# THREE-DIMENSIONAL ANALYSIS OF THE COLLAPSE BEHAVIOR OF KRAFT-COOKED NORWAY SPRUCE FIBERS

## Stig L. Bardage

**Research Scientist** 

## Geoffrey Daniel

Professor Wood Ultrastructure Research Center (WURC) Swedish University of Agricultural Science (SLU) Department of Wood Science P.O. Box 7008 750 07 Uppsala, Sweden

and

### Adya Singh

Research Scientist Forest Research Institute Private Bag Rotorua, New Zealand

(Received August 2001)

### ABSTRACT

Computerized reconstruction and measurement of cross-sectional compactness were used to analyze the collapse behavior of fibers in a kraft-cooked fiber bundle containing early- and transition wood fibers. The results show that the collapse behavior of delignified fibers may be determined by fiber structure and dimensions, and how these are affected by the action of external forces. Nevertheless, some deformation may also arise from internal stresses during drying. Cross-sectional compactness was shown to correlate with the collapse resistance of fiber walls. Results show that the collapse behavior of thin-walled fibers with similar cross-sectional compactness values may vary greatly along the fiber axis. Cross-sectional compactness was relatively higher and constant along the fiber axis for fibers with thick cell walls. Fibers with thin cell walls showed lower values of cross-sectional compactness, which seem to decrease towards the middle of the fiber. Cross-sectional compactness seems to increase towards the fiber tips. Fiber ends may become flattened after delignification independently of high values of cross-sectional compactness. Computerized 3D reconstruction techniques may be a valuable tool in understanding the collapse behavior of pulp fibers.

Keywords: Picea abies, 3D, reconstruction, kraft, fibers, collapse, cross-sectional compactness.

#### INTRODUCTION

The ability of papermaking fibers to conform to one another allows for better contact between fibers in the paper sheet. Lower resistance to bending improves fiber conformability. Since wood fibers are tubular, they bend easily when flattened or collapsed, making collapsibility or ability to become flattened a very important pulp fiber property. During chemical pulping, fibers are delignified and exposed to external forces that can change their native cross-sectional shape. Kraft pulp fibers, for instance, can collapse to different degrees after cooking, which may influence fiber flow and distribution prior to papermaking. Important kraft fiber determinants are collapse resistance, fiber perimeter, and wall thickness or wall area combinations, relative number of fibers, and fiber length (Kibblewhite 1999).

Wood and Fiber Science, 34(3), 2002, pp. 382-390

<sup>© 2002</sup> by the Society of Wood Science and Technology

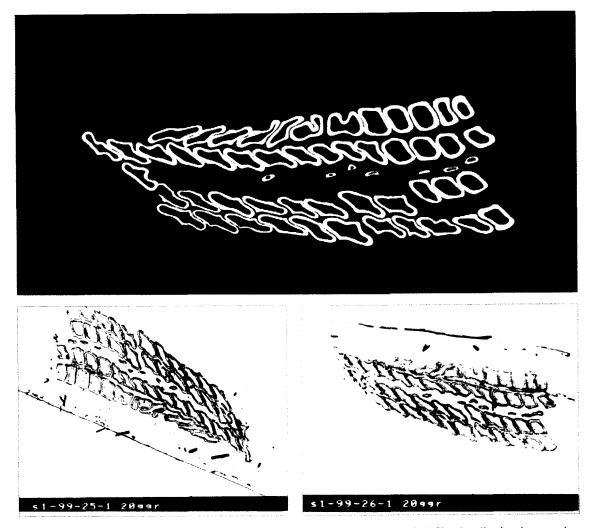


FIG. 1. Above, 3D reconstruction of fiber cell wall segments from a kraft-cooked fiber bundle showing morphological aspects of fiber collapse. Below, images of the two semi-thin section used to create the 3D reconstruction.

