

Digital image correlation as a strain measurement technique for fibre tensile tests

Delphine Depuydt*¹, Kevin Hendrickx*², Wouter Biesmans³, Jan Ivens², Aart Willem Van Vuure¹

¹ KU Leuven Department of Materials Engineering, Campus Groep T, Andreas Vesaliusstraat 13,
3000 Leuven

² KU Leuven Department of Materials Engineering, Technology Campus De Nayer, Jan De
Nayerlaan 5, 2860 Sint-Katelijne-Waver, Belgium

³ KU Leuven, Department of Electrical Engineering, Kasteelpark Arenberg 10, 3001 Leuven,
Belgium

* These authors contributed equally to this work

Keywords: A. Fibres, B. Fibre deformation, B. Optical properties/techniques, D. Mechanical testing

Abstract

A method is presented to test fibres in tension using direct strain measurement. This eliminates the need to test the fibres at multiple gauge lengths to correct for machine compliance, reducing the number of samples. Additionally, fibre slippage can contribute to the underestimation of the stiffness since this is not considered in the correction procedure. Steel fibres with a diameter of 30 μm , and a known stiffness of 193 GPa, were tested in tension using indirect methods and the direct strain method. Direct strain measurement resulted in a stiffness of 187 ± 12 GPa while the lowest and highest stiffness obtained by the indirect methods are 140 ± 2 GPa and 150 ± 4 GPa. The underestimation by the indirect measurement strain methods show the need for a new

method. To demonstrate the applicability of the new test method to natural fibres, the properties of *technical* flax and bamboo fibres were determined.

Introduction

Fibre tensile tests are a way to determine the fibre's mechanical properties. It is the preferred method when limited material is available, in material development stage. The fibre's tensile properties are needed to perform micromechanical analyses and mechanical modelling of these materials and their composites. For synthetic fibres, the fibre properties are often provided by the manufacturers. However, when testing new materials, and especially from natural resources, datasheets are often not available.

Natural fibres are becoming increasingly important as a reinforcing fibre in composite materials. Therefore, the need for consistent fibre properties increases. Additionally, fibre manufacturers, or cultivators in the case of natural fibres, should be able to rely on fibre properties to measure the quality of the produced material. Unfortunately, the literature reveals a large range of the measured natural fibre properties, as can be seen in Table 1. A part of this variation can be attributed to the inherent variability in plant materials, but this is unlikely to be a complete explanation. There is a need for a consistent fibre testing method where the scatter is only a function of the material variability and not of the testing setup. Summerscales et al. [1] provide a checklist for the material related data and testing conditions that always should be reported in order to compare the experimental data with literature.

There are a few problems when testing natural fibres, that can lead to a wide range of results [2, 3]. Firstly, the literature does not always clearly state whether tests are performed on an *elementary* fibre (a single plant cell) or a *technical* fibre (a bundle of *elementary* fibres). It is well recognised that *elementary* fibres have higher stiffness and strength compared to *technical* fibres

[4]. Secondly, when testing *technical* fibres at very short gauge lengths, *elementary* fibres can get gripped from end to end. The short gauge length reduces the probability of finding a weak location and reduces the effects of the weak inter-*elementary* fibre middle lamellae [3]. Besides the problems mentioned above, some other test related influencing factors are known: the strain rate (viscoelastic behaviour of the fibres), the environmental conditions (hygroscopic behaviour of natural fibres) and the gripping method (gripping of frame or fibre) should therefore always be reported [5, 6].

Haag et al. [2] summed up the previous issues, and added the importance of the determination of the cross sectional area. Via different optical techniques such as flatbed scanner, light microscopy and laser based analysis they determined, from the projected diameter, the cross-section of the fibre. Furthermore, the authors tried different calculations to determine the cross-sectional area, assuming either a circular or an elliptical shape. It was seen that the scatter induced by the different calculation methods, resulted in a large variation which is partly responsible for the high scatter on the natural fibre properties. Thomason et al. [7] embedded *technical* flax and sisal fibres after tensile testing, and traced the contour of the fibre to determine the cross sectional area. This method revealed a large difference between 'projected diameter techniques' and true diameter determination. In this research a gravimetric method was used, for the determination of fibre diameter, as is outlined in Defoirdt et al. [9]. It is believed that this method to determine the cross-section is user independent and can lead to a reduced spread on the fibre properties of natural fibres, avoiding the struggle of assuming a cross-sectional geometry of the irregular fibre and avoiding numerous measurements for each fibre.

