A Study of Flex Fatigue Characteristics of Nylon 6.6 Tire Yarns and Cords

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ABSTRACT: Accelerated flex fatigue testing was carried out on nylon 6.6 tire cords, under various applied tensions (stress), at standard atmospheric conditions of 65% relative humidity and 21°C. Fatigue lifetime was measured and scanning electron microscope used to investigate the nature of failure. The study concluded that there was an exponential dependence of fatigue life on stress. The mode of failure depended on the tension applied, the underlying modes being those of kink band at lower stress levels and tensile failures as the stress level increased. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 75: 1045–1053, 2000

Key words: tire fatigue; tire reinforcement; gel-spun PE

INTRODUCTION

The demand for certain characteristics, such as high endurance, long-term reliability, and safety of automobiles, trucks, and aircrafts make fatigue analysis of tire cords an important consideration in predicting tire performance. Since tire cords are not only subjected to axial tension but also to bending during the flexing of the side walls of the tire, it is important to study their fatigue life to determine the degree of endurance in bending. At the side walls of a tire, cords should flex and recover readily. In respect to this, nylon 6.6 is superior to most tire-reinforcing materials.

In recent years a comprehensive study of flex fatigue of various fibers was carried out at UMIST laboratories.[1–3] Miraftab[1] investigated the flex fatigue of nylon 6 hosiery monofilament, high tenacity industrial nylon 6.6 and polyester fibers. From the tests performed at room temperature and 65% relative humidity, he concluded that the non-uniformity in structure and variability from fiber to fiber may cause rupture to occur at varying stress levels. He also postulated that individual values of fatigue life that were experimentally observed normally scattered over a fairly wide range of flexing cycles.

Sengonul and Wilding[2] investigated flex fatigue of gel-spun high-performance polyethylene fibers. Their results showed that with low tension, time for failure was too long and unacceptable abrasion damage was evident. At high tension, failure time was very short suggesting that the breaks might have occurred predominantly due to the direct effect of tension rather than due to true fatigue. Sengonul and Wilding[3] also investigated the flex fatigue in gel-spun high-performance polyethylene fibers at elevated temperatures. The study concluded that the fatigue life was inversely related to temperature and the underlying mode of failure was axial splitting.

This paper reports on the flex fatigue life of nylon 6.6 tire yarns and cords at different stress levels at standard atmospheric conditions of 21°C temperature and 65% relative humidity. The tests were designed to obtain data within a prac-

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Table I Tensile Properties of Samples

	Linear Density (tex)	Tenacity (N/tex)	Elongation (%)	Breaking Load (N)	Twists/cm[a]	
					Plied	Single
Yarn[b]	140	0.700	15.5	98.2	—	—
Raw cord	313	0.568	21.5	177.7	3.2S	3.1Z
Dipped cord	320	0.552	21.6	176.6	3.4S	2.8Z

[a] S and Z show the direction of twist.
[b] The yarn was made out of 210 filaments, each 28 μm in diameter.

tical time scale and therefore, the yarn and cord samples were subjected to high level of stresses compared to the level otherwise the tire cords would experience in a real life situation.

EXPERIMENTAL

Materials

The materials used for experimental purposes were 140 tex nylon 6.6 filament yarn, untreated cord (known as raw cord), and treated cord (known as dipped cord as the cords were dipped in resorcinol formaldehyde latex (RFL) resin). The raw cords were made from twisting two 140 tex yarns together and subsequently they were dipped. All samples were obtained from Du Pont from their Doncaster (UK) manufacturing plant. The basic physical properties of the samples are given in Table I.

