

The Decrimping of Single Wool Fibers.

I. The Effects of Some Physical Treatments on Crimp Parameters

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Synopsis

The effects of relative humidity, temperature and longitudinal extension on the decrimping characteristics of single wool fibers are examined. Stress-strain properties are analyzed in terms of decrimping energy and decrimping force together with a parameter depending upon changes in crimp geometry. Decrimping properties are recovered even after single fibers are extended almost to their breaking strains.

INTRODUCTION

The stability of the crimped shape of wool fibers and its recovery after deformation has been briefly reported by Woods,¹ Goldsworthy and Lang,² Banbaji, Alexander, and Lewin,³ and Khan.⁴

Woods¹ noted that when a Merino wool fiber is hung freely in a closed vessel, the fiber begins to coil with changes in humidity and may even curl into a ball. Goldsworthy and Lang² demonstrated that coiling of wool fibers immersed in different liquids is due to relaxation of preexistent strains that could be resolved into reversible and nonreversible effects.

Khan⁴ studied the effect of hydration and swelling agents on the form of crimp and extended his work to the study of felting and compressional properties of wool treated in various ways. He concluded that, on hydration, there was a very considerable and permanent loss of crimp, principally in terms of crimp amplitude.

Banbaji, Alexander, and Lewin³ also investigated the recovery of crimp after various amounts of extension and the dependence of crimp on temperature as characterized by its decrimping energy and the stress to decrimp the fiber. These parameters were estimated from load-extension measurement in the crimp region.

The present work is an extension of the investigation made by Balasubramaniam and Whiteley⁵ and Banbaji, Alexander, and Lewin.³

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In this paper, the load-extension curve is characterized in terms of decrimping energy (E_c) and the forces to decrimp (F_c), but a parameter depending on crimp shape is also introduced. This parameter is dependent only on shape factors and is investigated before and after subjecting the fiber to physical changes such as strain, humidity, and temperature variations.

THEORY

Van Wyk and Venter⁶ have fitted an empirical relationship to stress-strain data in the decrimping region on the assumption that the fibers were not strained longitudinally, that the fiber cross sections were not distorted, and that the change in curvature was proportional to the bending moment due to the applied stress.

The relationship was expressed in the form

$$T = \frac{AEI}{S^2} \left\{ \frac{\cos^{-1} l_0/s}{\cos^{-1} l/s} - 1 \right\} \quad (1)$$

where T = tension, A = a constant, E = elastic modulus, I = moment of inertia of a cross section about a diameter, S = arc length of a quarter crimp, l_0 = original crimped length, l = crimped length at tension T , and s = straight length of the fiber.

From detailed observation on a series of wools, Walls⁷ derived an alternative equation for this region by considering the crimped wool fiber as a thin, uniform elastic rod in the form of a uniplanar sinusoidal wave, the plane of which rotates or oscillates about the axis. The relationship was approximated to a form which can be written as

$$QT \sqrt{1 + 1/P} + \sqrt{1 + 1/P} = \sqrt{s/(s - l)} \quad (2)$$

where P and Q are constants such that P depends only on fiber shape while Q depends both on shape and elastic modulus.

This crimp form and the assumptions on which eq. (2) is based have been criticized by Hannah⁸ in view of Holdaway's studies on helical configurations.

Holdaway,⁹ by examining the theoretical aspects of the decrimping of a uniform helical spring of small cross section, has been able to generalize an empirical relationship, which can be written in the form

$$T = a \frac{(l - l_0)}{(s - l)} \quad (3)$$

where for Holdaway's model

$$a = \frac{E'}{0.924} \left(\frac{\pi^3}{16} \right) \left(\frac{n}{l_0} \right)^2 d^4$$

and E' = apparent Young's modulus (involving a form factor), n = total number of free turns in the helix, and d = diameter of the fiber.

This relationship fitted experimental data well although there were relatively large deviations at low strains.

