Crystallite Orientation in Regenerated Cellulose Filaments. II. Effect of Degree of Polymerization and Stretch*

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Synopsis

Measurements of the degree of orientation in cellulose filaments prepared from fractions of secondary cellulose acetate having a very limited range of degree of polymerization have shown that the degree of orientation produced by a given effective stretch increases as the degree of polymerization increases. The orientation distributions of the (101) and (020) planes have been compared with those predicted by Kratky's theory for the deformation of a material containing rod-shaped crystallites, and it has been shown that the shapes of the observed and theoretical distributions are the same if the appropriate value of the degree of stretch is used. This value is always less than that calculated from the changes in the dimensions of the filaments produced by stretching. Kratky's theory cannot however account for the variation with degree of polymerization and does not account for the observed shape of the curves for orientation as a function of stretch. Consideration of the process by which the filaments were produced shows that the filaments were subjected to stretches additional to those intentionally applied to them. The results of the orientation measurements are compared with published figures for the birefringence and tenacity of similar filaments, and linear relations are found between birefringence and orientation and between tenacity and orientation.

INTRODUCTION

It has been known for a long time that two of the factors that influence the mechanical properties of a filament are the degree of orientation of the crystallites and the degree of polymerization of the material. In a regenerated cellulose filament the orientation of the crystallites is a result of the stretch that is given to the filament during its production at a time when it is in a plastic state. There is therefore considerable interest in the mechanism of the orientation process and in the effect of the degree of polymerization and the degree of stretch on the orientation produced.

Little work has been done on the effect of the degree of polymerization on the properties of cellulose filaments, but recently Cumberbirch and Harland¹ have made an intensive investigation of the mechanical properties of filaments of cellulose prepared from carefully fractionated cellulose acetate. They used the value of the birefringence as a measure of the de-

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gree of molecular orientation and related this to the mechanical properties, stretch, and degree of polymerization. The birefringence is, however, only a measure of the average molecular orientation and is somewhat insensitive to changes in the distribution of molecular orientation. We have therefore measured the crystallite orientation in filaments similar to those used by Cumberbirch and Harland by an x-ray method and related this to the stretch and degrees of polymerization of the filaments.

We have also considered the mechanism of the orientation process Two types of theory have been advanced to explain the mechanism of this process, one of which has been largely developed by $Kratky^{2-4}$ and the other by Hermans.¹¹⁻¹⁴ Attempts have been made²⁻¹⁰ to test these theories by means of x-ray measurements of the crystallite orientation in regenerated cellulose filaments. The results of the tests were not, however, conclusive, and more data are needed on the relation of the crystallite orientation to the conditions of production of the filaments. The work that will be described in this paper provides such data.

EXPERIMENTAL

Filaments were made either by Mr. R. J. E. Cumberbirch or under his direction from some of the fractions of secondary cellulose acetate that had been produced by Cumberbirch and Harland for their investigation on the mechanical properties of regenerated cellulose filaments.¹ The method of making these filaments has been described in detail.¹ A solution of secondary cellulose acetate (degree of substitution approximately 2.3) was slowly extruded into a coagulating solution. Care was taken not to apply any stretch to the filament at this stage and the filament that was produced was isotropic. Some of this isotropic filament, which had not been allowed to dry in the mean time, was then stretched in boiling water to produce an oriented filament of secondary cellulose acetate. This filament was then hydrolyzed to produce an oriented filament of cellulose. Reference filaments were produced by drying and then hydrolyzing some of the isotropic filament of secondary cellulose acetate that had not been immersed in boiling water.

The amount by which a filament has been stretched can be expressed as either the percentage stretch or the degree of stretch. If the length of the filament is L before stretching and L' after stretching, the percentage stretch is S, where

$$S = 100(L' - L)/L$$
 (1)

and the degree of stretch is v, where

$$v = L'/L \tag{2}$$

Thus,

$$S = 100(v - 1) \tag{3}$$

The values of L and L' can be measured at different stages in the manufacture of the filament and hence different values can be obtained for the stretch. Cumberbirch and Harland¹ defined the percentage effective stretch as S_t , where

$$S_{\rm t} = 100(d_1/d_2 - 1) \tag{4}$$

 d_1 is the mass per unit length of the dry, isotropic filament of cellulose (i.e., the reference filament) and d_2 is the mass per unit length of the dry, stretched filament of cellulose.