Testing Method

The testing method was simply flexing over a pin. The sample was pulled backward and forward over a pin under tension (at various stress levels between 20% and 60% of the breaking stress) so as to cause the fibers to follow the curvature of the pin surface (Fig. 1). This is considered to be one of the simplest techniques available to assess flexing characteristics of fibrous materials.[4]

Experimental samples were flexed by repeatedly pulling the specimen backward and forward over a pin while they were under tension. The pin used had a diameter of 10 mm. To minimize friction, the pin was allowed to rotate freely as the sample was pulled over it. Tension was applied to the sample by hanging a weight on it. There was a tendency for the weight to rotate. To avoid this, a horizontal elastic string was attached to the weight at one end, and to a fixed surface at the other, as shown in Figure 1. The testing speed was 40 cycles per minute. Each cycle included one forward and one backward movement. The length of each specimen was 35 cm. The part in contact with the pin as the sample was pulled backward and forward was 7 cm. It was along this part where the specimen broke. Tensions of 106 N, 71 N, 53 N, and 35 N were used for raw and dipped cords. These weights were equivalent to 60%, 40%, 30%, and 20%, respectively, of the breaking load of the cords ($\approx$ 177 N). For the yarn samples, tension weights equivalent to the above percentages of yarn's breaking load ($\approx$ 98 N), were 59 N, 39 N, 29 N, and 20 N, respectively. These various values are referred to as percentage stress levels in this paper. The number of cycles required for samples to fail were recorded for each stress level. Failed samples were examined using the scanning electron microscope (SEM).

Prior to fatigue testing, all specimens were conditioned for 24 h at 65% relative humidity and 21°C.

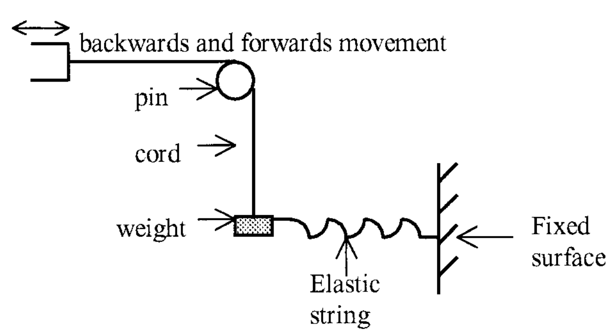

Figure 1 Schematic diagram of flex fatigue over a pin.

Table II Yarn Characteristics (Fatigue Life Cycles × 10^3)

	60% Stress	40% Stress	30% Stress	20% Stress[a]
Mean (average fatigue life)	0.490	3.211	19.522	40.070
Median	0.512	3.107	19.140	37.542
SD	0.174	1.420	5.710	13.099
CV (%)	35.7	44.2	29.2	32.7
Max endurance (cycles)	0.725	5.676	31.934	67.006
Min endurance (cycles)	0.215	1.555	12.642	24.397

[a] Percentage of stress, e.g., 60% stress, is the percentage of breaking load at which the sample was tested.

Table IV Dipped Cord Characteristics (Fatigue Life Cycles × 10^3)

	60% Stress	40% Stress	30% Stress	20% Stress
Mean (average fatigue life)	0.025	0.300	1.957	4.135
Median	0.026	0.279	1.918	3.444
SD	0.011	0.116	0.575	1.615
CV (%)	44.0	38.5	29.4	39.1
Max endurance (cycles)	0.045	0.501	3.234	7.225
Min endurance (cycles)	0.011	0.119	1.601	2.092

RESULTS AND DISCUSSION

Analysis of Experimental Results

As mentioned earlier, each type of yarn/cord was tested at four stress levels, the number of replications at each level being 10. For each stress level the mean, median, standard deviation, and coefficient of variation were calculated. All the calculated values of yarns, raw cords and dipped cords are given in Tables II–IV. Also given are the minimum and maximum numbers of endurance cycles of failure (fatigue life) for yarn, raw cord, and dipped cord. Unlike Miraftab's[1] observations (an obvious positive skewness in the scatter of the cycles), the results obtained here show that for 95% confidence limits of the population mean (Table V), the mean and the median lie within the limits. These limits were calculated using the formula

$$95\% \text{ conf. lim.} = \bar{X} \pm t_{n-1} \times (\text{SD}/\sqrt{n})$$

where $\bar{X}$ is the mean, n is the number of replication at each stress level ($n = 10$), $t_{n-1} = t_{0.025} = 2.262$, (obtained from the Critical Values of Student's t distributions),[5] and SD is the standard deviation. This suggests that the distributions are not very skewed (note: skewness shows the lack of symmetry in a set of data, e.g., if the mean $>$ median there is positive skewness; if mean $<$ median there is a negative skewness).

