DETERMINATION OF PAPER CROSS-SECTION STRESS-STRAIN CURVES USING ZERO AND SHORT SPAN TENSILE MEASUREMENTS

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ABSTRACT
Recently we have developed a new method for measuring the stress-strain curve of the paper cross-section (the stress-strain curve in the limit of the span length approaching zero). The method is to subtract the zero-span displacement-load curve from the short-span displacement-load curve, so as to obtain the displacement -load curve of the free span, which can then be converted into a stress-strain curve. If the span is small enough then this stress-strain curve will be the same as the span at zero length. We have applied this method to measuring the paper cross-section stress-strain curve for three kraft pulps: a never-dried, unbleached pulp; a never dried, bleached pulp and a once dried bleached pulp. Sheets were prepared from the unrefined pulps and after refining to 1000, 3000 and 6,000 PFI revolutions. The cross-section breaking strain of the paper made from the unbleached kraft increased with refining, while the reverse effect was seen with the paper made from the bleached kraft. In all three cases, the sheet cross-section breaking stress increased with the refining.

Keywords: bleached softwood kraft, unbleached softwood kraft, refining, fibre mechanical properties, zero span tensile test, short span tensile test.

INTRODUCTION
There have been a number of analytical models for the tensile strength of paper (1). All of the models agree that for well-bonded papers, the strength of the fibres is one of the critical factors determining the tensile strength of the sheet. Due to the large number of tests required and the difficulty of the tests, it is very tedious to determine an average fibre strength from testing single fibres. Accordingly, the zero-span tensile strength (ZSTS) has often been used (eg (2)) as a measure of single fibre strength. If the fibres are completely straight and free of defects then the measured zero-span strength of an isotropic sheet should be 3/8 of the strength of a paper where all of the fibres are aligned in the stress-direction (3).

One additional advantage of using the zero-span strength rather than an average single fibre strength is that because the zero-span strength is measured on paper the state of the fibres must be the same for both the tensile test and in the zero-span test. To measure an average single fibre strength, the fibres must be separated, dried and then mounted for testing. This process will change the fibre properties. In addition, in a sheet the fibres must conform around each other and so will have both in and out of plane curl. Perez and Kallmes (4) measured single fibre strength and compared it with the fibre strength estimated from the ZSTS for twelve pulps using the theory of Van den Akker (3). They found that the single fibre strength estimated from the ZSTS was 30-55% lower than the measured single fibre strength, a difference that they attributed to a proportion of the fibres being curled and so not bearing load during the test. The ZSTS also often increases after the fibres have been gently refined, as the refining straightens the fibres and removes defects (5).

If we wish to model the whole stress-strain curve of paper rather than just the tensile strength, then a method for measuring the stress-strain curve of the fibres is required. As discussed above, it is preferable if the stress-strain curve of the fibres within the sheet can be measured. We have recently developed a method to do this by modifying a commercial zero and short span tester to continuously measure load and displacement. In this paper, this new method will be applied to the measurement of the fibre stress-strain properties in paper made from several types of pulp. The effect of refining on the calculated stress-strain curves will also be examined. Full details of the new method have been submitted for publication elsewhere (6). However as the method has not yet been published we will first give a brief outline of the new method before showing the results obtained with the new method.

New method for measuring paper cross-section stress-strain properties

At the start of a zero-span test the two sets of jaws are in contact with each other. As the test proceeds an increasing jaw displacement occurs because the applied load is transmitted by friction from the surface of the jaws to the sample. For a given load, a finite span from the edge of the jaws, determined by the paper-jaw coefficient of friction and the applied force, exists before the tensile force in the sample falls to zero. The displacement between the jaws during the test is then due to the straining of this span. For a linear-elastic material, the sample behaves as if a residual span under the jaws is being strained. The residual span depends only on the clamping pressure and the jaw-paper coefficient of friction. Thus for a linear-elastic material the measured displacements could be converted to strains by calculating the residual span. However, if the material under the jaws is not linear-elastic then it can be shown that is not possible to obtain the stress-strain curve of the material from the measured load-displacement data.

