THE TENSILE TESTING OF SINGLE WOOD PULP FIBERS IN AIR AND IN WATER

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ABSTRACT

A technique for the tensile testing of single wood pulp fibers in air and in water is described. Successive loading-unloading tests with increasing loads showed that the immediate elastic recovery of the wood pulp fibers was a linear function of the removed load. The slope of the straight line is inversely proportional to the rigidity of the fibers. Creep curves were recorded at 50% relative humidity and in water. As the testing method involves the measurement of the elastic recovery, the effect on the strain of the irreversible extension of microcompressions and crimps is eliminated. The method presented in this paper may therefore be more accurate for the determination of rigidity than those which evaluate rigidity from stress-strain curves. The tensile testing technique was critically examined, with special emphasis on the reliability of the adhesive used in attaching the fibers to the testing apparatus. The calculated elastic moduli and the creep behavior agreed well with literature data.

Keywords: Cellulose fibers, elastic strength, stiffness, tensile tests.

INTRODUCTION

This paper describes a technique to measure the elongation on loading of single wood fibers in air and in liquids. Emphasis is laid on the experimental procedure, and a method is outlined for the evaluation of rigidity. The inverse stiffness is derived from the ratio of the immediate elastic recovery to the removed load. By measuring the elastic recovery, nonelastic deformations due to flaws in the fiber are eliminated. This nondestructive testing method allows successive measurements to be made on the same single fiber in both air and liquid.

BACKGROUND

Although the mechanical testing of single fibers is a routine in the textile branch of fiber physics, it is still much of a vocation in wood fiber research. This is chiefly due to difficulties encountered in the testing of the short, chemically and physically heterogeneous wood fibers. If the fibers are fixed by mechanical gripping or by gluing, there is always a risk of fiber damage, slipping, and misalignment. Accurate measurement of the elongation at low strain and of fiber cross-sectional areas is difficult and laborious. Much work therefore needs to be devoted to solving the practical problems involved in the tensile testing of wood fibers.

Gripping

The problem of attaching the single wood fibers to the testing apparatus has been solved either by direct mechanical gripping or by gluing the fiber to some supporting device that is easily attached to the testing machine. Direct gripping with pin-vice mechanical grips (Jayne 1959, 1960; Kellogg and Wangaard 1964; Tamolang et al. 1967) has been abandoned in favor of gluing. In early work with single fibers, commercial cellulose-based glues (Leopold and McIntosh 1961; Hart-

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ler et al. 1963; Duncker and Nordman 1965; Mathevet 1965) or specially designed glues (Van den Akker et al. 1958) were used to attach the fibers to paper and cellulose acetate tabs. Paper testing wax has also been used to attach single wood fibers to aluminum tags (Smith and Morton 1968), but with the development of two-component epoxy glues, other adhesives have been all but ousted from single fiber work. Epoxy resins have been used to attach wood fibers to tabs of paper (Van den Akker et al. 1958; Russell et al. 1964), manila (Armstrong et al. 1977), brass (McIntosh and Uhrig 1968), cellulose acetate (Leopold 1966; Leopold and Thorpe 1968), paper prepared with wet strength resin (Kallmes and Perez 1966), glass (Page et al. 1972, 1977; Kim et al. 1975) and steel pins (Jentzen 1964; Spiegelberg 1966; Hill 1967; Hardacker 1970). An epoxy glue has also been used in a semimechanical gripping system, where the fibers were gripped by means of small oval-shaped epoxy droplets at each end of the fiber (Kersavage 1973).

Air-dry fibers have usually been tested in rooms kept under temperature and humidity control. Measurements carried out at higher humidities require a separate chamber (Kersavage 1973) and wood fibers in the wet state have been tested by applying a drop of water to the fiber (Russell et al. 1964; Kallmes and Perez 1966; Kersavage 1973) or by immersing the fiber in water (Leopold and Thorpe 1968). The former method is limited to a few minutes of testing, which excludes creep measurements. In the presence of an adhesive, the latter method can be subject to doubt about the resistance of the adhesive to water.

Misalignment

Misalignment of the fiber in the measuring apparatus can lead to shear stress and untimely breaking of the fibers during loading. It is therefore desirable that the fibers lie straight in the pulling direction. Perfect alignment can be achieved only if the fiber is free to align itself on loading. Leopold and McIntosh (1961) attached the fiber assembly to the tensile testing device by fine jewellers' chains, which allowed the fiber to align in the direction of the applied load. Later McIntosh and Uhrig (1968) found, however, that loading through chains and paper tabs decreased the precision of the elongation measurements, and then tried a more rigid clamping system in which the bottom clamp was suspended in molten lead, which was allowed to cool when the fiber system was properly aligned. Kallmes and Perez (1966) constructed special clamps to fit an Instron tensile tester. The lower clamp was fixed to an adjustable table that could be turned and tilted to permit vertical alignment of the fiber prior to measuring. Kersavage (1973) designed fiber grips to support fibers to which oval epoxy droplets had been affixed. When the fiber was placed in the grips and loaded, the epoxy droplets and the grips formed two ball-type joints, which allowed the fiber to align under tension. Most loading experiments in tension have, however, been carried out without special arrangements for self-alignment. Frequent failure at the glue line on loading of fibers has generally been ascribed to stress concentrations due to misalignment or gluing problems. Kallmes and Perez (1966) found, that on axial loading to break of over a thousand fibers, half of the breaks were at the glue. However, collected mechanical data on fibers that did not fail at the glue line, and on those that did, showed negligible differences. The same conclusion had earlier been drawn by Hartler and co-workers (1963) and Leopold and McIntosh (1961), and

Mathevet (1965) on tensile testing of 200, 100 and 60 wood pulp fibers respectively.

