# Microscopic observations during longitudinal compression loading of single pulp fibers

**Irving B. Sachs**
Forest Products Technologist, USDA Forest Service, Forest Products Laboratory, One Gifford Pinchot Dr., Madison, Wis. 53705

**ABSTRACT** *Paperboard components (linerboard adn corrugating medium) fail in edgewise compression because of failure of single fibers, as well as fiber-to-fiber bonds. While fiber-to-fiber- bond failure has been studied extensively, little is known about the longitudinal compression failure of a single fiber. In this study, surface alterations on single loblolly pine kraft pulp fibers were observed during compurssion in a devise built for use in the scanning electron microscope. Distinct changes were observed in the fibers subjected to compression loading, including dislocations (slip planess), loosening of the fibrils, piling up of the bigrilar walls, and fibril fracture when loaded in compurssion. From these observations, the apparent failure sequence for a single pulp fiber is (a) formulation of dislocations, (b) loosening of the fibrils, piling up of the fibrillar walls, and eventual fibril fracture, and (c) coalescence of fractures, causing further reduction in fiber wall strength and eventual failure of the fiber. Direct observation of fibers under compression load suggests that sgrenghtening of the fiber wall, such as by use of chemical additives, could lead to improved fiber mechanical properties.*

**KEYWORDS**
Compression tests
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Scanning
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Fibrils
Fracture
Slip planes
Mechanical properties
Paper board properties

Fiberboard containers are subjected to compression loads in handling, storage, and shipment, The strength of the fiberboard container depends upon the strength properties and physical characteristics of the paperboard components that comprise the walls of the container, as well as fabrication and design features. The basic component of the paperboard is the pulp fiber itself. A recent study (*1*) shows that edgewise compression failure of paperboard components involves failure of fiber-to-fiber bonds as well as single fibers. While much information is available on the failure characteristics of fiber-to-fiber bonds(*2*), little information is available on the failure of single fibers in longitudinal compression loading (*3*).

Because failure of individual fibers is a contributing factor in edgewise compression failure of paperboard, we felt it was important to determine the failure sequence of individual fibers subjected to compression loading. Knowledge of changes in the pulp fiber wall may provide an indication of the compressive resistance of the finished container. Observation of external changeson the fiberwall should provide information about how and why pulp fibers fail under compressive load. Combining the knowledge gained from these compression studies on single fibers with information gained from studies on the edgewise compression of paperboard (*1,4*) may lead to a better understanding of container performance under compression loading.

The behavior of a single pulp fiber under longitudinal compression loading has not been studied in the dynamic mode. Although optical microscopic methods provide rapid techniques for assessingthe changes that occur under compression, the microscope's resolution is insufficient for detailed study of selected areas. The scanning electron microscope (SEM) can overcome this problem. In addition to high resolution, three-dimensional observations of SEM images permit the definite determination of pulp fiber surfaces. The SEM is widely used to study the topography as well as the internal structure of wood and pulp fibers. However, there is tittle SEM data on surface alteration of paperboard fibers following compression (*1-5*). Successful use of an SEM in this situation requires careful preparation of single pulp fibers and development of a device capable of holding and compressing individual fibers.

In this study, we observed the events occurring during compression failure of a single pulp fiber by using a single fiber cornpression device that was specifically designed for use in the specimen chamber of an SEM. This permits high-magnification, high-resolution examination of changes in the walls of individual pulp fibers as they undergo longitudinal compression. Loblolly pine kraft pulp fiber was subjected to longitudinal compression loading so that dislocations (slip planes), piling up of fibriIs, and fractures could be observed.

## Results and discussion

### Changes in cell wall of a single pulp fiber under longitudinal compression loading

When pulp fibers are compressed as individual columns. a normal cell wall displays dislocations, which form in the free span between the supported ends of the fiber. Figure 1A shows a single pulp fiber before longitudinal compression. Figure 1B shows the same fiber after longitudinal: compression, with compression dislocations appearing on the fiber wall. Figure 2 shows evidence of disturbance within the dislocation, including fibrils separating from one another, loosening, piling up, and fracturing.

