Tensile Testing of Single Regenerated Cellulose Fibres

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Summary: A tensile testing set-up was developed for the determination of the elastic modulus, tensile strength, and failure strain of single regenerated cellulose fibres. Since the accuracy of strain measurement is crucial for the measured elastic modulus and failure strain, strain measurements were performed mechanically and with a non-contacting optical method in parallel. The optical validation of mechanical strain measurement showed an agreement of measured strain >99%, confirming the accuracy and usefulness of the set-up and sample geometry developed for the test series.

Keywords: cellulose; fibres; strain measurement; tensile testing; video extensometry

Introduction

Regenerated cellulose fibres like Viscose and Lyocell are recently studied with regard to their potential use as reinforcement in polymer composites.[1–4] Considering glass fibre, the most widely used fibrous polymer reinforcement, cellulosic fibres show the disadvantage of weaker mechanical properties, whereas their renewability, sustainability and non-abrasiveness in processing are considered as important advantages.[5] Among cellulosic fibres, regenerated cellulose fibres are characterised by less variability of properties than natural fibres, but are usually less stiff than the latter, while being comparably strong.

The properties of regenerated cellulose fibres can be tuned in a controlled manner within a very wide range by varying production parameters. Viscose and Modal fibres are produced from wood pulp by dissolution following derivatisation (xanthogenation) and subsequent spinning into H_2SO_4. In a more environmentally friendly process, Lyocell fibres are produced by direct dissolution in *N*-methylmorpholine-*N*-oxide/water solvent. Both technologies are well developed to supply the fibres needed in the textile and nonwoven industry.[6] Rayon tire-cord, a viscose-type fibre, is optimised for high strength needed for tire-reinforcement.

Within a larger study dealing with the characterisation of Viscose, Modal, and Lyocell fibres with regard to their potential for polymer reinforcement, a large number of single fibre tests were performed. Tensile testing of single fibres is a routine procedure in industrial quality management. Testing is usually performed with dedicated machines (e.g. Lenzing Vibrodyn 400, www.lenzing-instruments.com) according to internationally recognised standards (BISFA– Testing methods viscose, modal, lyocell and acetate staple fibres and tows, ASTM D 3822, ÖNORM EN ISO 5079).[7–9] Since the modulus of elasticity is of particular relevance to our study, a dedicated set-up with reliable strain determination at reasonable testing time was desired. Single-fibre tensile tests usually rely on indirect strain measurement, relating cross-head movement of the testing machine to fibre length.[10] Since slippage of fibres in the grips of the testing machine can

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seriously affect strain measurement by over-estimating strain and thus under-estimating fibre stiffness, the method of fixing fibres to the grips is always paid particular attention. One group of authors uses paper frames, to which fibres are glued and which are cut open in order to allow testing after having been mounted to the testing machine. This method is very efficient in handling fragile fibres and transferring them to the testing machine without damage. A second group of authors applies a drop of polymer to the fibre ends, which is subsequently cured and used to secure the fibre ends to the testing machine (ball and socket method). A number of studies have also applied direct non-contacting strain measurement by video extensometry.[11–13] Non-contacting methods of strain measurement offer the advantage of being very accurate and not damaging fragile fibres during testing.

In the present study, we combine indirect strain measurement with simultaneous video extensometry in order to ascertain the validity of indirect strain measurement from regenerated cellulose fibres glued to paper frames.

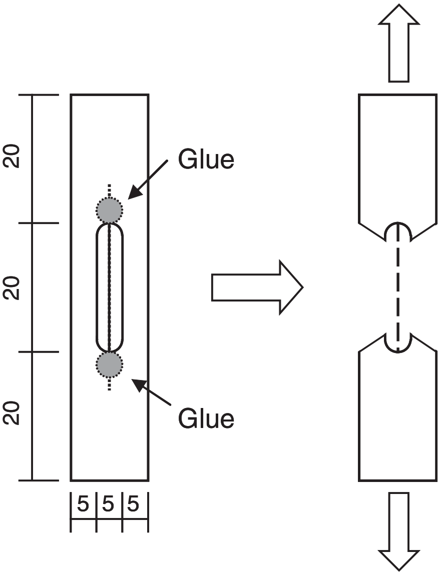

Figure 1.
Paper frame set-up for tensile testing of single fibres.

