

Bending Stress Relaxation and Recovery of Wool, Nylon 66, and Terylene Fibers

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Synopsis

The bending stress relaxation and subsequent recovery behavior were determined for merino wool, nylon, and Terylene fibers. The effect of four experimental parameters were investigated, viz., the level of bending strain (0.5–4%), the time of stress relaxation before release (1–1000 min), the relative humidity (0–85%), and the temperature (20°–60°C). For small strains the merino and nylon fibers displayed behavior characteristic of linear viscoelastic materials, while Terylene exhibited a degree of nonrecoverable set. It was possible to construct master recovery curves for fibers held bent for different times before release. These curves can be used as a more convenient means of presenting the results. A relationship was found, for each fiber type, between the percentage stress relaxation and the time taken to recover to a given level of set. This relationship appeared to be independent of the experimental conditions employed. Although the fibers were not linear viscoelastic under all conditions, recovery could be roughly predicted from their stress relaxation behavior at the particular test conditions using the Boltzmann superposition principle.

INTRODUCTION

It has been suggested^{1,2} that the rheological behavior of textile fibers in bending is of more practical interest than that in tension since many of the time-dependent properties in textile fabrics with which we are most concerned (e.g., wrinkle recovery, creasing, crease retention, flexibility, drape, and handle) are those which primarily involve fiber bending.^{2–4} However, because of the unavailability of any suitable commercial fiber-bending apparatus and because of the considerable experimental difficulties involved, rheological studies on fibers have been almost exclusively confined to tensile strains. Moreover, simple tensile experiments may be more directly interpreted in terms of the standard theories of linear and nonlinear viscoelasticity^{5,6} than bending deformations which involve complex nonhomogeneous strains. Some authors^{4,7,8} have assumed that bending behavior may be predicted from the appropriate tensile data, but others^{2,9,10} have shown that for many situations this assumption is invalid.

Single-fiber bending recovery studies in the past (see review in ref. 10) have usually involved low strains and a nonconstant bending moment along the specimen length and have been carried out only under standard test conditions (20°C, 65% R.H.). However, Suzuki¹¹ has obtained bending stress relaxation curves of yarns under approximately pure bending moment

conditions. In order to approach approximately pure bending moment conditions, we have utilized the single-fiber bending apparatus previously described.¹² The experiments reported here are studies of the bending stress relaxation and subsequent recovery of the textile fibers merino wool, nylon 66, and Terylene, carried out under carefully controlled temperature and relative humidity conditions. Although a creep and recovery sequence would have been more fundamental from a theoretical standpoint,⁵ the stress relaxation and recovery sequence was felt to have more practical relevance.

The parameters studied include the bending strain to which the fiber is taken, the time for which it is held strained, the relative humidity, and the temperature of the surrounding air.

EXPERIMENTAL

Materials

For these tests, merino wool fibers (of $\sim 20\text{--}25\ \mu$ diameter) were used. They had previously been washed several times in petroleum ether and dried and, after several washings in distilled water, had been allowed to dry in air before being stored for several years in an envelope in the laboratory. These fibers are therefore considered "aged."¹³⁻¹⁶ The nylon 66 fibers were obtained from an I.C.I. multifilament yarn and were approximately $53\ \mu$ in diameter. After "boiling off"¹⁷ the fibers in distilled water for half an hour, they were allowed to condition in air before mounting. This treatment renders the synthetic fibers closer to the mechanical state of fibers in a finished fabric. However, this treatment is known to affect the mechanical properties of wool fibers negligibly and so was not used for these. The Terylene fibers were from an I.C.I. multifilament yarn of medium tenacity and were around $23\ \mu$ in diameter. They were boiled off in the same manner as the nylon fibers. Since no appreciable aging effects were observed in these synthetic fibers, no particular experimental provisions were made to take account of such effects.

Method

The specimens were mounted as previously described.¹² A standard test procedure was defined for these specimens. This involved conditioning the fiber for 1 hr at 20°C and 65% R.H., then quickly bending it (within 1 or 2 sec) to 2% bending strain and leaving it bent for 10 min before release. This entire experiment was carried out at constant temperature and R.H. conditions. The nominal bending stress (expressed in megapascals) was calculated as previously described¹⁰ and plotted against log time, zero time being taken at the point at which the straining was commenced. Recovery was recorded by plotting against log time, the set remaining in the fiber at any given time as a percentage of the imposed strain (i.e., percentage set = 100-percentage recovery). Zero time for recovery was taken as the time of release. In order to assess the effect of

TABLE I
Test Values for the Four Parameters Under Investigation

Bending strain, %	Stress relaxation time, min	R.H., %	Temp., °C
0.5			
1	1	0	
2 (Standard test)	10	65	20
4	100	85	40
	1000		60

different values of these parameters, they were varied one at a time from their standard value as indicated in Table I; i.e., while any one parameter was being varied, all the others were retained at their standard values.

Since the preparation of each fiber specimen involves considerable time and skill, it was desirable to use each specimen for as many tests as possible. Because of the good recovery properties of wool and nylon fibers, it was possible to use one specimen for examining the whole range of any one parameter. (A fiber subjected to the standard test would give identical stress relaxation and recovery results after 1 hr of recovery, if re-tested.) The Terylene fibers displayed a small but significant permanent set ($\sim 1\%$ or 2%) and loss of reproducibility; and therefore for each of the temperature and R.H. values, a fresh fiber had to be used. However, the one fiber was used for examining the range of strains and of time, since any effect remaining in the fiber would be swamped by the next value of the parameter.

