Influence of Treatment Duration on Hydrophobic Recovery of Plasma-Treated Ultrahigh Modulus Polyethylene Fiber Surfaces

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ABSTRACT: One of the major disadvantages of ultrahigh modulus polyethylene (UHMPE) fibers is their low surface energy which makes them difficult to adhere to most of the resins used in composites. Therefore, UHMPE fibers are often treated with plasmas to improve their surface properties. However, aging of plasma treatment effect is a major concern for plasma-treated fibers. In this study, UHMPE fibers were treated for 30, 60, 90, and 120 s with Ar/O_2 plasma on a dielectric barrier discharge device. The change of the surface properties and adhesion characteristics of the fibers were investigated immediately after and 30 days after the plasma treatments using X-ray photoelectron spectroscopy, contact angle measurement, scanning electron microscopy, and micro-bond tests. Results show that aging of the plasma treatment effect was suppressed by increasing the plasma treatment duration. The interfacial shear strengths were increased from 4 MPa to 5.9, 7.8, 9.2, and 7.6 MPa for the 30, 60, 90, and 120 s treatment groups, respectively. After 30 days' aging, the IFSS for the 30, 60, 90, and 120 s treatment groups lowered 24, 22, 10, and 9%, respectively. Increasing the plasma treatment time will increase the thickness and saturation degree of the oxidized layer on the polymer surfaces, which might hinder the migration of the hydrophobic polar functional groups from the surface to the bulk of the polymer after the plasma treatment. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 995–1001, 2008

Key words: dielectric barrier discharge; UHMPE fibers; interfacial shear strength; XPS analysis; aging effect

INTRODUCTION

Ultrahigh modulus polyethylene (UHMPE) fibers have found increasing use because of their high tensile strength and modulus, and low specific density comparing with other reinforcing fibers.[1–3] The unique properties of these fibers are due to the fully extended and aligned chain configuration derived from the gel spinning production process.[4–6] However, UHMPE fibers have inert surfaces resulting in poor interfacial bonding to most of the resins in composite applications. To improve the interfacial shear strength (IFSS) for UHMPE fiber reinforced composites, many surface modification methods have been studied such as coating, etching, and chemical modification. Among various surface treatment techniques, plasma treatment is an environmentally friendly technique which introduces polar groups or increasing surface roughness without affecting the bulk properties.[7–11] However, most of the plasma systems operate at low pressure which makes a continuous operation difficult. Therefore, much attention has been paid to plasmas operating at atmospheric pressure due to advantages of eliminating an expensive vacuum system, on-line processing capabilities, high efficiency, and the scalability to a larger area.[12–16]

At atmospheric pressure, there are essentially two methods for producing nonthermal plasmas using either a corona discharge or a dielectric barrier discharge (DBD). DBD is superior to corona discharge because it is more homogeneous.[17–20] An important advantage of the DBD is the simplicity of its operation in strongly nonequilibrium conditions at atmospheric pressure and at reasonably high power levels, without using sophisticated pulse power supplies.[21,22] Therefore it could be an ideal surface treatment technique for fibrous materials.

It is well known that plasma treatments have an aging effect, namely, the plasma-treated polymers revert to their original surface properties gradually over the time. The polar groups on the surface have the tendency to rotate or bury themselves from the

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surface to the bulk of the polymer.[23–26] Up to now, limited number of papers has been published about aging effect of the atmospheric pressure plasma treatment and little has been reported on the relationship between the length of plasma treatment time and the aging effect of the DBD treatment. The objective of this study was to examine the aging effect of the DBD treated UHMPE fibers as a function of treatment time. The aging effects were studied over a period of 30 days. Samples were plasma treated for 30, 60, 90, and 120 s. Contact angle measurements were made to study the changes of surface free energy. Scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS) were used to study the surface morphology and the chemical composition of the plasma-treated fibers. IFSSs between the fiber and an epoxy resin were determined using the micro-bond technique. The tensile strengths of the fibers were measured before and after the plasma treatments to assess the potential damage to the fiber.

