

# Conversion of an Instron to mechanical testing of single fibres

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The object was to acquire the capability of performing tensile fracture and fatigue tests on single wood fibres. A benchtop model Instron was available with sufficient sensitivity to conduct these types of measurements. However, the grips were not rigidly aligned with each other and the resultant lateral movement generally broke the fibres before the test commenced. An attachment to the Instron was constructed to overcome the problem. In this paper, the design criteria for the Instron fitting are discussed, details of the design are given and some preliminary results showing the application of the new system are presented.

## Keywords

single fibre mechanical properties, fatigue testing, elastic modulus, plastic deformation, rayon mechanical properties, moisture effects

## Single Fibre Mechanical Properties

It is important to measure single fibre properties as considerable work in paper physics tells us that not only is the average value of a particular fibre property important but that the distribution around the mean also has a critical role to play. For example, in recent work (1) on the strength of paper made by recombining different fractions of pulp, the strength of the sheet of paper changed even though the pulps had been recombined so that the average fibre length was unaltered. The distribution of fibre strengths and elastic moduli of the fibres would also be expected to play a significant role in determining the strength of paper. Considering the failure of paper under load, it would be expected qualitatively that it would not directly be the mean of the fibre strength that would play the critical role but rather the distribution of fibre strengths below the average, since it is the weaker fibres that will fail first.

The most commonly used theories for the strength of paper e.g. Page (2) use only average fibre strengths and lengths, although recently there have been attempts to incorporate the effects of fibre property distributions into models of fibre mechanical properties (3,4,5).

Even the incorporation of only the average fibre strength into theories of paper strength is difficult since fibre strengths have been determined from the zero-span tensile strength. Theoretically (6) the zero-span strength is  $3/8$  of the strength that would have been obtained if all fibres had been aligned perpendicular to the jaw line. However, Perez and Kallmes (7) found that the zero-span strength is generally less than would be theoretically expected from measurements of the strength of the individual fibres, with the relationship between the theoretical and measured zero-span strengths being non-constant. The differences were explained by postulating that not all fibres that cross the jaw line actually bear load as some will be kinked, twisted or curled at the point where they cross the jaw line. More recently El-Hosseiny and Bennett (8) have shown theoretically that the average fibre strength and the distribution of fibre strengths affect the zero-span breaking length. Thus while there is a strong qualitative link between fibre strength and the measured zero-span value, it is not possible, without making assumptions, to work back from a measured zero-span value to obtain the average fibre strength.

Interest in single fibre fatigue testing became part of research into low consistency refining. This comes from research (9,10) which characterizes refiners by the number of impacts,  $N$ , and the energy expended per impact  $I$ , (called an intensity of impact). This description of refining as a fatigue type process has led to studies examining the development of fibre mechanical properties as a function of the number of straining cycles (11,12). There has also been considerable work to determine the forces imposed on the fibres in refining from considerations of bar geometry and refiner plate gap (13,14,15).

## Measurement techniques

The most significant work in measuring the mechanical properties of single fibres has been published in a series of papers by Page and co-workers (16,17,18). In the first of these papers, an extensive literature review of work undertaken prior to 1972 is given. Page and co-workers mounted their fibres on glass tabs, held together with stiff cardboard, using epoxy glue. After mounting the assemblage in the jaws of the tensile tester, the cardboard was then cut, leaving only the fibre to bear the load exerted by the jaws. The Instron used was described as 'heavily modified' but no details were given.

Recently, Mott and co-workers (19,20,21) have been using a technique in which a drop of epoxy glue was bonded to the fibre at each end of the fibre segment to be tested. Force was then applied to the fibre by gripping each of the epoxy drops with a two-pronged fork. Measurements were typically performed with the fibre being extended at only  $1\mu\text{m}$  per second until the fibre fractured. This type of grip arrangement is best suited to a horizontally oriented tensile testing system.

## EQUIPMENT

### Design criteria

The over-riding design criterion for the attachment to our benchtop tensile testing system (Instron model 5566) was that the load must only be applied uniaxially along the fibre. While our benchtop system has sufficient sensitivity to measure the small forces associated with single fibre testing, the grips supplied with the Instron were only the normal pneumatically activated type; they have considerable lateral movement built into them and then they rely on the strength of the sample for alignment as the test begins and the sample is taken under load.

When the existing grips were used for single fibre testing the strength of the fibre was insufficient to align the grips. Fibre fracture generally occurred immediately after removal of the support tabs. Van Den Akker et al. (6) overcame this problem by using rods attached to the

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crosshead that slotted through holes attached to the fixed grips. These guides eliminate lateral movement in the crosshead but added a frictional force that had to be subtracted from the measured force to obtain the force on the fibre. As the maximum load on a fibre is typically less than 0.5 N, great care must be taken with this type of a system to minimize frictional forces.

