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Characterizing the Surface Roughness of Thermomechanical Pulp Fibers with Atomic Force Microscopy[1)]

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Keywords

Atomic force microscopy (AFM)
Scanning electron microscopy (SEM)
Thermomechanical pulp (TMP)
Fiber surface morphology
Surface roughness
Refiner pressure
Fractals

Summary

Loblolly pine, separated into mature and juvenile portions, was refined at various pressures (4, 8 and 12 bar). Fiber surfaces were investigated using a Scanning Electron Microscope (SEM) and an Atomic Force Microscope (AFM). Refiner pressure had a significant effect on the fiber surfaces. SEM images showed an apparent increase in surface roughness with increased refiner pressure. This was shown quantitatively with data from the AFM that was analyzed using 5, 2.5 and 1.25 μm scan sizes. A scan size of 2.5 μm was found to be the most informative in terms of quantifying the effect of the different treatments on the two fiber types. The calculated surface roughness was greatest at 8 bar for both wood types. Juvenile fibers in general had higher surface roughness values than mature fibers. The results suggest that refining pressure may influence the failure mechanism of juvenile and mature wood differently.

Introduction

Wood composite materials are formed from fibers that have been generated either chemically or mechanically and are then brought back together to form a complex matrix. Matrix construction may be achieved by hydrogen bonding either between fibers as, for example, in paper production, or between the fibers and resin as occurs in the formation of medium density fiberboard. Due to the reliance on hydrogen bonding and secondary interactions, fiber surface chemistry and morphology is considered to have a direct effect on the quality of the bonding mechanism between adjacent fibers in the matrix and this ultimately determines product performance (Nissan and Sternstein 1964; Page 1990).

The analysis and design of wood fiber-based composites is complex due to the fact that the primary component is itself a bio-based composite. Wood cells are comprised of a thin primary wall and a three-layer secondary wall differing in thickness and varying in the chemical constituents cellulose, hemicellulose and lignin (Panshin and deZeeuw 1980). The cells are bound together by a lignin-rich middle lamella. Cellulose is present in the form of helically wound microfibrils encased in a matrix of lignin and coupled with hemicellulose. The angle and orientation of the microfibrils varies depending on the position in the cell wall (Harada and Côté, 1985; Abe *et al.* 1991; Megraw 1999).

There is much interest in the mechanisms involved in fiber separation during the mechanical breakdown of wood in the refining process. During the production of thermomechanical pulp (TMP), wood chips are subjected to extreme environmental conditions. The wood chips are softened at an elevated temperature and pressure in a saturated steam chamber, then forced at high pressure though a rapidly rotating disk to become defiberized. The pulp is then discharged at atmospheric pressure into a cyclone where the steam is removed, or blown through a blow pipe for minimal removal of steam from the system (Suchland and Woodson 1986). Changes in temperature, moisture and pressure, in conjunction with mechanical abrasion, affect the manner in which fibers become separated at the cellular level. This in turn may effect the surface structure of the fibers generated during refining.

Hypotheses have been developed to address what occurs during breakdown of wood chips during the refining process. Pearson (1983) believed that compression and expansion between the bars of the refiner plates together with excess steam cause cleavage at the S_2 layer. Sundholm (1993) proposed several steps including separation of the compound middle lamella to produce fines, followed by delamination and fibrillation of the fiber wall. Karnis (1993) however, suggested that the primary and S_1 layers were peeled away exposing the S_2 layer.

Goring (1971) showed that lignin becomes softened at elevated temperatures. Therefore, at high temperatures the middle lamella becomes a zone of weakness allowing for easy cleavage of adjacent fibers. Goring (1971) found that the temperature at which the plasticization of lignin occurred was determined by water content, the transition temperature being reduced with increased water content. This effect can be seen during refining where there is a reduction of power input required to fiberize wood chips at temperatures greater than 150 °C. As such, refining at varying temperatures and pressures may not only affect how fibers become separated, and which cell layer is the primary sur-

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face exposed, but may also have a significant effect on the chemical groups that are available for fiber bonding. Studying the effect of refiner pressure on fiber surface properties may increase our understanding of the mechanisms involved in fiber breakdown and the subsequent bonding mechanisms involved in the formation of composite materials.

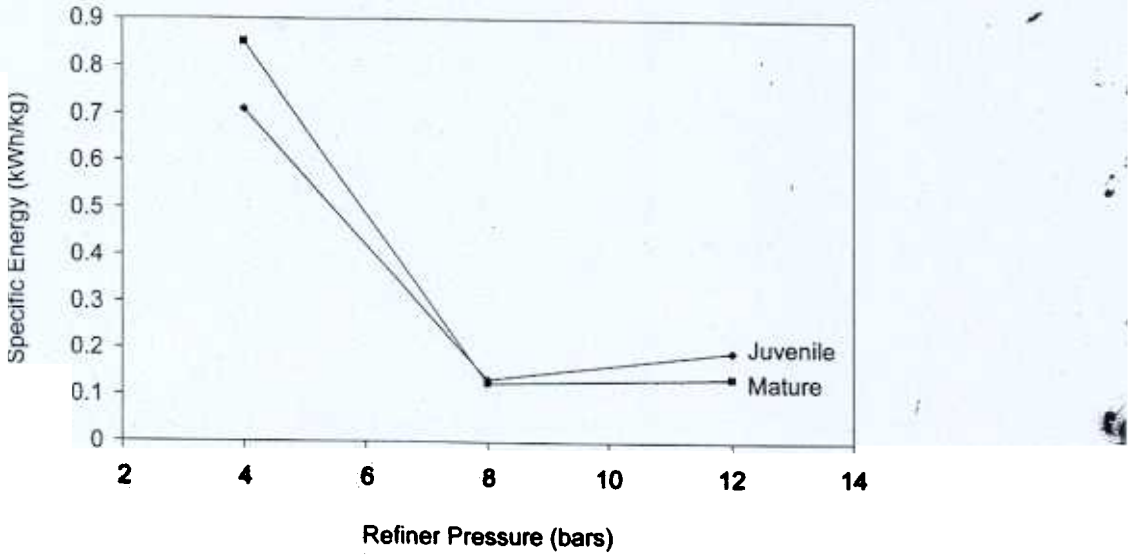

Fig. 1. Energy consumption during refining at 4, 8, and 12 bar.

