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# LOAD/ELONGATION PROPERTIES OF FIBRES

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***Synopsis***—The goal of this study was to gain a greater understanding of the changes in the mechanical properties of springwood fibres during the consolidation of the web. It was found that the elastic modulus of freely dried fibres $E$ is essentially tripled by any axial load applied during drying. Their tensile strength $t$ is increased and their stretch $e_f$ is decreased in proportion to the loads applied during drying up a twofold change. The elastic modulus measured at 90° to the fibre axis $E_\perp$ was found to be about 0.1 $E$. These fibre properties are of paramount importance to the stress/strain properties of paper, which are described quantitatively in terms of the fibre properties and sheet structure in our second contribution to this symposium.

*Nomenclature*

| | |
|---|---|
| $E$ | = axial elastic modulus of fibre, g/cm$^2$ |
| $E_\perp$ | = elastic modulus of fibre at rightangles to its axis, g/cm$^2$ |
| $K$ | = fraction of fibre strength to which a fibre is preloaded |
| $L_d$ | = load developed in a fibre after wetting and drying at constant elongation, g |
| $L_e$ | = expected failure load of a fibre, g |
| $L_q$ | = load developed in a fibre after 2 min at constant elongation, g |
| $L_w$ | = load developed in a fibre wetted at constant elongation, g |
| $S_e$ | = failure stress of a fibre, g/cm$^2$ |
| $t$ | = failure load of a fibre, g |
| $w$ | = weight per unit length of fibre, g/cm |
| $\alpha$ | = cross-sectional area of fibre, cm$^2$ |
| $\gamma$ | = density of cellulose, g/cm$^3$ |
| $\lambda$ | = fibre length, cm |
| $\omega$ | = fibre width, cm |
| $\delta$ | = fibre thickness, cm |

## *Introduction*

AT THE 1961 symposium, we presented the results of three studies whose overriding theme was that the lack of an understanding of paper structure was the most serious gap in paper technology. We believed that, once the structure of paper was well understood, it should be possible to formulate and

verify theories of many of its properties. We now realise that an understanding of paper behaviour requires far more information. By early 1963, we had combined a greatly improved model of paper structure[1] with a uniform strain theory of paper.[2] The theory predicted and limited data substantiated that the elastic modulus of idealised randomly formed sheets $Y$ formed from fibres with a constant modulus $E$, is not greatly affected by large changes in their degree of bonding.[3] As $Y$ can be greatly altered by purely mechanical treatments of the fibres or wet webs, we concluded that variations in $Y$ are due primarily to changes in $E$. This conclusion led to the concept that the mechanical properties of paper must be dependent on fibre properties to a far greater extent than is generally realised. (By fibre properties, are meant their actual stress/strain curves, not their flexibility as it affects the structure of the sheet.) Furthermore, it appeared that these fibre properties may be altered to as large—or possibly larger—extent by the tensions applied during drying as by beating. Therefore, a study was initiated in early 1963 to study the effects of drying under tension and other similar treatments on the load/elongation properties of single fibres.

It turned out that Jentzen[4] came to the same conclusion about fibre properties at the same time as we did. Thus, two quite similar programmes were carried out simultaneously at separate locations.

With a greater understanding of fibre behaviour as the project's principal objective, we decided to study the fibres of a single pulp in depth rather than those of several under limited conditions. We chose a never-dried, unbleached kraft pulp commercially prepared from a 50/50 mixture of western Canadian spruce and pine. Unbeaten fibres (0-level) and fibres beaten 90 min (90-level) in a Valley beater were tested. This particular pulp was selected principally because its thin-walled fibres readily dried down to a flattened ribbon. Such a fibre's cross-sectional shape can be approximated reasonably by a rectangle whose dimensions are readily measured under a microscope.

In mid-1964, we developed a new theory for the load/elongation behaviour of paper. This theory utilises the bonding-state model of paper,[5] the uniform strain theory of materials as first applied to paper by Van den Akker,[2] the load/elongation properties of fibres and the shear strength of interfibre contact areas.[6,7] Curves calculated for a considerable number of handsheets used in earlier studies were so close to the measured ones that we decided to present the theory in rudimentary form at this conference in our second contribution.

### *Experimental programme*

***Axial properties of fibres***—The experimental programme was largely carried out on the Instron tensile tester, using a special set of jaws whose

construction and operation are described in appendix 1. All testing was done at the constant rate of elongation of 0.0508 cm/min at 72°F and 50 per cent rh.

Determining the effects of drying fibres under different axial tensions on their axial load/elongation properties required that the expected failure load $L_e$ of each fibre be predetermined nondestructively. To calculate $L_e$, the mean failure stress $S_e$ of each type of fibre was measured first. Then, in the main experiments, $L_e$ was found from the equation—

$$L_e = S_e \, \delta\omega \qquad . \quad . \quad . \quad . \quad (1)$$

The fibre width $\omega$ was measured by a micrometer eyepiece and the double fibre wall thickness $\delta$ was calculated from the retardation colours observed when a fibre was viewed between crossed nicols and a quarter wave retardation plate (see elsewhere for experimental details[9]).

The fibres were tested by the cycles of the six programmes shown in Fig. 1 —for example, in one part of the second programme, each fibre was first strained to $0.5L_e$, held at constant elongation while the load came to an apparent equilibrium in about 2 min, wetted and allowed to dry, then unloaded and, finally, after another minute, strained to failure. Such testing sequences made it possible to observe many of the effects on individual fibres rather than from averages obtained on batches of fibres.

In all the programmes, the fibres were removed from a dilute suspension with a needle and dried freely before being mounted on tabs. In the first programme, the fibres were dried initially in two additional ways. These were (*a*) passing individual fibres through the TAPPI sheetforming process and drying them under restraint on a steel plate and (*b*) picking fibres from 60 g/m$^2$ handsheets dried freely or under restraint. The fibres picked from the handsheets were easily removable from surfaces that had been prepeeled with adhesive tape.

The six testing programmes (Fig. 1) were as follows—

*1.* This simply measured the load/elongation curve of fibres, wet or dry (see appendix 1).

*2.* Freely dried fibres were strained *dry* to 25, 50 or 75 per cent of $L_e$, maintained at constant elongation until the load had come to an apparent equilibrium in about 2 min, wetted and allowed to dry still under restraint, then either strained to failure directly (the *not-zeroed* case) or after removing the load momentarily (*zeroed*).

*3.* This was identical to *2*, except that the fibres were initially strained *wet* to 25 per cent or 75 per cent of $L_e$.

*4.* A conventional mechanical conditioning, in which the fibres were strained to $0.5L_e$ in each cycle, except that they were strained to failure in the last cycle.