Our design criteria for the system were then that it:

- impose only uniaxial tensile forces on the fibre;
- do so without generating frictional forces;
- could be used to test fibres wet (closer to the situation in a refiner) as well as dry and
- provide a mounting system for a microscope so that images of the fibre could be taken during the testing process.

### Equipment design

Schematics of the equipment are shown in Figure 1. The heart of the design is an adapted source drive used in Mössbauer spectroscopy. The casing of this drive is bolted on to a rigid frame, which in turn

is bolted on to the bedplate of the Instron. Within the drive casing there is a core attached to the case by three leaf type springs at the top and bottom. There is no other contact between the core and the casing of the drive. The spring arrangement allows the core to move easily along the axis of the drive while preventing any lateral movement of the core. As the core is suspended within the casing, no frictional forces must be overcome for uniaxial movement of the core. The top of the core is directly bolted to the Instron crosshead.

The fibre assemblage is mounted in two screw type grips, with the lower grip being attached to a thick bottom plate attached to the frame (effectively connected to the bedplate of the Instron). The upper grip is bolted to the lower end of the core. With this attachment, the load on the fibre is then purely uniaxial.

Testing in water is conducted using a small 50 x 40 x 35 mm<sup>3</sup> perspex bath which is glued to the bottom plate and caulked with silicon sealant to make it waterproof. Access to the sample grips is via a removable front window.

A support was added to the Instron to allow an Olympus microscope to be

horizontally mounted to view the sample as it is tested.

### EXPERIMENTAL METHOD

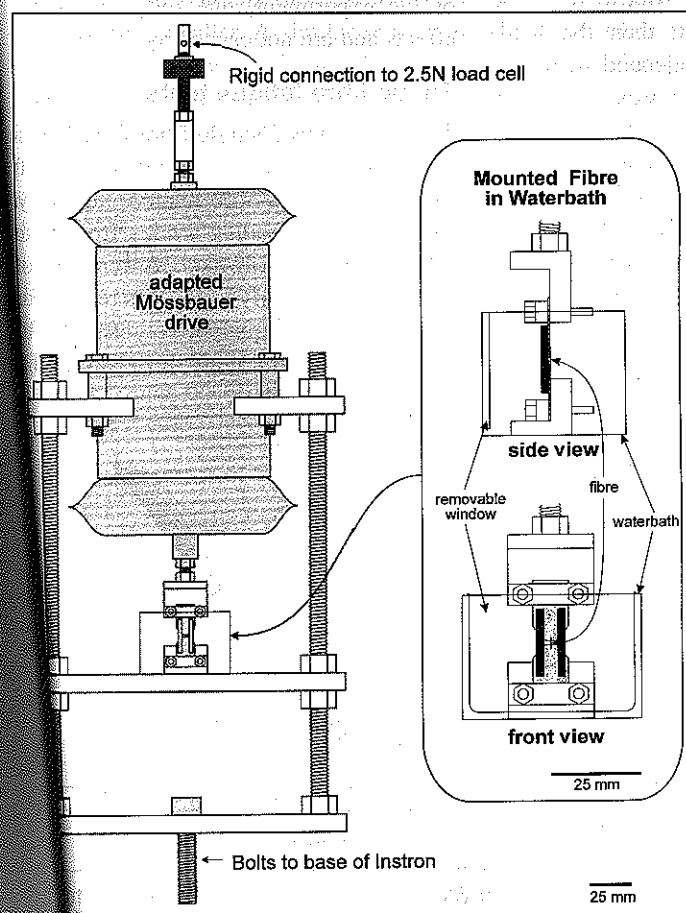
Fibres were mounted between clean steel tabs using epoxy glue which was allowed to set for three days to develop maximum strength. On either side of the fibre, plastic support strips were glued to allow the fibre testing assembly to be handled. After mounting the fibre for testing, the plastic support tabs were removed by melting with a hot wire. After removing the supports, the crosshead of the Instron was adjusted so that the fibre was slack between the grips.

Upon commencement of the test, the crosshead had to move some distance before the fibre was pulled taut and began to bear load. The slope of the load-displacement curve before the fibre began to bear load was determined for each test and used to subtract the spring constant of the apparatus from the measured load-displacement data.