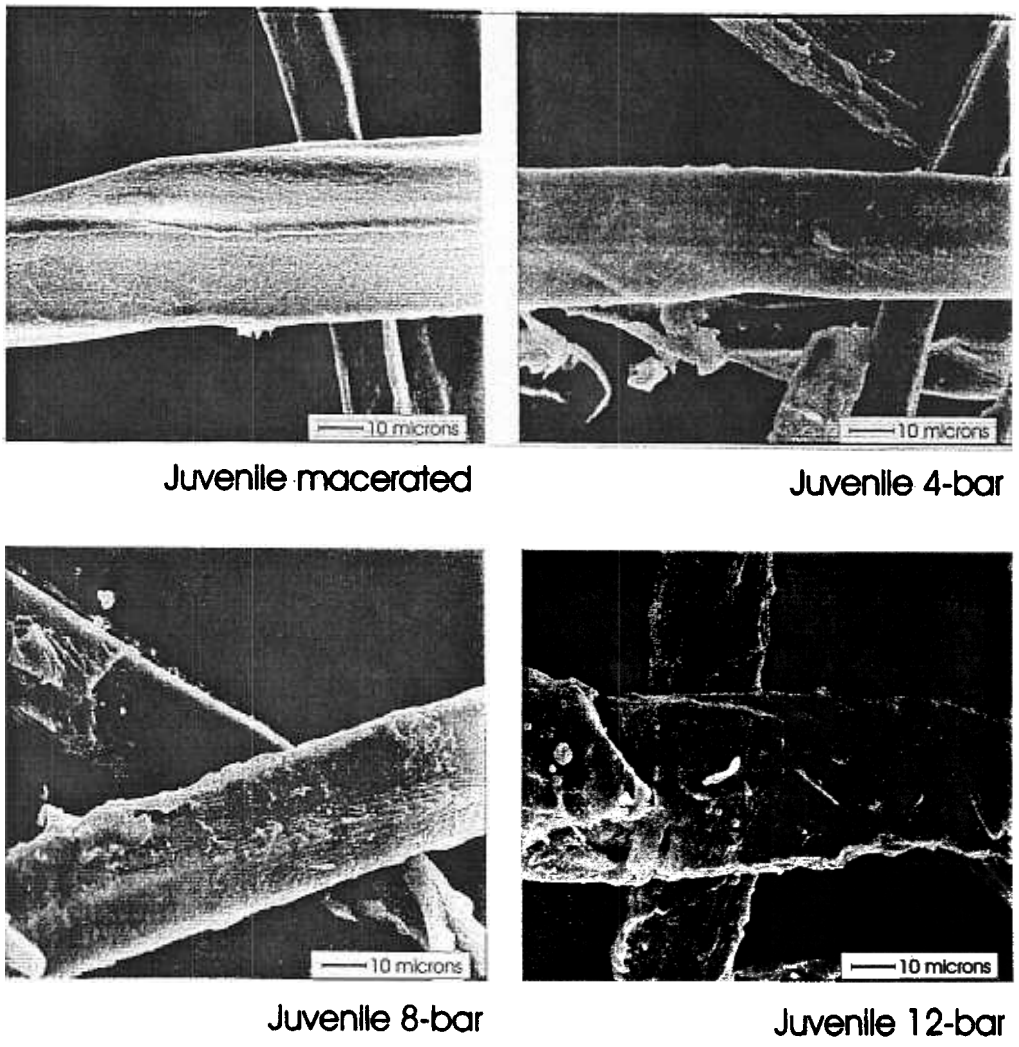

Fig. 2. SEM micrographs of juvenile fibers showing surface morphology after maceration and pressure refined at 4, 8, and 12 bar.

Detailed investigations of fiber surfaces are now possible using the atomic force microscope (AFM). The principles of the AFM are covered elsewhere in the literature (Beland 1996) and will not be detailed in this paper. The AFM enables surface topography to be recorded in three dimensions by detecting the vertical deflection of an oscillating tip that rasters across the sample surface. Quantitative surface measurements such as roughness can be calculated from height data, and direct comparisons can be made between samples. The AFM has been used to study surface topography of different wood fibers (Hanley and Gray 1994; Börås and Gatenholm 1999). However, little work has made use of quantitative data that is recorded during image capture. Pesacreta (1997) has used this technique extensively to quantify the effect of chemical treatments on cell wall roughness in cotton. In this work we study the effect of different refining treatments on fiber surfaces qualitatively with an SEM and quantitatively with an AFM.

Materials and Methods

Loblolly pine chips, separated into mature (growth rings 25 and beyond) and juvenile (pith to growth ring 10) portions, were refined at three pressures (4, 8, and 12 bar) in a continuous, pressurized, single-disc refiner at the BioComposites Centre, University of Wales. A 30 cm plate was used with fiber retention time of approximately 4 minutes. The plate gaps were set at 80, 250, and 50 µm for the 4, 8, and 12 bar pressures. Fibers were dried in a flash drier 80 meters in length to a moisture content of approximately 12 percent. The surface morphology of the treated fibers was investigated using a JEOL scanning electron microscope (SEM) and a Nanoscope IIIa AFM (Digital Instruments, Santa Barbara, Calif. USA) mounted on a pneumatic isolation table with an acoustic hood. Fibers were mounted on stubs and sputter coated with approximately 15-nanometer thick layer of gold and observed at 15 to 20 KV at a distance of 25 to 33 mm.