Materials and Methods

Viscose, Modal, and Lyocell staple fibres with a length of approximately 40 mm were obtained from Lenzing R&D (Lenzing, Austria). Under an incident light microscope, single fibres were separated and glued to paper frames as shown in Figure 1. A second paper frame was glued onto the first frame in a manner that fibres were sandwiched and glued between two frames at each side. The frames were then clamped into a Zwick/Roell universal testing machine equipped with a high resolution 50 N load cell. The paper frame was subsequently cut open as shown in Figure 1 in order to free fibres from the paper frame.

For strain measurement from cross-head displacement, the free fibre length before the start of the test was measured with a digital calliper at a precision of 0.01 mm. This reference length was entered into the testing software which recorded also the cross-head displacement and calculated strain from this input data.

Strain measurement from cross-head displacement was validated by means of video extensometry. Under an incident light microscope, two ink marks were made on the fibre mounted in a paper frame as shown in Figure 2. The testing machine was then programmed to load the single fibre in incremental steps of 20 MPa from zero MPa to 200 MPa, and continuing in steps of 50 MPa from thereon until failure. At each step, the cross-head was set to stop moving for 5 sec, meanwhile an image of the fibre as seen in Figure 2 was captured by means of a CCD camera with a resolution of 1300×1030 pixels. Tests were performed at a cross-head displacement rate of 1 mm min^{-1}. The distance between ink marks placed on single fibres (>8 mm) was evaluated using ImageJ software. Grey values along the fibre were plotted against fibre length and characteristic grey value peaks were identified (Figure 2). The distance between these peaks was

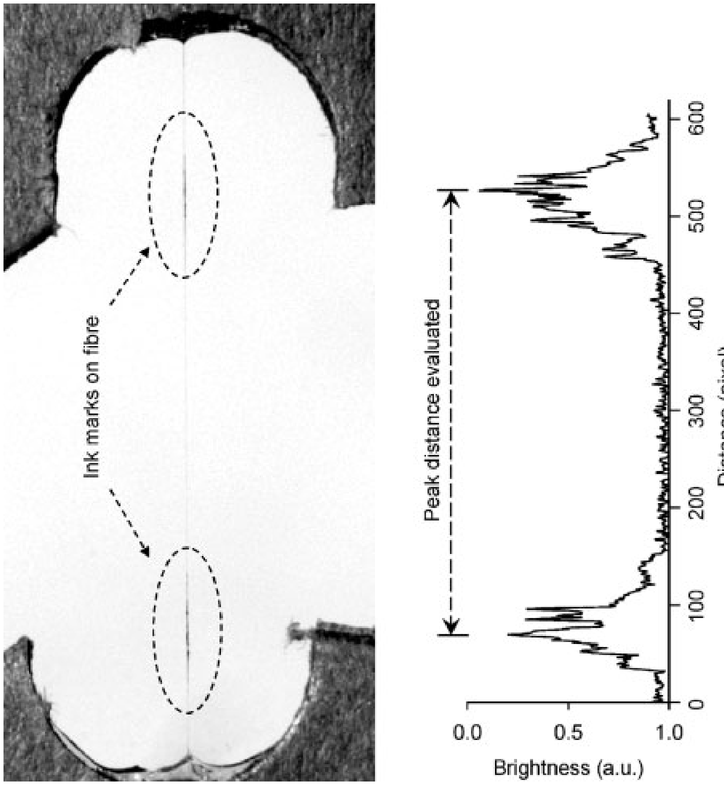

Figure 2.
Optical strain measurement by tracing the movement of ink marks on fibres using video extensometry. Left: Fibre with two ink marks mounted in paper frame. Right: Line-scan of grey values along the fibre length used to track the movement of ink marks during testing.

measured for each consecutive image and strain was calculated from the change in distance with regard to the first image taken at the beginning of the mechanical test.

