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IMAGING FIBRE DEFORMATIONS

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ABSTRACT

A new experimental technique is presented that allows the direct observation of fibre deformation during wet pressing. Pulp fibres were wet pressed onto a glass slide and the region where two fibres crossed was examined microscopically through the glass. While the underlying fibre was in contact with the glass slide down its length, the overlying fibre must span from the top of the fibre to the glass slide. The geometry of the intersection is controlled by both the local conformability of the overlying fibre and the deformability of the underlying fibre. It is not primarily controlled by the longitudinal flexibility of a fibre. The method provides new opportunities to investigate the effect of mechanical and chemical treatment on the papermaking properties of pulp fibres in both the dry and wet state.

INTRODUCTION

Fibre flexibility [1], conformability [2], compactability[3], collapsibility, pliability, and deformability are all words that have been used to describe properties which are linked to the ability of a paper sheet to consolidate and form a well bonded network. Fibre flexibility has become the predominant term used to describe the ability of pulp fibres to deform over one another. Many researchers have described methods to quantify fibre flexibility (e.g. [2, 4–8]). The most relevant and straightforward method was developed by Steadman

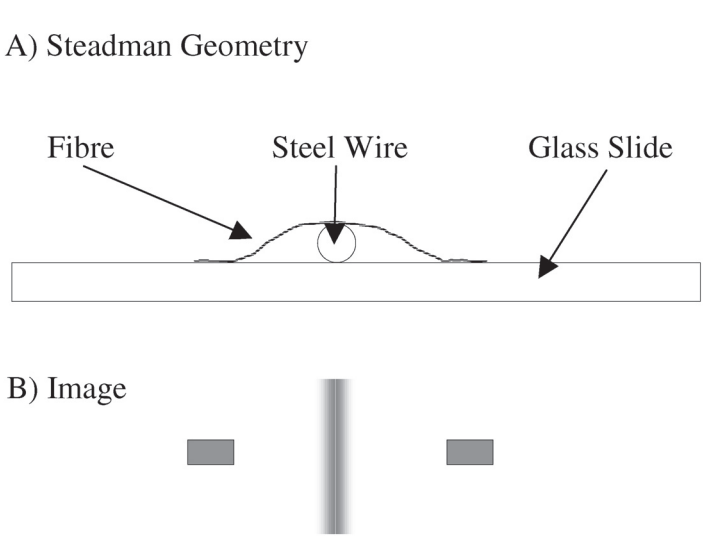

Figure 1 A schematic of the Steadman's geometry (A) and an illustration of an acquired image (B) [6].

and Luner [6]. In this method fibres are pressed and dried across a 25 μm diameter stainless steel wire wound around a glass slide. Figure 1 shows the geometry of the Steadman system (A) and the acquired image (B). The region of optical contact between the fibre and the glass slide is observed, and it is deduced that the closer the optical contact regions are to the wire the more flexible is the fibre. The length of the unbonded span is taken as a measure of flexibility.

One drawback of the Steadman and Luner method is that in a paper sheet the fibre deforms over a much smaller stepheight than 25 μm. While 25 μm is the approximate diameter of an uncollapsed pulp fibre, fibres in paper are always collapsed either partially or completely so that the step height is generally in the range 1–10 μm. As a consequence, the span over which the fibre is unbonded in actual paper should be much lower than in the Steadman and Luner experiments. It likely is on the order of a fraction of a fibre width (35 μm) rather than the 100 μm or so observed by Steadman and Luner [6].

It is more relevant to measure the deformation of a fibre when pressed against a glass slide crossing a fibre similar to itself rather than a 25 μm wire. This would simulate more closely the structure in a paper sheet. The unbonded span length under these conditions will be much lower than in Steadman's method and is more typical of the value that would occur in a

paper sheet. Thus any measurement of fibre flexibility by this technique is more relevant to the fibre deformations that occur in actual paper. Because of the higher demands on magnification, resolution and contrast, the method has been modified so as to optimize the optical conditions.

METHODS

Imaging fibre intersections

The research utilized a Leica DM-IRM inverted, reflected light microscope equipped with a Hamamatsu ORCA-ER digital camera and a 50 watt metal halide lamp. Samples were prepared by draining a dilute suspension of refined pulp fibres onto a piece of filter paper. The fibres were then wet pressed in a standard handsheet mold onto four glass slides for two minutes at 50 pounds per square inch (see Figure 2). The slides were allowed to dry and were kept under TAPPI Standard Conditions before and during imaging.

In order to acquire high contrast and high resolution images of the optical contact regions, several optical modifications were made. The first area of concern was the choice of an objective lens. When long spans are observed (e.g. with the Steadman and Luner method), low magnification objectives are suitable. However, with spans on the order of a few microns a high magnification objective is required. Unfortunately, objective lenses of high

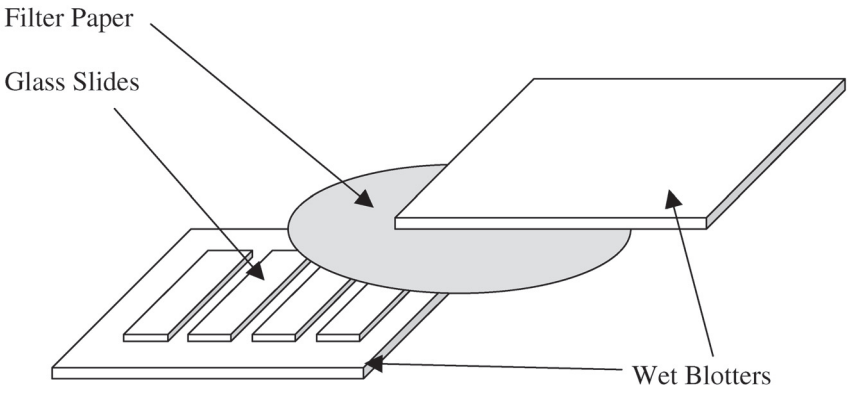

Figure 2 An illustration of the wet pressing setup.

numerical aperture suffer from spherical aberration when imaging though a glass slide. The off-axis light rays are focused in a different plane than the axial light rays. Modern objectives can be corrected for this spherical aberration via a correction collar, even when viewing through the thickness of a glass slide. A Leica 40X objective with such a correction collar was used for this research.