Evaluation of the elastic modulus

In all the published work on the elastic (tensile) modulus of single wood fibers, the modulus has been evaluated from load elongation curves, obtained at constant rate of loading or constant rate of elongation. Most load-elongation measurements have been made on commercial tensile testing machines at constant rates of elongation. The elastic modulus has been evaluated from the initial slope of the load-elongation curve (Russell et al. 1964; Leopold 1966; Kallmes and Perez 1966; McIntosh and Uhrig 1968; Page et al. 1977), or from the ratio of stress to strain over the linear portion of the curve (Jayne 1959, 1960; Kellogg and Wangaard 1964; Tamolang et al. 1967; Leopold and Thorpe 1968; Kersavage 1973). In work performed at the Institute of Paper Chemistry in Appleton, Wisconsin, the IPC "Fiber Load-Elongation Recorder" has been used (Jentzen 1964; Spiegelberg 1966; Van den Akker et al. 1966; Hill 1967; Hardacker 1970). In the IPC recorder, which was developed by Hardacker (1962), the fiber elongation is recorded at a constant rate of loading. The elastic modulus is evaluated from the slope of the initial portion of the curves. Duncker and Nordman (1965) designed and built an apparatus to record load-elongation curves at a constant rate of stretch. The elastic modulus was determined from the mean slope of the proportional part of the stress-strain curve. Armstrong and co-workers (1977) used a "microtensile testing stage," where elongation and load were measured at regular intervals during the test.

In order to calculate elastic moduli from stress-strain data, the cross-sectional areas of the fibers must be known. Cross-sectional areas have been measured by microscopy and by the compacting method introduced by Hardacker (1969) and modified by Kim and co-workers (1975). Microscopic measurements have been made of perpendicular microtome cuts of embedded fibers. McIntosh and Leopold and co-workers (1961, 1966, 1968) measured fibers embedded in cellulose acetate, while Smith and Morton (1968) used ice as an embedding agent. Photomicrographs of the sections were projected with high linear magnification and the areas were measured by planimetry. Jayne (1959, 1960) viewed the fibers end-on at 1,000× magnification and measured the area of the fiber using a calibrated micrometer eyepiece. Tamolang and Wangaard (1961) calculated the lumen width from fiber width and cell-wall thickness. The cross-sectional area was determined by assuming circular fibers and by subtracting the lumen area from the total area.

The method of Kersavage (1973) involves direct photography and measurement of the area from prints. Armstrong and co-workers (1977) photographed fiber cross sections in a scanning electron microscope and measured the area from the micrographs. In the compacting method, which has been used by Spiegelberg (1966), Hill (1967), Hardacker (1970) and by Page and co-workers (1977), the cross-sectional area is obtained from compacted fiber width and thickness. In this method large pores are eliminated and the cross-sectional area should therefore represent the actual fiber substance better than does the microscopically determined area. The compacting method gives an average cross-sectional area over the length of the fiber, and should therefore be more suitable for the evaluation

of the elastic modulus than is the area of one single section of the fiber. Kallmes and Perez (1960, 1966) determined the cross-sectional area of collapsed kraft pulp fibers as the product of width and double cell-wall thickness of noncompacted fibers. Spiegelberg (1966) based the elastic modulus on the cellulose area. The cellulose area was obtained by plotting the percentage of glucose in fibers of different hemicellulose content, versus average cross-sectional area and linearly extrapolating to 100% glucose.

In some cases the stiffness of single fibers has been evaluated without the measurement of cross-sectional areas. Jentzen (1964) evaluated the modulus of single fibers using cross-sectional areas that were derived by dividing the mass per unit length by the pycnometric density. Leopold and Thorpe (1968) used the concept of "initial load ratio" to describe the stiffness of wet fibers, the cross-sectional area of which they had not established, this ratio being the product of the elastic modulus and the cross-sectional area. Kellogg and Wangaard (1964) and Tamolang et al. (1967) considered their stress-strain data too uncertain to evaluate the stiffness in terms of elastic modulus. They preferred to use the concept of "strain at equivalent stress," which is inversely proportional to fiber stiffness.

The calculation of elastic moduli of fibers in the wet state requires a knowledge of the cross-sectional area of the wet fiber. No measurements of the cross-sectional area of wet fibers have been reported. Sakurada et al. (1964) in their work on ramie fibers neglected the increase of the cross-sectional area on wetting, and used the cross-sectional area of the air-dry fibers. According to Sakurada, this simplification is reasonable, since an important quantity determining the stress is presumably the total number of chains available to bear stress, and not the cross-sectional area of the fiber. Russell and co-workers (1964), Kallmes and Perez (1966), and Kersavage (1973) probably also used the dry cross-sectional area, as nothing is mentioned in their papers. Leopold and Thorpe (1968) chose to evaluate only the "initial load ratio" for the wet fibers tested.

Classification of fibers

In early single fiber work, much labor was devoted to the separation of early- and latewood fibers prior to mechanical testing. It was shown that early- and latewood fibers had different strengths and stiffnesses (Jayne 1959, 1960; Leopold and McIntosh 1961; Duncker and Nordman 1965; Leopold 1966; Leopold and Thorpe 1968; McIntosh and Uhrig 1968; Nordman and Qvickström 1970) and that only very careful separation of early- and latewood fibers within a growth ring could bring down the coefficient of variation of strength and stiffness between the individual fibers to a level of 10% (Nordman and Qvickström 1970). Later Page and co-workers (1972, 1977) found that the difference in strength and modulus between early- and latewood fibers could be explained by a difference between the fibril angles. Thus instead of classifying the fibers into early- and latewood fibers, Page and co-workers classified them according to their fibril angle. Page and El-Hosseiny (1973) measured the fibril angles using a mercury reflection technique. Another technique, involving scanning electron microscopy of fractured surfaces of axially loaded fibers, was used by Armstrong and co-workers (1977). Tamolang and co-workers (1967) determined the fibrillar orientation of hardwood fibers by microscopy. Bordered pits having canals with distinct elliptical