### RESULTS

#### Effect of cycle rate

Figure 2 shows the effect of loading-unloading cycle frequency on the load displacement curve for the apparatus alone. The tests included 10 loading and unloading cycles and were conducted at crosshead displacement rates of 1, 5 and 10 mm/min. The data sets for 5 and 10 mm/min have been displaced along the x-axis by 22 and 45 mm respectively to avoid overlap of the data from different sets. The load-displacement curves are linear and reproducible between cycles for all the tests conducted. There is little difference between the loading and



Attachment for single fibre tensile tests.

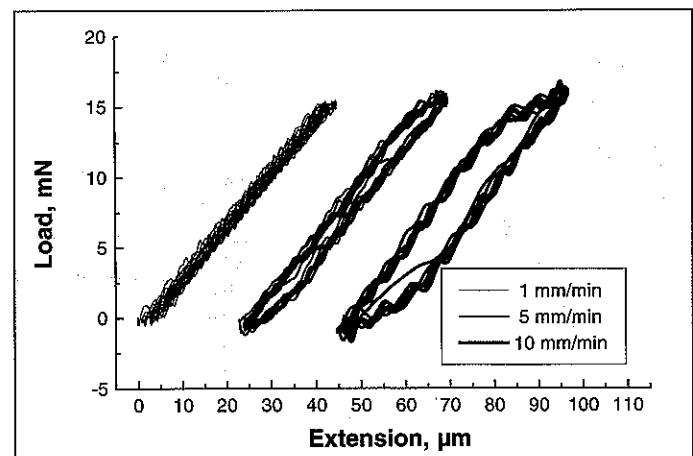


Fig. 2 Apparatus load displacement curves for 1, 5 and 10 mm/min Instron crosshead speeds.

**Table 1**  
**Results of fracture tests of different lengths of rayon fibres.**

Span (mm)	No. of tests	Extension at fracture mm	Strain at fracture	Breaking load
0.5	7	0.041 ± 0.006	8.2 ± 1.1	60 ± 6
0.8	8	0.066 ± 0.015	8.2 ± 1.9	59 ± 4
2.0	6	0.13 ± 0.03	6.7 ± 1.6	54 ± 3

Averages taken only from those tests where the rayon failed at loads greater than 50 mN and strains less than 15%.

unloading curves obtained at 1 and 5 mm/min. However, there is substantial hysteresis between the loading and unloading curves for a crosshead speed of 10 mm/min, suggesting that the maximum crosshead speed should be 5 mm/min or  $\approx 1$  Hz, if the crosshead is cycled from 0 to 40  $\mu$ m. For the results following a crosshead speed of 1 mm/min was used. The slope of the data gives a spring constant for the apparatus of 0.38 N/mm, which is an order of magnitude less than the stiffness of a typical radiata pine kraft fibre.

### Effect of glue

Thirty fracture tests of rayon fibres were conducted using test spans of 0.5, 0.8 and 2.0 mm (ten tests at each span). Rayon fibres were selected because the cross-section dimensions of the fibres in a given batch have greatly reduced variability compared to wood fibres, thus allowing results to be directly compared from fibre to fibre without the necessity of measuring the cross-section dimensions of the fibres. The rayon fibres were also of similar stiffness to wood fibres so as long as there is no slippage between glue and fibre the effect of the glue will be similar in tests on wood and rayon fibres.

The results of the tests are tabulated in Table 1, which displays the average extension, strain and force at the breaking point of the fibres in the different test conditions. The calculated averages given in this table exclude those tests where the sample broke prematurely at the glue-line (breaking load < 50 mN) or where there was significant slippage of the fibre in the glue (strains > 15%). These criteria gave an average success rate of 7 out of 10 tests. Slippage problems may be reduced with improved glues and/or sample preparation but fibre failure at the glue-line will remain an inherent limitation of the technique. When the average load and strain at fracture are compared it can be seen that they are almost independent of the span of the test.

If the glue does not contribute to the measured displacement, then the load-strain curve will be independent of the span over which the test was conducted. However, the differences observed with test span in Table 1 are not necessarily due to the glue. Fracture in general begins from defects, which are statistically distributed. As longer samples are statistically expected to have more defects than shorter ones, they will break at smaller stresses and strains. To test

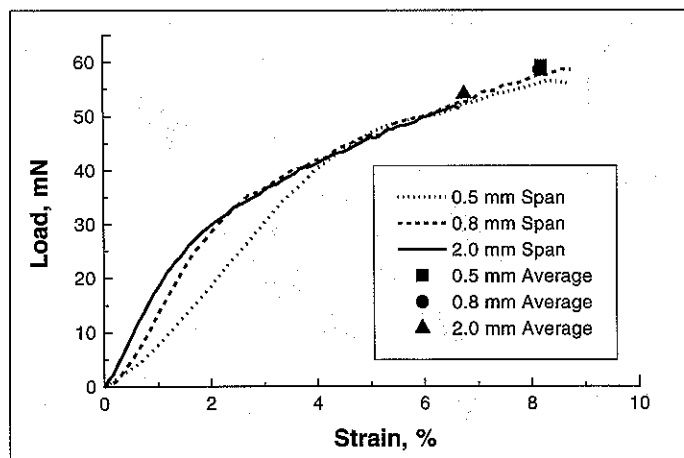
whether the differences between spans seen in Table 1 were due to the sample or due to the glue, typical load-strain curves measured at the different spans were compared with each other in Figure 3. The single points included in the Figure show the data given in Table 1 for the average forces and strains at fracture.