Whiteley and Balasubramaniam¹⁰ have noted that the two relationships represented by eqs. (1) and (2) behave similarly, regardless of whether or not they satisfy the decrimping properties of the fiber. This similarity was so striking as to suggest that the two equations could be closely related to each other over the decrimping ranges studied. They showed, by expanding $\cos^{-1}(l/s)$ terms of its sine function, that eq. (1) approximated to

$$\frac{TS^2}{AEI} [\sqrt{2}/\cos^{-1}(l_0/s)] + [\sqrt{2}/\cos^{-1}(l_0/s)] = \sqrt{s/(s-l)}. \quad (4)$$

Identifying (4) with (2), they found

$$Q = S^2/AEI \text{ and } \sqrt{1 + 1/P} = \sqrt{2}/\cos^{-1}(l_0/s).$$

These relations agree with Walls' conditions that P should depend on fiber shape while Q depends on shape and elastic modulus.

In this study, a wool sample whose fibers exhibited an excellent straight-line relationship between $\sqrt{s(s-l)}$ and T was selected so that Walls' equation could be adopted. This model is particularly useful as the value of P calculated from the intercept depends only on the crimp shape of the fiber and is independent of fiber diameter or elastic modulus, thus facilitating the study of changes in crimp shape after subjecting the fiber to various physical and chemical treatments.

MATERIALS AND METHODS

Decrimping Curve

The load-extension curves were carried out on a Cambridge extensometer in which the spring load had been replaced by a Statham force transducer connected to a chart recorder for load measurements. This recorder gave a maximum sensitivity of 100 mg for full-scale deflection.

The fiber was allowed to hang freely under its own weight in air, with its upper end attached to the Statham force transducer. The lower end was then attached to the extension arm of the Cambridge extensometer which was modified to provide straining rates of 0.2 cm/min.

Measurement of Decrimped Length (s)

The gradual extension of the crimped fiber was observed by a traveling microscope. When there was no sign of any crimp along the fiber, the length (L_0) was measured (Fig. 1).

The stress value (F_0) at this length was noted. The length L_0 represents the sum of the decrimped length (s) plus the contribution made by changes in arc length ($B'C$). The decrimped length s is therefore obtained by the subtraction of $B'C$ from L_0 . $B'C$ is measured from the intersection of the Hookean slope with the extension axis to the extension corresponding to the stress value F_0 (Fig. 1).

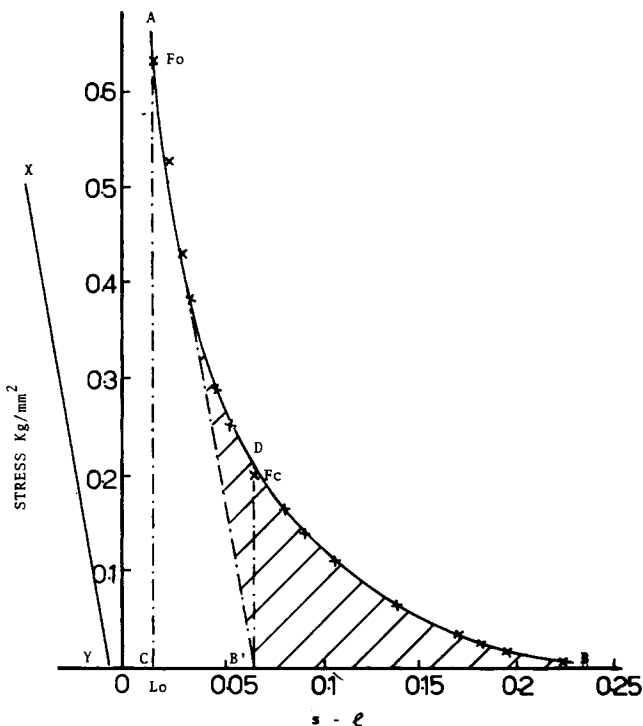


Fig. 1. Decrimping curves of a single wool fiber.

Determination of Hookean Slope

To improve the accuracy of the determination of crimp parameters, a second stress-strain cycle was necessary. While the first involved purely the decrimping region and maximum loads in range of 200 to 300 mg, the second extension cycle was carried out after a suitable rest period³ to a higher load range so as to enable accurate assessment of the slope of the Hookean region. Thus, in Figure 1, the line XY derived from the second extension cycle was drawn to correspond to the estimated Hookean slope but on the same scale of load as curve AB. A tangent to the upper limit of the decrimping curve was then drawn parallel to XY to give AB'.

