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STRENGTH AND CHEMICAL COMPOSITION OF WOOD PUMP FIBRES

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ABSTRACT

An analysis of published work, together with new data, has clarified the effect of chemical composition on the strength of wood pulp fibres. Among fibres of low fibril angle, and in nondegrading pulping processes, strength (expressed as breaking stress) is directly proportional to α-cellulose content over a wide yield range. This implies that the cellulose fibrils are the sole tensile-load-bearing elements; hemicellulose and lignin only serve as a matrix that transfers the stress under shear from fibril to fibril. However, pulping to a yield corresponding to an α-cellulose content higher than 80% tends to reduce fibre strength apparently because of the elimination of this stress-equalizing matrix. In cellulose-degrading processes, fibre strength falls below this expectation to an extent dependent upon the degradation. Thus a tool is provided that permits the degradative effect of new pulping or bleaching processes on fibre strength to be assessed. The value of zero-span strength as an index of fibre strength is confirmed.

THEORETICAL CONSIDERATIONS

The wood cell wall has a structure like that of a fibre-reinforced composite. In this analogy the "fibres" are the long slender highly crystalline cellulosic fibrils, and the "matrix" is the hemicellulose-lignin gel that surrounds them. This approach has been used successfully to explain the mechanical properties of wood by Mark (1) and of wood-pulp fibres by Page, El-Hosseiny et al (2, 3, 4, 5, 6, 7, 8).

In the S_2 layer of the cell wall, which comprises about 90% of the wall material in northern softwoods, the fibrils are locally parallel to one another and are helically wound at an angle to the fibre axis termed the fibril angle. Because of this structure, the strength of wood pulp fibres is a strong function of fibril angle, being at a maximum when the fibril angle is low, that is, when the fibrils are aligned with the fibre axis (3). Among slow-grown northern species, the average fibril angle is low, of the order of 10-15°; in most fibres it lies between 0° and 10° (9). The analysis given here is for fibres of low fibril angle.

By analogy with fibre reinforced composites

$$E_{fibre} = E_{fibril} V_{fibril} + E_{matrix} V_{matrix} \quad (1)$$

where E and V are the elastic moduli and volume fractions respectively of the phases. Since for all wood pulp fibres $E_{fibril} >> E_{matrix}$ and $v_{fibril} \geq v_{matrix}$, equation (1) may be abbreviated to

$$E_{fibre} = E_{fibril} V_{fibril} \quad (2)$$

Because the density of the fibril and the matrix are similar, V_{fibril} can be replaced by the weight fraction W_{fibril}, which is reasonably well determinable analytically either as α-cellulose (10) or as glucan, by enzymatic oxidation (11). Therefore

$$E_{fibre} = E_{fibril} W_{fibril} \quad (3)$$

If we assume that the failure mechanism is associated with strain energies in the fibrils and that the matrix plays little part in the failure mechanism of low fibril angle fibres, then

$$T_{fibre} = T_{fibril} W_{fibril} \quad (4)$$

where T is the tensile strength, expressed as breaking force/unit area of cross-section or breaking stress.

Thus, the breaking stress of wood fibres should increase upon pulping, being proportional to the weight fraction of fibrils in the fibre, provided that the pulping process does not degrade the fibrils and reduce T_{fibril}.

An alternative and equivalent statement is that, upon pulping, the breaking load per fibre should remain unchanged, since the cellulose portion of a fibre is essentially unchanged under conventional pulping procedures, and only the non-load-bearing hemicellulose and lignin are removed.

The literature contains measurements both of fibre breaking stress and of fibre breaking load, as a function of pulping, by which the theory may be checked, and this will now be reviewed. In some cases the measurements were made directly on single fibres. In others the indirect measure of zero-span tensile strength of handsheets was used.

