The effects of cyclic axial loading of single wood pulp fibers at elevated temperature and humidity

PETER M. WILD, JAMES W. PROVAN, RAN GUIN, AND SORIN POP

ABSTRACT

A single-fiber fatigue cell has been developed that allows single wood-pulp fibers to be cyclically loaded in axial tension at frequencies of up to 50 Hz, at temperatures of up to 100°C, and at a relative humidity of 100%. The cell is instrumented to measure fiber loading with an accuracy of ±4 mN and fiber elongation with an accuracy of ±2 µm. Parameters such as fiber stiffness and work done on the fiber can be calculated as testing proceeds. Initial tests, which have been performed on white pine TMP fibers, show that (a) the fibers exhibit strong hysteresis and stiffening behaviors during the initial few load cycles, (b) over long-term fatigue testing, fiber stiffness increases linearly as the logarithm of the number of loading cycles, and (c) under fatigue loading, the fibers exhibit a creep behavior that is similar to that of fibers under static loading.

Application:

The single-fiber fatigue cell is a unique platform for research into the mechanical behavior of wood-pulp fibers under the environmental conditions that exist in refining and papermaking.

IN MECHANICAL PULPING, FIBERS ARE extracted from wood chips due to the accumulated damage caused by the repetitive application of mechanical forces, or "fatigue." Through this process of extraction and as a result of continued mechanical action during the fibers' remaining residence time in the refiner, fiber properties are changed or developed. Developed fiber properties include axial stiffness, bending flexibility, transverse compressibility, and fibrillation.

The nature (direction, magnitude, and frequency) of the loading that individual fibers receive in a refiner is not well understood. The relationships between these different types of loading and the development of fiber properties (axial stiffness, bending stiffness, transverse compressibility, and fibrillation) are also not well understood. The long-term objective of this research program is to establish an understanding of these relationships by subjecting single fibers to modes of loading that are thought to occur in a refiner while monitoring the resulting changes in fiber properties.

LITERATURE REVIEW

There is a significant body of literature dealing with the behavior of single wood-pulp fibers under mechanical loading. Of particular note are the collected publications of Page *et al. (1-6)* in which a consistent portrait of the structure of a wood-pulp fiber is developed. According to Page, the principal structural component of a fiber, the S2 layer, is comprised of helically wound cellulose fibrils embedded in an amorphous matrix of lignin.

Previous authors had developed an analytical model of the relationship between fiber modulus and fibril based on the assumption that "the S2 layer is an orthotropically elastic material with one of the principal directions of symmetry along the fibrils" *(5)*. The S2 layer is, thus, similar to a filament-wound composite tube that has been wound in only one direction. Page conducted experiments that showed that the modulus of a fiber of a given fibril angle has an upper bound controlled by the fibril angle. The relationship between this upper bound and fibril angle was shown to be consistent with relations derived from orthotropic elastic theory. Fibers exhibiting moduli below this upper bound were found to "contain crimps, nodes, microcompressions or other irregularities of structure that would tend to lower the modulus" *(5)*.

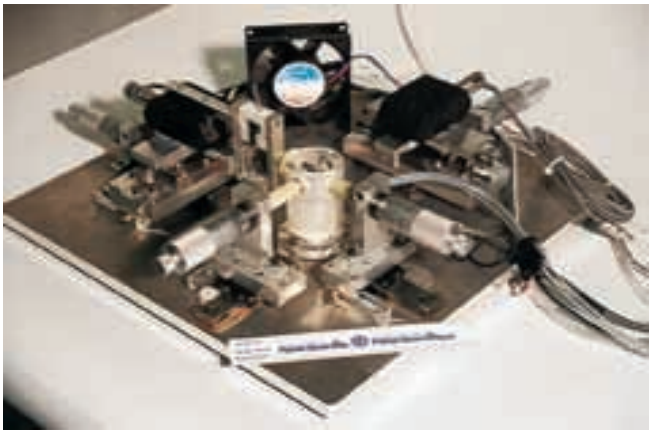
In related study, Page found that the strengthening of fibers (in particular fibers of high hemicellulose content) that are dried under tension is due to the shear flow between adjacent fibrils *(3)*. This shear flow permits irregularities of structure to be "pulled out," resulting in an initial increase in strength. In addition, this shear flow also permits a decrease in fibril angle, resulting in a further increase in strength.