The second area of concern was the low contrast in the acquired image because of light arising from regions in the specimen that do not contribute to a useful image. First, light is reflected from the bottom of the glass slide. This light is of similar intensity as the actual image leading to low contrast. The reflection can be eliminated by utilizing a quarter wave plate fashioned from a split sheet of mica. The mica sheet was brought into optical contact with the glass slide by applying microscopy immersion oil to the underside of the glass slide (see Figure 3). The specimen was then illuminated and viewed in crossed polars. The optical axis of the mica is set at 45 degrees to the plane of polarization. The polarized light reflected by the bottom of the glass slide is eliminated by the crossed polars. The light rays forming the image first pass through the mica sheet becoming circularly polarized. After reflection from the fibre-glass interface, they pass back through the mica sheet and become plane polarized in the direction of the analyzer. This technique only permits

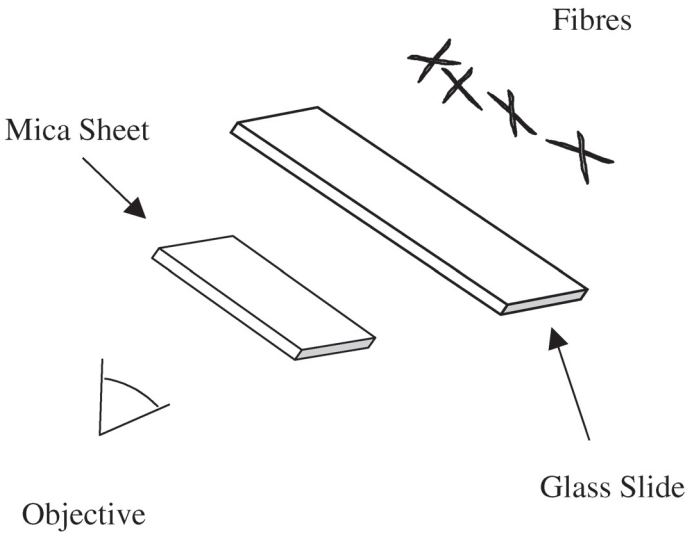

Figure 3 The imaging setup.

the desired reflected light (that from the fibre-glass interface) to reach the objective lens.

The final modification was to the fibres themselves. Pulp fibres transmit light that is in part reflected at their top surface and returned to the objective lens, thus reducing the image contrast. This source of unwanted light can be eliminated by dyeing the fibres before application to a glass slide.

Observing interference fringes

By incorporating these methods, high resolution images can be obtained. The areas of bonding between the fibres and the glass are dark compared with the bright regions of non-contact (see Figure 6). It has also been found that if a narrow band filter ($\lambda = 547 \pm 10$ nm) is incorporated, interference fringes can be observed. Figure 4 illustrates how the interference fringes are formed. Light at A passes through the glass slide and is absorbed by the dyed fibre if it is in optical contact with the glass slide; therefore, the intensity of the reflected light is zero. Light at B is reflected at the top glass-air interface and again at the fibre-air interface leading to an interference fringe. The local height of the air wedge, H, at each black fringe is given by Equation (1):

$$H = n\frac{\lambda}{2} \tag{1}$$

Where n is the order of the black fringe and λ is the wavelength of light.

Analysis of the fringe pattern thus allows an extremely accurate measurement of the height of the lower surface of the fibre in the freespan. These data have been used to determine the shape of the lower surface of the

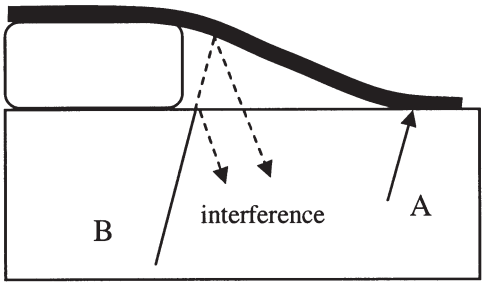

Figure 4 Interference fringe formation.

crossing fibre in the freespan region as well as the stepheight and the total freespan. Figure 5 illustrates the geometry of a fibre crossing. The stepheight (H) is defined as the height of the lower surface of the crossing fibre as it leaves underlying fibre. The freespan (F) is defined as the length of the fibre not in optical contact with the underlying fibre or the glass slide.

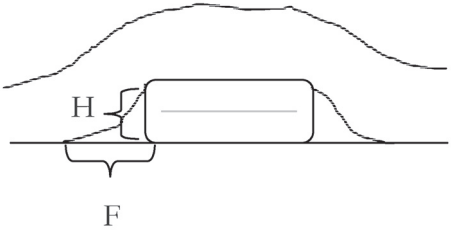

Figure 5 Stepheight (H) and freespan (F) in a fibre crossing.

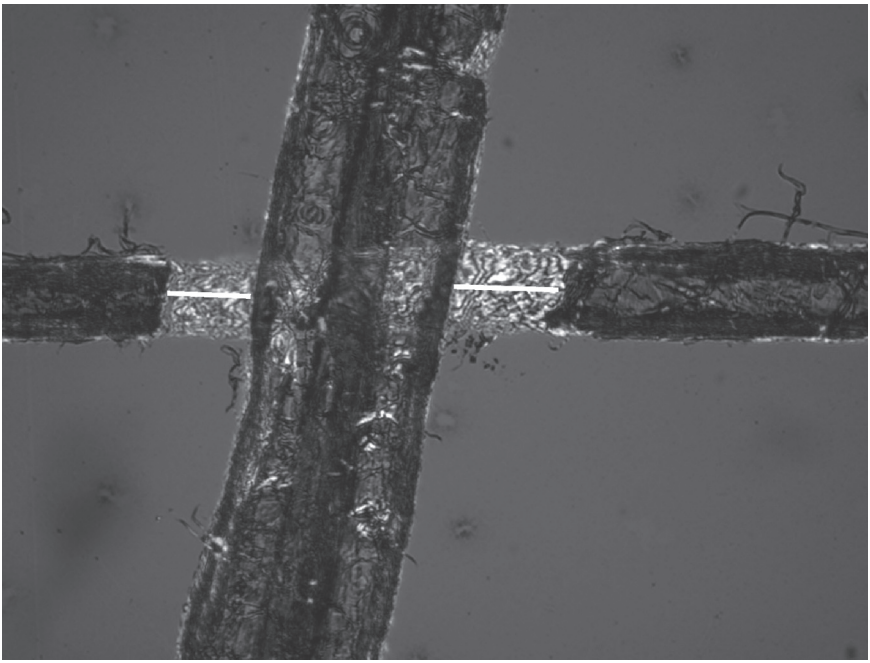

Figure 6 Dyed black spruce fibres refined to 300 revolutions. Two freespans are indicated by the white lines.

Experimental procedure

A mill produced, never dried, northern Canadian black spruce (*Picea mariana*) unbleached kraft pulp was washed and screened in a lab screen with 0.008 inch (0.2 mm) slots. The pulp's kappa number was 20. Four pulp samples were refined using a lab PFI mill with a 0.2 mm gap to 300, 1000, 2000, and 4000 revolutions. Five grams of wet pulp were removed from each sample and dyed using Chlorazol Black. Imaging slides were prepared as describe above after thoroughly washing the dyed pulp with deionized water. TAPPI standard handsheets were made with the remaining pulp. Freespans and stepheights were measured using image analysis software (see Appendix A). Tensile strength, density, and scattering coefficient were measured and are given in Appendix B.