Fiber preparation for the AFM was minimal. Individual fibers were carefully attached to AFM stubs using carbon tape. Three 5 µm scans, located in the middle and quarterpoints of 10 individual fibers, were collected for each treatment. Unrefined fibers were prepared by maceration in a solution of acetic acid/hydrogen peroxide for a period of approximately 48 hours, thus providing a baseline for comparison. Fibers were orientated with the long axis parallel to the raster scan direction. All AFM micrographs were taken at a resolution of 512 × 512 pixels. Images were obtained in intermittent-contact mode, also referred to as Tapping mode ™, in air. The probes were standard tapping-mode tips comprised of etched silicon. The probes had a nominal tip radius of curvature of 5 to 20 nanometers and a resonant frequency of about 300 kHz. A scan

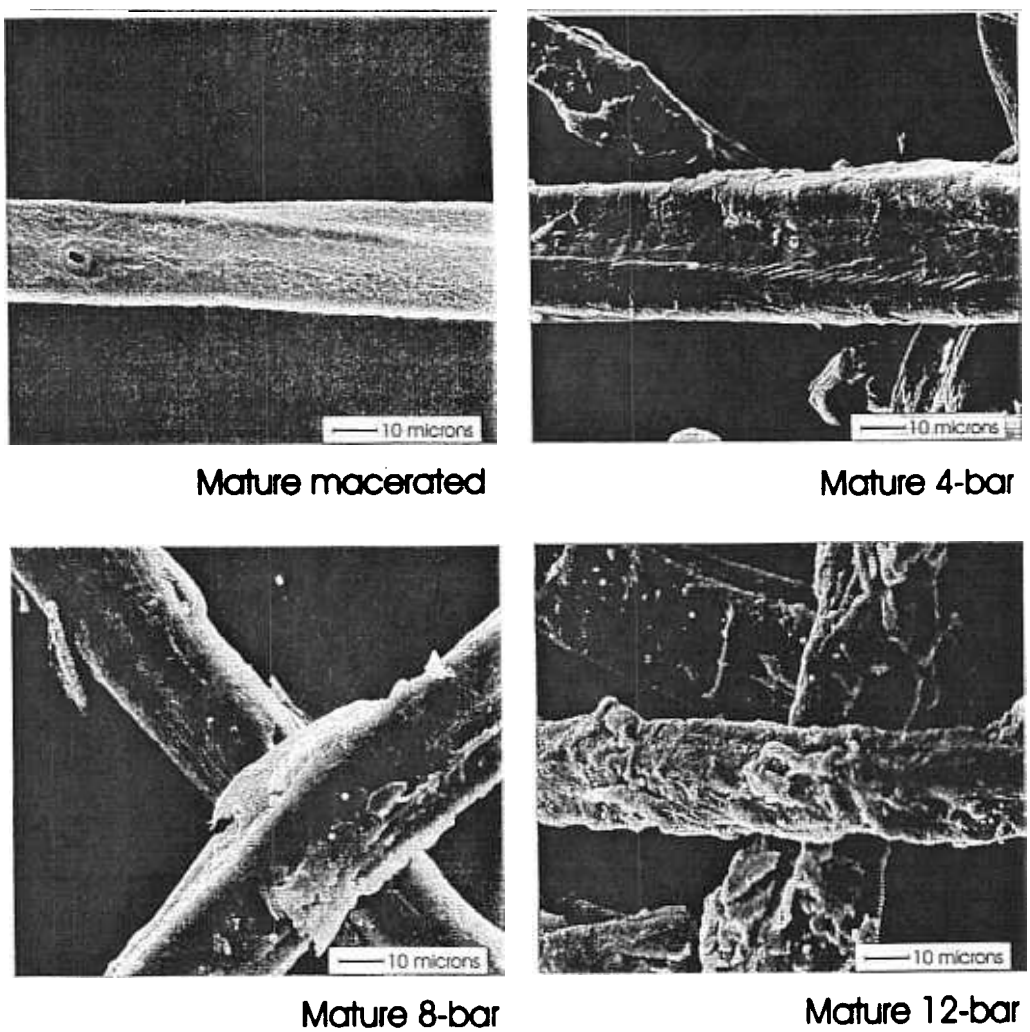

Fig. 3. SEM micrographs of mature fibers showing surface morphology after maceration and pressure refined at 4, 8, and 12 bar.

rate of 1 Hz was employed to generate a total of 240 scans. Three data channels, height, amplitude and phase shift were monitored during image acquisition. From each image a representative area of 2.5 by 2.5 µm and 1.25 by 1.25 µm were selected using the zoom feature and used for further analysis. A flatten order of 1 was applied to the height data, thus correcting for any 2-dimensional deviations about a flat plane while maintaining the integrity of individual features. The images were evaluated and statistically analyzed using the height data to quantify surface roughness (RMS), total power (TP) and fractal dimensions (FRAC).

Results and Discussion

Fiber throughput for the various fiber types was approximately 25, 35, and 40 kg/hour for the 4, 8, and 12 bar settings, respectively. The various refining levels resulted in large shifts in both energy consumption during refining and resulting fiber appearance. Figure 1 shows the energy consumption during refining drops by 80 percent from 4 bar to 8 bar. This drop is indicative of the glass transition temperature of lignin residing in this range. Increasing the pressure to 12 bar increased refiner energy slightly from 0.13 to 0.16 kWh/kg.

Visual differences between the different pressure treatments were immediately observed. There was a distinct progressive darkening in fiber color with increased refining pressure. Fibers refined at 4 bar were pale tan while 12 bar fibers were a deep rusty brown. There were no obvious differences between mature and juvenile fibers refined at the same pressure.