This is necessary as estimation of the slope of the linear portion of the graph obtained by decrimping, and the extrapolation back to zero stress, may become quite subjective, leading to errors in the estimate of the force to decrimp the fiber (F_c) and the decrimping energy (E_c).

Measurement of F_c , E_c , P , and Q

The stress to decrimp a fiber (F_c) was obtained by drawing the line B'D through the intersection of the Hookean slope on the strain axis to curve

AB (Fig. 1). The shaded area divided by length s gave the energy per unit volume to decrimp a fiber (E_e) and is expressed in units of Joules/cm³.

The plot of $\sqrt{s(s-l)}$ versus T gave a straight line (Fig. 2), and the intercept of the regression line of the strain function axis is equal to $\sqrt{1 + 1/P}$, thus enabling the value of P to be calculated. The parameter Q was obtained from the gradient of the regression line.

Wool Samples

The wool used was a Merino 64's chosen because of the exceptional uniformity in diameter along the length of its individual fibers and its well-defined and uniform staple crimp.

All fibers used in this section were carefully selected for uniformity of crimp amplitude and crimp frequency along the length of the fiber, and only fibers whose decrimping curves gave a good straight-line fit (correla-

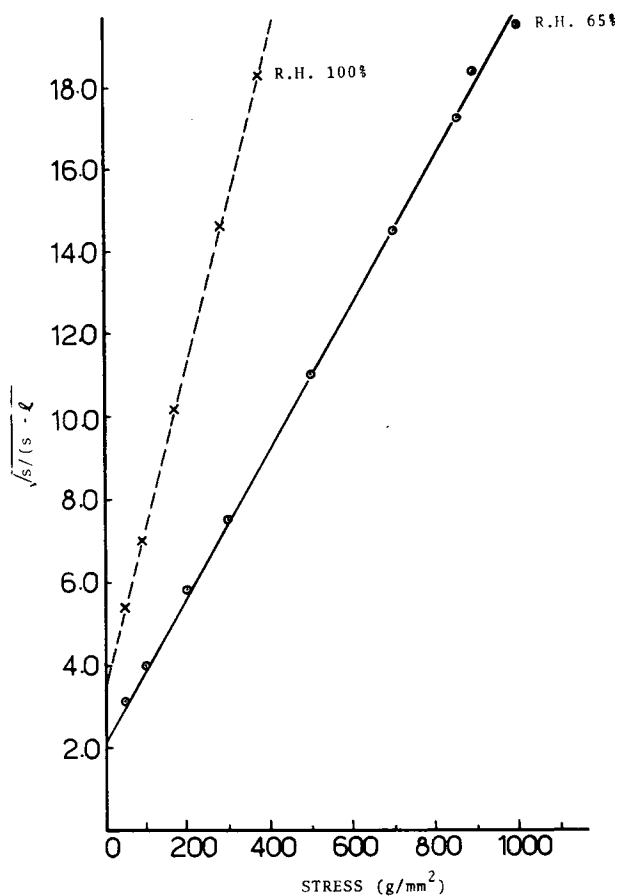


Fig. 2. Regression of $\sqrt{s(s-l)}$ vs. stress.

tion coefficient >0.95) to Walls' equation were used for recoverability studies.

The decrimped length s of the fiber was kept approximately constant (4 ± 0.1 cm).

Effect of Humidity

Five fibers were conditioned in water at 21°C for 20 hr and extended in water to find F_e , E_e , P , and Q . After relaxation for another 20 hr in water, the fibers were extended under similar conditions. Near-perfect reproducibility of the decrimping curves was obtained in agreement with Banbaji et al.³ and Walls.⁷

After a similar period of relaxation in water, the fibers were then conditioned to 65% R.H. at 21°C and extended at this relative humidity to find F_e , E_e , P , and Q . Subsequent stretching in water after resting for 20 hr revealed excellent reproducibility of the decrimping curve.

Crimp Recovery after Various Amounts of Extension with Zero Relaxation Time

The decrimping parameters of 20 wool fibers were measured in water at 20°C . After the required recovery time (20 hr in water at 21°C), the fibers were divided into four groups. Each group was extended in water to one of the final extensions: 2%, 10%, 30%, and 40%. After contraction, the fibers were immediately reextended to measure the second cycle decrimping parameters. Similar crimp recovery measurements were made on another group of fibers, extended to 1%, 2%, 10%, and 30% at 65% R.H. and 20°C .