RESULTS AND DISCUSSION

High contrast, high resolution images have been acquired using the methods described above. Over 200 images were collected and analyzed. Figures 6, 7,

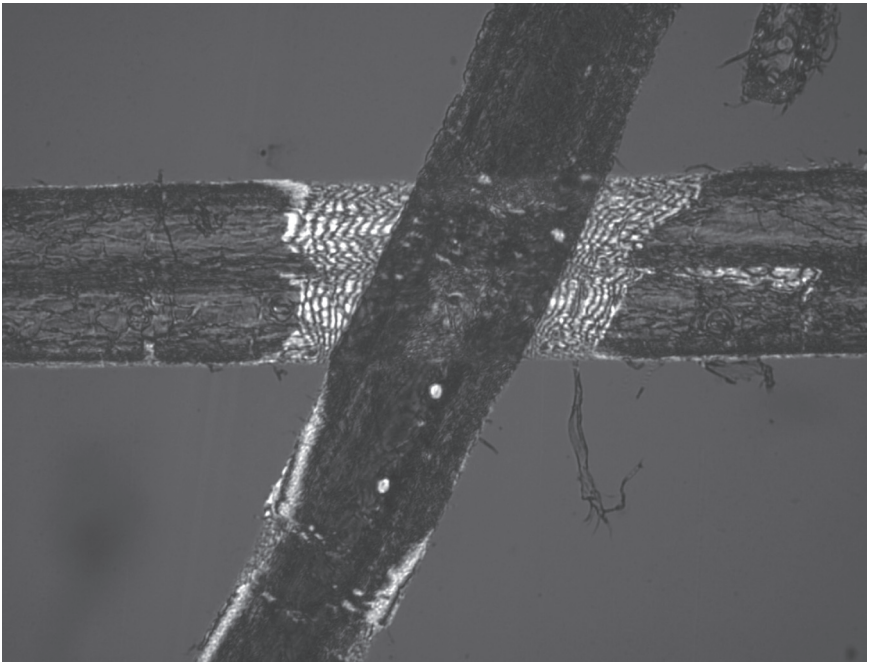

Figure 7 Dyed black spruce fibres refined to 1000 revolutions.

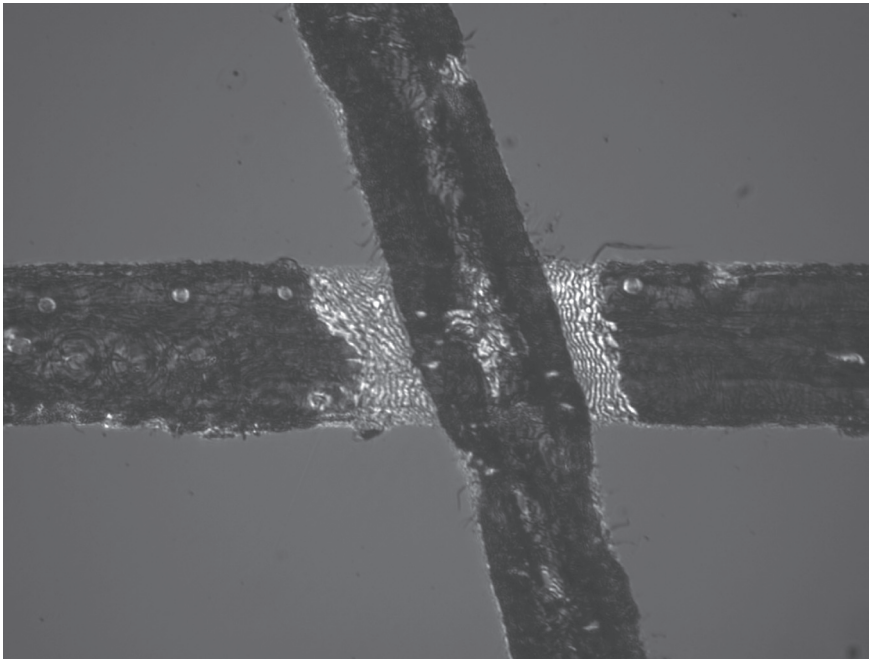

Figure 8 Dyed black spruce fibres refined to 2000 revolutions.

8, and 9 are representative micrographs showing fibres refined to 300, 1000, 2000, and 4000 revolutions respectively.

Stepheight versus freespan for a lightly refined pulp

Unbleached kraft black spruce fibres were refined in a PFI mill to 300 revolutions. Images were acquired and were analyzed by dividing each image into two areas of interest, one for each side of the intersection. Image analysis software was used to measure freespans and stepheights. The results are shown in Figure 10. A regression analysis line passes through the origin indicating that higher stepheights generally have higher freespans.

Figure 11 offers an interpretation of this result. Fibres in the unbeaten state after pressing are often uncollapsed [9] (Figure 11A) resulting in a high stepheight. Collapsed fibres will have a lower stepheight (Figure 11B). Assuming that all the fibres have the same flexibility, the stepheight controls the length of the freespan.

There is some scatter in the freespan data which is likely due to the differ-

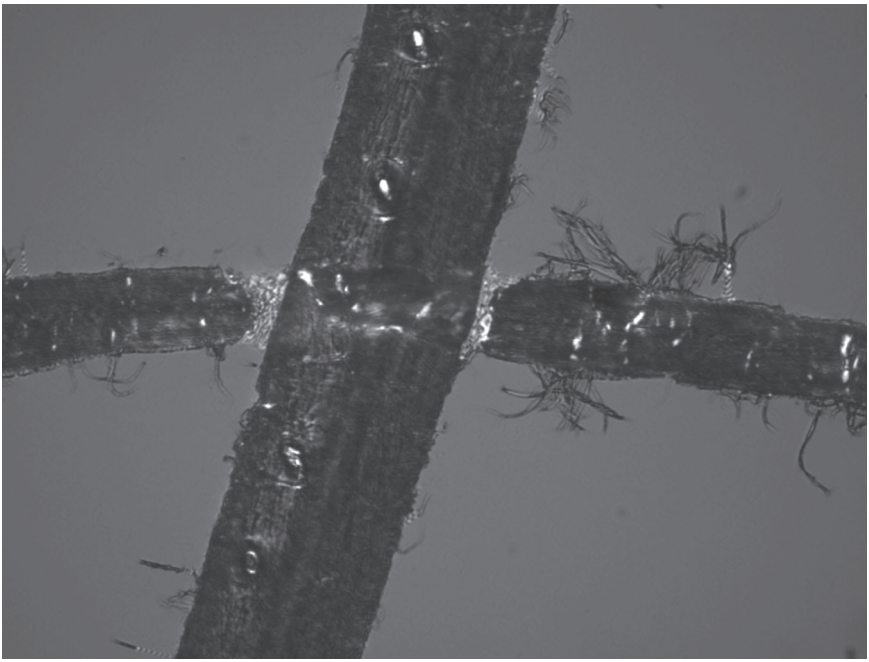

Figure 9 Dyed black spruce fibres refined to 4000 revolutions.

ences in fibre flexibility of the crossing fibres. The double wall thickness of an unbleached black spruce kraft fibre is about 2 μm which is slightly less than the average stepheight shown in Figure 10. While stepheight may primarily control the length of the freespan, flexibility also has some influence.

