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Effect of Drying on the Flexural Rigidity of Single Fibres

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THE importance of fibre flexibility to the consolidation of the paper web and to sheet formation has long been recognised and a number of methods have been developed to measure fibre stiffness.[1–4] The fundamental resistance of fibres to flexural deformations is given by the flexural rigidity EI, the product of modulus of elasticity and moment of inertia. The methods of Seborg & Simmonds[1] and Forgacs *et al.*[2] do not yield flexural rigidity values, that of Nethercut[3] requires fibres of 7–8 mm length and is therefore unsuitable for wood fibres; only the method of Samuelsson[4] has been successful in providing a quantitative measure of flexural rigidity. Duncker *et al.* (in the paper presented at this symposium) have used the Samuelsson method in studying the effect of drying on the flexural rigidity of fibres. It is the purpose of this contribution to describe a new method for measuring the flexural rigidity of fibres and to present some data on the effect of drying on flexural rigidity.

The method is based on the work of Seborg & Simmonds[1] and is illustrated schematically in Fig. 1. Fibres are erected as cantilever beams and a concentrated load is applied near the free end through a quartz helix spring. Loads are determined by measuring the spring extension and the span and the deflection of the fibre are measured microscopically. A series of readings of load and deflection are taken and are plotted as shown in Fig. 2. The flexural rigidity is determined from the slope of the linear portion of the load deflection diagram by taking a convenient load and associated deflection measurement and calculating the flexural rigidity from the equation for the deflection of a cantilever beam under a concentrated load—

$$EI = Pl^3/3\delta$$

where P is the load, l the span and δ the deflection at load P.

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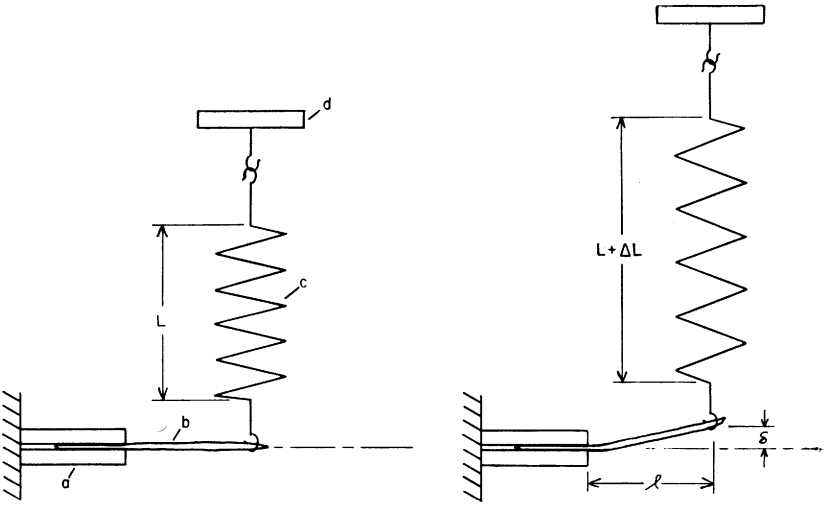

On the left, the unloaded system is shown with fibre clamp (*a*), fibre (*b*), quartz helix spring (*c*), and movable head (*d*); on the right, the system is shown after load has been applied. The deflection δ is measured at increasing levels of load as determined from the spring extension ΔL, which together with the span *l* yield the data for calculating the flexural rigidity.

Fig. 1—Schematic drawing of method for determining the flexural rigidity of fibres

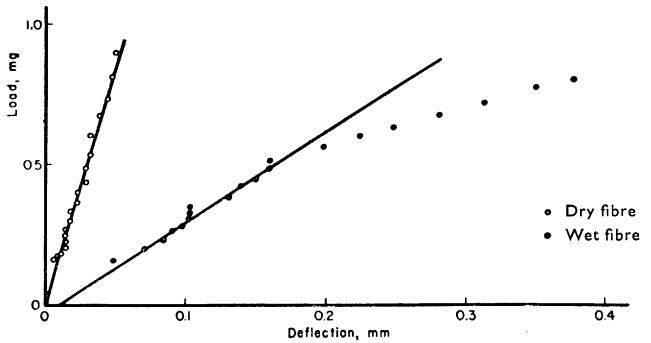

Fig. 2—Load deflection graphs for a beaten summerwood fibre—deflection values for the wet fibre were adjusted to equivalent values corresponding to a span identical with that used in the dry test

