MATERIAL DAMAGE DUE TO ELECTRON BEAM DURING TESTING IN THE ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE (ESEM)

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ABSTRACT

This study describes the development of cell-wall damage, i.e., the creation of cracks across or in the vicinity of pits during the testing of twenty microtomed spruce (*Picea abies* karst.) samples in the Environmental Scanning Electron Microscope (ESEM). Samples were investigated both in an unloaded condition and under a constant tensile load and at different moisture levels. Regions of the moisture-cycled samples that had been exposed to an electron beam during image acquisition showed damage running through pits and their surroundings. Specimens loaded in the green condition and dried in the chamber for 2 h without beam exposure except during imaging showed no noticeable cell-wall damage. The results indicate that the electron beam may be a major source of damage initiation. Therefore, it is essential to note the circumstances of the test when explaining the observations made in ESEM studies.

Keywords: Constant load, damage, drying, electron beam, environmental scanning electron microscopy, pit, wetting

INTRODUCTION

Due to its wide structural applications, the long-term behavior of wood has been a research area for many decades. However, the relationship between the long-term behavior of wood under varying load and moisture conditions and its anatomical structure at the cell-wall level has not yet been clearly understood. Conventional Scanning Electron Microscopy (SEM) and ESEM are convenient tools that can be applied for such investigations.

The SEM technique has been utilized to reveal the failure morphology of wood under different loading conditions. The effect on the failure behavior of wood of the low moisture content in the specimen and the high vacuum involved in the SEM procedure has been studied by Kyanka (1976) and others (see Hoffmeyer and Hanna 1989). In the past, Côte and Hanna (1983), Zink et al. (1994), and Kifetew et al. (1997) have used the SEM technique to characterize wood fracture surfaces. Hoffmeyer and Hanna (1989) have utilized the SEM method to study the influence of an electron beam on the failure morphology of spruce (*Picea abies*).

Recent improvements have led to the development of ESEM as a suitable technique for investigating biological tissues that contain water in the form of vapor or liquid. Compared to the conventional SEM technique, the drying and coating of the specimen are unnecessary and studies can be made in the chamber at low vacuum. Taking advantage of

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this development, Mott et al. (1995) and Shaler et al. (1996) have tested individual fibers in tension using ESEM. Their observations suggest that tension failure is related to pits. Accordingly, the present authors have tested microtomed green samples of normally grown spruce (*Picea abies*) in the ESEM in order to relate the surface deformation of wood under constant tensile load and varying moisture conditions to its anatomical structure. During the testing in the ESEM, damage (microcracks) has been observed at or in the vicinity of pits. However, most SEM fracture surface studies have demonstrated that the pits provided a resistance to transwall failure, i.e., to failure that runs all the way through a pit. A literature review of the subject has been presented by Kifetew (1996). Nevertheless, damaged pits have been detected by Turkulin and Sell (1997) in SEM studies on Scots pine and Norway spruce samples exposed to natural and artificial weathering. Feist and Hon (1983) have observed diagonal microcracks passing through the bordered pits after UV irradiation for 1000 h. Jenkins and Donald (1997) have investigated the sensitivity of cellulosic fibers to the electron beam. They believe that damage mechanisms are enhanced by the continual presence of water vapor in the ESEM chamber. Therefore, the principal objective of the present investigation was to study the influence of electron beam, moisture variation, and loading condition on the cell-wall structure of wood.

MATERIAL AND METHODS

Twenty sapwood samples of normally grown spruce (*Picea abies* Karst.) were cut in the green condition using a sliding microtome along the fiber direction. The cross-sectional dimensions were 0.2 mm by 10 mm, and the length was 45 mm. Five test samples were air-dried for 2 days, and the remaining fifteen specimens were preserved in the green state prior to tensile loading parallel to the grain. During the test, each sample was mounted on a microtensile stage that fits inside the ESEM. The microtomed specimen is gripped and held rigidly using a cyanoacrylate adhesive from Mega Metal.