In general, as a result of fiber collapse, the cross-sectional area of the fiber lumen is reduced or even eliminated. Such fiber flattening can be estimated and characterized in different ways (Robertson 1963; Page 1967; Kibble-white and Hamilton 1984; Miller 1989; Evans et al. 1997; Jang et al. 1995; Jang and Seth 1998). Degree of fiber collapse is often calculated through the correlation of randomly measured geometrical parameters without any further analysis of the origin or nature of collapse. For instance, Runkel's ratio and Luce's shape factor have been used as a measure of

collapse resistance (Evans et al. 1997). These two parameters are based on cross-sectional measurements assuming separated fibers to be circular in cross section. The presence of cross-sectional compactness has also been suggested as a parameter that would give an indication of the flexibility and collapsibility of fibers (Sirvö 2001). However, very little is known about the properties of cell-wall collapse of whole pulp fibers. Anatomical features such as bordered and crossfield pits (Sirviö and Kärenlampi 1998) as well as fiber dimensions, fiber chemistry, and microfibril an-

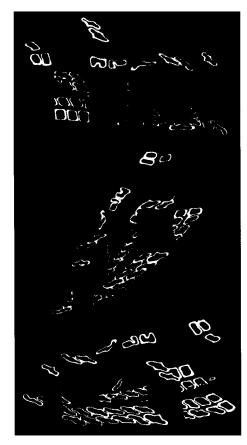


FIG. 2. 3D reconstruction showing the shape of several tracheid segments from three different perspectives.

gle (Evans et al. 1997) may also influence the collapse behavior of fiber cell walls.

In this study, we attempt to analyze the collapse behavior of kraft-cooked Norway spruce fibers using computerized three-dimensional reconstruction and discuss the concept of degree of fiber collapse. The results reflect features present on pulp fibers directly after cooking.

### MATERIAL AND METHODS

A radial section (5 mm thick and 30 mm wide) covering the diameter of a Norway spruce (*Picea abies* [L.] Karst) wood disc was prepared with a knife. The sample was carefully immobilized between two perforated metal plates with screws. Thereafter, the sam-

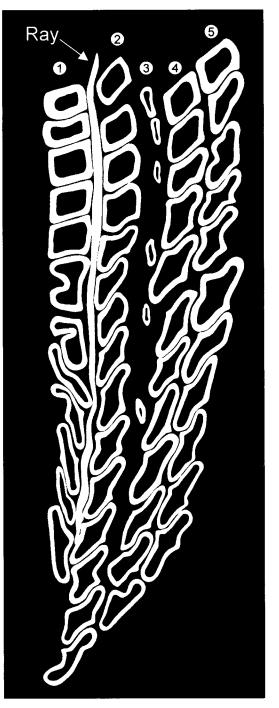


FIG. 3. 2D reconstruction of the first transverse section of the fiber bundle studied. Fiber rows are numbered 1-5. The presence of ray tissue is also displayed.

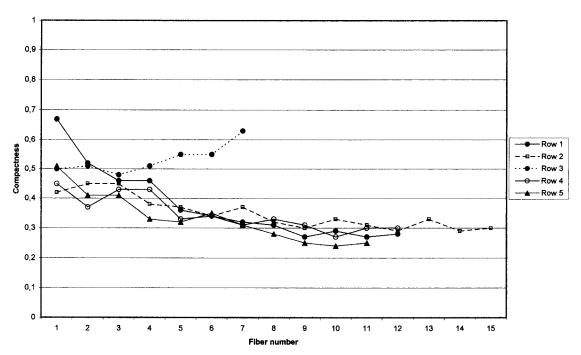


FIG. 4. The diagram shows the cross-sectional compactness of each fiber in relation to its position in the transverse section displayed in Fig. 3 and the trend in variation of cross-sectional compactness along fiber rows.

ple was placed in a laboratory circulation digester and kraft-cooked together with other Norway spruce wood chips to a Kappa number (bulk) of 30.