Compression loading causes dislocation formations in the gold-coated fiber, exposing non-gold-coated areas beneath the fiber surface to the SEM gun's electron beam.[1] When the electron beam impinges upon these non-gold-coated areas, it causes the buildup of an electrical charge. Visually, the charging shows up on micrographs as streaks or charging lines across the fiber surface. While these charging lines originate from dislocation areas, the straightness of the charging Fines does not mean that the dislocations occur in straight lines across the fiber surface. Indeed, these dislocations occur randomly over the fiber surface. The straightness of the charging lines is an artifact of the arrangement of the SEM electron gun, the fiber specimen, and the electron collector (*6*). This charging effect shows clear widen—of dislocations, as seen in Fig. 1B. Formation of voids or tears in the fiber can also cause charging.

[1]The fiber surface needs to be electrically grounded. Therefore, it is coated with a thin layer of gold prior to viewing in the SEM. Temperature damage of the specimen during the gold-coating procedure and subsequent examination in the SEM is quite small (*6*). The specimen in subject to a temperature of only one or two degrees centigrade over ambient temperature during both the gold-coating procedure and the SEM examination (*7*).

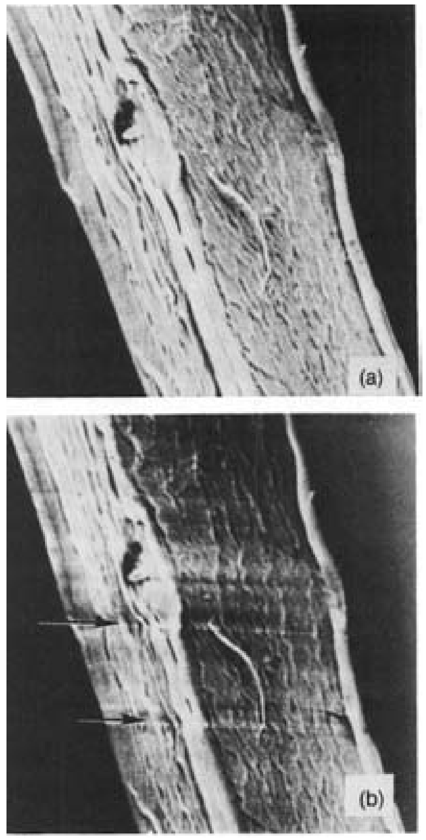
Because so little is known about the behavior of single pulp fibers subjected to longitudinal compression, it is interesting to compare their behavior with that of wood under compression. It is generally recognized that the first discernible stage in the mechanical failure of wood under compression stress is the appearance of minute dislocations in the structure of the cell wall (*8*). Dislocations are found in wood that has been compressed parallel to the grain. It is, therefore, not too surprising to observe that one of the first changes in pulp fibers under compression is similar to that of wood under compression. This is because the pulp fiber wall is a homologue of the wood cell wall in spite of chemical and physical alteration resulting from fiber processing.

### Fracture propagation

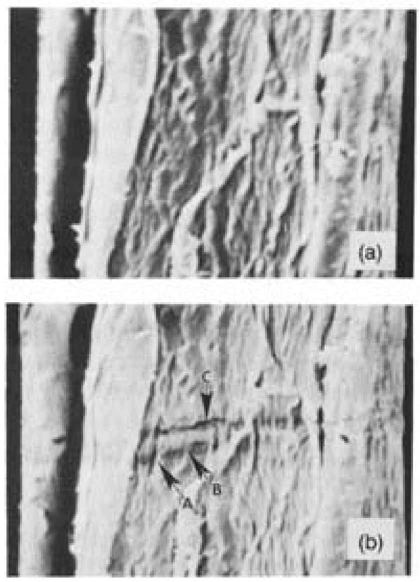
Early in the dynamic longitudinal compression failure sequence, fracture of the fibrillar walls is observed in the fibers as dislocations are formed (Fig. **3).** As the dislocations develop, the fibrillar wall in the area of the dislocation appears to pile up, which creates voids or tears in the fibrillar wall. As compression continues, the voids or fractures appear to coalesce with neighboring fibrillar wall fractures, further weakening the cell wall and causing the split to proceed until it eventually encircles the fiber, as seen in Fig. 4B (see enlargement in Fig. 4C). A comparison of Figs. 4A and 4B shows evidence of thickening and a slight twisting of the fiber. Severing of the fibrillar wall is apparently a slow process and appears to depend upon the uniformity of stress upon the cell wall.