Stepheight versus freespan for refined pulps

Figure 12 illustrates two hypothetical extremes of the effect of refining. In Figure 12A, refining only serves to increase the flexibility of fibres reducing the freespan at a given stepheight. In Figure 12B, refining makes fibres more collapsible reducing the stepheight and, hence, the freespan. Figure 13 shows a plot of stepheight versus freespan for each refining level. Figure 14 shows the average for the same data. Clearly, the effect of refining is to lower the stepheight as proposed in Figure 12B. There is no indication that refining increases the longitudinal flexibility of fibres which would lead to a lower freespan at a given stepheight.

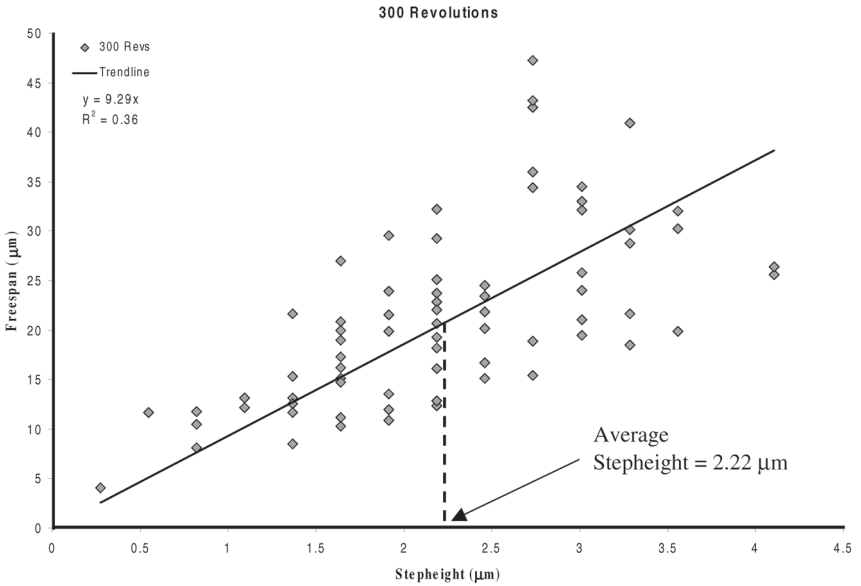

Figure 10 Stepheight versus freespan for lightly beaten (300 revolutions) black spruce fibres.

Conformability and deformability in a fibre intersection

The data for the refined pulps do present a challenge in interpretation. Upon refining the average stepheight falls to 1.1 µm (see Figure 14) and many values of stepheight are below 0.5 µm. Indeed, there are cases in which the stepheight is essentially zero as shown in Figure 15. Since this measurement is calibrated to the wavelength of light, there can be little doubt as to its accuracy.

A stepheight of zero presents some difficulty when one considers that the double wall thickness of a collapsed unbleached black spruce kraft fibre is about 2 µm. The lower bound on stepheight should be 2 µm not 0 µm. Two mechanisms can help explain this apparent anomaly. First, the overlying fibre may conform to the fibre beneath by overlapping as shown in Figure 16A. Second, the underlying fibre may be compressed and deformed as shown in Figure 16B. One or both of these phenomena must occur to explain the data, but the extent of each is not certain.

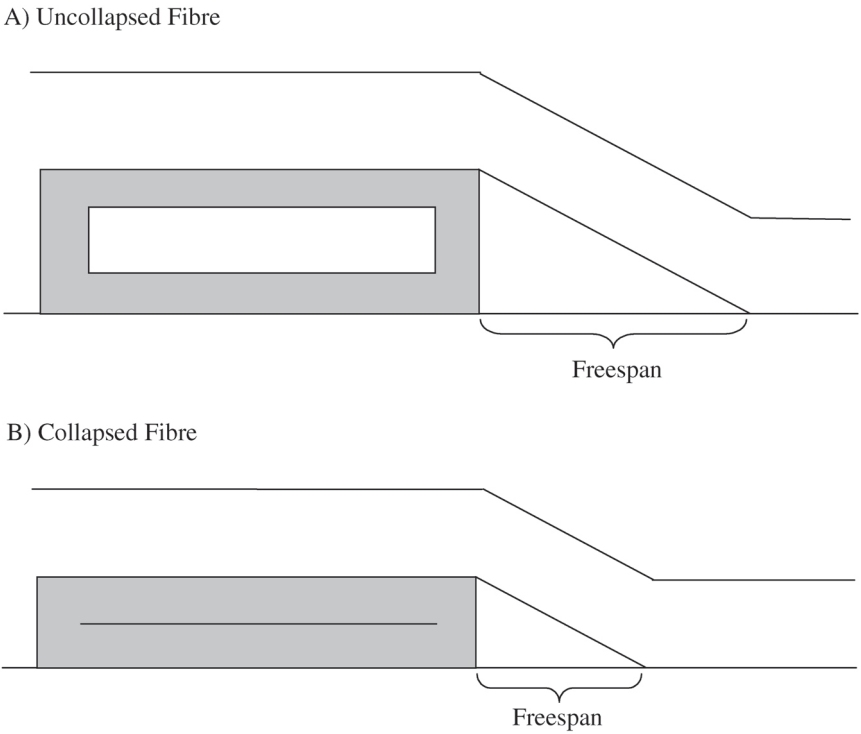

Figure 11 The effect of fibre collapse on stepheight and freespan.

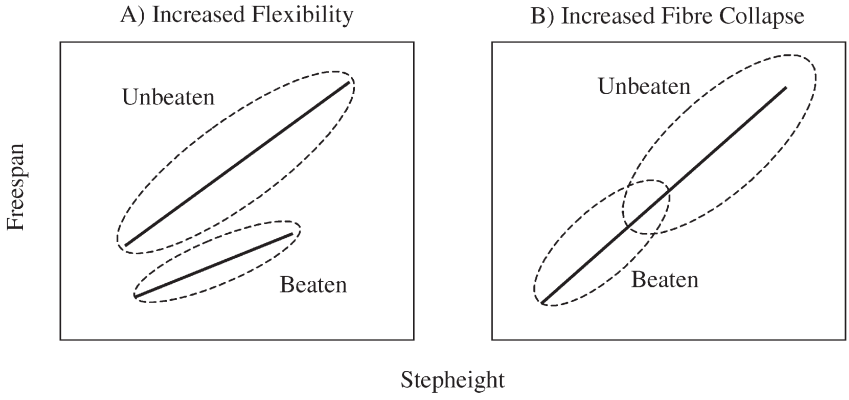

Figure 12 Two potential effects of refining.