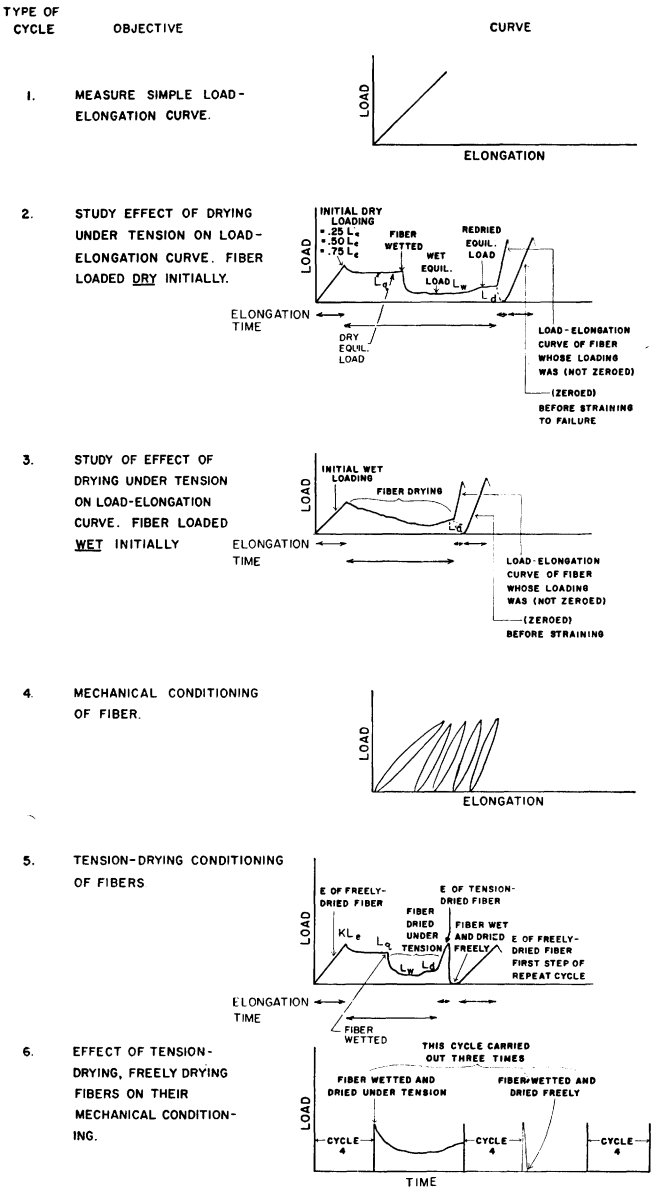


***Fig. 1***—Test cycles

*5.* The fibres were alternately (*a*) wetted, strained to $0.5L_e$, dried under restraint and (*b*) wetted and freely dried to determine if the effects produced by drying under tension were nullified by further wetting and free drying.

*6.* This was a combination of *4* and *5*.

Some of the programmes were repeated with 30 min intervals between steps to test the permanence of certain treatments.

It should be noted that the fibres were not representative of the whole pulp, but more typical of the long fibre fraction because the hole of the mounting tab was 1.6 mm in diameter (see appendix 1). Another bias was introduced into the data by the failure of some fibres during the initial tensioning to $KL_e$ in the wetting/drying cycles. This problem became more severe as $K$ increased. These failures occurred because $L_e$ is a mean value and the true failure load of many fibres is below $L_e$.

Because of the basic similarity between Jentzen's study[4] and large parts of ours, we tabulated his results alongside ours whenever possible. Jentzen worked with separated springwood and summerwood fibres obtained from a single disc of a longleaf pine tree. Both of these samples were pulped by an eight-month holocellulose procedure at room temperature. The much greater uniformity of Jentzen's samples prepared for an academic study resulted in much less scatter in his data than in our study on a commercial pulp.

There was a further difference between Jentzen's study and ours. We worked only with once-dried fibres, as all treatments were applied to the fibres mounted on the Instron tester; this procedure gave us the wide scope of testing conditions of this highly versatile instrument. On the other hand, most of Jentzen's work was carried out on never-dried fibres. He developed an ingenious apparatus and technique for mounting and tensioning never-dried fibres under water before drying them. As it turned out, never-dried fibres did not differ in their properties from once-dried fibres within the precision of Jentzen's techniques.

It is quite common in the testing of materials to measure strain by an instrument other than that elongating the sample—that is, by an extensometer. The purpose of this extra measurement is to check for jaw slippage, which would be particularly serious in the testing of single fibres, because the span under test is about the same size of the 'jaws', the glue spots. We therefore checked the Instron-measured strain of single fibres by determining on double exposures the movement of fibres bonded to one being strained. The type of fibre network tested is shown in Fig. 2. Although it was difficult to obtain good precision in the strain measurements on the photographs, the data in Table 1 indicate no slippage.

TABLE 1—STRAIN MEASUREMENTS OF SINGLE FIBRES

| *Instron-measured strain, cm* | *Strain measured on photographs, cm* | |
|---|---|---|
| | *Operator* 1 | *Operator* 2 |
| 0.0035 | 0.0031 | 0.0035 |
| 0.0035 | 0.0037 | 0.0030 |
| 0.0030 | 0.0028 | 0.0034 |
| 0.0030 | 0.0041 | 0.0037 |
| 0.0040 | 0.0044 | 0.0044 |
| *Average* 0.0034 | 0.0036 | 0.0036 |

### *Cross-direction modulus of fibres*

THE response of fibres to strains applied at rightangles to the fibre axis (the cross-directional properties) is also important to the characteristics of paper. The lack of information on the cross-directional properties is due to the difficulties involved in making such measurements, but an estimate of the cross-directional modulus of fibres can be obtained from the following two experiments.

Craver & Taylor[10] and ourselves in unpublished work have found that the elastic modulus of paper determined by a sonic technique is 50–75 per cent larger than that measured on the Instron tester. On applying the sonic technique to sheets in which the fibres were aligned uniaxially, a measure of the fibres' elastic modulus parallel $E$ and at rightangles $E_\perp$ to the fibre axis was obtained. Sheets with an essentially uniform fibre orientation distribution—90 per cent of the fibres were oriented within 10° of one axis—were formed on a laboratory device loaned to us by Stone & de Montigny.[11]

Another experiment to verify the order of magnitude for the ratio of $E:E_\perp$ was performed as follows. When a single fibre is extended axially in the usual manner with other fibres bonded to it, the load developed at a given strain should be greater than the load that would have developed if no fibres had been bonded to it. (See such a network in Fig. 2.) The contribution of the bonded fibres can be ascertained by first extending a fibre with the others bonded to it to a point below failure (such as $0.5L_e$), then releasing the load and carefully removing the bonded fibres with a pair of tweezers; finally, by measuring the modulus of the now free fibre. This experiment was performed with the bonded fibres in two different orientations—(*1*) random to the axis of the strained fibre (see Fig. 2) and (*2*) at rightangles to it (see Fig. 2). The first type of 'network' was obtained from 2-D sheets, the second by the technique of forming crossings of Schniewind *et al.*[12] using shives. In both cases, about one quarter of the surface of the fibre under tension was bonded.