In Figure 3 it can be seen that the initial sections of the load-strain curve differ with the span. While these differences are relatively small for the 0.8 and 2 mm spans, the loads measured for a 0.5 mm span are significantly lower for strains up to 4%. This effect probably comes from how the sample takes up the load. If there were any misalignment between the top and the bottom grip then this would increasingly affect the initial load-strain curve at very short spans. As a span of 1 mm was typically used in our tests, no problems would be expected from this source.

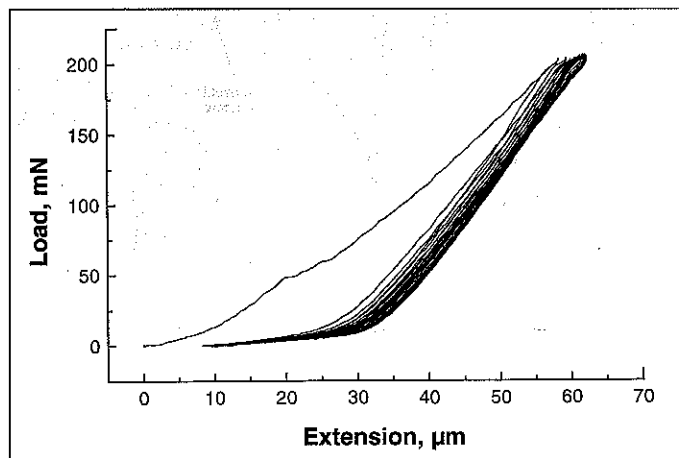
For strains greater than 4%, the three curves are nearly coincident and the points giving the average strains and loads at fracture lie almost exactly on these three curves. Thus the differences in observed breaking loads and strains at different spans are, within the sensitivity of the experiment, due only to sample effects and are not caused by the glue.

### Single fibre fatigue tests

Figure 4 shows a single fibre fatigue test conducted on a dried radiata pine kraft fibre at 23°C and 50% relative humidity. In the test, 10 loading cycles from 0 to 200 mN were performed. A number of fibres were similarly tested, the results for each fibre were qualitatively similar to those presented in Figure 4. In examining



**Fig. 3** Typical load-strain curves for rayon fibres as a function of span. Average failure strains and loads are marked.



**Fig. 4** Typical dry pine fibre load-extension curves, 10 cycles.

the data, it can be seen that there are very large differences between the initial and subsequent loading cycles. The loading curve for the first cycle has a substantially lower slope than the unloading curve, resulting in substantial plastic strains in the fibre. In the second and subsequent cycles, the loading and unloading curves have similar slopes to the unloading curve of the first cycle.

To clarify the results shown in Figure 4, the average stiffness and plastic deformation of the fibre as a function of the cycle number has been plotted in Figure 5. To calculate these results the plastic deformation for each cycle was taken as the point on the loading curve where the slope of the load-displacement curve first reached 2 N/mm. This somewhat arbitrary definition was adopted because the fibre does not instantaneously take up the load, which makes it hard to define the point where the fibre first becomes fully taut. The stiffness was calculated as the average slope of the loading curve, after excluding the initial part of the load displacement curve.

From the data, there has been a large change in the stiffness after the first straining cycle, with the change in stiffness in the second and subsequent cycles being much smaller. A similar trend can be observed in the results for the plastic deformation. The two possible causes of an increase in plastic strain and stiffness are the removal of defects from the fibre and/or a change in the fibril angle of the fibre. For example, the removal of a crimp will simultaneously lengthen the fibre as well as increase the stiffness by improving the uniformity of the fibre cross-section. Reducing the fibril angle has the same result: the fibre becomes

longer and narrower and fibres with lower fibril angles have a higher elastic modulus (18). At present, it is not possible to directly determine the contributions of defect removal and/or fibril angle reduction to the observed increase in the stiffness of the fibre.