A final influencing factor, which is normally not addressed, is the importance of a correct strain measurement method, and this is investigated in this paper. The strain in a fibre tensile test is

often measured indirectly via the grip displacement, due to the difficulties in measuring it in a direct manner. To compensate for the system compliance, ASTM C1557-14 [8] prescribes a procedure to perform the data reduction. Defoirdt et al. [9] have developed an alternative procedure to correct the strain values to calculate the fibre properties. However, for both methods it is necessary to test at least at 3 different gauge lengths. Hence, the method requires a significant number of fibres. It remains to investigate whether the correction on the strain for system compliance covers all the effects occurring during a tensile test. Fuentes et al. [10] developed a technique to measure local *technical* fibre properties by applying a speckle pattern on the fibre itself. The analysed length hereby is a few millimetres, therefore giving insight in the local behaviour of the fibre. With the strain mapping it was also possible to indicate movements of *elementary* fibres.

In this research, a novel method is presented for direct measurement of strain during single fibre tensile testing. The new technique is compared to the existing methods, including the different ways of data reduction for tensile fibre tests. Cold drawn steel, a material with known properties, is used to assess the accuracy of each method. Finally, two types of natural fibres, bamboo and flax are characterised by the new technique.

1 Materials & Methods

1.1 Steel fibres

Cold drawn stainless steel (type 316L) fibres with a Young's modulus of 193 GPa and a diameter of 30 μm were supplied by NV Bekaert SA. The continuous steel fibres are produced in a bundle drawing process in which steel wires are embedded in a copper matrix and subsequently cold drawn to reduce the bundle diameter. After this process the copper matrix is removed. The cross-sectional area of the fibres after drawing has a constant value but has a polygonal appearance

[11]. Optical microscopy showed that the fibre diameter did not vary along the fibre length. The average cross sectional area of the fibre (A) was calculated by equation 1.

$$A = \frac{m}{\rho l} \quad (1)$$

In the above equation m is the fibre mass, ρ is the fibre density and l is the fibre length. The fibre mass was determined with an analytical balance (Mettler AT 261 DeltaRange, Mettler Toledo) accurate to 10^{-5} g. The density of the steel fibres was 8.00 g/cm^3 .

1.2 Flax

Technical flax fibres (*Linum usitatissimum* L.) were sampled from FlaxTape 200, a unidirectional flax fibre tape for composite applications with an areal density of 200 g/m^2 , supplied by Lineo NV. The tape consists out of scutched and aligned *technical* fibres, originating from different harvests to average out variations in the fibre properties. The density of the fibres was determined using a gas pycnometer, Beckman model 930, in which helium gas at a pressure of 0.5 bar was used as the displacement medium. Prior to the density measurement, the fibres were cut to a length of 1 cm and vacuum dried for 19 hours at 60°C . The measured density was $1.47 \pm 0.01 \text{ g/cm}^3$. For the tensile tests, the fibres were cut to a length of 10 cm, dried for 24 hours at 60°C and subsequently conditioned at 50% relative humidity (RH) and 21°C for at least 24 hours. In the latter condition, the mass of the fibres was measured to calculate the average cross sectional area using equation 1.

1.3 Bamboo

Bamboo fibres (*Guadua angustifolia* Kunth) were sourced from Columbia, the Coffee Region, at 1300 meters above sea level with an annual average temperature of 23°C and an annual average precipitation of 2200 mm and a relative humidity of 80% according to the environmental authorities of the region [12]. From the bamboo culms, which have an average culm diameter of

11 cm and a height of 20-23 meter, fibres were fully mechanically extracted from the middle part of the culm using an in house developed technique [12]. The density and average cross sectional area of the fibres was determined following the same procedure and the same specimen preparation as for the flax fibres. The measured density was $1.36 \pm 0.02 \text{ g/cm}^3$.

1.4 Fibre tensile test set-up

To compare the different existing methods, steel fibres with known Young's modulus were chosen to experimentally determine the fibre modulus. The fibre was glued onto abrasive paper (PS11A grain 1000, Klingspor) frame using a double-sided glue roller (Permanent Pritt glue roller, Henkel). Although, this method of gripping may lead to premature failure of the fibre due to damage, this is irrelevant to determine the fibre modulus. Figure 1 shows the frame dimensions for a test gauge length of 50 mm and the position of the fibre in this frame. The frame facilitates sample mounting and fibre alignment in the grips, whereas the abrasive paper minimises fibre slippage during the test.