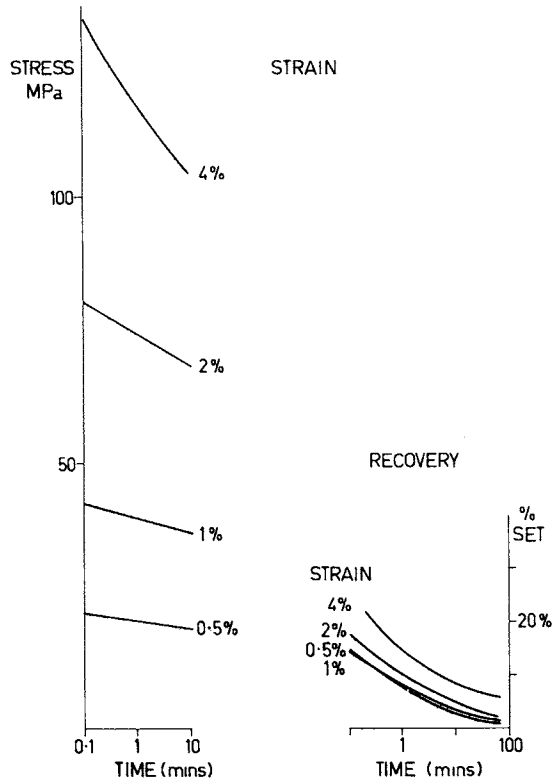
When one fiber was used to examine the whole range of any one parameter it was conditioned to the appropriate atmosphere for 1 hr, and then the tests were done in increasing order of severity. Thus, to study the effect of strain, a fiber would first be bent to 0.5% strain, all other parameters retaining their standard values. After 100 min of recovery, the fiber was then bent to 1% strain and recovered. Following this, the fiber was bent to 2% and 4% strain.

In a similar way, a fiber was held bent for increasing lengths of time, recovery being allowed after each test. For the investigation of temperature, the sequence used was 20° , 40° , and 60°C . However, for the relative humidity studies, it was considered that 85% was less severe than 0% , and the humidity variation was done in the order 65% , 85% , and 0% .

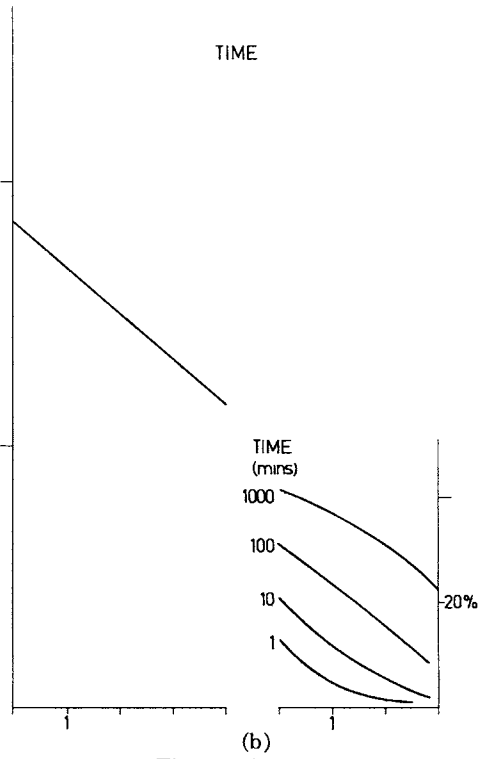
The results are summarized in Figure 1 where all sets of recovery curves have been drawn adjacent to their appropriate stress relaxation curves. Each curve is the mean for three fibers.

RESULTS AND DISCUSSION

The results are presented as an array in Figure 1 and are self-explanatory. Several points are worth noting, and these will be discussed below.



(a)



(b)

Fig. 1. (continued)

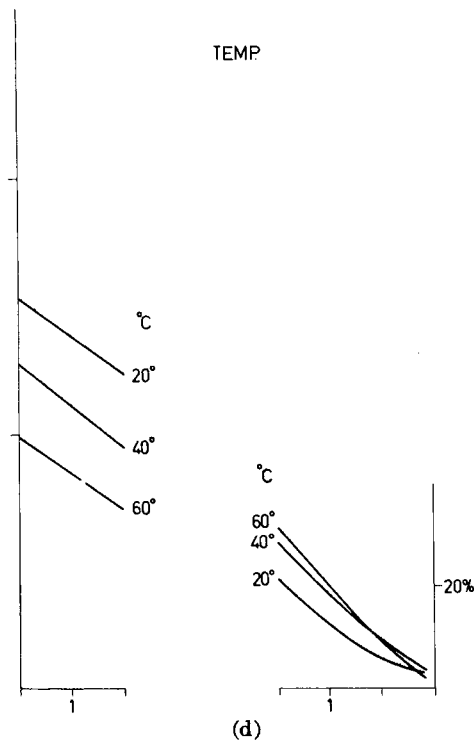
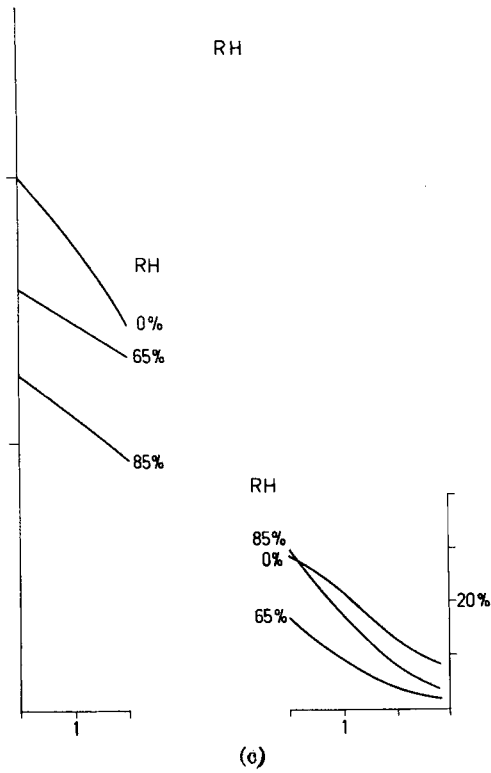


Fig. 1. (continued)