EXPERIMENTS RELATING TENSILE STRENGTH T_{FIBRIL} TO CELLULOSE CONTENT W_{FIBRIL} WITH CHANGING YIELD

Cowan (12) studied the zero-span strength of handsheets of spruce, kraft-pulped in the yield range 65%-50%. W_{fibril} was determined from the α-cellulose content. Cowan showed that T_{fibril}/W_{fibril} was a constant independent of yield, a result which confirms equation (4) and which he anticipated by reasoning similar to that given earlier.

Stone and Clayton (13) measured the zero-span strength of longitudinal sections of red spruce springwood, kraft-pulped in the yield range 100%-43%. They found that the lower the yield, the lower was the ratio of zero-span strength to α-cellulose content, the value dropping about 20% over the yield range. They concluded that while the chief contribution to fibre strength was provided by the α-cellulose content, the other constituents also made a contribution. They attributed Cowan's result to the use of an inadequate yield range.

Leopold and McIntosh (14) measured the breaking stress of loblolly pine summerwood and springwood holocellulose fibres after caustic extractions of increasing severity. The yield range based on wood was from 72%-49%. Fibre strength fell with increasing α-cellulose content. They concluded that the hemicelluloses are important for the internal cohesion of the cell wall and that their removal weakens the fibre by reducing interfibrillar adhesion.

Spiegelberg (15) measured breaking stress of longleaf pine holocellulose fibres, caustic-extracted to various extents. The strength fell with increasing α-cellulose content. Spiegelberg deduced that since the cellulosic fibrils are not degraded by the extraction, the loss in strength is caused by the poor stress distribution in the fibre as the flexible fibril-hemicellulose-fibril bonds are replaced by inflexible fibril-fibril bonds.

EXPERIMENTS RELATING BREAKING LOAD PER FIBRE TO CHANGING YIELD

McIntosh (16) investigated the breaking load per fibre of loblolly pine that was initially kraft pulped in the yield range 94%-46% and subsequently delignified by a peracetic acid treatment to give a final yield range of 64%-44%. The fibre strength fell with decreasing yield.

Leopold (17) measured breaking load per fibre in the yield range 89%-75% from spruce pulped by the neutral sulphite semi-chemical process. He found no change in fibre strength and concluded that the lignin and hemicellulose removed in this yield range made no contribution to strength.

Leopold and Thorpe (18) measured the breaking load per fibre in the yield range 84%-48% from Norway spruce pulped by the kraft, bisulphite and acid sulphite processes. They found that in all processes, the fibre strength dropped uniformly with yield.

Usuda et al. (19) measured the breaking load per fibre of sulphite and kraft pulps of red pine in the yield range 66%-43%. They found that the fibre strength decreased with decreasing yield, but more rapidly among the suphite pulps than among the kraft.

COMMENTARY ON THE LITERATURE

At first glance equation (4) does not appear to be well supported by the literature. The data of Cowan (12) and Leopold (17) support it, but there is evidence from the other work cited that, upon pulping, fibre strength falls below the expected value. In particular, rather severe losses in strength occur upon caustic extraction.

Equation (4) cannot be tested critically with the cited data for three reasons. First, equation (4) is expected to be valid only for fibres of fibril angle lower that 15°, since for higher fibril angles, failure is initiated by shear in the matrix rather than by failure of the fibrils (3). Unfortunately fibril angle was not reported with any of the cited data.

Secondly, the cited data do not all cover the wide yield range that is necessary to bring out the functional relationship between cellulose content and strength.

Thirdly, techniques of single fibre testing (2) and of zero-span strength measurement (20) have developed considerably since the cited work, and might be expected to provide improved data.

Two pieces of work have therefore been carried out on specimens of known low fibril angle, over the complete yield range from 100% (stone groundwood or refiner mechanical pulp) to 43% yield chemical pulp, using improved methods of fibre strength measurement and zero-span testing. These data have been used to test equation (4).