Tensile tests were performed on an Instron 5943 equipped with a 100 N load cell according to the ASTM C1557-14 [8] standard in a conditioned environment at 50% RH and 21°C. The frame was pneumatically gripped with a gripping force of 200 N. A pre-load of 0.01 N was applied to the fibre to straighten it. Fibre straightness is critical when the strain is to be derived from the crosshead displacement. Furthermore, the upper grip is connected to the load cell with a ball joint which ensures the alignment of the fibre in the first stages of the test. The crosshead displacement rate was chosen according to the ASTM C1557-14 standard, which suggests to achieve fracture within 30 seconds of testing [8]. For the investigated fibres, this translates to a crosshead displacement rate of 1.5 mm/min.

1.5 Indirect strain measurement

In indirect strain measurement, the crosshead displacement is assumed equal to the axial extension of the fibre. When indirect strain measurement is used, the measured deformation can be partly due to the deformation of the test machine and slippage of the sample in the grips. To correct for the deformation of the test machine, samples with different gauge lengths are needed. Four different gauge lengths were used in this study: 10 mm, 20 mm, 35 mm and 50 mm. For the respective gauge lengths, 17, 20, 20 and 22 steel fibres were successfully tested.

1.6 Direct strain measurement

A method was developed to optically measure the fibre strain. The technique requires some additional modifications to the samples and the test set-up. The specimens were attached to an abrasive frame as outlined above. Additionally, two optical flags with an approximate diameter of 3 mm were attached to the fibre surface using white correction fluid (correction pen Tipp-Ex, Bic World). On these white flags a black speckle pattern was applied with spray paint, as shown in Figure 1.

The gauge length was fixed to 50 mm to manipulate the fibres easily, 22 steel fibres were successfully tested. This method requires only one gauge length to be tested since deformations due to slippage between fibre and grips are not registered. Moving to shorter or longer gauge lengths will increase or decrease the strength respectively, but leave stiffness unaffected for synthetic fibres. In natural fibres also the stiffness can be affected when the gauge length becomes so small, that at least one *elementary* fibre is gripped from end to end, this results in an increase in the stiffness [3]. In this research 24 bamboo fibres and 18 flax fibres were successfully tested.

Rotations or movements in the plane of the sand paper do not affect the strain measurement as in the case of indirect strain measurement. The out-of-plane motion (forward and backward fibre

movement relative to the camera) does affect the strain measurement significantly and care should be taken to limit this movement during the test [5].

For the registration of the images during tensile testing, a digital camera (*Limess messtechnik & software GmbH*, Krefeld, Germany) with a spatial resolution of 96 dpi, equipped with a Componon-S 2.8/50 lens (Schneider Kreuznach, Bad Kreuznach, Germany), were used. The distance between sample and camera was 1 m. Samples were illuminated frontally with a 350 W halogen lamp. Subsequent images and force registration were taken during the test with an interval of 150 ms using acquisition software (LimShot, *Limess messtechnik & software GmbH*, Krefeld, Germany).

Data reduction

The accuracy of a tensile test depends on the accuracy of the force and strain measurement during the test. When the strain of the fibre is indirectly measured via the crosshead displacement a correction for the system compliance is made to deduce the fibre strain and fibre stiffness. Three different methods were applied to correct for the system compliance. The fourth method is based on direct strain measurement and does not require this correction.

- Method 1: Method as described in ASTM C1557-14 [8],
- Method 2: Method as described by Defoirdt et al. [9],
- Method 3: Modification of method 2
- Method 4: Digital image correlation

1.6.1 Method 1: Data reduction according to ASTM C1557-14 [8]

In ASTM C1557-14 [8], the method to determine the fibre modulus takes into account the deformation of the test set-up. The crosshead displacement resulting from this increase in deformation per unit of force (F) is designated as the system compliance (C_s). The system

compliance is assumed to be constant for a given test apparatus, gripping system and fibre type. Consequently, $C_s F$ is the part of the total crosshead displacement (ΔL) that is not attributable to fibre deformation. Considering the previous, equation 2 is valid which expresses the total crosshead displacement as the sum of fibre elongation (Δl_f) and set-up elongation ($C_s F$).

$$\Delta L = \Delta l_f + C_s F \quad (2)$$

Dividing equation 2 with the applied force and applying Hooke's law to replace the fibre elongation, equation 3 is obtained in which l_0 is the initial gauge length and E_f is the fibre Young's Modulus.

