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Effect of Internal Structure and Local Defects on Fiber Strength

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INTRODUCTION

The strength of fibers from high polymers has received considerable attention during the past thirty years. Past work clearly demonstrates that fiber strength is strongly influenced by such structural factors as the polymer from which the fiber is made, the degree of molecular orientation, and the crystallinity. Alfrey,[1] for instance, has discussed the relationship between fiber tenacity and molecular orientation. It is a well-known fact that drawn, crystalline, oriented, polymeric fibers are many times stronger than randomly oriented samples from the same polymer. The importance of molecular orientation in such synthetic fibers as nylon and polyethylene terephthalate, is well-known and so is that in natural fibers.[2,13]

In addition to these internal structural factors, there is the inevitable fact that fibers break at their weakest point, and so fiber tenacity is expected to be influenced by the severity and distribution of local defects along the length of filaments.

Many investigators have made use of strength measurements to characterize the level of defects in fibers. Attempts have been made to rate local defects by studying the effect of test specimen length and of sample history on fiber tenacity.[4–7]

It is certain that both the internal fiber structure and local defects influence fiber tenacity. However, the relative role of these two factors in the well-known fibers is not generally understood. In the present work it is shown that, in the case of nylon 66 and polyethylene terephthalate fibers, tenacity is controlled by either of these two factors, depending on the temperature of measurement, but that at no temperature is the tenacity substantially controlled by both. In particular, the following has been shown:

1. Fiber tenacity at relatively high temperatures (depending on the fiber) is controlled by the internal fiber structure; the character of local defects is of only minor importance. These internal structural factors presumably include the fiber molecular orientation, the crystallinity, and the presence of second-order transitions at certain temperatures.

2. At very low temperatures (depending on the fiber) the severity of local defects, and not the overall structure of the fiber, controls the fiber

strength. Furthermore, at the low temperatures the fiber tenacity is independent of the actual temperature of measurement and is an unambiguous measure of the defect severity.

Particularly interesting is the fact that at very low temperatures the fiber strength measurements were extremely dependent on the test specimen length, whereas little dependence was found in measurements at relatively high temperatures. Also, analysis of the strength measurements at low temperatures indicated that the tenacity data are often distributed about two most probable values; i.e., a bimodal distribution of defects was observed. In contrast, tenacity measurements at relatively high temperatures showed only a monomodal distribution, indicating the presence of only one strength-controlling mechanism.

EXPERIMENTAL PROCEDURE

In this work stress–strain properties of single filaments were measured at an elongation rate of 10%/min. and at temperatures ranging from about 250 to −196°C. Samples were preconditioned at 21°C. and 65% r.h. The test temperature was established by placing filaments in various liquid baths whose temperatures were accurately known. At −196 and −186°C. the filaments were submerged in liquid nitrogen and argon, respectively. At somewhat higher temperatures various liquid hydrocarbon baths were used, at their melting points and in equilibrium with the solid phase. At room temperature and above, the filaments were placed in a silicone oil bath which was preheated to the desired temperature. Table I summarizes the baths and the cooling agent used. The tensile properties of samples were shown to be insensitive to the hydrocarbon and silicone baths by room temperature measurements.

TABLE I
Control of Temperature

Temp., °C.	Bath	Cooling agent	Method of maintaining temp.[a]
−196	Liquid N_2	Liq. N_2	BP
−186	Argon	Argon	BP
−161	2Me-butene	Liq. N_2	MP of bath
−130	*n*-Pentane	Liq. N_2	MP of bath
−94	*n*-Hexane	Liq. N_2	MP of bath
−90	*n*-Heptane	Liq. N_2	MP of bath
−70	*n*-Heptane	Acetone–Dry Ice	SP of Dry Ice
−31	*n*-Decane	Acetone–Dry Ice	MP of bath
0	*n*-Heptane	Ice water	MP of water
21	Air	—	Room conditions
Above 21	Silicone oil	—	Heat control

[a] BP, boiling point; MP, melting point; SP, sublimation point.