So far, measurements have been made with a temporary apparatus. Details for a more permanent apparatus, which will lend itself to a more efficient gathering of a large number of data, are at present being worked out. Tests on dry fibres have been made by conditioning and testing the fibres in a standard paper testing room (72°F and 50 per cent rh). Wet fibres were tested by submerging them in water, but, in view of the fact that Seborg *et al.*[5] had to correct the calibration of their quartz springs at high relative humidities, the springs were kept in air. This caused some difficulties with surface tension where the end of the spring passes through the surface of the water, which were remedied by the application of a wetting agent at that point. It is possible that deflections observed by Seborg *et al.* were due merely to the weight of water adsorbed on the springs and, if it can be ascertained that the spring constant itself does not change under these conditions, future tests will be made by submerging both fibre and spring in water.

Some measurements were made on white fir (*Abies concolor*) kraft pulp fibres. Both springwood and summerwood fibres were measured, some of which were taken from unbeaten pulp (715 CSF), others came from pulp that had been beaten in a Valley laboratory beater to 380 CSF. The fibres were first tested dry in equilibrium with 72°F and 50 per cent rh, then rewetted and tested under water. Care was taken that the dry fibres were loaded just high enough to obtain a number of points sufficient for determining the slope of the load deflection diagram, but to keep the load below the limit of proportionality to avoid permanent damage. The wet fibres were generally loaded until the hook of the spring slipped off the fibre, which occurred usually after the load/deflection graph had gone well past the proportional limit. Fig. 2 shows the load/deflection graphs in the dry and the rewetted state of a beaten summerwood fibre. The spans used were generally between 1 and 2 mm. Since it was not possible to achieve identical spans for dry and wet tests on the same fibre, the deflections of the wet test in Fig. 2 were adjusted to a span identical with that used in the dry test—that is, 1.1 mm. The difference in slopes therefore is directly proportional to the difference in flexural rigidity in the wet and dry states. The graphs in Fig. 2 are fairly typical; some of the curves for data from other fibres are more regular and others somewhat less.

Some results are shown in Table 1. The variability of the test is very large. Two of the groups show a standard deviation that is larger than the mean. The flexural rigidity values depend to a great extent on the size of the fibres, since the moment of inertia is one of the factors. For a circular rod, for instance, the moment of inertia is directly proportional to the fourth power of the diameter. It might therefore be expected that there would be a correlation between flexural rigidity values in the dry and wet states because both of these were obtained on the same fibre. Inspection of the values in Table 1 shows

that there is some tendency in this direction, particularly in the wet, beaten springwood fibres. For this group, the correlation is significant at the 95 per cent confidence level. Table 1 shows an estimate of the number of fibres that would be required to obtain sample means within ± 10 per cent of the population mean at the 95 per cent confidence level. In most cases, this is between