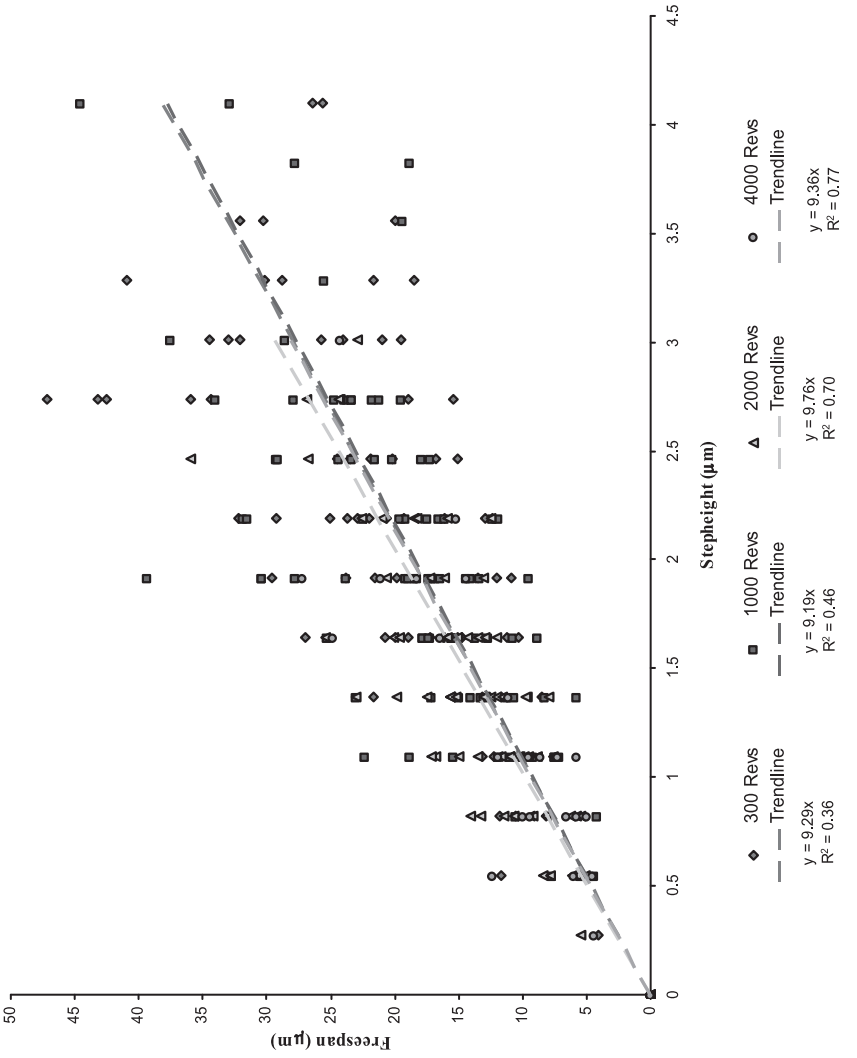

Figure 13 The effect of refining.

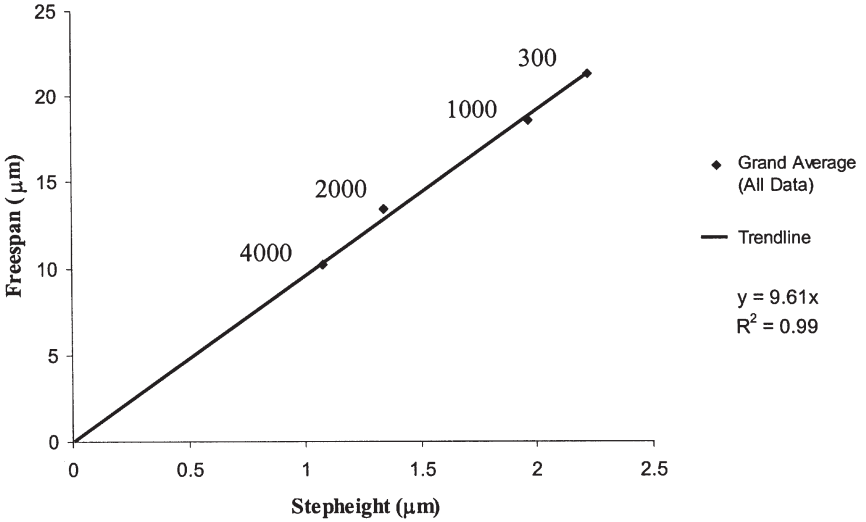

Figure 14 Average stepheight versus average freespan for 300, 1000, 2000, and 4000 revolutions.

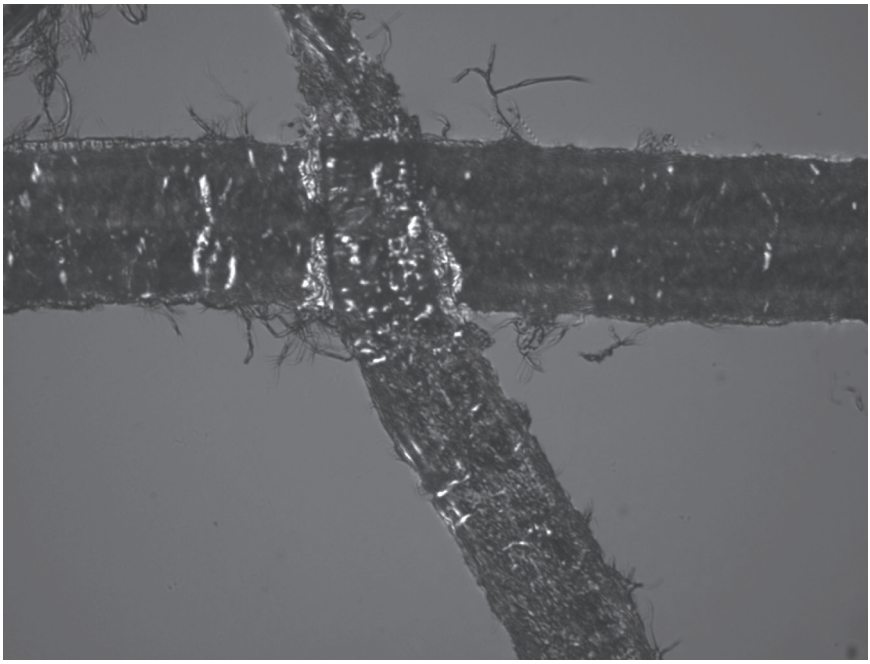

Figure 15 Dyed black spruce fibres refined to 4000 revolutions showing essentially no freespan or stepheight.

