

Measurement of wet fiber flexibility of mechanical pulp fibers by confocal laser scanning microscopy

DONGBO YAN, KECHENG LI, AND YAJUN ZHOU

ABSTRACT: We measured the fiber flexibility of mechanical pulp with confocal laser scanning microscopy (CLSM) based on Steadman's method. Pulp fibers were wet pressed and bent over a glass wire and CLSM was used to image the three-dimensional deformation of the fiber. A fiber neutral bending plane was defined for freespan length measurement, which was subtracted from a transverse optical section of a fiber. We measured the moment of inertia of the fiber from the cross-sectional geometry and calculated the elastic modulus of the fiber wall from the fiber flexibility and the moment of inertia. Results show that the flexibility of chemithermomechanical pulp (CTMP) fibers correlated well to interfiber bonding strength and sheet structural properties. A bleaching or refining process significantly increased the fiber flexibility, mainly through altering the elastic modulus of the fiber wall.

Application: The wet fiber flexibility of mechanical pulp fibers such as BCTMP can be measured more accurately.

Fiber flexibility is crucial in determining fiber-fiber interactions and interfiber bonding, because it correlates strongly to most of paper's structural properties such as bulk, opacity, and paper physical strengths. The fiber flexibility of mechanical pulp, in particular bleached chemithermomechanical pulp (BCTMP) fibers, is more important when the BCTMP is used in wood-free fine paper grades as a substitute for kraft pulp. The use of BCTMP reduces the production costs and improves the bulk of the paper; however, excessive use of BCTMP may lower interfiber bonding and surface roughening may occur in the coating or the offset printing process [1, 2].

Past research has proposed several methods for fiber flexibility measurement, either for a small amount of pulp or individual fibers [3-10]. The flexibility of individual fibers is usually measured according to small deflection beam theory by measuring the magnitude of fiber deflection when subjected to a transverse bending moment. The measurement can be obtained by either hydro-dynamic method [6-8] or mechanical pressing method [9, 10]. Of these methods, the conformability testing method appears to be the most straightforward. Mohlin [9] proposed the method and it was further developed by Steadman and Luner [10]. The method has several advantages: it can provide a numerical measure of flexibility in an engineering unit, it does not require handling individual fibers, and the microscopic observation used is convenient to conduct. Therefore, the conformability method has been widely used in evaluating fiber flexibility, and it provides results that correlate well with the paper properties for chemical pulp fibers [11, 12].

In the Steadman method, a 25- μm -diam. steel wire mount-

ed on a glass slide is used as a support; fibers are wet pressed on the wire and arced over it. Noncontact length, i.e., the free span that appears bright under incident light, is measured using a conventional light microscope. The fiber is treated as an elastic beam and the flexibility is then expressed as the reciprocal of bending stiffness (Eq. 1)

$$\text{Flexibility} = \frac{1}{EI} = \frac{72d}{qL^4} \quad (1)$$

where E is the elastic modulus of the fiber wall, I is the moment of inertia of the fiber cross-section, d is the fiber deflect height, and L is the freespan length of the fiber bending over a support wire [10].

Using this method, we assumed that because the fiber deflection ratio is small, the small deflection beam theory is applicable. The fiber was also assumed to be fully collapsed, thus the thickness of the fiber can be neglected. Lawryshyn and Kuhn [13] noticed that using the small-deflection theory for flexibility analysis may introduce an error of 10% at a 20% deflection ratio. In the case of a typical chemical pulp fiber, the actual deflection ratios may be as large as 40% when supported by a 25- μm -diam. wire, so the error is expected to be larger.

Lowe et al. [14] proposed using real pulp fibers instead of support wires to simulate the situation relevant to the fiber deformation in actual paper structure by reducing the deflection ratio. Since the diameter or thicknesses of the support fiber were unknown, sophisticated microscopy techniques were needed for measuring it.

BCTMP fibers are rich in lignin so the wall structure is rigid compared with chemical pulp fibers, which usually col-

lapse completely during pressing. Mechanical pulp fibers only collapse partially with variable thickness that may vary from a few microns to 30 μm [15, 16]. For mechanical pulp, fiber thickness was comparable to the diameter of support wire. For accurate measurement of fiber flexibility, the effect of fiber wall thickness should be taken into account in determining the fiber deflection.