This definition is equivalent to measuring L' on the dry, stretched filament after hydrolysis to cellulose and L on the dry, unstretched filament after hydrolysis to cellulose. We have also measured the applied stretch, where L' is measured immediately after stretching, before the filament is removed from boiling water, and L is measured at the time that the filament is attached to the stretching frame, before it is immersed in boiling water.

 TABLE I

 Degree of Polymerization, Degrees of Stretch, and Orientation Coefficient of Specimens

	Degree of polymerization	Degrees of stretch calculated from the measured dimensions		Orientation coefficient	Degrees of stretch calculated from Kratky's rodlet theory	
Specimen	N	$v_{\mathbf{a}}$	v_{t}	Ω_{50}	$v_{ m KD}$	v_{KP}
$\gamma 2/0$	729	1.0	2.14	0.045		1.83
$\gamma 2/20$		1.2	2.42	0.070	2.09	2.51
$\gamma 2/40$		1.4	3.00	0.089	2.70	2.92
$\gamma 2/120$		2.2	4.25	0.123	3.32	3.62
$\beta 4/0$	614	1.0	1.57	0.032	_	1.40
$\beta 4/40$		1.4	2.47	0.077	<u> </u>	2.79
$\beta 4/80$		1.8	3.10	0.089		2.92
$\beta 4/120$		2.2	4.20	0.117		3.50
δ3/60	569	1.6	3.01	0.088		2.90
δ3/100		2.0	3.87	0.095		3.05
δ3/140		2.4	4.14	0.112	<u> </u>	3.38
δ3/160		2.6	4.11	0.097		3.09
$\delta 4/0$	430	1.0	1.86	0.037		1.59
$\delta 4/40$		1.4	2.94	0.071		2.54
$\delta 4/80$		1.8	3.41	0.093		3.00
$\delta 4/120$		2.2	4.28	0.097	_	3.09
A3/0	292	1.0	1.81	0.031		1.37
A3/40		1.4	2.32	0.053		2.05
A3/80		1.8	2.59	0.072		2.57
A3/100		2 .0	3.36	0.076	—	2.66
D3/0	189	1.0	1.97	0.030		1.32
D3/40		1.4	3.10	0.042		1.74
D3/80		1.8	3.86	0.059	_	2.22
D3/100		2.0	4.43	0.065	2.18	2.38

Filaments that had been stretched to different extents were obtained from each of six fractions covering a range of degree of polymerization from 190 to 730. Details of the degree of polymerization, N, the applied stretch, $v_{\rm a}$, and the effective stretch, $v_{\rm t}$, of the filaments that were used are given in Table I.

From these filaments, x-ray specimens consisting of bundles of short lengths of filaments were prepared, and x-ray diffraction photographs were taken in the way that was described in Part I.¹⁵ Normal photographs, suitable for determining the distribution of intensity in the paratropic reflections (i.e., hol reflections), were taken for each of the specimens. In addition, inclined photographs suitable for determining the distribution of intensity in the 020 reflection were taken for a few of the specimens. The azimuthal intensity distribution of the 020 and 101 reflections of these specimens were compared with those predicted by Kratky's theory for the deformation of a material consisting of rodshaped micelles embedded in an isotropic medium.³ The 40%, 50%, and 60% orientation coefficients (the reciprocals of half the breadth of the distribution curve at 40%, 50%, and 60%, respectively, of the maximum height) were measured for the (101) planes of all the specimens. Three photographs were taken for each specimen, and both the 101 reflections were measured on each photograph, so that the final orientation coefficient was the mean of six results. The mean orientation coefficients were plotted against the percentage effective stretch and interpolated values at constant values of the percentage effective stretch were plotted against the degree of polymerization.

RESULTS AND DISCUSSION

Relation of Orientation to Effective Stretch and Degree of Polymerization

The curves for orientation coefficient against percentage effective stretch were found to be represented quite well by parabolae having equations of the form

$$(\Omega + A)^2 = C(S_t - B)$$
(5)

where Ω is the orientation coefficient and A, B, and C are constants. It was further found that the three orientation coefficients were related by the equations

$$\Omega_{40} = 0.84\Omega_{50} \tag{6}$$

$$\Omega_{60} = 1.19\Omega_{50} \tag{7}$$

It is therefore sufficient to consider one of the orientation coefficients. The one that has been chosen is Ω_{50} ; the values obtained are listed in Table I. Two examples of the curves for orientation coefficient against percentage effective stretch are shown in Figure 1, and the values of A, B, and C for the different fractions are given in Table II.