All the samples have their values of fatigue life scattered over a wide range of flexing cycles. This could be due to a number of factors, such as variability of twist numbers in the cords, contributing to nonuniform building up of stress in the cord structure. It could also be due to the nonuniformity in the microstructural levels of fibers where fatigue originates.

Table III Raw Cord Characteristics (Fatigue Life Cycles × 10^3)

	60% Stress	40% Stress	30% Stress	20% Stress
Mean (average fatigue life)	0.044	0.483	2.371	5.416
Median	0.043	0.499	1.945	4.399
SD	0.016	0.141	0.797	2.324
CV (%)	36.4	29.2	33.6	42.9
Max endurance (cycles)	0.072	0.701	4.016	10.632
Min endurance (cycles)	0.019	0.298	1.562	3.007

Table V 95% Confidence Limits of the Population Mean (Fatigue Life Cycles × 10^3)

	Limits	60% Stress	40% Stress	30% Stress	20% Stress
Yarn	Min	0.366	2.195	18.831	30.700
	Max	0.614	4.227	20.211	49.439
Raw cord	Min	0.035	0.382	1.801	3.754
	Max	0.053	0.584	2.941	7.078
Dipped cord	Min	0.007	0.112	1.027	1.522
	Max	0.043	0.488	2.887	6.74

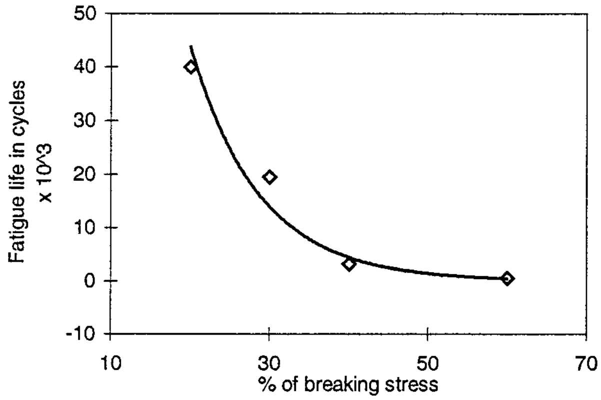

Figure 2 Stress–fatigue life relationship for yarn.

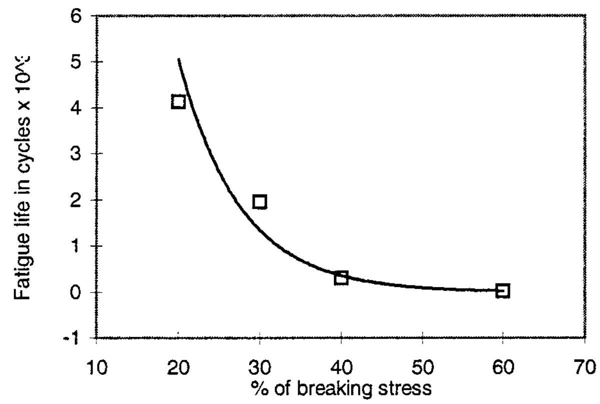

Figure 4 Stress–fatigue life relationship for dipped cord.

It is clear from the experimental data that as the stress increases, fatigue life decreases. This behavior is shown in Figures 2–4. As the stress level in the cord samples increases, the free volume between the filaments decreases, enhancing the level of abrasion between the filaments. Also, at higher stress levels (e.g. 40–60%) greater contact between cord fibers and pin surface accelerates the process of abrasion, causing an early failure of a sample (not necessarily due to fatigue).