Several regions, referred to as A, B, C, etc., were selected for image acquisition. On each sample, a distinguishable earlywood zone with several pitted cells was selected as region A, and the initial image was recorded prior to loading and changing the moisture state. This was done in order to record any damage that might have been introduced onto the specimen surface during sample preparation and air-drying. Another region of interest, B, was selected as a standstill position for the electron gun during the drying and wetting periods. At this position, the gun alignment, which normally has x,y-coordinates of (0,0), was changed to another x,y-coordinate position in order to avoid direct beam exposure of this region. Region B was chosen as a standstill position during the drying and wetting periods in order to protect region A from long-term beam exposure. This was done because we were not sure whether the change in electron gun alignment at region A would achieve the desired goal and prevent the region from beam exposure. Other regions were chosen as comparison sites in case damage was detected in region A during the test procedure.

The accelerating voltage during the tests was 15 kV. The samples were wetted by circulating cold water through the tensile stage until condensed water was seen on the sample surface. The samples were dried at 666 Pa and 30°C. Each sample was loaded at a rate of 100 μm/sec to a level of 15 N. In most cases, images were recorded at a magnification of 300×, but some images were also acquired at higher magnifications.

The experimental procedures for each group can be summarized as follows:

a) *Six unloaded and moisture-cycled green samples:* On each sample, region A was selected and an initial image was acquired in the green condition. To protect region A from long-term beam exposure, a new region of interest B was selected as a standstill position. The specimen was dried for 30 min, and a

FIG. 1a. Micrographs of region A after 5.1 min. See Fig. 1b next page.

second image of region A was recorded. The sample was then exposed to two moisture cycles of 10 min wetting followed by 30 min drying. During the wetting/drying procedure, the electron gun alignment was directed away from region B. Images of region A in the dry state were recorded after each wetting/drying cycle. Additional sites were also selected and images were recorded for comparison purposes.

b) *Tensile loading and moisture cycling of five green samples:* An initial region A image was first recorded on each sample and the sample was loaded. After loading, a second image of the region was acquired. A new region B was selected as a standstill position, and the sample was dried for 30 min, and the dried image of region A was recorded. The sample was then exposed to two 10-min wetting periods followed by 30-min drying cy-