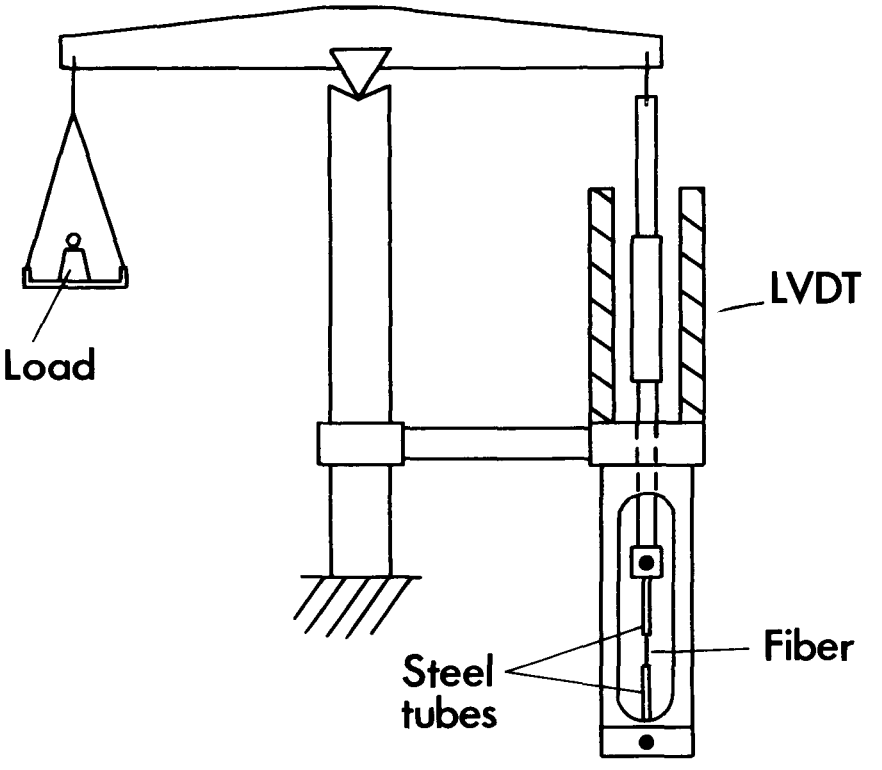

FIG. 1. A schematic drawing of the tensile testing apparatus.

apertures were measured for their angle of inclination from the fiber axis. The long axis was assumed to be parallel to the mean microfibrillar direction of the S_2 layer.

EXPERIMENTAL

Fibers

The wood fibers used in the present study were Asplund-type spruce pulp fibers, which had been delignified to different lignin contents, using buffered sodium chlorite solutions. The chemical and physical properties of the delignified fibers have been discussed in detail in earlier papers (Ehrnrooth 1982). The fibers were selected and prepared for testing in the following manner. Air-dry fibers were suspended in distilled water to make up a very dilute suspension. A few drops of the fiber suspension were poured onto a black plastic sheet, on which the fibers were allowed to dry. The fibers were viewed on the plastic sheet with a stereomicroscope and long single fibers without visible damage were chosen for testing. To straighten out the fiber, the dry fiber was gripped at one end with tweezers and pulled through a droplet of water on the black plastic sheet into a straight groove created by the tweezers as they passed across the sheet. The straight, air-dry fibers were glued with a freshly mixed two-component epoxy adhesive (Araldite) between the points of stainless steel pins with a separation of 0.8 to 1.6 mm.

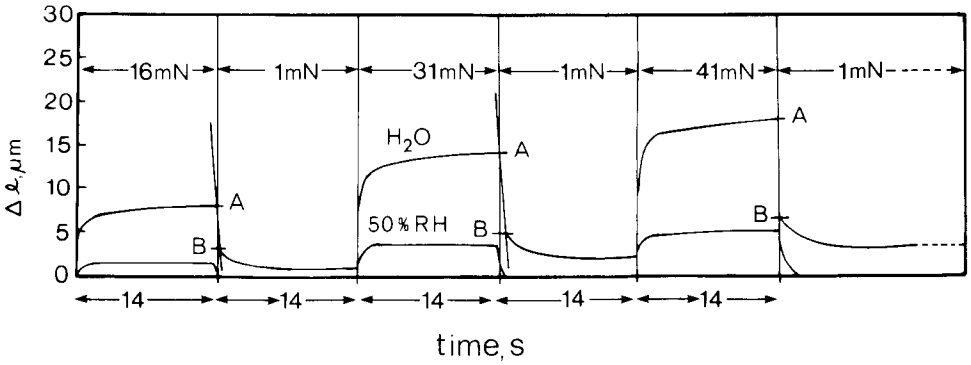

FIG. 2. Successive loading-unloading with increasing load of a wood pulp fiber at 50% RH and in water. Chart speed 120 mm/min. AB = immediate elastic contraction.

The gluing was performed under a stereomicroscope. The glue was allowed to cure for at least 24 hours at room temperature before the fibers were tested.

The cross-sectional areas of wood pulp fibers were determined. In the microscopy method, the fiber, which was glued to a steel pin, was immersed in a droplet of water on a wooden cutting board, and frozen at −30 C. A razor blade was used to cut the frozen fiber in the ice drop. This cutting procedure is not perfect with regard to the cutting angle. After being cut, the fiber was allowed to thaw and dry and was viewed end-on in a microscope. The cross section was projected onto a paper where it was drawn at about 1,000× total magnification. The cross-sectional area was determined from the drawn image by planimetry. The cross-sectional areas were also determined using the compacting method of Kim and co-workers (1975). The determinations were made on fibers that had not been mechanically tested. The fibers had, however, been chosen in the same mode as the actually tested fibers and should therefore represent the tested fiber material. Measurements were made on 3 to 4 fibers of each species using parts of the same individual fibers in both methods.

In the present paper, reference is made to measurements on rayon and ramie fibers. The rayon fibers were furnished by Prof. Erich Treiber at the Swedish Forest Products Research Laboratory. The fibers had been stretched 70% in the wet state and their crystallinity was assumed to be about 60%. The ramie fibers were furnished by Dr. Volker E. Stöckmann. They were native fibers, which had been purified in 1 to 2% sodium hydroxide for several hours (Stöckmann 1971). The compacted cross-sectional areas of the rayon and ramie fibers were $6.5 \cdot 10^{-10}$ and $5.1 \cdot 10^{-10}$ m^2, respectively.

