MICROSTRUCTURE, QUANTIFICATION AND CONTROL OF DISLOCATIONS IN BAST-TYPE PLANT FIBRES

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Keywords: natural fibres, kink bands, microstructure, physical treatments, mechanical properties

Abstract
Bast-type plant fibres are increasingly being used for structural composite applications where high quality fibres with good mechanical properties are required. A central aspect for this application is the existence of dislocations in the cell wall of plant fibres, i.e. regions of misaligned cellulose microfibrils, which are believed to form weak points leading to reduced mechanical properties. In the present study, microstructural observations of dislocations are made using high-magnification scanning electron microscopy. An experimental protocol using polarized optical microscopy and image analysis is presented for the quantification of dislocations in plant fibres. The protocol is evaluated with respect to its robustness, and the uncertainty of the determined content of dislocations. Based on in-situ straining of fibres under the optical microscopy, findings are presented to show that this leads to a reduction in the content of dislocations. This is indicating that dislocations in the cell wall of plant fibres are changeable structures. Preliminary work is presented where plant fibres are exposed to physical treatments involving moisture and mechanical straining in order to change the content of dislocations. The effect of the treatments is evaluated by tensile testing of single fibres.

1. Introduction

The industrial use of plant fibres as reinforcement in composites has increased during the past two decades, and is supported by numerous scienctific studies. In the case of plant fibre composites for structural applications, high quality fibres with good mechanical properties are required, and the existence of dislocations in the cell wall of plant fibres is a central aspect. Dislocations are believed to be caused by compressive loading of the fibres, taking place both during growth of the plants, and during the applied mechanical processing steps to extract the fibres from the plants. Due to the layered construction of the fibre cell wall with variable angles of the cellulose microfibrils, compressive loading of the fibres leads to local lateral bulging (or kinking) of the cell wall. Thus, dislocations are regions of misaligned cellulose microfibrils, which are believed to form weak points leading to reduced mechanical properties.

In the present study, the morphology and content of dislocations in bast-type plant fibres are investigated by using microscope and image analysis techniques. Straining of the fibres, together with increased moisture content, is applied to change the dislocation content, and the effect is evaluated by tensile testing of the fibres.

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2. Materials and methods

Flax and hemp bast-type plant fibres from textile yarns were used in the present study. Flax yarns were purchased from Safilin (France) and Linificio e Canapificio Nazionale (Italy). Hemp yarn was purchased from HempAge (Germany).

Scanning electron microscopy of dislocations on the surface of the fibres was done using a field emission scanning electron microscope (FE-SEM), Supra 35 (Carl Zeiss, Germany) with an in-lens secondary electron detector. More details of sample preparation and microscope settings can be found in the study by Madsen et al. [1].

Quantification of the dislocation content in the fibres was done by polarised optical microscopy. An established protocol was used, which is detailed described in the studies by Mortensen and Madsen [2] and Lester [3]. As shown in Fig. 1a, the fibre samples were prepared by placing a single fibre on a glass slide and by fasten it with double-sided tape at the ends of the slide. A droplet of demineralised water was placed onto the fibre, and another glass slide was then placed on top to encapsulate the fibre and the water. It was found that more clear images were obtained when the fibre was fully water wetted between the glass slides. The effect of absorbed water in the fibre cell wall on the observed dislocations must however be considered. In the optical microscope, two images were acquired at a given fibre location: (i) an image using dark field illumination to have the best possible contrast between the fibre edges and the surrounding background in order to quantify *the fibre area*, and (ii) an image using polarised light to be able to visualize the dislocations within the fibre in order to quantify *the dislocation area*. The image analysis algorithms of the seeded region growing method and the Otsu's method were applied to the images. The dislocation content was determined from the ratio of the measured dislocation area and fibre area. An outline of the applied steps in the protocol is shown in Fig. 1b. By using a custom-made fixture developed in the study by Mortensen [4] for the glass slide with the fibre sample, the fibre could be strained in-situ while positioned under the microscope.

For one sample of hemp fibres, physical treatments were performed involving a combination of strain and moisture. Weights of either 2.6 or 7.0 g were clamped to the ends of single fibres, and thereby, the fibres where strained to values of about 0.8 and 2.0 %, respectively, for 30 minutes. The fibres were wetted with distilled water every 5 minutes. More details can be found in the study by Lester [3].