The equations that are obtained by substituting the appropriate values of the constants in eq. (5) enable the orientation coefficient to be predicted for any given stretch and any one of the fractions with reasonable accuracy.

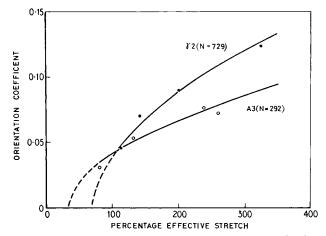


Fig. 1. Orientation against effective stretch at constant degree of polymerization.

 TABLE II

 Values of the Constants in the Equations Relating the 50% Orientation Coefficient and the Percentage Effective Stretch

Fraction	А	В	$C \times 10^{\circ}$
$\gamma 2$	0.010	66	71.7
$\beta 4$	-0.002	69	52.6
δ3	-0.003	66	47.6
δ4	-0.001	52	36.9
A3	0.005	30	30.1
D3	0.012	12	26.5

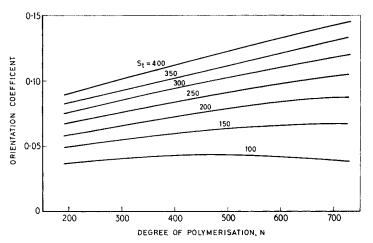


Fig. 2. Orientation against degree of polymerization at constant effective stretch.

The average difference between the predicted value and the observed value is 0.006 and the average standard deviation of an experimental determination is also 0.006. Values were calculated by means of eq. (5) for all the fractions at constant values of the percentage effective stretch and plotted against the degree of polymerization. Some of the curves that were obtained are shown in Figure 2. The curves for $S_t = 100$ and $S_t = 400$ are limiting examples that could not be observed in practice, since the observed values of the percentage effective stretch were mostly greater than 100 and less than 400.

Comparison with Kratky's Theory

Kratky³ considered the crystallite orientation produced by the deformation of a material consisting of rod-shaped crystalline micelles embedded in a continuous medium. He assumed that each volume element of the material, however small, was deformed in the same way as any other volume element and obtained expressions for the distribution of orientation of the diatropic and paratropic planes as a function of the degree of stretch. Although Kratky assumed that the material contained rod-shaped crystalline micelles, his theory is equally applicable to a material that contains no crystalline regions. All that it requires is that the material shall contain rod-shaped units and that the deformation shall be homogeneous. The units could be bundles of parallel chain molecules or even single chain molecules. During deformation the axes of the units will follow the deformation of the environment and become oriented. The axes of the molecules will normally be parallel to the axis of the unit and when, at a later stage, crystallization takes place, this direction will become the direction of the b axis of the crystalline region. The theory can therefore be applied to the filaments that were used in this investigation, in which there was probably little or no crystalline material at the time when they were stretched.

When Hermans and Kratky²⁻⁷ tested the theory they attempted to obtain the appropriate values of the degree of stretch from the measured dimensions of the filament. The process by which the filaments are produced is however a complex one and it is probable that the values for the degree of stretch that are obtained in this way will not be the appropriate ones for the theory. It is, however, possible to obtain the degree of stretch from the orientation distribution curve for the (020) planes and to use this to calculate the distribution of orientation that is to be expected for the (101) planes.

The expression obtained by Kratky for the distribution of orientation of a diatropic plane, i.e., a plane whose normal is parallel to the b axis, in a material that had been subjected to a simple stretching process was

$$J(\beta) = v^3 J_0 / [1 + (v^3 - 1) \sin^2 \beta]^{3/2}$$
(8)

where $J(\beta)$ is a density function such that the number of crystallites for

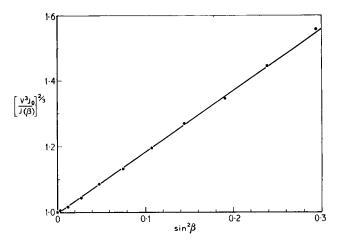


Fig. 3. The azimuthal intensity distribution of the 020 reflection of specimen $\gamma 2/40$ compared with Kratky's rodlet theory.

which the b axis makes an angle between β and $\beta + d\beta$ with the direction of stretch is dn, where

$$dn = 2\pi \sin\beta J(\beta)d\beta \tag{9}$$

v is the degree of stretch, and J_0 is a constant. The value of $v^3 J_{\theta}$ can be obtained from the peak value of the curve since, when $\beta = 0$,

$$J(0) = v^3 J_0 (10)$$

If then $[v^3J_0/J(\beta)]^{2/3}$ is plotted against $\sin^2 \beta$, a straight line should be obtained. The value of v can be found from the slope of this line. This has been done for the 020 reflections of four representative specimens. Figure 3, which is an example of the graphs obtained, shows that the shape of the azimuthal intensity distribution is very well represented by Kratky's equation. The values of v that were obtained for the four specimens are given in Table I. They have been called $v_{\rm KD}$.