This proves the linear relation between $\frac{\Delta L}{F}$ and $\frac{l_0}{A}$. Let $\frac{\Delta L}{F}$ be the inverse of the slope taken in the linear part of the measured force-displacement curve then the slope of the linear regression line of $\frac{\Delta L}{F}$

$$\frac{\Delta L}{F} = \frac{l_0}{A E_f} + C_s \quad (3)$$

versus $\frac{l_0}{A}$ for different gauge lengths equals $1/E_f$. The method is graphically illustrated in Figure 2.

In order to apply this method, tests on different $\frac{l_0}{A}$ ratios are necessary. ASTM C1557-14 suggests testing at least 3 different gauge lengths [8].

1.6.2 Method 2: Data reduction according to Defoirdt et al. [9]

An alternative method to perform the data reduction to obtain the fibre modulus has been proposed by Defoirdt et al. [9]. For each sample the apparent fibre modulus is calculated as defined in equation 4.

$$E_{app} = \frac{Fl_0}{A\Delta L} \quad (4)$$

$$\lim_{l_0 \rightarrow \infty} \Delta L = \Delta l_f \quad (5)$$

At infinite gauge length, equation 3 transforms into equation 5, where ΔL equals Δl_f as $C_s F$ becomes negligibly small. The regression line of E_{app} versus $\frac{1}{l_0}$ intersects the y-axis at E_f . The method is illustrated in Figure 2, where E_{app} is the stiffness found as the slope in the linear part of the stress-strain curve, derived from the measured force and displacement of the machine. Apart from a constant system compliance it also assumes the existence of a linear relationship between E_{app} and $\frac{1}{l_0}$.

1.6.3 Method 3: Modification to the data reduction by Defoirdt et al. [9]

By following the substitutions outlined below, equation 7 is obtained which reveals that the true relationship between E_{app} and $\frac{1}{l_0}$ is a rational function.

$$E_{app} = \frac{Fl_0}{A\Delta L} = \frac{l_0 F}{A(\Delta l_f + C_s F)} \quad (6)$$

Combining equation 6 with $F = \frac{E_f \Delta l_f A}{l_0}$ and dividing by Δl_f

$$E_{app} = \frac{E_f}{1 + C_s E_f A \frac{1}{l_0}} \quad (7)$$

Finding E_f is identical to the unmodified method except that nonlinear least-squares fitting must be performed to fit the data to the rational function. The Levenberg-Marquardt algorithm [13, 14] was used to fit the experimental data to $E_{app} = \frac{A}{1+B*\left(\frac{l}{l_0}\right)}$ with A and B free variables. The optimized A value equals the effective fibre modulus E_f . The method is schematically represented in Figure 2.

1.6.4 Method 4: Digital image correlation

Images were processed with Vic 2D 2009 (*Correlated Solutions*, Columbia, USA) correlation software to determine the pixel displacement of both optical flags. The subset window and step size were set to 15 and 3 pixels, respectively. Finally, the relative displacement of the optical flags was calculated and the fibre strain, based on the relative pixel displacements, was extracted.

1.7 Statistical analysis

To determine whether the sample data has been drawn from a normally distributed population, a Shapiro-Wilk test is performed. The null hypothesis for the test is that the data are normally distributed. For a chosen alpha level of 0.05, the null hypothesis is rejected when the p-value is less than 0.05. For normally distributed data, the results are summarized using the mean \pm standard deviation notation.

To detect significant differences between group means, one sample, two-sided t-tests were performed. It was assumed that the population mean of the steel fibres was known and equal to 193 GPa. 95% confidence intervals of methods 1 to 3 were constructed according to the methods described in [15]. The 95 % confidence interval for the developed method was based on the t-statistic. To compare the sensitivity of the estimated modulus to random variations in the measured sample values, a Monte Carlo simulation was run for all three testing methods.