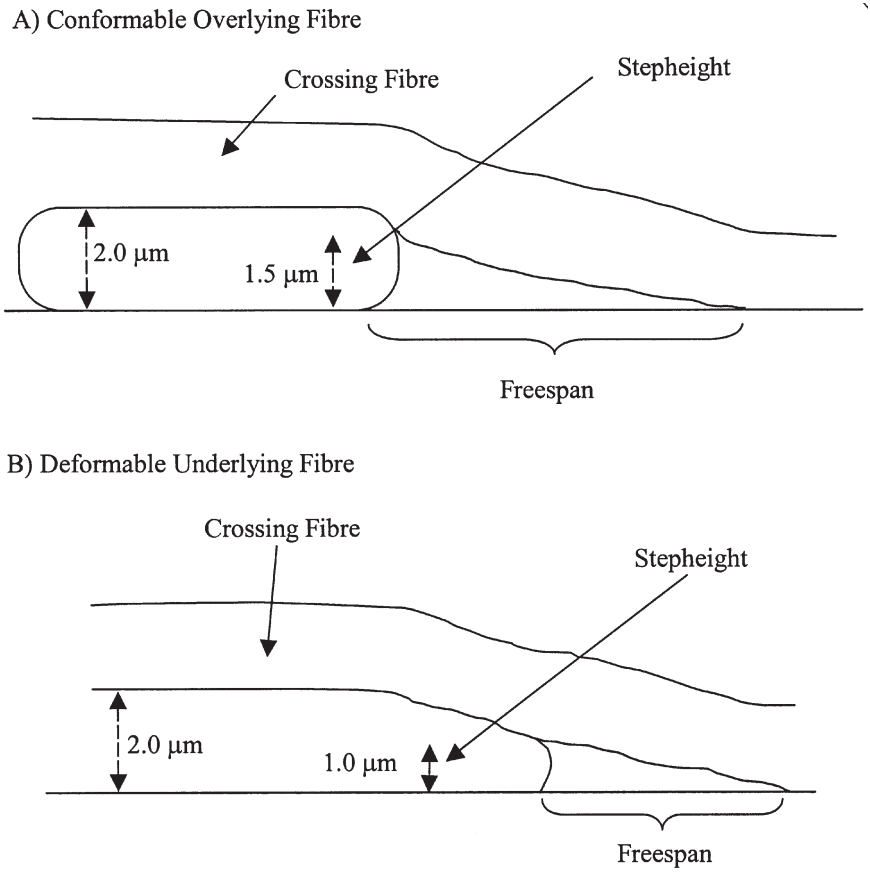

Figure 16 Conformability and deformability in a fibre intersection.

The shape of the fibre in the freespan region

The shape of the fibre in the freespan region can be determined from the interference fringes. Previously it has been suggested that the fibre deforms via bending and the diagrams (e.g. Figure 2 of Steadman and Luner [6]) seem to confirm this concept. Recently it has been suggested by Waterhouse and Page [10] that this is incorrect. In paper the transverse deformation of fibres occurs more by shear than by bending. This work allows the shear deformation hypothesis to be checked by analyzing the spacing of interference fringes in the freespan region. The two modes would show different spacing

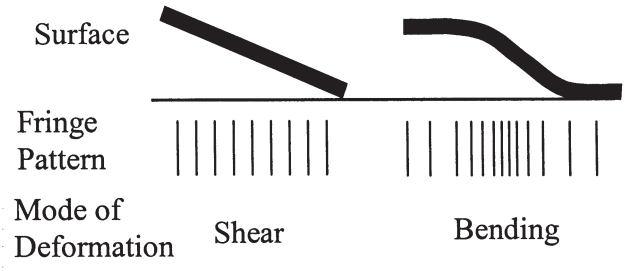

Figure 17 Fringe patters for different modes of deformation.

between the interference fringes as shown in Figure 17. Shear deformation will result in more evenly spaced fringes. Bending deformation would show fringes that are spaced more closely at the center of the span and further apart near the crossing fibre and near the contact with the glass. Upon close inspection of images (Figures 7, 19 and other unpublished images) the fringes appear approximately evenly spaced. This physical evidence thus tends to support the theory presented by Waterhouse and Page [10].

Imaging of fibre intersections in water

It is also important to note that this technique can capture images of fibre intersections immersed in water. Figure 18 shows the same fibre intersection dry (A) and after re-wetting (B). Although the contrast is greatly reduced the

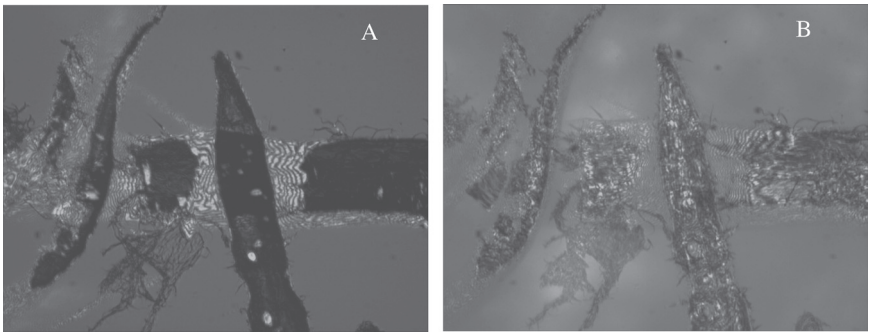

Figure 18 Dyed black spruce fibres refined to 1000 revolutions dry (A) and wet (B).

interference fringes are apparent in both cases. This result has implications with respect to wet pressing. Ultimately, the goal of this research to image fibre intersections during the dynamic conditions that occur during wet pressing.

CONCLUSIONS

A new experimental procedure has been developed that allows fibre deformability to be measured using geometries that realistically simulate deformations in paper. Contrary to earlier opinion, the freespan of a fibre intersection is not controlled by the flexibility of the overlying fibre. Instead it appears to be controlled partly by the local conformability of the overlying fibre as it wraps around the underlying fibre and partly by the collapse and deformability of the underlying fibre. Observed interference fringe patterns indicate the predominant mode of deformation of a fibre freespan in a paper sheet is shear.

The technique has provided some surprising results in the light of all the previous work on fibre flexibility. It provides new opportunities to investigate the effect of mechanical and chemical treatment on pulp fibres in both dry and wet states.

ACKNOWLEDGMENTS

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Appendix A

Image analysis

The described technique allows the effect of any number of mechanical and/or chemical treatments to be analyzed. Often with mechanical treatments, the fibre surface becomes damaged leading to difficulty in counting the interference fringes. When this situation arose, image analysis software was utilized to help count fringes. Figure 19 shows an intersection with broad, distinct fringes, while Figure 20 shows fringes that were difficult to resolve.

The image analysis software includes a feature that determines the intensity at each pixel or picture element. When there was difficulty in determining the number of fringes, a line of pixels was selected on the image and the intensity data was graphed. Figure 21 shows an enlargement of the region indicated in Figure 20 along with an intensity profile. The dashed line indicates the line of pixels (160 in total) that were analyzed. Each pixel is assigned a value based

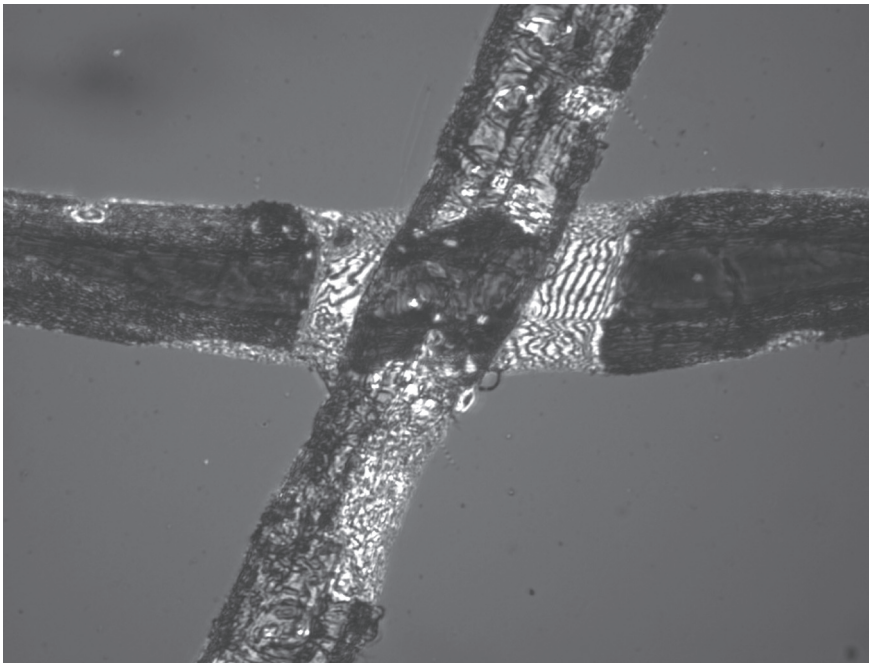

Figure 19 Dyed black spruce fibres refined to 300 revolutions showing distinct interference fringes.

