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Tensile Strength of Radio Frequency Cold Plasma Treated PET Fibers—Part I: Influence of Environment and Treatment Time

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This article reports on a series of experiments with polyethylene terepthalate (PET) treated in a radio frequency plasma reactor using argon and oxygen as a gas fuel, for treatment times equal to 5 s, 20 s, 30 s, and 100 s. The mechanical strength modification of PET fibers, evaluated by tensile tests on monofilaments, showed that oxygen and argon plasma treatment resulted in a decrease in the average tensile strength compared with the untreated fibers. This reduction in tensile strength is more significant for argon plasma and is very sensitive to the treatment time for oxygen plasma. Scanning electron microscopy (SEM) used to analyze the effects of cold plasma treatment on fiber surfaces indicates differences in roughness profiles depending on the type of treatments, which were associated with variations in mechanical strength. Differences in the roughness profile, surveyed through an image analysis method, provided the distance of roughness interval, D_{ri}. This parameter represents the number of peaks contained in a unit length and was introduced to correlate fiber surface condition, before and after cold plasma treatments, and average tensile strength. Statistical analysis of experimental data, using Weibull cumulative distribution and linear representation, was performed to explain influences of treatment time and environmental effects on mechanical properties. The shape parameter, α, and density parameter, β, from the Weibull distribution function were used to indicate the experimental data range and to confirm the mechanical performance obtained experimentally.

Keywords argon, oxygen, PET fiber, plasma treatment, statistical analysis

1. Introduction

Reinforcement of a thermoplastic polymer like polymethyl methacrylate (PMMA) with continuous polyethylene terepthalate (PET) fibers, results in a composite material with some improved characteristics such as low embrittlement, stability, and high mechanical strength.[1] The characteristics of chemical inertness and low surface energy of the PET fibers result in a weak interfacial bond with the polymers in the production of composites.[2,3] It is well known that the specific high strength of composites, which results from the interaction between the constituents, is directly related to the interfacial condition achieved or to the interphase between them. The adhesion between fiber and matrix affects the performance of the composite,[4-9] considering that this region is responsible for the stress transfer from the matrix to the fiber.[10] Many treatments to modify the surface of the fibers are used to enhance the interfacial shear strength with consequent influence on the mechanical properties of the composite.[11-13]

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The plasma treatment can be used to modify the chemical and physical state of the material surface without altering the bulk properties. These attributes indicate the plasma treatment of fiber surface as an important technique for the control of interfacial adhesion in composites.[2,14] Occhiello et al. observed that no real increase in pull strength with treatment time was obtained for more than 5 s or 10 s.[15] Some authors also observed that treatment times longer than 180 s, with argon plasma, cause heavy degradation on the poly(tetrafluoroethylene) fiber surface.[16]

It is recognized that fiber strength must be described statistically, given the fact that fiber fracture is controlled by the statistical distribution of the surface defects.[17] As stated by Tanaka et al. and Lipson and Sheth,[18,19] Weibull statistics were used to analyze the tensile strength results obtained for PET monofilaments treated in a cold plasma reactor, using two gases and various treatment times.

These PET filaments, after treatments, were tested in tensile mode and the results obtained were statistically analyzed by Weibull distribution function, according to Eq 1, which considers the $f(\sigma)$ as an accumulated distribution function of failures.

$$f(\sigma) \equiv 1 - \exp\left\{-\left[\frac{\sigma - \sigma_s}{\beta}\right]^{\alpha}\right\} \quad \text{(Eq 1)}$$

The density of data (β), the shape of distribution (α), and the largest stress at which the probability of failure is zero (σs) are dependent of the materials and change with the surface condition.[14]

In this work, σ_s considered null, resulted in the highest values of the correlation coefficients (R), which indicates that

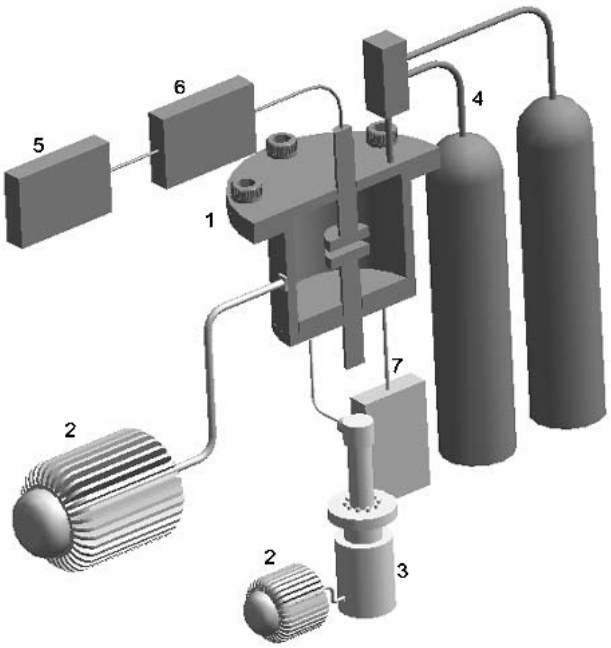


Fig. 1 Plasma reactor schema: (1) reaction chamber; (2) mechanic pump; (3) turbomolecular pump; (4) mass flow controller; (5) radio frequency generator; (6) impedance controller; (7) pressure controller

the best correlation of experimental data was obtained when the material constant σ_s is not included in the Weibull function.