SEM observations

SEM images show that, in general, fibers refined at 4 bar have a much smoother surface than those refined at 12 bar. The 12 bar fibers have surfaces that are fragmented, a result of intercellular delaminations. This is more noticeable in the juvenile than the mature fibers. There appears to be a progressive increase in the surface roughness with increased refiner pressure; juvenile fibers are shown in Figure 2 with mature fibers represented in Figure 3. The surface of the juvenile fibers refined at 4 bar looks to be much smoother than the mature fibers. Delamination is evident in some places on the fiber surface for both maturity fiber types refined at 8 bar. Surface layers have peeled and flaked off, as described by Karnis (1993). Surfaces for both juvenile and mature fibers refined at 12 bar look extremely rough, with large globular deposits and a broken crust-like appearance. The macerated fibers have an extremely clean surface, interrupted mainly by natural features such as pits. It can be seen from all the images of the pressure-refined fibers that

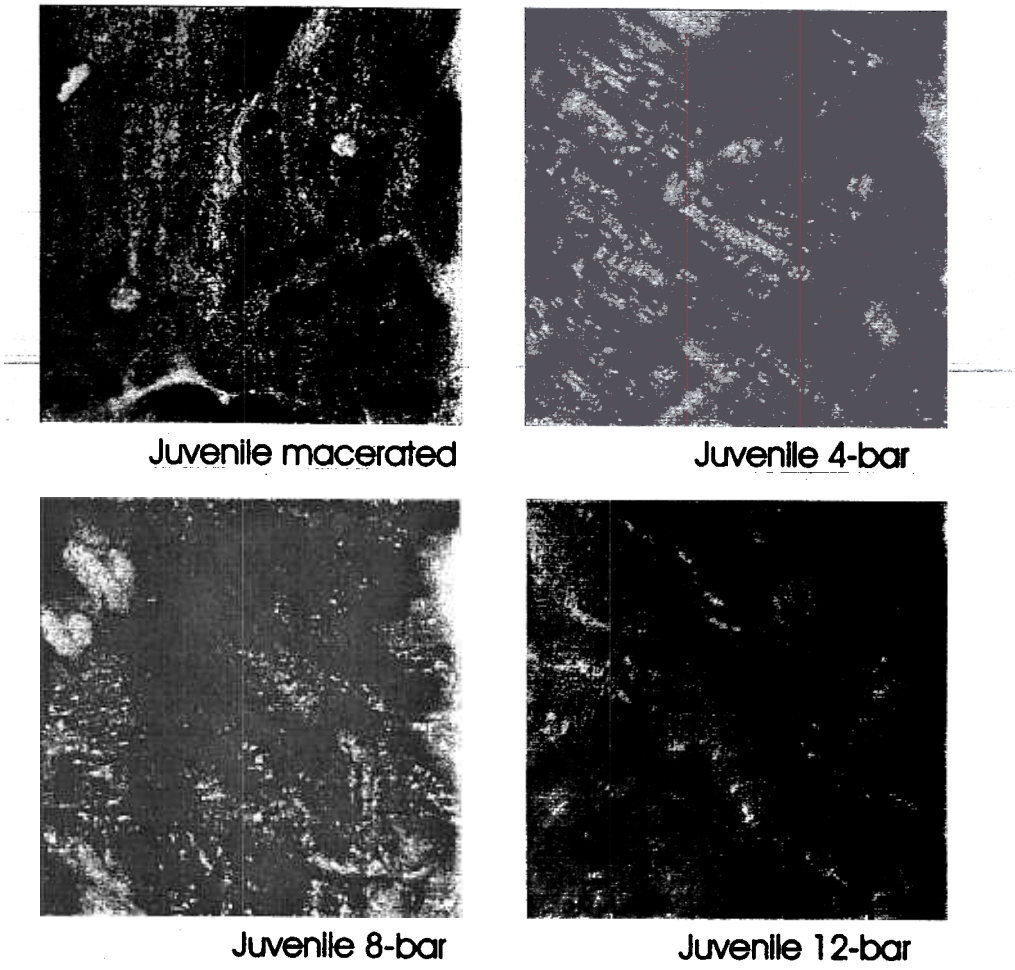

Fig. 4. Five-micron AFM scans of juvenile fibers showing surface morphology of macerated and pressure refined fibers at 4, 8, and 12 bar.

surface morphology varies greatly along the fiber axis and between fibers.

AFM observations

The AFM images for juvenile and mature fibers are shown in Figures 4 and 5, respectively. Although the AFM images were difficult to interpret visually at such a high magnification, an overall trend could be seen. Macerated fiber surfaces looked extremely different from the refined fibers. Microfibrils were evident over the entire surface for both the juvenile and mature macerated fibers. The microfibrils were oriented in a rather random pattern indicating that the primary cell wall layer had been exposed. The microfibril angle for the fibers refined at 4 bar was shown to be approximately 30 degrees to the fiber axis suggesting that the S_2 layer was predominant on the fiber surface. This is more evident for the juvenile fibers than the mature. Microfibril angle was difficult to see for the 8 and 12 bar pressure treatments. The surfaces at these pressures appeared to be covered with a smooth layer that had possibly been either smeared or re-deposited during refining at the higher pressures. The temperatures corresponding to the refining pressures from steam tables are 144 °C, 170 °C and 188 °C for 4, 8, and 12 bar pressures (Weast 1975). The smooth layer observed could be lignin that had been softened and redeposited as the glass transition temperature of native lignin (148.9°) is between 4 bar and 8 bar. The microfibrillar structure may also be obscured by low molecular weight extractives that have migrated from the interior wall.