Although Page's work is certainly comprehensive, it does not specifically address the effects of multiple, controlled-load applications to a single fiber. In fact, only a few studies deal with the response of single fibers to multiple load cycles and none that deals with multiple loading modes under the environmental conditions that occur in a refiner.

Kallmes and Perez *(7)* and Jentzen *(8)* conducted studies in which fibers were subjected to cyclic axial loading and observed a "strain hardening" effect, which was characterized by a tensile modulus that increased with each cycle. These tests were conducted at room temperature and humidity, and the numbers of cycles were restricted to only



1. A fiber held in the fork grips



2. Single-fiber fatigue cell

six and three, respectively. Tam Doo and Kerekes *(9)* applied cyclic bending forces to fibers in the form of a fluid flowing normal to the fiber axis. It was found that bending flexibility increased with the number of bending cycles. Naito, Usuda, and Kadoya *(10)* applied cyclic torsional loads to a fiber using a torsional pendulum. Torsional compliance, which was inferred from changes in the period of the pendulum, was found to increase as cyclic loading proceeded.

Similar results have been obtained in studies that have investigated fatigue by comparing the properties of fibers that have been subjected to differing levels of refining. Samuelson *(11)* and Tam Doo and Kerekes *(9)* found that bending flexibility increases with beating time, while Hardaker (12) found that tensile modulus increases with beating time.

The only study that has addressed multiple loading modes was qualitative in nature. Provan and Hamad *(13)* performed an assessment of cyclic loading based on morphological changes observed with a confocal laser scanning microscope. This work suggested that cyclic shear loading was a particularly important mechanism in fiber fibrillation.

Because fatigue loading can be of relatively long duration, studies of fiber creep are also relevant to this work. The most comprehensive treatment of pulp fiber creep under constant tensile loading is covered by Hill *(14).* Hill identified a characteristic pattern of creep in which the limiting behavior, termed *logarithmic creep,* appears as a straight line on a plot of elongation vs. the logarithm of time. This is preceded by a period of initial creep, which also appears as a straight line but has a smaller slope. The time required to reach the transition from initial to logarithmic creep decreases as load increases. Ehrnrooth and Kolseth *(15)* found a similar transition in creep behavior, particularly for those fibers tested at elevated humidity. No studies that address the issue of creep in the context of cyclic loading have been found.

Many different methods for the tensile testing of individual wood-pulp fibers appear in the literature. Key issues that these methods address include gripping the fiber, applying a load to the fiber, measuring the load, and measuring the elongation. For a comprehensive review of these methods, refer to Mark and Gillis *(16),* Kersavage *(17),* and Mott, Shaler, and Groom *(18).* However, none of these methods is capable of high-frequency quantitative cyclic loading of wood-pulp fibers at elevated temperature and humidity. The device reported here is the first to allow a quantitative investigation of the effects of cyclic loading, although only axial, at elevated temperatures and humidities such as those that occur in a refiner.