Confocal laser scanning microscopy (CLSM) is a technique originally developed for rapid and nondestructive three-dimensional imaging of living cells. In recent years, it has been used increasingly in pulp and paper research for imaging fiber cross-sections and evaluating fiber collapse behavior [15, 17], paper cross-section analysis [18], and chemical distribution across the fiber wall [19], as examples. The optical sectioning function makes CSLM ideal for imaging fiber deformation and cross-section geometry with higher accuracy. In our previous study [20], we developed a new method of fiber flexibility measurement by CLSM. With this method, we also measured the transverse profile of fibers to calculate the contribution of shear deformation to overall flexibility [21].

In this paper, we discuss the measurement of the flexibility of BCTMP fibers with CLSM. Glass fibers of 10- μm diameter instead of 25- μm diameter used in the original Steadman and Luner method were used as support wires to reduce the fiber deflection ratio, thereby eliminating the error associated with analysis by small deflection theory. Because all geometric parameters were measured with the image analysis software from binary CLSM images, subjective errors were avoided.

MATERIALS AND METHODS

Materials

We obtained two commercial pulps, aspen CTMP and BCTMP, from the same production line of a Canadian paper mill. The BCTMP was further refined by a PFI mill at 4% consistency to 3000 revolutions and at 10% consistency to 4000 revolutions denoted as LCR and MCR, respectively. The Canadian Standard Freeness (CSF) of the pulps was tested in accordance with the TAPPI standard (T227 om-94). The CSF of LCR and MCR are 236 mL and 268 mL, respectively. Bleached jack pine kraft pulp (BKP) was obtained from a Canadian mill and was used for comparison. The BKP was refined at 6000 revolutions to a CSF freeness of 500 mL and denoted as BKP-6K.

Sample preparation

Acetone washed glass fibers were deposited on glass slides (Fisher brand precleaned microscope slide) as support wires prior to depositing pulp fibers on them. Glass fibers (0.5 g, CDS Analytical 1001-0345) were suspended in 1 L of distilled water and drained onto a piece of filter paper (Fisher brand Q8) by a TAPPI standard handsheet former. The suspension was swirled before draining to orient glass fibers in parallel on the filter paper. The glass fibers were transferred onto microscope slides by placing and gently tapping the filter paper onto the slides.

To enhance the fluorescence intensity, pulp fibers (0.3g

o.d.) were stained in 20 mL 0.1 % Safaranin-O for 24 h at room temperature and then diluted to 0.03% consistency and drained onto a filter paper as was done for the glass fibers. The filter paper with fibers was placed on two pieces of dry blotting paper and then pressed onto eight glass slides by a standard handsheet press (Labtech) for 5 min. Before pressing, the glass slide and the filter paper were arranged so that the pulp fibers and the glass fibers crossed each other perpendicularly. Slides were air dried and kept under TAPPI standard conditions before CLSM imaging.

CLSM imaging

Image scanning was carried out with a Leica TCS-SP2 confocal laser scanning microscope using a dry objective lens (HC FLOUTAR 50 $\times$) with a numerical aperture of 0.8. Excitation wavelength of 514 nm from an Ar laser was used. The pinhole size was set at the optimum value by Leica Confocal Control software. The emission light collected by detector (PMT) was set from 525 to 760 nm. The gain and offset of PMT were automatically adjusted for each fiber by software to ensure a constant image contrast and brightness. The CLSM was operated in XZ or YZ mode to obtain a transverse or cross-sectional image. Scanning step size in Z direction is 0.12 μm . For each pulp sample, at least 40 fibers were measured.

Image processing

Image processing was performed with the image processing toolbox in Matlab 7.0 (Mathworks Inc.). CLSM images were smoothed using low-pass filtering and then converted into binary format. The threshold for binarization was determined automatically by the double peak histogram method [24]. All measurements were carried out on binarized images.