***Results and discussion***

***Axial properties of fibres***—It was obvious from a cursory inspection of the raw data that the mechanical properties of fibres are highly variable. To determine the validity of statistical tests based on the normal distribution, 17 sets of data—including some of every property—were compared with normal curves with the same mean and standard deviation. Ten were not significantly different according to the $\chi^2$ test; the rest to only a small extent. Therefore, we felt justified in using Student's $t$ test as a test of significance.

Originally, the data were divided into two classes—data on fibres that did not fail at the glue line, data on those that did. Without exception, the differences between the properties in the two classes were negligible, so the data were combined for this paper. It might be of interest that half of 1 020 breaks were at the glue line.

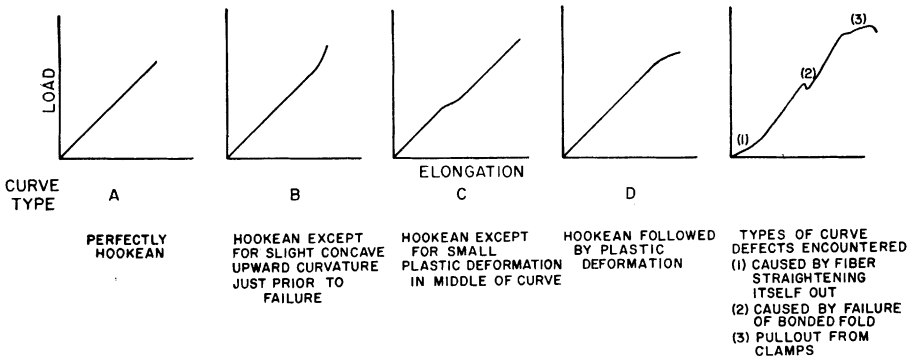


***Fig. 4***—Shape of load/elongation curves

It is clear from Table 2 that the properties of fibres dried under the first three sets of conditions applied—freely dried, plate-dried and in handsheets—do not differ significantly from one another. Therefore, when calculating the properties of handsheets in terms of those of single fibres, it appears to be valid to use data on fibres obtained from any of these three sources.

The differences in the properties between unbeaten and highly beaten fibres measured in the wet and dry states were somewhat surprising. The strength properties of freely dried fibres were greater than those of the wet fibres in the case of the 0-level pulp; the converse was true for the 90-level pulp. This reversal of differences between wet and dry measured properties was supported by the initial modulus values obtained in later experiments on the much larger numbers of fibres that are indicated in the parentheses after the following values. For the 0-level pulp, $E_{wet} = 11.8 \times 10^7$ g/cm$^2$ (58) and

TABLE 2—FIBRE PROPERTIES

(Number in parentheses is percentage standard deviation*)

| *Treatment* | *No. tested* | *Curve** shape* | *Elastic modulus, $g/cm^2 \times 10^{-7}$* | | *Modulus ratio* | *Failure load, g* | *Failure stress, $g/cm^2 \times 10^{-5}$* | *Stress ratio* | *Stretch per cent* |
|---|---|---|---|---|---|---|---|---|---|
| | | | *Initial* | *Final* | | | | | |
| **0-level fibres** | | | | | | | | | |
| Wet-tested fibres | 28 | A,B | — | 14(55) | — | 8.1(48) | 81(33) | — | 6.8 |
| Freely dried fibres | 40 | A,B | — | 20(60) | — | 10.8(74) | 93(41) | — | 4.7 |
| Plate-dried fibres | 58 | A,B,C | — | 19(55) | — | 9.0(42) | 92(45) | — | 6.0 |
| Fibres from plate-dried sheet | 42 | A,B,C | — | 15(57) | — | 10.0(48) | 83(72) | — | 5.6 |
| Fibres dry-strained to $0.25L_e$ (Z) | 34 | C,D | 11(46) | 26(36) | 2.6(36) | 11.6(49) | 100(35) | 1.1 | 4.4 |
| Fibres dry-strained to $0.25L_e$ (NZ) | 39 | C,D | 14(46) | 38(55) | 3.1(40) | 11.5(48) | 97(33) | 1.0 | 4.9 |
| Fibres wet-strained to $0.25L_e$ (Z) | 30 | A,C,D | 9(18) | 40(16) | 4.3(23) | 10.6(41) | 82(30) | 0.9 | 2.8 |
| Fibres dry-strained to $0.50L_e$ (Z) | 34 | D | 18(33) | 39(21) | 2.7(43) | 12.1(34) | 105(38) | 1.1 | 4.1 |
| Fibres dry-strained to $0.50L_e$ (NZ) | 27 | D | 16(34) | 53(23) | 3.7(30) | 13.5(27) | 113(29) | 1.2 | 4.9 |
| Fibres dry-strained to $0.75L_e$ (Z) | 33 | D | 24(65) | 63(36) | 3.1(62) | 14.7(32) | 163(34) | 1.8 | 3.3 |
| Fibres dry-strained to $0.75L_e$ (NZ) | 32 | D | 20(35) | 72(24) | 3.4(44) | 14.6(37) | 123(54) | 1.3 | 3.9 |
| **90-level fibres** | 47 | A,B | — | 23(57) | — | 7.8(41) | 103(69) | — | 4.6 |
| Wet-tested fibres | 47 | A,B | — | 11(46) | — | 7.0(46) | 58(47) | — | 5.3 |
| Freely dried fibres | | | | | | | | | |
| Plate-dried fibres | 83 | A,B,C | — | 17(42) | — | 10.7(42) | 79(47) | — | 4.6 |
| Fibres from plate-dried sheet | 66 | A,B,C | — | 15(54) | — | 9.6(42) | 83(55) | — | 6.1 |
| Fibres from freely dried sheet | 47 | A,B,C | — | 12(72) | — | 8.5(41) | 65(57) | — | 5.8 |
| Fibres dry-strained to $0.25L_e$ (Z) | 74 | D | 11(54) | 30(21) | 3.1(35) | 10.8(37) | 78(37) | 1.4 | 4.3 |
| Fibres dry-strained to $0.25L_e$ (NZ) | 54 | D | 12(67) | 47(34) | 3.9(38) | 9.8(43) | 94(45) | 1.6 | 4.8 |
| Fibres wet-strained to $0.25L_e$ (Z) | 30 | D | 9(17) | 40(16) | 4.1(21) | 10.5(38) | 82(30) | 1.4 | 3.3 |
| Fibres wet-strained to $0.25L_e$ (NZ) | 80 | D | 8(35) | 53(25) | 7.1(33) | 11.1(38) | 84(33) | 1.4 | 3.2 |
| Fibres dry-strained to $0.75L_e$ (NZ) | 70 | D | 18(42) | 63(24) | 3.8(36) | 13.1(33) | 104(29) | 1.8 | 2.5 |
| Fibres wet-strained to $0.75L_e$ (NZ) | 75 | D | 19(24) | 74(29) | 4.0(35) | 14.3(29) | 118(22) | 2.0 | 2.9 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Jentzen's holocellulose fibres** | | | | | | | | | |
| Summerwood fibres, freely dried | 40 | A | — | 40(26) | — | 47.6(24) | 114(24) | — | 3.2 |
| Summerwood fibres, dried under 1 g | 40 | D | — | 55(13) | 1.4 | 55.7(17) | 130(18) | 1.1 | 3.0 |
| Summerwood fibres, dried under 3 g | 36 | D | — | 65(8) | 1.6 | 58.2(13) | 145(13) | 1.3 | 2.9 |
| Summerwood fibres, dried under 5 g | 36 | D | — | 58(8) | 1.4 | 58.9(20) | 135(16) | 1.1 | 3.0 |
| Springwood fibres, freely dried | 41 | A | — | 17(55) | — | 17.5(30) | 56(27) | — | 4.5 |
| Springwood fibres, dried under 1 g | 39 | D | — | 48(10) | 2.9 | 26.1(22) | 91(17) | 1.6 | 2.7 |
| Springwood fibres, dried under 3 g | 41 | D | — | 56(11) | 3.4 | 30.4(22) | 110(13) | 1.9 | 2.8 |