The linear representation of the experimental data was obtained by taking lnln of $f(\sigma)$ as follows:

$$\ln\ln[1 - f(\sigma)]^{-1} \equiv \alpha\ln(\sigma - \sigma_s) - \alpha\ln\beta \quad \text{(Eq 2)}$$

Experimental data were plotted as $\ln\ln (1 - p_i)^{-1}$ versus $\ln\sigma_i$, in which p_i is associated with the cumulative distribution and is calculated as $p_i = i/(n + i)$, where n is the number of experimental values and σ_i is the average tensile strength in an ascendant sequence.[14].

Roughness measurements through image analysis, based on mathematical concepts,[20] that provide a quantitative approach to the image were performed.

2. Materials and Methods

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

A 36×10^3 cm^3 reaction chamber, which contains 13 cm diameter electrodes, provides a 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

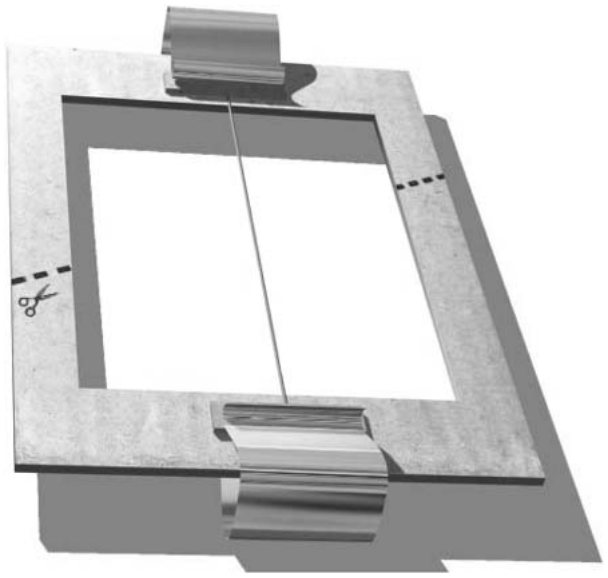


Fig. 2 Tensile specimen of the PET single fiber

gaseous species in the glow discharge.[21] 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 controller maintained 3.33×10^{-7} m^3/s gas flux, and the treatment time varied from 5-100 s.

For the tensile tests on the monofilaments, an INSTRON 4204 (Department of Materials and Production Engineering-University of Naples, Italy) at a constant speed of 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, as demonstrated in Fig. 2.

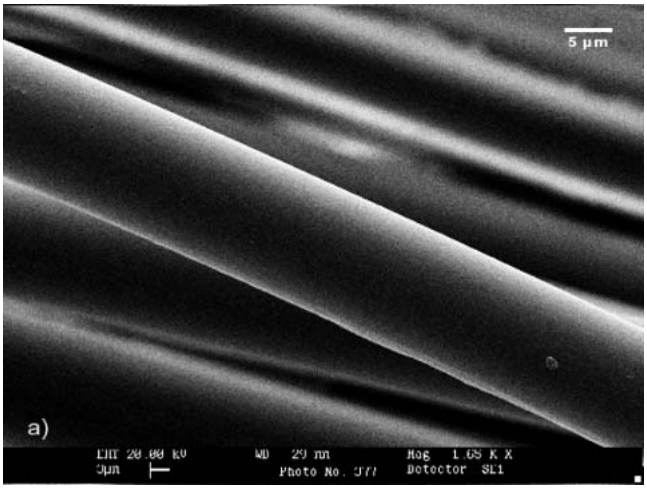
Surface morphology analysis was developed using scanning electron microscope (SEM) LEO 220S (University of Naples). From the captured surface images of the fibers, the roughness profile of the treated and untreated fibers was produced.

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 evaluated 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)-11(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 and Discussion

Table 1 shows tensile strength test results for PET fibers treated with radio frequency cold plasma, using oxygen and