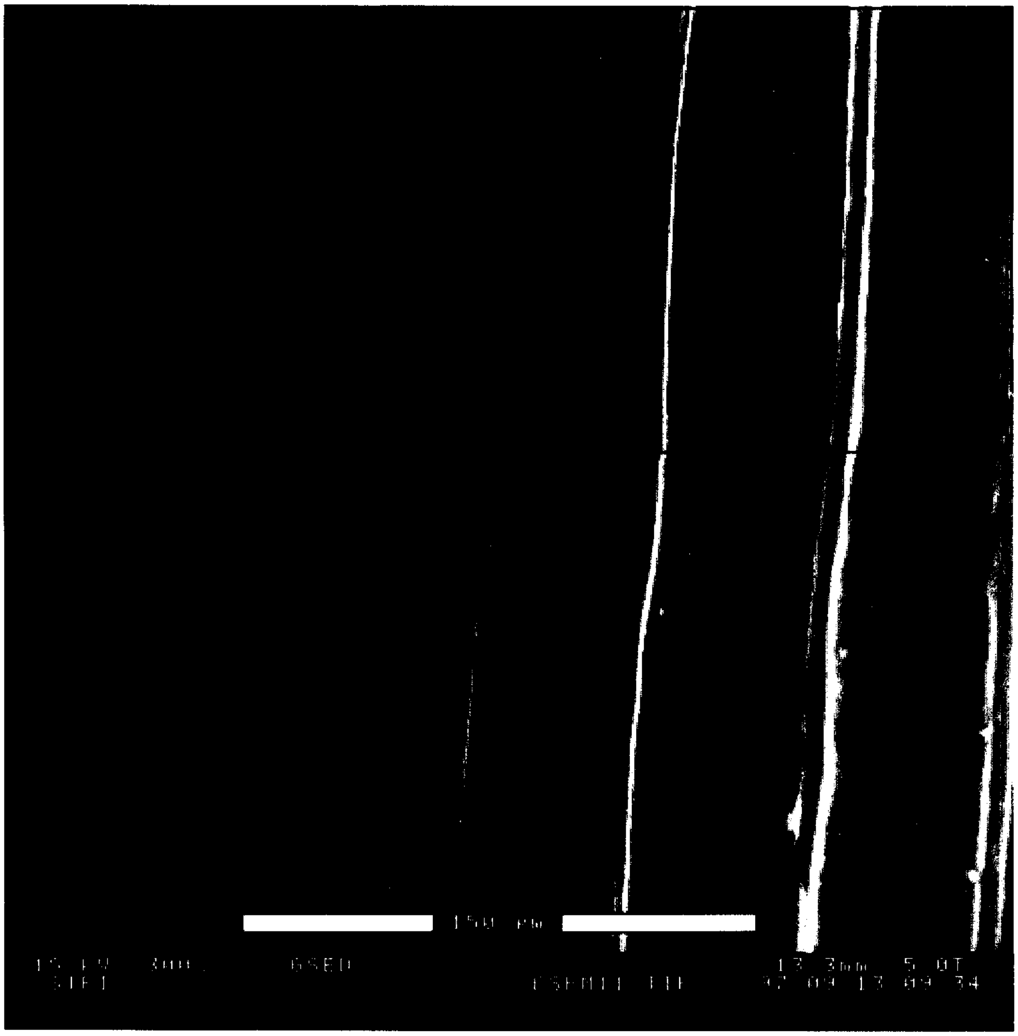

FIG. 1b. Micrographs of region A after 8.25 min of beam exposure.

cles. Images of position A in the dry state were recorded after each wetting and drying cycle. Additional regions of interest were selected, and images were recorded in order to identify changes.

c) *Five air-dried tension-loaded and moisture-cycled samples:* For each sample, an initial image of region A was recorded; the sample was then loaded and a second image acquired. The sample was then dried for 30 min in the chamber. The procedure that followed after drying the sample for 30 min, i.e., the selection of other regions, the wetting and drying cycles and the acquisition of images, were the same as for item b).

d) *Tensile loading and drying of four green samples:* Region A was selected and an initial image was recorded; the sample was loaded and the second image acquired, and the sample was then dried for 2 h. During the drying period, the beam was completely shut off. After 2 h of drying, images of region A were recorded. Additional regions were also selected for image acquisition for descriptive purposes.

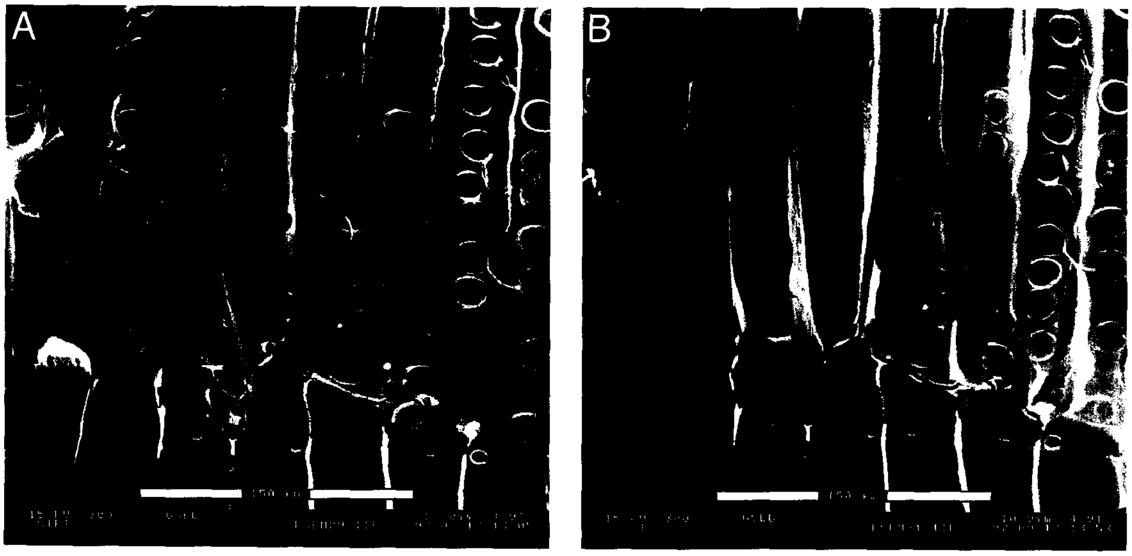

FIG. 2. Micrographs of region A after a) 4.0 and b) 7.0 min of beam exposure.

OBSERVATIONS

For each sample, images were recorded at several regions, but the analysis presented in this study was concentrated to region A. Micrographs of other regions were considered when a relative explanation became essential due to damage in region A. A total of twenty samples were tested and a number of micrographs were recorded, and the pattern of the damage (formation of cracks) observed was identical.