ZERO-SPAN STRENGTH OF PULPS OF VARIOUS YIELDS

A series of pulps prepared from a common source of slow-grown black spruce, from Quebec, Canada, were available from previous work. This wood is known to have an average fibril angle of around 12°; in 50% of the fibres, the fibril angle lies between 0° and 10°.

Eight kraft pulps in the yield range 66-44%, a refiner mechanical pulp, and a holocellulose were used. The cellulose content of the kraft and mechanical pulps were obtained from a knowledge of their yields, using the relationship found by Rydholm (21) between yield and enzymatically determined glucan content in similar black spruce pulps. The glucan content of the holocellulose sample was determined enzymatically.

Zero-span strength of handsheets was measured using the Pulmac zero-span tester. Among pulps of higher yield, the zero-span strength was found to rise with handsheet pressing

pressures and level out at a plateau that was considered to be the correct measure of fibre strength.

The plot of zero-span strength against cellulose content is shown in Figure 1. The results confirm with remarkable precision the validity of equation (4) for these pulps. The points representing refiner mechanical pulp, holocellulose, and kraft pulp all fall on a common line passing through the origin. The kraft and holocellulose pulping processes used in this experiment were deliberately chosen because they are not expected to degrade the cellulose; thus T_{fibril} would be constant and equation (4) could be tested. The sole point beyond 80% cellulose content falls below the line. While comment is not justified on this result alone, the drop in fibre strength beyond 80% cellulose content is confirmed by other work and will be discussed later.

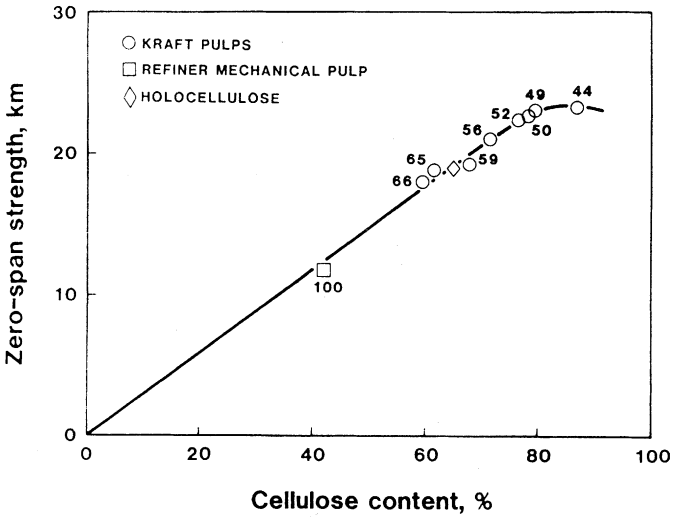

Fig 1—Zero-span strength of well-bonded handsheets of black spruce pulps, plotted against cellulose content, derived enzymatically. The figures indicate % yield.

SINGLE FIBRE STRENGTH OF PULPS AT VARIOUS YIELDS

During a program of work on single fibres, a series of fibre strength measurements were made using the same low fibril angle wood source pulped in a variety of ways. The outer rings of a single black spruce tree over 100 years old were used and the fibril angle was found to be below 10° in all fibres. Kraft, holocellulose and neutral sulphite pulps were prepared by conventional procedures. Unpulped wood fibres were obtained by stripping single fibres away from the wood block under hot water. Glucan content was determined enzymatically.

The techniques of fibre preparation and strength measurement that have been developed are described elsewhere (2). The preparation method avoids application of stress to the fibre during mounting, glueing and clamping of the mount. It ensures linearity of the fibre during drying so that a truly axial stress is applied during testing. The test methods ensure precision of cross-sectional area measurement and tensile load. The span length in these tests was fixed at 0.35 mm.

The results are shown in Figure 2. They confirm the hypothesis that fibre strength is directly proportional to cellulose content. Again it must be noted that the pulping processes used here are not expected to degrade the cellulose.