Fiber cross-section geometry

The fiber aspect ratio (AR) was used for fiber collapsibility evaluation (Eq. 2) according to Jang et al. [15].

$$AR = \frac{D_{\min}}{D_{\max}} \quad (2)$$

where $D_{\min}$ is the fiber thickness (shortest Feret diameter) and $D_{\max}$ is the fiber width (longest Feret diameter), which were obtained from a binarized fiber cross-sectional image.

Because of the irregular shape of the fiber cross-section, the moment of inertia (I) of fiber in relation to the neutral bending plane was calculated based on the location of each pixel (Eq. 3) [26].

$$I = \sum I_i = \sum \left(\frac{ab^3}{12} + A_i x^2 \right) \quad (3)$$

where a and b is the width and height of the pixel. A_i is the area of a pixel and x is the distance of pixel i to the neutral bending plane.

Fiber and handsheet characterization

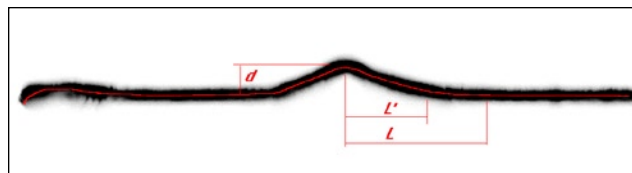
We made the handsheets and tested the physical properties, including bulk, light-scattering coefficient, Scott bond strength, and breaking length, were carried out in accordance with TAPPI standards. The flexibility of wet fiber was calculated according to Eq. (1). The elastic modulus of individual fiber was calculated from the flexibility and the moment of inertia of each fiber according to [20].

RESULTS AND DISCUSSION

Freespan length measured by CLSM

Figure 1 shows how the freespan length can be measured by CLSM. With optical sectioning function, fiber transverse view can be obtained directly and thus the freespan length can be measured easily. If a light microscope was used, the freespan length identified would be “ L' ” as shown on the image that is the distance between the center of the support wire and the points of optical contact between the fiber and the glass slide surface. However, for mechanical pulp fibers, the cell wall is thick and rigid and the thickness of the fiber wall cannot be neglected when determining the freespan length. Previously, we proposed using the neutral bending plane to identify the freespan length [20]. The fiber neutral bending plane was approximated as the geometrical middle plane in the fiber cross-section. The neutral bending plane was subtracted by applying a skeleton operation to binarized images [25]. **Table I** shows that the difference in freespan length measured by CLSM and LM ranges from about 5%-25% for different pulp fibers. As shown in the Fig. 1, if the neutral plane is used, then the freespan length will be “ L ”, which is much larger than “ L' ”.

The difference increases significantly with fiber thickness as listed in Table I. According to Steadman's analysis (Eq. 1), fiber flexibility is inversely proportional to the 4th power of the freespan length (L) and proportional to support height (d);



1. Difference in freespan length measured by conventional light microscope and CLSM. L' – measured by conventional light microscope at contact surface; L – by CLSM at neutral bending plane. Aspen BCTMP, 50X lens, image size 300×65μm.

thus, error in freespan measurement will be multiplied exponentially in the calculated flexibility results [13]. Fiber deflection height, d , was assumed to be a constant that equals the diameter of the supporting steel wire in the Steadman method. We measured it directly from the transverse CLSM images.

Flexibility of various CTMP fibers

With CLSM, the true freespan length can be measured at the neutral bending plane, which will improve the accuracy of the flexibility measurement. We used four types of aspen CTMP pulps in our investigation to compare the fiber flexibility change with different processing (**Table II**). The histograms in **Fig. 2** show a wide distribution of flexibility values, so we present median values for flexibility comparison.

The flexibility of aspen CTMP fiber is $0.74 \times 10^{11} \text{ N}^1\text{m}^{-2}$, which is the same for mechanical pulp fibers measured by the hydrodynamic method [7]. However, the flexibilities of BCTMP fibers and the PFI-refined BCTMP fibers (MCR and LCR) are much higher than the CTMP fiber. The higher flexibility is probably due in part to the pressing process used, which compressed fiber in the thickness direction and decreased the moment of inertia, I , particularly for fibers having lower elastic modulus.