Fiber bundles of delignified early- and latewood were carefully separated with forceps using a stereomicroscope. Fiber bundles were thereafter dehydrated in acetone and embedded in Spurr's (1969) low viscosity resin. This procedure allowed for fibers to remain together and be aligned in the same direction after delignification. As the collapse resistance of latewood fibers is considered to be high, a sample comprised of both early- and transition wood fibers was chosen. Serial semi-thin (4µm) sections from embedded fibers were cut using a Reichert FC4 ultramicrotome fitted with a Diatome diamond knife. Sections were transferred individually onto glass slides on a drop of water, and subsequently dried with heat. A total of 272 consecutive sections were made.

Sequential images of fiber transverse sections were acquired using a light microscope

fitted with a CCD-camera attached to a computer (Pentium II). Three-dimensional (3D) reconstructions of tracheid segments were generated combining CAD (computer aided design) and visualization software. The transverse shape of fibers was extracted from each image and positioned on top of each other. 3D reconstruction was accomplished by separating the serial planes in the Z direction with a corresponding gap of 4  $\mu$ m in between and linking the serial tracheid segment shapes using Non-Uniform Rational B-Splines (NURBS). Bordered and cross-field pits were not included in this work as their presence adds significantly to the complexity of fiber reconstructions. Observations at higher magnification are needed to improve microstructural details. Reconstructions from pit regions will be published in a subsequent paper. Visualization was accomplished with a high degree of freedom, although here reconstructions are displayed only in tonalities of the grayscale or in black and white.

Cross-sectional compactness of fiber seg-

FIG. 5. Five neighboring fibers numbered 1–5 (shaded) with varying degrees of cell-wall collapse were chosen for further analysis.

ments lacking pits was calculated from  $4\pi A/P^2$  where A is cross-sectional cell-wall area and P fiber perimeter (Sirviö 2001).

Image analysis was performed using software ImagePro PLUS (version 4.0) and Rhinoceros (version 1.1).

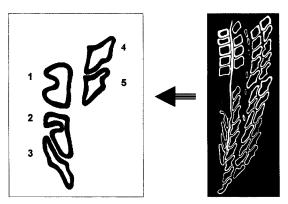
#### RESULTS AND DISCUSSION

The immobilization of sections from the wood discs prior to kraft-cooking allowed for pulped fibers to remain together after cooking supported by each other and by ray tissue facilitating the serial sectioning of several pulped fibers (Fig. 1). Fiber walls may have shrunken a little due to the dehydration process, but this shrinkage may have a marginal effect on fiber shape. However, the fibers were probably affected by some minor mechanical strains during the careful separation of fiber bundles with forceps, although under industrial condition deformations are likely to be much greater.

Observations on computerized reconstructions of transverse sections of the fiber bundles studied showed that earlywood pulp fibers are subject to collapse to different extents after kraft-cooking depending on the action and intensity of external forces (Figs. 1 and 2). Visual analysis performed on a whole cross section of the fiber bundle revealed a trend on the collapse behavior of the fibers (Fig. 3). Thinwalled tracheids with a large cell lumen area collapse more than transition wood fibers that are more thick-walled with a smaller lumen. This is consistent with the findings of Jang and Seth (1998), who concluded that for a given wall thickness, fibers with large lumen perimeters are likely to collapse more readily than those with small perimeters. This trend was confirmed by measurements of cross-sectional compactness that varied from 0.24 to 0.67, and with the exception of row 3 (Fig. 3), to increase from earlywood towards transition wood (Fig. 4). Cross-sectional compactness in row 3 varied from 0.48 to 0.63, which is relatively high compared with the other rows. This may have some relation to the fact that row 3 contains transverse sections of fiber ends suggesting that fiber ends show higher

 TABLE 1. Cross-sectional compactness of the fiber transverse section displayed in Fig. 3.