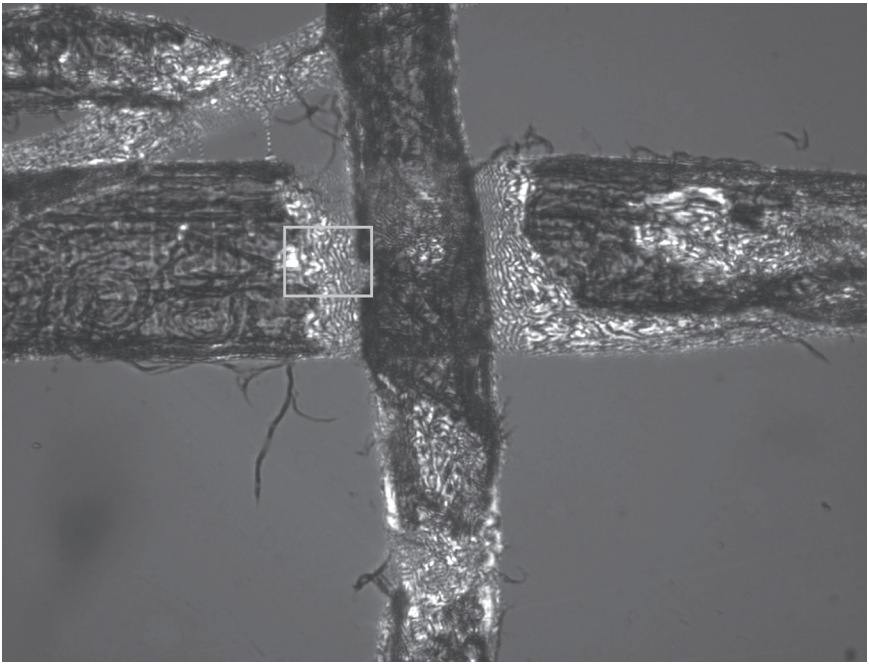

Figure 20 Dyed black spruce fibres refined to 300 revolutions showing indistinct interference fringes. The boxed region indicates the area used in Figure 21.

on its intensity. Line A represents where the overlying fibre leaves the glass slide, while line B represents where the underlying fibre begins. Both line A and B are zeroth order fringes. Dark and light fringes are indicated by a definitive change in the direction of the intensity profile. Each successive line between A and B indicates where a dark fringe occurs and its corresponding position on the intensity profile.

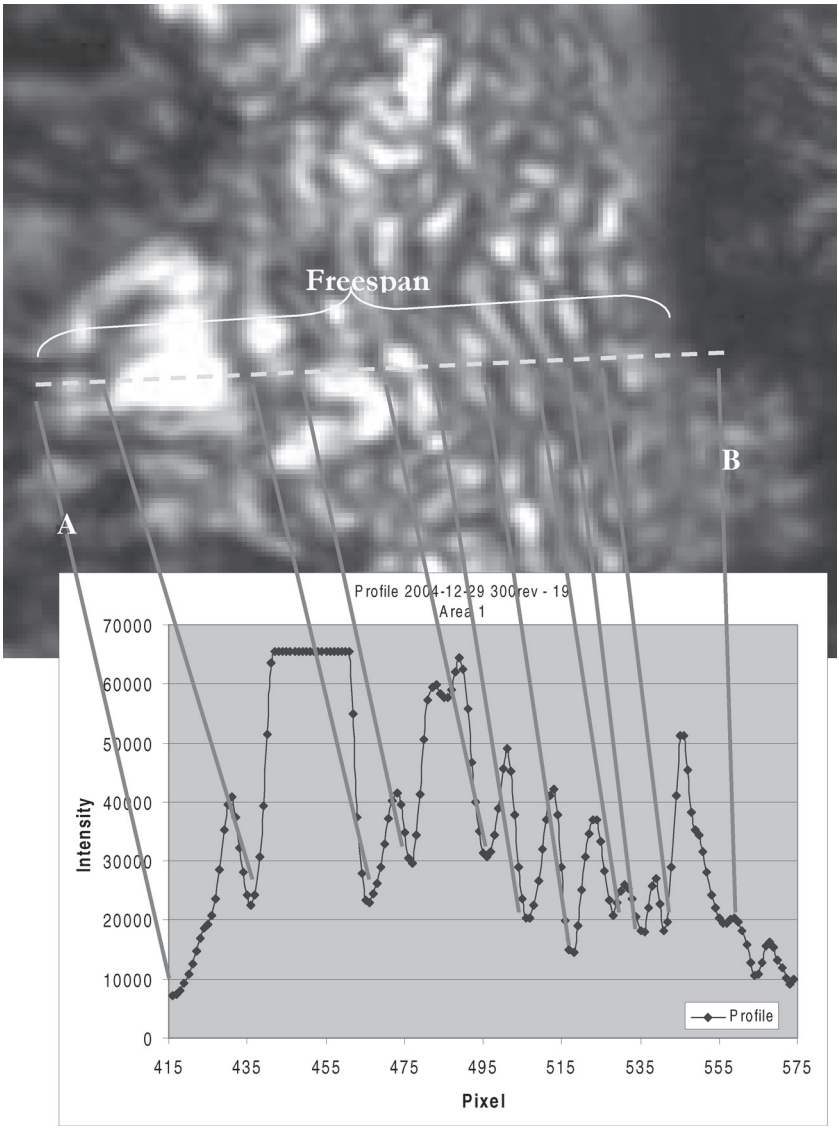

Figure 21 Using intensity analysis to aid in counting interference fringes.

Appendix B

Physical and optical properties

Table 1 shows some physical and optical property data that were collected. The data trend as expected. Refining leads to higher densities, higher tensile indexes, and lower scattering coefficients.

Table 1 Physical and optical properties.

PFI Revs	Density g/cm^3	Tensile Index Nm/g	Scattering Coefficient m^2/kg
300	0.75	57.45	25.31
1000	0.84	74.35	21.58
2000	0.94	88.42	18.42
4000	1.01	94.45	16.17

Transcription of Discussion

IMAGING FIBRE DEFORMATIONS

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Ho Fan Jang PAPRICAN

Thank you very much for presenting a very interesting technique.