Load-elongation measurements

The apparatus used in tensile loading of the fibers, is schematically drawn in Fig. 1. The steel pins holding the fiber are inserted into a fixed clamp and a movable clamp that is suspended from an ordinary beam balance. The deformations of the fibers were measured with a linear variable differential transducer (LVDT), its core being a part of the connection between the movable clamp and the beam. To improve the alignment of the system, PTFE bushings were mounted

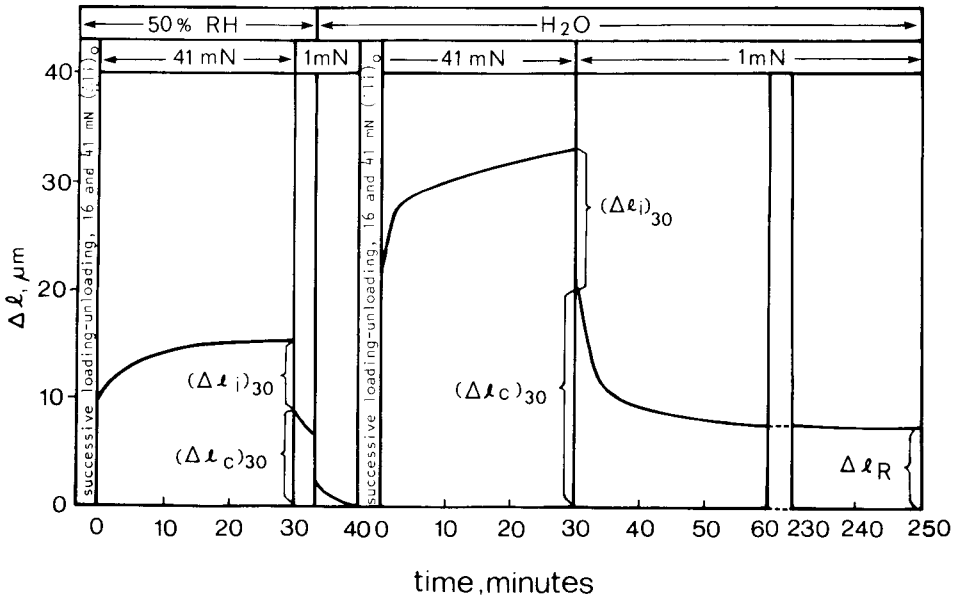

FIG. 3. The creep and recovery of a wood pulp fiber under 41 mN load at 50% RH and in water.

at each end of the LVDT core. A minimum load of 1 mN (0.1 g) was used to balance the system and straighten the fiber. A cathetometer was used to calibrate the signal from the LVDT and to measure the absolute fiber length. The resolution of the recorded LVDT signal was better than 1 μm. The experiments were carried out in a room kept at 23 $\pm$ 1 C and 50 $\pm$ 5% relative humidity. Measurements in water were performed by raising a beaker containing water to a position where the fiber was just immersed. The loads were calibrated copper rings. They were applied and removed by means of a lever that was moved by a motor-driven eccentric cam. The automatic system was designed to give smooth and sufficiently rapid loading and unloading. Before the actual load-elongation measurements, the fibers underwent a standard conditioning treatment, consisting of soaking the fiber in water for 15 minutes and drying at 50% relative humidity for 12 hours with the minimum load of 1 mN. During this conditioning prior to creep tests, the fibers were tensile loaded with 41 mN for 14 seconds in water.

Two different kinds of load-elongation measurements, successive loading-unloading with increasing load, and creep tests at constant load, were carried out with wood fibers at 50% relative humidity and in water. Loading tests were performed with loads between 5 and 65 mN at 5 or 10 mN intervals. A low load was applied and left on the fiber for 14 seconds. The load was taken off and left off for 14 seconds. The procedure was then repeated for successively higher loads. For each individual fiber, the loading test was carried out at 50% relative humidity and then in water. The loading is schematically illustrated in Fig. 2. The immediate contraction, Δl_i, is defined as the difference between the fiber extension before releasing of the load, and the fiber extension at the point where the time-extension curve departs from the tangent AB. From a series of Δl_i values at different loads, the rigidity can be determined.

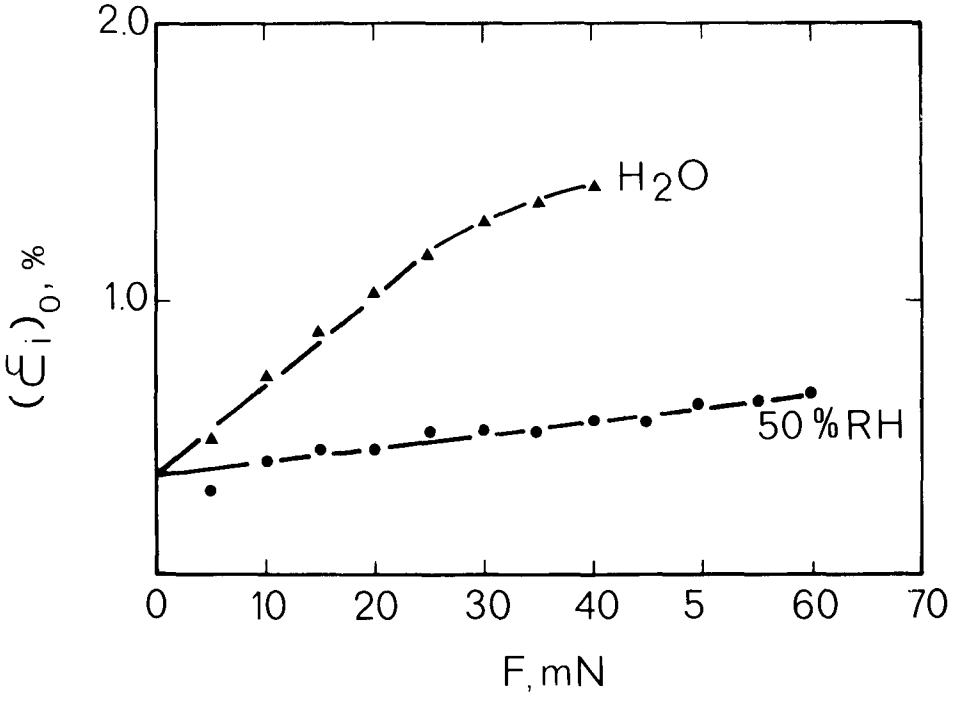

FIG. 4. Elastic recovery versus removed load for a wood pulp fiber at 50% RH and in water.