All specimens were mounted at room temperature and then submerged in a bath at the desired temperature. It was generally found that at temperatures substantially above the second-order transition temperature T_g, a small "shrinkage force" developed before the actual test was begun. Figure 1 illustrates the shapes of typical stress–strain curves obtained below T_g (Fig. 1a) and above (Fig. 1b). The small shrinkage force found at temperatures above T_g was subtracted from the stress–strain data; that is, the dashed line in Figure 1b was taken as zero force for computing all tensile properties.

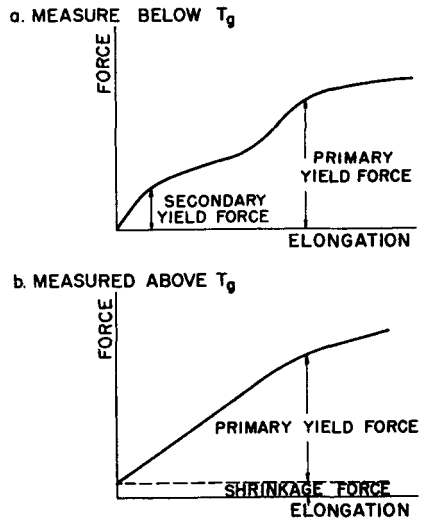

Fig. 1. Typical stress–strain curves, nylon 66 and polyethylene terephthalate.

INFLUENCE OF INTERNAL FIBER STRUCTURE ON STRENGTH

In the case of the fibers studied, the tenacity at relatively high temperatures was controlled by the rupture of the internal structure of fibers and not by the simple propagation of localized defects along the length of the filaments. In demonstrating this fact it is helpful to make use of two stress–strain properties, the initial modulus and the "primary yield force," which are discussed below.

Relationship of Strength to Primary Yield Force

Figure 1 illustrates typical stress–strain curves obtained at relatively high temperatures for the nylon 66 and polyethylene terephthalate. The stress–strain curves show two pronounced yield points. The first yield point is designated "the secondary yield point." The force at this point is moderately dependent on temperature and the yield point vanishes at temperatures somewhat above the second-order transition temperature of the fiber, ca. 380°K. for nylon 66 and polyethylene terephthalate.

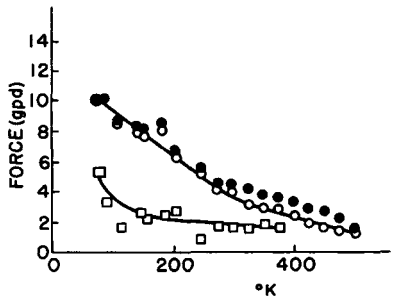

Fig. 2. Effect of temperature on stress–strain properties of a nylon 66 fiber: (O) primary yield force; (□) secondary yield force; (●) actual tenacities.

The yield point at higher elongations, Figure 1, has been termed the "primary yield point;" this yield point persists even at temperatures approaching the crystalline melting point.

At all temperatures the fiber tenacity is but slightly greater than the primary yield force. This is shown in Figures 2 and 3 by the fact that the open circles lie only slightly below the closed circles at all temperatures. Since little force is developed after the primary yield point, the mechanism which controls the primary yield phenomenon also controls the fiber strength, for all practical purposes.

When a fiber is elongated to the primary yield point, it yields over its entire length and not at some local point on the sample. This can be seen from the fact that single filaments may exhibit large amounts of elongation after the primary yield point. This elongation may be as great as 30% for some drawn nylon fibers at temperatures near the second-order transition temperature, 70°C. If the yielding were associated with only a few local points along the fiber length, the sample would have to "neck down" severely at the isolated points to produce this large amount of elongation; but this does not happen. Filaments that break at elongations past the primary yield point break sharply across the diameter without any evidence of necking down.

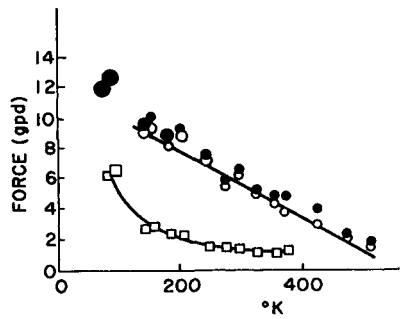

Fig. 3. Effect of temperature on stress–strain properties of a polyethylene terephthalate fiber: (O) primary yield force; (□) secondary yield force; (●) actual tenacities.