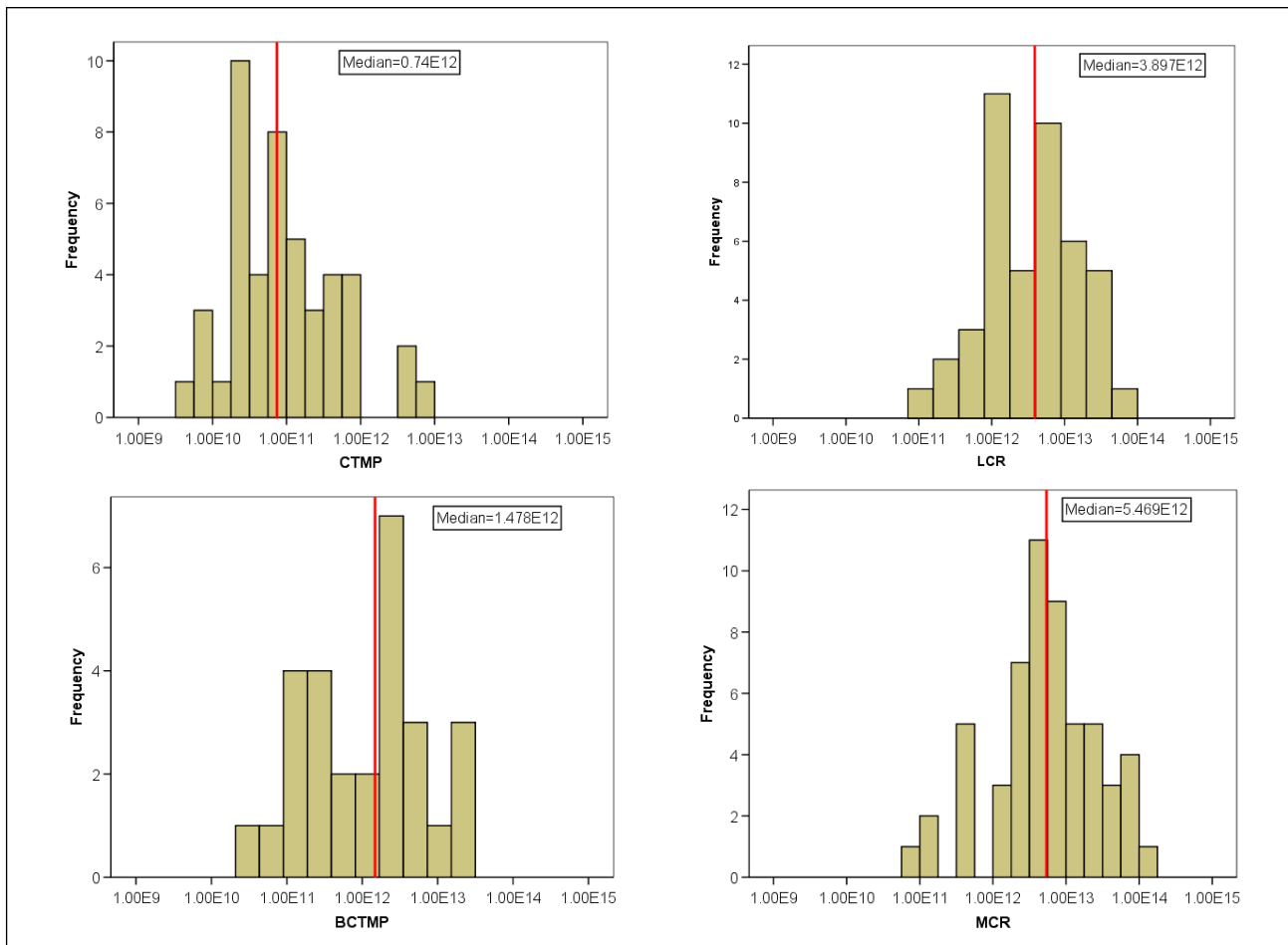
Collapse analysis based on the cross-section CLSM images also revealed the difference in the collapse behavior of

	Mean fiber thickness (μm)	Mean freespan length by CLSM (μm±SD)	Mean freespan length by LM (μm±SD)	Difference in freespan length (%)
CTMP	9.02	103.54±15.32	82.07±14.25	26.16
BCTMP	7.08	65.29±24.10	49.24±22.86	32.60
BKP	4.64	32.48±7.37	28.64±6.21	13.41
BKP-6K	2.69	18.31±2.78	17.48±3.45	4.75

I. Comparison of measured freespan length by CLSM and conventional light microscope (LM). SD: Standard deviation; 15 measurements for each fiber along the fiber width direction, using CLSM transverse slicing mode.