REEXAMINATION OF DATA FROM THE LITERATURE

In view of the unambiguous implication of the results of Figures 1 and 2 it was felt worthwhile to reassess the published data, by plotting the values of fibre strength (or zero-span strength) against cellulose content, and comparing the results with those of Figures 1 and 2.

ZERO-SPAN STRENGTH AND CELLULOSE CONTENT

Cowan's data for spruce kraft pulp are plotted in Figure 3. The range is very narrow; nevertheless the data are consistent with the hypothesis of a linear relationship through the origin with a drop in strength for the lowest yield sample, occurring at about 80% cellulose content.

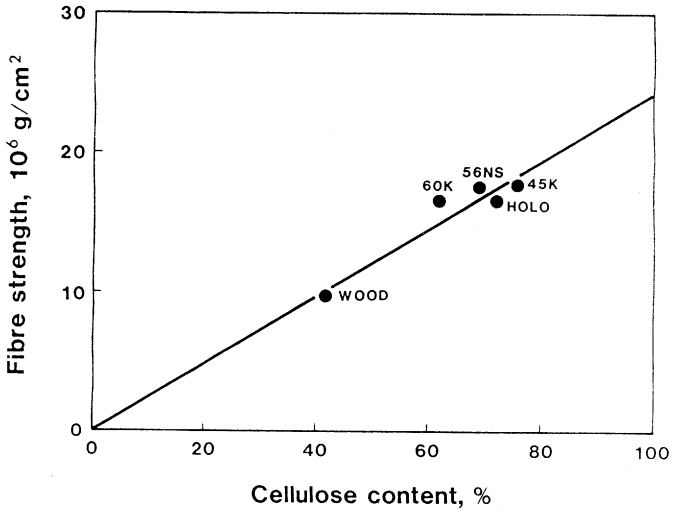

Fig 2—Single fibre strength of pulps from the same region of single black spruce log, plotted against cellulose content, derived enzymatically.

Wood - fibres stripped mechanically from the wood
60K - 60% yield kraft
45K - 45% yield kraft
56NS - 56% yield neutral sulphite
HOLO - acid chlorite holocellulose.

SINGLE FIBRE STRENGTH AND CELLULOSE CONTENT

The data of Leopold and McIntosh (14), McIntosh (16) and Speigelberg (15) are plotted in Figures 4, 5 and 6. The yield range is again small. Nevertheless these data considered in combination are consistent with the proposal that fibre strength is proportional to cellulose content, up to about 80% cellulose content, after which it falls. It should be noted that the slope of the line from the origin is different for the

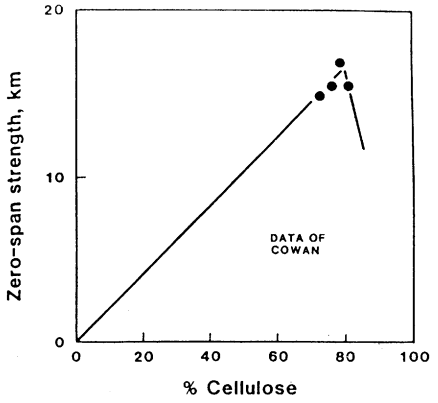

Fig 3—Zero-span strength of handsheets plotted against α-cellulose content for spruce kraft pulps (**12**)

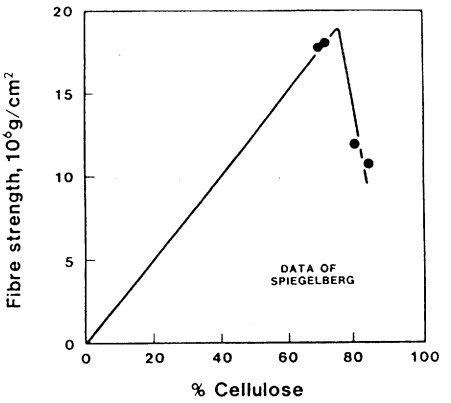

Fig 5—Fibre strength of loblolly pine kraft pulped and bleached to varying degrees, plotted against α-cellulose content (**16**)