TABLE 1—FLEXURAL RIGIDITY OF WHITE FIR KRAFT PULP FIBRES

	Summerwood fibres				Springwood fibres			
	Unbeaten		Beaten		Unbeaten		Beaten	
	Dry (*mg mm*²)	*Wet* (*mg mm*²)	*Dry* (*mg mm*²)	*Wet* (*mg mm*²)	*Dry* (*mg mm*²)	*Wet* (*mg mm*²)	*Dry* (*mg mm*²)	*Wet* (*mg mm*²)
	4.84	0.85	1.11	—	0.93	—	0.537	0.061
	6.98	2.88	1.81	—	1.14	0.18	0.659	0.056
	7.66	1.03	4.35	1.29	1.19	0.37	0.833	0.022
	8.13	—	4.69	0.58	1.46	1.34	1.38	0.151
	8.53	—	6.53	0.46	2.58	0.18	1.98	0.375
	8.65	0.05	6.59	—	3.06	0.71	2.56	0.215
	11.08	0.003	7.01	1.14	3.52	2.04	2.84	0.494
	12.81	0.79	8.32	—	4.90	1.50	3.18	—
	14.38	1.34	9.41	1.64	5.00	1.29	3.20	0.370
	14.75	5.03	9.64	2.61	5.82	0.97	4.31	1.25
	15.25	2.60	10.23	3.94				
	18.70	4.03	15.11	3.19				
	21.96	0.39	17.99	3.59				
	22.41	8.96	22.05	1.38				
	28.98	1.76						
	31.72	7.38						
Average[1]	15.73 (14.80)	2.65	10.70 (8.92)	1.98	3.19 (2.96)	0.95	2.03 (2.15)	0.33
Standard deviation[2]	8.01	2.78	5.95	1.26	1.81	0.65	1.28	0.38
Percentage of dry values[3]	100	17	100	18	100	30	100	16
Percentage of unbeaten[3]	100	100	68	75	100	100	64	35
No. of tests required[4]	102	427	173	157	147	178	138	511

[1] Average based on paired dry and wet values; the averages of all dry tests are shown in parentheses

[2] Based on all available values

[3] Based on averages from paired values only

[4] Number of tests required to estimate population mean within ± 10 per cent at the 95 per cent confidence level

100 and 200, but two groups gave larger numbers. The order of magnitude of the variability seems to be about the same as that found by Samuelsson.[4]

In spite of the large variability and the somewhat limited number of tests performed, there is no question that there are substantial increases in flexural

rigidity upon drying. This applies to both springwood and summerwood fibres and to beaten and unbeaten fibres. The order of magnitude of the drying effect agrees quite well with that found by Duncker *et al.*

In the dry state, the summerwood fibres show substantially higher flexural rigidity values than do the springwood fibres. In the wet state, the difference is much less, but seems to be consistently present. This applies to both the beaten and the unbeaten fibres.

The effect of beating seems to be most pronounced in the wet springwood fibres, but all groups show reductions in flexural rigidity values after beating, the reductions ranging 65–25 per cent. For the dry summerwood fibres, the effect of beating was statistically significant at the 5 per cent level, but for the other groups the effect was not significant. An analysis of variance using all the results might have shown a generally significant effect of beating, but this could not be done, owing to the evident non-homogeneity of variance. Samuelsson found larger reductions,[7] but, since the pulps and the method of beating were different, the results are not directly comparable.

There seems to be some question about the validity of the solids content values plotted in Fig. 6 (page 536) of the paper by Duncker *et al.* Sharp increases in fibre stiffness occurred between solids contents of 15 and 35 per cent, which would correspond to moisture contents (based on oven-dry weight) of 567 and 186 per cent, respectively. The fibre saturation point of pulp is generally between 25 and 30 per cent and of wood between 21 and 33 per cent moisture content,[6] representing a range of solids contents of 75–86 per cent. Above the fibre saturation point, wood does not shrink nor show any changes in elastic properties with changes in moisture content and there is no reason to believe that pulp fibres should show a fundamentally different relationship to moisture content. This suggests that the fibres of Duncker *et al.* were subjected to further drying between the time they were removed from the webs of given moisture content and the time the stiffness tests were made. Their fibres were tested under silicone fluid, a hydrophobic substance. That a hygroscopic material can be dried in a hydrophobic substance is attested to by the fact that wood can be dried by boiling it in oil. Although this is an extreme example, the quantity of water vapour that must be removed from single fibres to dry them is so small that this might very well have taken place in the experiments of Duncker *et al.*

Further work is planned to study the flexural rigidity of fibres in equilibrium with a range of relative humidity values, for which the method described here should be very well suited.

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Discussion

Chairman—Van den Akker and his co-workers have shown a considerable change to occur in the property of the fibres when they are alkali-extracted, which they attribute to decrease in hemicellulose content. If you treat a fibre with 8 per cent potassium hydroxide, is not a considerable proportion of the native cellulose converted to cellulose II? Could not the onset of mercerisation be more responsible for the effect than that portions of hemicellulose are extracted?