Results and Discussion

Figure 3 shows a plot of a stress-strain curve of a single lyocell fibre comparing strain calculated from the displacement of the machine cross-head and strain measured by video extensometry. While there is poor agreement at small strain, the agreement between strain measured with both methods improves considerably with increasing strain. Due to the high scatter of small strain measured in the elastic region by video extensometry it is evident that this method can not be used for the determination of the elastic modulus. Only at strain >1% are the changes in distance between the ink marks applied sufficient to be reliably captured by the CCD camera used in this study. For strain measurement at higher resolution, a CCD with more pixels would be required. Nevertheless, the resolution used in the present study is sufficient to measure strains >1% very accurately.

A comparison of strain data calculated from the displacement of the machine cross-head and strain measured by video extensometry acquired from tensile tests with 20 single fibres is shown in Figure 4. The agreement between both methods of strain measurement is excellent, with a coefficient of determination of 0.99. While the offset of the linear regression trend line

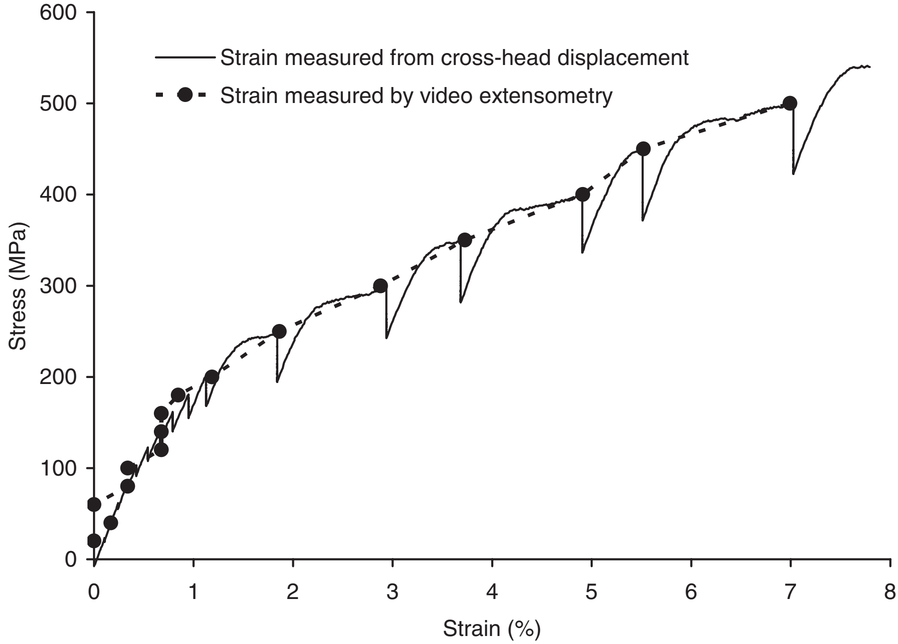

Figure 3.
Comparison of stress-strain curve of a single lyocell fibre using strain calculated from the displacement of the machine cross-head and strain measured by tracking the movement of ink marks on the fibre with video extensometry.