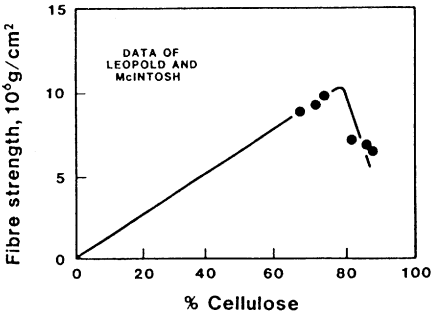

Fig 4—Single fibre strength plotted against α-cellulose content for loblolly pine holocellulose after caustic extractions of increasing severity (**14**)

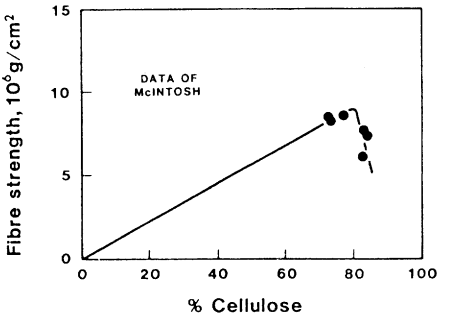

Fig 6—Fibre strength of longleag pine holocellulose, caustic extracted to various degrees, plotted against α-cellulose content (**15**)

data from different sources. The reasons for this may be associated with differences in testing methods and fibre source and will not be considered here.

DISCUSSION

To a remarkable degree the data of this paper combined with a reassessment of published work provide a clear picture of the way in which the strength of low fibril angle pulp fibres is modified by progressive removal of the matrix material by means that do not affect fibril strength. Over the cellulose content range of 44% to 80% the fibre strength is directly proportional to cellulose content as expected. Above 80% cellulose content the fibre strength falls off. It appears that this pattern did not emerge clearly from earlier work because no single author who measured both fibre strength and cellulose content covered a wide enough yield range, but generally spanned the range around 80% cellulose content in which the fibre strength first increases and then decreases.

The reason for this fall-off of strength at 80% cellulose content is not completely clear. Leopold and McIntosh suggest it is caused by inadequate adhesion between the fibrils as hemicellulose is removed. Spiegelberg suggests it is due to poor stress distribution in the structure as flexible cellulose-hemicellulose-cellulose bonds are replaced by more rigid cellulose-cellulose bonds. This suggestion of Spiegelberg is supported by two pieces of evidence. First, a fibril content of 80% would be expected to coincide at least approximately with the point at which fibrils contact one another without an intervening matrix. Secondly it is known that fibres such as ramie and flax which contain almost no matrix material increase in strength with increasing relative humidity indicating that a softening of the fibril-fibril bond improves the stress distribution in the fibre.

Of the cited data, only two sets, those of Stone and Clayton and Leopold and Thorpe do not conform to the pattern of the remainder. In their work fibre strength fell consistently below the line given by equation (4) over a wide yield range. If one excepts the possibility of experimental error, then perhaps the most likely reason is that these authors chose wood samples of high fibril angle. This hypothesis cannot now be

tested other than by repeating the work with fibres of known fibril angle.

THE EFFECT OF PULPING PROCESSES THAT DEGRADE CELLULOSE

Data have been obtained from similar black spruce samples pulped by processes that are expected to degrade the cellulose and are shown in Figure 7. The high-yield bisulphite pulps fall only slightly below the line of Figure 1, but the low-yield acid sulphite pulps fall appreciably below the line. Clearly, cellulose-degrading processes reduce the strength of fibres below their potential. This may be a powerful tool for investigating the usefulness of novel pulping processes. By

