Mechanical Strength of PET Fibers Treated in Cold Plasma and Thermal Exposed

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As a followup to previous work, experiments with argon and oxygen Radio Frequency plasma treated polyethylene terepthalate (PET) exposed to 100 °C after cold plasma treatment were performed. Tensile tests results in monofilaments treated in oxygen and argon plasma for 5 s, 20 s, 30 s, and 100 s showed a decrease in the average tensile strength compared with the untreated fibers. It was also observed that the reduction in mechanical strength is more significant for argon plasma and very sensitive to the treatment time for oxygen plasma. Experimental data obtained from tensile tests in samples thermal exposed to 100 °C after plasma treatments indicate the same influence of treatment times on mechanical strength, as observed for cold plasma treated fibers. Furthermore, an increase in tensile strength when compared with the samples unexposed to 100 °C was observed. Scanning electron microscopy, used to analyze effects of cold plasma treatment on fibers surfaces, indicates differences in roughness profiles depending on the type of treatment. The distance of roughness interval, D_{ri}, was a parameter introduced to relate the fibers surface condition and average tensile strength. Statistical analysis of experimental data was performed to explain influences of treatment time, and environmental and temperature effects on mechanical properties.

Keywords PET fiber, plasma, statistical analysis, thermal treatment

1. Introduction

To obtain a material with a strong interface, the use of fibers and matrix of the same chemical nature is generally recommended. The chemical inertness of thermoplastic materials, which promotes a low interfacial adhesion for composites application, must be considered.[1-2] It is well know that this location is responsible for the load transfer from the matrix to the fiber.[3-9]

Consequently, many treatments are studied with the objective of modifying the superficial condition of the fibers to enhance the interfacial shear strength between fibers and matrices. The direct response is an effective influence on the mechanical properties of the composite.[10-12].

The plasma treatment is an interesting technique used to control the interfacial adhesion in composites.[1,13,14] Moreover, treatment time is an important parameter to consider.[15] For monofilaments, more specifically PET fibers, experimental data presented by Cioffi et al.[16] indicated that the oxygen and argon plasma treatment resulted in a decrease in the average tensile strength compared with the untreated fibers.

Given that a polymerization process is needed to obtain the composite matrix, the influence on mechanical properties of thermal treatment on cold plasma treated fibers in oxygen and argon gases must be evaluated.

This research analyzes the effect of thermal treatment at 100 °C for 1 h on the mechanical strength of PET fibers treated by cold plasma using oxygen and argon gases by 5 s, 20 s, 30 s, and 100 s. In this work the Weibull Distribution Function was used to statistically describe the fiber strength according to Ref. 17 and 18.

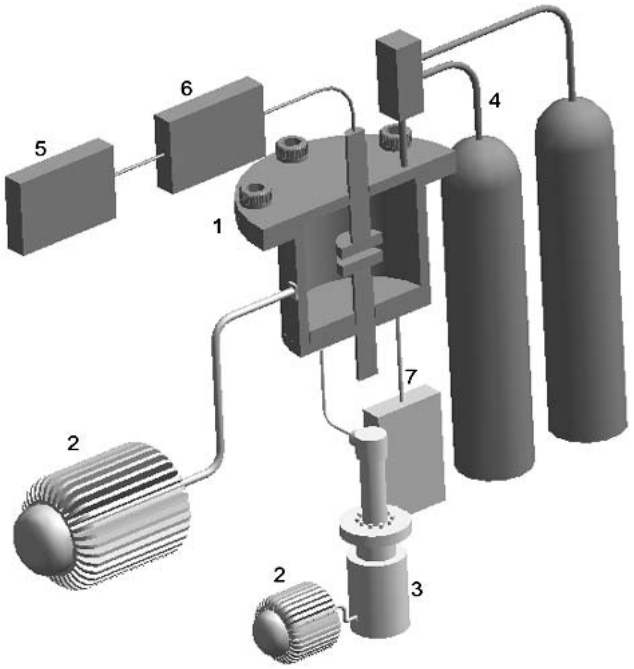

Fig. 1 Plasma reactor schema: **(1)** reaction chamber; **(2)** mechanic pump; **(3)** turbomolecular pump; **(4)** mass flow controller; **(5)** radio frequency generator; **(6)** impedance controller; **(7)** pressure controller

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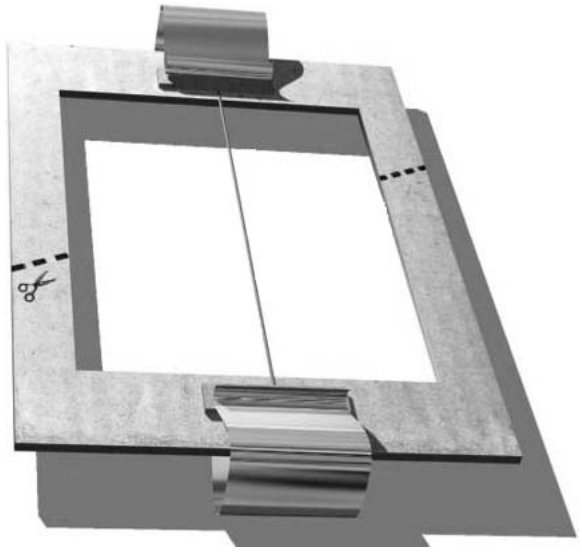
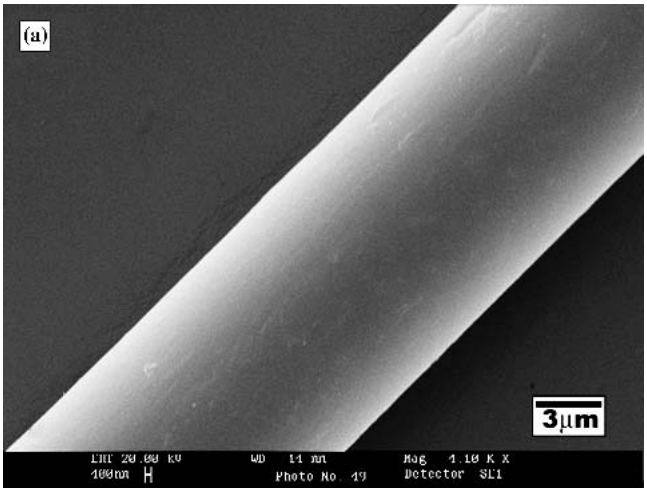