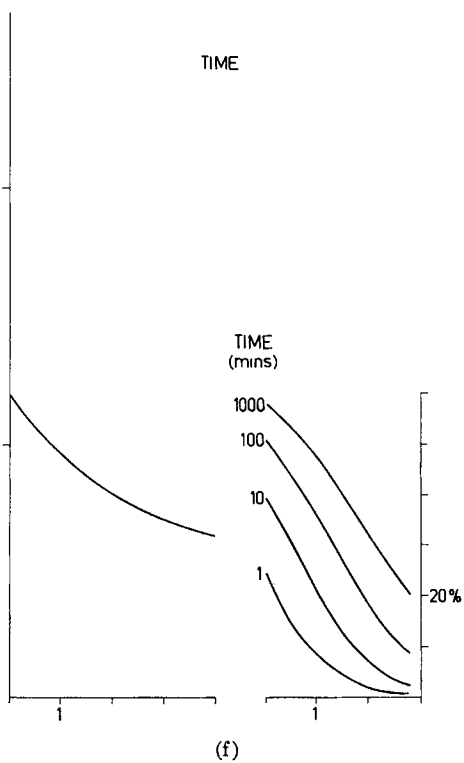
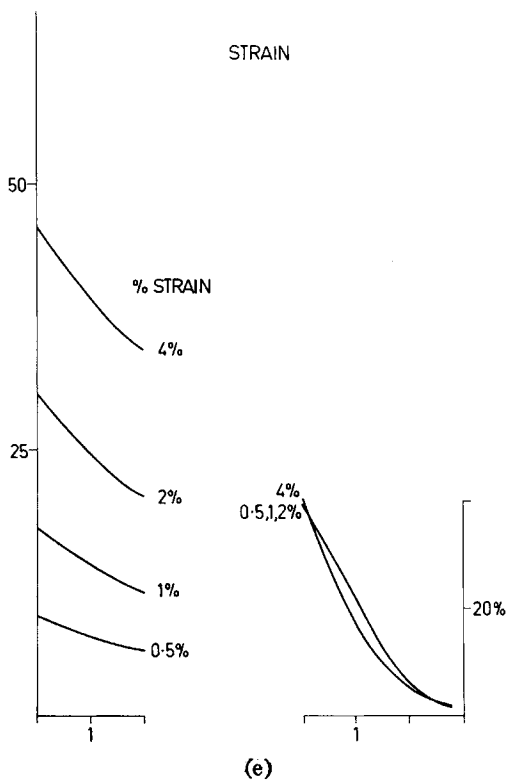


Fig. 1. (continued)

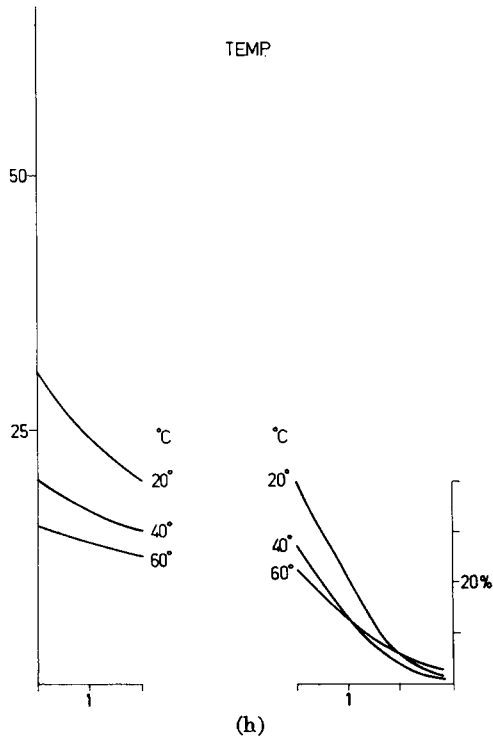
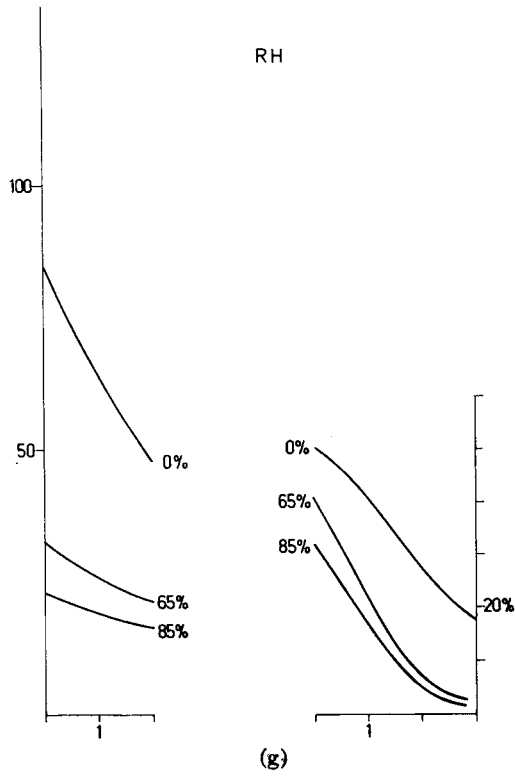


Fig. 1. (continued)