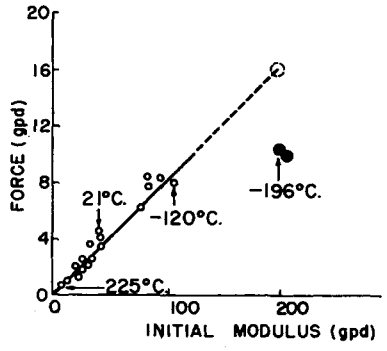

Fig. 4. Relationship between primary yield force and initial modulus for a nylon 66 fiber: (●) tenacity; (O) primary yield force. The dotted circle represents the theoretical yield force for modulus of 200 G.D.P.

Clearly, the primary yield force is controlled by the disruption of the internal structure of the fiber and not by the character of any local defect. As discussed earlier, the primary yield force comprises most of the fiber strength, which means that the break tenacity is controlled by the same internal structural mechanism.

Relationship of Primary Yield Force to Initial Modulus

The initial fiber modulus was taken as the initial slope of stress–strain curves illustrated in Figure 1. It will now be shown that temperature has virtually the same effect on both the primary yield point and the initial modulus.

The fact was demonstrated by plotting the primary yield force (measured at any one temperature) against the initial modulus (measured at the same temperature). As shown by the open circles in Figures 4 and 5, the primary yield force is approximately proportional to the initial modulus for the nylon 66 and polyethylene terephthalate fibers.

The slope of the line relating modulus to the primary yield force is not constant for a given polymer but varies widely with fiber processing con-

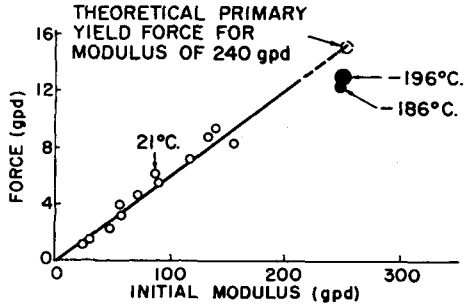

Fig. 5. Relationship between primary yield force and initial modulus for a polyethylene terephthalate fiber: (●) tenacity; (O) primary yield force.

ditions. In Figure 6, for instance, greatly different slopes are shown for three nylon 66 fibers.

In summary: The slopes of the lines in Figures 4, 5, and 6 are the primary yield force per unit modulus and are important in a detailed analysis of the effect of fiber structure on fiber tenacity as suggested by the following: (*1*) The fiber tenacity is virtually equal to the primary yield force (Figs. 2 and 3). (*2*) The primary yield force is linearly related to the fiber modulus, measured at various temperatures (Figs. 4, 5, and 6); the slope of the line relating them is a temperature-independent parameter of practical importance, determined by the internal structure of the fiber, and not by the defect character of the sample. (*3*) The remainder of such an analysis of fiber strength would consist of an interpretation of the modulus–temperature curve, which is basically a simpler problem than the initial one and is much better understood. For instance, the effects of crystallinity and orientation on fiber modulus are discussed by Charch and Moseley.[8] In addition, the effect of second-order transitions on the relationship between

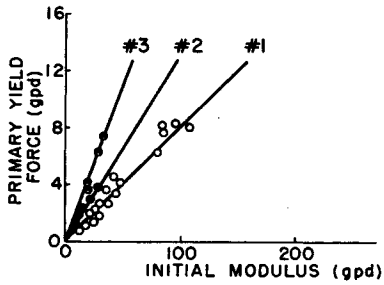

Fig. 6. Relationship between primary yield force and initial modulus for three different nylon 66 fibers.

modulus and temperature for many polymers has been extensively discussed.[9–12] Most of the studies reported in the literature make use of the dynamic modulus rather than the initial fiber modulus measured in a one-directional tensile test. However, a similar interpretation should apply to the effect of temperature on the initial stress–strain modulus.