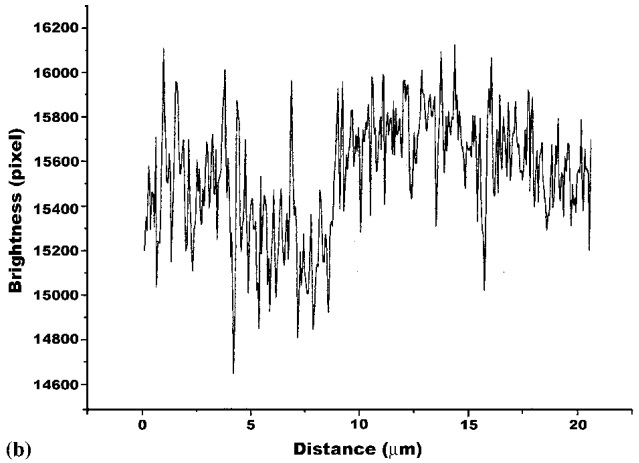
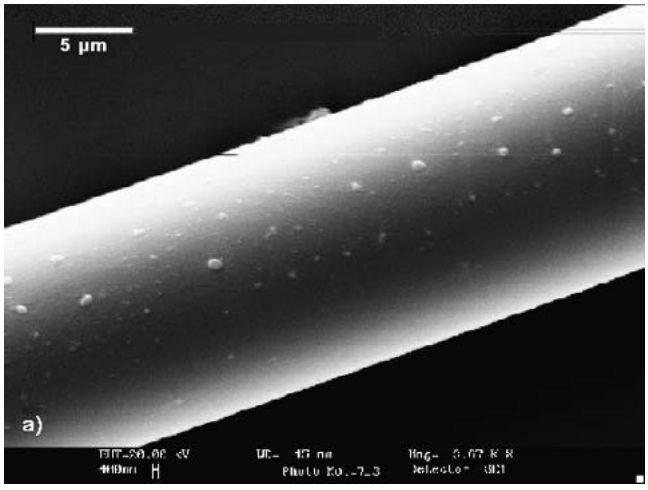


Fig. 3 Untreated PET fibers: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber



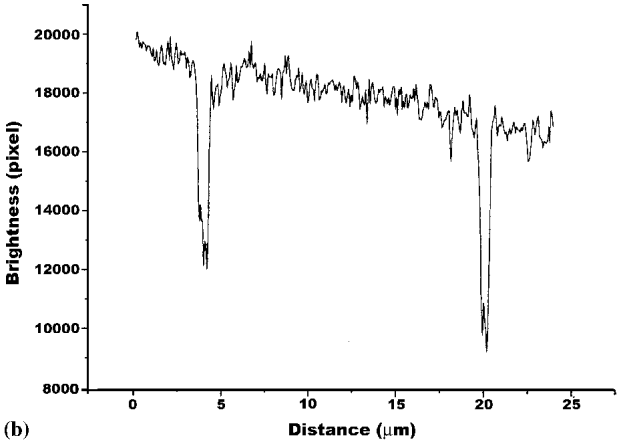


Fig. 4 Oxygen plasma treated PET fibers for 5 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

Table 1 Tensile Strength Values of PET Fibers Treated in Cold Plasma Using Oxygen and Argon Gases

Treatment	Samples	σ, MPa Average	Sdt, MPa	σ*max*, MPa	σ*min*, MPa	%, 1 (a)	%, 2 (b)
0″	41	998	177	1383	615	…	18
O_2 5″	57	858	121	1229	536	–14	14
O_2 20″	27	984	159	1231	614	–2	16
O_2 30″	10	747	52	846	693	–25	7
O_2 100″	9	590	149	846	460	–41	25
A_r 5″	18	819	161	1073	462	–18	20
A_r 20″	17	859	68	923	691	–14	8
A_r 30″	10	732	110	846	539	–27	15
A_r 100″	10	762	112	847	614	–24	15

(a) Variation of the average tensile strength in relation to the untreated material (998 MPa); –, reduction in the tensile strength.
(b) Ratio between standard deviation and average tensile strength.

argon gases, for times equal to 5 s, 20 s, 30 s, and 100 s. The number of specimens tested, standard deviation (Sdt), the higher and lower tensile strength values (σ*max* and σ*min*), the variation of average tensile strength of treated fibers in relation to average tensile strength of untreated fibers, and the ratio between Sdt and average tensile strength for each condition, are also indicated.

Experimental results represented in Table 1 show that cold plasma treatments, using oxygen and argon gases, were responsible for lower average tensile strength values in comparison to the untreated fibers. Moreover, for both gases, higher tensile strength reduction was observed for longer treatment times and higher average tensile strength values were obtained for treatment times equal to 20 s.

Data analyses indicate that for PET fibers treated with cold plasma using oxygen and argon gases, the average tensile strength shows an initial decrease for 5 s treatment time in comparison with the average tensile strength value for the untreated fibers.

Afterwards, an increase in the average tensile strength occurred for treatment time equal to 20 s, but was still lower than that for the untreated fibers. For both gases, experimental results for 30 s and 100 s treatment times indicate a decrease in the average tensile strength. For PET fibers treated in 5 s, Table 1 shows that reductions in average tensile strength compared