EXPERIMENTAL

Materials

The UHMPE yarns were composed of 240 monofilaments with a single fiber diameter around 28 μm supplied by Ningbo Dacheng Chemical Fibers Company (Zhejiang, China). The yarns were soaked in acetone for 10 min and then dried in a vacuum oven to remove residual acetone. The cleaned yarns were wound onto glass frames to facilitate the subsequent plasma treatments. The epoxy resin was a mixture of DER 311 (bisphenol-A type epoxy) and DER 732 (polyglycol diepoxide) and the curing agent DEH 26 supplied by Dow Chemical Company (Shanghai, China).

Plasma treatment

The plasma treatments were carried out using a DBD plasma treatment system fabricated by the Academy of Opto-electronics, Chinese Academy of Sciences. A schematic diagram of the DBD plasma treatment apparatus is shown in Figure 1. In the DBD treatment processes, the frame with UHMPE fiber bundles were fed in between the two discharge plates in the treatment chamber. The distance between two plates was about 3 mm. The discharge power was around 1.75 kW, and the treatment gas was a mixture of argon as the carrier gas (5.17 L/min) and oxygen as the secondary gas (36 mL/min). The UHMPE fibers were treated for 30, 60, 90, and 120 s, respectively. After the treatment, the fibers were immediately placed into a clean plastic bag to minimize potential contamination. The control and

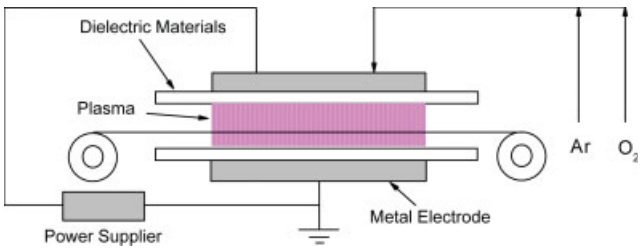

Figure 1 Schematic diagram of the DBD plasma treatment apparatus. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the treated samples were stored at the standard condition of 20°C and 65% relative humidity to perform the aging experiments.

Scanning electron microscopy

A JSM-5600LV SEM system was used to inspect the surface modification of the fibers directly after the plasma treatments. All of the fiber specimens were gold coated prior to conducting the SEM observation.

Contact angle measurements

The wettability was determined by measuring the water contact angle on the fibers using the sessile drop technique with a JC2000A Stable contact angle analyzer (Powereach Digital Equipment, Shanghai, China). Each contact angle reported was an average of at least 15 different measurements on at least five different fibers.

Surface chemistry analysis

The chemical composition of the fiber surface was determined using an X-ray photoelectron spectrometer (XPS) model ESCALAB 250 (Thermo Electron VG Scientific, Waltham, MA). The X-ray source was Mg Kα (1253.6 eV), operating at 300 W. The analysis was carried out under 10^{-7} to 10^{-8} Pa. Photoemitted electrons were collected at a take-off angle of 45° and curve fitting was performed in the C1s peaks.

IFSS test

The IFSS values of UHMPE/epoxy interface were measured using the micro-bond technique. To investigate the aging process of the plasma-treated UHMPE surfaces, two sets of samples were prepared in 1 h and 30 days after the initial plasma treatments, respectively.

The micro-bond specimens were prepared on a frame as described by Qiu et al.,[6] with DER 331 and DER 732 at a ratio of 70 : 30, and 12 phr (parts per hundred resin parts) of hardener DEH 26. After placing the epoxy beads on the fibers, the specimens

were cured for 2 h at 80°C and postcured for 0.5 h at 100°C. The diameters of the fibers and the lengths of the epoxy beads were measured using an Olympus CH-2 microscope equipped with a Panasonic WV-GP410/A digital photomicrography system. The micro-bond test was carried out at an upper clamp displacement rate of 1 mm/min on an XQ-1 fiber tensile testing machine (Shanghai Lipu Research Institute, China).