Pulp	Fiber aspect ratio	Fiber moment of inertia, I , m^4 , $\times 10^{21}$	Elastic modulus E , GPa	Fiber flexibility N^1m^{-2} , $\times 10^{-11}$
CTMP	3.06	1.20	17.17	0.74
BCTMP	3.76	0.85	2.27	14.78
LCR	3.85	0.75	0.56	38.97
MCR	3.53	0.81	0.22	54.69

II. Median values of the properties of aspen mechanical pulp fibers.

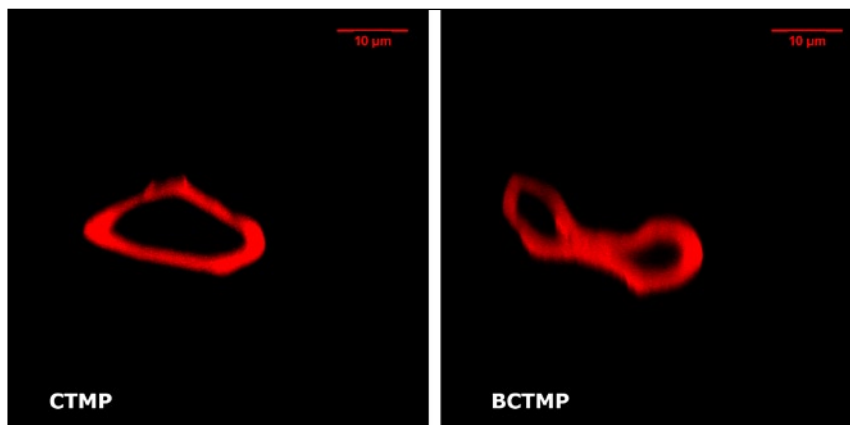


2. Distribution of measured flexibility values of aspen mechanical pulp fibers.

different pulp fibers. The bleaching process removed certain chemical components in the fiber wall and modified the lignin structure, rendering it more hydrophilic. Mechanical refining delaminates the fiber wall, which alters the elastic modulus of the fiber wall and makes it liable to collapse. **Figure 3** shows typical CLSM optical slices of CTMP and BCTMP fibers, revealing the difference in collapse behaviors.

Table II shows that the elastic modulus of BCTMP, 2.27 GPa, is much lower than 17.17 GPa of CTMP. Refining also increased the fiber flexibility substantially. With low-consistency refining, fiber flexibility increased from about $15 \times 10^{11} \text{ N}^1\text{m}^{-2}$ to about $40 \times 10^{11} \text{ N}^1\text{m}^{-2}$. With medium-consistency refining, the increase was even greater to about $55 \times 10^{11} \text{ N}^1\text{m}^{-2}$.

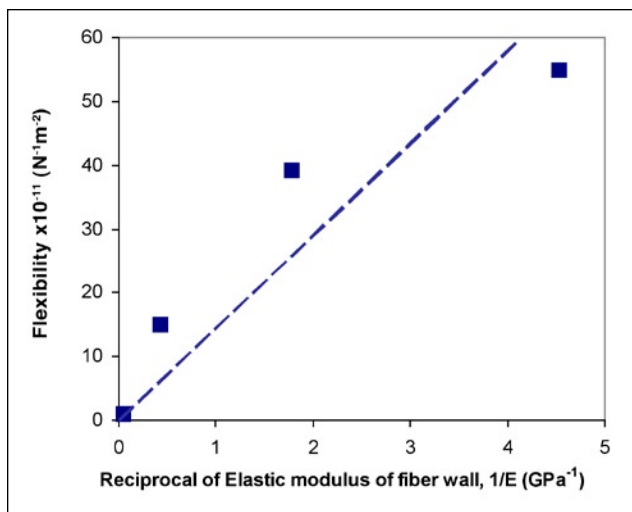
Table II shows that the increase of flexibility by refining is mainly achieved