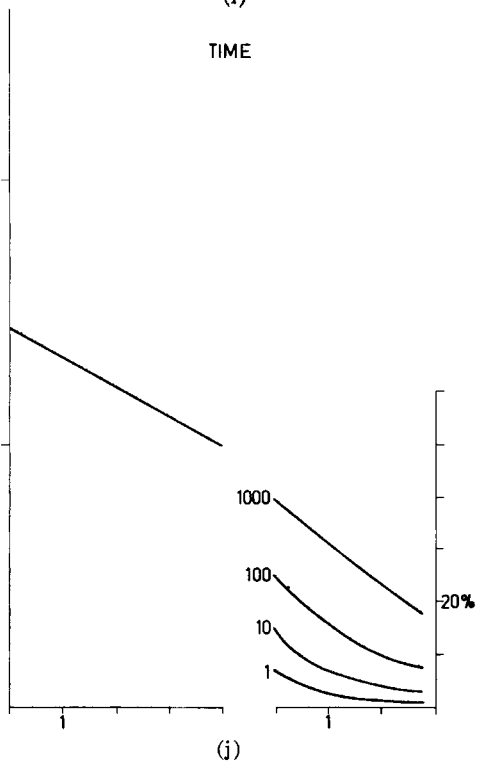
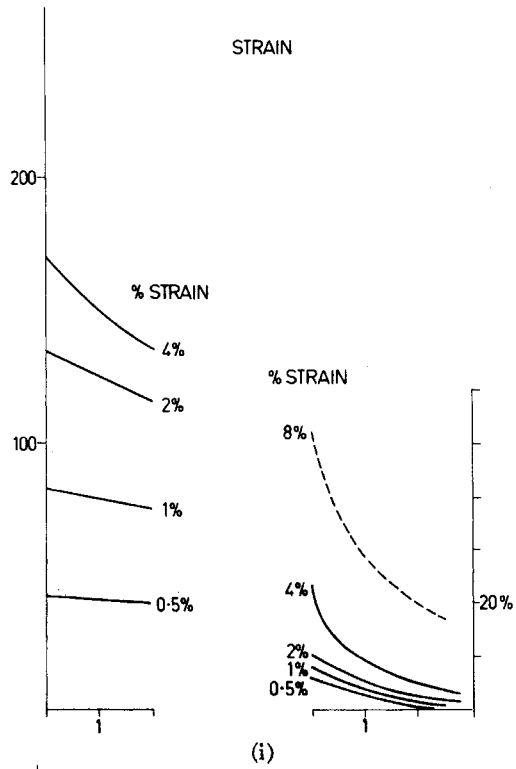
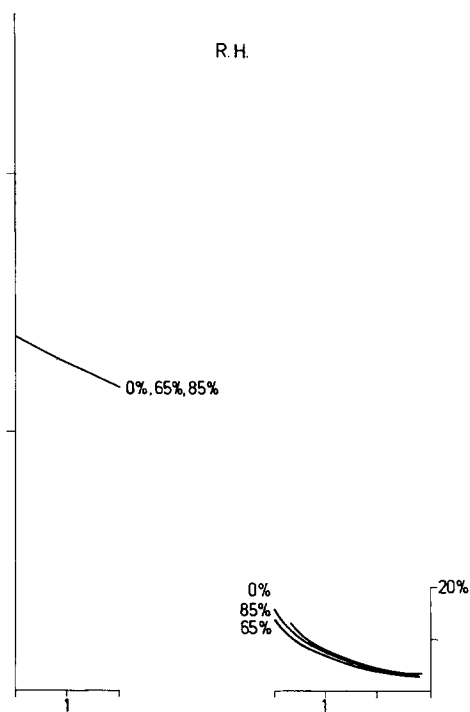
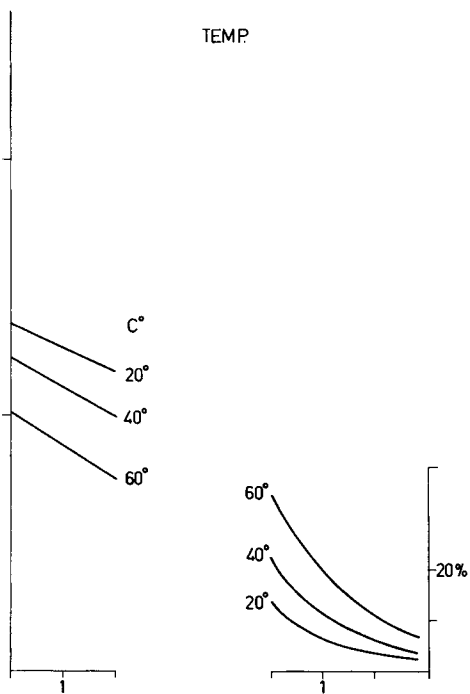


Fig. 1. (continued)



(k)



(l)

Fig. 1. Bending stress relaxation and subsequent recovery curves for various strains, times of stress relaxation, relative humidities, and temperatures for (a)–(d) merino wool; (e)–(h) nylon 66; (i)–(l) medium-tenacity Terylene.

Effect of Strain

The stress relaxation (bending) moduli calculated at 1% strain at the 1-min level for the nylon, wool, and Terylene samples are 1.4, 4.0, and 8.0 GPa, respectively. (Stress relaxation modulus is the stress divided by the strain at which the specimen is held, expressed as a function of time.) Stress relaxation is linear with log time for wool and Terylene at all strain levels, except 4% where a curvature (concave upward) is noted. This curvature is evident at all strain levels for nylon.

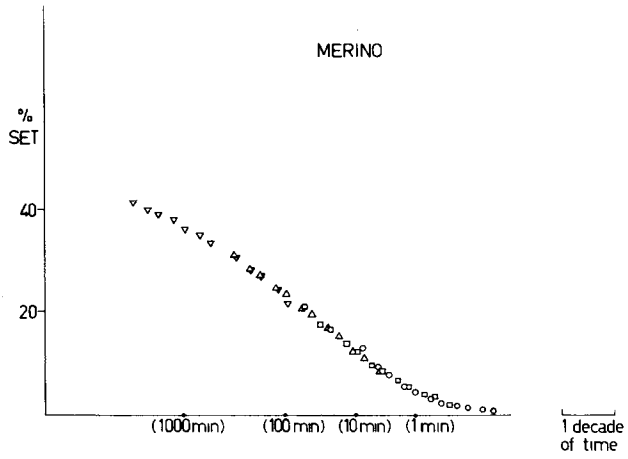
A necessary consequence of linear viscoelastic behavior⁵ is that the stress relaxation modulus and the resultant recovery curves should be independent of strain level. This is seen to be true for wool and nylon at 0.5% and 1% strain. (For nylon at 2% strain, the recovery curve is still the same as for the lower strain levels, but the stress relaxation modulus is reduced slightly.) Terylene does not satisfy this criterion and so cannot be considered linear viscoelastic at these strain levels. Increasing the strain level results in progressively poorer recovery for wool and Terylene, but the (percentage) recovery of nylon at 4% is as good or better than for the lower strains over most of the log time range studied. (Note that the result for 8% strain for one Terylene specimen is shown dotted in Fig. 1i.)