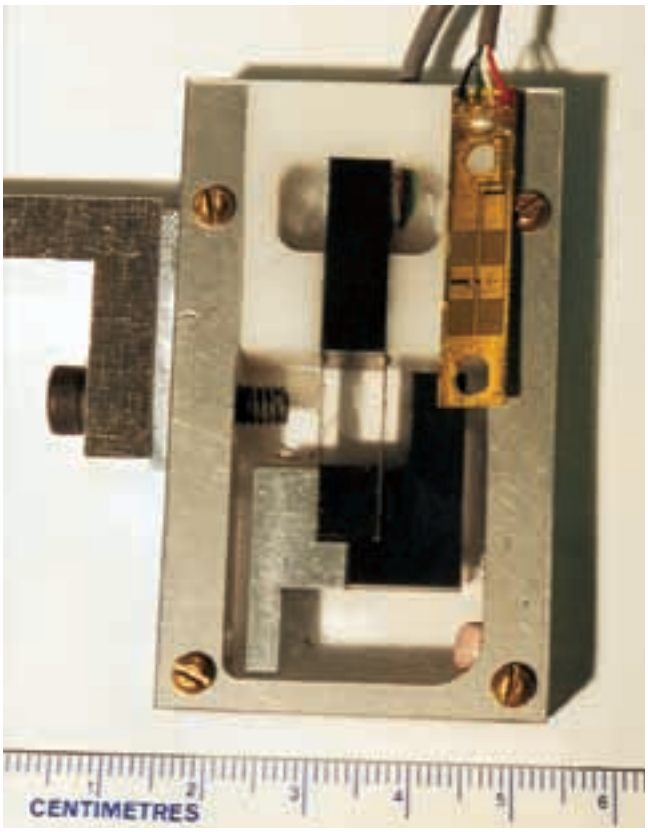
THE SINGLE-FIBER FATIGUE CELL

A single-fiber fatigue cell has been developed in which quantitative cyclic axial loading of single wood-pulp fibers can be conducted at frequencies up to 50 Hz, at temperatures up to 100°C, and at a relative humidity of 100%. The cell includes a piezoelectric actuator that tensions the fiber and sensors that measure fiber elongation and loading. The method by which the fiber ends are gripped is based on the epoxy droplet method developed by Kersavage *(15)* and refined by Mott *et al. (16).* Actuator control and data acquisition are provided by a PC-based interface using National Instruments' LabView™ software.

The fiber-gripping method developed by Kersavage and Mott involves placing an approximately spherical droplet of epoxy at each end of the fiber and then locating the fiber between two small forks, as shown in **Fig. 1.** When the forks are

drawn apart, the epoxy spheres locate against the forks, creating a self-aligning ball-and-socket type of grip. The principal benefits of this method are (a) relative ease of loading the fiber into the grips and (b) self alignment of the fiber, minimizing stress concentrations at the point of grip.

The fiber is held between the fork grips, which are located in an environmental chamber, as shown in **Fig. 2.** One end of the fork grips is fixed to a Burleigh PZS-200 piezoelectric actuator, which provides up to 200 µm of displacement at frequencies of up to 1 kHz. The maximum operating frequency is, however, determined by the natural frequency of the load cell, which is approximately 55 Hz. The opposing end of the fork grip is fixed to a micrometer head, which is used for coarse adjustment of displacement. The fiber load cell, shown in detail in **Fig. 3,** consists of a strain-gauged dual cantilever, which provides milli-Newton resolution. The load cell also accommodates a Kaman KD2300 displacement transducer, which measures fiber elongation with micron resolution. Also shown in Fig. 2 are the fixtures for cyclic loading of the fiber in the transverse direction. This capability is not yet fully developed.



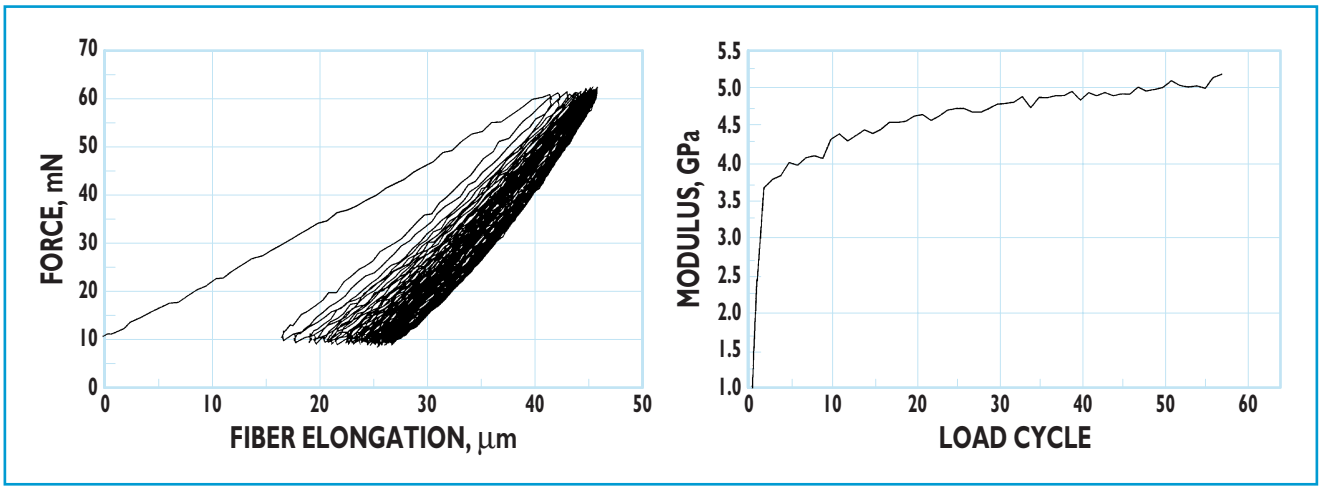
3. Load cell

The environmental chamber contains a small reservoir of water in which a resistive heater is immersed. The chamber is sealed to the stationary fork grip and has a clearance hole to allow free movement of the fork grip, which is fixed to the actuator. As steam is able to escape past this fork grip, the water in the reservoir must occasionally be replenished.