Furthermore, it is clear from the observations that the dipped cords offer less resistance to fatigue, resulting in shorter lives as compared to their untreated counterparts. Available information in the published literature has shown that dipping stiffens the fiber structure and creates more microcracks and voids. These make the total structure more susceptible to an early failure. On the whole, the failure trends of the yarns are similar to that of the raw cords and dipped cords, although their fatigue lives are different (Figs. 2–4).

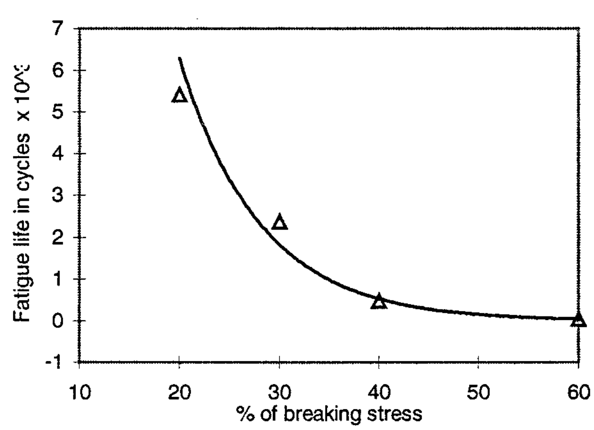

Figure 3 Stress–fatigue life relationship for raw cord.

To enable prediction of fatigue life of tire cords at any stress level, regression analysis of experimental data was used. Figures 2–4 clearly highlight that there is an exponential dependence of fatigue life on stress; therefore, variation of fatigue life can possibly be explained by an exponential equation of the type, $y = ae^{bx}$, where x is the stress level and y is the fatigue life.

To test this suggestion, the values of $\ln y$ were plotted against x in Figure 5. The resulting plots are reasonably linear, which confirms that the suggestion is reasonable. The equations of curves defining stress–fatigue life relationships for yarn, raw cord, and dipped cord are given in Table VI, where the values of a and b of the exponential equation, $y = ae^{bx}$, were calculated using least squares technique.

The coefficient of determination R^2, which measures the total variations of fatigue life that is explained by the equation, $y = ae^{bx}$, lies between 0.980 and 0.993, showing a very high dependence of fatigue life on stress. A perfect dependence is achieved when $R^2 = 1$. The values of R^2 were calculated using the Microsoft Excel computer program. This program enables the creation of graphs and also provides their equations and coefficient of determination.

SEM Observations

Scanning electron microscope examinations of failed regions of various samples were found to

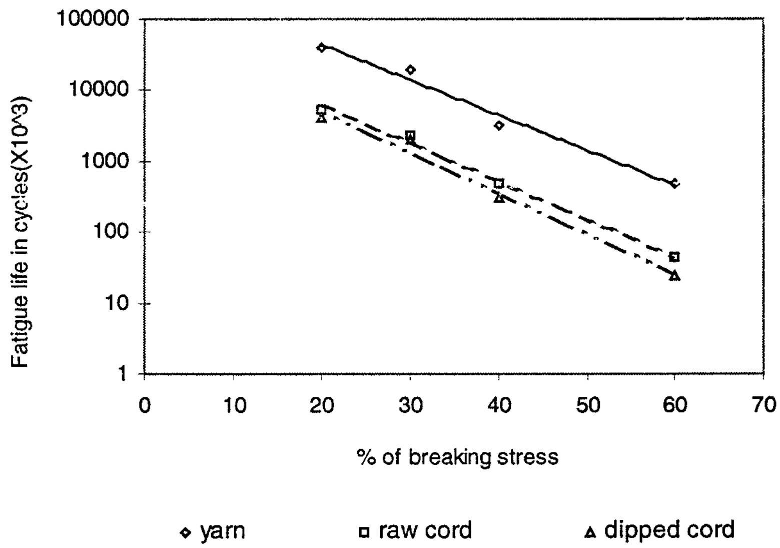

Figure 5 Testing the suggestion that the stress–fatigue life relationship is explained by equation $y = ae^{bx}$.