The crimp parameters of the second cycle were expressed as a fraction of the first cycle, and this recovery coefficient term was plotted against the selected final extensions.

Crimp Recovery with Time

The decrimping parameters of 20 wool fibers were measured in water at 20°C . These parameters were also measured on another group of fibers at 65% R.H. and 20°C . After contraction and relaxation, the first group was extended to 40% extension in water and the second group to 30% at 65% R.H., respectively. The groups were then each divided into subgroups for testing recovery after 0, 1, 2, 5, and 20 hr.

Dependence of Decrimping Parameters on Temperature

Decrimping curves were obtained on 24 fibers in water at 20°C . The fibers were divided into six subgroups, and each subgroup was conditioned for 30 min and extended to the limits of the Hookean region (2% strain) in water at one of the following temperatures: $30^\circ \pm 1^\circ\text{C}$; $40^\circ \pm 1^\circ\text{C}$; $50^\circ \pm 1^\circ\text{C}$; $60^\circ \pm 1^\circ\text{C}$; $70^\circ \pm 1^\circ\text{C}$; $80^\circ \pm 2^\circ\text{C}$. After contraction and a 20-hr

relaxation with zero strain in water at 20°C, the fibers were reextended to the Hookean region at 20°C in water for recovery testing.

RESULTS AND DISCUSSION

Humidity Effects

The results for the decrimping force F_c , the decrimping energy E_c , and values of P and Q are summarized in Table I. The value of P at 100% R.H. had decreased to about half its value at 65% R.H., while Q had increased to about twice its original value. It is also seen from the table that the decrimping energy of wool fibers decreases on saturation of the fibers by a factor of 5 and the decrimping stress, by a factor of 3 to 4; compared to the ratio of wet to dry (65% R.H.) Young's modulus for wool fibers, these values are high. However, when it is recalled that not only modulus but also shape factors are involved in the determination of E_c , it would appear reasonable to suggest that the principal mechanical process involved in decrimping is one of bending rather than torsion.

The behavior of the crimp shape factor P on wetting is parallel to that of the decrimping energy; and it may be deduced that, on wetting, the decrease in decrimping energy is partly attributable to a decrease of the geometric crimp parameters brought about, presumably, by the differential lateral swelling of the ortho- and paracortex components.

The effect of humidity on Q is complex due to the simultaneous variation in modulus and crimp shape.

Recovery Coefficients of Decrimping Parameters

The recovery coefficient of the decrimping parameters for various final extensions with zero relaxation time is illustrated in Figures 3 and 4 for both 100% R.H. and 65% R.H.

For the wet fiber, the recovery coefficient decreases linearly with extension. However, for the "dry" fiber (65% R.H.), the curve shows a rapid decrease in the recovery coefficients of the decrimping parameters between 0% and 10% extension, followed by a less rapid change between 10% and 30% extensions.

It should be noted that the strains imposed in the course of these experiments range from 2.5% to 40% strain; and, in terms of the deformation of the fibrils alone, this wide range of extension involves only a very small change in stress. It is not surprising that excellent linear relationships are obtained if we assume little or no contribution from the amorphous matrix in relationship to crimp stability in the wet state. This would assume, in addition, that the matrix contribution which produces the post-yield phase in longitudinal stress-strain properties does not contribute to crimp stability.

In other words, it is suggested that the "wet" crimp stability may be solely attributable to microfibrillary influences.

TABLE I
Effect of Humidity on Decrimping Parameters

Fiber no.	Relative humidity 100%				Relative humidity 65%			
	F_c , kg/mm ²	$E_c \times 10^6$, Joule/m ³	P	Q	F_c , kg/mm ²	$E_c \times 10^6$, Joule/m ³	P	Q
1	0.195	0.256	0.119	0.0130	0.720	1.28	0.243	0.0077
2	0.225	0.368	0.103	0.0064	0.800	1.97	0.205	0.0043
3	0.200	0.232	0.106	0.0121	0.800	1.48	0.200	0.0062
4	0.235	0.512	0.200	0.0096	0.820	2.37	0.396	0.0064
5	0.230	0.264	0.101	0.0096	0.820	1.61	0.219	0.0060
Mean	0.217	0.33	0.126	0.0101	0.792	1.74	0.252	0.0061