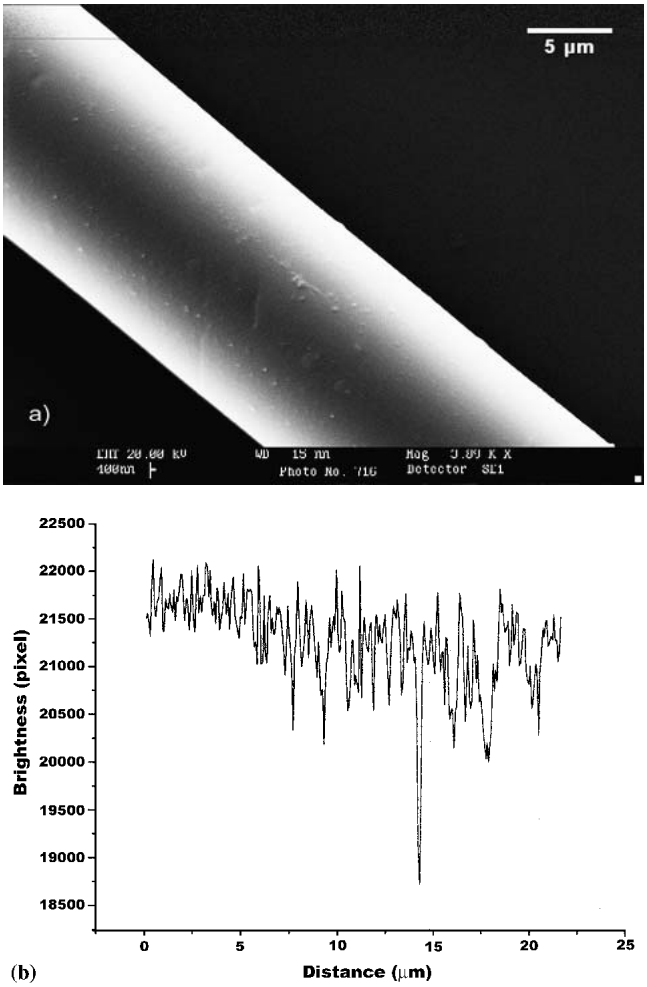


Fig. 5 Oxygen plasma treated PET fibers for 20 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

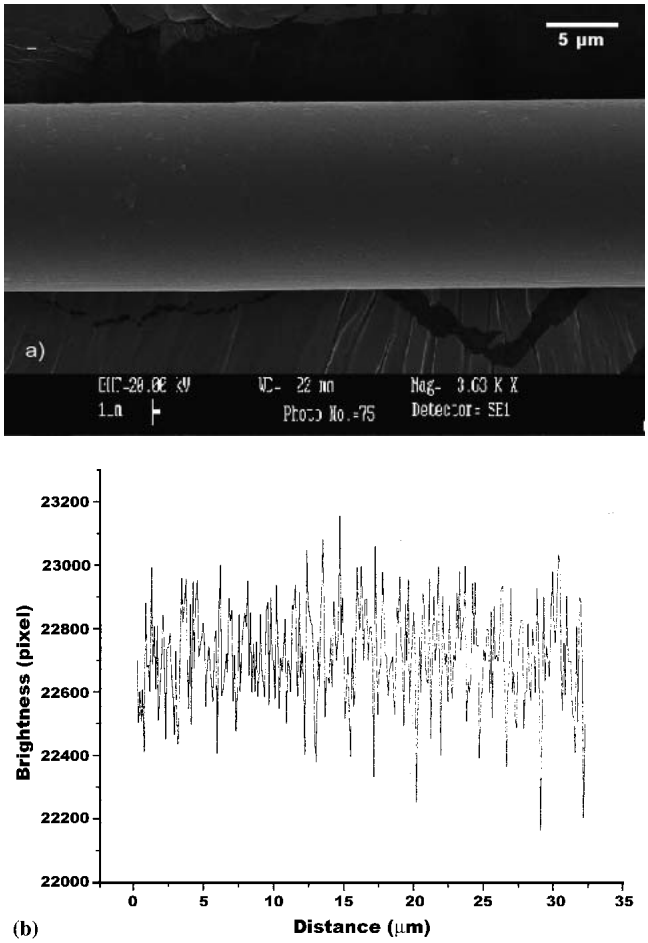


Fig. 6 Oxygen plasma treated PET fibers for 30 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

with the untreated fibers are 14% and 18% for oxygen and argon gases, respectively.

For the 30 s treatment time, reductions in average tensile strength compared with the untreated fibers are 25% and 41% for oxygen and argon gases, respectively. For the 100 s treatment, the reductions are 27% and 24% for oxygen and argon gases, respectively, The negative values for the average tensile strength variation of treated fibers in relation to the average tensile strength of untreated fibers indicate that a reduction in strength occurred after cold plasma treatment using oxygen and argon gases. For both gases, 20 s of treatment time is the condition in which the lowest decrease in the average tensile strength was observed.

Experimental results from tensile strength tests, σmax and σmin, show the same tendency indicated for the average tensile strength; cold plasma treatments using oxygen and argon gases were responsible for lower values of σmax and σmin in comparison to the untreated fibers, with a more pronounced effect for treatment time equal to 5 s, excepting 20 s treatment time for cold plasma argon gas.

The tendency observed for the treated fibers' tensile strength values may be explained by an important stress concentration effect induced by the plasma treatments. In the 5 s treatment time, impact of ions on the fiber surfaces[2,22] produce defects that have an intense concentration effect and as a consequence the fiber mechanical strength is affected. The increase in treatment time to 20 s for both gases is responsible for smoother fiber surfaces in comparison to the fibers that are cold plasma treated for times equal to 5 s; thus confirmed by the average range of brightness levels observed.