The essence of the new method is that in a short span test, the measured displacement will be the sum of the displacement from under the jaws and the displacement for straining the free span. Thus by subtracting (at the same load) the zero-span from the short-span displacement, the displacement due to straining the free span at that force can be obtained. Repeating this procedure over the whole force range of the test yields a complete set of load-displacement data for straining the free span, which can be converted to stress-strain, given that the span is known. The utility of this method is that it eliminates the need to analyse the mechanical behaviour of the sample under the jaws. One inherent assumption of the technique, for which experimental evidence is at the moment lacking, is that the z-direction stress-distribution in the free span is independent of span length. Further analytical and experimental work are required to validate this assumption.

If the strain in the free span in a short span test is ε then it can be shown (6) that the load, F_L is given by

$$F_L = E_p\left(1+(1-c)\frac{32}{9\pi}\frac{G}{\overline{l|_G}}\right)\varepsilon \qquad (1)$$

where E_p is the elastic modulus of the paper in the limit of a free span length of 0, G is the span between the jaws, $\overline{l|_G}$ is the average length of the set of load-bearing elements that are long enough to span between the jaws and c is a factor that accounts for bonding, and is equal to 0 for an unbonded (wet) sheet and 1.0 for a perfectly bonded paper. The term 'load-bearing element' is used rather than fibre length as fibres may be composed of segments, joined at defects such as kinks and twists across which the fibre will be unable to bear any load. If that is the case, then it is the length of these load-bearing elements (or fibre segments) that determines the sheet mechanical properties. If the fibres are completely straight and free from defects, the load-bearing element length will be equal to the fibre length.

If $(1-c)(32/9\pi)\left(G/\overline{l}\big|_G\right)\approx 0$ then $F_L = E_p\varepsilon$ and the stress-strain curve determined by subtraction will equivalent to that at a span of zero. This calculated stress-strain curve will be the stress-strain curve of the paper cross-section and thus independent of the bonding between the fibres. The stress-strain curve calculated in this manner will be related to the stress-strain properties of the individual fibres, although the exact relationship will depend on the orientation of the fibres in the sheet and the number of fibres which do not take up load due to in-plane or out-of-plane curl and other defects. In order to minimise $(1-c)(32/9\pi)\left(G/\overline{l}\big|_G\right)$ it is necessary to have well bonded sheets of paper made of straight fibres, which are long compared to the span, G. However, the span should also not be too small, since the displacement from straining the free span will be directly proportional to the span.

For full details of the derivation see (6, 7).

EXPERIMENTAL

Force-displacement curves were measured on a Pulmac Zero and short span tensile tester. The tester comes with a computer controlled X-Y table, which allows 24 tests to be performed automatically. The width of the test jaws is 25 mm. The force was calculated from the pressure in the piston driving the jaws apart, using the calibration given by the manufacturer. The separation of the jaws was continuously measured by a Kaman Multi-VIT (Multi-purpose Variable Impedance Transducer). This is a contactless displacement transducer, which was attached to the moving jaw and provided a measurement of position relative to an aluminium target attached to the stationary jaw. The Multi VIT was statically calibrated against a dial gauge. Test data was continuously recorded using PicoLog v5.05.1, and a 12-bit A-D card. The data was collected at a sampling rate of 10 ms, but to reduce noise data was recorded as block averages of five data points.

The following pulps were measured

- Never dried unbleached softwood kraft (A)
- Never dried bleached softwood kraft (B)
- Once dried bleached softwood kraft free dried in sheets from pulp B, reslushed and formed into handsheets (C)
- TMP, 120ml CSF (D)
- TMP, 54ml CSF (E)

The two TMP samples were taken from SCA's Ortiviken mill. Handsheets were prepared from the two TMP pulps only from the as-received pulps. The TMP samples did not prove suitable for obtaining stress-strain curves by subtraction due to the low breaking loads and small displacements. The results from these samples will not be further discussed.