Effect of Time

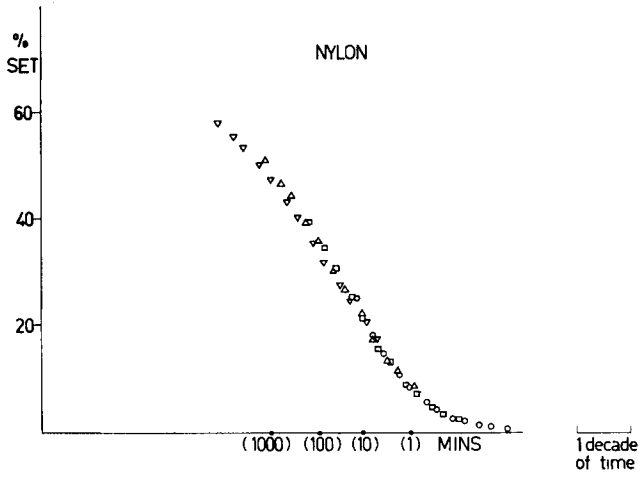
Over the four decades of time studied, the bending stress relaxation of wool and Terylene are linear with log time, while for nylon the relationship is curved (concave upward). These results indicate⁵ that over the time interval 0.1–1000 min, wool has a nearly constant distribution function of relaxation times, while for nylon we are looking at a tail of the distribution which is centered at a time shorter than we can measure, i.e., we are near the end of the stress relaxation process.

The recovery curves give the impression that they are portions of a master recovery curve which is shifted along the log time axis for various times of stress relaxation. The master curve can be built up in the standard way by shifting each recovery curve along the log time axis. This has been done for merino in Figure 2a. If we take the point ($t = 1$ min, % set = 0) as the origin of our graphs, then the respective origins corresponding to the stress relaxation times 1, 10, 100, and 1000 min may be plotted on the same graph as the master curve. The shifts necessary to form these master curves may then be calculated from the separation of these points.

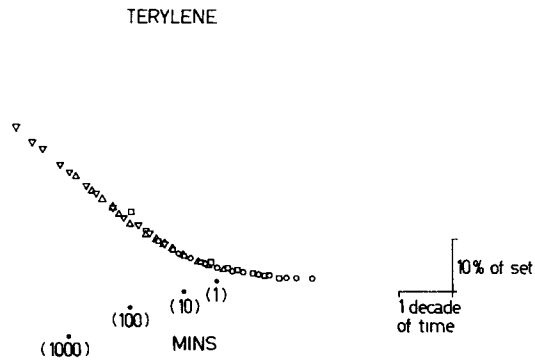
The recovery curves for Terylene will superpose only if both a horizontal and vertical shift are employed. The appropriate origins are marked on this master curve, which is given in Figure 2c. These origins move progressively back along the log time axis and drop below it with increase in stress relaxation times. These results are consistent with the idea that there is a viscoelastic component of recovery controlled by the master curve, in addition to a nonrecoverable viscous component (permanent set) which increases with time of stress relaxation. The nylon results do not superpose as satisfactorily, and this is shown in Figure 2b.



(a)



(b)



(c)

Fig. 2. Master recovery curves constructed from results for different times of stress relaxation for (a) merino; (b) nylon, and (c) Terylene fibers: (○) 1 min S.R. time; (◻) 10 min S. R. time; (Δ) 100 min S. R. time; (∇) 1000 min S. R. time.

TABLE II
Slopes of Master Recovery Curves

	Slope (% set/decade of time)	Standard deviation of slope (% set/decade of time)
Merino	8.7	1.8
Nylon	17.8	0.1
Terylene	8.6	0.6

Strictly speaking, these master recovery curves are valid only between one decade before and two decades after each appropriate origin and are an alternative and convenient way of representing the original results from Figure 1. However, they do suggest that the validity extends over a greater time interval. Justification for this suggestion will be presented below. Each master curve shown has been constructed from Figure 1, and the slopes of these curves are given in Table II. It may be noted that Terylene and nylon show little variation between fibers, while there is considerable variation between merino fibers. This probably indicates true variation in the physical properties of keratin itself.

Effect of R.H.

Wool is seen to recover better at 65% R.H. than at 0% or 85% R.H.; it seems probable that this is merely a reflection of the fact that the fibers are aged at 65%. When conditioned to 85% R.H. or 0% R.H. for 1 hr, the fibers are unaged under these conditions. Unaged fibers have been shown to have a greater bending stress relaxation and poorer recovery than when aged.¹⁵

Nylon is seen to recover better the higher the humidity, but Terylene is virtually unaffected by relative humidity. Any differences shown are within experimental error.

Effect of Temperature

Linear viscoelastic materials show a time-temperature equivalence⁵ such that stress relaxation curves for different temperatures should superpose to give a master curve. This master curve then gives the stress relaxation behavior of the material at one temperature over many more decades of time than it is convenient or possible to measure experimentally. Since in the present work we are dealing with nominal bending stresses¹⁰ (i.e., all calculations are based on the dimensions of the fibre at 65% R.H. and 20°C), any stresses shown for conditions other than 65% R.H. and 20°C will not be actual values and so cannot be validly compared since the dimensions will have altered.

Since Terylene does not absorb a significant amount of moisture,¹⁸ its dimensions should be virtually unaffected by different conditions; so all moduli would be actual moduli and could be compared directly. Nylon

swells only slightly,¹⁸ hence errors should be small; while wool swells markedly and even an approximate comparison would not be valid.

The stress relaxation curves for Terylene for different temperatures will not superpose, as can be seen from Figure 1. This is further evidence for its nonlinear viscoelastic behavior. The curves for nylon, on the other hand, do superpose reasonably well to give a curve which is close to that obtained for stress relaxation for 1000 min. This behavior is consistent therefore with its behavior at the various levels of strain.

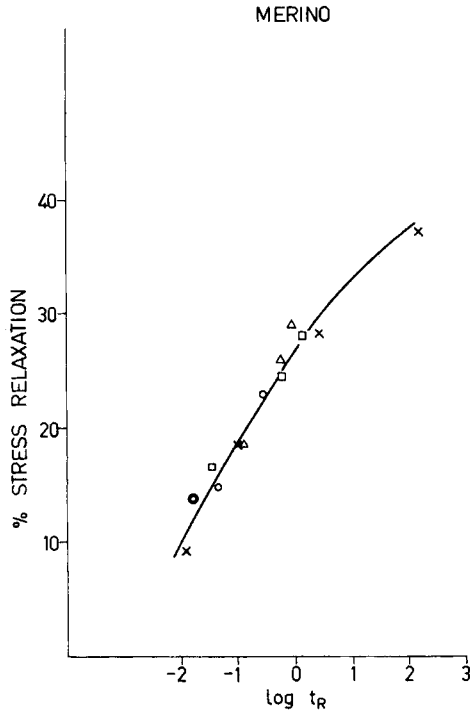
Wool at 20°C shows better recovery than at the higher temperatures, but this is probably again a reflection of the fact that the fiber is aged under the lower temperature conditions but deages when the regain changes at higher temperatures. In the early stages of recovery, nylon is better at higher temperatures but the curves cross over at longer times. Terylene shows a distinct decrease in recovery as the temperature is raised.