2 Results and discussion

2.1 Determination of the fibre modulus using indirect strain measurement

The results for the modulus of the steel fibres, according to the three different methods, are presented in Table 1: Mechanical properties of flax and bamboo technical fibres.

| Young's modulus (GPa) | Strength (MPa) | Failure strain (%) | Comment | Ref. |
|-----------------------|---------------------------|---------------------------|--|------|
| Flax | | | | |
| 31 ± 12 | 305 ± 120 | 1.3 ± 0.4 | Gauge 75mm, 1mm/min, green flax | [4] |
| 32 ± 12 | 310 ± 120 | 1.1 ± 0.4 | Gauge 75mm, 1mm/min, retted flax | [4] |
| Bamboo | | | | |
| 33.37 | 639 ± 175 - 813 ± 94a | 2.0 ± 0.6 - 2.9 ± 0.7a | Gauge 5,15,25,35, 1mm/min, steam explosion | [9] |
| 43 | 775 ± 103 - 860 ± 119a | 1.7 ± 0.2 - 1.9 ± 0.3a | Gauge 5,10,25,40, 1mm/min, mechanical extraction | [16] |
| 35.91 | 503 | 1.4 | Mechanical extraction | [20] |
| 19.67 | 341 | 1.73 | Chemical extraction | [20] |
| 35.9 ± 13.1 | 441 ± 220 | 1.3 | 1mm/min, steam explosion | [21] |

^{aweakest} test length – strongest test length

Table 2, whereas Figure 3 to Figure 5 indicate how the results were obtained. The modulus for the steel fibre obtained via Method 1 is 147 ± 4 GPa and via Method 2 140 ± 2 GPa. Both methods show only a minor spread on the results, reflected in their low standard deviation. However, the estimate of the Young's modulus is poor, since the real fibre modulus (193 GPa) is significantly different from the test result ($p=10^{-64}$). In paragraph 1.6.3 the assumption of a linear relation between the apparent stiffness E_{app} and the inverse of the gauge length ($\frac{1}{l_0}$) was rejected and has been demonstrated to follow a rational relationship. Figure 5 shows the fit of this rational function to the experimental data. The predicted fibre stiffness based on method 3 is 150 ± 4 GPa, which is 7.3% larger than for method 2. Though the accuracy increases, this value still underestimates the effective stiffness of the steel fibres by as much as 25%.

It must be concluded that neither method 1, nor method 2 or 3 is able to adequately correct the measured strain values and obtain a correct value for the Young's Modulus. This is caused by the assumption of a constant system compliance. The system compliance was created as a constant that corrects for the elastic deformations of the machine parts. However, it does not correct for slippage that can occur between fibre and frame or between frame and grip. It could be argued that a different sample preparation method would lead to a decreased contribution of slippage to the total deformation. Indeed, this might be correct but slippage effects are generally too small to detect visually, requiring advanced techniques to correct for the previous, generating an additional disadvantage of the indirect method. The crosshead displacement attributable to slippage is unpredictable and is not necessarily a function of the applied force. Therefore, equation 2 no longer holds rendering all indirect strain methods invalid. The increase in crosshead displacement not attributable to fibre elongation, generated by this effect can lead to a significant underestimation of the fibre modulus. This effect is especially pronounced for very stiff fibres where the material elongation is small. Here, a small increase in crosshead displacement

attributable to slippage can lead to an extremely high relative error on the material elongation and hence on the fibre stiffness.

Considering the challenge it would be to take slippage effects into account, the authors hypothesise that the solution must be found in an alternative, direct strain measurement method.

Sensitivity analysis

To compare the sensitivity of the estimated modulus to random variations in the measured sample values, a Monte Carlo simulation was run for all three testing methods. In this simulation, fixed, experimentally obtained values were chosen for the parameters depicted in Table 3. From these values and by using equation 2, the average $F/\Delta L$ was calculated for each gauge length.

Gaussian noise was added to the simulated values $F/\Delta L$, modeling random variations in the measured samples. This way, a simulated set of 17 samples is created for each gauge length. The standard deviations of the Gaussian noise were chosen equal to the experimentally determined standard deviations for each gauge length. To justify the generation of Gaussian distributed data a Shapiro-Wilk normality test was performed on the experimental data of $\Delta L/F$ and $F/\Delta L$. It was concluded that both data series originate from a normally distributed population ($\alpha=0.05$). From the generated samples, the modulus was calculated according to the different methods. This process was repeated 10 000 times.