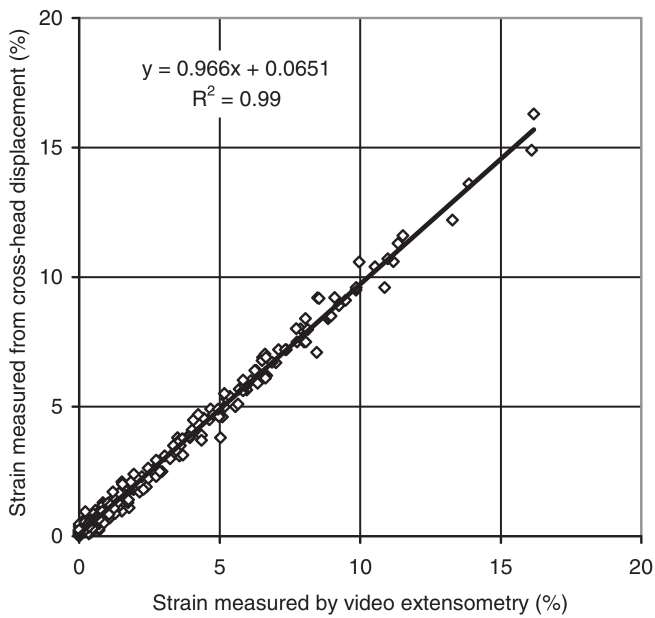

Figure 4.
Strain calculated from the displacement of the machine cross-head and strain measured by video extensometry compared for tensile tests with 20 fibres.

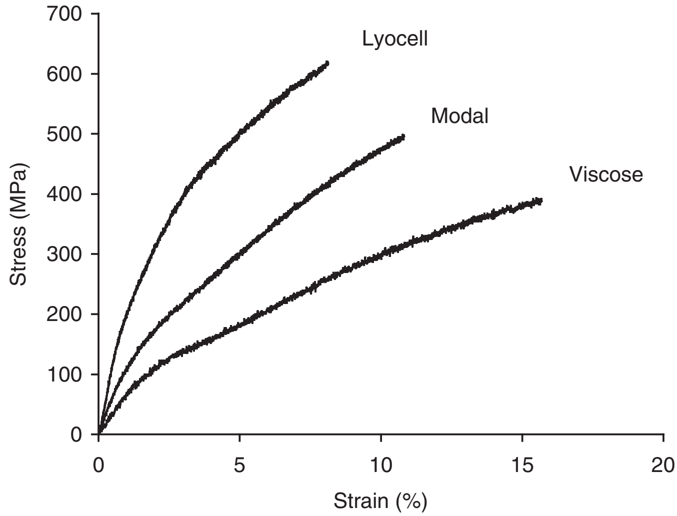

Figure 5.
Representative stress-strain curves of Lyocell, Modal, and Viscose fibres (linear density was 1.3 dtex for all fibres).

is negligibly small, the slope of the trend line deviates slightly from 1. This indicates that in spite of the fact that free fibre length was measured from glue droplet to glue droplet, strain measured from cross-head displacement is slightly higher than strain measured by video extensometry. Since we assume that image analysis is accurate, the observed difference is accounted for by the compliance of the paper frame and the glue used for fixing fibres.

Therefore, stress-strain curves and mechanical properties of selected regenerated cellulose fibres presented in Figure 5 and Table 1 may be considered accurate and representative for mechanical behaviour of these fibres. Owing to their different structural and chemical characteristics caused by the specific variables of their respective production process, a trend of increasing modulus of elasticity and increasing tensile strength is observed in parallel with a decrease in elongation at break.

Conclusion

From the validation with video extensometry of indirect strain measurement based on machine cross-head displacement performed in the present study we conclude that indirect strain measurement is sufficiently accurate to deliver reliable strain and modulus data. Accurate measurements are achieved, when slippage of fibre ends in the machine grips is minimised or avoided completely, such as is possible with the dedicated paper frame set-up developed for this study.

Table 1.
Mechanical properties of selected single regenerated cellulose fibres.

Fibre type	Diameter (μm)	E-Modulus (GPa)	Tensile Strength (MPa)	Elongation at break (%)
Viscose	10.4 ± 0.4	12 ± 2	400 ± 62	15 ± 2.2
Modal	10.2 ± 0.5	14 ± 2	515 ± 65	11 ± 2.2
Modal	9.2 ± 0.6	15 ± 2	482 ± 70	10 ± 1.4
Lyocell	9.7 ± 0.9	27 ± 4	614 ± 72	8 ± 1.4
Lyocell	8.7 ± 0.8	18 ± 5	515 ± 97	6 ± 1.7

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