Fig. 2 Tensile specimen of the PET single fiber



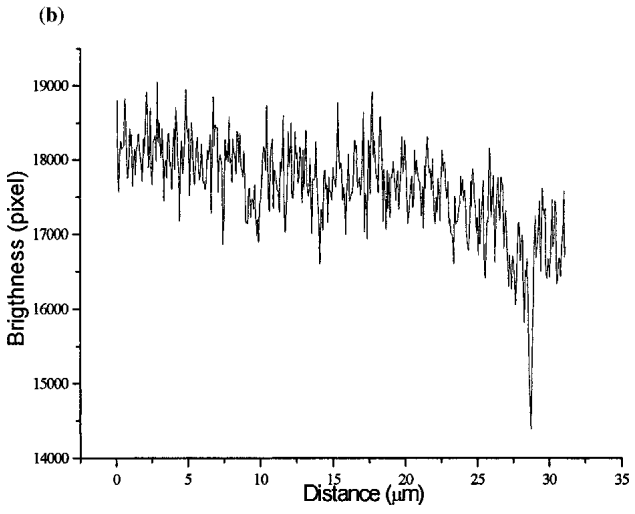
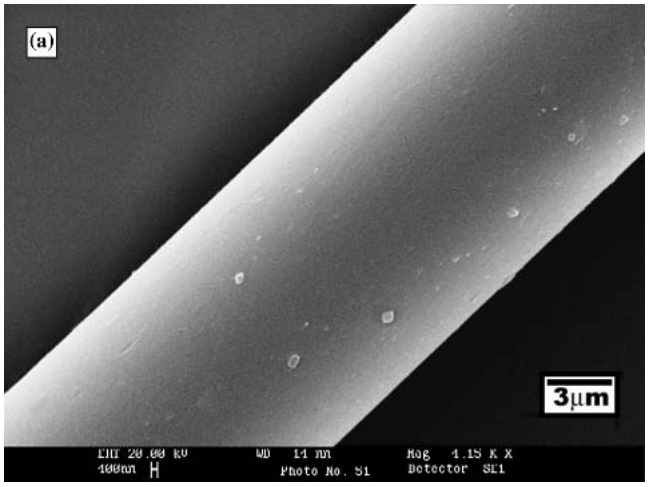


Fig. 3 Oxygen plasma treated PET fibers for 5 s post heated at 100 °C **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber



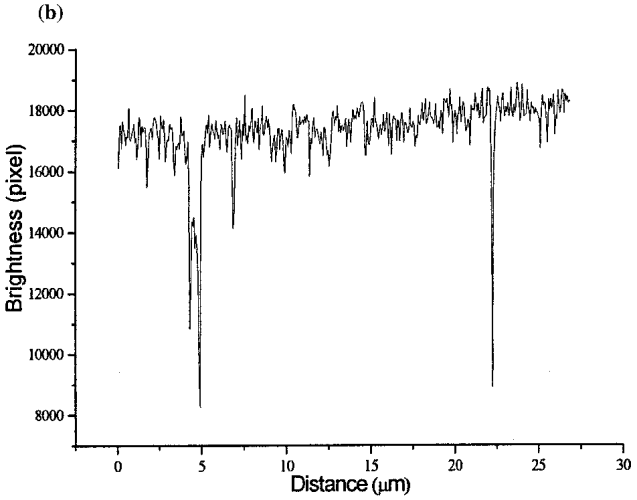


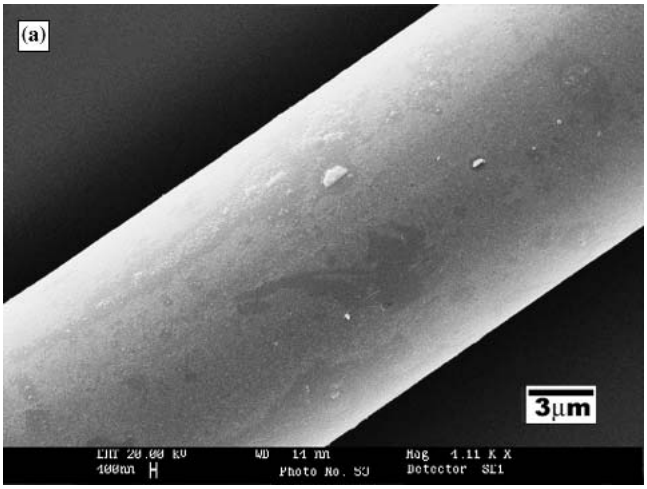
Fig. 4 Oxygen plasma treated PET fibers for 20 s post heated at 100 °C **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

2. Materials and Methods

Polyethylene terephthalate (PET) fibers were provide by Montefiber Spa (Acerra, Naples, Italy), with a filament diameter of about 13 μm and elastic modulus equal to 1 GPa. PET filaments were treated in a radio frequency cold plasma reactor (Fig. 1).