These observations are made while the fibers are in the vacuum environment of the scanning electron microscope. The vacuum in the specimen chamber ($10^{-4}$ mm Hg) is maintained in order to: (a) prevent collision between the electron beam and gaas molecules, thus eliminating such problems as decreased resolution, chromatic aberation, and reduced contrast of the image, (b) avoid high-voltage breakdown of the electron microscope, and (c) preserve the life of the electron gun's tungsten filament. It may be that the vacuum in the specimen chamber is a contributing factor in the observed fracture results, How- ever. even if the vacuum environment does hasten fracture growth, it does not affect the manner En which the

1. A single loblolly pine kraft pulp fiber before (A) and after (B) longitudinal compression loading (Magnification = 2000X) The arrows in 1(B) indicate charginglines that originate from dislocations in the fiber wall following compression.




2. A loblolly pine kraft pulp fiber before (A) and after (B) longitudinal compression loading (Magnification = 2000X). The arrows in 2(B) indicate results following compression. Arrow A shows separation of fibrils from one another: ;rrow B shows piling; and Arrow C shows fracture of the fibrils.



pulp fiber responds to longitudinal compression loading. The pulp fibers showed no apparent brittleness, and there was no evidence of snapping or shattering of the fiber wall under longitudinal compression loading when viewed on the microscope viewing screen.

### Compression failure sequence

The fiber's failure sequence can be constructed by combining the observed changes in (a) the outer cell wall from compression of a single pulp fiber and (b) the fracture propagation after fibrillar wall failure. The failure sequence appears to be initiated by the formation of dislocations in the pulp fiber cell wall. Under increasing compressive load, these dislocations lead to separation and loosening of the fibrils, piling up of the fibrillar wall, and eventual fibril fracture. These fractures or cracks in the fibrillar wall appear to weaken the pulp fiber. As the cracks grow together, the cell wall's strength diminishes and the fiber ultimately fails. Fiber buckling appears to be the last sequential event of the failure.

### Additional ultrastructural details of single-pulp-fiber cell walls under compression

The changes occurring within a single pulp fiber show that deformation dislocations and fractures are the result of compressive forces. In the zone of dislocation, separation or tearing apart of the fiber wall appears to be a result of slippage. Therefore, separation may constitute a secondary rather than a primary result of loading. Fracture of the fibrillar wall is accompanied by dislocation ridges.

In the zone of rupture, ray cells are generally more resistant to failure than fibers. Their resistance to compression parallel to the axis of the fiber has been explained by the fact that their cells are circular (*9*). Thus they are compressed in the plane of their diameter, unlike fibers, which are compressed along their length. However, in this study it was found that where longitudinal compression of single fibers in association with ray cells occurred, fracture of the ray cell's wall appeared to coincide with the fiber's plane of rupture.

Furukawa (*5*) found that single wood fibers under tensile stress broke at the bordered-pit chamber. In this study we observed that, when single pulp fibers were longitudinally compressed, the dislocations enlarged after buckling of the fiber. Most of these dislocations occurred between the bordered pits, as illustrated in Fig. 5.