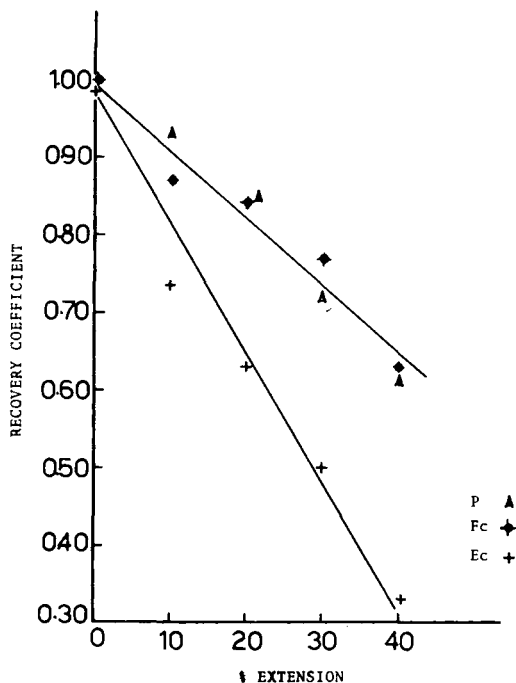


Fig. 3. Recovery coefficients of decrimping parameters P , F_c , and E_c after extension to various levels at 100% R.H.

It would be most interesting to confirm this latter hypothesis by examining strains in the range 0% to 2% in which one would expect almost perfect recovery and therefore a sharp deviation from the relationship observed at higher strains.

By comparison, the crimp stability at 65% R.H. is more complex. It is well established that the dehydration process causes marked changes in the stress-strain curve at low strains, indicating a large hydrogen bond contribution to the Hookean region over and above that provided by the crystalline microfibrils. In addition, the transition from the Hookean to the yield region in the dry state is not so clearly defined, possibly due to the poorer orientation of the hydrogen bonds in the matrix. The relation between recovery coefficient and crimp parameters and % extension in the dry state suggests a complex of matrix and fibrillary components.

The dependence of the recovery coefficients of the decrimping parameters above 40% extension (100% R.H.) and above 30% extension (65% R.H.) on recovery time is shown in Figures 5 and 6.

Two-stage recovery at both 65% relative humidity and 100% relative humidity is observed (Figs. 5 and 6). The wet recovery may be likened to recovery in longitudinal stress-strain^{11,12} in which there is a rapid initial phase associated with recoiling of the alpha helices during which time the strength of the fiber is largely recovered, followed by a slower recovery

period associated presumably with the recrystallizing of reformed alpha helices. Thus, a crimp recovery coefficient of 0.9 is obtained after 2 hr (100% R.H.), followed by perfect recovery after 5 hr.

In the dry state, we can assume additional delays in recovery due to the reformation of hydrogen bonds in the matrix where the degree of orientation is poor. Under these circumstances, we find a recovery coefficient of 0.6 after 4 hr, whereas complete recovery is only achieved after 20 hr.

A comparison of fast and slow processes at 100% R.H. and 65% R.H. indicates the relative contribution of matrix and fibril to the recovery procedure. It is remarkable, however, that one is able to extend dry fibers almost to their breaking strain and still obtain, eventually, 100% recovery of all decrimping parameters.

Effect of Temperature

The decrimping parameters at various temperatures are expressed as a ratio of their respective values at 20°C. The plot of this ratio with temperature is shown in Figure 7. Complete recovery was obtained after