A 36×10^3 cm^3 reaction chamber, which contains 13 cm diameter electrodes, provides 2×10^3 cm^3 of plasma supplied by a radio frequency generator. Oxygen and argon gases were used to produce the sputtering and etching mechanisms. In the sputtering mechanism, from glow discharges the ion collides with the solid surface producing a series of collisions between atoms of the surface, leading to the ejection of one of these atoms. The plasma etching, in contrast to sputtering, deals with the chemical combination of the solid surface with the active gaseous species in the glow discharge.[14]

The treatment was performed according to the following conditions: excitation frequency was 13.56 MHz, the power of the electrical field was 50 W, the pressure of treatment was kept at 40 Pa by a double stage mechanic pump, the mass flow



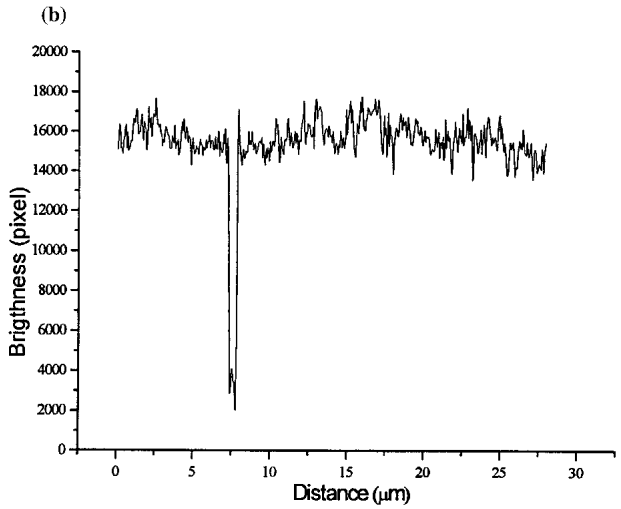
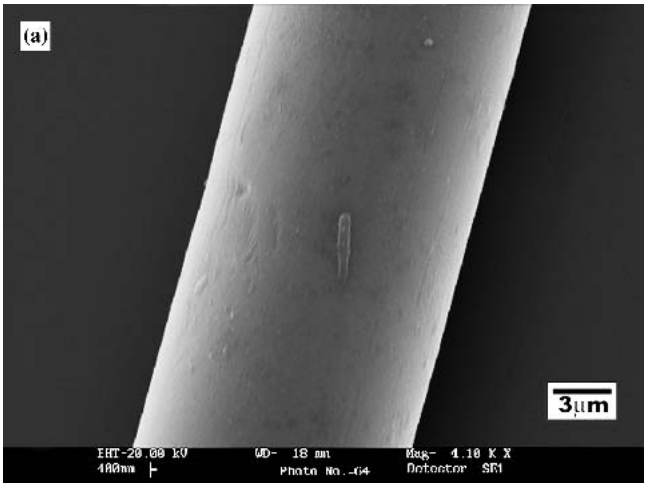


Fig. 5 Oxygen plasma treated PET fibers for 30 s post heated at 100 °C **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber



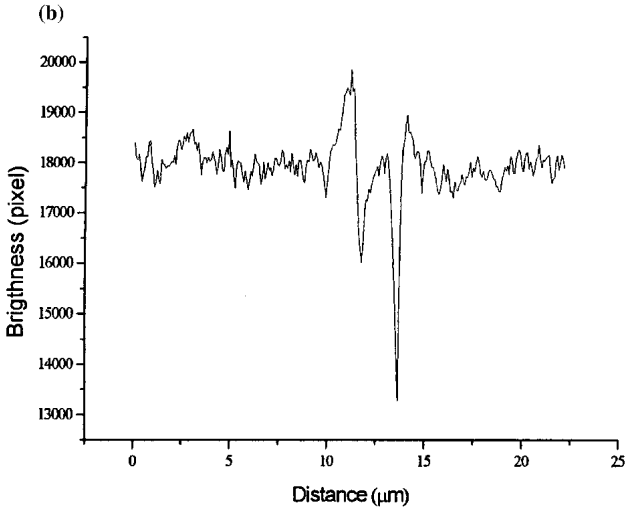


Fig. 6 Oxygen plasma treated PET fibers for 100 s post heated at 100 °C **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

Table 1 Tensile Strength Values of the PET Fibers Treated in Cold Plasma Using Oxygen and Argon Gases Post Exposed to 100 °C for 1 h

Treatment	Samples	σ (MPa) Average	Std, MPa	(%)1	(%)2	σ_{Max}, MPa	σ_{Min}, MPa
0 s	41	998	177	…	18	1383	615
O_2 5 s	9	1033	381	3.5	37	1689	538
O_2 20 s	8	1112	337	11	30	1844	691
O_2 30 s	9	1024	292	2.6	28	1771	766
O_2 100 s	9	990	157	−0.8	16	1230	766
Ar 5 s	9	871	138	−13	16	1000	693
Ar 20 s	8	1013	274	1.5	27	1510	843
Ar 30 s	10	953	194	−4.5	20	1463	777
Ar 100 s	8	976	475	−2.2	49	1847	616

(%)1, Variation of the average tensile strength in relation to the untreated material (998 MPa)
(%)2, Variation of standard deviation related to average tensile strength
−, Reduction in the average tensile strength
σ, Ultimate tensile strength

controller maintained 3.33×10^{-7} m^3/s gas flux, and the treatment time varied from 5 s to 100 s. Subsequently, PET fibers were exposed to a thermal treatment of 100 °C for 1 h.