The three kraft pulps were from SCA's Östrand mill. Four sets of samples were made for each of the kraft pulps. One set was made from the pulp as it was received from the mill and three other sets were made after refining the pulps in a PFI mill to 1000, 3000 and 6000 PFI revs. Handsheets were made under standard conditions except that drying was performed between teflon sheets, which allow the sheets to freely shrink. For each sample, 24 tests were conducted at zero span and at spans of 50, 101, 160 and 300 μm. In order for the subtraction technique to be applied successfully, an average curve for all 24 tests in each data set must be calculated. To do this, a spline function was fitted to. To remove the effect of load-takeup, the maximum slope of each spline fitted curve was determined by linear regression. All data below this point of maximum slope were disregarded. The tangent at the point of maximum slope was then extrapolated to find the displacement at 0N force, with the displacement determined in this manner then subtracted from all other displacements. This removes both load take up effects as well as the initial span of the test, if any. Both zero and short span curves corrected in this manner then have a common starting point at 0 μm displacement. After removing the load take-up, each curve was then represented as fifty points, equally spaced in displacement. An average curve for the whole data set was then constructed by averaging all 24 curves for each of the fifty points.

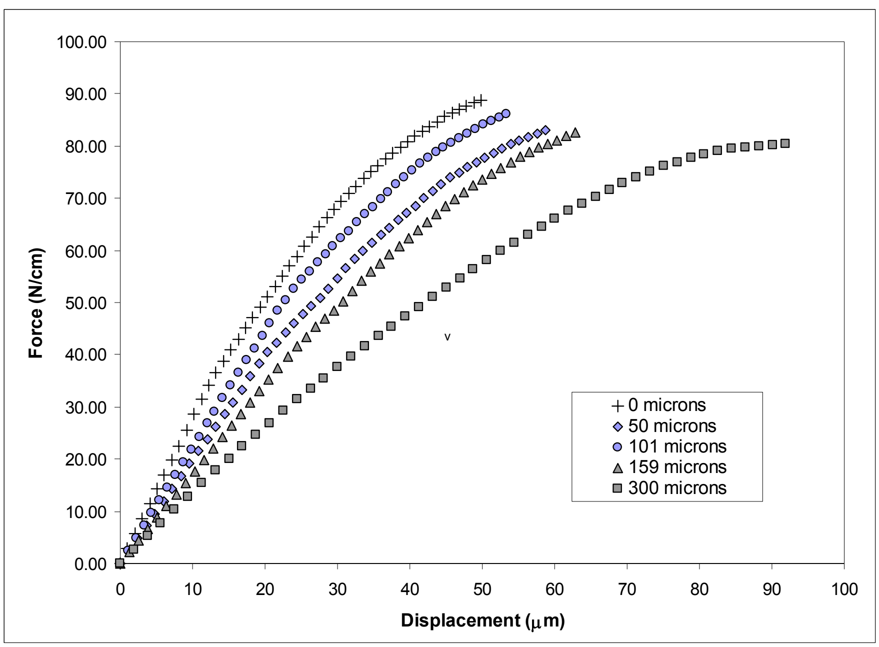

Fig 1. Average load-displacement curves determined for tests on sample B refined to 1000 PFI revolutions. The legend indicates the short span at which the test was conducted. The zero microns data is for the zero-span test.

RESULTS

Fig. 1 shows the zero and short span load-displacement curves measured for the sheets made from the never dried bleached pulp (B) refined to 1,000 PFI mill revolutions. The load-displacement curves are mostly as expected, with the zero-span curve having the least displacement for a given load and the 300 μm span curve showing the most displacement. This is as expected because the displacement from straining the free span should be directly proportional to the span. The one discrepancy is that the 50 and 101 μm span curves are reversed from their expected order. This discrepancy probably arises from the uncertainties in determining an average load-displacement curve. Some of these uncertainties are discussed later in this paper.