* Percentage standard deviation = (standard deviation/mean) × 100
** See Fig. 4

*Z* = Load zeroed before final elongation (see text)
*NZ* = Load not zeroed before final elongation

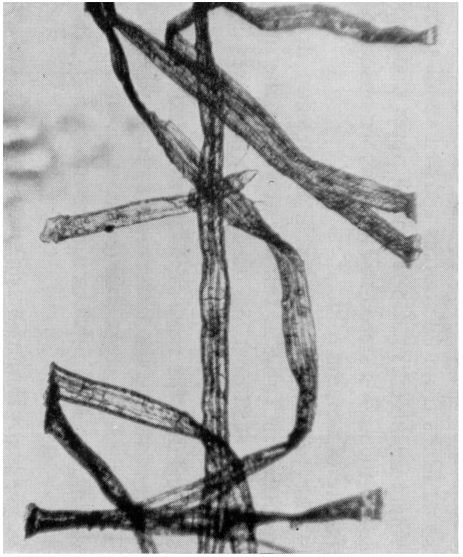

*Fig. 2*

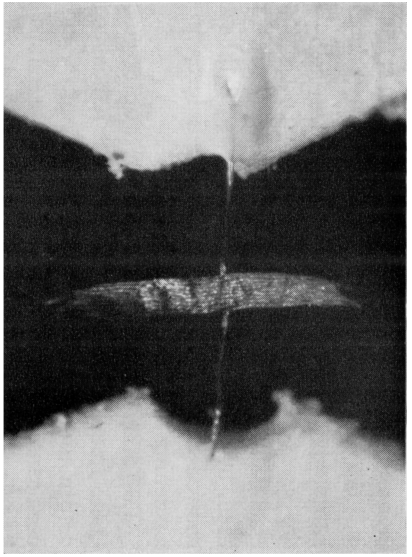

*Fig. 3*

$E_{dry} = 17.6 \times 10^7$ g/cm$^2$ (239); for the 90-level pulp $E_{wet} = 16.2 \times 10^7$ g/cm$^2$ (202) and $E_{dry} = 13.3 \times 10^7$ g/cm$^2$ (245). Both increases and decreases of strength properties as a result of wetting cellulose fibres are reported in the literature.[13]

The shape of a fibre's load/elongation curve gives some insight into its behaviour under strain. Jentzen[4] pointed out that two mechanisms cause load/elongation curves to deviate from Hookean. One is creep or plastic deformation in time. It causes the load at a fixed elongation to decrease and it tends to make the curve concave toward the *strain* axis. The other is work hardening, which stiffens a fibre and tends to make the curve concave toward the *stress* axis. We concur with Jentzen's[4] conclusion that the two effects essentially cancel each other out in freely dried fibres at the rates of loading used, but we did find that quite a few of the fibres *not* dried under tension showed some stiffening just before failure, probably because of the work hardening effect (Fig. 4B).

Two other unusual phenomena were observed in the load/elongation curves. 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, a subject discussed more fully in our second contribution. We found also that some fibres from all sources showed small sharp drops in load almost anywhere in the curve. On watching fibres undergoing straining on the Instron tester through a stereo-microscope, we observed this drop was caused by the failure of bonded folds.

The behaviour of fibres in the tension drying cycles is of interest and recorded in Table 3. Immediately after the initial elongation to $KL_e$ (where $K = 0.25$, 0.50 or 0.75), the load in the fibres dropped sharply, then came to an apparent equilibrium $L_q$ in about 2 min (cycle 2, Fig. 1). For the fibres strained dry, the ratio $L_q/KL_e$ was around 0.7.

On rewetting the fibres under restraint, the load decreased further to $L_w$. The ratio $L_w/L_q$ appeared to increase with increasing $KL_e$. During subsequent drying, the load increased once more to $L_d$; the ratio $L_w/L_d$ averaged about two.

The loads in the fibres during drying from $L_w$ to $L_d$ were appreciably lower than $LK_e$. For example, the ratio $L_w/L_e$ of the 0-level fibres initially strained dry to $0.25L_e$ averaged about $0.023L_e$. When the fibres were strained wet initially, they were, of course, at $KL_e$ before their final drying. Thus, the tensions in the fibres before their final drying varied over an extremely wide range, $0.01L_e$–$0.75L_e$.

With the modulus ratios ranging 2.6–4.3 (no pairs of values differed significantly) for drying tensions ranging $0.01L_e$–$0.75L_e$, we concluded that *as long*

*as there is an axial load on springwood fibres during drying, their modulus increases three or four fold.* Since the modulus of the springwood fibres picked from handsheets dried under restraint were the same as those of freely dried fibres, there appears to be little axial load on the fibres of standard handsheets during drying. Yet the machine-direction (MD) modulus of many machine-made papers exceeds that of handsheets and is often double the cross-direction (CD) modulus. Part of the difference between MD and CD sheet moduli is undoubtedly due to preferred MD orientation of fibres, but another is probably attributable to the larger modulus of the springwood fibres observed in Jentzen's study and ours. It is known that handsheets dried under tension show appreciable modulus increases. For example, Schultz[14] found that the

TABLE 3—FIBRE BEHAVIOUR DURING TENSIONING AND DRYING

| *Treatment, K* | *Load ratios* | | | |
|---|---|---|---|---|
| | *Equilibrium/Initial,* $L_q/KL_e$ | *Wet/Equilibrium,* $L_w/L_q$ | *Wet/Expected,* $L_w/L_e$ | *Dry/Wet,* $L_d/L_w$ |
| **0-level fibres** | | | | |
| Dry-strained, 0.25 | 0.74 | 0.12 | 0.02 | 1.7 |
| Wet-strained, 0.25 | — | — | 0.25 | 1.8 |
| Dry-strained, 0.50 | 0.65 | 0.20 | 0.06 | 2.6 |
| Wet-strained, 0.50 | — | — | 0.50 | — |
| Dry-strained, 0.75 | 0.72 | 0.23 | 0.12 | 2.0 |
| **90-level fibres** | | | | |
| Dry-strained, 0.25 | 0.70 | 0.06 | 0.01 | — |
| Wet-strained, 0.25 | — | — | 0.25 | 1.8 |
| Dry-strained, 0.75 | 0.78 | 0.24 | 0.14 | 2.0 |
| Wet-strained, 0.75 | — | — | 0.75 | 1.1 |

modulus of handsheets of a moderately beaten alpha pulp of western hemlock increased from 800 to 1 100 kg/mm$^2$ (a 37 per cent increase) when the degree of wet straining before drying was increased from 1 to 7 per cent; incidentally, this modulus increase was accompanied by a decrease in the degree of bonding.