Distance (µm)	Cross-sectional compactness						
	Fiber 1	Fiber 2	Fiber 3	Fiber 4	Fiber 5		
First Layer	0.33	0.30	0.33	0.36	0.38		
320	0.33	0.30	0.33	0.38	0.42		
560	0.33	0.29	0.31	0.40	0.39		
800	0.35	0.29	0.30	0.36	0.43		
976	0.32	0.29	0.31	0.33	0.39		
Average	0.33	0.29	0.32	0.37	0.40		
Max.	0.35	0.30	0.33	0.40	0.43		
Min.	0.32	0.29	0.30	0.33	0.38		
SD	0.011	0.005	0.013	0.026	0.022		
Total Max.	0.43						
Total Min.	0.29						



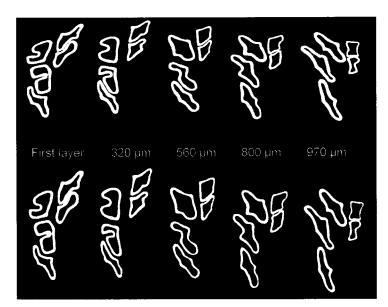


FIG. 6. 3D and 2D reconstructions of fiber segments at 5 levels along the fiber axis. The reconstructions show variable collapse of cell walls of individual fibers within a length of 970  $\mu$ m (i.e., ca. ½ of fiber length).

cross-sectional compactness than regions more distant to the tips.

Further analyses of collapse along the fiber length were performed on five neighboring fibers showing different degrees of collapse (Fig. 5). Measurements were made at a distance of 320, 560, 800, and 970 µm from the first transverse section. The cross-sectional compactness between fibers along this distance varied from 0.29 in fiber n° 2 to 0.43 in fiber n° 5 (Table 1). The cross-sectional compactness of fiber n° 1-3 did not vary as much as for fibers  $n^{\circ}$  4 and 5 (Table 1). This may be due to the position of fibers in relation to the fiber rows. The transverse sections of fibers 1, 2, and 3 are at the same level due to the fact that these fibers belong to the same radial row. Fibers 4 and 5 belong to a neighboring row and their transverse sections most certainly do not reflect the same levels as for fibers 1-3.

Observations on 3D reconstructions of these fiber segments revealed that pulp fibers may collapse differently along their axis and that cross-sectional compactness seems to correlate with the ability of the fiber walls to collapse (Fig. 6 and Table 1). However, cross-sectional compactness, and most probably any other type of estimation of fiber flattening, can not predict how the collapse of fiber walls develops. In the present study, irregular collapse was developed in fibers with cross-sectional compactness values lower than 0.40. The irregular development of fiber flattening that was observed along fibers 1-3 supports this assumption (Fig. 6). In the case of fibers 4 and 5, the higher values of cross-sectional compactness were reflected in less deformed crosssectional areas still resembling the original fiber form (Fig. 6). However, fibers 4 and 5 were in contact with a ray at the 320-µm level (Fig. 5), which may have influenced collapse development. The results also indicate that the morphology of collapse may be a result of anatomical and structural features of the delignified fiber cell wall, and action of external shear and compression forces. These findings indicate that the collapse behavior of fiber walls may vary greatly along the fiber axis regardless of similar cross-sectional compactness values.

Another interesting feature was the presence of cell-wall corners after delignification and partial separation of fibers. Persistence of the

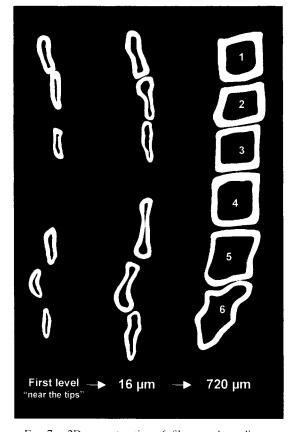


FIG. 7. 2D reconstructions 6 fibers at three distances along the fiber bundle (first level; 16  $\mu$ m from first level; and 720  $\mu$ m from first level). Two layers are located at the fiber ends and the third layer is distant (i.e., ca. ¼ of fiber length) from the fiber ends.

cell corners may be related to a difference in cell-wall structure, for instance, the packing and agglomeration (aggregation) of cellulose microfibrils of delignified fibers (Hult et al. 2001).