Dr J. A. Van den Akker—Spiegelberg observed that the 002 diffraction peak was not disturbed by treatment with 8 per cent potash solution, thus showing that the cellulose remained cellulose I. He was very concerned, of course, with both this and the problem of degradation, which he handled as described in the paper.

It is difficult to resolve the differences between the data of Duncker, Hartler & Samuelsson and ours (as someone has asked), because the bases were different. In the Swedish work, the fibre was quickly extended a fixed distance, whereas in our laboratory the fibre was subjected to a fixed load and so permitted to extend. When a fibre of helical structure is dried under constant load and is free to extend, the structure after drying is different from that of a fibre dried under fixed strain and would have different properties. Without a doubt, a complete resolution of the two sets of results must await more information on the visco-elastic properties of wet and dry fibres.

Chairman—Indeed, the trouble is that we do not understand the background of visco-elastic properties of any material, whatever it is, so the problem is really of quite a magnitude.

Dr B. Leopold—Van den Akker referred to the work that McIntosh and I have done as being along the same lines as his own. What I have to say refers to the Chairman's question on the effect of alkali extraction. Our results show that the most drastic effect of extraction on fibre strength occurs at the lower end of the concentration range; in other words, long before cellulose II has

a chance to appear. We observe no change once the concentration range is reached when mercerisation is likely to occur, which I think supports what Van den Akker said. We have been bolder than he in trying to attribute the changes to specific fractions of the hemicellulose and think that the removal of xylan is mainly responsible for the fall in fibre strength.

Dr C. A. Jentzen—Referring first to the Chairman's question, after extracting cotton fibres with the same treatments used for the pulps, an increase in the modulus occurred, whereas extraction of the pulp caused a decrease in modulus. Then, additional experiments were run to confirm the results of sudden extension at the start of drying. Measurements taken at several span lengths gave the same percentage elongation, which indicated no fibre slippage in the clamping jaws. The exact time of extension and its amount were predictable and reproducible.

Prof. H. W. Giertz—We have to keep in mind that the hemicellulose, which is supposed to be located between the microfibrils, is plastic when wet, but stiff when dried. Thus, the dried fibre is composed of two solid phases—the anisotropic microfibrils (which are extremely strong in their length direction) and the more or less isotropic hemicellulose. When the fibre is loaded, the microcompressed areas will elongate and the hemicellulose in these parts will be broken down. This flow elongation will proceed until the microfibrils are straightened. With this approach, the flow properties of paper should be referred to the irreversible microcracking of the interfibrillar hemicellulose material.

Mr D. H. Page—Had Giertz not commented as he did a few minutes ago, I would probably have said exactly the same thing, except that I would have left out the word hemicellulose.

I would like to refer to Fig. 4C of the paper on load elongation properties of fibres by Kallmes & Perez and to the comment on it in the text—'Quite frequently, fibres picked from handsheets underwent a small plastic deformation somewhere in the middle of the curve (Fig. 4C). This may have been caused by a removal of microcompressions or straightening of the fibres . . .' Now, if we have a fibre with a microcompression in it and it gives under test a stress/strain curve like that of Fig. 4C, this implies that the stress/strain curve of the microcompressed region of the fibre would be quite similar to the curve for paper, having an initial elastic region followed by an appreciable plastic region. There would seem to be a case for the view that we expressed at Oxford that the whole stress/strain curve of a paper containing appreciable

microcompressions can be explained by the stress/strain curve of microcompressed fibres and that the random structure of paper merely acts to produce an averaging effect.

Mr J. A. S. Newman—In the investigation of the stress/strain characteristics of fibres, we appear to be at the same point we were ten years ago in the investigation of the stress/strain characteristics of paper. The explanation of paper strength properties is not being validly based on the statistical geometry of the fibre network. Equally, I believe that the theory of the strength properties of fibres should be based on the concept of the statistical geometry of a network of fibres or of microfibrils inside the fibre itself, which are cross-linked or bonded together at discrete points.