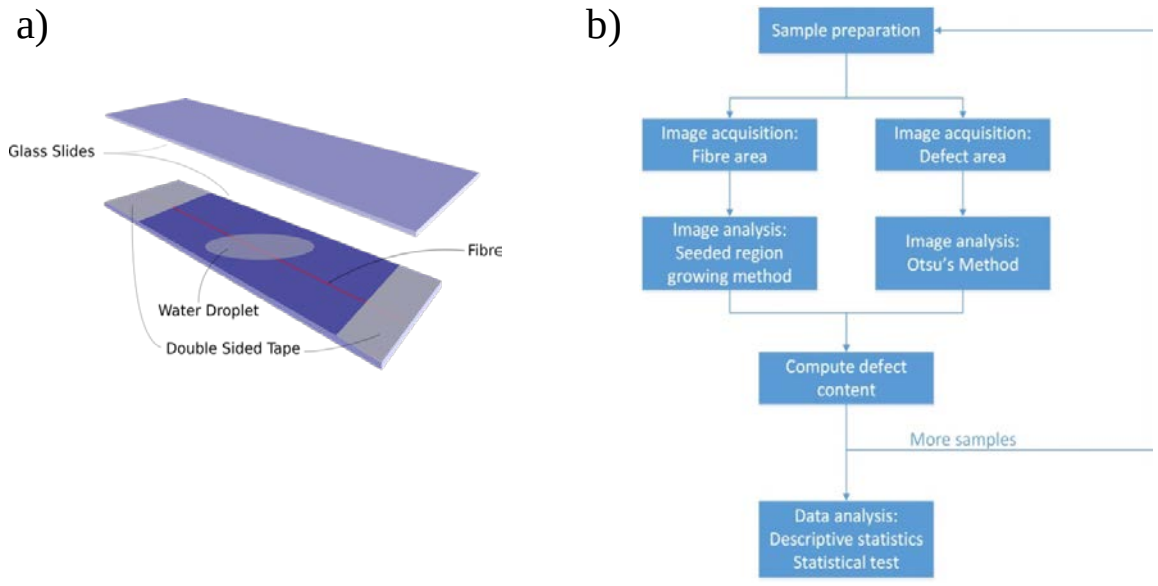

Figure 1. Protocol for quantification of dislocations in plant fibres: (a) sample preparation, (b) outline of steps in protocol. Modified from Mortensen and Madsen [2].

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Mechanical properties of single fibres were measured with a Textechno (Germany) tensile test machine for automated test of single textile fibres. The machine and method are detailed described in the study by Beauson et al. [5]. In the present study, the fibres were tested with a gauge length of 10 mm, and by using a displacement rate of 1 mm/min and a load cell of 6.1 N. The tests were done at room temperature. The cross-sectional area of the fibres, used to convert load into stress, were determined from the measured fibre widths by the above mentioned optical microscopy protocol, and by assuming that the fibres are circular.

3. Results and discussion

The FE-SEM images in Figs. 2, 3 and 4 show the typical appearance of dislocations at the surface of bast-type plant fibres. Bulging bands, which will be referred to as kink bands, with variable widths are distributed along the fibres, as shown in Fig. 2. In Fig. 3, a typical characteristic of kink bands are shown where the bands are having a larger width on the concave side of a bent fibre, giving them a wedge-shaped appearance. This is also shown in Fig. 4 that shows a few fibres arranged in a bundle with a joint wedge-shaped kink band. The wedge-shaped appearance of kink bands can be explained by a more marked kink band formation on the compression side of fibres when they are bent.