The IFSS, τ_i, was calculated using the following equation, derived from the well-known shear-lag model:[27]

$$\tau_i = \frac{nP_{\max}\coth(nL/r)}{2A}, \quad (1)$$

where $P_{\max}$ is the peak load, A is the cross-sectional area of the fiber, L is the embedded length, r is the equivalent fiber radius calculated from the fiber cross-sectional area, and n is defined as:

$$n = \left[\frac{E_m}{E_f(1+\nu_m)\ln(R/r)}\right]^{1/2}, \quad (2)$$

where E_m and E_f are the Young's modulus of the matrix (1.4 GPa) and that of the fiber (130 GPa), ν_m is Poisson's ratio of the matrix (0.4),[6] and R is the radius of the epoxy beads.

Single-fiber tensile test

Single-fiber tensile test was performed to inspect the impact of the plasma treatment on fiber strength. The test was carried out at a gauge length of 10 cm and a strain rate of 10 mm/min using the Universal Materials Testing Machine (Model H5K-S, Hounsfield, USA) with a load cell capacity of 50 N. An Olympus CH-2 microscope equipped with a Panasonic WV-GP410/A digital photomicrography system was used to take the longitudinal image of the UHMPE fibers at 400× magnification to determine the diameter of each fiber. At least 20 specimens were successfully tested for each treatment group.

Statistical analysis

One-way analysis of variance (ANOVA) and Turkey's pairwise multiple comparisons were used to compare the contact angles, the tensile strengths and the IFSS among different treatment groups. P values less than 0.05 were considered significant.

RESULTS AND DISCUSSION

Fiber surface morphology

SEM images of UHMPE fibers before and after the plasma treatments are shown in Figure 2. A significant etching effect of plasma could be seen and some cracks were generated on the surfaces of fibers. As the treatment time increased, more, longer, and wider cracks were developed. Similar results were reported by Jensen et al.,[28] who found many micro cracks developed on the surfaces of the UHMPE fibers treated with He/O_2/air DBD plasmas. Therefore, it could be due to the ablation and chain scission of the treated fiber surfaces as a result of DBD treatment. Another possibility was that local temperature on the surface of the fibers could exceed the melting temperature of the fiber and create heat stress on the fiber surface, resulting in the development of the cracks. The cracks are developed only along the direction perpendicular to the fiber axis. This is because the fibers often develop residual stress along their longitudinal direction due to elongation of the fibers in the spinning and stretching processes. When etching or surface melting occurs, the cracks are induced only perpendicular to the residual stress direction or the fiber longitudinal direction.

Contact angle measurements

The contact angles of the UHMPE fibers before and immediately after the DBD treatment are shown in Table I. The contact angles for the untreated samples decreased from 119.2° to 73.7° and 69.2° after 30 s and 60 s exposures to plasma, respectively. No further decrease in water contact angle was observed when plasma treatment time further increased.

Furthermore, as shown in Figure 3, the water contact angles increased for all the treated samples compared with those immediately after the plasma treatments. For the samples treated for 30 s and 60 s, the contact angle increased from 73.7° and 69.2° to 88.3° and 82.2°, respectively, after 30 days, whereas the 90 s and 120 s treated samples showed a smaller degree of hydrophobic recovery (from 67.9° and 66.9° to 76.8° and 75.1°, respectively). It may be concluded that the duration of the treatment time has a significant influence on the aging effect of the plasma treatment. Longer plasma treatment duration introduced more polar groups on the UHMPE fiber surface. The abundance of polar groups near the surface of the polymer may hinder their migration from the surface to the bulk. Therefore, the speed of hydrophobic recovery of the plasma-treated surface with longer treatment time could be lower than those treated for shorter time.