For the tensile tests on the monofilaments, an INSTRON 4204 (Dept. of Materials and Production Engineering/ University of Naples "Federico II," Italy) at a constant speed of

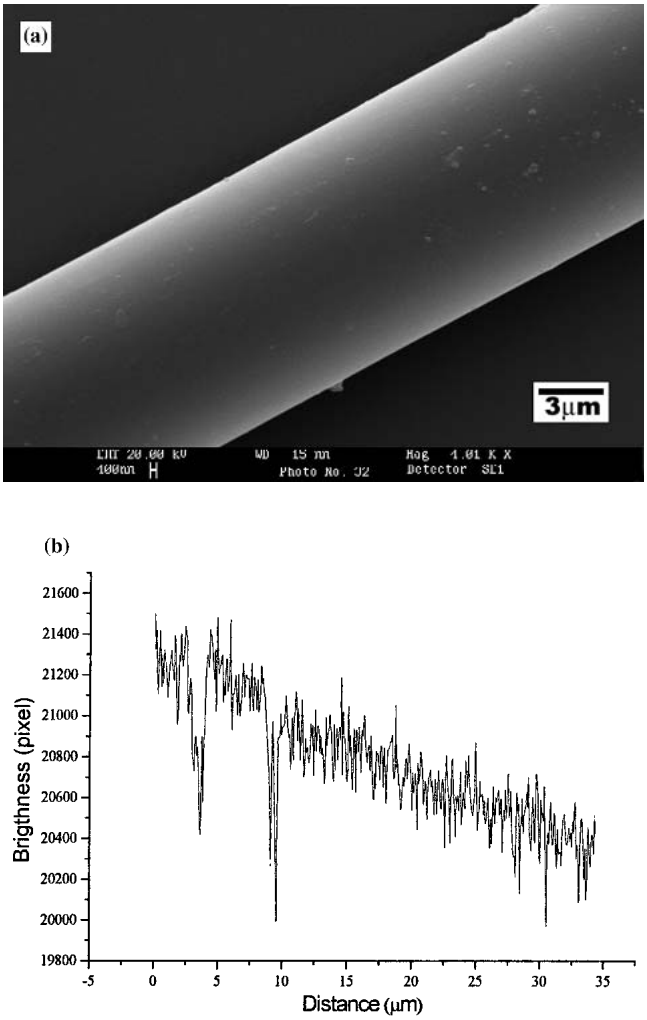


Fig. 7 Argon plasma treated PET fiber for 5 s post heated at 100 °C. **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

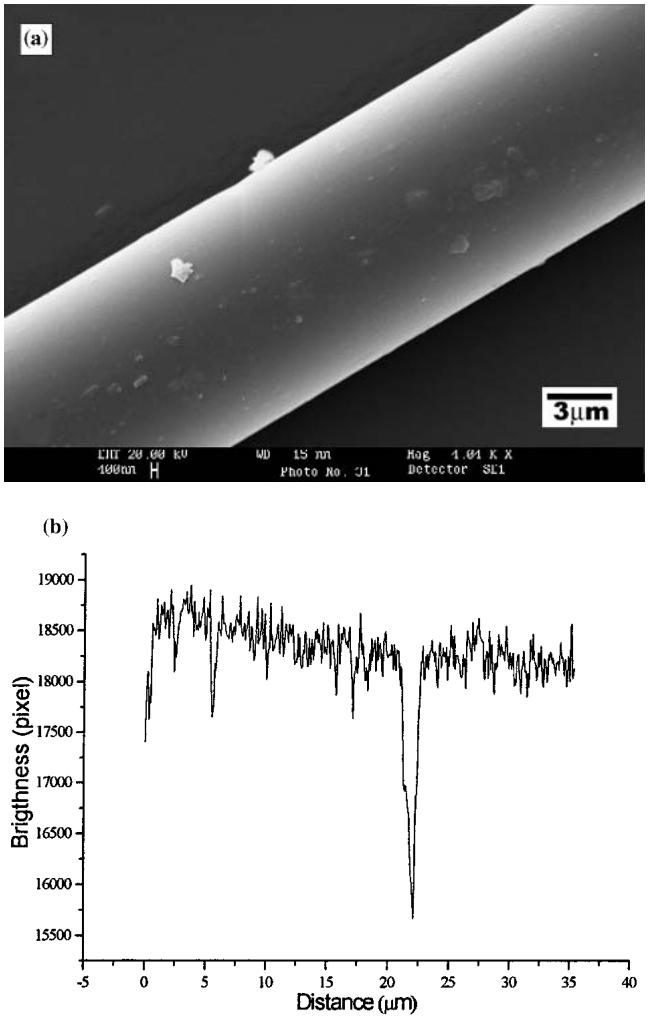


Fig. 8 Argon plasma treated PET fiber for 20 s post heated at 100 °C. **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

Table 2 Tensile Strength for PET Fibers Treated in Cold Plasma Post Heat Exposed to 100 °C (Higher and Lower Values Are Not Included)

Treatment	Samples	σ (MPa) Average	Std, MPa	(%)1	(%)2	σ_{Min}, MPa	σ_{Max}, MPa	σ_{Max}-σ_{Min}, MPa
0 s	41	998	177	…	18	615	1383	768
O_2 5 s	7	1010	284	1.2	28	767	1463	696
O_2 20 s	6	1059	116	6.1	11	999	1288	289
O_2 30 s	7	954	61	−4.4	6	844	999	155
O_2 100 s	7	987	121	−1.1	12	843	1154	311
Ar 5 s	7	889	107	−1.1	12	393	999	306
Ar 20 s	6	959	135	−3.9	14	843	1228	385
Ar 30 s	8	912	65	−8.6	7	843	999	156
Ar 100 s	6	892	327	−1.1	37	616	1535	919

(%)1, Variation of the average tensile strength in relation to the untreated material (998 MPa)
(%)2, Ratio between standard deviation and average tensile strength
−, Reduction in the average tensile strength
σ, Ultimate tensile strength

2×10^{-4} m/s with a 10 N load cell was used. The single fiber was assembled on rectangular cardboard tabs of 40 mm gauge length and fixed between the grips according to ASTM D3379 (Fig. 2).