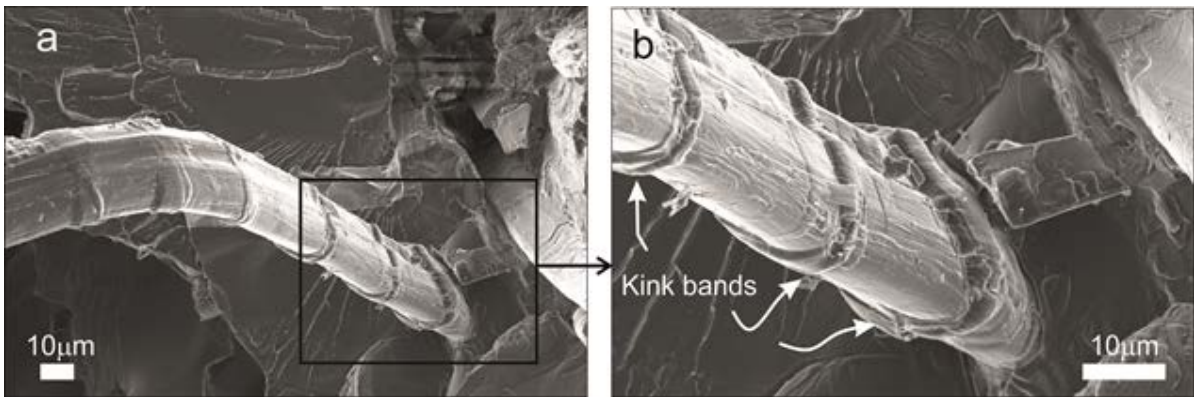

Figure 2. FE-SEM image of a pulled-out flax fibre on a composite fracture surface. Kink bands are distributed along the length of the fibre. Modified from Madsen et al. [1].

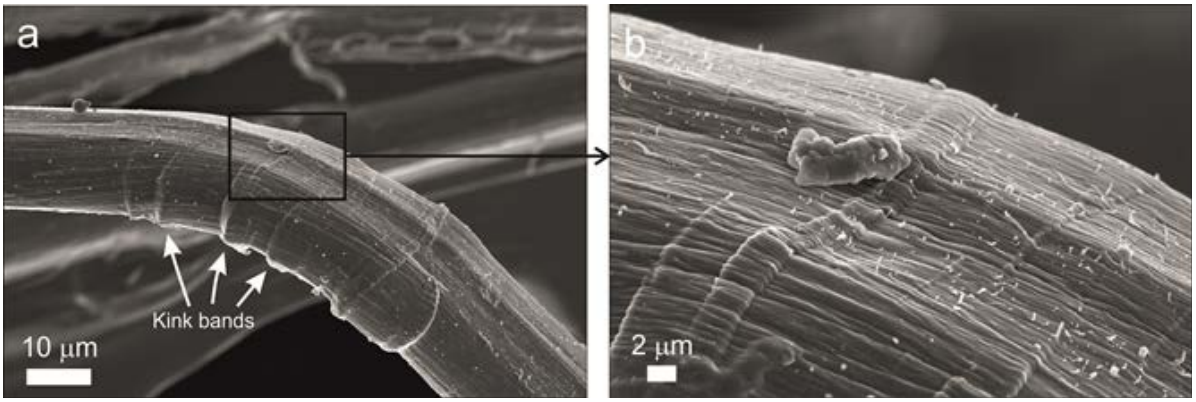

Figure 3. FE-SEM image of kink bands on the surface of a bent flax fibre. Modified from Madsen et al. [1].

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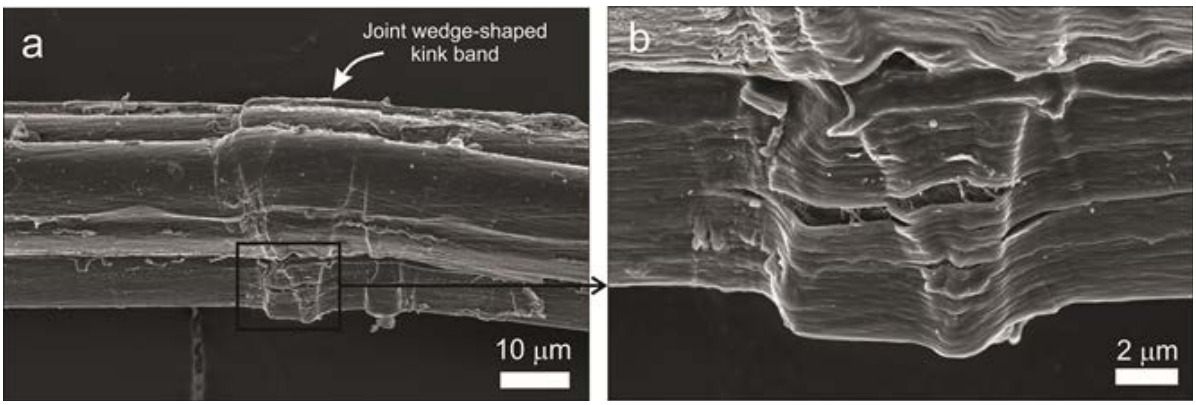

Figure 4. FE-SEM image of a joint kink band in a bundle of flax fibres. Modified from Madsen et al. [1].