XPS analysis

Table II shows the relative chemical composition of the fibers right after the plasma treatments. The survey scan for the control UHMPE fiber consists of

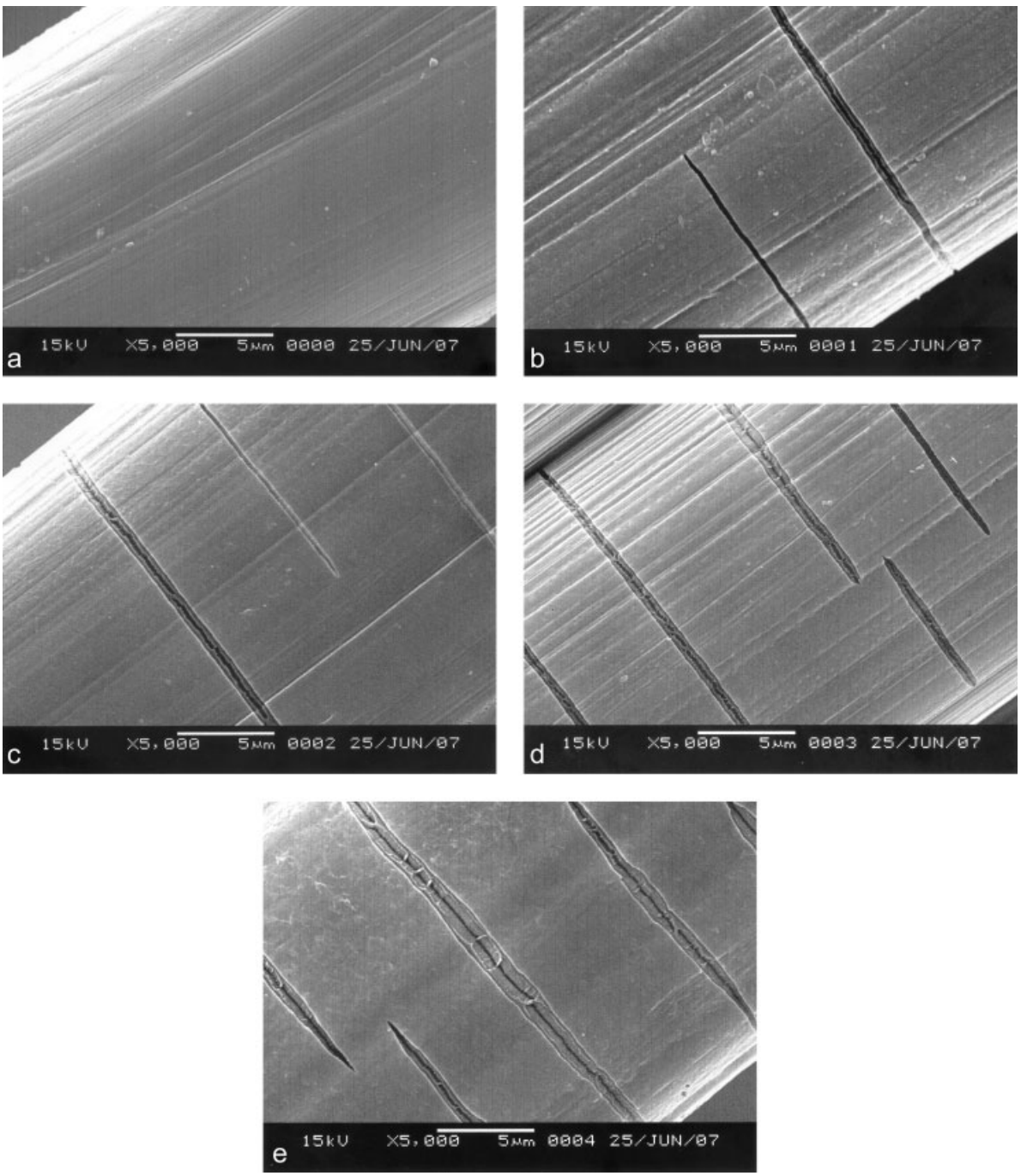

Figure 2 The SEM photographs (×5000 magnification) of the DBD treated UHMPE fibers: (a) control; and plasma treated for (b) 30 s, (c) 60 s, (d) 90 s, (e) 120 s.