Mr P. A. Tydeman—I do not want to appear ungracious after the extensive reference to microcompression made by Kallmes & Perez, but I have one comment to make on the cross-direction or transverse modulus of fibres, which has obvious importance. I think that there is an anomaly. The argument is that, by bonding other fibres at random to your tensile test fibre, its modulus is doubled. The implication that at least as much fibrous material again is bonded to the test fibre seems unlikely upon consideration of your method. Can this apparent anomaly be explained?

Prof. B. G. Rånby—May I first refer to the question of visco-elasticity. I am not as pessimistic as the Chairman about the state of knowledge of visco-elasticity in polymer systems: I think the theory is in good shape for amorphous polymers. For partly crystalline polymers, the interpretation is more difficult. For papermaking fibres, the problems are even more difficult, because the virgin wet fibres are thixotropic.

I would like to make one remark about the properties of papermaking fibres by referring to the *Summing up* at the Oxford symposium in 1961—published, but not much observed. There is accumulating evidence that the hemicelluloses are fairly well ordered, in the native fibres, although they are *not* X-ray crystalline. It can be shown that the hemicelluloses carry part of the load when the pulp fibres from wood are stretched or dried under tension. There are close relationships between the cellulose microfibrils and the hemicelluloses—for example, well-ordered hydrogen bonds. Our recent NMR data gives support to this concept and they will be published elsewhere.

Dr O. J. Kallmes—First Tydeman's comments. The measurements consisted of taking a small section out of a thin sheet, then one fibre from the

sheet, with numerous fibres bonded to it, was strained to a point below failure and relaxed. All the fibres were carefully pulled off and its stress/strain curve measured. The fibre was not completely bonded, but there were several fibres almost parallel to that being strained. This showed whether or not this technique would be sensitive to fibres bonded to it.

In reply to Hudson's comment, fibre properties varied throughout the sheet to a far greater extent than is generally realised. This is what I was referring to.

The curves referred to by Page are purely speculative so far as curves in paper go. We have never measured stress/strain curves of fibres in paper, though we have developed a technique of measuring the size of strains in paper and found the variation of strain along a fibre to be very marked. Some stretch tremendously, some parts do not stretch at all, so it is from this idea that I drew this curve. There are many ways you could draw the curve and Fig. 4C was purely speculative. These are the only two that have been measured and they were measured on fibres in air.

It is going rather far to say that microcompression explains the stress/strain behaviour of paper. This picture does not take into account, for example, bond failure, nor does it take into account the parts of the fibre that are stiff and straight. In other words, in a sheet dried under tension in this direction, fibres lying in this direction did not shrink and there are no microcompressions.

Chairman—It is easy to speculate. May I remind you that, about 15 years ago, Eyring and a few others were working on stress/strain properties of different fibres. Some types of wool exhibited unusual kinks in their stress/strain curves. Within half a year, Eyring & Halsey produced a beautiful absolute reaction kinetic theory that explained this new feature. It was found that the diameter of this specific form of wool fibre was not uniform.

We are dealing with natural fibres, which are very complicated in their build-up; when we measure stress and strain on single fibres, we are dealing with something that is rather poorly defined material geometrically. It is probably easier to make theories than to make observations. We have many new observations today and I think the future will bring more.

Mr L. G. Samuelsson—In preparing our specimens for fibre stiffness measurements, thin, wet paper sheets were dried in a desiccator to different solids contents by varying the time. It is important to remember this, because no conditioning of the specimens occurs under these circumstances. The drying conditions could rather be compared with those present on the paper-machine. Since we cannot expect an even moisture content in the fibre material, the outer fibre surface, which is most efficiently exposed to the drying

medium, can very well have a high solids content, even if the inner parts of the fibre are still wet. We believe, therefore, that the increase in fibre stiffness during drying can occur within the range 15–35 per cent solids content, since it is the outer layer of the fibre that most probably determines the stiffness of the fibre.