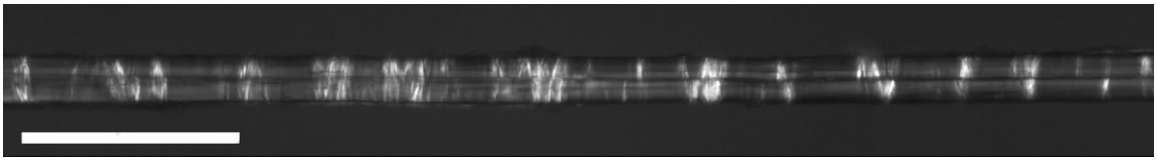

Figure 5. Polarised optical microscope image of a flax fibre showing dislocations located along the fibre. Scale bar is 100 μm. From Mortensen and Madsen [1].

Next, polarised optical microscopy was used to quantify the content of dislocations in the fibres by using image analysis techniques. An example of a polarised optical microscopy image of a flax fibre is shown in Fig. 5. The dislocations in the bulk part of the fibre are clearly visible.

A protocol for quantification of dislocations in plant fibres has been established, and it has been used for various types of fibres [2, 3]. The robustness of the protocol has been evaluated by doing repeated measurements at the exact same location of a fibre, and by resetting focus and brightness of the microscope between the measurements. Results from seven repeated measurements on the same location of a hemp fibre are shown in Table 1. It can be observed that the *fibre area* is measured with low deviation between the repeated measurements. The standard deviation is 180 μm^2 which compared to the overall mean on 13,300 μm^2 gives a coefficient of variation on about 1%. For the measured *dislocation area*, the deviation is however much larger between the measurements. The standard deviation is 90 μm^2, which compared to the overall mean on 130 μm^2 gives a coefficient of variation on about 70%. The standard deviation of the calculated *dislocation content* is 0.003, which compared to the overall mean on 0.010 gives a coefficient of variation on about 30%. Thus, the method in itself has some uncertainty, and this must be taking into account when comparing dislocation contents between fibre types and treatments.

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Table 1. Repeated measurements of dislocation content at the same location of a hemp fibre. Values are mean ± stdv. The overall stdv. in the last row are calculated by taking the stdvs. of the single measurements into account.

Measurement no	Fibre area (μm^2)	Dislocation area (μm^2)	Dislocation content (-)
1	13,280 ± 340	210 ± 70	0.016 ± 0.005
2	13,230 ± 340	150 ± 50	0.011 ± 0.004
3	13,270 ± 320	110 ± 70	0.008 ± 0.005
4	13,240 ± 320	110 ± 70	0.008 ± 0.005
5	13,350 ± 310	100 ± 40	0.008 ± 0.003
6	13,290 ± 340	110 ± 60	0.008 ± 0.004
7	13,450 ± 320	120 ± 50	0.009 ± 0.004
Mean ± stdv.	**13,300 ± 180**	**130 ± 90**	**0.010 ± 0.003**

From the microscopy observations it is known that the distribution of dislocations is largely non-uniform both along single fibres, and between fibres, and therefore, a large number of fibres must be examined to obtain a representative value. Table 2 shows the typical approach used where fibres from two sections of a hemp fibre yarn have been examined, covering 16 fibre samples with 4-6 measurements each, giving 78 measurements in total. It can be observed that there is a considerably large deviation in the determined mean values of dislocation content between measurements, even within the same fibre sample. This deviation is believed to originate both from the method itself (see above), and from the non-uniformity of the material. On the fibre sample level, the difference between mean values is getting smaller. The determined dislocation contents of the two yarn sections are almost similar at 0.02 ± 0.01, which is then also the final value for the yarn batch.

Table 2. Overview of measured dislocation contents of a hemp yarn. 78 measurements were done on 16 fibre samples from 2 yarn sections of a hemp fibre yarn batch.