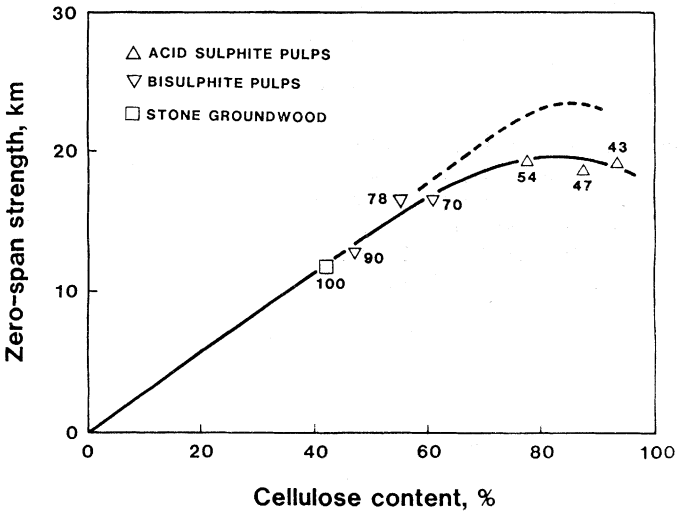

Fig 7—Zero-span strength of well-bonded sheets of stone groundwood, high yield bisulphite and low yield acid sulphite pulps of black spruce, plotted against enzymatically determined glucan content. The figures indicate yield. The dotted line is from Figure 1.

determining the zero-span strength and the cellulose content of a novel pulp, and comparing the plot with values for kraft pulping of a similar wood sample, the extent of degradation of the cellulose can be assessed. Moreover the effect of modifications to the process can be rapidly determined.

The single-fibre data of Figure 8, obtained from the same wood samples as the data of Figure 2, confirm that cellulose-degrading processes yield pulps that fall below the line of the remainder. Three pulps fall into this category, a 63% yield acid sulphite, a 52% yield soda-oxygen pulp and a kraft pulp prehydrolyzed with sulphuric acid.

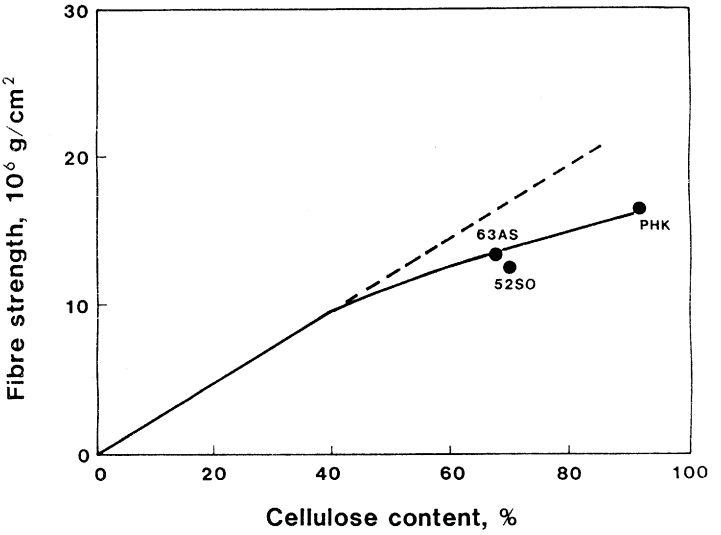

Fig 8—Fibre strength of pulps from the same wood sample as Figure 2. The fibre strengths for these cellulose-degrading processes fall below the dotted line obtained in Figure 1.

63AS - 63% yield acid sulphite
52SO - 52% yield soda-oxygen
PHK - low yield kraft, prehydrolyzed.

CONCLUSION

The problem of fibre strength and chemical composition may be much simpler than it has seemed from the rather complex literature. As long as the fibril angle is low, and the pulping process does not degrade the cellulose, fibre strength is directly proportional to cellulose content up to a value of 80% cellulose. Beyond this value fibre strength is reduced apparently because of the loss of a stress-equalizing matrix. For processes which are degrading, fibre strength falls below this line depending on the extent of degradation.