have considerable value in the evaluation of actual failure mechanism. Randomly selected failed raw and dipped cord samples were analyzed under SEM to develop a qualitative view of the general pattern of creep failure. Yarn samples could not be examined due to technical problems encountered with the failed samples (on failure the filaments at the broken ends became entangled and curled up). All observations were recorded at 60× magnification. Figures 6–8 illustrate various fractures of failed samples at 20%, 40%, and 60% stress levels, respectively. Major features observed in failed samples are (a) axial splitting, (b) abrasion and peeling, (c) kink bands, (d) brittle fracture, and (e) high-speed breaks.

Looking at the failure characteristics and the experimental variables, it is somehow reasonable to class the failure behavior into two categories:

Table VI Equations Defining Stress-Fatigue Life Relationships and Their Coefficient of Determination

	Equation of Curve	Coefficient of Determination (R^2)
Yarn	$y = 430.45e^{-0.114x}$	0.980
Raw cord	$y = 74.28e^{-0.124x}$	0.993
Dipped cord	$y = 71.28e^{-0.132x}$	0.986

1. Fatigue fracture failure, normally occurring at low stress level (e.g., 20–30% of breaking stress), and
2. Tensile fracture failure, normally occurring at high stress level (e.g., 40–60% of breaking stress).

Failure of a specimen occurs due to a combination of reasons. Apart from the stress level, the actual failure is also significantly influenced by the physical state of the specimen (e.g., dipped cords were more prone to tensile failure than raw cords, as they were more stiff and brittle). Perhaps it is important to mention here that semicrystalline systems (such as tire yarns and cords) can continue to yield and reorganize with stress application up to large deformations, which otherwise can be called creep,[6] before fracture initiates and failure occurs.

At low stress levels, (especially when the polymer is at a glassy state), localized plastic deformation can occur by the formation of void network[7] with molecular segments of fibrils oriented in the direction of the principal stress. This is why axial splitting is a common fracture type, with low-stress failed samples. Also kink banding in cords is seen as an important feature of failure, which is caused by combination of the presence of shear stress component arising from twist, and a compressive stress component, arising from bending deformation. From the micrographs, it is evi-