The minimization in the stress concentration effect of the defects explains higher values for the tensile strength for fibers treated during 20 s in comparison to 5 s of treatment time. Fiber degradation observed for treatment time equal to 30 s and 100 s are correlated to the decrease in mechanical strength for cold plasma treated fibers.

The distance of roughness interval (D_{ri}) was used to analyze the influence of cold plasma treatment times, using oxygen and argon gases, on the tensile strength of the fibers. Through image analysis from fiber surfaces, represented in Fig. 3(a)-11(a), fiber roughness profiles were performed and are represented in Fig. 3(b)-11(b).

Table 2 shows D_{ri} as a function of treatment time for cold plasma treated fibers in oxygen and argon gases. The distance

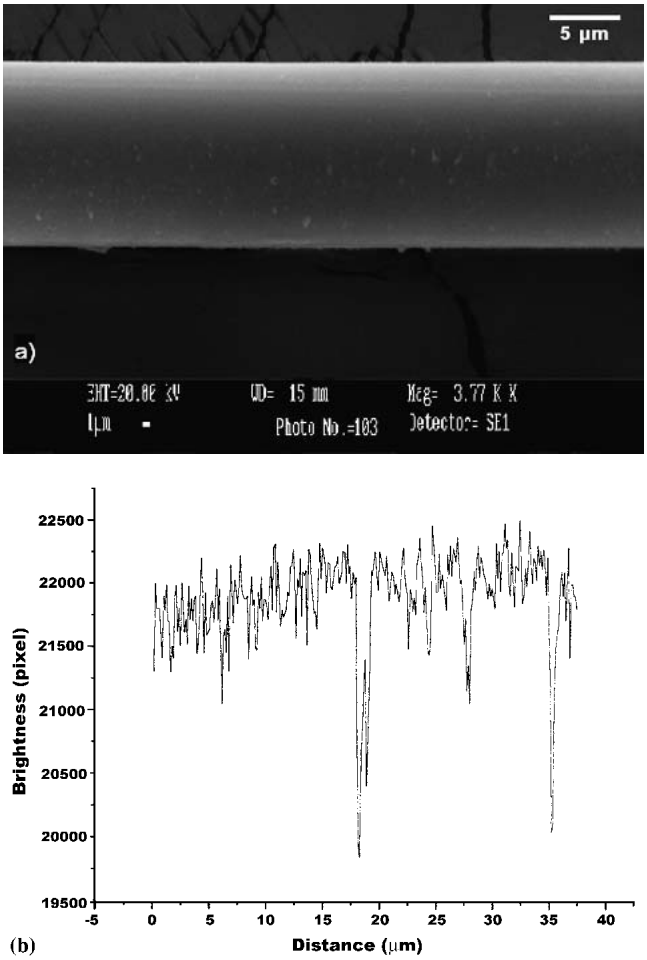


Fig. 7 Oxygen plasma treated PET fibers for 100 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

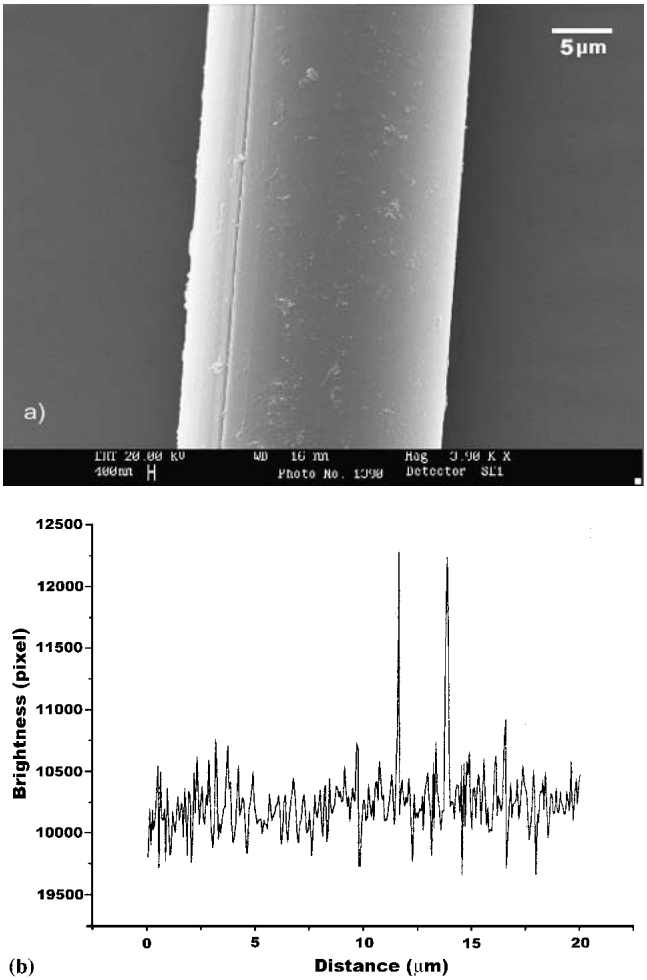


Fig. 8 Argon plasma treated PET fiber for 5 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

Table 2 Distance of Roughness Interval (D_{ri}) Values of Cold Plasma Treated PET Fibers

	Distance of Roughness Interval (D_{ri})	
Treatment Time	**Oxygen**	**Argon**
5 s	0.45	0.35
20 s	0.37	0.30
30 s	0.37	0.46
100 s	0.44	0.38

of roughness interval (D_{ri}) values are comparative data instead of absolute results and should be interpreted this way.