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Transcription of Discussion

SESSION 1 PULP continued
FIBRE AND SHEET PROPERTIES
Chairman T. Lindström

Strength and Chemical Composition of Wood Pulp Fibres
by D.H. Page, R.S. Seth and F. El-Hosseiny

Dr. O. Kallmes MK Systems, Danvers, U.S.A.

In the 1960's, I was involved with measuring single fibres at St. Regis, and we found large effects of drying tensions on single fibre properties. Would you like to comment on how those kind of results fit in with the work you described today?

Dr. D.H. Page We published an extensive paper on that in 'The Journal of Applied Polymer Science', 19, 1549 (1975). We showed that the major effect of tensile load, during drying on fibre strength, was one of taking out the stress concentrations, particularly the crimps and kinks that are put into fibres during drying. Since all the data we have presented in this paper were for plate-dried sheets of laboratory made never-dried pulps, which contain few kinks and curls and microcompressions, we have avoided this problem.

Kallmes Does that effect really exist? In other words, does drying tension play a large part in sheet properties?

Page Oh, yes certainly it exists. Drying tension removes the microcompressions, kinks, and small-scale curl in fibres and this is what is affecting sheet properties.

A.A. Ibrahim Papyrus Inc., Westerville, U.S.A.

Dr. Page, in some of your earlier articles about the effect of tensile strength and compressive strength of paper, you mention that at low degree of fibre bonding, sheet strength depends on fibre bonding. At a high degree of fibre bonding, then the tensile or compressive strength depends on the intrinsic fibre strength.

Page Correct, I still hold to that.

Ibrahim Now with your findings you showed us that the intrinsic strength of the fibre depends on so many other new factors, so the original statement should be expanded not only on the intrinsic strength of the fibre, but with these new findings. Is that not so?.

Page Both statements hold, there is no problem. It is just that fibre strength is a variable.

R.H. Reeves James River Corp., Neenah, U.S.A.

Were these wet zero span tensile tests that you took on the handsheets because there is an effect of the bonding that takes place also?

Page No. There are two ways in which people have tried to handle the problem of bonding. One is to wet the sheet so that the bonding is reduced to zero. In that case, you measure the zero span strength of wet fibres (if that is what you want to measure). The other way of doing it is to make the bonding so high that zero span strength is not affected by the bonding, and that is what we did. We refined the pulps and measured zero span strength as a function of wet pressing pressure. The zero span strength quoted is the plateau value that was reached.

B. Radvan Wiggins Teape R & D, Beaconsfield, England

When you said low fibril angle, you really meant negligibly low angle. Calculation by Dr. Salmén has shown the difference between zero and ten degrees is negligible. You also showed us an earlier slide where the difference between one degree and ten degrees was far from negligible, something like 30%. Now, assuming that these data are right, two things will follow. First of all, how long are the fibrils compared with the radius of the fibre, i.e. is there an appreciable curvature of the fibril? If there was such a curvature, this would explain this large difference between one degree and ten degrees. If you accept that, the correlation you showed should not be as good as this because it contains anything between zero and ten degrees if I understood correctly.

Page First of all, the single fibre plots which you saw of one degree and twelve degrees were each for a single fibre. As indicated in our own published data, there is no significant difference in strength between fibres of 1^{o} and 12^{o}.

You raised the question of the length of the fibrils; this is an excellent question to raise because for all of the work that we are doing and that Dr. Salmén and his co workers are doing, we need to know the length of the fibrils and we don't know it. If somebody can tell us, then we can use it.

Prof. M.L. Miller Miami University, Oxford, U.S.A.

You made the comment that we now have a good tool to use to explain pulping. I wonder if you could elaborate on that. Secondly, I would like to know if you are doing some work in that area.

Page Secondly, we are doing some work in that area. Firstly, very simply, it follows directly from what I have said. You take a wood sample of low fibril angle and pulp it by the kraft or holocellulose process. You obtain the line through the origin of zero span strength plotted

against cellulose content. For the same wood, you use your new pulping process and obtain its line on this plot. If your data fall on the same line as the kraft or holocellulose line, the new process is non degrading. If it falls below the line, it is a cellulose-degrading process, and the extent of degradation can be assessed. This should be a much better way of assessing new pulping processes than existing methods, such as for example, viscosity.