Surface morphology analysis was developed using scanning electron microscope (SEM) LEO 220S (Dept. of Materials and Production Engineering/University of Naples "Federico II,"

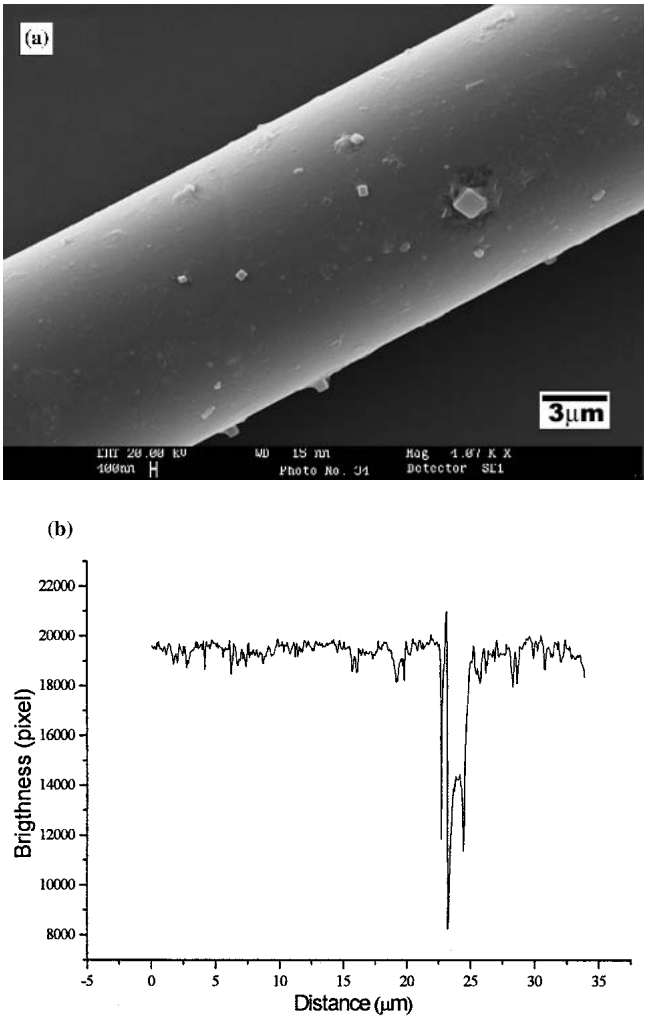


Fig. 9 Argon plasma treated PET fiber for 30 s post heated at 100 °C. **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

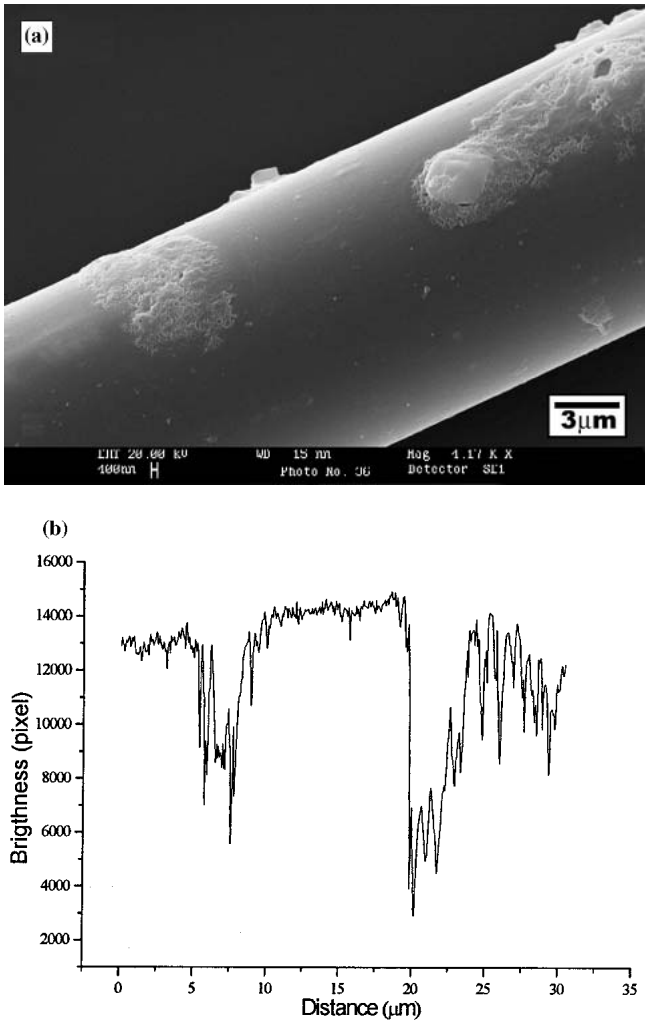


Fig. 10 Argon plasma treated PET fiber for 100 s post heated at 100 °C. **(a)** scanning electron microscopy of the fiber surface. **(b)** roughness profile of the PET fiber

Italy) and from the fibers surfaces images captured, the roughness profile of the treated and untreated fibers was produced.