Unloaded and moisture-cycled green sample

During the recording of the initial green state image, region A was exposed to the electron beam for 2.1 min, but the region showed no damage. Nor did the second image recorded after the sample had been dried for a period of 30 min and exposed to the electron beam for a total of 5.1 min (Fig. 1a) reveal any noticeable damage. Damage started to appear on the image (Fig. 1b) taken after the first wetting/drying cycle and a 8.25-min beam exposure. After two wetting/drying cycles and a total of 15.55 min of beam exposure, region A exhibited more damaged areas.

Tensile-loaded and moisture-cycled green sample

While the initial and the second (Fig. 2a) images were recorded, region A was exposed to the electron beam for 4.0 min, but neither image revealed any noticeable damage. Nor did the image recorded after the sample had been dried for 30 min, exposing the region to an electron beam for a total of 5.5 min, exhibit any damage. An image that revealed some damaged pits and its surroundings was recorded after the first wetting/drying procedure and after beam exposure of the region for a total of 7.0 min (Fig. 2b).

Tension-loaded and moisture-cycled air-dried sample

The first undamaged image of region A was recorded after 3.5 min of beam exposure prior to loading and additional drying. The second image was recorded after loading and exposure of the region for a total of 5.0 min (Fig. 3a). These images showed no noticeable damage, but the image recorded after the first wetting/drying cycle and a total of 6.5 min of beam exposure showed some slightly damaged areas near the pits. Damaged areas can

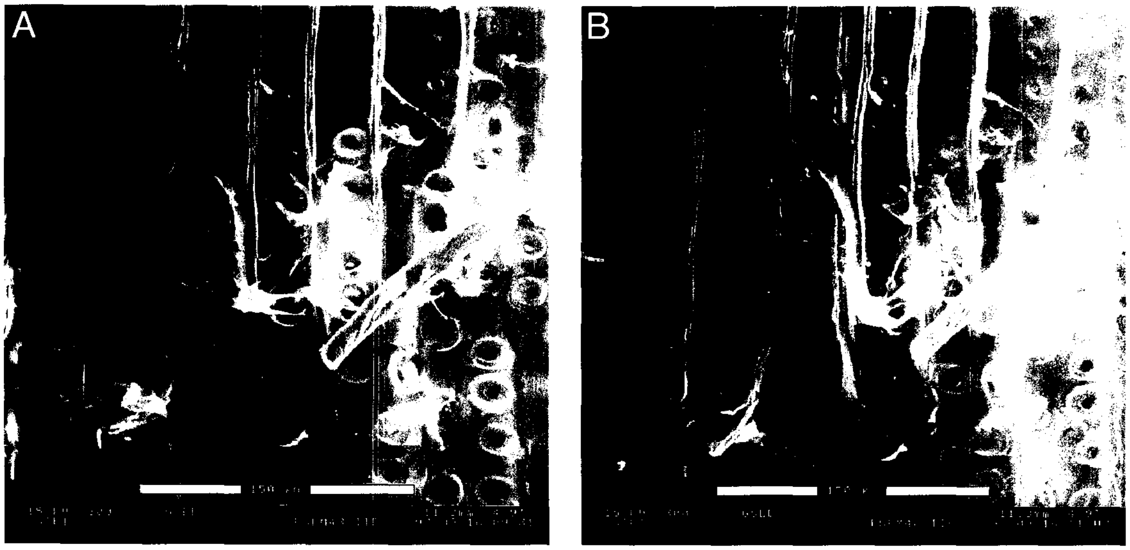

FIG. 3. Micrographs of region A after a) 5.0 min and b) 8.0 min of beam exposure.

clearly be observed on images recorded after the second wetting/drying cycle (Fig. 3b) and 8.0 min of beam exposure.

Tensile-loaded and dried green sample

To acquire the reference image, 4.5 min of beam exposure was required and the region showed no damage. The specimen was then dried for 2 h in the chamber while the electron beam was completely shut-off, i.e., 0 kV. Images were acquired to detect any damage or structural changes on the surface of the region of interest, but no noticeable structural change or damaged surface could be detected after the region of interest A had been exposed for a total of 9.5 min (Fig. 4).

FIG. 4. Micrograph after two hours of drying and 9.5 min of beam exposure.