4. LabView™ VI

PC-based interfaces, or virtual instruments (VIs), developed on the LabView software, control the actuator and perform data acquisition, as illustrated in **Fig. 4.** To control the piezo-actuator, a VI generates a waveform of the desired frequency, amplitude, and offset, which is then amplified by the piezo power supply. VIs that allow cyclic loading under either displacement or force control have been developed. Due to the creep behavior of the fibers, most testing is conducted under force rather than displacement control. Signals from the force and displacement transducers are amplified, low-pass filtered, and then acquired by the VI. The sampling frequency for data acquisition is 1 kHz. Once the data are acquired, they can be processed, displayed, and/or downloaded to a spreadsheet. Two parameters that the VI calculates and displays, as a fatigue test proceeds, are fiber stiffness and work done on the fiber. The strength of the



5. (left) Typical force–displacement plot; (right) Fiber modulus vs. load cycle

LabView interface is its flexibility. New or modified VIs can be developed as testing requirements evolve.

EXPERIMENTAL PROCEDURES

Fiber preparation

All of the experiments reported here were conducted with white pine thermomechanical pulp (TMP) fibers prepared in a Sunds Defibrator 12-in. laboratory refiner and fractionated to R12 in a Bauer MacNett fractionator. To separate individual fibers, a small amount of never-dried pulp was grasped with tweezers and added to 100 mL of water. This was mixed well to separate the fibers into suspension. The suspension was then poured into a filter containing a fine-filter membrane and was allowed to drain. When the water had drained, the filter membrane was removed from the filter and inverted onto the back of a petri dish. When the membrane was removed from the petri dish, the fibers were left behind on the petri dish and allowed to dry.

The dried fibers are examined under the stereo microscope to select only fibers that are straight, relatively unkinked, and long. The selected fibers are spanned between two microscope slides and held in place using double-sided tape. Droplets of epoxy are applied to either end of the fiber by dipping tweezers into the epoxy and spreading the tips of the tweezers over the fiber. The epoxy spans the tips of the tweezers as they open, and this thread of epoxy is lowered over the fiber. The epoxy is deposited onto the fiber and quickly forms a ball. This is repeated until the ball is the correct size (~300 μm). When hardened, the epoxy droplets are approximately spherical. Each slide will fit ~30 fibers. When all of the fibers have epoxy droplets, they are cured as required. This is the mounting method described by Mott *(16).*

Cyclic load range, mN	SPECIFIC REFINING ENERGY 6.7 MJ/kg	10.5 MJ/kg
10–40	Test condition #1	Test condition #4
10–60	Test condition #2	Test condition #5
10–80	Test condition #3	Test condition #6