Observations on the collapse behavior of fibers at three distances from the fiber tips revealed that regions near the fiber tips were more flattened than regions more distant from the tips (Fig. 7). Cross-sectional compactness was relatively high and constant along the axis for fibers with thick cell walls (fibers 1 and 2), while in fibers with thinner cell walls, this feature tended to decrease towards the middle of the fiber (Fig. 7 and Table 2). Moreover, cross-sectional compactness (i.e., within a distance of  $0-32 \ \mu m$  from the fiber tips) seems to increase towards the tips. It has been reported that collapse of dried spruce pulp fibers occurs more frequently at fiber ends due to the thinner cell walls in this region (Page 1967). However, the fact that cross-sectional compactness was relatively high in this region suggests that this fiber region should not be prone to deformation, and that the collapse of fiber ends must be related to fiber morphological structure (Bardage 2001) or to the action of external or internal strains. For instance, collapse and shrinkage at moisture contents above the fiber saturation point (FSP) take place during drying where water causes tension within the fiber lumen (Kauman 1964). The fibers studied here were dehydrated from a moist state. As stated by Page (1967), the

Level	Distance _ (µm)	Cross-sectional compactness						
		Fiber 1	Fiber 2	Fiber 3	Fiber 4	Fiber 5	Fiber 6	
Fiber tip	0		_		_			
First level	20		_		0.55	0.55	0.65	
	32	0.50	0.51	0.48	_			
16µm	36				0.35	0.39	0.38	
	48	0.47	0.42	0.39		_		
720µm	740		-	_	0.42	0.33	0.30	
	752	0.48	0.48	0.40		_		
	Average	0.58	0.50	0.37	0.43	0.35	0.45	

TABLE 2. Cross-sectional compactness of fiber transverse sections displayed in Fig. 7. Fibers 4–6 are displaced by  $12 \mu m$  in relation to fibers 1–3.

resistance of fibers to collapse is a function of the rigidity of the fiber wall in its transverse plane. Furthermore, thin-walled tracheids, which also show slightly higher microfibril angles (MFA) compared to latewood tracheids (Paakari and Serimaa 1984; Sahlberg et al. 1997), are likely to be mechanically prestressed due to greater lignification (Gindl and Wimmer 2000). For example, the lignin content in secondary walls was found to be positively correlated to tracheid diameter and negatively correlated with cell-wall thickness (Gindl and Wimmer 2000). On the other hand, recent results by Bergander and Salmén (2000) showed that the fibril angle of the  $S_{2}$ layer of the wood cell wall of Norway spruce does not influence the transverse elastic modulus of the fiber wall. Rather their results indicate that the  $S_1$ - and also the  $S_3$ -layer (with much higher MFA compared with the S<sub>2</sub>-layer) would be more involved in the large variation of transverse elastic modulus. Nevertheless, fiber dimensions and anatomical features like cell-wall corners, bordered and cross-field pits together with the transverse properties of the lignin-hemicellulose matrix (Bergander and Salmén 2000) may also influence how fibers collapse along their axis.

The use of 3D reconstruction techniques allowed for a better interpretation of the collapse behavior of pulp fibers. The results in the present study show that the collapse behavior of fibers is most likely determined by fiber structure and dimensions in association with degree of delignification and action of external forces. Nevertheless, some deformation may also arise from internal stresses during drying. The disposition for collapse of fibers coming from an annual ring in wood may be ordered in a gradient pattern from early- to latewood. Thinwalled fibers will collapse more than thickwalled fibers up to a certain limit of crosssectional compactness. Thin-walled fibers with large cell lumens may collapse irregularly along their axis depending on the action of external forces, and independently of similar cross-sectional values. Fiber ends may become flattened after delignification independently of high values of cross-sectional compactness. All these features will have important implications on fiber flow and fiber formation along the paper line. Industrial cooks where tons of pulp are being produced simultaneously are likely to impose much greater mechanical strains on the fibers resulting in even greater fiber collapse.