The roughness profiles were obtained through the scanning line method where one considers a unit of linear length in which the roughness is linked with the brightness level. The calibrated method considered the diameter of the fiber as a reference.

Roughness level was evaluate throughout the distance of roughness interval (D_{ri}), which was calculated considering the same unit of linear defined length used to obtain the brightness (pixel) versus distance (µm) curve. The curves, represented from Fig. 3(b) to Fig. 10(b), present peaks for the different brightness levels represented on surface images of the fibers with reference to their superficial roughness. D_{ri} values are obtained as the ratio between the linear length and the number of peaks contained in this length, counted on the curve. This means that a lower D_{ri} is associated with a rougher surface.

3. Results

Table 1 shows the average tensile strength results for plasma treated and thermal exposed PET fibers. The number of specimens tested in each condition, standard deviation, variation of tensile strength values related to the untreated material, maximum and minimum tensile strength values, and variation of the standard deviation related to the average tensile strength are also indicated in Table 1.

Analysis of Table 1 shows that, for cold plasma oxygen gas, an increase in the average tensile strength for treatment times equal to 5 s, 20 s, and 30 s and a decrease for 100 s, in comparison with the untreated fibers. For cold plasma argon gas, the experimental results from Table 1 indicate that, for treatment time equal to 5 s, the average tensile strength is lower than that for the untreated fibers, increasing for treatment time equal to 20 s and decreasing again for 30 s and 100 s. Experimental data indicate, for both gases, that the increase in the cold plasma treatment time is responsible for a decrease in the

Table 3 Tensile Strength for PET Fibers Treated in Cold Plasma Post Heat Exposed to 100 °C (Lower Value and the Two Lowest Values Are Not Included)

	Without the Lower Value							Without Two Lowest Values						
Treatment	Sample, s	σ, MPa, Average	Std, MPa	(%)1	(%)2	σ_{Min}, MPa	σ_{Max}, MPa	Sample, s	σ MPa, Average	Std, MPa	(%)1	(%)2	σ_{Min}, MPa	σ_{Max}, MPa
O_2 5 s	8	1095	356	10	33	767	1689	7	1142	357	14	31	767	1689
O_2 20 s	7	1171	315	17	27	999	1844	6	1200	335	20	28	999	1844
O_2 30 s	8	1056	294	5.8	28	844	1771	7	1087	304	8.9	28	922	1771
O_2 100 s	8	1018	142	2.0	14	843	1230	7	1042	133	4.4	13	844	1230
Ar 5 s	8	903	107	−9.5	12	693	1000	7	932	70	−6.6	7	843	1000
Ar 20 s	7	1038	242	4.0	23	843	1510	6	1070	248	7.2	23	921	1510
Ar 30 s	9	973	193	2.5	20	843	1463	8	989	200	−0.9	20	844	1463
Ar 100 s	7	1028	469	3.0	46	667	1847	6	1088	483	9.0	44	691	1847

(%)1, Variation of the average tensile strength in relation to the untreated material (998 MPa)
(%)2, Ratio between standard deviation and average tensile strength
−, Reduction in the average tensile strength
σ, Ultimate tensile strength

Table 4 Tensile Strength for PET Fibers Treated in Cold Plasma Post Heat Exposed to 100 °C (Higher Value and Two Highest Values Are Not Included)

	Without the Higher Values							Without Two Highest Values						
Treatment	Samples	σ, MPa, Average	Std, MPa	(%) 1	(%) 2	σ_{Min}, MPa	σ_{Max}, MPa	Samples	σ, MPa, Average	Std, MPa	(%) 1	(%) 2	σ_{Min}, MPa	σ_{Max}, MPa
O_2 5 s	8	951	311	−4.7	33	538	1463	7	878	251	−12	29	538	1308
O_2 20 s	7	1007	175	0.9	17	691	1288	6	960	135	−3.8	14	691	1072
O_2 30 s	8	931	87	−6.7	9	767	999	7	922	89	−7.6	9	767	999
O_2 100 s	8	960	137	−3.8	14	767	1154	7	932	121	−6.6	13	767	1077
Ar 5 s	8	855	138	−14	16	616	999	7	833	135	−17	15	616	999
Ar 20 s	7	1038	242	4.0	23	921	1228	6	895	40	−10	5	843	922
Ar 30 s	9	896	77	−10	9	767	999	8	882	72	−12	8	767	999
Ar 100 s	7	852	317	−15	37	616	1535	6	738	107	−26	15	616	922

(%), Variation of the average tensile strength in relation to the untreated material (998 MPA)
−, Reduction in the average tensile strength
σ, Ultimate tensile strength

average tensile strength in comparison with the untreated material.

Very high standard deviations obtained in some cases showed the need to perform a data processing to obtain a reliable experimental tendency.[16]

Experimental data analysis for fibers exposed to 100 °C after cold plasma treatments are represented in Tables 2-5.

Table 2 was prepared excluding the higher and lower tensile strength values. Table 3 refers to data processing excluding the lower value and two lowest values. Table 4 was prepared excluding the higher tensile strength value and two highest tensile strength values, in each condition. Analyzing the average tensile strength data as a function of cold plasma treatment time from Table 2, for oxygen and argon gases, the average tensile strength values are lower than for untreated fibers (998 MPa), except for oxygen gas 5 s and 20 s. Furthermore, there is an initial tendency of the same behavior observed fibers treated in cold plasma using oxygen and argon gases without thermal treatment (Table 1). In Table 4, the data processing without the two highest values shows an average tensile strength behavior similar to that obtained in the case of cold plasma treated fibers.