Figure 6 shows how the 2.5 and 1.25 µm scans were obtained using the zoom function on the AFM. Statistical calculations using numbers obtained from off-line analysis using height data, showed that there were differences in the surface roughness (RMS), total power (TP) and fractal dimensions (FRAC).

RMS is the root mean squared of standard deviation of the height (z) data and gives an overall indication of surface roughness. TP is roughness amplitude squared and defines which surface features have the greatest influence on the surface topography. FRAC is the log of the dimensions of the entire surface of each scan versus the scan size; it defines the total surface area, the larger the fractal number the more complex the surface.

Figures 7 and 8 show the RMS value frequency distribution of the juvenile and mature fibers, respectively, for the different refiner pressures at a scan size of 5 µm scans. There are a wide range of RMS values for fibers within the same treatment. Even for the same fiber, RMS values were found to vary considerably. This was to be expected as the

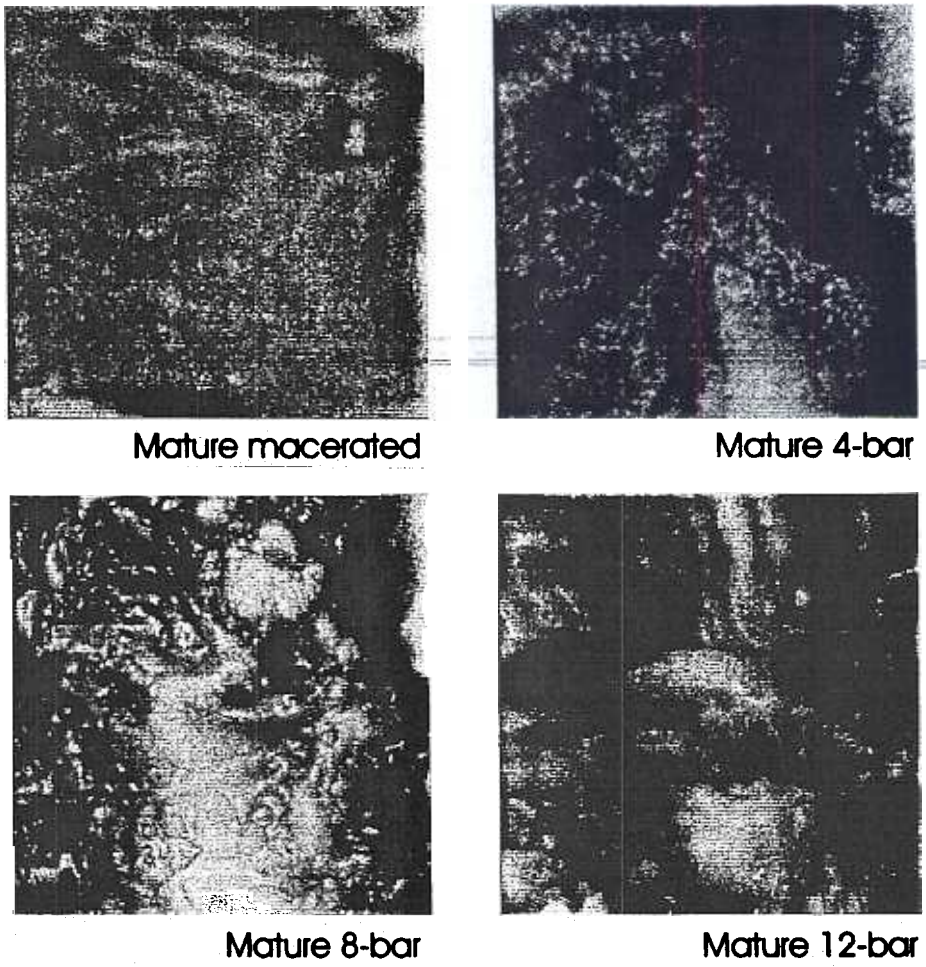

Fig. 5. Five-micron AFM scans of mature fibers showing surface morphology of macerated and pressure refined fibers at 4, 8, and 12 bar.