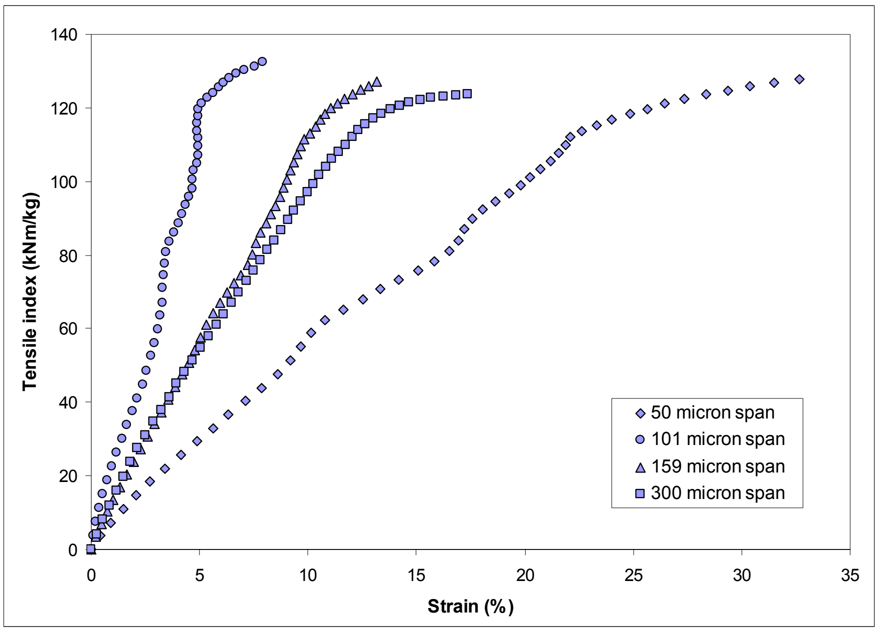

Fig. 2 Stress-strain curves determined for sheets made from bleached pulp B refined to 1000 PFI mill revs.

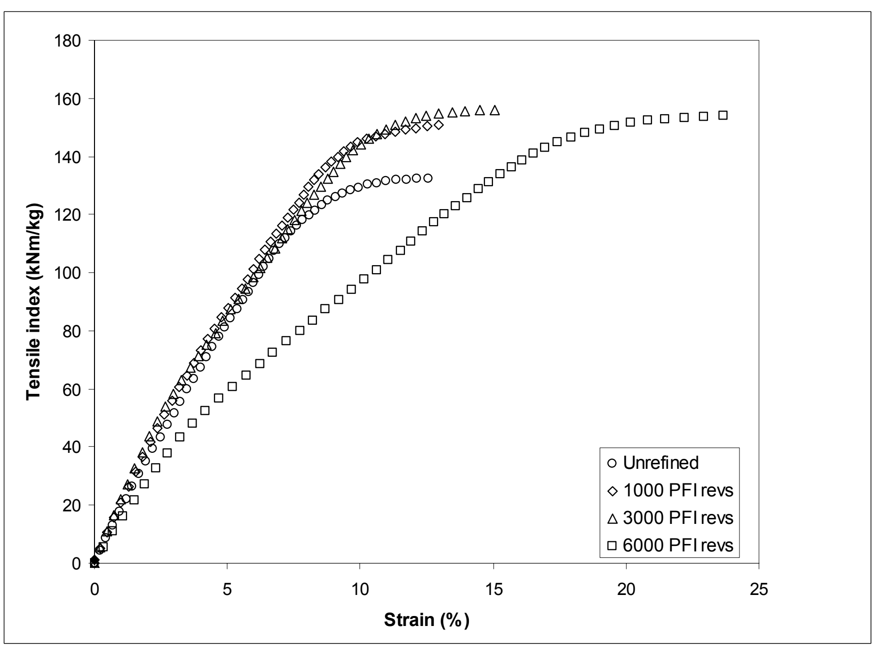

Fig. 3. Cross-section stress-strain curves of paper made from unrefined and refined unbleached softwood kraft pulp (A)

Fig. 2 shows the four curves that were determined by the subtraction of the zero-span displacement-load curve from the corresponding short span displacement-load curve. Theoretically, all the curves should be coincident. It can be seen in the figure that this is not the case. While the stress-strain curves obtained by subtracting from the 159 and 300 μm spans are reasonably close together over the whole range of strain, the 50 and 101 μm curves diverge from these two curves. This reflects that when the span is small, the difference in displacement, at a given load, is also small, affecting the accuracy of the subtraction. Accordingly, the most accurate determination of the stress-strain curve will be when the zero-span curve is subtracted from the 300 μm span curve, because these two curves will have the largest difference in displacement.