Comparison sites

Before any conclusions were drawn about the structural change due to the development of damage, images of some new and randomly chosen regions on each sample were recorded. These regions were considered to be comparison sites for the changes noticed on the surface of region A on each sample. The maximum time required to expose a region to the electron beam during the recording of an image was 2 min. As shown in Fig. 5, none of the images of the newly selected regions revealed any noticeably damaged area.

DISCUSSION

It is recognized that recent improvements and the use of ESEM have made it possible

FIG. 5. Micrograph of a comparison site.

to investigate wood and wood fibers containing water in the form of vapor or liquid. However, to acquire an image, exposure of the sample to the electron beam cannot be completely avoided.

The micrographs presented in this study showed a clear difference in the occurrence of damage depending on whether or not the specimens had been exposed to the electron beam for a long time, i.e., a minimum of 6.5 min and a maximum of 8.25 min, and had been wetted and dried. The specimens that were tested with or without a constant applied load and had experienced two wetting/drying cycles showed distinct pit damage and damage to their surroundings. However, other sites used for comparative purposes showed no sign of cell-wall damage.

This investigation has revealed the development of damage to the pit and its surroundings during testing in the ESEM. Therefore, the study suggests that precautions must be taken in identifying the weakest links and in labeling the sources of failure initiation in wood cell-wall structure under direct ESEM studies.

CONCLUSIONS

The aim of this investigation was to study the effect of an electron beam, constant loading and repeated drying, and wetting on cell-wall damage, i.e., the creation of microcracks. The micrographs of both loaded and unloaded samples exposed to moisture cycles and an electron beam have revealed damage that in most cases runs through a pit and its surroundings. In contrast, a green sample tested under constant load during 2 h of drying without electron beam exposure, except during the period when images were acquired, showed no noticeable damage. These results indicate that the electron beam may be a major cause of damage initiation. Care should therefore be taken when explaining the observations made in ESEM studies.

REFERENCES

CÔTE, W. A., AND R. B. HANNA. 1983. Ultrastructural characteristics of wood fracture surfaces. Wood Fiber Sci. 15(2):135–163.

FEIST, W. C., AND D. N.-S. HON. 1983. Chemistry of weathering and protection. Pages 401–451 *in* Roger Rowell, ed. The chemistry of solid wood. The 185th Meeting of the American Chemical Society. March 20–25, Seattle, WA.

HOFFMEYER, P., AND R. B. HANNA. 1989. Electron beam damage during testing of wood in SEM. Wood Sci. Technol. 23:211–214.

JENKINS, L. M., AND A. M. DONALD. 1997. Use of the environmental scanning electron microscope for the observation of the swelling behaviour of cellulosic fiber. Scanning 19(2):92–97.

KIFETEW, G. 1996. Some aspects on the deformation behaviour of wood in relation to its structure. Doctoral thesis, KTH, Stockholm, Sweden, TRITA-TRÄ R-96-19.

———, F. THUVANDER, L. BERGLUND, AND H. LINDBERG. 1997. The effect of drying on wood fracture surface from specimen loaded in wet condition. Wood Sci. Technol. 32:83–94.

KYANKA, G. 1976. Fracture behavior of single fibers and paper sheets in the scanning electron microscope. Proc. 2nd Int. Cong. on Mech. Behavior of Materials. Pp. 1354–1357.

MOTT, L., S. M. SHALER, L. H. GROOM, AND B. H. LIANG. 1995. The tensile testing of individual wood fibers using environmental scanning electron microscopy and video image analysis. TAPPI 78(5):143–148.

SHALER, S. M., L. H. GROOM, AND L. MOTT. 1996. Microscopic analysis of wood fibers using environmental scanning electron microscopy and confocal microscopy. Pages 25–32 *in* Proc. Woodfiber-Plastic Composites Symp. Forest Prod. Soc., Madison, WI.

TURKULIN, H., AND J. SELL. 1997. Structural and fractographic study on weathered wood. An application of FE ESM microscopy to the "Thin strip" method. EMPA Bericht 115/36. Dübendorf, Switzerland.

ZINK, A. G., P. J. PELLICANE, AND C. E. SHULER. 1994. Ultrastructural analysis of softwood fracture surfaces. Wood Sci. Technol. 28:329–338.