It should be noted that Jentzen[4] found the modulus ratio of summerwood fibres to be much smaller than that of springwood fibres, 1.36 at $0.021L_e$, 1.63 at $0.063L_e$ and 1.43 at $0.105L_e$; we tested no summerwood fibres.

Although the stress ratio—the ratio of the average failure stress of the tension-dried fibres to that of the freely dried fibres—of both the unbeaten and beaten fibres increased with increasing drying tension, the improvements were statistically not significant. This was particularly true for the 0-level fibres. In the case of the 90-level fibres, the differences were close to the border

of significance. We placed little value on this significance because of the sample bias mentioned earlier.

Jentzen found that the stress ratio of springwood fibres increased appreciably (and significantly) up to 1.94 at $0.17L_e$. The difference between the stress ratios in Jentzen's study and ours was probably due both to the nature of the tensile test and the care Jentzen gave his fibres during pulping. The tensile strength of a material is a measure of its weakest part. Jentzen handled his fibres with 'kid gloves', whereas our pulps were commercially prepared. Thus, there was much greater likelihood of mechanical damage to our fibres than to his.

TABLE 4—FIBRE MODULUS CHANGES DUE TO SUCCESSIVE STRAIN HARDENINGS

| | 0-*Level fibres* | | | 90-*Level fibres* | |
|---|---|---|---|---|---|
| | *Immediate cycling* | *Equilibrium cycling* | *After ½ h* | *Immediate cycling* | *Equilibrium cycling* |
| No. of fibres tested | 22 | 21 | 15 | 11 | 15 |
| Initial modulus, $g/cm^2 \times 10^{-7}$ | 13 | 16 | 15 | 14 | 22 |
| Failure stress, $g/cm^2 \times 10^{-5}$ | 82 | 82 | — | 118 | 132 |
| Stress ratio | 0.9 | 0.9 | — | 2.0 | 2.3 |
| Dry cycle No. | Modulus ratios* | | | | |
| 1 | 2.65 | 2.26 | 1.58 | 2.27 | 1.96 |
| 2 | 2.82 | 2.44 | | 2.47 | 2.07 |
| 3 | 2.85 | 2.58 | | 2.48 | 2.09 |
| 4 | 2.83 | 2.60 | | 2.51 | 2.09 |
| 5 | 2.92 | 2.69 | | 2.58 | 2.15 |
| 6 | 3.09 | 2.72 | | 2.57 | 2.22 |

* Ratio of modulus in the cycle indicated at left to the initial modulus

The stress ratios of the 90-level fibres appear to be deceptively large because of the low failure stress of its freely dried fibre, 58 kg/mm$^2$. Lower ratios would have been obtained on the basis of the failure stress of 0-level freely dried fibres, 93 kg/mm$^2$. Although we lack statistically significant data to substantiate the argument, we believe that tension drying does increase the intrinsic failure strength considerably (up to twofold at drying tensions approaching $L_e$), but this increase is masked to a considerable extent by weak spots caused by beating.

Usually, the values of the failure stretch decreased with increasing drying tension. This result, which is in general agreement with Jentzen's, is as expected.

In the dry mechanical conditioning cycle (programme 4), each fibre was successively strained to $0.5L_e$ and relaxed five times, then strained to failure.

In three separate experiments, the interval between successive elongations was varied. In the first, the fibres were strained immediately after relaxing and the moduli measured are shown in the column labelled *immediate cycling* of Table 4; in the second, the fibres were strained at 1 min intervals and the results are in the column under *equilibrium cycling*; in the third, at half-hourly intervals.

The most marked modulus increases were obtained by the first dry cycle with only minor increases thereafter. Although the original gain was of the same order of magnitude as developed by tension drying, it was only partially permanent. After 1 min under no load, the gain in modulus dropped 8–18 per cent; after $\frac{1}{2}$ h, 40 per cent. Thus, it appears that the stiffening effects of strain-hardening are partly temporary.

TABLE 5—EFFECT OF REWETTING AND DRYING UNDER NO LOAD ON THE MODULUS OF 12 TENSION-DRIED FIBRES

| *Cycle* | *Modulus of freely dried fibres,* $g/cm^2 \times 10^{-7}$ | | *Modulus ratio of tension-dried fibre** |
|---|---|---|---|
| 1 | 13 | Fibre rewetted and | 3.25 |
| 2 | 15 | ← dried under no load | 2.83 |
| 3 | 16 | | 2.62 |
| 4 | 16 | | 2.79 |
| 5 | 16 | | 2.60 |
| 6 | 16 | | 2.69 |

* Same ratio as in Table 4

In the experiment illustrated in Fig. 1 (programme 5), fibres were alternately wetted and tension-dried, wetted and freely dried (see Table 5). Each tension drying of a previously freely dried fibre produced about a threefold modulus increase; each free drying of a previously tension-dried fibre negated most of the previous improvement. It appears as if the first two cycles may have caused a minor irreversible modulus increase from $13 \times 10^7$ to $16 \times 10^7 g/cm^2$. Jentzen[4] made a similar observation. This irreversibility may be one cause of the well-known irreversible differences between sheets of never-dried and once-dried fibres as the fibre's modulus is of considerable importance to the entire load/elongation curve of paper.

In the last experiment, fibres were again alternately wetted and freely dried, wetted and tension-dried. Between successive wettings, they were mechanically conditioned four times to $0.5L_e$ to determine the effect of strain-hardening on freely and tension-dried fibres.

The results for three fibres are shown in Table 6. Each row gives the four moduli measured after the application of the drying condition listed at the

left; the dry cycles were run in immediate succession so that the fibres were not allowed to come to equilibrium.