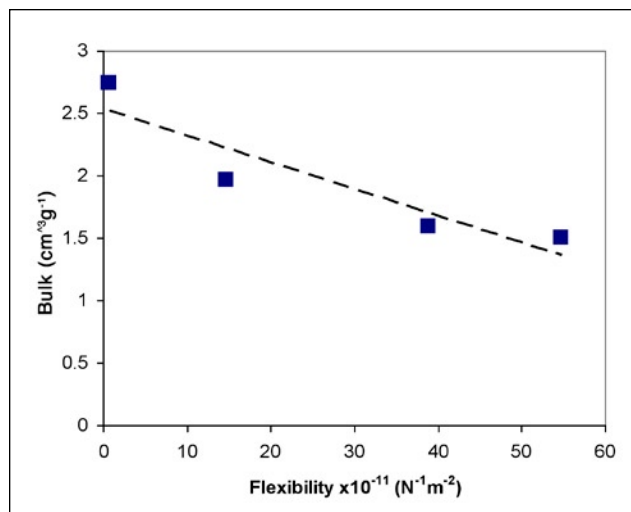
3. Cross-sectional CLSM images of typical aspen CTMP and BCTMP fibers that were wet pressed onto glass slides.

by decreasing the elastic modulus of fibers and the change of moment of inertia has a minimal effect. For the four types of CTMP pulps, the moment of inertia varied only by less than 40%, from $1.20 \times 10^{-21} \text{ N}^1\text{m}^4$ to $0.75 \times 10^{-21} \text{ N}^1\text{m}^4$, while the elastic modulus de-

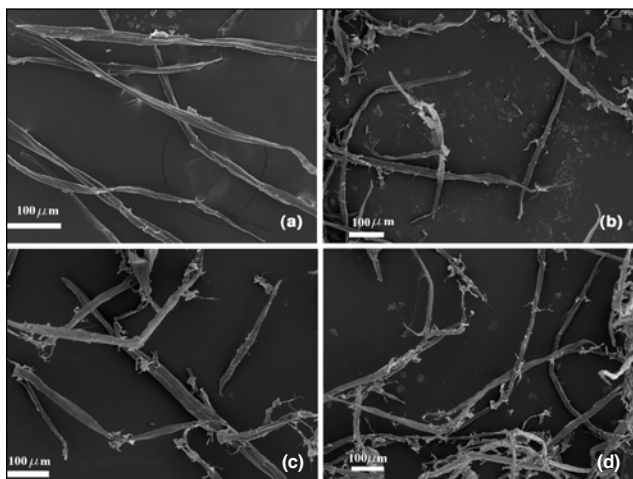
creased by more than 98%, from 17.17 GPa to 0.22 GPa. This can also be illustrated by a plot of flexibility against the reciprocal of elastic modulus of the fiber wall (**Fig. 4**). Fiber flexibility is almost linearly related to it, indicating the insignificant effect of the moment



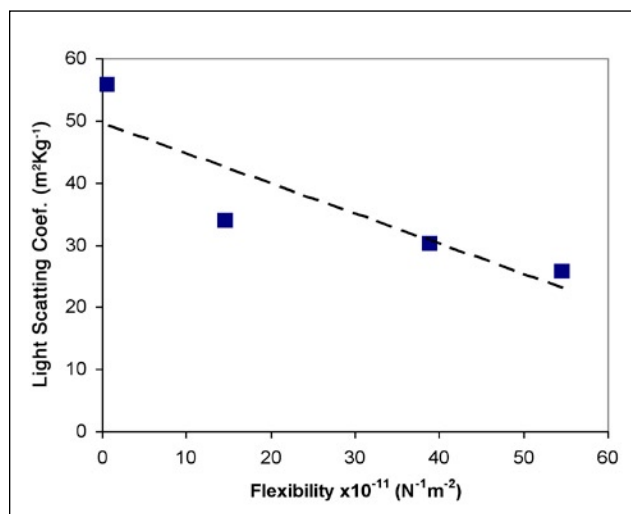
4. Wet fiber flexibility vs. the elastic modulus of fiber wall. Aspen mechanical pulp fibers.