General

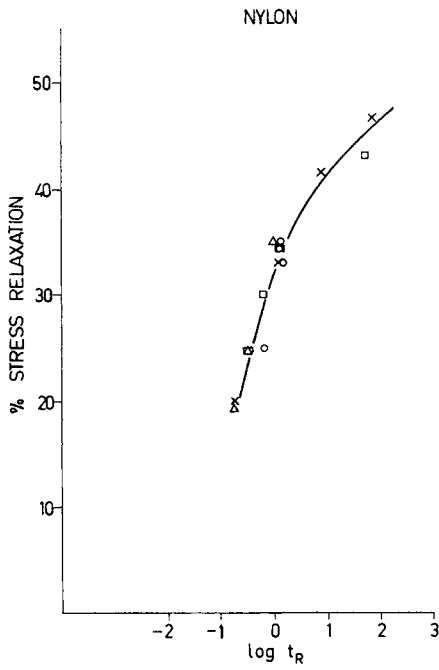
Each of the four graphs for each fiber type in Figure 1 contains the result for one (averaged) standard test. A comparison of these four results should give an indication of the amount of variation to be expected from this type of experiment. The coefficients of variation (between fibers) of the stress level at 1 min were found to be 11% for merino, 5% for nylon, and 2% for Terylene. The high variation between merino fibers may represent real variation in the keratin material as suggested above, but could also be caused by nonuniformity along the fiber length making determination of fiber dimensions difficult. Accurate determination of transverse fiber dimensions is essential, since modulus is dependent on the fourth power of this dimension.¹⁹ The error in modulus will be about four times the percentage error of measurement. Another possible source of error is the assumption that the fibers are elliptical in cross section, with either the major axis or the minor axis in the plane of bending.¹⁰ Bearing in mind the possible sources of error involved, the level of variation obtained is quite acceptable. From the results for all standard tests, the standard deviations in the vertical position of the recovery curves were found to be 1.8% for merino, 2.0% for nylon, and 1.3% for Terylene.

It was of interest to determine whether any relationship existed between the bending stress relaxation and subsequent recovery results recorded in Figure 1. As in previous work,¹⁵ we have utilized as a measure of recovery, t_R , the time taken for the fiber to recover to a certain level of set. For convenience, this level has been taken as 20% set for wool and nylon and 10% set for Terylene. (These levels were chosen because the corresponding times could be found from Figure 1 with at most a small extrapolation.) The measure of stress relaxation chosen was the percentage stress relaxation occurring between 0.1 min and the instant of release.

For all the experimental curves of Figure 1, the percentage stress relaxation has been plotted against the appropriate $\log t_R$ for wool, nylon, and Terylene in Figures 3a, 3b, and 3c, respectively. For each fiber type, the



(a)



(b)

Fig. 3. (continued)

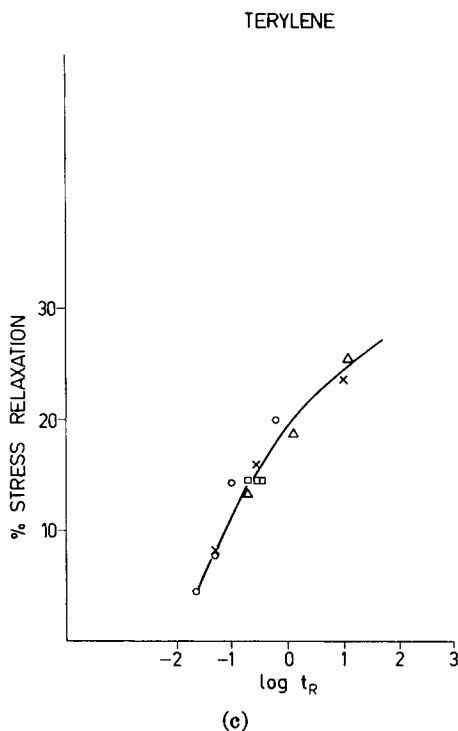


Fig. 3. Plots of percentage bending stress relaxation vs. logarithm of time taken for the fiber to recover to a given level of set. The level of set chosen was 20% for merino (a) and nylon (b) and 10% for Terylene (c): (○) strain experiments; (×) time experiments; (□) relative humidity experiments; (△) temperature experiments.

points for all experiments are seen to lie close to a single curve. These results suggest that irrespective of the conditions of the experiment (i.e., irrespective of the strain, time of stress relaxation, relative humidity, or temperature) the relationship between percentage stress relaxation and t_R is unique.

Application of the Theory of Linear Viscoelasticity

Since both wool and nylon fibers exhibit certain properties characteristic of linear viscoelastic materials, it is of interest to determine to what extent the theory can be applied. From a knowledge of the form of the stress relaxation function over a given time range utilizing the Boltzmann superposition principle,⁶ it is possible to compute the recovery behavior which would be expected (see Appendix). This may be done, provided the stress relaxation behavior is known, or can be estimated up to a time equal to the sum of the times allowed for stress relaxation and for recovery. Where necessary, second-order extrapolations have been made to the observed stress relaxation functions to estimate values outside the experimental range.