Creep tests were carried out under constant loads of 16 or 41 mN. In the series at 16 mN, fibers were tested under load for periods of up to 24 hours duration in either air at 50% relative humidity or in water. In the series at 41 mN, both creep and recovery phenomena were studied as illustrated in Fig. 3. In order to determine the rigidity of the fiber before creep, the fiber was initially subjected to successive loading-unloading under 16 mN and 41 mN. The fiber was then allowed to creep under a load of 41 mN for 30 minutes, after which it was allowed to recover for ca. 5 min and then soaked in water. After the rigidity in water had been determined by successive loading-unloading under 16 mN and 41 mN as before, the fiber was allowed to creep under a load of 41 mN for 30 minutes. Finally the recovery in water was studied.

From these creep tests, four deformation characteristics were derived: the immediate contraction in successive loading-unloading, $(\Delta l_i)_0$, the immediate contraction after creep, $(\Delta l_i)_{30}$, the creep extension, $(\Delta l_c)_{30}$, and the residual extension after recovery, Δl_R. The elastic recovery, ϵ_i, is the immediate contraction, Δl_i divided by the initial fiber span length, l_0. In the present study, the initial dry fiber length has been used in calculating the elastic recovery both at 50% relative humidity and in water. The creep deformation, CD, is the creep extension, Δl_C, divided by the initial fiber span length, and the permanent deformation, PD, is the residual extension after recovery, Δl_R, divided by the initial fiber span length. After successive loading-unloading tests or creep tests, wood fibers were loaded to break at 50% relative humidity or in water. The loading to break was performed by adding lead-shot at a constant rate of 1 lead-shot every 5 seconds. The calculated mean loading rate was 1.57 mN/5 sec.

TABLE 1. *The slopes, $k = (E \cdot A)^{-1}$, of 20 wood fibers at 50% relative humidity and in water.*

Lignin content %	Degree of delignification %	Earlywood (EW)–latewood (LW)	Slope, $k = (E \cdot A)^{-1}$ 50% RH N^{-1}	Slope, $k = (E \cdot A)^{-1}$ H_2O N^{-1}
26.3	0	–	0.147	0.389
26.3	0	–	0.136	0.429
26.3	0	EW	0.080	0.240
26.3	0	LW	0.064	0.304
26.3	0	LW	0.100	0.247
24.7	6.1	EW	0.095	0.600
24.7	6.1	LW	0.054	0.316
22.5	14.4	LW	0.058	0.220
22.5	14.4	LW	0.050	0.334
22.5	14.4	LW	0.060	0.334
22.5	14.4	LW	0.076	0.447
22.5	14.4	–	0.060	0.367
14.0	46.8	EW	0.164	0.900
14.0	46.8	EW	0.106	0.730
14.0	46.8	LW	0.039	0.318
9.9	62.4	–	0.209	1.340
9.9	62.4	EW	0.164	0.593
9.9	62.4	LW	0.100	0.753
9.9	62.4	LW	0.172	0.559
9.9	62.4	LW	0.084	0.364

RESULTS AND DISCUSSION

Fiber stiffness

The elastic recovery of fibers.—The successive loading-unloading test on a single wood pulp fiber at 50% relative humidity and in water is illustrated by Fig. 2. On loading the fiber, there is an immediate elongation followed by a slower creep deformation. On unloading there is an immediate contraction followed by a slower recovery. The residual deformation increases with the load and is higher in water than at 50% relative humidity.

The elasticity of a body is defined as the ability of the body to recover its size and shape after deformation. The tensile elasticity of the fiber at any given stress is the ratio of the extension recovered in a reasonable time to the total extension. Meredith (1956) has called this ratio the elastic recovery. The elastic recovery can be divided into two components: the immediate elastic recovery and a delayed creep recovery.

The immediate elastic recovery, ϵ_i, is shown in Fig. 4 versus the removed load, ΔF, for one wood pulp fiber at 50% relative humidity and in water. For small loads, the immediate recovery for every wood pulp fiber tested at 50% relative humidity and in water is a linear function of the load, $\epsilon_i = k \cdot F + a$. There is deviation from linearity at higher loads in water. For a given fiber, the intercept, a, is approximately the same in water and at 50% relative humidity, but the magnitude of the intercept differs for different fibers. Measurements with rayon fibers (Ehrnrooth, unpublished results) have shown that the intercept decreases with increasing fiber length and improving alignment, while the slopes are in-

TABLE 2. *The mean slopes, $\bar{k}$, of wood fibers at 50% relative humidity and in water. (Coefficient of variation = (standard deviation/mean value) × 100, in parentheses.)*

Lignin content %	Degree of delignification %	$\bar{k}_{50\%\ RH}$ N^{-1}	$\bar{k}_{H_2O}$ N^{-1}
26.3	0	0.105 (34)	0.322 (26)
24.7	6.1	0.075	0.458
22.5	14.4	0.061 (15)	0.340 (24)
14.0	46.8	0.103 (61)	0.649 (46)
9.9	62.4	0.146 (36)	0.722 (52)

dependent of alignment and fiber length. The shift of the straight lines from the origin is theoretically treated in the appendix. It is shown that misalignment of the fiber and the LVDT core can lead to a shift of the same magnitude as the intercept recorded in the present work. Thus it is suggested that the correct values for the immediate elastic recovery are obtained by shifting the curves vertically to the origin. The relationship between load and elastic recovery can, however, be derived from the original data as presented in Fig. 4. As the immediate elastic recovery is a linear function of the removed load, Young's formula relating stress, strain and elastic modulus can be applied. It follows that the inverse of the slope, k^{-1}, equals the product of the modulus and the cross-sectional area of the fiber, $E \cdot A$.