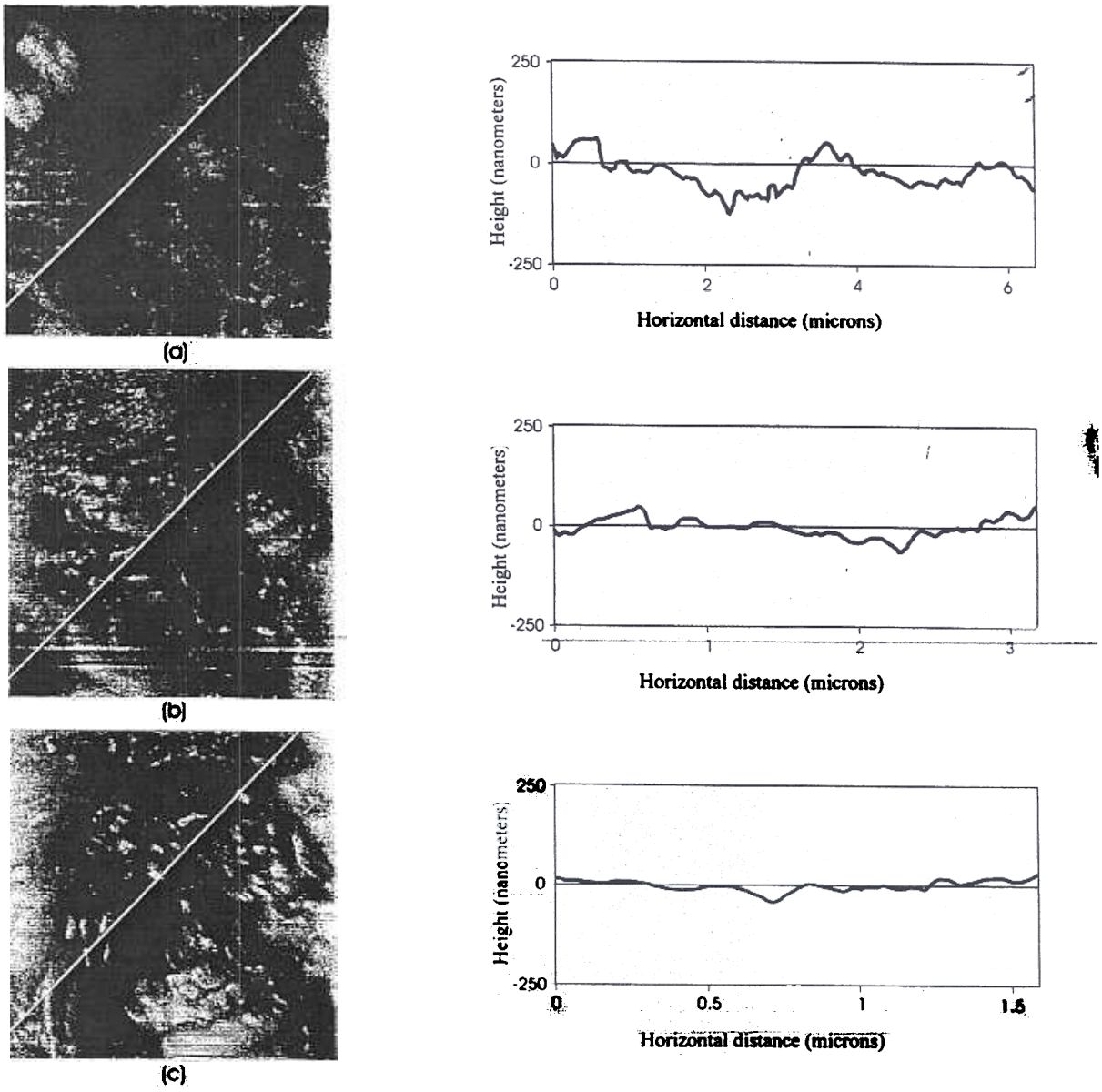

Fig. 6. Surface plot of juvenile fiber refined at 8 bar showing (a) 5 (b) 2.5 and (c) 1.25 μm scan size (zoom location shown by white box), with corresponding surface line trace (position shown by diagonal white line).

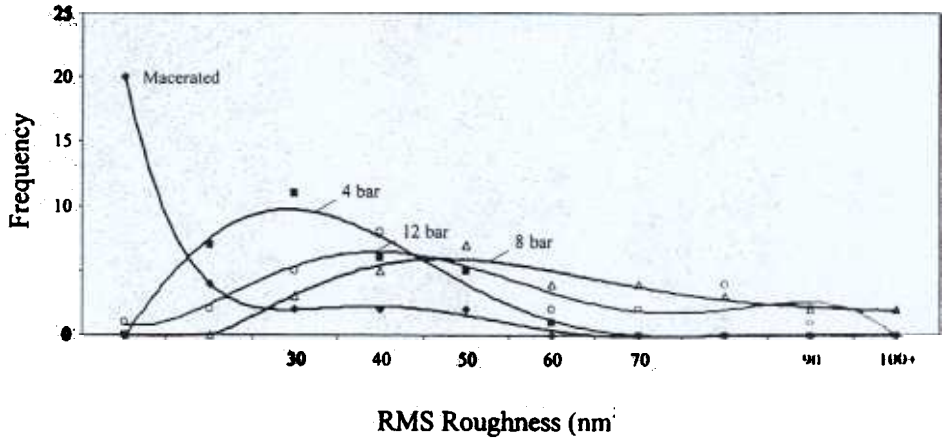

Fig. 7. Frequency distribution of juvenile fiber roughness macerated and refined at 4, 8, and 12 bar.

SEM images showed that the surface is not homogeneous. The variation is reflected in the standard deviation of the mean RMS in Table 1. Analysis of variance of the full RMS data set showed that refining pressure does have a significant effect on the surface roughness ($p = 0.0001$), this is also affected by the maturity of the fibers ($p = 0.0499$).

Using Fisher's LSD tests run on the analysis of variance (anova), each parameter RMS, TP and FRAC produce values that enable differentiation between the different treatments to a certain extent (Table 2). RMS values prove to be the most useful. Fibers refined at 4 and 8 bar have significantly different surface roughness to each other for the 5 µm scans. This is reflected in the frequency data in the respective juvenile and mature fibers in Figures 7 and 8 where the 4 bar fibers have a greater frequency of small RMS values while 8 bar fibers have a larger proportion of high RMS values. This trend is also found when comparing 4 bar with 12 bar treatments for juvenile but not mature fibers. RMS values for 8 bar fibers are not significantly different to 12 bar fibers for either juvenile or mature fibers. This again can be seen from the frequency distribution curves in Figures 7 and 8. Evaluation of the data using anova for the defined 2.5 µm

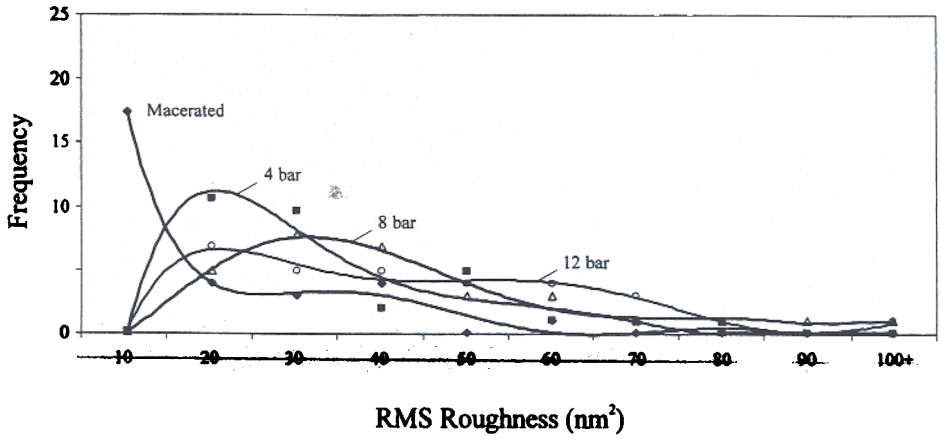

Fig. 8. Frequency distribution of mature fiber roughness macerated and refined at 4, 8, and 12 bar.