The untreated fibers, which presented the higher average tensile strength, also show the higher D_{ri} value, 0.55 µm. Figure 3(a) and (b) indicates the surface aspect and roughness profile for this initial condition.

For oxygen plasma treatment times 5 s, 20 s, 30 s, and 100 s, D_{ri} values were 0.45 µm, 0.37 µm, 0.37 µm, and 0.44 µm respectively, which indicate rougher surfaces compared with the untreated fiber surfaces. This is in accordance to the mechanical strength tendency shown in Table 1. With the increase in the treatment time from 5-20 s for both gases, a lower D_{ri}, associated with a rougher surface, was obtained. However, data analyses from Table 1 indicate an increase in the average tensile strength from 5-20 s treatment times for fibers cold plasma treated in oxygen and argon gases.

This behavior may be explained from the brightness versus distance curves in Fig. 4(b) and 5(b) for oxygen plasma treated fibers at 5 s and 20 s and in Fig. 8(b) and 9(b) for argon plasma treated fibers for the same treatment times. The main conclusion is that the surface roughness, which results from treatment time equal to 5 s, acted in a more effective way as stress concentration decreased the mechanical strength of the fibers. The same tendency is observed for argon plasma treated fibers. Differences in the distance of roughness interval (D_{ri}) for oxygen and argon plasma were associated with the mechanisms involved in the treatments: sputtering for argon plasma, and sputtering and etching for oxygen plasma.

Another important observation from surface analysis through SEM is that fiber surfaces after argon plasma treatment were attached in a more effective way than those treated using oxygen plasma.

The increases in the plasma exposure treatment time to 30 s

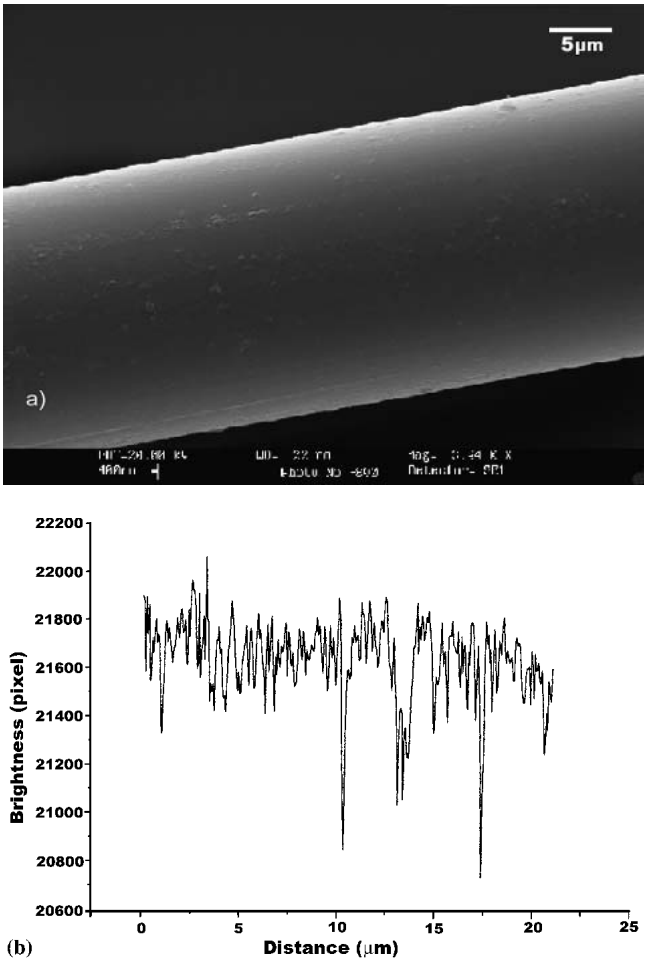


Fig. 9 Argon plasma treated PET fiber for 20 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