The distribution of a paratropic plane, i.e., a plane whose normal is perpendicular to the b axis, was found by Kratky to be given by

$$F(\alpha) = J_0 v^{-3/2} E(k) / (1 - k^2)$$
(11)

where $F(\alpha)$ is a density function such that the number of crystallites for which the normal to the paratropic plane is inclined at an angle between $\pi/2 - \alpha + d\alpha$ and $\pi/2 - \alpha$ to the direction of stretch is dn', where

$$dn' = 2\pi \cos \alpha F(\alpha) d\alpha \tag{12}$$

k is defined by the equation

$$k^2 = \cos^2 \alpha \ (v^3 - 1)/v^3 \tag{13}$$

E(k) is the complete elliptic integral of the second kind of modulus k.

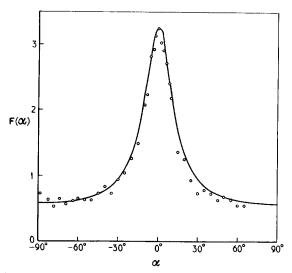


Fig. 4. The azimuthal intensity distribution of the 101 reflection of specimen $\gamma 2/40$ compared with Kratky's rodlet theory.

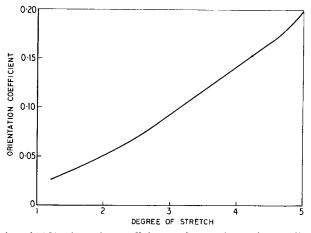


Fig. 5. Relation of 50% orientation coefficient to degree of stretch according to Kratky's rodlet theory.

By means of equation (11) the values of $F(\alpha)$ have been calculated, and graphs of $F(\alpha)$ against α have been plotted for the four specimens for which the value of $v_{\rm KD}$ has been determined. The curves that were obtained were compared with the azimuthal intensity distribution curves for the 101 reflections (an example is shown in Fig. 4). From the agreement in the shapes of these curves it can be concluded that (a) Kratky's "rodlet" theory gives a satisfactory description of the shape of the orientation distribution curves for the (101) planes of the specimens used in this investigation, and (b) the filaments whose orientation is being studied show little if any selective orientation of the (101) planes. The value of $v_{\rm KD}$ is in each instance greater than the degree of applied stretch, $v_{\rm a}$ and less than the degree of effective stretch, $v_{\rm t}$. The significance of this fact will be discussed later.

Kast⁸⁻¹⁰ found that Kratky's rodlet theory predicts that the orientation coefficient will be a linear function of the stretch. This is only approximately true, however. If the orientation coefficients are measured from the theoretical orientation distribution curves for a paratropic reflection in exactly the same way as is used for the experimental curves, the relation between the 50% orientation coefficient and the degree of stretch is found to be that shown in Figure 5. The shapes of the experimental curves for orientation coefficient against stretch (Fig. 1) are not the same as that of the curve in Figure 5, and it is therefore necessary to consider the relation between the observed degrees of stretch and the degree of stretch that is appropriate to Kratky's theory.

Relation of Observed and Theoretical Degrees of Stretch

From Figure 5 a value for the degree of stretch can be obtained such that if this degree of stretch were applied to a material to which Kratky's rodlet theory applied, it would produce a material having the same value of the 50% orientation coefficient as the experimental material. This value of the degree of stretch, $v_{\rm KP}$, has been obtained for all the specimens used in this investigation and is listed in Table I. The theoretical orientation distribution curves have been computed for these values of the degree of stretch and compared with the corresponding experimental curves. In all instances the agreement is satisfactory. There is some discrepancy between the values of $v_{\rm KP}$ and $v_{\rm KD}$ given in Table I. This arises from the experimental error in the determination of the azimuthal intensity distribution curves and the fact that the value of $v_{\rm KD}$ was obtained from one such curve whereas that of $v_{\rm KP}$ was obtained from the mean of six.