two intense photoelectron peaks, the C1s at 285.0 eV and the O1s at 532.4 eV. The control fiber showed a certain amount of oxygen on the surface which is likely due to some contamination or residual finishing on the fiber surface as well as certain degree of oxidation. After the DBD treatment, the oxygen content increased significantly for all the plasma-treated groups. The oxygen content increased as the treatment time increased. However, when the treatment time exceeded 90 s, the oxygen content did not change substantially. To investigate what chemical functional groups are introduced to the UHMPE fiber surface after the DBD treatment, the concentrations of the functional groups with C1s were calculated by deconvolution analyses using XPSPEAK software and the detailed data are shown in Table II. The major peak at 285 eV was assigned to alkyls (C—C/C—H) and the smaller peaks were assigned to C—O at 286.5 eV, C=O at 287.9 eV, and O=C—OH at 289.2 eV according to Beamson and

TABLE I
Water Contact Angles of the UHMPE Fibers Untreated and Treated with Atmospheric Pressure Plasmas

Treatment	Number of specimens	Water contact angle (degree)	
		Mean	Standard deviation
Untreated	19	119.2[a]*	6.3
30 s	19	73.7[b]	3.8
60 s	22	69.2[c]	5.0
90 s	21	67.9[c]	5.0
120 s	19	66.9[c]	6.2

* Means with different letters are statistically different at $P < 0.05$.

Briggs.[29] The increase of the hydrophilic functional groups indicates that some of the C—C bonds in polymer surface could be broken by the plasma treatments. Then the carbon radicals, formed by the abstraction of hydrogen atoms from the polymer chains, may be combined with oxygen atoms generated in DBD resulting in the formation of the oxygen-containing polar functional groups on the fiber surface.[30,31]

The surface chemical composition and the results of the deconvolution analyses for all the fibers 30 days after the initial plasma treatment are presented in Table III. Clear differences between freshly treated samples and the aged ones were detected. It was found that the oxygen contents on the fiber surfaces for all the treatment groups decreased after 30 days of aging. Among the four plasma-treated groups, the oxygen concentration for the 30 s treatment group decreased from 15.4% to 9.9%, which was the most pronounced, whereas that for the 120 s treatment group (from 17.9% to 16.4%) was the smallest. Therefore, it was concluded that a longer plasma treatment duration led to a slower decay of oxygen content on the UHMPE fiber surfaces. The results were in accordance with those reported by Lawton et al.,[32] who found the length of time that polydimethylsiloxane film was plasma oxidized had a substantial effect on hydrophobic recovery and a shorter plasma duration could result in a greater aging effect. In addition, it was interesting that certain amount of nitrogen appeared on the fiber surface after aging for 30 days, but it could not be found freshly after the DBD treatment, which might be due to the reaction between the nitrogen in air and the free radicals on the fiber surfaces introduced by the plasma treatment during storage. From the results of the deconvolution analyses (Table III), one may find that a substantial reduction of the relative percentages of the C—O and C=O groups, which is more significant than the percentage change of O—C=O groups, suggesting that the C—O and C=O groups could be flopped or diffused into the polymer bulk faster than the O—C=O groups in the ambient air as found in our previous research.[33]

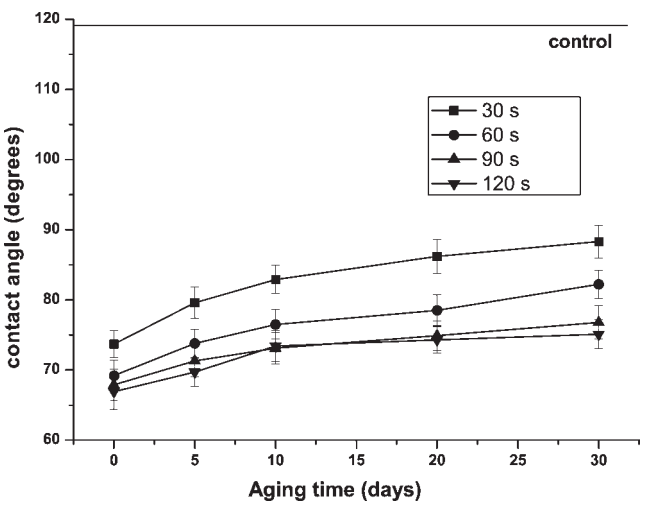

Figure 3 Change of the water contact angles for different treatment groups during aging.

