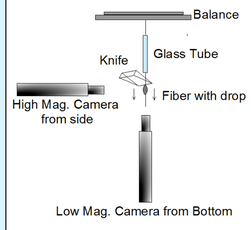


Representative XPS spectra for a) untreated, and b) 2 minutes plasma treated Spectra filaments.

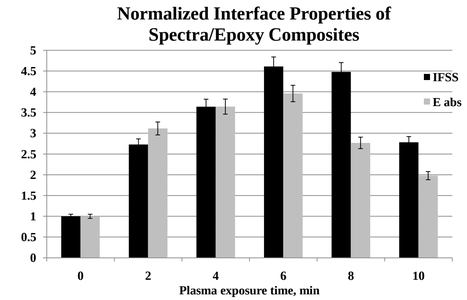
XPS data for the oxygen plasma treated Spectra fibers.

Plasma exposure time, min	[-OH], a.u.	[=C=O], a.u.	[-COOH], a.u.
0	4.20±0.69	0.0±0	0.0±0
2	16.38±5.59	9.33±5.85	3.28±1.64
4	16.62±6.28	8.37±5.69	4.05±4.48
6	13.81±4.55	3.62±1.18	2.90±1.65
8	15.22±2.86	5.05±0.98	6.17±1.50
10	13.50±1.51	10.94±6.44	6.65±1.46

Microdroplet Test Data



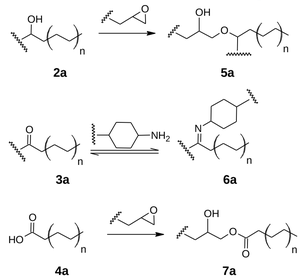
$$IFSS = \frac{F_c}{\pi \cdot d_f \cdot l_e}$$



IFSS and EA values for the Spectra/Epoxy composites obtained via microdroplet test techniques.

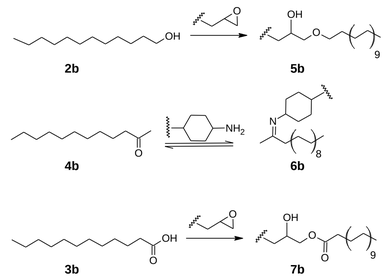
Best combination of IFSS and energy absorption is reached after 6 min plasma exposure (1LPM flow rate).

Plasma Treated PE Surface Chemistry



Possible reactions of PE fiber surface functionalities with epoxy/amine resin.

Modeling of Plasma Treated PE Surface Chemistry with Small Molecules Analogues

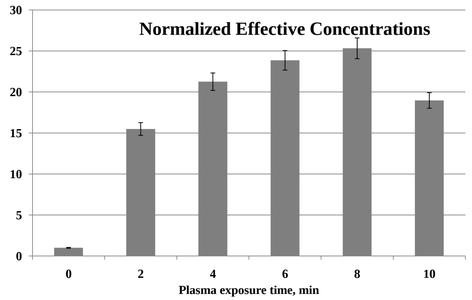


Possible reactions for the dodecyl derivatives that mimic the reactivity of PE fibers surface functionalities.

Conversion Coefficients and Effective Concentrations of Surface Functionalities on PE Surface

Plasma exposure time, min	[-OH], a.u.	[=C=O], a.u.	[-COOH], a.u.	-OH group conversion, c_i	$[EC]_i = [-COOH]_i + c_i[-OH]_i$, a.u.
0	4.20±0.69	0.0±0	0.0±0	0.13±0.02	0.55±0.09
2	16.38±5.59	9.33±5.85	3.28±1.64	0.26±0.03	8.52±1.25
4	16.62±6.28	8.37±5.69	4.05±4.48	0.46±0.03	11.69±1.46
6	13.81±4.55	3.62±1.18	2.90±1.65	0.74±0.05	13.13±1.85
8	15.22±2.86	5.05±0.98	6.17±1.50	0.51±0.04	13.93±1.84
10	13.50±1.51	10.94±6.44	6.65±1.46	0.28±0.03	10.43±1.60

Effective Concentrations of Surface Functionalities on Plasma Treated PE Surface

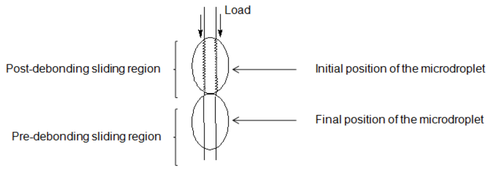


Highest effective concentration of surface functional groups is reached after 6 min plasma exposure (1LPM flow rate).