## Conclusions

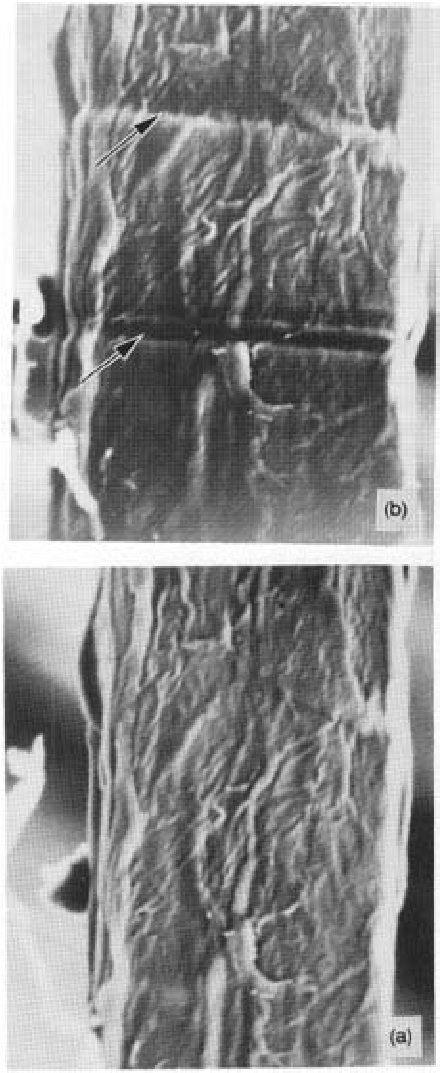
The apparent failure sequence of a single pulp fiber subjected to increasing longitudinal compression loading is:

**1.** Formation of dislocations in the pulp fiber cell wall

**2.** Loosening of the fibrils, piling up of the fibrillar walls, and eventual fracture of the fibril

**3.** Coalescence of fibril fractures, causing further reduction in cell wall strength until the fiber eventually fails.

3. A loblolly pine kraft pulp fiber before (A) and after (B) longitudinal compression loading (Magnification = 1800X). The arrows in 3(B) show fractures of fibrillar walls in the slip plane of the dislocations.



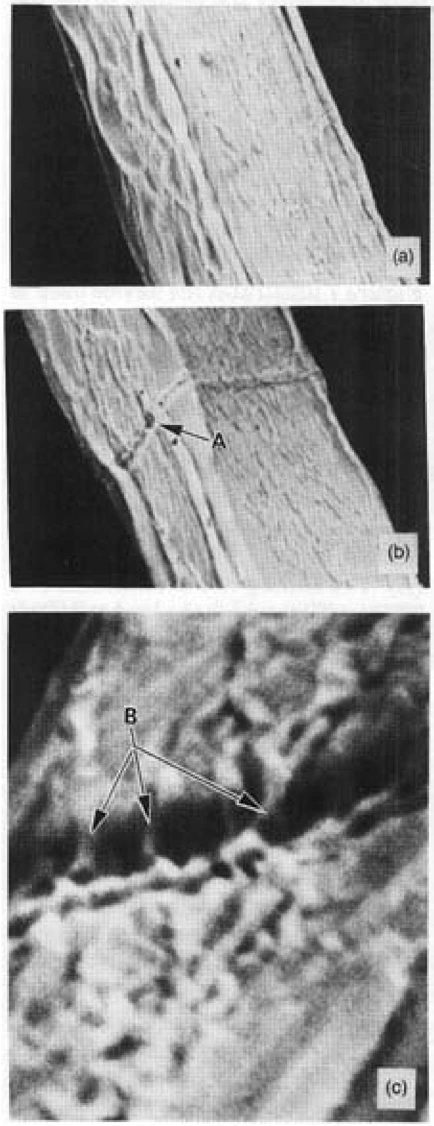

Failure of a single pulp fiber during longitudinal compression loading appears to result from the separation and fracture of the fibrils within the fiber wall. Buckling of the fiber appears to be the last sequential event of the failure.