I have one question: considering that the optical sectioning of confocal microscopy can allow us to generate highly-resolved two- and three-dimensional images for wood pulp fibres, would you comment on why this technique is used instead of using confocal microscopy?

Rob Lowe

The ultimate goal of this research is to image fibre intersections during wet pressing. We want to capture movies and confocal cannot do that.

We were also looking for a new way to look at deformability. Confocal probably does do some of the similar things, but this is optical, we are actually seeing these visually.

Richard Kerekes The University of British Columbia

Some of those earlier methods were an attempt to measure fibre flexibility as it is defined scientifically: the modulus of elasticity multiplied by the area moment of the inertia. It is useful for many purposes and only partly explains deformability. I think you are very correct in pointing this out. What you are looking at, as far as I can see, is a combination of large amplitude bending and collapsibility. When these come together around that sharp corner you show, bending is further compounded by the fact that many of our earlier estimates did not consider shear within the fibre. I think Waterhouse and

Discussion

Page showed that this is really important and is something that earlier workers missed. This has been a real addition. But, I wonder, at the end of the day when you have made your measurement, what units do you have for deformability?

Rob Lowe

We do not have units of deformability at this point. We have looked at the data and tried to figure out what was going on. We do not have an index or anything like that right now.

Richard Kerekes

Do you think you will ultimately be able to relate it to a combination of collapsibility and flexibility? Perhaps to some basic fibre properties?

Rob Lowe

Something measurable like a reportable index, yes that will be ultimate goal.

Ingunn Omholt PAPRICAN

I am sure you have thought of this and discussed it when you developed your technique, but I was wondering if you could comment on the chance that the shrinkage of the underlying fibre might have affected the apparent flexibility of the crossing fibre and whether it might have stretched the free span and made it appear straighter, less bent than it was originally?

Rob Lowe

That is a very good point. The only way to get around that I think is to look at the fibre crossing while it is still wet. Again, there is some difficulty in trying to resolve the techniques but we hope to do that. When the fibre dries it is possible for some of this stretching to occur but this also happens in paper when it dries.

Steven Keller SUNY-ESF/ESPRI

I am sure you selected the green wavelength for some reason. Did you try other wavelengths?

Rob Lowe

No, green is really the predominant filter – 547 *nm* is almost always used for inferometry.

Steven Keller

Have you ever considered using multiple wavelengths or changing the wavelength and seeing if your results will be the same?

Rob Lowe

They should be exactly the same because all it is going to do is shift where the interference fringe is slightly.

Steven Keller

Right. Could you use other wavelengths in order to get better resolution or may be even determine more details of the result you are getting?

Rob Lowe

I am not sure where more information would come from.

Steven Keller

I think there was a Toyo Seiki instrument which used optical contact and changed the wavelengths in order to look at different depths and get different optical contacts for surface roughness.

Rob Lowe

If we are talking about optical contacts, that is different from interference technique images.

Gary Baum PaperFuture Technologies LLC

Thanks, Rob, for that presentation. Would you not expect that the step height would be very dependant upon the surface tension forces existing between the two fibres and that you really also need to consider the chemistry of the system?

Discussion

Rob Lowe

Do you mean the fibre-fibre bond?

Gary Baum

As the fibres dry, the wrapping of one fibre on another.

Rob Lowe

Sure. There should be something that you can consider. That is going to become important. When we start looking at other pulps. Right now, it is all the exact same pulp we used.

Gary Baum

If you add little a little surfactant to the system, do you think you will get the same step height?

Rob Lowe

That gets back to the PAPRICAN question I think, that it is going to change how the fibres are bonded to each other so it may change the apparent step.

Kecheng Li University of New Brunswick

Just a response to Ho Fan's question. We have done some similar work recently, but we used a confocal microscope because if you use a confocal, you can have a focal plane, so if you direct your focal plane on the glasses slide surface, actually it is much easier to identify the free span.

Wolfgang Bauer Graz University of Technology

I have two questions. The first part of the question is the influence of the fibre width on the free lengths. Did you see any influence there?

Rob Lowe

We did not look for the influence of fibre width. It may have an influence, but, I think that when it comes down to it and we do all the averages it works its way out.

Wolfgang Bauer

The second part of the question is to get a feel for the means. How many fibre crossing did you evaluate to get one mean value that you reported?

Rob Lowe

For each refining level about 50 data points were used to get an average. We took lots of images, probably 400 images or so, to get the data for all refining levels.

Kaarlo Niskanen KCL

I was just wondering would you comment on the fact that there are not really any interference fringes at the edges of the fibres. Might that indicate that the fibres are pulled against the glass plate? If the fibres had sort of elliptical cross section, you might expect to see some (interference fringes).

Rob Lowe

That is going to happen when the fibres dry, when it comes off the glass side a little bit, only slightly. As a fibre is dried it can lift off the slides a little bit and so we get the first interference fringe.

Kaarlo Niskanen

Yes, but my point is that when the fibres are drying on the glass plate, they might be deformed to cross-sectional shapes that are different from what they would be in a paper sheet.

Rob Lowe

Oh, sure of course, the fact that these fibres are flat and bound to the glass slide, is a little bit different from what is going on in the paper sheet. So, the structure is going to change in the paper sheet, but we are trying to find a method to look at the deformability of a fibre crossing. This is as close as anyone has come so far.

Kaarlo Niskanen

Yes, I think the work is beautiful, I should say. If I may continue, one more, did you measure the widths of the underlying fibres at the crossings?

Discussion

Rob Lowe

We did not measure the widths, but we still have the images. So, it would not be a problem to get the widths.

Kaarlo Niskanen

Yes, because you do not see any deformation at the crossing with the underlying fibre which might take place if there was a pressure between them.

John Roberts University of Manchester

I am just following on from Gary Baum's comment about the possible influence of surface chemistry, how much of an impediment do you think the Chlorazol dye molecules are going to be when looking at surface characteristics?

Rob Lowe

We used Chlorazol Black because it is known not to change the fibre chemistry very much, but again, it is a molecule that is on the surface, so it probably is affecting the crossing to some extent.

Alan Button Buttonwood Consulting

It is nice to see the work coming from the early discussions to where you are now, some really cool stuff. I am picking up on Dick Kerekes' comment. I am wondering whether there would be value in looking at this as a mechanical problem, much like folks have done earlier, and also bringing in microfibril angle to see if you can predict how these fibres would behave from a geometry and structure point of view?

Rob Lowe

Of course, I think that would be possible but hopefully it will be somebody else's PhD. One thing that I do want to stress is that I think that we need to be thinking about fibre deformations on a much more local scale. When we talk about fibre flexibility, or rigidity, we are implying that the entire fibre is involved and that's simply not what we should be concerned with when we talk about fibre deformations.

Ron Lai Eka Chemicals

I am just curious in terms of future work. Will you consider other types of refining because as far as I understand PFI refining is not very indicative of commercial refining techniques?

Rob Lowe

Correct, you are right, it is not very indicative, but we needed a quick lab scale refining that we could do. The PFI is pretty much standard. It would be interesting to expand it to other refining types, unfortunately, for other refining types you usually need a lot more fibre.