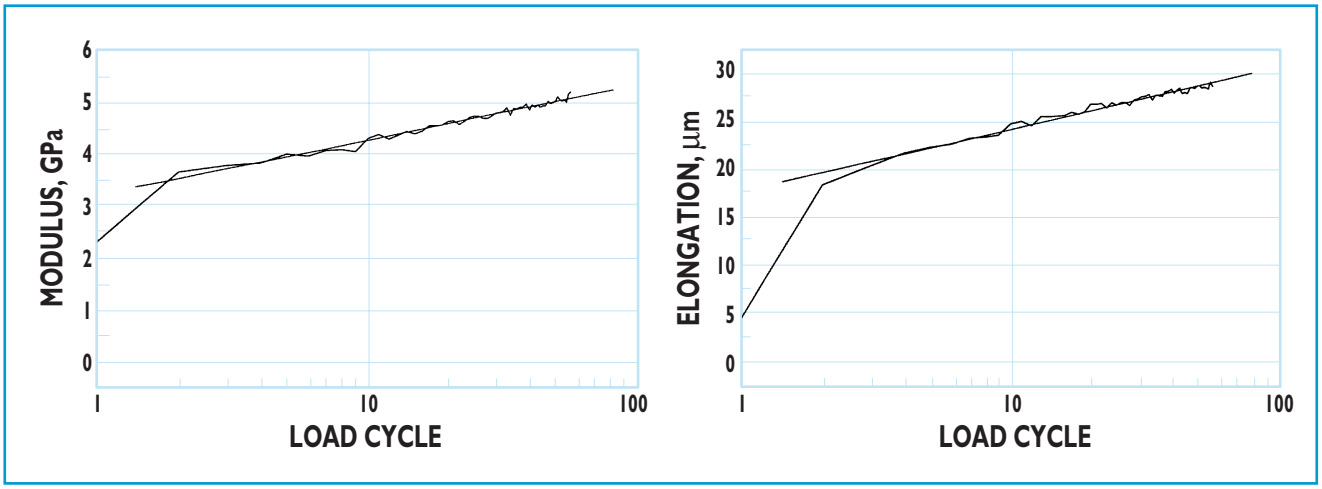
I. Fiber test conditions

Epoxy selection

In Mott's work, which was conducted at relatively low temperatures and humidities, Devcon 2-ton epoxy was found to be the most suitable adhesive. This epoxy is not, however, suitable for this work as it softens and loses its grip on the fiber at elevated temperatures and humidities. Identification of an adhesive that did not soften under these conditions, had an appropriate viscosity, and had an acceptable cure procedure proved to be a difficult task *(19).* Magnabond 6371 was found to provide adequate performance and was used throughout the experiments that are described here. This is a clear two-part epoxy, which is designed for injection repair of composite structures.

Fiber test methods

Once the epoxy droplets have been mounted on the fiber and the epoxy has been cured (3–4 hours at 65°C), the fiber can be placed on the fork grips. This is done using tweezers to grip the fiber by one of the ends that projects from the droplets. For tests at high temperature and humidity, the environmental chamber is covered and the heating element raises the water temperature to the boiling point. As is discussed later in this paper, the fibers exhibit a considerable amount of unrecoverable axial extension, particularly in the first



6. (left) Fiber modulus vs. log of load cycle; (right) Fiber elongation vs. log of load cycle

few load cycles. As a result, cyclic load tests are conducted under force rather than displacement control. In all of these tests, a preload is applied to the fiber by manually adjusting the micrometer heads while visually monitoring the axial load on the fiber. Once the preload is established, automatic cyclic loading is initiated, and the force–displacement data are automatically logged as testing proceeds.

Confocal laser scanning microscopy

After being tested, all fibers were examined using the confocal laser scanning microscope (CLSM) at the Vancouver Laboratory of the Pulp and Paper Research Institute of Canada. Prior to testing, the fibers were dyed with a 0.002% solution of Acridine Orange, a fluorescent dye that improves the quality of CLSM images. The primary objective of these examinations was to determine the cross-sectional area of the fibers and thus determine fiber moduli. Three cross-sectional images were taken along the length of each fiber. The areas determined from these three images were averaged to reduce the effect of variation in cross-sectional area along the length of the fiber.

INITIAL EXPERIMENTAL PROGRAM: CYCLIC AXIAL LOADING OF WHITE PINE TMP FIBERS

Cyclic axial loads of three different magnitudes were applied to white pine TMP fibers of two different refining energies, resulting in six different test conditions, as summarized in **Table I.** Ten fibers were subjected to each of these six test conditions. The number of load cycles in each test was 60. All tests were conducted at 100°C and 100% RH.

In all tests, the fibers showed successive increases in axial modulus with each load cycle. A typical force displacement plot, in this case for test condition no. 6, is shown in **Fig. 5(a).** As shown in **Fig. 5(b),** the largest increase in modulus occurs during the first load cycle. Beyond the first cycle, the modulus of the fiber continues to increase with successive load cycles but by diminishing amounts. These data show a good linear fit on a semi-log plot, as shown in **Fig. 6(a).** Corresponding with these diminishing increments of modulus are diminishing increments of elongation, which, as shown in **Fig. 6(b),** also show a good linear fit on a semi-log plot.