TABLE 6—EFFECT OF FREE AND TENSION DRYING, ALSO STRAIN HARDENING ON MODULUS

| *Fibre No.* | *Drying conditions* | *Fibre modulus, g/cm² × 10⁻⁷* | | | |
|---|---|---|---|---|---|
| | | *Dry cycle No.* | | | |
| | | 1 | 2 | 3 | 4 |
| 1 | Freely dried (FD) | 27 | 31 | 35 | 34 |
| | Tension-dried (TD) | 38 | 42 | — | 45 |
| | FD | 27 | 38 | 37 | 38 |
| | TD | — | 47 | 47 | 48 |
| | FD | 30 | 38 | 40 | 38 |
| | TD | 49 | 47 | 47 | — |
| 2 | FD | 18 | 34 | 37 | 37 |
| | TD | 54 | 54 | 54 | — |
| | FD | 24 | 44 | 48 | 47 |
| | TD | 54 | 54 | 61 | — |
| | FD | 26 | 45 | 44 | 46 |
| | TD | 58 | 59 | 58 | — |
| 3 | FD | 18 | 41 | 42 | 44 |
| | TD | 52 | 55 | 56 | — |
| | FD | 40 | 60 | 63 | 61 |
| | TD | 72 | 69 | 70 | — |
| | FD | 42 | 57 | 63 | 60 |
| | TD | 68 | 74 | 71 | — |

The results confirm previous findings. Free drying of fibres previously tension-dried once or twice causes a small irreversible increase in the modulus of freely dried fibres. Mechanical conditioning causes a partial increase in the modulus of freely dried fibres, but has no effect on tension-dried fibres.

### *Cross-direction modulus of fibres*

THE two sonic moduli measured on the sheet with the uniformly aligned fibres were $15.4 \times 10^7$ g/cm² and $1.40 \times 10^7$ g/cm², respectively, a ratio of 11:1. Although it is impossible to conclude that $E_\perp = 1.4 \times 10^7$ g/cm², it is legitimate to conclude that $E$ is one order-of-magnitude larger than the $E_\perp$. This conclusion was indirectly confirmed by the experiment in which fibres were strained with other fibres bonded to them.

The mean $E$ of 62 fibres with others bonded to them *at random* (Fig. 2) was $37 \times 10^7$ g/cm², a value almost double that of any of the sets of freely dried fibres. We concluded from this result that the technique should pick up the cross-direction modulus of fibres, if it is of the same order of magnitude as $E$.

Because the shives and fibres used in the experiments to measure $E_\perp$ were

dyed so that their degree of bonding could be measured readily (80–100 per cent of the 25–50 per cent of the area of the tensioned fibres crossed by others was bonded), it was not possible to measure $\delta$ by the retardation method, which is based on colours.[9] Therefore, instead of $E_\perp$, we measured the load of $e_f = 2$ per cent. With other fibres bonded to them, it averaged 2.3 g. After the shive was removed and the once-strained fibre had been free for $\frac{1}{2}$ h to eliminate some of the strain-hardening effect, the load at 2 per cent strain averaged 3.1 g. In other words, the residual stiffening from strain-hardening was all that was detected; there was no measurable effect of straining the shive. This result confirmed the conclusion that $E > E_\perp$.

These above results were partly confirmed by Mark,[15] who calculated the ratio of $E:E_\perp$ to be 8:1 for crystalline cellulose. He found also that the shear modulus $G$ to be about 1/35 of $E$, instead of $\frac{1}{3}-\frac{1}{2}$ as is the case for *isotropic* materials. This result is of considerable importance to the calculation of the forces arising in fibres during their deflection at rightangles to the fibre axis. It means that these forces, which would be up to 25 per cent of the tensile forces if the fibres were isotropic, are one order-of-magnitude lower (about 2–3 per cent) and can therefore be neglected. This matter will come up again in our second contribution.

*Acknowledgements*

We are indebted to Mrs Doris Mellor, who did the fibre testing; to Dr Ivar Stockel, of St. Regis Paper Co. for critically reviewing the paper; in particular to Dr K. A. Arnold, St. Regis Paper Co., for permission to publish the findings.

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### *Appendix—Instron accessories for testing single fibres*

THE INSTRON accessories for conveniently measuring the load/elongation curves of single fibres consisted of two specially constructed clamps and of jigs for holding the paper tabs on to which the fibres were glued. The functions of the parts are best understood from the following description of the testing of a fibre.

The fibre to be tested, picked by a needle from a suspension, a steel plate or a sheet, is easily mounted on to a jig under a $\times 20$ stereomicroscope as follows. A paper tab (Fig. 5*a*), pretreated with a wet strength resin to make it insensitive to water, is secured in the jig (Fig. 5*b*) as shown in Fig. 5*c* by tightening the fork

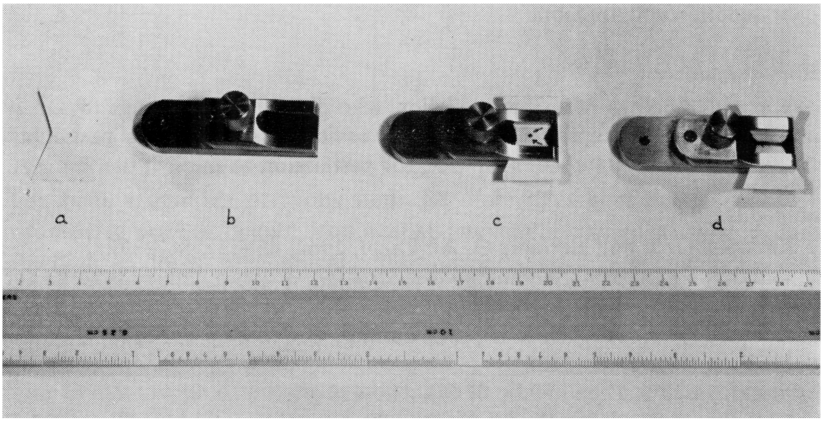


***Fig. 5***—Paper tab and jig

to the base plate. A film of epoxy resin adhesive* is placed by a dissecting needle at the two points indicated by the arrows of Fig. 5*c*. After allowing the adhesive to set for 15 min, the fibre is placed across the hole by another needle so that equal lengths are embedded in the two adhesive spots. With a little practice, it is quite easy to mount the fibre with a small bow out of the plane of the tab to insure its safety during subsequent handling.