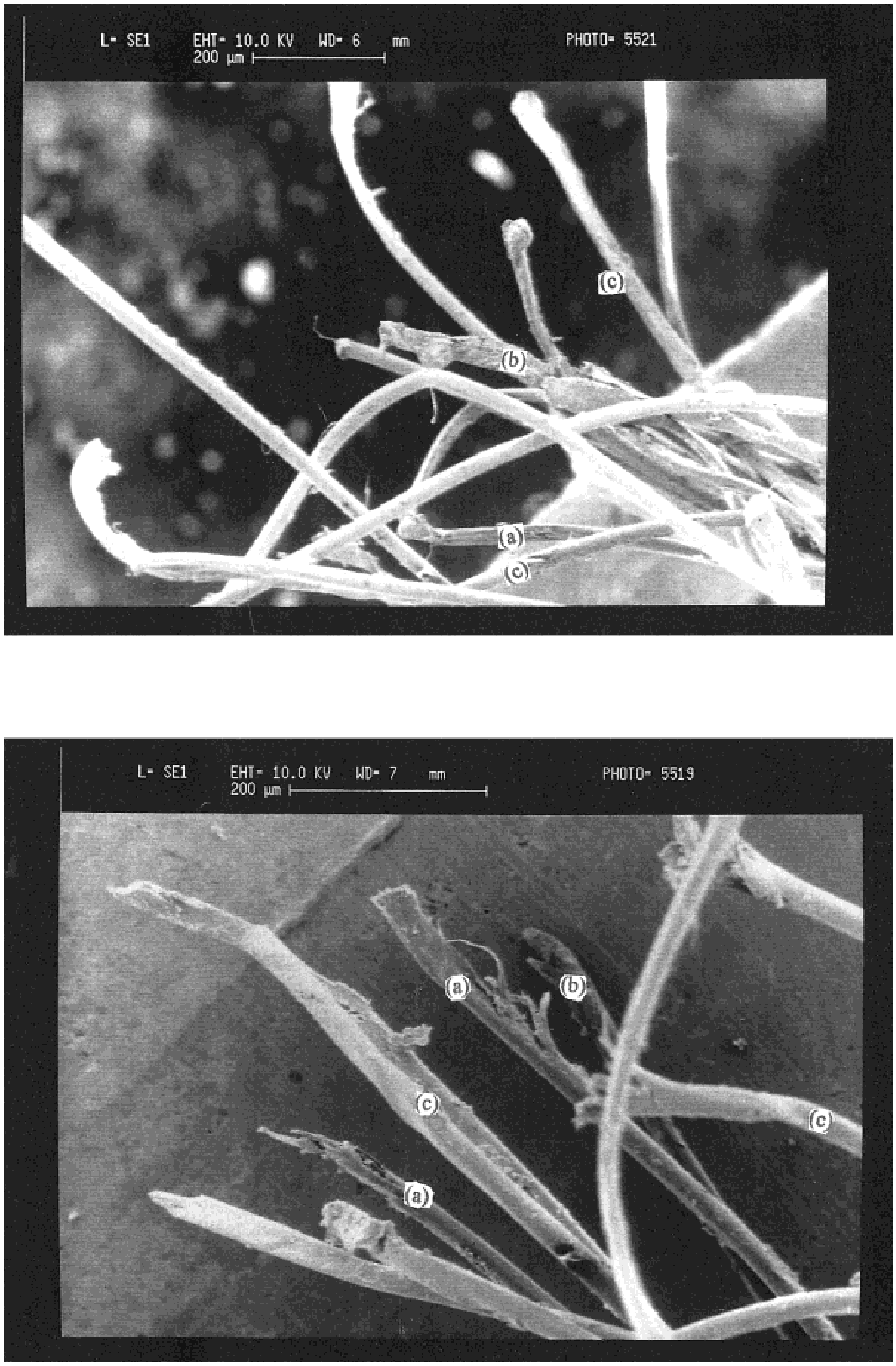

Figure 6 SEM observations of raw cord (top) and dipped cord (bottom) at the 20% stress level: (a) axial splitting; (b) abrasion and peeling; (c) kink bands.

dent that at a low stress level, kink bands appear on the surface, followed by a region that may show axial split lines with surface roughness and peeling. The possible reason for surface roughness and peeling come from abrasion between cord surface and the pin.