Yarn batch	Yarn sections	Fibre samples	Measurements
0.020 ± 0.010	0.023 ± 0.013	0.021 ± 0.011	0.019 ± 0.010
			0.022 ± 0.020
			0.012 ± 0.007
			0.022 ± 0.021
			0.031 ± 0017
		…	...
		0.039 ± 0.022	0.076 ± 0.041
			0.022 ± 0.020
			0.041 ± 0.024
			0.017 ± 0.008
	0.017 ± 0.009	0.014 ± 0.007	0.013 ± 0.006
			0.014 ± 0.011
			0.015 ± 0.011
		…	…
		0.021 ± 0.015	0.019 ± 0.017
			…
			0.011 ± 0.003

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Fig. 6 shows an example of the use of the protocol to evaluate the difference in dislocation content between two differently processed bast-type plant fibres. The figure shows results for two types of flax yarn fibres: (i) short technical fibres, a by-product from the scutching process (sample code ESTLYS), and (ii) long technical fibres, a direct product of the scutching process (sample code LML). It can be seen that although the distributions of dislocations content for the two fibre types are overlapping, they are shifted with respect to each other with values for the median on 0.058 and 0.032 for the ESTLYS and LML fibres, respectively. The unequal spacing between the quartiles and fractiles indicates that the distributions are slightly skewed. This is most pronounced for the ESTLYS fibres which are skewed toward the lower dislocation contents. In addition, it can be observed that the spread of data is larger for the ESTLYS fibres. The mean ± stdv. for the dislocation content is determined to be 0.069 ± 0.050 and 0.039 ± 0.031 for the ESTLYS and LML fibres, respectively. Thus, it is indicated that the shorter ESTLYS fibres, being a by-product in the yarn process, show larger dislocation contents than the longer LML fibres.

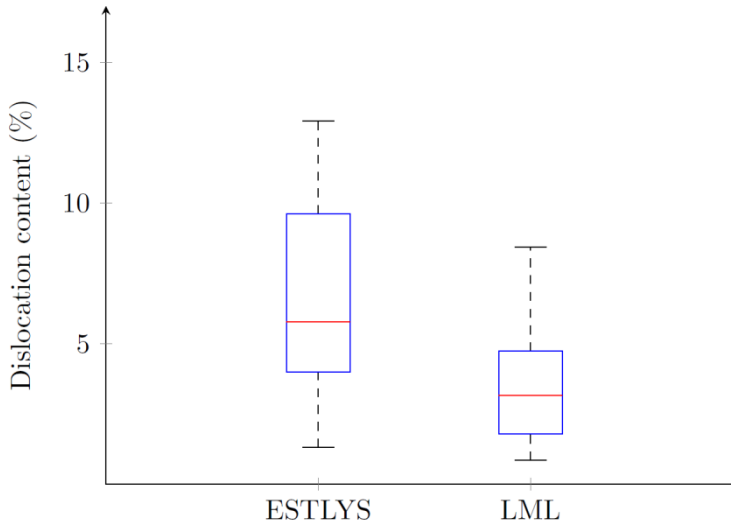

Figure 6. Box plot showing results for the determined dislocation contents for two types of differently processed flax fibres, ESTLYS and LML. The horizontal sides of the blue box are first and third quartiles, the horizontal red line is second quartile (median), and the horizontal black short lines are 0.05 and 0.95 fractiles. From Mortensen and Madsen [2].

Knowing that dislocations in plant fibres are local regions of misaligned cellulose microfibrils, it can be expected that straining of fibres might lead to some degree of microfibril re-alignment, and thereby a reduction in the dislocation content. This was evaluated by straining fibres in-situ while positioned under the polarised optical microscope. Fig. 7a shows results from six flax fibres where the normalised dislocation content is plotted as a function of the strain applied to the fibres. The fibres were strained in small steps up to about 1.2 %. The results are showing that the dislocation content is reduced to low levels of 0 - 20 % of the initial content after having applied strain in the range 0.4 – 0.6 %. For one fibre, it was investigated how the dislocation content was influenced by first straining the fibre to 1.2 %, then de-straining the fibre back to 0 %, and finally, straining it one more time. These results are shown in Fig. 7b. It can be seen that after de-straining back to 0 %, the dislocation content is about 30 % of the initial content. Straining the fibre once more, reduce the dislocation content again to a low level, by following the same curve as for the de-straining. Altogether, the presented results in Fig. 7 demonstrate that dislocations in the cell wall of bast-type plant fibres are changeable structures.