Influence of Local Defects on Fiber Strength

When the test temperature was lowered to some low value (between room temperature and −100°C.), the fiber tenacity became *constant* with further decreases in temperature down to −196°C. in the case of all fiber studies. This temperature-independent tenacity, at relatively low temperatures, was found to be controlled by the propagation of local defects in the fiber samples.

In demonstrating the influence of local defects on tenacity it is helpful to make use of the plots in Figures 4, 5, and 6, which show a linear relationship between the primary yield force and the initial modulus measured at

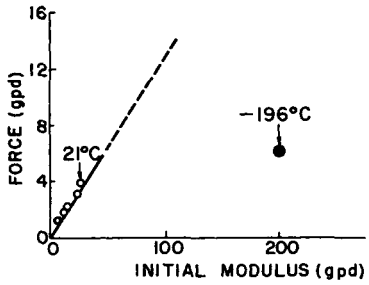

Fig. 7. Relationship between primary yield force and initial modulus for an 8-mil nylon 66 monofil: (●) tenacity; (O) primary yield force.

various temperatures. The lines drawn through the open circles in Figures 4 and 5 are dashed beyond the data. This portion of the curve represents the expected relationship between the primary yield force and the initial modulus at temperatures below −100°C.

Actual measurements at −196°C. show a modulus of about 200 g./denier (gpd) for nylon 66 and about 240 gpd for the polyethylene terephthalate fiber. As indicated in Figures 4 and 5, the expected primary yield force at these moduli is about 16 gpd, for both fibers. However, when actual measurements were made at −196°C., the fibers broke before any primary yield point was exhibited, and the tenacities were less than the expected primary yield force. This is indicated by the solid points in Figures 4 and 5 which show that the nylon fiber broke at about 9 gpd for the polyethylene terephthalate fiber broke at about 13 gpd for measurements at −196° and −186°C. Similar data are shown in Figure 7 for an 8-mil nylon monofil.

In the experiments described below, it is shown that local defects along the length of the fiber samples cause them to break at forces less than the expected primary yield force when they are stretched at low temperatures. To study the influence of defects on the fiber strength, various imperfections were introduced into single filaments in a controlled manner, and the stress–strain properties were measured at several temperatures.

First of all, nicks, 1 mil deep, were cut in the 8-mil nylon monofil (Fig. 7) and the stress–strain properties were measured at certain temperatures. The results are indicated by the solid triangles in Figure 8. At −196°C.

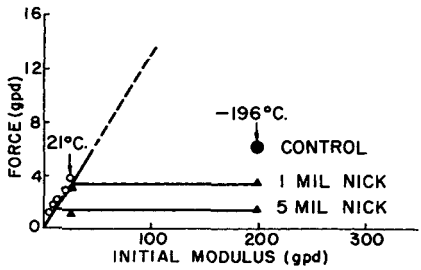

Fig. 8. Effect of nicks on the breaking behavior of an 8-mil nylon 66 monofil: (●) tenacity; (O) primary yield force; (▲) tenacity, nicked sample.

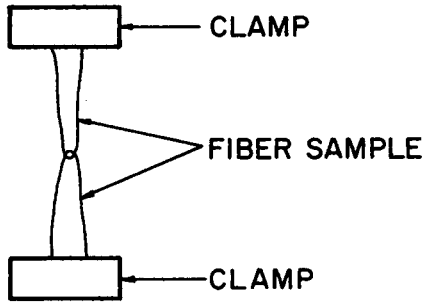

Fig. 9. Schematic of loop test.

the tenacity dropped from about 6 gpd to about 3 gpd. The tenacity at 21°C. dropped only slightly. The nicked sample broke shortly before the primary yield point in the room temperature measurement, and the strength was constant between room temperature and −196°C. for the nicked sample. At temperatures above 21°C. the nicked sample broke after the primary yield point; the primary yield force and the initial modulus were identical to the control at these higher temperatures.

Figure 8 shows an analogous result when a 5-mil nick was placed in the monofil. In going from the 1-mil nick to the 5-mil nick the room temperature tenacity and the tenacity at −196°C. were lowered by the same amount, and in both cases the tenacity was constant between 21 and −196°C.