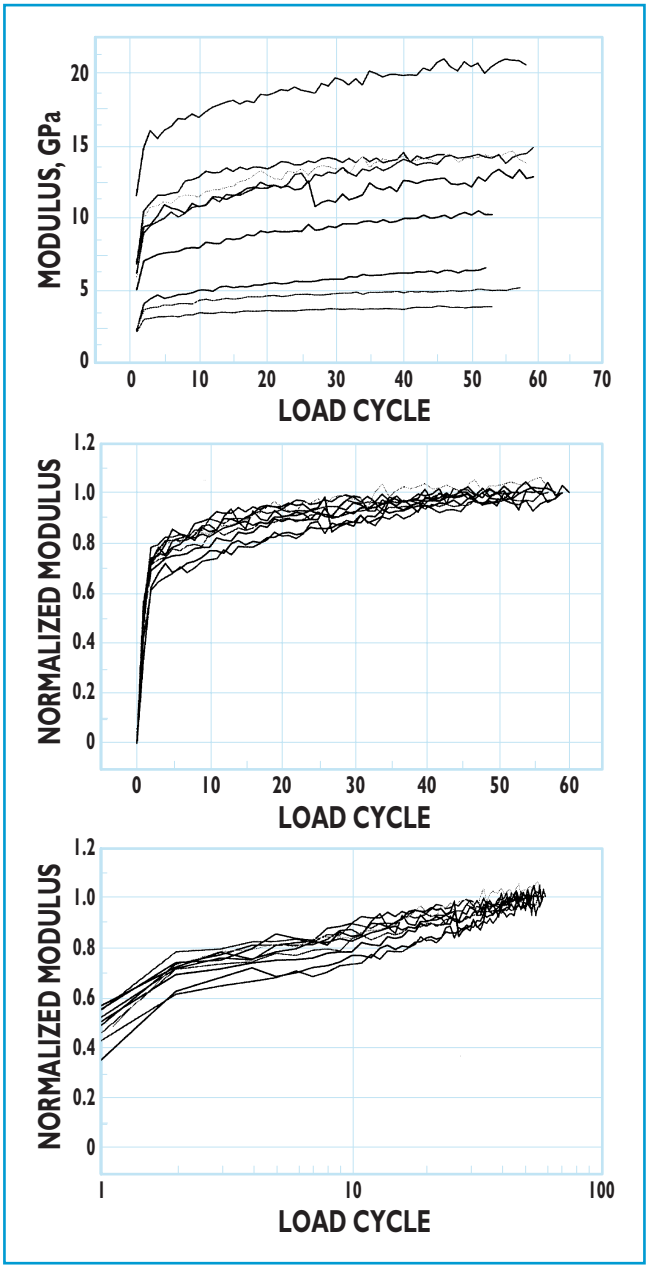
In absolute terms, the data from the different test conditions showed great variation among the fibers tested under each condition. The modulus-vs.-load-cycle curves for test condition no. 2, shown in **Fig. 7 (a),** are typical of all test conditions. The data were then normalized, with respect to the modulus on the final load cycle and, as shown in **Figs. 7 (b) and 7(c),** collapse relatively well into a common trend. However, comparison of either normalized or absolute data from different test conditions reveals no consistent trends.

DISCUSSION

The findings of this study are as follows:

- The fibers exhibit strong hysteresis and stiffening behaviors during the initial few load cycles.
- Over long-term fatigue testing, fiber stiffness increases linearly with the logarithm of the number of loading cycles.
- Under fatigue loading, the fibers exhibit a cyclic creep behavior that is similar to the creep behavior of fibers under static loading.

The stiffening behavior during the initial few load cycles is similar to the results obtained by Kallmes and Perez *(7)* and Jentzen *(8)* for fibers in the dry state. This observation is believed to be due to the "pulling out" of crimps and kinks in the fibers, which have been introduced by the mechanical action of refining. Page *(20)* observes that "crimps and kinks are incapable of transmitting load," and these features, therefore, effectively reduce the modulus of a fiber. However, by repeatedly loading the fiber in axial tension, these features are "pulled out," and the observed increase in tensile modulus is achieved.



7. (top) Modulus vs. load cycle for test condition no. 2; (middle) Normalized modulus vs. load cycle for test condition no. 2; (bottom) Normalized modulus vs. load cycle for test condition no. 2

After these initial few load cycles, the rate of fiber stiffening is diminished but is not zero. These trends in elongation resemble static creep data for wet or dry fibers reported by Hill *(14)* and Ehrnrooth and Kolseth *(15)*. The mechanism for this long-term stiffening is believed to be due to structural changes within the fiber rather than simply the realignment of the fiber mass, as occurs when kinks and crimps are pulled out. Under the influence of repeated cyclic loading or fatigue, the bonds between microfibrils and the surrounding matrix in the S2 layer are believed to become weakened, allowing shear flow between adjacent microfibrils. This leads to a reduction in the fibril angle and the observed increase in tensile modulus.