## Experimental procedures and apparatus

### Preparation of single pulp fibers

Loblolly pine (*Pinus taeda* L.) unbleached kraft pulp (kappa no. 47) was refined in a Valley beater to a freeness of 600 mL CSF. The pulp was

4. A loblolly pine kraft pulp fiber before (A) and after (B) longitudinal compression loading (Magnification = 1000X). Figure 4(C) is an enlargement of 4(B) (Magnification = 3000X). Arrow A in 4(B) shows where the fracture areas have coalesced and encircled the fiber. Arrow B in 4(C) shows that some remnants of the wall are still intact. The depth of the fracture is unknown.



placed in a petri dish and air dried. Individual fibers were separated from fiber clumps with the aid of fine tweezers. Pulped summerwood fibers were selected for the compression studies because of the fragility of pulped springwood fibers. The individual pulp fibers of loblolly pine ranged from 3.0 to 4.6 mm in length and from 32.0 to 45.0 $\mu$m in diameter.

### Fiber mounting technique

The fibers selected for testing had few or no dislocations in their walls caused by fiber processing. Fibers that had twisted into irregular shapes during the drying process were discarded. The straight shaft from nontwisted fibers was cut free into segments that averaged 1.5–2.0 mm in length. Figure 6 illustrates the method used to mount the fibers prior to testing. One end of the straight shaft of the pulp fiber was embedded in a drop of cellulose acetate cement on the flat surface of a small (4 mm diam. x 5 mm long) brass cap (Fig, 6a). After the cement dries, the cap and fiber are slipped onto rod A. A second cap with a drop of cellulose acetate cement is slipped onto rod B. Quickly, rod A is moved so that the free end of the fiber on cap A is guided into the cement of cap B (Fig. 6b). The cement is then allowed to cure.

### Compression testing apparatus

Figure 7 illustrates the device used to individually compress single pulp fibers. The device is made of brass and is small enough (66 mm long x 31 mm wide x 21 mm high) to fit into the specimen chamber of an SEM. The pulp fiber sample (K) is cemented to caps that slip onto rods (A and B) of 2.5-mm diameter. The rods are made of steel, one sliding (B) and one fixed (A). The sliding rod is connected to a “T” bar (C) to which the load cell (D) is attached.

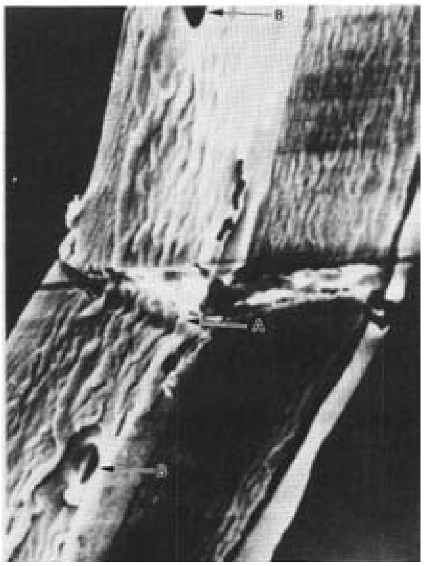
As the microscope rotation knob (located outside the microscope specimen chamber) is hand-turned, the lead screw (E) advances brass bar (F) and pushes the load cell (D) forward. Rod B, extending from the “T” bar (C) with connecting load cell (D), is pushed towards the fixed rod (A) compressing the pulp fiber sample.

The load cell (D) is used to measure the compression load. The load cell is a thin band of aluminum, 40 mm long x 5 mm wide x 0.25 mm thick. Four strain gauges (I) are bonded to the aluminum load cell, forming an electronic bridge. Lead wires (G) from the strain gauges to terminal points (J) can be connected through a port in the column of the microscope to an amplifies signal conditioner, a digital voltmeter, and a chart recorder to record the load.