The essential conclusions from this experiment are that (*1*) the tenacity at low temperatures is sensitive to defects and is independent of the actual temperature of measurement and (*2*) the high-temperature tenacity is controlled by the primary yield force which is not affected by defects. Clearly, the temperature at which the strength-controlling mechanism changes is dependent on the severity of the defects in a fiber with a given internal molecular structure. With the 1-mil nick, defects were operative between 21 and −196°C.; with the 5-mil nick, between 200 and −196°C.

A second technique was used to cause the sample to break via defect propagation. In this case the same nylon monofil was simply tested in a loop arrangement illustrated in Figure 9. An examination under the

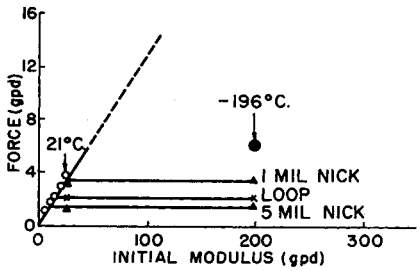

Fig. 10. Effect of loop and nicks on the breaking behavior of a nylon 66 monofil.

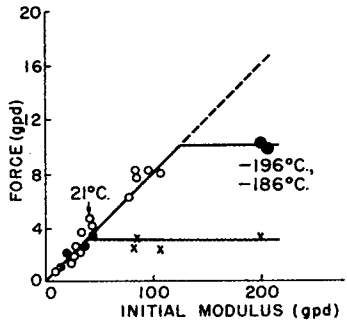

Fig. 11. Effect of loop on the breaking behavior of nylon 66 single filament: (O) primary yield force, control; (●) tenacity, control; (⊗) primary yield force, loop test; (×) tenacity, loop test.

microscope revealed that defects were actually formed at the loop when samples were stressed by a very small amount, ca. 1 or 2 gpd. When samples were further stressed in the loop test, they invariably broke at this defect. The loop data for the monofil are compared, in Figure 10, with the nick and control data. The loop and the nick data indicate a constant tenacity at 21 to −200°C., even though the modulus increased substantially. The defect-controlled tenacity for the loop test lies between the value for the 1-mil nick and the 5-mil nick.

In the case of single filaments of very small diameters, it was difficult to introduce nicks in a reproducible fashion. However, it was possible to study the influence of defects in small single filaments with the loop test described above. Figures 11 and 12 show the control and the loop data for the nylon 66 and the polyethyleneterephthalate samples discussed earlier. At low temperatures (high modulus), the loop strength (indicated by X) was substantially lower than the control strength (solid circles), and was virtually insensitive to the temperature of the measurement. However, the primary yield force at higher temperatures (lower modulus) is identical for the control and loop tests, as indicated by the circled X's in Figures 11 and 12.

If the low-temperature tenacity is a measure of defect severity, it must

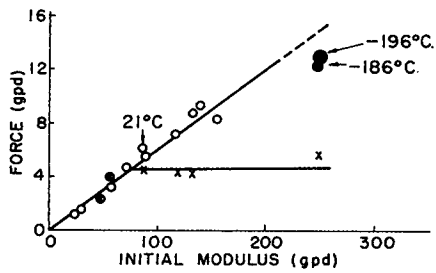

Fig. 12. Effect of loop on the breaking behavior of a polyethylene terephthalate single filament: (O) primary yield force, control; (●) tenacity, control; (⊗) primary yield force, loop test; (×) tenacity, loop test.

certainly decrease regularly when a fiber is subjected to progressive amounts of mechanical damage. To illustrate this fact, single filaments of a polyethylene terephthalate fiber (Fig. 12) were placed in the IBM electric typewriter and were repeatedly hit with the letter *l* to produce a small flat spot on the filament. This situation looked something like the following:

The typewriter was set to produce only a small amount of damage for each hit. Single filaments were hit a varying number of times (100, 500, 1000 times) and in each case the stress–strain properties of nine filaments were measured both at 21 and at −196°C. The actual fatigue operation was conducted at 21°C. in all cases.