Evaluation of fiber stiffness.—The slopes, k, have been evaluated from the experimental data in the following manner. At 50% relative humidity all the experimental points have been used to calculate the straight lines by means of the least squares method. The correlation coefficients for the straight lines varied between 0.93 and 1.00. In water, the points used in the calculations have been chosen graphically by excluding all points beyond the load at which the deviation from linear behavior becomes obvious. Based on the theoretical considerations in the appendix, it is stipulated that the intercept should be equal in water and at 50% relative humidity. Thus the intercept of the straight line at 50% relative humidity has been included in the points used for the calculation of the straight line for measurements made in water. The slopes calculated in this manner are called "statistical" slopes. The slopes for wood fibers of different lignin contents at 50% relative humidity and in water are shown in Table 1.

In one set of creep experiments, elastic recovery was measured at two loads, 15 mN and 40 mN, only. The slopes, k, were in this case evaluated in the following manner. At 50% relative humidity the slope was calculated from the two experimental points. In water the slope was calculated from the straight line between the intercept at 50% relative humidity and the experimental point at 15 mN. The slopes calculated in this manner are called "geometrical" slopes. The geometrical slopes were also calculated for the set of fibers for which the statistical slopes had been determined. The results show that the mean values of the slopes evaluated by the two methods are of the same magnitude. The standard deviation, however, is greater for the geometrical slopes. Nevertheless, the slopes derived from the geometrical evaluation of two experimental points are comparable with slopes derived statistically from a greater number of experimental points. In Table 1,

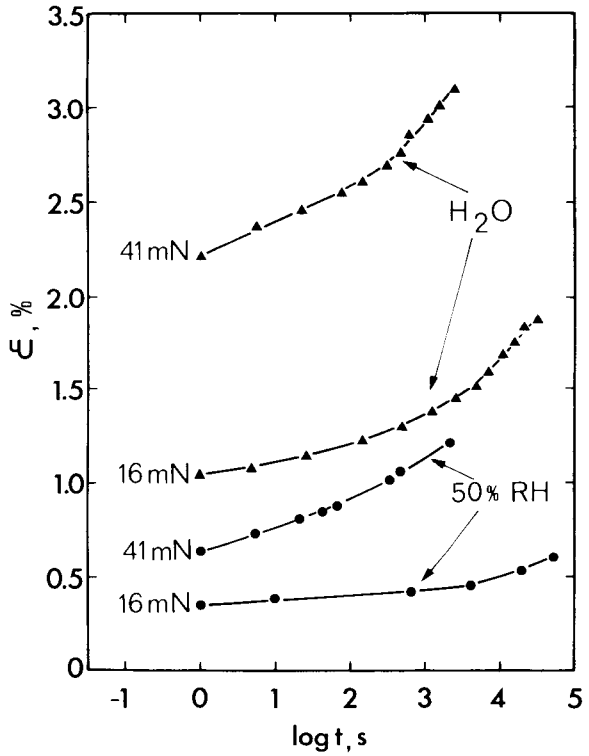

FIG. 5. Elongation versus log time at 16 and 41 mN load at 50% RH and in water for two wood pulp fibers of 9.9% lignin content.

which shows the slopes of the linear load-recovery curves of wood fibers, the geometrical slopes have been included for fibers for which statistical slopes were not available.

Some of the fibers used in the successive loading-unloading tests were later loaded to break at 50% relative humidity in water. In a few cases the fibers slipped from the glue at about 250 mN load. As no distinction was observed between the slopes of the fibers that eventually slipped and those that broke, the slopes of all fibers of both categories were included in our data. Microscopic investigations of the fiber cross-sectional areas provided a tool for the qualitative classification of the tested fibers. Available data have been included in Table 1. The mean slopes of wood fibers at 50% relative humidity and in water and the coefficient of variation of the experimental data are reported in Table 2.

Creep

The strain versus log time under loads of 16 and 41 mN at 50% RH and in water for two wood fibers of 9.9% lignin content is shown in Fig. 5. The creep deformation increases with load and on wetting of the fiber. There is a low-deformation initial creep, followed by a high-deformation logarithmic creep linearly related to log time. The time until the onset of logarithmic creep decreases with increasing load and with immersion in water. At 50% relative humidity, a

TABLE 3. *The mean cross-sectional areas of wood pulp, rayon and ramie fibers measured by compacting and by microscopy. (Coefficient of variation = (standard deviation/mean value) × 100%, in parentheses.)*

Fiber	Lignin content %	Degree of delignification %	Microscopy cross-sect. area $m^2 \times 10^{12}$	Compacting cross-sect. area $m^2 \times 10^{12}$	Number of fibers measured
Wood	26.3	0	402 (20)	340 (30)	4
Wood	24.7	6.1	359 (20)	294 (32)	3
Wood	22.5	14.4	278 (27)	215 (18)	3
Wood	14.0	46.8	356 (20)	264 (3)	3
Wood	9.9	62.4	364 (39)	319 (31)	4
Rayon			637 (2)	652 (0)	3
Ramie			611 (13)	505 (24)	3

load of 16 mN (~50 MPa stress) is not sufficient to start logarithmic creep of the fiber below 10^5 seconds. In water the logarithmic creep is initiated after 30 minutes under load.

CRITICAL EXAMINATION OF THE FIBER TESTING METHOD

The fibers used in the mechanical tests came from a spruce pulp that contained fibers of all seasons. The homogeneity of the wood fiber samples, from the point of view of lignin content, has previously been discussed (Ehrnrooth 1982). For one fiber sample of about 10% lignin content, the scatter in the calculated lignin content was ±3%. The fibers selected for testing were studied by microscopy. Of all fibers mounted for testing 70% were latewood fibers. Fibers that have thin walls and high fibril angle exhibit a theoretically predictable buckling, which is manifested as a yield point on loading to break in air (Page et al. 1971). In the present work, seven wood fibers were loaded to break at 50% relative humidity. Since all loading curves were linear, we assume that the presence of extreme fibril angles in the fiber population is unlikely.