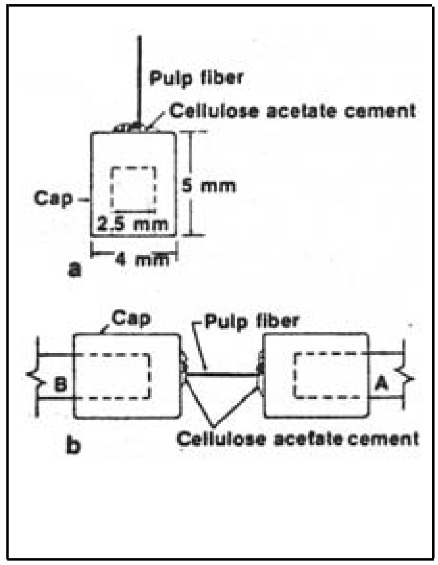
Because the single pulp fiber is cemented at each end to the caps at the tips of the steel rods (Fig. 6b), the pulp fiber is directly in line with the applied load. The free span between the supported ends of the fiber was estimated to be between 0.1 mm and 0.8 mm. A commercially available diffraction grating was used to verify magnification.

To prevent charging within the microscope from the electron beam, the pulp fiber samples were coated with a grounding metal (approximately 100 Å of gold). When operating the microscope, the electron beam strikes the coated pulp fiber, and the microscope collector receives electrons emerging from the sample. The collector in turn

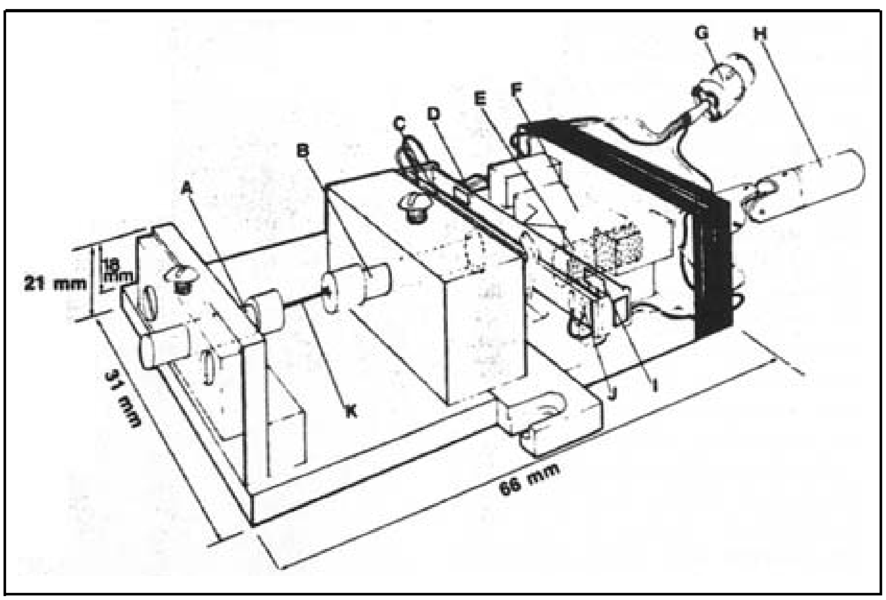
5. Most of the dislocations occurred between bordered pits (Magnification = 2000X). Arrow A shows dislocation situated between the bordered pits indicated by Arrows B.




6. Method used to mount fibers prior to testing. Figure 6a shows a pulp fiber with one end glued to a brass cap. Figure 6b shows a pulp fiber that has been glued to brass caps at the ends of Rods A and B.




7. Apparatus for longitudinal compression loading of a single pulp fiber. A, fixed rod; B, sliding rod; C, “T” bar; D, load cell; E, lead screw; F. brass bar; G, lead wires from strain gauges; H, microscope rotation knob; I, strain gauges; J, terminal points; K, single pulp fiber sample.



sends these to a photo-multiplier that generates the display signal. This allows direct observation of the pulp fiber as it is being compressed. The coated fibers were studied and photographed using a Cambridge Mark II scanning electron microscope at an accelerating voltage of 20 kV.

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