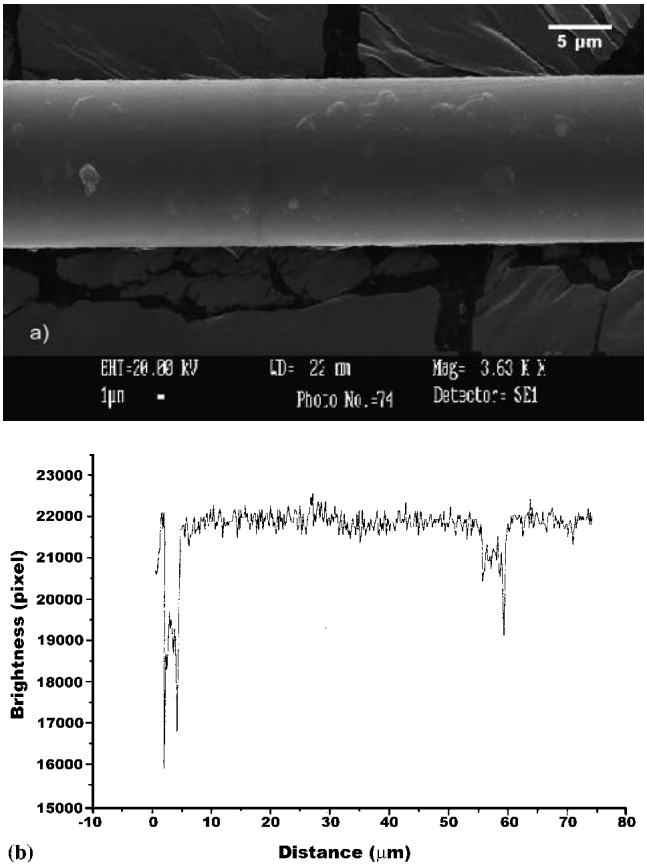


Fig. 10 Argon plasma treated PET fiber for 30 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

Table 3 Weibull Constants Calculated for the Linear Representation and Cumulative Distribution of the Tensile Strength Values. PET Fibers Treated in Cold Plasma Using Oxygen and Argon Gases as Fuel

	Linear Representation			Cumulative Distribution			σ Average,
Treatment	α	β	R	α	β	R	MPa σ_i
0″	6.0	1062	0.97	7.5	1050	0.97	998
O_2 100″	2.0	896	0.97	3.5	650	0.97	590
O_2 30″	5.6	841	0.88	9.5	815	0.91	747
O_2 20″	6.3	1057	0.98	7.0	1045	0.98	984
O_2 5″	8.3	900	0.98	7.5	890	0.98	858
A_r 100″	3.2	989	0.92	5.5	820	0.92	614
A_r 30″	4.2	889	0.94	6.2	770	0.96	539
A_r 20″	12.1	870	0.94	9.8	895	0.94	859
A_r 5″	5.0	810	0.98	6.4	880	0.98	819

and 100 s show D_{ri} values slightly higher in comparison to treatment times equal to 5 s and 20 s. In these conditions, degradation of fiber surface increased the width of roughness peaks and consequently decreased the number of roughness peaks in a linear unit, which resulted in higher D_{ri} values and lower average tensile strength, as indicated in Table 1.

For the Weibull distribution function, Table 3 indicates the shape (α), density (β), the correlation coefficient (R) calculated for the linear representation and cumulative distribution of the tensile strength values, and the average tensile strength (σ_i).

Considering the oxygen plasma treated fibers, variations in values of α are from 6.0, for untreated fibers, and increase to 8.3→6.3→5.6→2.0 for 5 s, 20 s, 30 s, and 100 s of treatment times, respectively.

The increase in α to 8.3 for 5 s of treatment time accompanied by a decrease in β is explained by the plasma effect on the fiber tensile strength. This means that some defects induced by plasma treatment on the fiber surface effectively perform as stress concentration. As a consequence, higher data concentration, reduction in the density of defects, and a decrease in the average tensile strength occurred.

The increase in treatment time to 20 s shows a decrease in α (6.3) and an increase in β (1057), which means higher density of defects and a larger range of experimental points. In fact, this occurrence was confirmed by the increase in the Sdt indicated in Table 1 and also by the reduction in the D_{ri} value in Table 2. This indicates a rougher surface, but as seen in Fig. 5(b), lower average roughness depth.