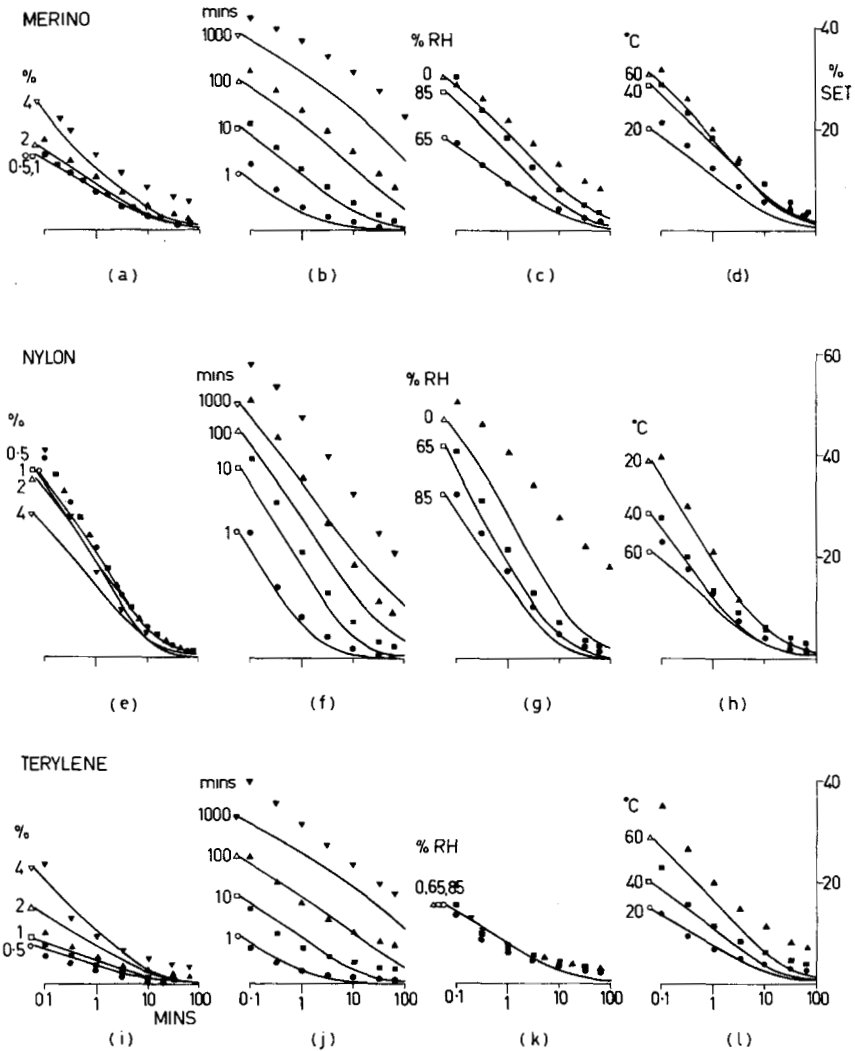


Fig. 4. Recovery curves predicted from stress relaxation data of Fig. 1. For comparison, the experimental recovery curves of Fig. 1 have been plotted as solid points (\bullet , \blacksquare , \blacktriangle , \blacktriangledown). The calculated recovery curves corresponding to these are indicated by the open points (\circ , \square , \triangle , \triangledown), respectively.

Where the stress relaxation moduli begin to drop above 1% strain level, it was obvious that the theory would not be strictly applicable. However, we have calculated recovery curves based on each of the experimental stress relaxation curves obtained under each appropriate set of conditions assuming the principle of superposition. The same approach was used to calculate recovery curves from the stress relaxation data obtained for Terylene, even though it was obvious that this material was not linear viscoelastic. Figure 4 shows the computed recovery curves obtained from

the stress relaxation data for merino, nylon, and Terylene fibers, and these can be compared directly with the recovery curves of Figure 1, which have been plotted as experimental points in Figure 4. Thus, we see that theoretical recovery curves for wool at 0.5% and 1% strain are exactly as observed. However, at 2% and 4% strain, departure from the theory is becoming significant with the experimental curves displaying poorer recovery than the predictions. For nylon, the predicted recovery for strains up to 2% is close to that observed while the superior recovery for 4% strain is consistent with the computed curve. The experimental results for Terylene, although in the same general region as the computed curves over most of the log time range, are of a distinctly different shape.

The computed recovery curves for wool (at 2% strain) up to stress relaxation times of 100 min (Fig. 4b) are slightly lower than the experimental ones. This is consistent with the observations on the effect of strain noted above. However, at a stress relaxation time of 1000 min, the predicted curve is much too low. This departure from the theory appears to be a result of the fact that we are working somewhat outside the region of linear viscoelasticity.

Other results²⁰ indicate that even for long times of stress relaxation, provided strains do not exceed 1%, the theoretical and experimental curves for wool agree surprisingly well. Note that the recovery curves for different times of stress relaxation are superposable by shifts along the log time axis. Although this superposition is not exact, the deviations are not great. Recovery calculations using different rates of stress relaxation have shown that this feature is characteristic of linear viscoelastic materials with linear stress relaxation-versus-log time relationships.

Significant departures from the theoretical predictions for nylon occur for stress relaxation times of 100 and 1000 min, although the general shape is still consistent. However, the agreement for 1 min and 10 min is quite good. Note that these predicted recovery curves do not even superpose well. Again we note that the computed curves for Terylene, although of a different shape, are in the same general region as the experimental results over the log time region.

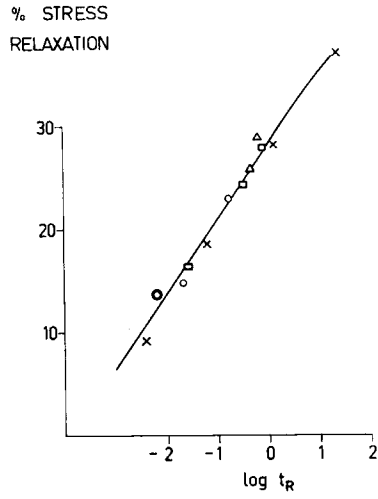
Except for nylon at 0% R.H., the predicted behaviors for wool and nylon at different relative humidities and temperatures are consistent with the experimental curves. As previously indicated, Terylene shows rough general agreement, but the predicted curves are of a different shape from those observed.

Figure 5 shows plots of percentage stress relaxation against $\log t_R$ for the computed curves, and the same general relationships are seen to exist as for the plots in Figure 3 obtained from the experimental curves. Thus, these relationships appear to reflect an aspect of superposition.