The cross-sectional areas of wood fibers, evaluated by microscopy and by compacting, are shown in Table 3. Data for rayon and ramie are also included. The results show that the cross sections of wood and ramie fibers obtained by microscopy are 15 to 35% higher than those obtained by compacting, and that the coefficient of variation of cross-sectional areas of native fibers is high for both methods. The cross sections of rayon fibers show very little variation and are moreover the same for both methods. The uniform results obtained for rayon seem to suggest that the variation in the measured cross-sectional areas of native fibers is due to real structural differences between the individual fibers.

Any mechanical fiber test involving the use of adhesives should be carefully scrutinized in order to identify any participation of the adhesive in the tensile deformations investigated. The glue used in the present work was a commercial two-component adhesive, Araldite AV 129 + HV 953 B. It was reported to have a dry modulus of 8,000 MPa and to be inert to water at room temperature for 30 days (CIBA-GEIGY publication No. 31455/d). In the present study, 14 wood fibers were loaded to break in water and five wood fibers at 50% relative humidity. The loading in some cases led to slippage of the fiber from the glue. Results of this study and of earlier work with rayon and ramie fibers (Ehrnrooth, unpublished

TABLE 4. *Elastic moduli, E, of dry and wet single fibers of rayon, ramie and wood.*

Fiber species	Fiber treatment	Dry testing conditions	E_{dry} GPa	E_{H_2O} GPa	Reference
Rayon	70% stretched	23 C, 50% RH	9.6	0.4[1]	present study
Rayon	textile viscose	24 C, 50% RH	10.6	0.3	Bryant, Walter (1959)
Ramie	1–2% NaOH	23 C, 50% RH	39.2	15.1[2]	present study
Ramie	bleached	20 C, 80% RH	24.0	12	Sakurada et al. (1964)
Norway spruce	Asplund pulp	23 C, 50% RH	26.7	9.2[2]	present study
Norway spruce	62.4% delign holocellulose	23 C, 50% RH	20.8	4.3[2]	present study
Norway spruce, latewood	unbl sulphite	20 C, 65% RH	18.0	—	Duncker, Nordman (1965)
Norway spruce, latewood	unbl bisulphite		25.1	—	Leopold (1966)
Norway spruce, latewood	unbl kraft		16.1	—	Leopold (1966)
Scotch pine, latewood	unbl kraft	20 C, 65% RH	18.0	—	Duncker, Nordman (1965)
Scotch pine	TMP		12.8	—	Armstrong et al. (1977)
Douglas fir, latewood	holocellulose	22 C, 66% RH	28.4	15.5	Kersavage (1973)

[1] 100% lateral swelling in water assumed.
[2] 0% lateral swelling in water assumed.

results) show that slippage with wood fibers occurred with the strongest fibers and it occurred more often in water than at 50% relative humidity. Slippage never occurred with rayon fibers. The mean load at slippage of wood and ramie fibers was not conclusively lower in water than at 50% relative humidity. It is therefore believed that the slippage is due to poor contact in the gluing zone. The higher frequency of slippage in water would thus be due to softening of the fiber material in the gluing zone rather than to softening of the glue in water.

Creep and successive loading-unloading tests were earlier carried out (Ehrnrooth, unpublished results) with rayon and ramie fibers at 50% relative humidity and in water. The rayon fibers exhibited creep and recovery behavior that generally agreed with that reported in the literature (Leaderman 1943; Press 1943; O'Shaughnessy 1948). At 50% relative humidity, the creep deformation under small loads was totally recoverable after unloading and the recovery was speeded up by wetting of the fiber. The rigidity of 15 rayon fibers, measured at fiber lengths between 1 and 10 mm, was independent of the fiber length, and the elastic recovery was independent of the wetting time in water. The wood fibers show creep behavior similar to that shown by Hill (1967) for holocellulose fibers of a longleaf pine pulp. These results would not have been obtained if deformation of the glue had appreciably added to the fiber elongation.

The mean inverse rigidity, $\bar{k} = (E \cdot A)^{-1}$ for rayon and ramie fibers at 50% relative humidity was 0.16 and 0.05 N^{-1} respectively, and $\bar{k}$ for rayon and ramie in water was 2.16 and 0.13 N^{-1} respectively (Ehrnrooth, unpublished results). The elastic moduli of the rayon and ramie fibers, $E = \sigma/\epsilon$, were calculated using the mean values of the compacted cross-sectional areas. The calculated moduli are shown in Table 4, which also includes calculated moduli for the wood fibers at 0 and 62.4% delignification, and literature data for rayon, ramie and wood fibers. The

results show that the moduli measured for wood fibers in the present study and those reported in the literature are of the same magnitude. Moreover there is a fair agreement between the moduli of rayon and ramie fibers obtained by the testing method used in the present study and moduli of rayon and ramie fibers reported in the literature. Bryant and Walter (1959) used a textile fiber testing apparatus to which rayon fibers were attached without the use of adhesives. The testing span was 50 mm. Sakurada and co-workers (1964) worked with fiber bundles mounted without adhesives, and fiber spans of 35 mm. The fact that the results in the present study for short rayon and ramie fibers glued to steel pins agree with those obtained with different testing techniques in the absence of glues suggests that the adhesive used in the present study does not influence the elongation of the rayon and ramie fibers in air and in water. As the rigidity of ramie and wood fibers should be of the same magnitude, it can be concluded that the tensile measurements carried out with wood fibers in the actual study are not influenced by the glue.

CONCLUSIONS

The tensile testing technique outlined above provides seemingly reliable data on the rigidity of single fibers both in air and in water. The testing method has some advantages compared with conventional stress-strain testing. As the immediate recovery is used to measure elastic strain in the sample, nonelastic structural yielding is excluded from the recorded strain. Moreover the testing method permits successive testing of the same fiber in air and in water. The influence of the glue does not apparently influence the measurements as complete recovery is obtained at small loads and the measured moduli compare well with literature data.

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APPENDIX

This appendix analyzes the load-elongation characteristics of the tensile testing apparatus shown in Fig. 1. If the true fiber elongation is to be measured with the LVDT, it is crucial that the point of attachment of the lower end of the fiber is positioned vertically below the upper attachment. Any horizontal displacement will lead to inaccurate fiber elongation readings from the LVDT, because there will then also be a component from straightening of the system. It is shown that this additional elongation mainly takes place at loads lower than 4 mN.