The defect tenacity (−196°C.) decreased regularly with an increasing number of hits, while the tenacity at 21°C. remained unchanged. This is shown by the solid circles in Figure 13, in which tensile measurements made on both the fatigued and control specimen are compared. Considering the above results, it seems proper to call the low-temperature strength of fibers and "defect tenacity."

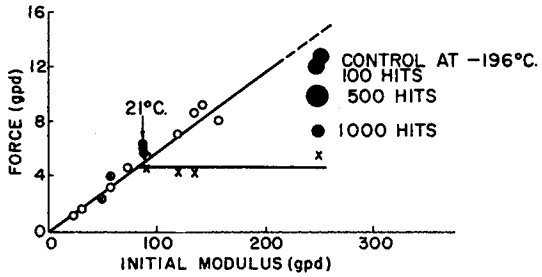

Fig. 13. Effect of typewriter damage on the breaking behavior of a polyethylene terephthalate single filament: (O) primary yield force, control; (●) tenacity; (⊗) primary yield force, loop test; (×) tenacity, loop test.

The fact that the defect tenacity is independent of the test temperature should be indicative of the mechanism by which defects propagate and cause fiber failure. This insensitivity to temperature may be rationalized in the following way. For a relatively small force on the filament, the tension at the apex of a crack or nick will be very high owing to the concentration of stresses.[13] Indeed, for a relatively small force on the filament it is entirely conceivable that the stress at the apex of a crack will approach the strength of molecular chains, and may therefore lead to molecular chain rupture.

It is a well-known fact that the strength of a polymer molecular chain does not vary appreciably over the temperature range of absolute zero to room temperature. This means that if fiber breakage is controlled by the

rupture of molecular chains, the fiber tenacities should be independent of temperature over the range of temperatures under consideration.

SUMMARY OF STRENGTH-CONTROLLING MECHANISMS

When the stress–strain test temperature is varied from near 200°C. to near room temperature the internal structure of fibers stiffens, as evidenced by an increase in modulus. Associated with this stiffening is an increase in the primary yield force and, as a consequence, the fiber strength increases.

When the temperature is decreased to very low values, the filaments stiffen greatly and at sufficiently low temperatures the primary force is greater than the force required to propagate the most severe local defect, the latter force being independent of temperature. This is true regardless of whether the most severe defect is a nick, a defect formed in a loop test, or a flat spot introduced by hitting the filament. The extent of damage at the local defect determines the fiber tenacity at low temperatures. For this reason the low temperature tenacity is called the "defect tenacity."

In one sense the molecular structure may actually influence the low-temperature strength, and the statement that the low-temperature tenacity is controlled by defects and not by the internal fiber structure may depend on the frame of reference used in describing defects. The low-temperature strength is clearly a measure of the force required to propagate defects and is not a measure of the actual size of a local imperfection. It is quite likely that fibers with the same geometrical defects, but with different internal structures, may have different low-temperature strengths. In this sense the internal molecular structure may influence the low-temperature tenacity.

EFFECT OF TEST SPECIMEN LENGTH ON TENACITY

In a study of the effect of test specimen length on fiber tenacity, the stress–strain properties of single filaments of a severely damaged nylon fiber were measured at 1-in. test length and at 3.5-in. test length. Measurements were made at both room temperature and −196°C.

There was no significant effect of test specimen length on the primary yield force at room temperature. Both the test lengths 1 and 3.5-in. gave a primary yield force of 6.1 ± 0.2 gpd.

In contrast, measurements at −196°C. showed a substantial dependence on test length. Samples tested at 1-in. lengths had an average defect tenacity of 12 gpd for 15 observations. The average defect tenacity for the 3.5-in. test length was 6.5 gpd.

Before these tests were conducted, the defect character of the sample was unknown. As a result of the measurements at −196°C. it was clear that the fiber sample had severe defects at an average interval greater than 1 in. along the length of the filaments.