Interfacial shear strength

As shown in Figure 4, the IFSS values for all the groups increased significantly immediately after the DBD treatment ($P < 0.05$). It is due to the combined

TABLE II
XPS Analysis of the Untreated UHMPE Fiber and After DBD Treatment

Group	Atomic concentration (%)		Relative area under C1s envelop (%)			
	C	O	C—C	C—O	C=O	O—C=O
Untreated	90.5	9.5	84.3	12.8	1.9	1
30 s	84.6	15.4	76.1	19.7	2.6	1.6
60 s	82.7	17.3	67.7	27.9	3.3	1.1
90 s	81.9	18.1	70.2	23.5	4.5	1.8
120 s	82.1	17.9	71	21.7	4.7	2.6

TABLE III
XPS Analysis of the Untreated UHMPE Fiber and 30 Days After DBD Treatment

	Atomic concentration (%)			Relative area under C1s envelop (%)			
Group	C	O	N	C—C	C—O/C—N	C=O	O—C=O
Untreated	90.5	9.5	–	84.3	12.8	1.9	1
30 s	92.1	9.9	0.4	80.6	16	1.9	1.5
60 s	83.4	15.4	1.2	76.2	21.4	1.6	0.8
90 s	84.3	14.4	1.3	77.1	18.2	3.2	1.5
120 s	82.1	16.4	1.5	73.5	20	3.5	3

effect of etching and surface modification produced by the plasma treatments, which increased both the mechanical interlocking and the wettability of the fiber surface. The IFSS values of the control UHMPE was around 4 MPa, it increased as the length of plasma duration increased and the peak value of 9.2 MPa was reached when fibers were treated for 90 s. When the UHMPE was treated by plasma for 120 s, the IFSS value decreased to 7.6 MPa. Similar results were also reported by Moon and Jang,[4] who found that the interlaminar shear strengths of UHMPE fiber/vinylester composites showed a maximum value at a plasma treatment time of 1 min and then decreased with a further increase of the treatment time. This phenomenon could be attributed to a change of the surface morphology of the UHMPE fiber with the plasma treatment. In the surface areas other than the cracks, the longer treatment time could result in a smoother surface due to the equilibrium of plasma etching and re-deposition.

After 30 days, the IFSS of all plasma-treated groups decreased compared with that immediately after the plasma treatment, mainly due to diffusion of the polar functional groups from the fiber surface to the bulk.[23,24,26] The decay rates of the IFSS were different for four treatment groups. Thirty days after the initial plasma treatment, the IFSS for the 30, 60, 90, and 120 s treatment groups reduced 24, 22, 10, and 9%, respectively. The duration of the plasma treatment appeared to significantly affect the aging behavior of the fiber surfaces. These results are in good agreement with the results of the contact angle measurement and the XPS analyses.

After the plasma treatment, the surface chain mobility enables the surface to be reoriented in response to the environment. The polar groups have a tendency to rotate and bury themselves below the surface, so as to reduce the surface free energy.[34] It is possible that the duration of plasma treatment may have a significant influence on the thickness of the oxidized layer on the polymer surface. The shorter the plasma duration, the thinner the oxidized layer, and therefore the more easily the polar functional groups diffuse from the surface to the bulk. Increasing the plasma treatment time might increase thickness of the oxidized layer on the polymer surfaces and the saturation degree of the oxidation of the treated surfaces. Normally, after a certain treatment time, the effect of plasma treatments could level off. Fritz and Owen[35] suggested that if a substantial portion of 30 nm film is oxidized, the amount of unoxidized polymer remaining in the film will be insufficient for complete recovery. Lawton et al.[32] also found that the length of plasma treatment time had a greater influence on the aging of the plasma-treated surface than the environment in which the treated polymer was stored.