The geometry of the problem is shown in Fig. 6, where the active forces have also been included.

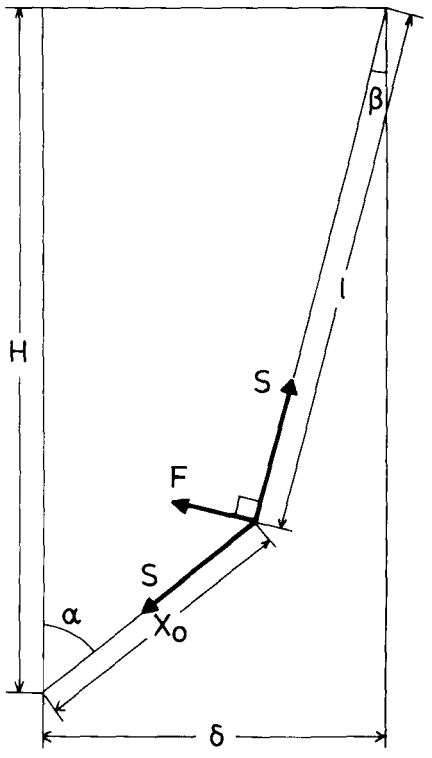

FIG. 6. Geometry of the tensile testing apparatus.

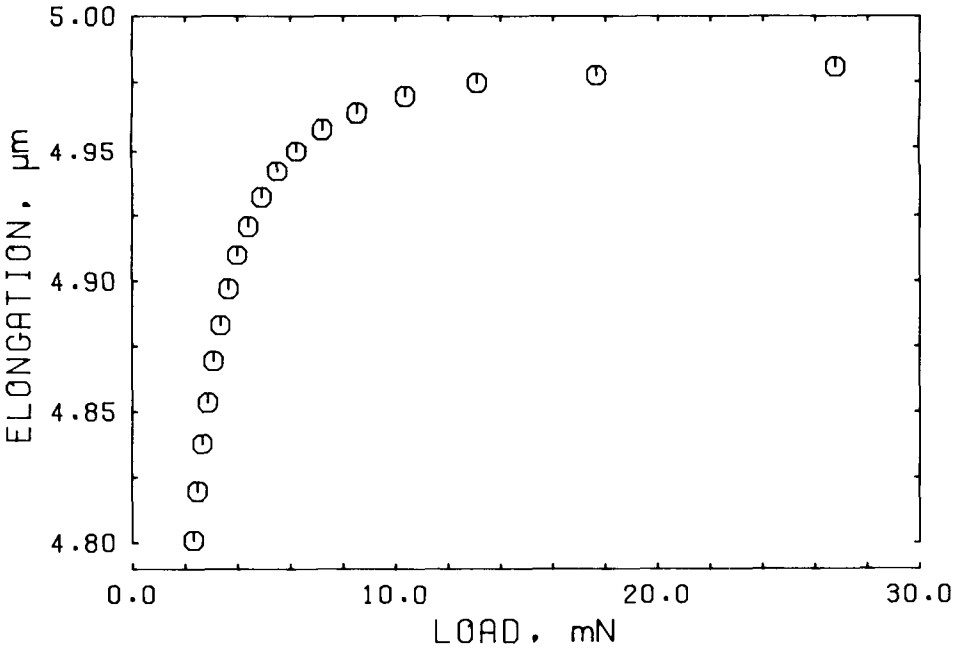

FIG. 7. Elongation due to 0.1 mm misalignment for a fiber with 1 mm free span.

The tension S, acting along the core-rod and the fiber, is essentially equal to the applied load if the misalignment is small. It can be shown that the force F acting perpendicularly to the core-rod is:

$$F = S \cdot \sin(\alpha - \beta) \tag{1A}$$

where α and β are defined in Fig. 6. The vertical distance H between the lower attachment point of the fiber and the upper attachment point of the core-rod is:

$$H = x_0 \cdot \cos \alpha + l \cdot \cos \beta \tag{2A}$$

where x_0 is the unstrained fiber length and l is the length of the core-rod. The force F is balanced by the gravitational forces acting on the core-rod according to:

$$F = (l'/l) \cdot m \cdot g \cdot \sin \beta \tag{3A}$$

where l' is the distance from the upper attachment point to the mass center of the core-rod, m is the mass of the core-rod and g is the acceleration of gravity. If Eqs. (1A) and (3A) are combined, the following relation between S and α and β is obtained:

$$S = (l'/l) \cdot m \cdot g \cdot \sin \beta / \sin(\alpha - \beta) \tag{4A}$$

Now, α and β are interrelated through x_0, l and the horizontal misalignment δ:

$$\sin \alpha = \delta / x_0 - l / x_0 \cdot \sin \beta \tag{5A}$$

where

$$0 \leq \beta \leq \sin^{-1}[\delta/(l + x_0)] \leq \alpha \leq \sin^{-1}(\delta/x_0).$$

Numerical calculations were carried out for different magnitudes of the misalignment δ. The angle β was varied between 0 and $\beta_{max} = \sin^{-1}[\delta/(l + x_0)]$, and the necessary tension S and the resulting distance H were calculated for each β. The result for a misalignment of 0.1 mm is shown in Fig. 7, where the elongation (i.e., $H(\beta) - H(0)$) is plotted versus applied load.

The graph shows that initially there is a relatively large elongation for small loads. When the load exceeds 4–5 mN, the further increase in elongation is small. It must therefore be reasonable to assume that the incremental elongation recorded above 4–5 mN is primarily due to fiber strain, if the misalignment is moderate (0.1 mm or less).

The elongation calculated from the theoretical analysis is the movement of the upper end of the core-rod. The actual elongations are, of course, measured at the core of the LVDT. This means that the full capacity of the second term on the right-hand-side of Eq. (2A) is not included in the recorded data. The contribution from the second term is, however, less than 1% of that from the first term. The discrepancy is thus negligible.