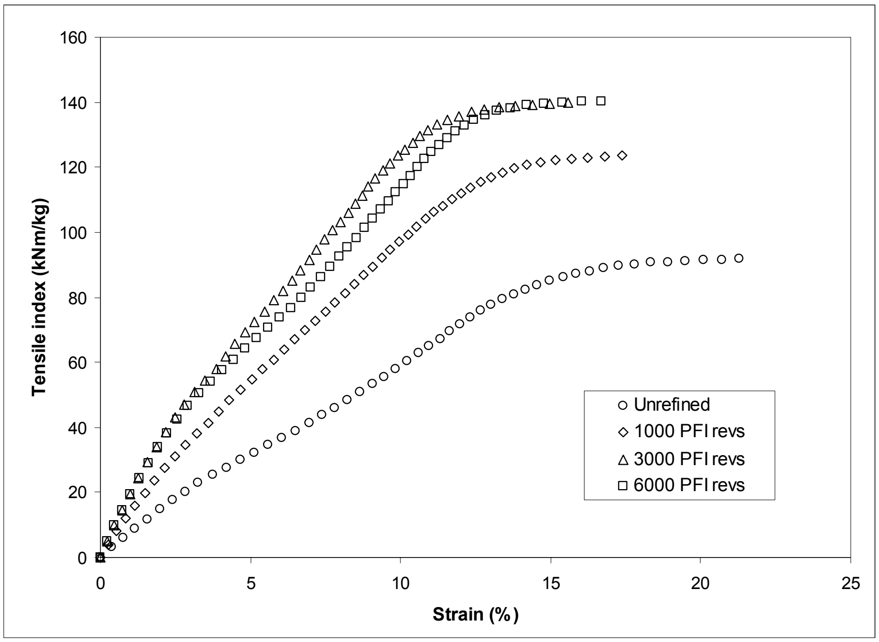

Fig. 4. Cross-section stress-strain curves of paper made from unrefined and refined bleached softwood kraft pulp (B)

When considering the reasons, as to why these curves are not coincident, it is important to recognise that each of the curves is an average of 24 measurements, which were measured on three small disks, each of 6.25 cm diameter. If there is systematic strength variation in the samples at this length scale, then this may bias the results. Variation could occur in both machine and laboratory papers from grammage variations. For such samples it may be necessary to make more measurements to improve the accuracy of the fitted average load-displacement curves. These sorts of errors are most significant when the subtraction is attempted at smaller spans.

Effect of refining on paper cross-section stress-strain curve

Figs. 3-5 show the effect of refining on the three kraft pulps. In generating these figures, the curves determined by subtracting the zero-span curve from the 300 μm span curve were used, as these will provide the most reliable data. When these figures can be examined, some very interesting trends can be observed.

The stress-strain curves for the sheets made from the unbleached pulp (Pulp A) are shown in Fig. 3. In this figure, the cross-section stress-strain curves for the sheets made from the unrefined pulp and from the pulps refined to 1000 and 3000 PFI revs are linear-elastic until shortly before fracture, with the same modulus. The yield stress is approximately 120 kNm/kg for the sheets made from the unrefined pulp and 140 kNm/kg for the sheets made from the pulp refined to 1000 and 3000 PFI revolutions. The tensile index at fracture increases after refining for 1000 PFI revs, compared to the sheets made from the unrefined pulp, and then remains constant with increased refining. This increase is probably due to the refining process straightening fibres, and removing defects, thus increasing the number of fibres bearing load at fracture and increasing the strength (5). The stress-strain curve for the sheets made of pulp refined to 6000 PFI revs shows a much lower modulus than the sheet made from the other three pulps as well as an apparent yield stress around 40 kNm/kg. The fracture strain was calculated to be 23% compared to 12% for the unrefined sample.

When Figs. 4 (Pulp B- bleached pulp) and 5 (Pulp C- bleached pulp, dried and reslushed) are examined it can be seen that they both display similar trends. In both cases, the breaking strain is higher for the sheets made from the unrefined pulp than it is for the sheets made from the refined pulp. The breaking strain for the unrefined pulps of both samples is around 21%. After refining, the cross-section breaking strain of the sheets made from pulp B has been reduced to 17%, irrespective of the level of refining. The cross-section breaking strain for the sheets made from the same pulp after it has been once dried (Pulp C) shows a very similar trend with the breaking strain of the sheets made from the pulps refined to 1000 and 3000 PFI mill revs also coming to 16%. The breaking strain of the sheets made from the most heavily refined pulp fell even further to around 10%. In addition, the refining can be seen to have greatly increased the tensile index at fracture for sheets made of both pulps. In the case of the sheets made from the once-dried pulp C, the effect of the refining was to double the tensile index at fracture.