6. Wet fiber flexibility vs. sheet bulk. Aspen mechanical pulp fibers.



5. SEM images of aspen fibers showing fiber morphology change with refining: (a) aspen CTMP; (b) aspen BCTMP; (c) low-consistency refined (LCR) aspen BCTMP, at 3000 revolutions; and (d) medium-consistency refined (MCR) aspen BCTMP, at 4000 revolutions.



7. Wet fiber flexibility vs. sheet light scattering coefficient. Aspen mechanical pulp fibers.

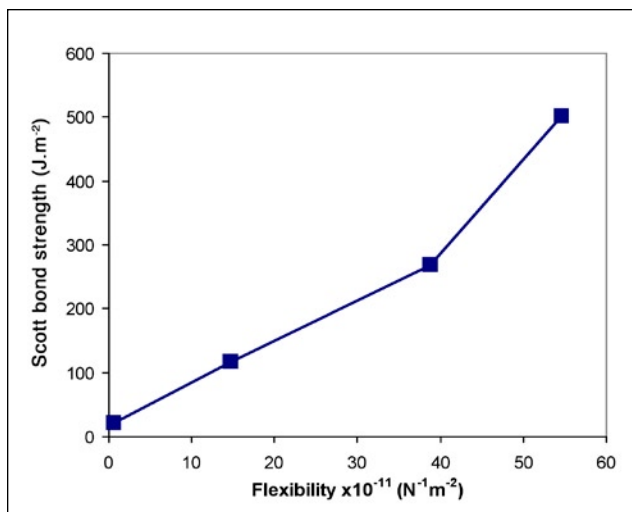
of inertia. This also confirms Paavilainen's assumption that the E and I have different effects on fiber flexibility [23]. We know that refining causes fiber internal fibrillation or cell wall delamination [22, 27]. Alteration of cell wall structure, of course, changes the elastic modulus of a fiber that is modeled as a beam in the flexibility measurement method. The present results, on the other hand, indicate that the new method with CLSM can determine the flexibility of mechanical pulp fibers and its change in the bleaching and pulp refining process.

The increase in fiber flexibility can also be seen with the fiber morphology change (Fig. 5). The original aspen BCTMP fibers appeared rigid. After low-consistency (LC) refining, more kinks were observed but each segment between the kinks still appeared rigid. After medium-consistency (MC)

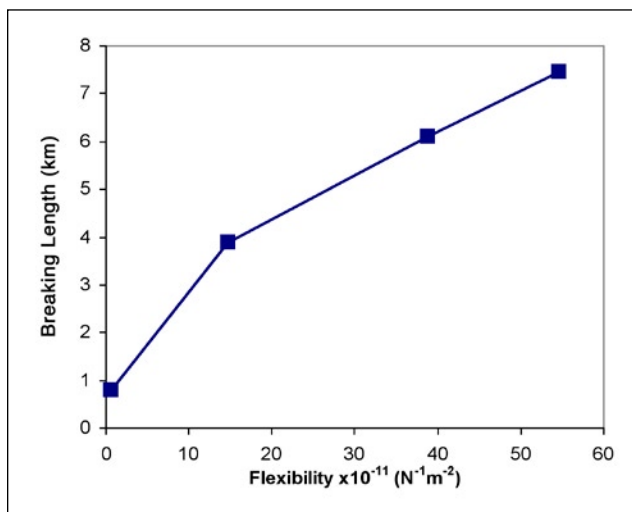
refining, most fibers appeared to be curl-shaped, indicating higher flexibility.