After the adhesive has set overnight, two strips are cut out of the tab so that

* Recommended by K. Hardacker of The Institute of Paper Chemistry

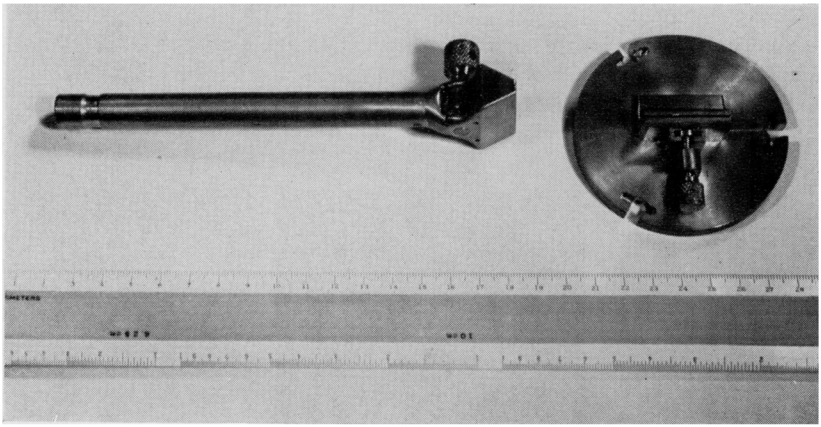

***Fig. 6***—Upper clamp and top piece of lower clamp

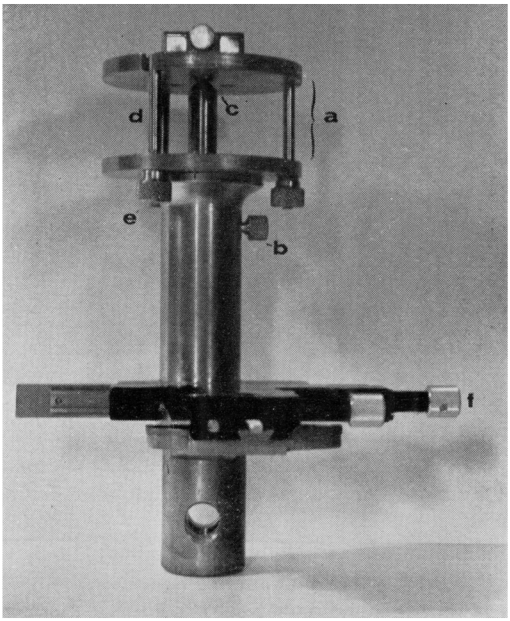


***Fig. 7***—Lower clamp

the fibre becomes the only link between the now two parts of the tab (Fig. 5*d*). The fibre is now ready for testing.

Because the original clamps have no means for fine positioning, the special ones shown in Fig. 6 and 7 were constructed. One end of each fits into the tester; the other has a pair of spring-loaded jaws that automatically move apart when loosened.

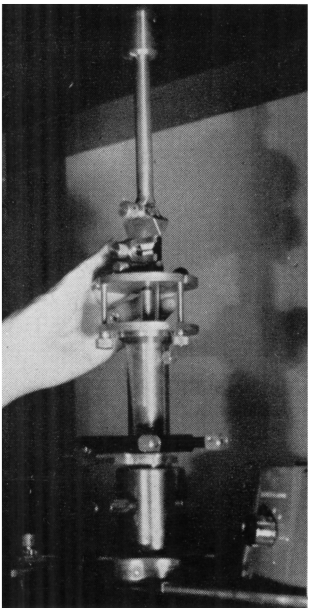

***Fig. 8***—Slipping jig into position on Instron tester

The lower clamp has four adjustments for precise alignment of the fibre before testing (Fig. 7). The entire table *a* can be rotated 360° and fixed by the knob *b*. The top of the table rests on the pivot point *c* and the three posts *d* whose length is adjustable by means of the knobs *e*. Thus, the table can be tilted to permit vertical alignment of the lower end of the fibre. (The upper end of the fibre must be set vertically into the upper clamp; see below.)

The entire upper portion of the clamp is fastened to the microscope stage *f*, whose two verniers permit placement of the lower end of the fibre exactly beneath the upper. The stage, in turn, is fastened to the bar that fits into the Instron cross-head.

The distance between the two sets of jaws is set about a millimetre larger than the width of the jig. With the entire clamping zone visible through the stereo-microscope, the jig is slipped between the clamps so that the tabs protrude into the two pairs of jaws (Fig. 8). After insuring that the top end of the fibre is set