Table 5 Distance of Roughness Interval (D_{ri}) Values of Cold Plasma Treated PET Fibers Post Heated at 100 °C

	Distance of Roughness Interval (D_{ri})	
Treatment Time	Oxygen	Argon
5 s	0.40	0.46
20 s	0.40	0.60
30 s	0.37	0.56
100 s	0.40	0.49

For cold plasma treatments, average tensile strength values are lower in comparison with the average tensile strength for the untreated fibers. For oxygen gas, 5 s is the treatment time in which the lowest average tensile strength is obtained. In both cases an increase in the average tensile strength occurs for 20 s and decreases again for 30 s and 100 s. It is also important to observe lower values for the standard deviation in comparison with Table 1. Table 3 indicates the same tendency for the average tensile strength in data processing excluding the lower tensile strength and two lowest strength values. It is not pos-

Table 6 Linear Representation of Weibull Parameters, α and β, and the Correlation Coefficient, R, for Five Simulation of the Tensile Strength. Cold Plasma Treated PET Fiber Post Heat Exposed to 100 °C

	Linear Representation																	
Treatment	**αint**	**βint**	**Rint**	**α>&<**	**β>&<**	**R>&<**	**α2>**	**β2>**	**R2>**	**α>**	**β>**	**R>**	**α<**	**β<**	**R<**	**α2<**	**β2<**	**R2<**
0 s	4.4	1205	0.96	4.3	1255	0.97	**4.7**	**1197**	**0.97**	4.7	1199	0.89	4.9	1224	0.88	5.3	1208	0.88
O_2 5 s	1.8	1641	0.93	1.6	1549	0.88	**2.2**	**1507**	**0.93**	2.0	1400	0.93	1.9	1643	0.87	1.0	1678	0.93
O_2 20 s	2.1	1625	0.83	3.1	1289	0.69	3.6	1284	0.86	**3.3**	**1291**	**0.89**	1.9	1786	0.65	1.9	1824	0.71
O_2 30 s	2.2	1525	0.72	7.1	1063	0.94	**6.7**	**1057**	**0.98**	7.0	1056	0.98	2.0	1603	0.64	1.8	1735	0.59
O_2 100 s	4.3	1176	0.95	3.8	1202	0.96	4.9	1155	0.95	4.7	1147	0.96	4.7	1213	0.94	**5.2**	**1223**	**0.98**
Ar 5 s	4.1	1123	0.98	3.7	1088	0.97	**3.9**	**1097**	**0.97**	4.0	1049	0.98	5.4	1051	0.97	8.4	1031	0.93
Ar 20 s	2.4	1595	0.72	2.6	1296	0.72	**2.7**	**982**	**0.88**	3.8	1177	0.70	2.4	1460	0.73	2.2	1548	0.70
Ar 30 s	3.1	1265	0.74	6.6	1032	0.90	8.0	969	0.93	**7.8**	**1001**	**0.94**	2.9	1305	0.68	2.8	1331	0.70
Ar 100 s	1.4	1428	0.79	1.2	1600	0.80	**4.3**	**955**	**0.91**	1.7	1353	0.76	1.4	1803	0.80	1.4	1901	0.83

sible to draw conclusions about the tendency of experimental data from Table 3, in which high values for standard deviations are indicated.

Experimental data indicated in Table 4, which exclude the two highest values, and confirmed by fracture surface analysis, show that 100 s treatment times with argon plasma may cause an important degradation on the fiber surface. Average tensile strength values are slightly higher than those for fibers treated in cold plasma, indicating that the thermal treatment at 100 °C is responsible for an increase in the mechanical strength of the fibers, probably associated with the minimization of stress concentration effect.

Comparison between unexposed data and the data processing in which two highest values are not considered from Table 4 shows, for almost all results, a lower reduction in the average tensile strength for cold plasma treated material and thermal exposed. It is also interesting to observe that for oxygen and argon plasma, 20 s treatment time is the condition in which the lower reduction in average tensile strength values occurs. Figure 3(a) to Fig. 10(a) represent scanning electron microscopy of the surface for treated fibers.

Roughness profiles obtained through the scanning line method are represented in Fig. 3(b) to Fig. 10(b), and the average distances of the roughness interval for each condition were calculated. Table 5 shows D_{ri} values for PET fibers treated in cold plasma and subsequently exposed to a thermal treatment of 100 °C for 1 h.

As a comparison parameter, it is important to note that D_{ri} is equal to 0.55 μm for untreated fibers. Higher D_{ri} means fewer roughness peaks in the reference unit length; closely spaced surface defects are associated with low values of D_{ri}. Data analysis from Table 5 indicates higher D_{ri} values for argon gas in comparison with oxygen gas, which, theoretically, indicates a smoother surface originated by the cold plasma treatment process.

On the other hand, surface analysis from Fig. 3(a) to Fig. 10(a) shows an intense surface degradation on fibers subjected to argon plasma. Roughness profile represented in Fig. 10(b) for argon plasma treatment 100 s and thermal exposed to 100 °C confirms the reduction in the average tensile strength. Table 4 shows that the average tensile strength for PET fibers cold plasma treated in argon gas and thermal exposed to 100 °C are always lower than those for oxygen gas, for the same treatment times.

Despite the fact that D_{ri} values are higher for argon gas in comparison with the oxygen gas (Table 5), surface degradation explains lower average tensile strength for PET fibers treated in argon plasma and thermal exposed to 100 °C.