Effect of fiber flexibility on paper properties

Flexible fibers form close contact with one another in the paper sheet, so fiber flexibility relates closely to the paper properties. Steadman and Luner [10] found that the sheet's apparent density and the fiber flexibility were linearly related. We obtained results showing that the data scatters about the straight line (Fig. 6). The sheet density is more sensitive to fiber flexibility when the flexibility is low. The deviation from a linear relationship may be explained by the fact that in the paper sheet containing mechanical pulp, some fibers do not collapse. In our previous study [28], we found that, in a sheet cross-section image, quite a few TMP fibers retained their tubular shape after a supercalendering



8. Wet fiber flexibility vs. inter-fiber bonding strength. Aspen mechanical pulp fibers.



9. Wet fiber flexibility vs. sheet strength. Aspen mechanical pulp fibers.

process. Therefore, for paper grades containing mechanical pulps, fiber flexibility as well as fiber collapsibility will affect the paper structure and, in turn, the paper properties.

Figure 7 shows the relationship between the fiber flexibility and the sheet light-scattering coefficient; the relationship is similar to the relationship between fiber flexibility and sheet density. The light-scattering coefficient indicates the total unbonded fiber surface area in the sheet, including some lumen surfaces, and further supports a role for fiber collapsibility. Nevertheless, when only kraft pulp is used, almost all the fibers in the paper sheet are collapsed, and the paper structure is mainly determined by fiber flexibility.

Figures 8 and 9 show the effect of fiber flexibility on inter-fiber bonding strength and paper tensile strength. More-flexible fibers will form greater contact area for bonding between fibers, thus resulting in higher interfiber bonding strength and tensile strength. The general trend in the

relationships supports this notion, although there is some subtle difference between these two curves. In Fig. 8, inter-fiber bonding strength increases slowly at the beginning and then more quickly toward the end; whereas, in Fig. 9, the tensile strength increases quickly at the beginning and then slowly toward the end. The difference implies that factors other than the fiber flexibility, such as fiber surface chemistry [29] and fiber length, may also influence interfiber bonding. For the BCTMP fiber after MC refining, both surface chemistry and fiber flexibility were changed from CTMP due to bleaching and refining so the interfiber bond strength was increased by greater specific bond strength and bonded area. In terms of tensile strength, fiber length also has an effect. Although the interfiber bonding strength was increased, the fiber length was decreased by refining, indicating the increase of the tensile strength slows after both LC and MC refining.

CONCLUSIONS

Because mechanical pulp fibers are stiffer and have thicker cell walls than kraft pulp fibers, the wall thickness cannot be neglected using the Steadman method to measure the fiber flexibility. With CLSM, it is possible to observe the freespan and the deflection height directly from the fiber transverse section. With the fiber transverse image, a neutral bending plane was defined that gives more accurate measurements of the freespan length. The difference in freespan length measured with CLSM and LM can be as much as 25%. Furthermore, the collapse behavior and the moment of inertia of wet fiber can be measured from the cross-section geometry of the fibers. It is possible to evaluate elastic modulus, flexibility, and collapsibility for the same fiber within one test.

Results show that the flexibility of four types of CTMP fibers correlates well to the paper structure with regard to paper bulk and light scattering coefficient, and to the paper physical strength with regard to interfiber bonding strength and paper tensile strength.

We also found that the flexibility of CTMP fibers was significantly improved by either a bleaching or a refining process, mainly because of the reduced elastic modulus of the fiber wall by bleaching or refining. The change of the moment of inertia due to the processes is minimal. **TJ**

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INSIGHTS FROM THE AUTHORS

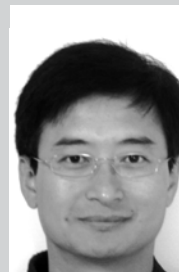
Until now, there has not been a method for measuring the flexibility of mechanically pulped fibers, although methods are available for measuring chemically pulped fibers. The most difficult part of our work was to make sure that the fibers did not move after pressure was removed from the supporting wire. We found that CLSM can provide much more accurate measurements than conventional light microscopy.

The CLSM method will allow mechanical pulp producers to measure fiber flexibility directly. The next step is to try to develop a new instrument for the measurement.

Yan is a doctoral candidate and Li is associate professor,



Yan



Li



Zhou

Department of Chemical Engineering and Limerick Pulp and Paper Research and Education Center, University of New Brunswick, Fredericton, NB, Canada. Zhou is research scientist, Tembec Inc., Temiscaming, QC, Canada; e-mail Li at kecheng@unb.ca.