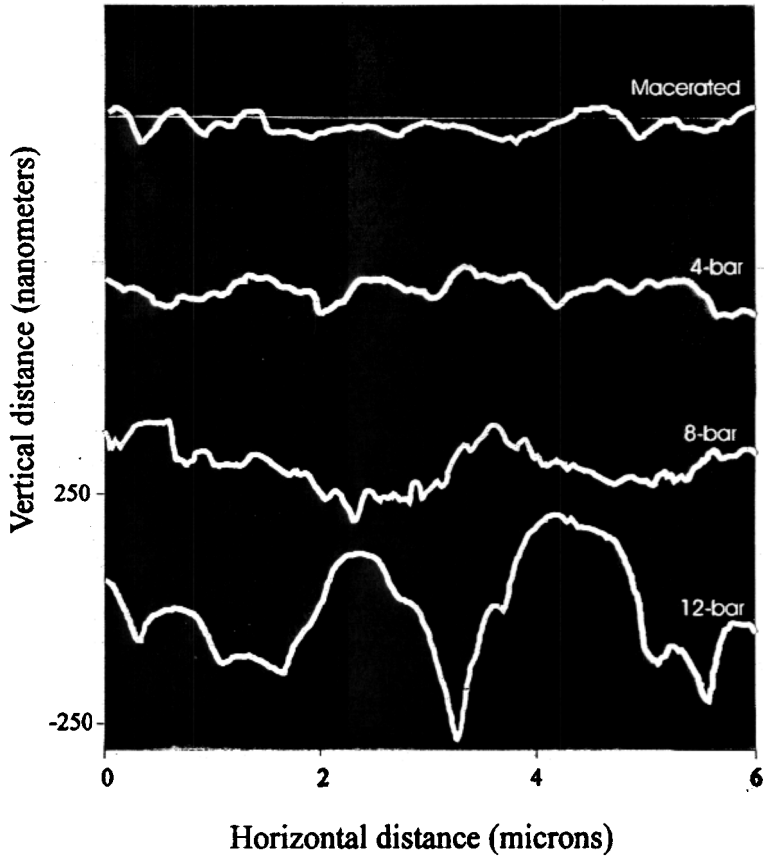

Fig. 9. Surface line traces for juvenile fibers shown in Figure 4 taken perpendicular to the microfibril angle (approx 60° to fiber axis).

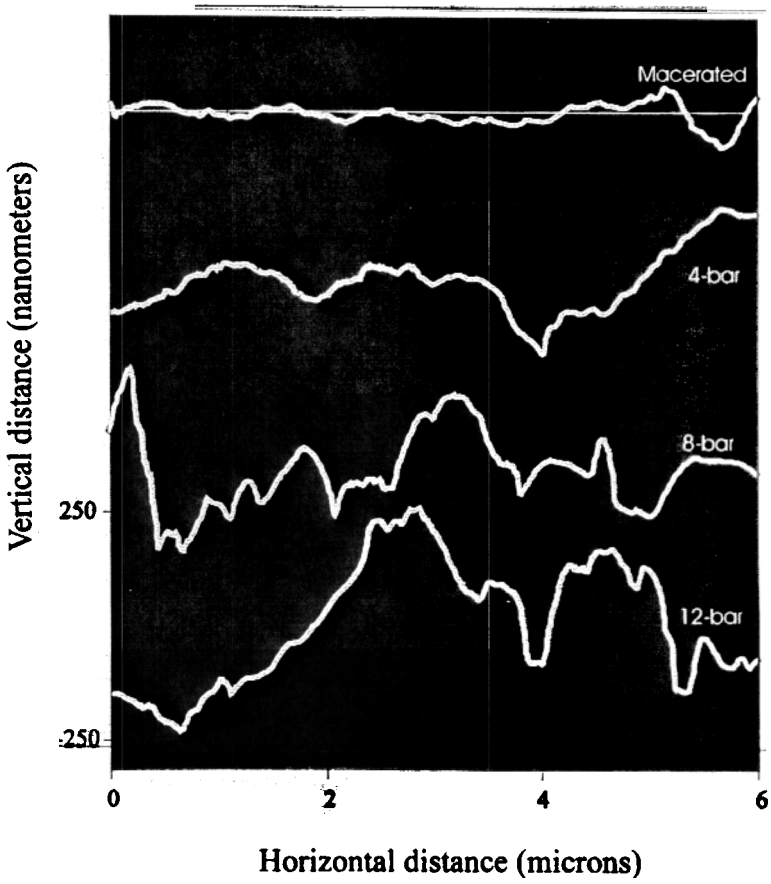

Fig. 10. Surface line traces for mature fibers shown in Figure 5 taken perpendicular to the microfibril angle (approx 60° to fiber axis).