Single-fiber tensile strength

Table IV shows the results of single fiber tensile tests of the UHMPE fibers from different treatment groups. No statistically significant difference between the tensile strengths of the control and those of the fibers treated for 30 and 60 s. However, once the treatment time exceeded 90 s, the tensile strength of the treated fibers decreased about 10% compared with that of the control group.

Plasmas introduce chemical and physical modifications only at the outmost surface (a few

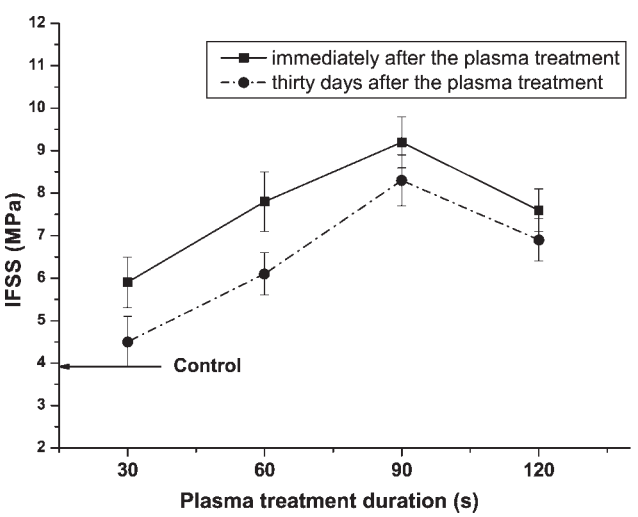

Figure 4 Comparison of IFSS of UHMPE fibers to epoxy freshly treated and 30 days after the plasma treatment.

TABLE IV
Single Fiber Tensile Strengths of Untreated and Plasma Treated UHMPE Fibers

Group	Number of specimens	Tensile strength (GPa)	Standard deviation (GPa)
Untreated	25	3.38[a]*	0.41
30 s	25	3.30[a]	0.43
60 s	25	3.35[a]	0.32
90 s	25	3.03[b]	0.34
120 s	25	2.96[b]	0.27

* Means with different letters are statistically different at $P < 0.05$.

nanometers in depth); a moderate plasma etching of fibers can improve the surface properties without degrading the fiber mechanical properties. An intensive and prolonged plasma treatment, however, may induce property changes in greater depth into the fiber, resulting in deterioration of the fiber tensile strength.[2] For a short DBD treatment time, though a certain degree of etching and cracking occurred because of the plasma treatment, as shown in the SEM images, the bulk properties of the UHMPE fibers were not influenced; however, when the treatment time was longer than 90 s, deep cracks were developed on the fiber surfaces leading to reduced mechanical properties of the UHMPE fibers.

CONCLUSIONS

The UHMPE fibers were treated by DBD to improve the surface wettability and the adhesion to epoxy. The fibers were treated for different time scales at 30, 60, 90, and 120 s. The fiber surfaces were etched significantly after the plasma treatment and the number, width, and length of the cracks on the fiber surfaces increased as the increase of the DBD treatment time. The water contact angles of the fibers decreased as the plasma treatment duration increased, whereas there was no further significant decrease in water contact angle when the treatment time was longer than 60 s. The IFSS values for all the groups increased significantly freshly after the DBD treatment, due to the combined effect of etching and the oxygen-based polar functional groups introduced by plasma treatments. The treated surface showed a gradual hydrophobic recovery over 30 days aging. The duration of the plasma treatment had a significant influence on the aging effect of the DBD plasma treatment: Shorter plasma treatment duration led to a faster aging tendency. There appeared to be no statistically significant difference in the tensile strengths between the control and the 30 and 60 s treatment groups. However, when plasma treated for longer than 90 s, the UHMPE fibers showed significantly decreased tensile strengths.

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