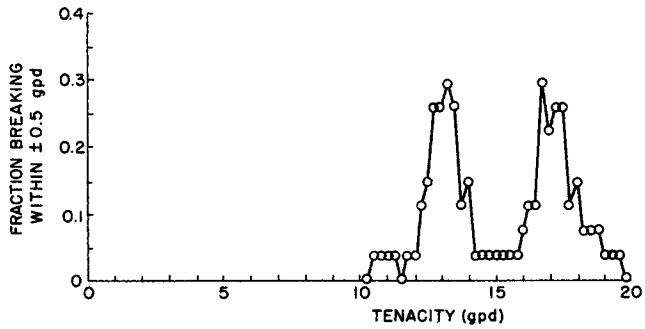

Fig. 14. Distribution of tenacity measurements at −196°C. for an undamaged nylon 66 filament, unsmoothed data.

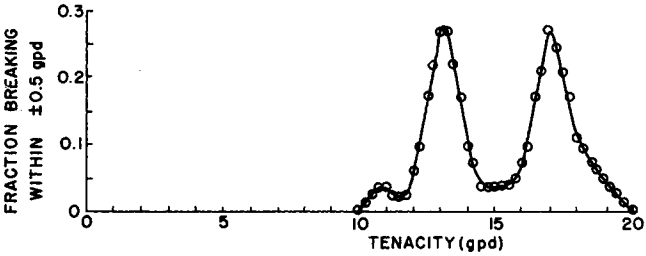

Fig. 15. Data of figure 14, smoothed.

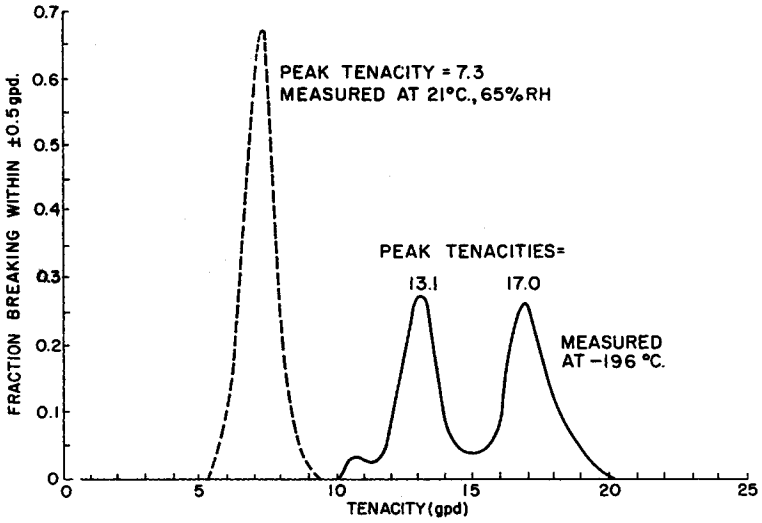

Fig. 16. Comparison of tenacity distribution of a nylon 66 single filament measured at 21°C. and −196°C.

DISTRIBUTION OF LOCAL DEFECTS IN FILAMENTS

The distribution of defects in various nylon filaments was studied at a constant sample length of 3.5-in. by plotting the fractional number of filaments that broke at a given tenacity (within ±0.5 gpd) against that tenacity measured at −196°C. Distribution curves of the type shown in Figure 14 were obtained. Forty to fifty single-filament tests had to be made in order to obtain an experimentally significant distribution curve.

The data shown in Figure 14 were averaged to obtain the smooth curve shown in Figure 15. As an illustration of this averaging process, the number of breaks at 12.75 ± 0.5 gpd, the number of breaks at 13.00 ± 0.5 gpd, and the number of breaks at 13.25 ± 0.5 gpd were all averaged, and this average was plotted at 13.0 gpd. This process was carried out for various tenacity values and gave the smooth curve shown in Figure 15.

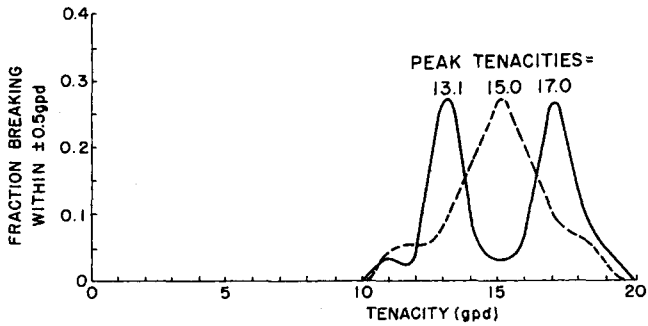

Fig. 17. Defect tenacity distribution for two different nylon 66 single filaments measured at −196°C.