The objective of the six different test conditions was to ascertain if the amount of energy expended during the pulping process had a noticeable influence on the mechanical properties of single fibers. However, due to the scatter in the data under each test condition, no definitive trend can be stated at this time.

During the course of conducting these tests and the exhaustive evaluation of their results, we have surmised that the large variation observed in the mechanical responses of individual fibers may result from variations in the fibril angle of each specimen, as reported by Page *et al. (6)*. Although the 60 fibers themselves were viewed in the CLSM to determine their cross-sectional area characteristics, no determination of fibril angle was made, and therefore, at this stage, no conclusive information as to the influence of the fibril angle on the reported test results can be issued. In the near future, it is hoped that the fibril angle of each fiber will be measurable. With this additional information, the mechanical responses of fibers could be regrouped so as to exhibit the differences between the high-energy– and low-energy–produced fibers. A future objective of the single-fiber fatigue testing program is to shed light on this hypothesis. **TJ**

Wild is an assistant professor at Queen's University, Department of Mechanical Engineering, McLaughlin Hall, Kingston, ON K7L 3N6, Canada. Provan is a professor, Guin is a research engineer, and Pop is a research engineer at the University of Victoria, Department of Mechanical Engineering, P.O. Box 3055, MS 8895, Victoria, BC V8W 3P6, Canada.

The authors wish to acknowledge the National Centres of Excellence for Mechanical and Chemimechanical Wood-Pulps for their support of this research.

Received for review Nov. 28, 1997.
Accepted May 9, 1998.

LITERATURE CITED

1. Page, D. H., El-Hosseiny, F., Winkler, K., *et al., Pulp Paper Mag. Canada* 73(8): 72(1972).
2. El-Hosseiny, F. and Page, D. H., *Fiber Sci. and Tech.* 8: 21(1975).
3. Kim, C. Y., Page, D. H., El-Hosseiny, F., *et al., Appl. Poly. Sci.* 19: 1549(1975).
4. Page, D. H. and El-Hosseiny, F., *Svensk Papperstid.* 14: 471(1976).
5. Page, D. H., El-Hosseiny, F., Winkler, K., *et al., Tappi* 60(4): 114(1977).
6. Page, D. H. and El-Hosseiny, F., *J. Pulp Paper Sci.* 9(4): TR99(1983).
7. Kallmes, O. J. and Perez, M., *Proceedings of the Technical Section British Paper Board Makers' Association,* Vol. 1, British Paper Board Makers' Association, London, 1966, p. 507.
8. Jentzen, C. A., *Tappi* 47(7): 412(1964).
9. Tam Doo, P. A. and Kerekes, R. J., *J. Pulp Paper Sci.* 15(1): 36(1989).
10. Naito, T., Usuda, M., and Kadoya, T., *Proceedings of the 1983 International Paper Physics Conference,* TAPPI PRESS, Atlanta, p. 197.
11. Samuelsson, L. G., *Svensk Papperstid.* (67)23: 943(1964).
12. Hardaker, K. W., *Tappi* (Special Technical Association Publication) 8: 201(1970).
13. Provan, J. W. and Hamad W., *Cellulose* 2: 153(1995).
14. Hill, R. L., *Tappi* 50(8): 432(1967).
15. Ehrnrooth, E. M. L. and Kolseth, P., *Wood Fiber Sci.* 16(4): 549(1984).
16. Mark, R. E. and Gillis, P. P. in *Handbook of Physical and Mechanical Testing of Paper and Paperboard* (R. E. Mark, Ed.), Vol. 1, Dekker, New York, 1983, pp. 409–495.
17. Kersavage, P. C., USDA Bulletin 790, USDA, Beltsville, MD, 1973.
18. Mott, L., Shaler, S. M., Groom, L. H., *et al., Tappi J.* 78(5): 143(1995).
19. Rees, M., Internal report, University of Victoria, Department of Mechanical Engineering, Victoria, BC, September 1997.
20. Page, D. H., *Svensk Papperstid.* 23: 30(1985).