D_{ri} values confirm tensile strength results obtained for untreated and treated fibers. For treatment time equal to 5 s, D_{ri} for both gases is lower than for the untreated fibers (D_{ri} = 0.55 μm), and so are average tensile strength values.

Increasing treatment time to 20 s results in an increase in D_{ri} or, in other words, a less rough surface; for 30 s and 100 s, surface analysis shows degradation associated with cold plasma treatment.

For oxygen gas, Table 5 indicates a reduction in D_{ri} value for treatment time equal to 30 s accompanied by a decrease in the average tensile strength (Table 4).

Comparisons between D_{ri} values for oxygen plasma treated PET fibers exposed and unexposed to 100 °C indicate very similar results. This behavior is not followed by the average tensile strength, which shows, for treatment time equal to 30 s and 100 s, significant differences.

For argon gas, Table 5 shows an increase in D_{ri} for treatment time equal to 20 s, 30 s, and 100 s in comparison with 5 s. For 20 s and 30 s, the increase in D_{ri} is accompanied by an increase in the average tensile strength; for 100 °C the average tensile strength decreases in comparison with 5 s.

Comparisons between D_{ri} values for argon plasma treated PET fibers and exposed to 100 °C with plasma treated and unexposed material indicate higher values for the former, which shows the important effect of the thermal treatment on the fiber surface condition.

The effect of thermal exposure, after plasma treatment, on mechanical properties of PET fiber was also analyzed using the Weibull Distribution Function. Table 4 shows Weibull parameters, α, β, and the correlation coefficient, R, for the five following data processing: complete experimental data, excluding the lower tensile strength value, excluding the higher tensile strength data, and excluding two lowest and two highest points. Analyses of Table 6 indicate that higher correlation coefficients are obtained when the higher and two highest tensile strength results are not considered.

Table 7 was prepared from results indicated in Table 5, using the highest correlation coefficients as a selecting parameter. Comparison between the Weibull parameters shows higher values for β in the thermal treated condition, for oxygen

Table 7 Best Weibull Linear and Cumulative Parameters, α and β, for Highest Coefficient Correlation, R, for Five Simulations of the Tensile Strength Values of Cold Plasma Treated PET Fiber Post Heat Exposed at 100 °C

	Linear Representation			Cumulative Distribution		σ Average, MPa
Treatment	α	β	R	α	β	σ_i
0 s	6.0	1062	0.97	7.5	1050	998
O_2 5 s	2.2	1507	0.93	2.1	1215	878
O_2 20 s	3.3	1291	0.89	5.3	1050	960
O_2 30 s	6.7	1057	0.98	8.2	1005	922
O_2 100 s	5.2	1223	0.98	6.9	1110	932
Ar 5 s	3.9	1097	0.98	3.8	1000	833
Ar 20 s	2.2	982	0.88	2.8	1230	895
AR 30 s	7.8	1001	0.94	8.0	940	882
Ar 100 s	4.3	955	0.91	4.9	900	738

and argon cold plasma treated fibers, related to these Weibull parameter obtained for plasma treated samples presented by Cioffi et al.[16] Values of β for the plasma treated and thermal exposed fibers were frequently higher than β for untreated fibers, which confirmed the increase of defects amount on the fiber surface with the thermal treatment.

Analyses of α indicates lower values for 5 s and 20 s and higher for 30 s and 100 s of treatment times compared also with unexposed samples. In relation to the untreated fiber, α values of plasma treated post thermal exposed fibers were generally lower except for the 30 s treatment time for both gases. The thermal exposure gives the fiber a wide range of tensile strength values.

Whereas the differences in the behavior of α and β occurred after thermal exposure, the tendency of the average tensile strength remained the same as cold plasma treated and unexposed fibers. Interestingly, for oxygen and argon gases, 20 s of treatment time is the condition in which the highest average tensile strength was obtained.

Experimental results from tensile tests in fibers treated in cold plasma, exposed and unexposed to 100 °C, indicate that initially, for 5 s treatment time, the fiber surface was damaged and the mechanical strength reduced. The increase in treatment time to 20 s reduced the stress concentration effect, which increased tensile strength compared with 5 s. For treatment times equal to 30 s and 100 s, degradation of fiber surface further decreased in tensile strength.

4. Conclusions

- PET fibers treated in oxygen and argon plasma for 5 s, 20 s, 30 s, and 100 s and subsequently exposed to a thermal treatment of 100 °C for 1 h were found to have lower average tensile strength values compared with the untreated fibers.
- Experimental data treatment resulted in the same behavior observed for PET fibers cold plasma treated and unexposed to 100 °C; an initial reduction in the average tensile strength for a treatment time of 5 s was followed by an increase for 20 s and decrease again for 30 s and 100 s treatment times.
- PET fibers treated in oxygen and argon plasma for 5 s, 20 s, 30 s, and 100 s and subsequently exposed to a thermal treatment of 100 °C showed higher average tensile strength results in comparison with fibers treated and unexposed.
- Thermal exposure of 100 °C after cold plasma treatment showed for oxygen gas, D_{ri} values close to unexposed fibers; on the other hand experimental data for argon gas indicated an important influence of thermal treatment on the fiber surface conditions.
- Parameters α and β from Weibull Distribution were calculated to understand experimental behavior obtained from tensile tests in oxygen and argon cold plasma treated fibers thermal exposed to 100 °C. Thermal treatment is responsible for a wide range of tensile strength values, confirmed by the decrease in α due to the increase in the fiber surface roughness and, consequently, the increase in β.

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