The values obtained for $v_{\rm KP}$ confirm that the theoretical degree of stretch, $v_{\rm K}$, which is measured by $v_{\rm KP}$ or $v_{\rm KD}$, is greater than the degree of applied stretch, $v_{\rm a}$, but less than the degree of effective stretch, $v_{\rm t}$. This can arise from the changes that are to be expected in the degree of swelling.¹⁶ The relation between the theoretical degree of stretch and the degree of applied stretch would then be

$$v_{\rm K} = (q_1/q_3)^{1/3} v_{\rm a} \tag{14}$$

where q_1 is the degree of swelling of the filament before stretching and q_3 is the degree of swelling of the filament after stretching. The relation between the theoretical degree of stretch and the degree of effective stretch would be

$$v_{\rm K} = (\lambda/q_3^{1/3})v_{\rm t}$$
 (15)

where λ is defined by

$$l_{\rm d} = \lambda l_{\rm w} \tag{16}$$

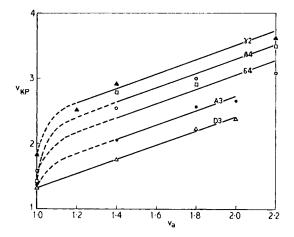


Fig. 6. Theoretical stretch against applied stretch.

 $l_{\rm w}$ is the length of the wet, stretched filament, and $l_{\rm d}$ is the length of the dry, stretched filament. Since q_3 depends on v_{α} and λ depends on the degree of molecular orientation and hence on $v_{\rm K}$, the relation between the theoretical degree of stretch and the observed degrees of stretch will not be simple.

When v_a is unity, q_3 will be equal to q_1 , and λ will be equal to $q_1^{1/3}$. Both $v_{\rm K}$ and $v_{\rm t}$ should therefore also be unity, but this was not observed. However, because of the nature of the process by which the filaments used in this investigation were produced, additional stretching can occur. Consider for example the stage of the process between the time when the filament is attached to the stretching frame and the time when it is removed from the stretching bath. A length l_1 of the filament is attached to the stretching frame so that it is just taut. The frame and filament are then immersed in the stretching bath of boiling water and allowed to attain the temperature of the bath. During this time the temperature of the filament and probably also its degree of swelling are changing. The two effects may occur at different rates, may involve either expansion or contraction of the filament, and may affect one another. The fact that the filaments for which $v_{\rm a}$ is unity all have values of $v_{\rm K}$ and $v_{\rm t}$ greater than unity suggests that the filament may have tried to contract but been prevented from doing so by the stretching frame, and has therefore been stretched. It was observed however that when these filaments were removed from the stretching bath they were slack, showing that the total effect during this stage of of the process was an expansion to a length l_2 . The filament was then stretched to a length l_a and removed from the stretching bath. The degree of applied stretch was measured as l_3/l_1 , but the true degree of applied stretch was unity unless l_3 was greater than l_2 , when it was l_3/l_2 . The values obtained for the degree of applied stretch for those filaments that were not intentionally stretched $(v_a = 1)$ are therefore not consistent with those obtained for filaments which were intentionally stretched $(v_{\rm a} > 1)$. This is illustrated in Figure 6, where the theoretical degree of

stretch is plotted against the degree of applied stretch; only the points for which $v_a > 1$ appear to be related. In any future work on the stretching of hot, wet filaments this stage of the process should receive further attention.

Comparison with Birefringence and Tenacity

Values of the birefringence and the tenacity for filaments similar to those used in this investigation have been published by Cumberbirch and Harland.¹ The results given by them for filaments with zero percentage effective stretch relate to reference filaments (i.e., isotropic filaments that had not been immersed in boiling water during their production). As a result of the additional stretches that occur in the process of production it is not possible to make filaments with effective stretches in the range 0 to S_0 , where S_0 is the percentage effective stretch for a filament for which the measured percentage applied stretch was zero. It is not therefore to be expected that the same relation will necessarily exist between any measured property of the filaments that is affected by stretch and the percentage effective stretch on both sides of this range. The results for the birefringence and the tenacity of filaments of zero effective stretch were therefore omitted, and it was found that, when the other results were plotted against the effective stretch, the points lay on curves similar to those found for the x-ray results. Interpolated values of the birefringence and the tenacity were then compared with corresponding values of the x-ray orientation coefficient and linear relations were found to exist:

$$n = p\Omega + q \tag{23}$$

$$T = r\Omega + t \tag{24}$$

0.19

0.13

0.11

0.44

0.31

0.36

where n is the birefringence and T is the dry tenacity of the filaments. The values of the coefficients p, q, r, and t are given in Table III.