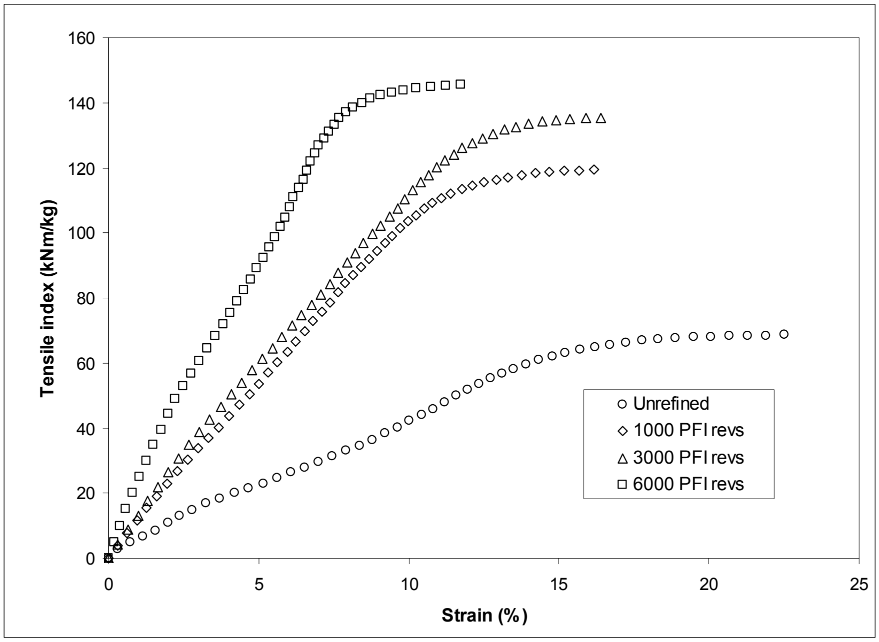

Fig. 5. Cross-section stress-strain curves of paper made from unrefined and refined previously dried, bleached softwood kraft pulp (C)

We wish to emphasise that the results in Figs 3-5 should not be generalized beyond the samples tested here. The sheets of paper tested here were not restrained during drying and it well known that the level of drying restraint has a strong influence on the stress-strain properties of the fibres.

At this juncture an explanation of the differences between the unbleached and the bleached pulps must be somewhat speculative. Certainly in the unbleached pulp much more of the lignin matrix will be intact than in the bleached pulp, producing a stiffer fibre than for the bleached pulp. The effect of extensive refining may then be to help break up this lignin matrix, increasing the ductility of the fibre. Such a mechanism will not be as important for the bleached pulps with their reduced yield. Instead the refining process may have plastically strained the fibres, increasing their length and reducing their ductility. Certainly the data seems to suggest that refining bleached fibres will do very little to improve their reinforcement capability, since the ultimate stretch of the fibres will have been reduced. It should be noted that single fibre tests on previously dried fibres shows similar results (8), in that after the fibres had been put through an initial loading cycle and plastically deformed, then the stiffness in the subsequent loading cycles was approximately double. It is clear that much more work will need to be done before all of the results presented here can be fully understood.

CONCLUSION

A new method has been developed for measuring the stress-strain curve of the paper cross-section (the stress-strain curve in the limit of the span length approaching zero) by subtracting an average zero-span displacement-load curve from a short span displacement-load curve. The new method was used to measure the effect of refining on cross-section stress-strain characteristics for sheets made from several commercial Scandinavian softwood krafts. The breaking strain of the sheets made from an unbleached kraft pulp increased with refining, as did the fracture stress. The breaking strain of the sheets made from a bleached, never-dried and bleached, dried pulps, decreased with refining, but the fracture stress increased.

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