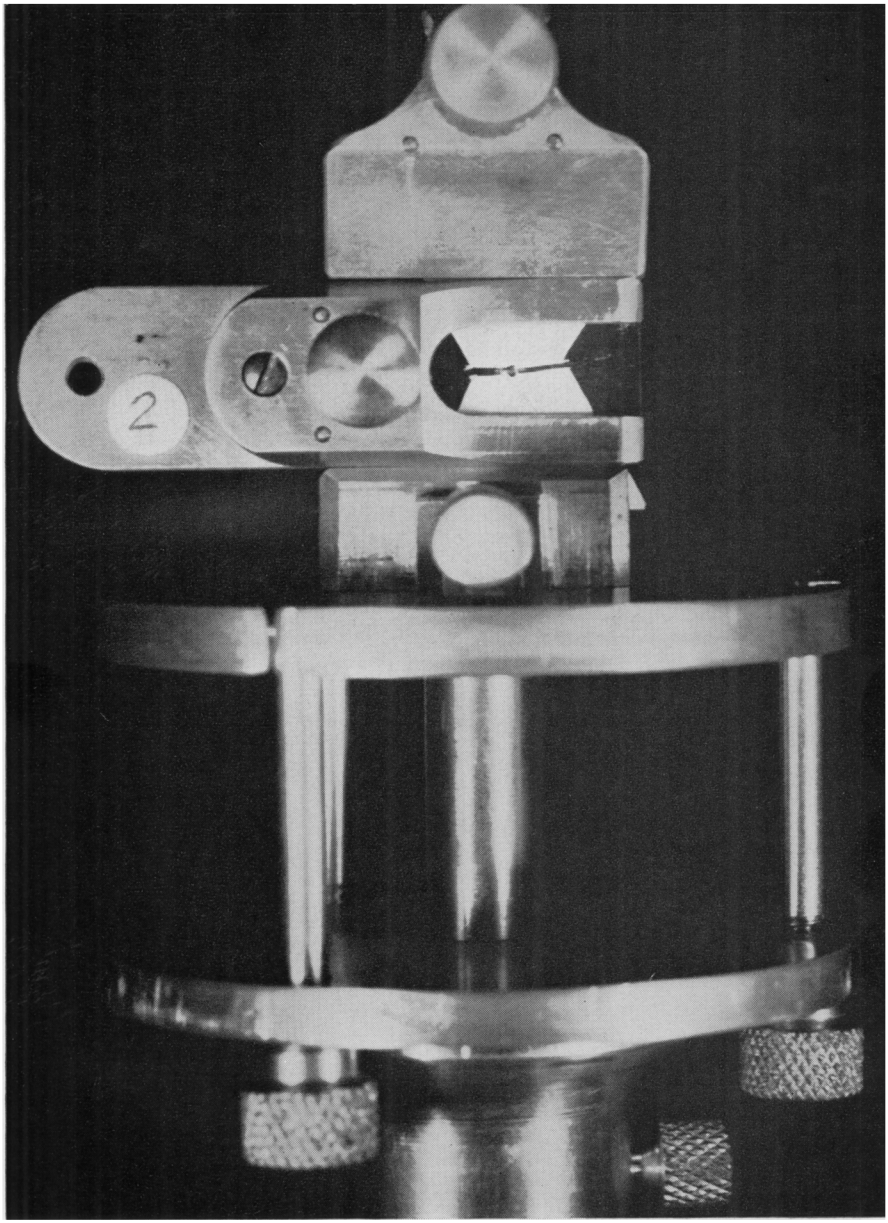


***Fig. 9***—Jig in place in clamps

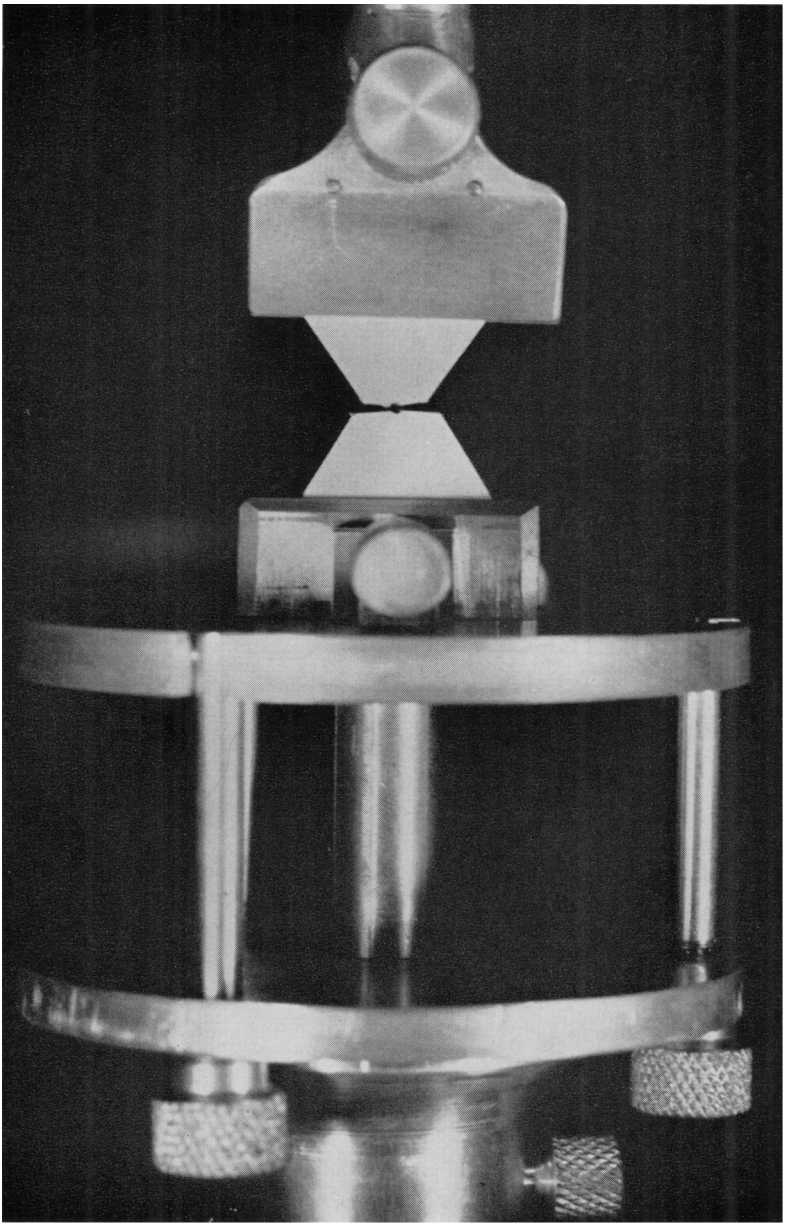

***Fig. 10***—Fibre ready for testing

vertically, the upper clamp is tightened. Then the lower one is tightened slowly so that the jaw faces contact the tab simultaneously; this step often required vernier adjustments (see Fig. 9).

With the tabs gripped in place, the fork of the jig is loosened and the jig slipped out. This is the only difficult step of the technique; our best technician lost about an average of one fibre in 20 in this step. Once the jig is removed, the cross-head is lowered and other necessary adjustments made to insure the vertical alignment of the fibre during the test. The length of fibre between the glue lines is measured by a micrometer eyepiece in the stereomicroscope. The fibre is now ready for treatment and for testing (see Fig. 10).

The wetting of a fibre before or during a test cycle was accomplished by means of the hypodermic needle mounted on the positioning device shown in Fig. 11. With two rotary and three linear vernier motions, a drop of water was transferred easily from the tip of the needle on to a fibre.

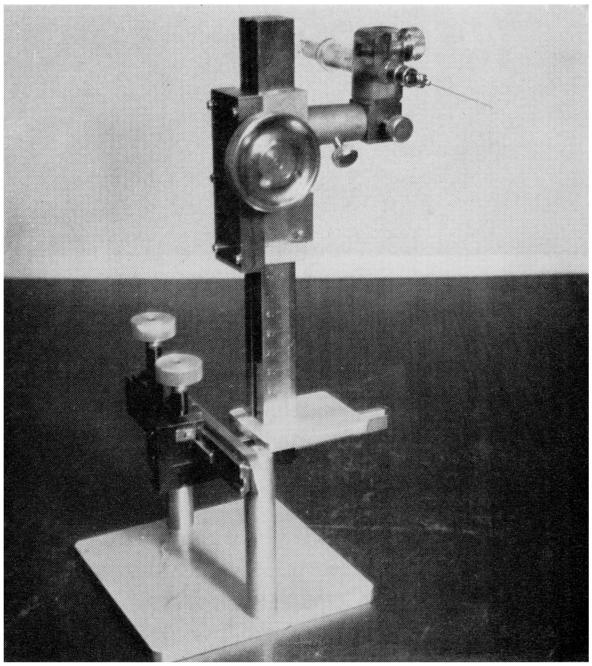

***Fig. 11***

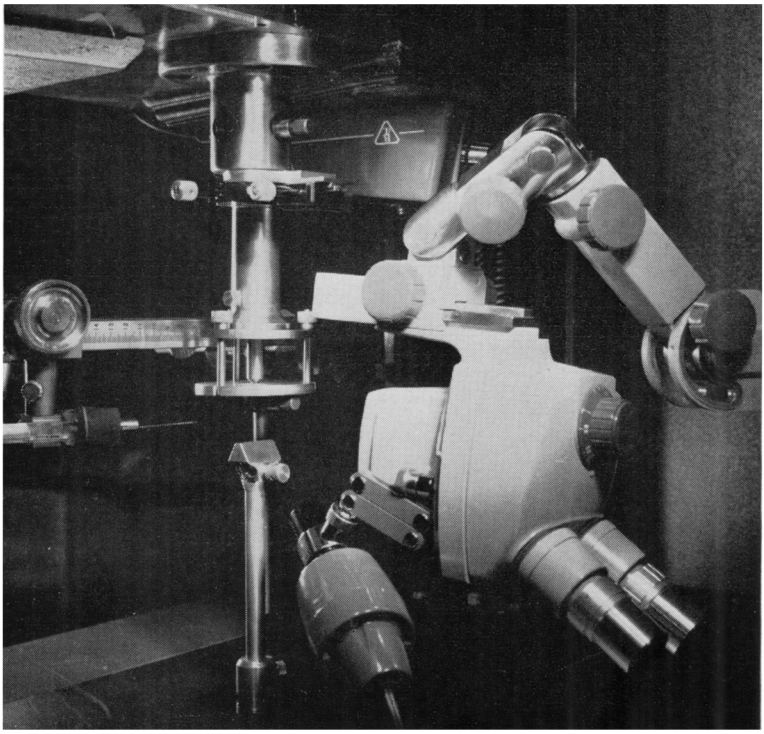

***Fig. 12***—Set-up for the wet testing of fibres

# 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.