It has been hypothesised by Page (22) that the removal of defects is the primary cause for the improvement, upon refining, of the sheet strength of previously dried chemical pulps. The defect free fibres are expected to be stiffer and straighter and thus better able to carry load within the sheet. The results presented in Figure 5 are interesting because while the fibre stiffness increases with mechanical treatment, almost all of the change in fibre stiffness occurs during the first straining cycle, with very little change occurring in subsequent cycles. Thus the improvement in fibre mechanical properties measured here did not occur through a fatigue mechanism, which has been proposed as the underlying effect in kraft pulp refining (9,10,11).

#### Wet and dry fatigue tests

Figure 6 shows stiffness and plastic deformation results from wet and dry tests on the same fibre. After being mounted dry, the fibre was tested in water for 10 load cycles of 0 to 200 mN. After completion of this set of tests, the water was removed and the fibre allowed to dry without restraint. A further ten loading cycles to 200 mN were then performed.

Similar trends to Figure 5 can be seen in the plastic deformation and stiffness data for wet cycling, i.e. large increases in plastic deformation and stiffness (over 100% increase) in the first wet cycle with only small increases in these quantities in

the remaining cycles. During drying, the fibre shrank 3% while the stiffness of the fibre has increased. Approximately 1% of the shrinkage upon drying was then recovered during the subsequent loading cycles.

#### CONCLUSIONS

An attachment for an Instron was designed and constructed to allow tensile testing and fatigue testing of single fibres. The attachment ensures that load is only applied uniaxially to the fibre. A central rod, which transmits load from the crosshead to the fibre, is supported by springs allowing it to move uniaxially without frictional forces. The effect of the glue used to mount the fibres was investigated and found to be negligible.

Results from a single fibre tested under cyclic loading conditions are presented. Large plastic strains, accompanied by an increase in tensile stiffness were induced during the first loading cycle only, subsequent loading cycles produced very little change in either property. These results are inconsistent with the hypothesis that fibre development in low consistency chemical pulp refining takes place through a fatigue type process.

Results from a single fibre cyclically loaded under both wet and dry conditions are also presented.

#### ACKNOWLEDGEMENTS

We acknowledge useful discussions with Alan Holland in designing the rig as well as the assistance of Gerry Skipper in building the rig. The financial support of the Commonwealth Government of Australia through the Australian Research Council is gratefully acknowledged.

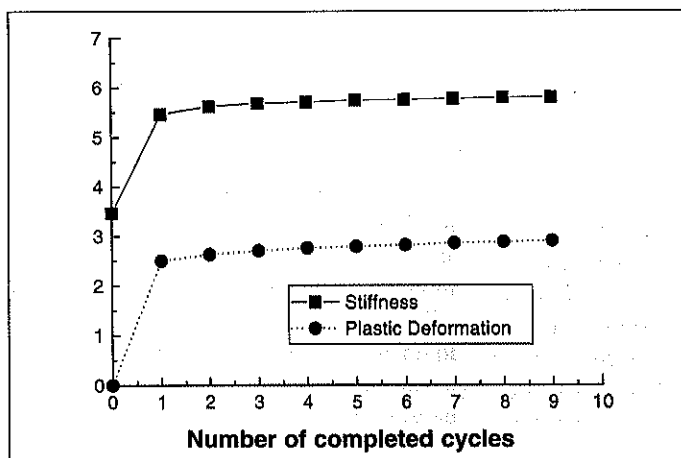


Fig. 5 Stiffness and plastic deformation of a single dry pine fibre.

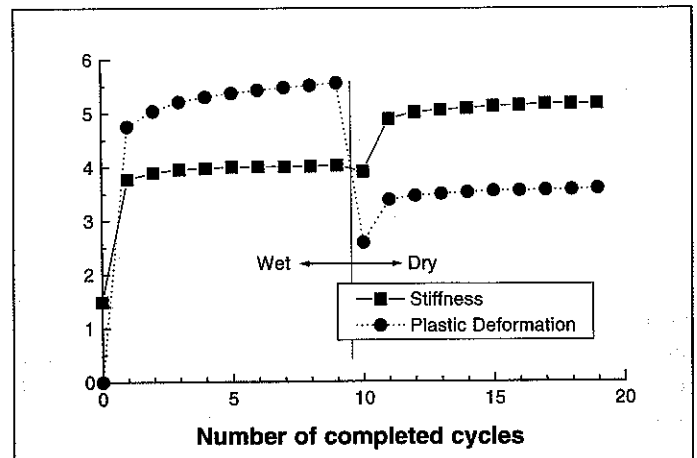


Fig. 6 Stiffness and plastic deformation of a single pine fibre in wet and dry conditions (10 cycles wet, 10 cycles dry).

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Revised manuscript received for publication 6.5.99.