Table 1. Mean and standard deviation (std) of RMS values for 5, 2.5 and 1.25 μm scans for juvenile (J) and mature (M) fibers macerated (mac) or refined at 4, 8, or 12 bar. Thirty images (3 scans from 10 fibers) were taken for each treatment

	J-mac	J-4 bar	J-8 bar	J-12 bar	M-mac	M-4 bar	M-8 bar	M-12 bar
5 μm scan mean	23.7	73.1	127.0	116.8	25.1	68.7	111.1	87.8
std	25.1	32.5	55.6	60.9	22.3	31.2	81.5	57.4
2.5 μm scan mean	12.4	28.9	55.1	42.8	13.7	27.9	38.7	38.3
std	12.4	10.5	22.4	20.5	12.8	13.3	22.8	20.4
1.25 μm scan mean	7.2	12.5	22.9	18.7	6.3	14.0	18.3	16.7
std	7.2	5.5	10.3	8.2	5.0	8.4	10.5	9.5

region of the 5 μm scan show significant differences for all the treatments ($p = 0.0001$) and fiber maturity ($p = 0.0106$). Fisher's LSD tests (Table 2) confirmed that all treatments for both juvenile and mature fibers were significantly different except for mature fibers refined at 8 or 12 bar. The differences for fiber maturity at a scan size of 1 μm can no longer be distinguished ($p = 0.1957$). However, the effects of the treatments are still significant ($p = 0.0001$). Table 2 shows that although the effect on juvenile fibers is significant for the 4 and 8 bar treatments, no differences were apparent for mature fibers. The results suggest that 2.5 μm scans reveal the most useful information enabling differentiation between the refining treatments.

Total power and fractals were not such useful parameters for analyzing the data. Total power was more effective in distinguishing differences between the juvenile fibers than the mature fibers especially for the smaller scan size (Table 2). The surface features of the juvenile fibers for all treatments were distinctly different at 2.5 μm and 1.25 μm, whereas no statistical differences were found for the mature fibers. Fractals showed differences only for juvenile fibers between the 4 and 8 bar pressure treatments for all the scan sizes.

Surface profiles were examined for each of the AFM images in Figures 4 and 5 by conducting a surface line trace perpendicular to the microfibril angle, approximately 60

Table 2. Treatment comparisons using Fisher's LSD test run on analysis of variance of surface roughness (RMS), total power (TP) and fractal (FRAC) data generated from the 5, 2.5 and 1.25 µm scans. Mature and juvenile fibers were analyzed separately. Asterisks signify treatments that have significant p-values ($p < 0.05$). NS = not significant

		RMS		TP		FRAC	
Treatment Comparisons		Juvenile	Mature	Juvenile	Mature	Juvenile	Mature
5 µm							
4 bar	8 bar	*	*	*	*	*	NS
4 bar	12 bar	*	NS	*	NS	NS	NS
8 bar	12 bar	NS	NS	NS	NS	NS	NS
2.5 µm							
4 bar	8 bar		*		NS	*	NS
4 bar	12 bar		*		NS	NS	NS
8 bar	12 bar		NS		NS	NS	NS
1.25 µm							
4 bar	8 bar	*	NS		NS	*	NS
4 bar	12 bar	*	NS		NS	NS	NS
8 bar	12 bar	NS	NS		NS	NS	NS

degrees to the fiber axis, and are shown in Figures 9 and 10, respectively. It is possible to see from the surface profiles the differences in surface topography of the area scanned. Macerated fibers have a relatively flat surface whilst the treated fibers get progressively more undulating. The change in surface roughness increases up to refiner pressures of 8 bar for both fiber types; the frequency of oscillations increasing with increased pressure. The surfaces of fibers refined at 12 bar have a much larger variation in height, but there are fewer surface oscillations. Although evident in mature fibers, these surface features are especially notable for the juvenile fibers. This was to be expected as the SEM images and AFM 5 micron scans showed that the surfaces of fibers refined at 12 bar looked smooth but bulbous. Surface line traces are also shown in Figure 6 for a 5 µm scan of juvenile 8 bar fibers and the subsequent 2.5 and 1.25 µm images obtained using the zoom function. It can be seen that with increased magnification the surface trace becomes flatter. This may explain the more statistically significant results for the 2.5 µm scan size for roughness. Surface features below 2.5 µm may no longer be small enough to be recorded.

General discussion

The combination of the two microscopy methods were found to be complementary. Although the SEM requires that the sample is desiccated and coated with gold, it allows groups of fibers to be seen in detail and a larger surface area can be observed. Images obtained from the AFM were difficult to interpret visually and are, for practical reasons, restricted to individual fibers and scan sizes of approximately 25 µm or less. SEM images therefore gave an indication of the overall effect of refining on the fiber surface, whilst the AFM allows for quantitative measurements of the fiber surface at a much higher magnification. SEM images show that the surface can vary greatly along the length of the fiber. This would account for the high variability found for the quantitative measurements. SEM images show fibers refined at 12 bar were very fragmented with lots of breaks and lumps on the surface, whereas the 8 bar fibers showed mainly delamination.

One of the limitations of the AFM is that the maximum vertical deflection of the cantilever is 6 µm. Thus, some areas randomly chosen for AFM analysis had to be avoided due to excessively large features. Debris on the fiber surfaces also proved to be problematic occasionally due to vibrational interruptions of the cantilever. Thus, areas with loose debris also had to be avoided. Also if any loose material causes vibrations, a bad signal is obtained, so areas with delaminated debris could not be sampled. This is probably why the results showed that 8 bar fibers were rougher than 12 bar fibers.

Conclusion

The results from this study show that refining at different pressures has a significant effect on the surface morphology of wood fibers. Raising the pressure, in general, was found to increase the surface roughness. Most of the surface changes occur at refiner pressures between 4 and 8 bar. Fibers could be statistically distiguished best scanned at 5 µm and analyzed at 2.5 µm. Further study is now required to see how the increased roughness affects the bonding strength in structural fiberboards. A subsequent study is currently underway utilizing the phase images of the 5 µm scans in an attempt to discover the usefulness of these data as indicators of the chemical groups that are present on the surface of the treated fibers.

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