Although Sirviö (2001) observed that the cross-sectional compactness of Norway spruce fibers was quite invariant along the fiber length axis, this study shows that the cross-sectional compactness of thin-walled early-wood fibers may vary along the fiber axis allowing for localized flattening or collapse.

### ACKNOWLEDGMENTS

This work was carried out within the framework of the Wood Ultrastructure Research Centre (WURC), a competence center at the Swedish University of Agricultural Sciences and the SLU Wood Chain Program. The authors also want to thank Peter Sandström from SCA (Svenska Cellulosa Aktiebolaget) for carrying out kraft-cooking of the spruce material used in this study.

#### REFERENCES

- BARDAGE, S. L. 2001. Three-dimensional modeling and visualization of whole Norway spruce latewood tracheids. Wood Fiber Sci. 33(4):627–638.
- BERGANDER, A., AND L. SALMÉN. 2000. Variations in transverse fibre wall properties: Relations between elastic properties and structure. Holzforschung 54(6):655–661.
- EVANS, R., R. P. KIBBLEWHITE, AND S. STRINGER. 1997. Kraft pulp fiber property prediction from wood properties in eleven radiata pine clones. Appita J. 50(1):25– 33.
- GINDL, W., AND R. WIMMER. 2000. Relationship between lignin content and tracheid morphology in spruce. Pages 163–168 in H. C. Spatz and T. Speck, eds. Plant Biomechanics, Freiburg-Badenweiler, Aug. 27–Sept. 2.
- HULT, E.-L., P. T. LARSSON, AND T. IVERSEN. 2001. Cellulose fibril aggregation—An inherent property of kraft pulps. Polymer 42:3309–3314.
- JANG, H. F., AND R. S. SETH. 1998. Using confocal microscopy to characterize the collapse behaviour of fibers. TAPPI 81(5):167–173.
- , R. C. HOWARD, AND R. S. SETH. 1995. Fiber characterization using confocal microscopy—The effect of recycling. TAPPI 78(12):131–137.

- KAUMAN, W. G. 1964. Cell collapse in wood. Part 1: Process variables and collapse recovery. Holz. Roh-Werkst. 22(5):183–196.
- KIBBLEWHITE, R. P. 1999. Designer fibers for improved papers through exploiting genetic variation in wood microstructure. Appita J. 52(6):429–435.
- —, AND K. A. HAMILTON. 1984. Fiber cross-section dimensions of undried and dried *Pinus radiata* kraft pulps. N.Z. For. Sci. 14(3):319–333.
- MILLER, P. 1989. Fiber and sheet properties resulting from refining stock concentration variation. Appita J. 42(2): 125–130.
- PAGE, D. H. 1967. The collapse behaviour of pulp fibers. TAPPI 50(9):449-455.
- PAAKARI, T., AND R. SERIMAA. 1984. A study of the struc-

ture of wood cell by X-ray diffraction. Wood Sci. Technol. 18:79–89.

- ROBERTSON, A. A. 1963. The physical properties of wet webs. Sven. Papperstidn. 66:477–497.
- SAHLBERG, U., L. SALMÉN, AND A. OSCARSSON. 1997. The fibrillar orientation in the S<sub>2</sub>-layer of wood fibres as determined by X-ray diffraction analysis. Wood Sci. Technol. 31:77–86.
- SIRVIÖ, J. 2001. Variation of cross-sectional properties within single Norway spruce tracheids. Wood Fiber Sci. 33(1):16–25.
- , AND P. KÄRENLAMPI. 1998. Pits as natural irregularities in softwood fibers. Wood Fiber Sci. 30(1):27–39.
- SPURR, A. R. 1969. A low viscosity embedding medium for electron microscopy. J. Ultrastruct. Res. 26:31–43.

## **IMPORTANT NOTICE**

A number of price changes will take effect on January 2003. Subscription price for the journal will be \$250. Dues for full members will be \$75 per year; student dues will be \$25; and dues for retired members will be \$40.

For articles received after January 1, 2003, page charges will be \$100 for SWST members and \$135 for nonmembers.