This bimodal distribution curve clearly indicates that about half the samples had a strength near 13 gpd and the remaining half had a strength near 17 gpd. In contrast, the distribution curve for room temperature measurements shows a monomodal distribution, indicating that breakage was controlled by a single mechanism (Fig. 16).

It was found that defect-tenacity distribution curves varied widely among otherwise similar yarn samples from the same polymer. As an illustration, the bimodal distribution curve of Figure 16 is compared with a monomodal distribution curve in Figure 17. These samples were prepared in the same manner and were very similar in their room temperature mechanical properties. Neither sample received any specific damage treatment.

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Synopsis

Tensile measurements on fibers from nylon 66 and polyethylene terephthalate have shown that the fiber tenacities at relatively high temperatures (about 250 to −100°C.) are controlled by the internal molecular structure of the fiber and not by any local defect. While it is true that fibers always break at some local defect, the actual strength was determined by a yielding of the molecular structure which occurred before fiber breakage. In contrast, the tenacity at low temperatures (generally −100 to −196°C.) was controlled by local defects which propagated under stress, causing fiber failure before the internal molecular structure exhibited any yield. The temperature at which the mechanism changed was dependent on the severity of local defects and on the internal molecular structure as well. The low-temperature tenacity was found to be independent of the actual temperature of measurement, and this constant level of tenacity, at low temperatures, was shown to be a measure of the severity of the most critical local defect in the fiber sample.

Résumé

Des mesures de tensions effectuées sur les fibres du nylon 66 et du téréphthalate de polyéthylène ont montré que les tenacités des fibres à températures relativement élevées (entre environ 250°C et −100°C) sont contrôlées par la structure moléculaire interne de la fibre et non par un quelconque défaut local. Bien qu'il soit vrai que les fibres se rompent toujours à l'un ou l'autre défaut local, la force actuelle est déterminée par un arrangement de la structure moléculaire, qui se produit avant la rupture de la fibre. Au contraire, la tenacité à basse température (généralement de −100° à −196°C) est contrôlée par des défauts locaux qui se protègent sous l'influence de la tension en produisant la rupture de la fibre, avant que la structure moléculaire interne ne montre quelque arrangement. La température à laquelle le mécanisme change dépend de l'importance des défauts locaux ainsi que de la structure moléculaire interne. On a trouvé que la tenacité à basse température est indépendante de la température de mesure et on a montré que ces niveaux constants de ténacité à basse température sont une mesure de l'intensité des défauts locaux les plus critiques dans les échantillons.

Zusammenfassung

Zugmessungen an Nylon-66 und Polyäthylenterephthalatfasern haben gezeigt, dass die Faserfestigkeit bei verhältnismässig hohen Temperaturen (zwischen etwa 250°C und −100°C) durch die innere, molekulare Struktur der Faser und nicht durch irgendwelche lokalen Defekte bestimmt ist. Es ist zwar richtig, dass Fasern immer bei irgendeiner lokalen Defektstelle reissen, die tatsächliche Festigkeit wird aber durch ein Nachgeben der molekularen Struktur bedingt, das vor dem Reissen der Faser eintritt.

Im Gegensatz dazu wird die Festigkeit bei niedrigen Temperaturen (im allgemeinen −100°C bis −196°C) durch lokale Defekte bedingt welche unter Spannung wachsen und einen Faserriss verursachen, bevor noch die innere molekulare Struktur überbeansprucht wird. Die Temperatur, bei welcher eine Änderung des Mechanismus auftritt, hängt vom Grade der lokalen Defekte und von der inneren, molekularen Struktur ab. Die Festigkeit bei niedriger Temperatur erwies sich als unabhängig von der jeweiligen Messtemperatur und diese konstante Höhe der Festigkeit bei niedriger Temperatur bildete ein Mass für den Grad der kritischen lokalen Defekte in der Faserprobe.

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