Dr. D. Reeve University of Toronto, Canada

I thought you were rather too categorical about whether a pulping process was degrading of cellulose and wonder if you measured the viscosity in order to determine the extent of cellulose degradation. My second question is, you have plotted the pre-hydrolysed Kraft point (Fig. 8) as being on a straight line or at least a curving line and yet it is over 80% alpha cellulose. Could it not be plotted otherwise?

Page It could indeed. We haven't done enough work to show what family of curves it belongs to. On the first point, we did not measure the viscosity. It would be interesting to see how our measure of cellulose degradation relates to viscosity and we intend to do this.

Prof. R.H. Atalla IPC., Appleton, U.S.A.

I would like to make two comments. One, in connection with drying under tension. We did some work in 1980 on drying cotton under tension and recently we repeated it drying ramie under tension; the Raman spectra indicated that there is a re-alignment of molecular organisation in the transverse direction as a consequence of that. So there is some kind of aggregation caused by the drying under tension that may contribute. The other is in relation to the hemi-celluloses. Looking at molecular models there are good reasons for believing that many of the beta-1,4 linked polysaccharides are capable of co-aggregating with cellulose on the surface and contributing to the strength.

Page I don't think the hemicelluloses contribute to the tensile strength even though, as we have shown, they are oriented parallel to the fibrils. This is probably because the chain length is too short. You need quite long chains for the shear stresses to build up the tensile load in an oriented molecule and the chain length of hemicellulose is not long enough.

Prof. G.A. Baum IPC, Appleton, U.S.A.

I assume you got your well bonded sheets by wet pressing?

Page Yes, can I check with my co-author, Dr. Seth. That is correct isn't it? (Affirmative)

Baum What would be the effect of refining if you had carried out your experiments at low, moderate and high levels? Would you expect the results to be the same?

Page I think we would expect them to be much the same, would you agree with that Dr. Seth?

Dr. R.S. Seth PPRIC, Pointe Claire, Canada

The pulps were beaten to various levels and sheets made at different pressing pressures. The plateaus in zero span strength were obtained that way.

Baum So they were beaten to a number of different levels?

Seth Yes. They almost reached the plateau values by about 500 CSF.

Baum Could you make any predictions as to what the Z-direction tensile might be based on your model?

Page No, I think you are going to do that tomorrow.

Baum No, I don't think so.

Reeves As you changed the yield, you are changing the coarseness or the weight per unit length very dramatically and the number of fibres that you are testing on your zero span is also changing. Have you tried to normalise any of this data, taking that into account?

Page Well, we look at it from the other way around, that is we consider the fibre strength per unit area as determined from the zero span strength.

Reeves There is a very large difference in the number of fibres involved in the test.

Page The number of fibres is irrelevant. The strength is measured as a specific strength per unit of fibre cross-sectional area.

Dr. R.H. Marchessault Xerox Research, Mississauga, Canada

Dr. Page, could you comment on the relative probabilities of microfibrillar structure being composed of folded or linear chain molecules?

Page I am not sure that our work relates to that specifically, but I do have some comments in response that I can make. When chain folding was first discovered in polyethylene, everybody looked for chain folding in other polymers and they found it. In many polymers, crystals were grown that were chain folded and the automatic reaction was therefore to assume that chain folding must exist in native cellulose. However it is just a historical accident that it occurred that way because many years later, it was shown that extended chain crystals existed in polymers and you can make very strong, stiff fibres with extended chain crystals of many polymers and these are now

commercially available. These have extremely high modulus, and strength and low stretch to break, exactly like native cellulose. If those extended chain polymers had been found before chain-folded polymers were found, I don't think any question would have been raised that cellulose was chain-folded.