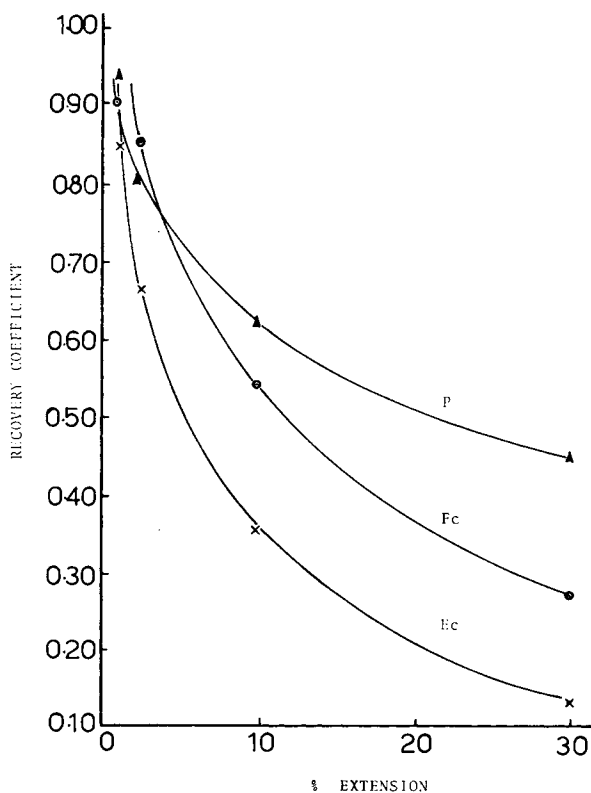


Fig. 4. Recovery coefficients of decrimping parameters P , F_c , and E_c after extension to various final levels at 65% R.H.

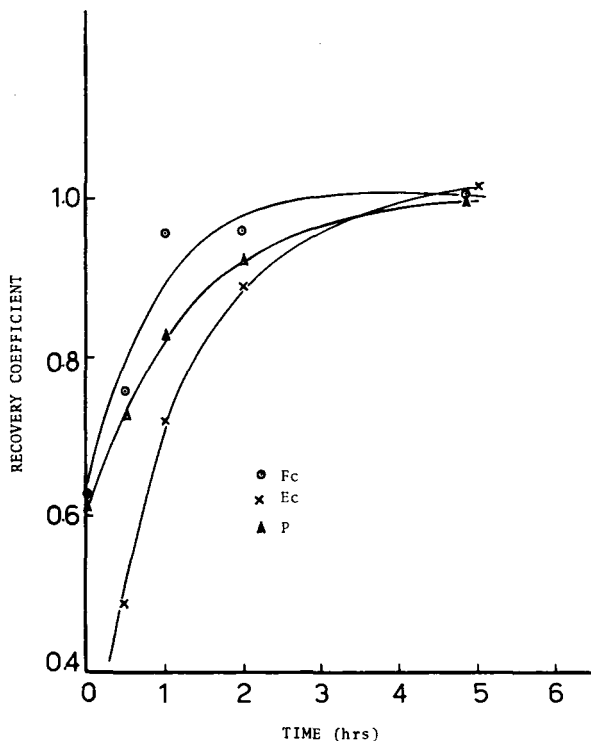


Fig. 5. Recovery coefficients of decrimping parameters P , F_c , and E_c after extension to 40% and relaxing for various times at 100% R.H.

treatment at all temperatures, with the exception of the 80°C treatment (Table II).

Over the range from 40°C to 80°C, the effect of temperature on crimp parameters appears to be comparatively linear, in agreement with the observation of Banbaji et al.³ This relationship holds independently of the fact that fibers do not recover from the 80°C treatment.

The interesting feature of the curve is that considerable changes appear to occur between 20°C and 40°C, indicative of marked alterations to the hydrogen bond network. This is most noticeable for P and E_c , but there is also a slight effect observable for F_c which is not indicated in Figure 7. It is interesting, however, to note that a similar disproportionate change over this range has also been reported for Hookean modulus by both Sherman, Balasubramaniam, and Whiteley (1968) and Feughelman and Mitchell¹³, the results of these two researches being in very close agreement.

CONCLUSION

The shape of the load-extension curve of the decrimping region is described completely by two constants: P and Q . P depends only on the