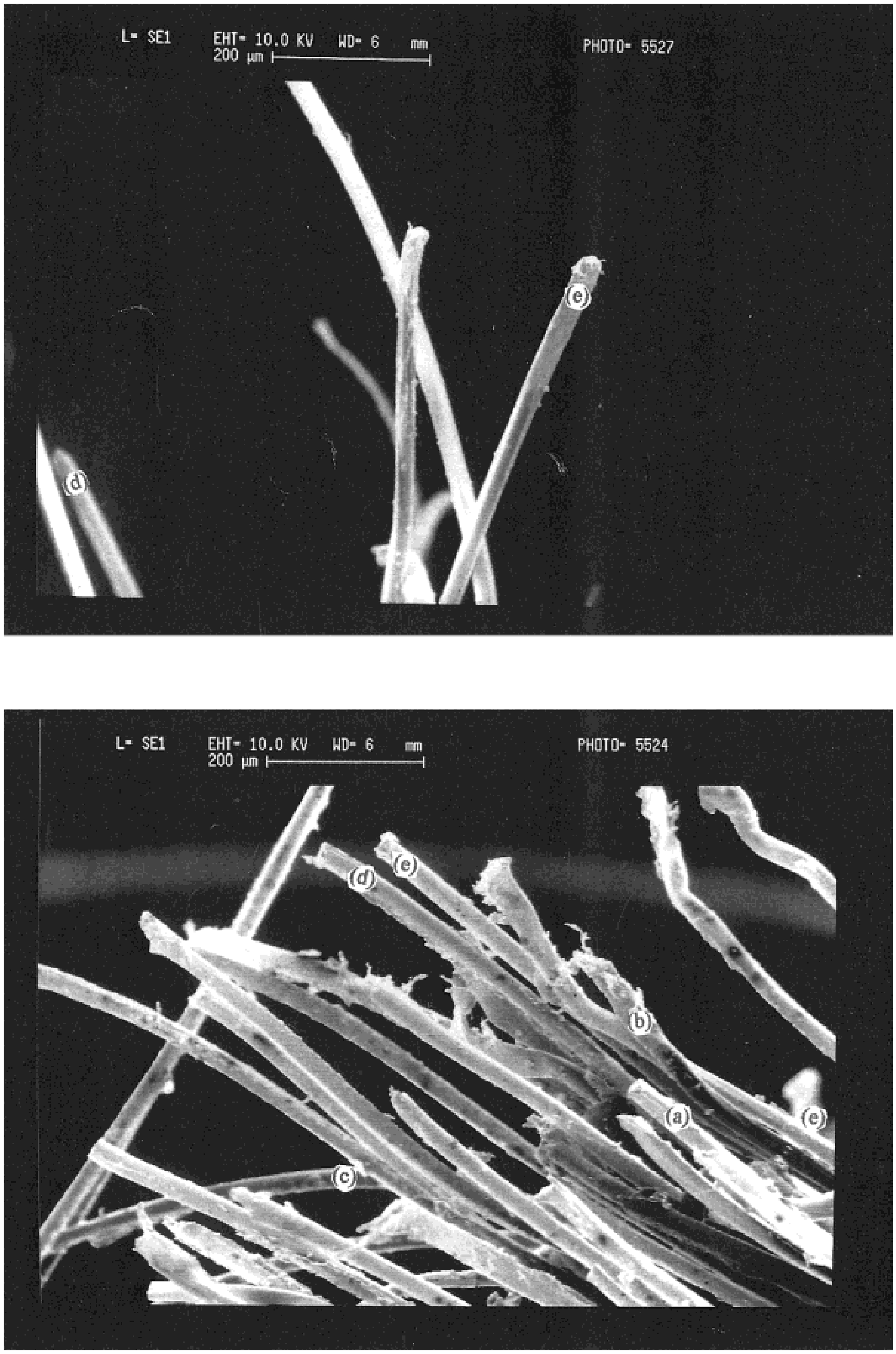

Figure 7 SEM observations of raw cord (top) and dipped cord (bottom) at the 40% stress level: (a) axial splitting; (b) abrasion and peeling; (c) kink bands (d) brittle fracture; (e) high-speed breaks.

At a high level of stress, tensile fracture failure is predominantly seen through high-speed breaks and axial splitting and peeling. Kink bands are virtually nonexistent. At a high level of stress, tensile fracture occurs, starting with a sudden breakdown of a void or a defect, often leading to a