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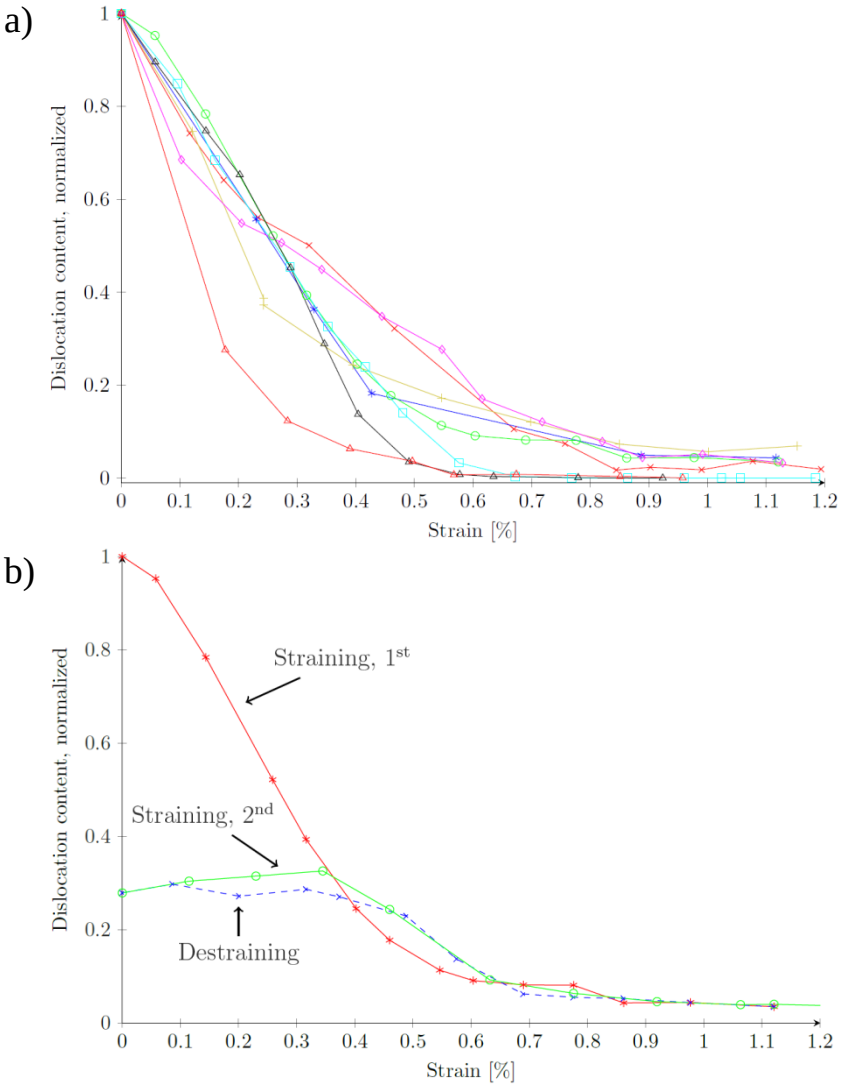

Figure 7. Normalised dislocation content in flax fibres as a function of fibre strain for (a) six different fibres strained one time, and (b) a single fibre strained, de-strained, and then strained again.

Based on the findings on the effect of fibre straining, preliminary work was done to examine whether physical treatments, involving straining together with moisture, can be used to change the dislocation content in plant fibres. The dislocation content of untreated hemp fibres was measured to be 0.020 ± 0.010. After being fully wetted in water, and allowed to dry at room temperature under either low or high strain (as specified in the Materials and methods section), the dislocation content was measured to be 0.016 ± 0.010 and 0.012 ± 0.007, respectively. Thus, there is a tendency that the dislocation content is reduced by the treatments, and that the effect is larger for the larger strain level. The effect of the physical treatments was also evaluated by tensile testing of the fibres. The measured stress-strain curves are shown in Fig. 8. It clearly shows that the stress-strain curves for the treated fibres are shifted upwards with respect to the curves for the untreated fibres. The change is larger for the fibres that were exposed to the low strain level. Stiffness was determined to be 19 ± 5, 32 ± 8 and 27 ± 4 GPa for the untreated, low strained and high strained fibres, respectively. Strength was determined to be 420 ± 160, 540 ± 160 and 460 ± 90 MPa for the untreated, low strained and high strained fibres, respectively. There is a tendency that the high strained fibres are showing lower scatter in mechanical properties than the low strained fibres, which might be due to the lower dislocation content making the fibres more uniform. Further work needs to validate and elaborate the findings from this preliminary work.