SURFACE STUDY OF PLASMA TREATED POLYETHYLENE FIBERS BY AFM

(Continued)

Surface Roughness Measurements for the plasma Treated PE Fibers

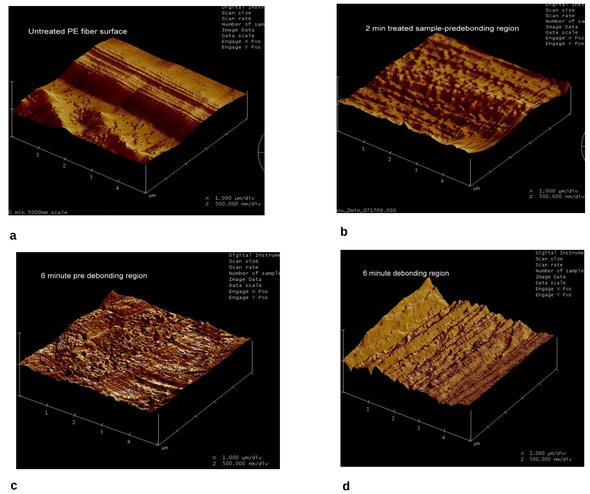


Schematic representation of pre- and post-debonding regions in a microdroplet composite.

Pre- and post-debonding surface roughness for the plasma treated PE fibers (determined by AFM).

Plasma exposure time, min	Pre-debonding RMS roughness, nm	Post-debonding RMS roughness, nm
0	32.50±11.76	32.50±11.76
2	44.03±7.44	42.13±15.78
4	39.12±8.23	16.28±3.64
6	25.45±11.01	61.88±8.04
8	41.64±13.45	35.48±15.35
10	46.82±17.16	53.81±6.65

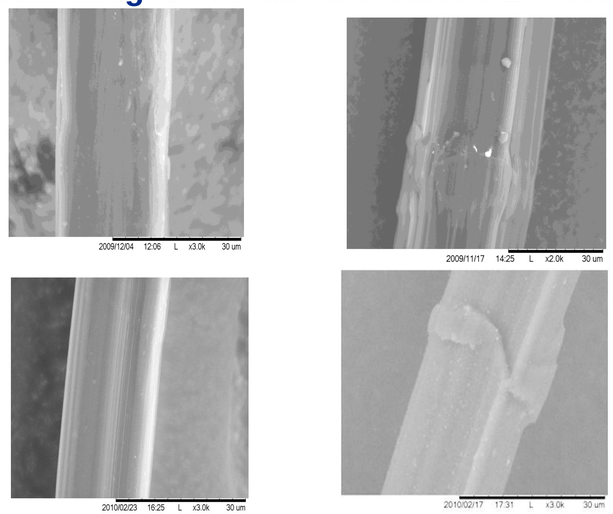
AFM Images of Plasma Treated PE Fibers



a b c d

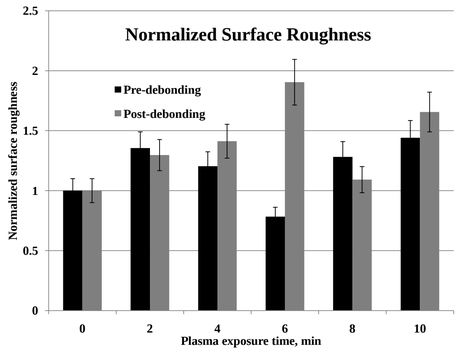
AFM images of a) untreated Spectra fiber; b) after 2 minutes exposure, pre-debonding region; c) after 6 minutes exposure, pre-debonding region; d) after 6 minutes exposure, post-debonding region.

SEM Images of Plasma Treated PE Fibers



SEM images of a) after 2 minutes exposure, pre-debonding region; b) after 2 minutes exposure, post-debonding region (mostly adhesive failure); c) after 6 minutes exposure, pre-debonding region; d) after 6 minutes exposure, post-debonding region (mixed adhesive/cohesive failure).

SUMMARY



Pre- and post-debonding regions' surface roughness obtained with AFM microscopy.

Highest post-debonding roughness is observed after 6 min plasma exposure

Conclusions

•The concentrations for the three major surface functionalities: hydroxyl, carbonyl and carboxyl were determined by XPS analysis.

•Determination of conversion coefficients for the reaction between epoxy resin and surface moieties showed 100% conversion of –COOH group, 0% conversion of =C=O group, and variable conversion of –OH function.

•Calculated effective concentrations of surface reactive species exhibit good correlation with the IFSS and energy absorption mechanical

Conclusions (continued)

data obtained through the microdroplet test techniques, as well as the pre- and post debonding surface roughness determined by the AFM.

•At high effective concentrations of surface groups and high post-debonding surface roughness there is a failure mode switch from predominantly adhesive to mixed adhesive/cohesive type, which suggests that the crack has to propagate through the more torturous path leading to the highest IFSS and EA of the interface.

ACKNOWLEDGEMENTS

This research was sponsored by the Army Research Laboratory under the joint CMR program with the Center of Composite Materials (CCMT 332179), for which we are grateful. The views and conclusions in this document are those of authors and should not be interpreted as representing the official policies, either expressed or implied, of the Army Research Laboratory or the US Government. The US Government is authorized to reproduce and distribute reprints for Government purposes notwithstanding any copyright notation hereon.