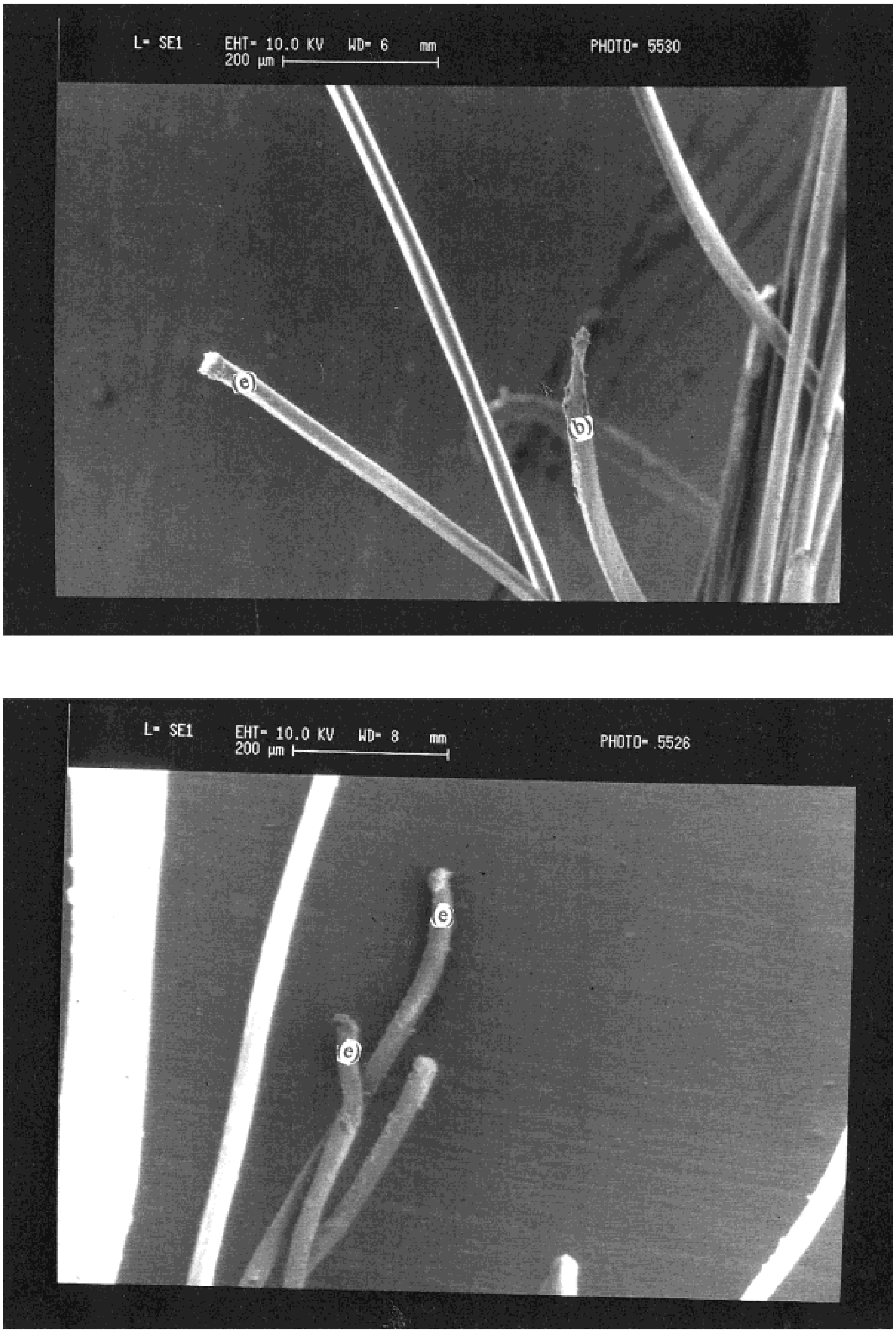

Figure 8 SEM observations of raw cord (top) and dipped cord (bottom) at the 60% stress level: (b) abrasion and peeling; (e) high-speed breaks.

catastrophic failure. It is known that in a polymeric system high levels of stress can bring about the earlier onset of crack initiation leading to poor resistance to fatigue fracture.[7]

Finally, in the dipped cord, the presence of rubbery component dispersed on the surface in the form of particles can lead to earlier crack initiation, resulting in a reduction in fatigue life cycles.

CONCLUSION

There was a pronounced decrease in fatigue life as applied stress increased. There is no doubt that twisting and dipping the cord in RFL resin has a significant effect on fatigue life. Regression analysis has shown that there is an exponential dependence of tire cord fatigue life on applied stress.

The SEM observations showed that all specimens experienced more than one kind of mechanisms leading to failure. However, further extensive experiments on controlled samples are necessary to develop a better understanding of failure mechanisms of tire cords.

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