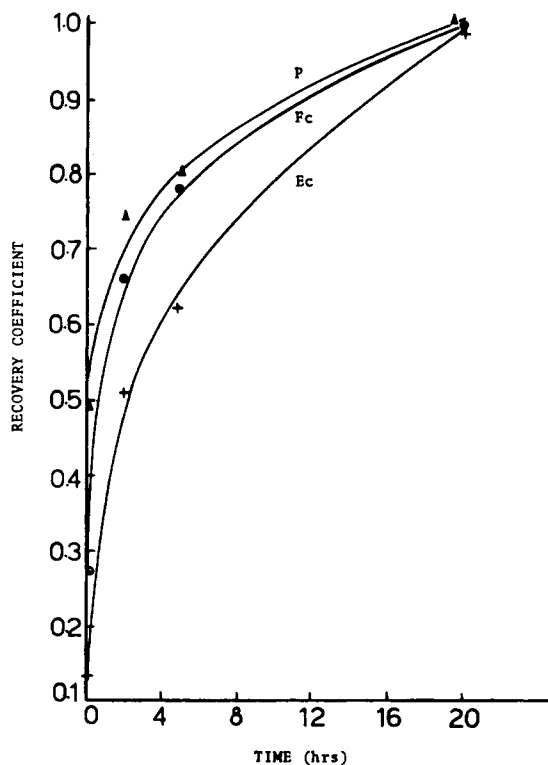


Fig. 6. Recovery coefficients of decrimping parameters P , F_c , and E_c after extension to 30% and relaxing for various times at 65% R.H.

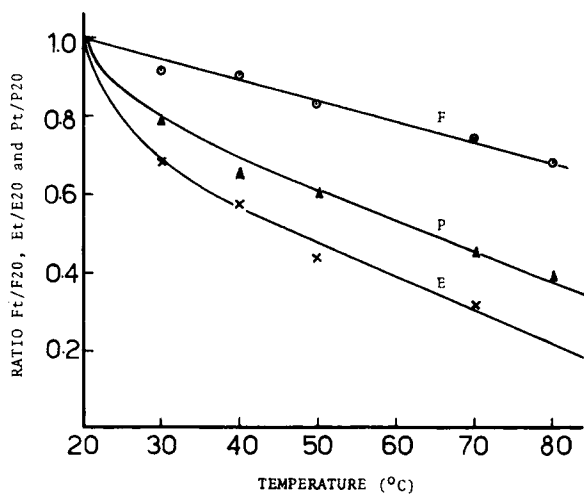


Fig. 7. Regression of decrimping parameters P , F_c , and E_c on temperature.

TABLE II
Effect of Decrimping at 80°C on Decrimping Properties at 20°C

Fiber no.	Before treatment			After treatment			Recovery coefficient			
	F_c , kg/mm ²	$E_c \times 10^5$, Joule/m ³	P	F_c , kg/mm ²	$E_c \times 10^5$, Joule/m ³	P	F_c , kg/mm ²	$E_c \times 10^4$, Joule/m ³	P	
1	0.275	0.31	0.112	0.260	0.31	0.092	0.945	1.000	0.821	
2	0.315	0.29	0.104	0.290	0.26	0.089	0.921	0.896	0.856	
3	0.350	0.36	0.109	0.280	0.30	0.090	0.800	0.833	0.826	
4	0.300	0.31	0.113	0.295	0.26	0.098	0.983	0.838	0.867	
5	0.300	0.30	0.111	0.280	0.24	0.094	0.933	0.800	0.847	
							Mean	0.916	0.873	0.843

crimp shape of the fiber and is independent of fiber diameter and elastic modulus.

For the fibers used in this investigation Walls' equation⁷ adequately describes the decrimping curve of wool fibers from the line of the Hookean slope down to loads of about 20 mg in air and about 5 mg in water, this range being greater than predicted by Walls.⁷ The goodness of fit of this relationship did not vary with the treatments applied.

In the treatment experiments, the crimp shape parameter P correlates well with the decrimping energy. This suggests that the decrease in decrimping energy observed is closely related to changes in crimp geometry.

The crimp behavior can be satisfactorily explained in terms of a two-phase theory of keratin structure involving the mechanical behavior of the crystalline alpha helices and the noncrystalline matrix.

Alternatively, the properties of the decrimping region do not appear to relate closely to the zone theory of Feughelman and Haly.^{11,12} This is revealed by the relationship between % extension and crimp recovery where it was noted that a linear relationship was obtained for extension and decrimping up to 40% in the wet state. This range of extension involves a transition from the X- to Y-zones. These higher levels of extension are considered to involve disulfide exchange and permanent loss of fiber strength, so that it would not have been surprising to see a deviation from linearity.

This work was financed in part by a grant from the Wool Research Trust Fund.

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Received February 14, 1974

Revised May 22, 1974