As a consequence of the increase in treatment time to 30 s

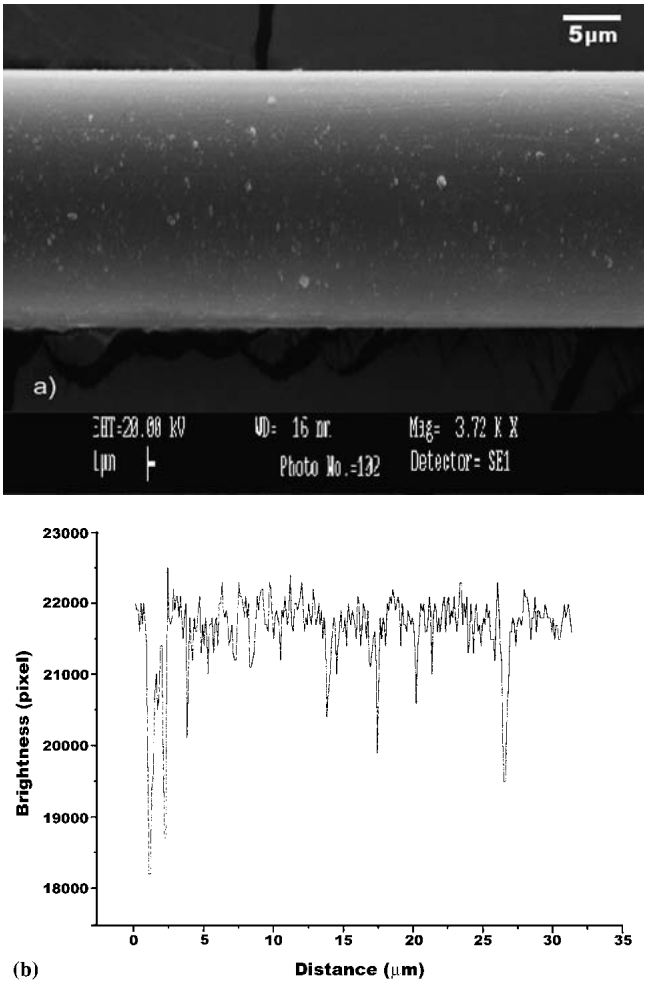


Fig. 11 Argon plasma treated PET fiber for 100 s: **(a)** SEM of the fiber surface; **(b)** roughness profile of the PET fiber

and 100 s, a reduction in α was observed, which represents a larger range of the experimental points and a significant density of defects, in accordance with the reduction of the standard deviation in these cases, shown in Table 1. The elevated exposure time to the plasma treatment produced fiber surface degradation through the appearance of effective defects providing a decrease in the fiber tensile strength and less concentration of experimental data, increasing D_{ri} values.

Taking into account the cumulative distribution, the same tendency of the linear representation was observed, excepting for 30 s, which provided higher α value. Due to the elevated exposure time to the plasma treatments, fibers were degraded, thus explaining the difference in α obtained in the cumulative distribution and in the linear representation.

The average tensile strength value of argon plasma treated fibers for 5 s showed a slight decrease in α and a strong reduction in the β parameter when compared with the untreated fibers. Nevertheless, given the larger range of experimental data, this condition presented a reduction of standard deviation indicated in Table 1, in accordance to the reduction of the density of defects, β. Table 2 also indicates a decrease in D_{ri} in comparison with the untreated fibers.

The condition that provided the smaller range of experimental data was that for 20 s of treatment time, which is in accordance with the lower Sdt presented. In this case the decrease in β, when compared with the untreated material, occurs due to the defects introduced by the treatment on the fiber surfaces. Average tensile strength for argon plasma treated fibers for 20 s increased slightly in comparison with the 5 s treatment time as a result of the minimization of stress concentration effects due to the higher exposure interval. This means that less depth roughness is obtained and that 20 s of treatment time almost completely removes the initial fibers' superficial layer.

The increase in treatment times to 30 s and 100 s is responsible for a decrease in α and an increase in β. The larger range of experimental data is confirmed by higher amount of defects acting to decrease the fiber tensile strength in accordance with the strong superficial degradation induced by the high exposure time to the plasma treatment.

Weibull analysis showed that argon plasma treatment at 5 s of exposure produces a decrease in the density of defects, β, introduced on the fiber surfaces, despite the large experimental data range. At 20 s of treatment time there is a significant experimental data concentration, α, and higher density of defects, β. For 30 s and 100 s, Table 3 shows smaller α and higher β. This statistical tendency is in accordance with the mechanical fiber disposition.

Table 3 shows differences in the behavior of α and β for oxygen and argon cold plasma treatments, regardless of the fact that the influence of treatment time on mechanical strength is the same for both gases. This phenomenon is related to differences in mechanisms associated with the cold plasma treatments: for oxygen gas, sputtering and etching, and for argon gas, only sputtering. Weibull parameters for the cumulative distribution are in accordance with the observations already stated.

Conclusions

- Oxygen and argon cold plasma treatments were responsible for lower average tensile strength values in comparison with the untreated fibers. Moreover, higher tensile strength reduction was observed for longer treatment times, for both environments.
- At 5 s of oxygen and argon plasma treatment times, a decrease of fibers' tensile strength in comparison with the untreated fibers occurred, which is associated with the stress concentration induced by the defects introduced on fiber surfaces during plasma treatment. A better mechanical performance is obtained after 20 s of treatment time, for both oxygen and argon cold plasma treatments, due to the minimization of stress concentration effects with the higher exposure times to plasma treatment. The increase in the treatment time to 30 s and 100 s, for both gases, resulted in a strong degradation of the fiber surfaces, which indicates that any attempt to establish a mechanical tendency would be unreal due to the high level of error.
- The distance of roughness interval (D_{ri}) was a parameter used to associate fiber surface condition, after cold plasma treatment, and average tensile strength. For oxygen and argon cold plasma treated fibers, D_{ri} is lower in compari-

son with the untreated fibers, which explains decreases in tensile strength. In some cases, higher roughness depth enhances concentration effects and influences mechanical behavior.

- Weibull parameters were permitted to confirm the stress concentration effect induced by the defects introduced on the fiber surfaces and to conclude that there were two different mechanisms acting during the plasma treatment, etching and sputtering.

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