SUMMARY

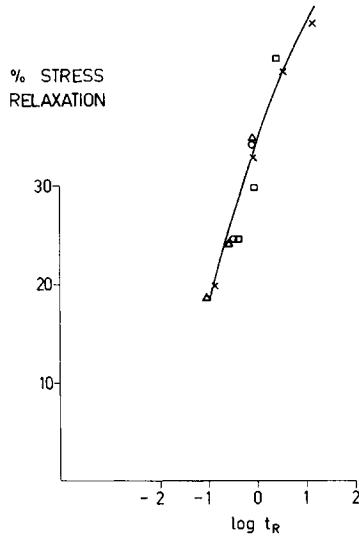
Bending stress relaxation and subsequent recovery of merino wool, nylon, and Terylene fibers were investigated at several strain levels for different

MERINO



(a)

NYLON



(b)

Fig. 5. (continued)

TERYLENE

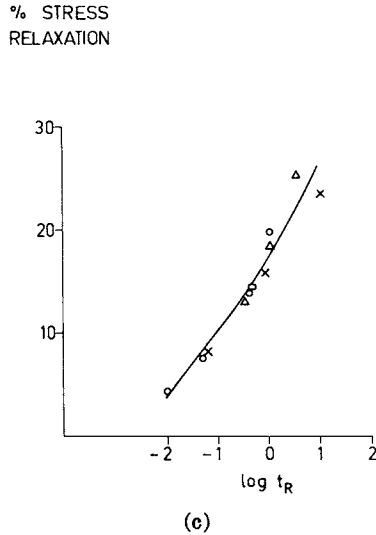


Fig. 5. Plots of percentage bending stress relaxation vs. log time as in Fig. 3 but using data obtained from the computed curves of Fig. 4.

times of stress relaxation, for various temperatures, and various humidities. Both wool and nylon at low strains exhibit behavior which is characteristic of linear viscoelastic materials.

Master curves for recovery were constructed from the recovery curves obtained for different times of stress relaxation. These results suggest that during stress relaxation Terylene undergoes a degree of viscous flow which results in a nonrecoverable component of strain or permanent set.

For any one fiber type, the relationship between the percentage stress relaxation and the time taken to recover to a fixed level of set appears to be independent of the conditions of the experiment.

Even though these fibers are not linear viscoelastic under all conditions, the recovery curves can be roughly predicted from the stress relaxation behavior through the principle of superposition.

Appendix

Calculation of Recovery from Stress Relaxation Data

The Boltzmann superposition principle may be expressed in incremental form⁶ as follows:

$$f(t) = \sum_{i=0}^n \Delta \epsilon_i G(t - \tau_i) \quad (1)$$

where $f(t)$ is the stress at time t , $G(t)$ is the stress relaxation modulus, $\Delta\epsilon_i$ are all the n incremental strains applied at times τ_i before time t .

Assume the specimen is subjected to an instantaneous strain $\Delta\epsilon_0$ at time $t = \tau_0 = 0$ and allowed to stress relax for time τ_1 . At the end of this interval, it is allowed to recover under a fixed stress f_0 . (This stress $f_0 = 0$ under free recovery.)

We approximate the recovery process by adding incremental strains $\Delta\epsilon_n$ at times τ_n such that

$$\lim_{t \rightarrow \tau_{n+1}} f(t) = f_0$$

i.e., at the end of each interval $(\tau_{n+1} - \tau_n)$ (before the application of the incremental strain at τ_{n+1}) the stress in the specimen is equal to the constant value f_0 . From eq. (1),

$$f_0 = \sum_{i=0}^n \Delta\epsilon_i G(\tau_{n+1} - \tau_i) = \Delta\epsilon_n G(\tau_{n+1} - \tau_n) + \sum_{i=0}^{n-1} \Delta\epsilon_i G(\tau_{n+1} - \tau_i)$$

or

$$\Delta\epsilon_n = \frac{1}{G(\tau_{n+1} - \tau_n)} \left\{ f_0 - \sum_{i=0}^{n-1} \Delta\epsilon_i G(\tau_{n+1} - \tau_i) \right\}.$$

Since the strain $\Delta\epsilon_0$ and time τ_0 for stress relaxation are known, $\Delta\epsilon_1$ may be computed from the above equation for any chosen τ_2 . Thus,

$$\Delta\epsilon_1 = \frac{1}{G(\tau_2 - \tau_1)} \{ f_0 - \Delta\epsilon_0 G(\tau_2) \}.$$

Similarly,

$$\Delta\epsilon_2 = \frac{1}{G(\tau_3 - \tau_2)} \{ f_0 - \Delta\epsilon_0 G(\tau_3) - \Delta\epsilon_1 G(\tau_3 - \tau_1) \}$$

and so on.

Values of τ_n are conveniently chosen such that the values of recovery time $(\tau_i - \tau_1)$ are equally spaced logarithmically. The more closely spaced these points, the more accurate will be the calculation.

The strain remaining in the specimen at time t where $t_n \leq t \leq t_{n+1}$ is $\sum_{i=0}^n \Delta\epsilon_i$, and the percentage set S is therefore given by

$$S = \frac{\sum_{i=0}^n \Delta\epsilon_i}{\Delta\epsilon_0}.$$

It is clear that the function $G(t)$ should be known within the interval $(\tau_2 - \tau_1) < t < \tau_n$, where τ_n is the sum of the stress relaxation time τ_1 and time allowed for recovery.

For the calculations reported here, each stress relaxation curve has been extrapolated back from 10^{-1} to 10^{-2} min using a second-order extrapolation technique. The first time of recovery $(\tau_2 - \tau_1)$ has therefore been set at 10^{-2} min, and subsequent values of recovery time $(\tau_n - \tau_1)$ have been set 0.1 of a decade apart. Steps of this size were found to give sufficient accuracy. For the theoretical recovery curves of Figure 4, the first decade (10^{-2} to 10^{-1} min) has been discarded, since any errors due to the approximate methods will be greatest here. The errors introduced into the value of recovery at 10^{-1} min by extrapolating the stress relaxation curve back one decade were thought to be less than if we had calculated using an initial recovery time of 10^{-1} min.

For those stress relaxation curves whose forms are not known beyond 10 min, the accuracy of the recovery curves are subject to a similar extrapolation from 10 min. For example, a recovery time of 10 min requires a small extrapolation of about 0.3 decade. As the recovery time becomes greater, the uncertainty increases.

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