TABLE III Values of the Coefficients in the Relations between Birefringence and Orientation and

Tenacity and Orientation					
	Degree of Polymerization	p	q	r	t
$\gamma 2$	729	0.21	0.013	0.21	1.00
δ3	569	0.24	0.008	0.25	0.60
A2	407	0.33	0.001	0.30	0.19

0

0

0.004

	Polymerization	p	q	r	t
$\gamma 2$	729	0.21	0.013	0.21	1.00
δ3	569	0.24	0.008	0.25	0.60
A2	407	0.33	0.001	0.30	0.19
B1	386	0.32	0.002	0.27	0.20
ε2	372	0.38	0.002	0.20	0.69
A3	292	0.34	0.002	0.21	0.42
C2	243	0.36	0.001	0.27	0.01
C3	187	0.39	0	0.14	0.55

0.33

0.33

0.24

D3

F1

E3

189

125

111

Equations (23) and (24), together with eqs. (16)-(22) enable the birefringence and tenacity for a filament of given effective stretch prepared from any of the fractions to be predicted with reasonable accuracy. The average difference between the predicted value and the observed value is 0.0007 for birefringence and 0.03 for tenacity. These differences are less than the experimental error in the determination of the birefringence and the tenacity respectively.

The values of the coefficients p, q, r, and t appear to vary with the degree of polymerization, but the values obtained are not sufficiently accurate to enable any conclusions to be drawn about this variation.

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Résumé

Les mesures du degré d'orientation dans les filaments de cellulose, préparés à partir de fractions d'acétate de cellulose secondaire possédant un domaine très limité du degré de polymérisation, ont montré que le degré d'orientation produit par une tension effective augmente quand le degré de polymérisation augmente. Les distributions d'orientation des surfaces planes (101) et (020) ont été comparées à celles prévues par le théorie de Kratky pour la déformation d'un matériau contenant des cristallites de forme baguette, et on a montré que les formes des distributions observées et théoriques sont les mêmes si on utilise la valeur appropriée du degré de tension. Cette valeur est toujours plus petite que celle calculée à partir des changements dans les dimensions des filaments produits par tension. La théorie de Kratky n'explique cependant pas la variation avec le degré de polymérisation et n'explique pas la forme observée des courbes pour l'orientation en fonction de la tension. L'examen du processus par lequel les filaments ont été produits montre que les filaments ont été soumis à des tensions supplémentaires à celles qui leur ont été appliquées intentionnellement. Les résultats des mesures d'orientation sont comparés aux résultats publiés pour la biréfringence et la ténacité de filaments semblables, et on a trouvé des relations linéairés entre la biréfringence et l'orientation et entre la ténacité et l'orientation.

Zusammenfassung

Aus Messungen des Orientierungsgrades in Cellulosefasern, die aus sekundär-Celluloseacetatfraktionen mit einem sehr begrenzten Polymerisationsgradbereich hergestellt wurden, geht hervor, dass der durch eine gegebene wirksame Dehnung hervorgerufene Orientierungsgrad mit steigendem Polymerisationsgrad zunimmt. Die Orientierungsverteilung der (101)- und (020)-Ebenen wurde mit derjenigen verglichen, die von der Kratkyschen Theorie für die Deformation eines stabförmige Kristallite enthaltenden Materials vorausgesagt wird. Dabei ergab sich, dass beobachtete und theoretische Verteilung bei Verwendung des entsprechenden Wertes für den Dehnungsgrad von derselben Form sind. Dieser Wert ist stets kleiner als der aus der Dimensionsänderung der Faser bei der Dehnung berechnete Wert. Die Änderung mit dem Polymerisationsgrad sowie die beobachtete Form der Kurven für die Abhängigkeit der Orientierung von der Dehnung können jedoch mittels der Kratkyschen Theorie nicht erklärt werden. Wie aus einer Untersuchung des Herstellungsprozesses der Fasern hervorgeht, wurden die Fasern, zusätzlich zu den mit Absicht angewandten Dehnungen, weiteren Dehnungen Die Ergebnisse der Orientierungsmessungen wurden mit den in der unterworfen. Literatur angegebenen Kurven für die Doppelbrechung und die Zähigkeit ähnlicher Fasern verglichen. Zwischen Doppelbrechung und Orientierung sowie zwischen Zähigkeit und Orientierung bestehen lineare Beziehungen.

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