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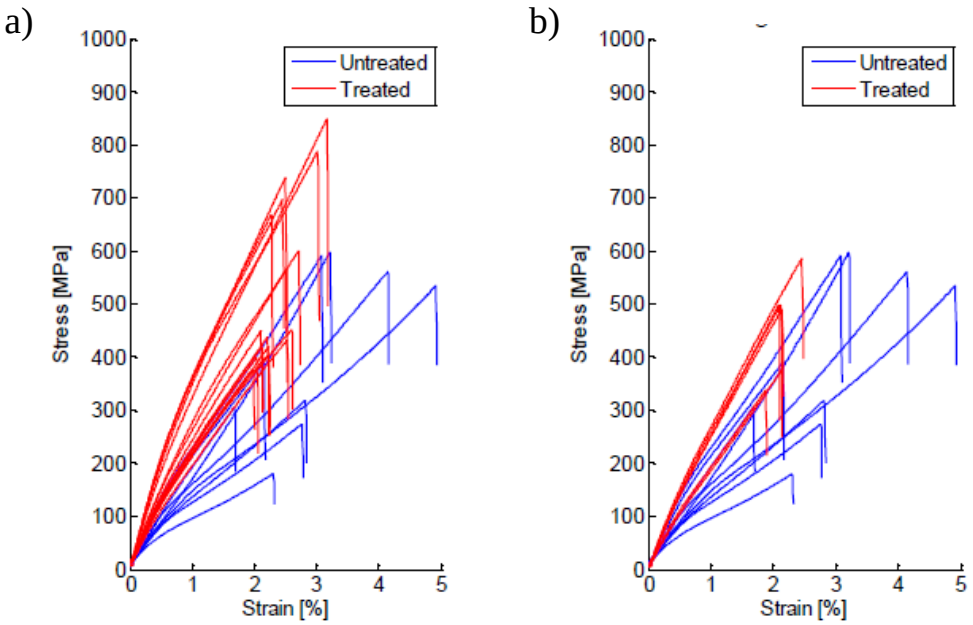

Figure 8. Stress-strain curves of single hemp fibres exposed to physical treatments of being fully wetted in water, and then allowed to dry at room temperature under either (a) low strain, or (b) high strain.

4. Conclusions

Observations by field-emission scanning electron microscopy are presented to show that dislocations are distributed along bast-type plant fibres in the form of kink bands width variable widths. Some kink bands are having a wedge-shaped appearance, which can be explained by a more marked kink band formation on the compression side of bent fibres. The content of dislocations in the fibres was quantified by using an experimental protocol including polarised optical microscopy and image analysis. For two types of differently processed flax fibres, the dislocation content was measured to be 0.069 ± 0.050 and 0.039 ± 0.031, which indicates that the fibre process is influencing the dislocation content. The change of the dislocation content in a fibre by straining the fibre was investigated. The results are consistently showing that the dislocation content is reduced to low levels of 0 - 20 % of the initial content after applying strain in the range 0.4 – 0.6 %. Based on these findings, physical treatments were applied in an attempt to change the dislocation content. For hemp fibres, after being fully wetted in water, and allowed to dry at room temperature under either low or high strain , the dislocation content was measured to be 0.016 ± 0.010 and 0.012 ± 0.007, respectively. The dislocation content of untreated hemp fibres was measured to be 0.020 ± 0.010. Thus, there is a tendency that the dislocation content is reduced by the treatments, and that the effect is larger for the larger strain level. The effect of the treatments was evaluated by tensile testing of the fibres. Stiffness was determined to be 19 ± 5, 32 ± 8 and 27 ± 4 GPa for the untreated, low strained and high strained fibres, respectively. Strength was determined to be 420 ± 160, 540 ± 160 and 460 ± 90 MPa for the untreated, low strained and high strained fibres, respectively. This is consistently showing that the mechancical properties of the fibres are increased by the physical treatments.

Acknowledgments

The authors are grateful to the Danish Council for Independent Research supporting the CelFiMat project (IP No. 12-127446: “High quality cellulosic fibers for strong biocomposite materials”).

Excerpt from ISBN 978-3-00-053387-7

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Excerpt from ISBN 978-3-00-053387-7