To evaluate the accuracy of the different methods, the root-mean-square error (RMSE) was calculated from equation 8. In this equation, E_i represents the fibre modulus that is calculated in each iteration of the Monte Carlo simulation. By comparing the RMSE for the different methods in Table 4, it can be seen that method 2 is the most sensitive to variations of the force-displacement

data. To explain this difference, a second indicator, the relative bias (RB), was calculated according to equation 9. For high iteration numbers, the bias on the modulus should converge to zero.

$$\text{RMSE} = \sqrt{\frac{\sum_{i=1}^n (E_i - E_f)^2}{n}} \quad (8)$$

$$\text{RB} = \frac{\sum_{i=1}^n (E_i - E_f)}{n E_f} \quad (9)$$

Figure 6 shows the relation of the bias to the number of Monte Carlo iterations. It can be seen that method 2 is constantly underestimating the stiffness by around 8%, which explains the high RMSE for this method. Method 1 and method 3 both have a negligible bias and an equally low RMSE, from which we can conclude that the methods are equally accurate in the estimation of the fibre modulus.

Determination of the fibre modulus using direct strain measurement

The steel fibre modulus obtained by digital image correlation is 187 ± 12 GPa, as listed in Table 5. Even though the actual modulus (193 GPa) falls within the confidence interval of the test result, a larger variability is found in comparison with the previous three methods. However, the bias on the result is significantly lower. Comparing the relative bias on the results of Table 2, 21%-26% with the relative standard deviation found in the digital image correlation experiment, 6%, it becomes clear that the relative bias on method 1-3 is bigger than the relative standard deviation found in the digital image correlation method. However, possible routes to further decrease the variability of the method are described in Table 6. The proposed technique is promising although further optimization is required to decrease the standard deviation on the mean. In this case,

digital image correlation is the preferred method for the determination of the strain and stiffness.

Digital image correlation is in this case the preferred method for the determination of the strain and stiffness.

The mechanical properties of *technical* bamboo and flax fibres were measured using digital image correlation. For the bamboo *technical* fibre, a modulus of 51.8 ± 6.8 GPa was measured with an average failure strain of 1.33 ± 0.20 %. The stiffness exceeds the values measured in literature [16], due to the improved measurement of the fibre strain. Flax *technical* fibre modulus was determined to be 40 ± 11 GPa with a strain to failure of 1.64 ± 0.32 %. This result corresponds well with data found in literature for *technical* flax fibres [4, 17]. However, the values in the previous references should be interpreted with caution as they utilise method 1 to calculate the fibre stiffness. Note that this stiffness value is not representative for the behaviour of flax fibres in composite materials where the behaviour of *elementary* fibres dominates the composite stiffness instead of the *technical* fibres. This has been extensively discussed in Shah et al. [3].

Opportunities in direct strain measurement on fibres

Direct strain measurement is especially valuable when testing natural fibres. A large portion of the natural fibres has not been screened for their potential use as a reinforcing fibre in composites.

Using the conventional indirect strain methods, testing a new fibre requires large amounts of fibres and is time consuming [18]. Moreover, extracting the fibres from the plants can be difficult.

ASTM C1557 advises to use at least 3 different gauge lengths. Using the presented method there is no need to vary the gauge length of the fibres hence, therefore three times fewer samples are required.

In conjunction with this, the usefulness of testing single *technical* natural fibres is sometimes questioned. Indeed, certain natural fibres, such as *technical* flax fibres, underperform in single fibre tests relative to their performance in composites. Nevertheless, fibre testing can serve to first screening tool and to investigate potential extraction issues.

Optical strain measurement allows to measure local strains directly on the fibre surface [10]. It has been shown that appropriate speckle patterns can be produced, even at the micro-scale [19]. This technique is promising to produce full-field strain maps of the fibres during deformation and to investigate features such as defects.

As discussed in previous sections, during optical strain registration some environmental or experimental parameters may influence the test result. A summary of the most important factors, together with possible mitigations, is given in Table 6.

3 Conclusions

Measuring mechanical properties of fibres is crucial to gain more insight in the composite mechanics. Although standardized, the current measurement and especially data reduction methods are prone to significant error. According to the data reduction method proposed by ASTM C1557-14 [8] the modulus of 316L steel fibres was underestimated by 25%. This was due to an amount of crosshead displacement that did not result in fibre deformation. Instead, the additional displacement could be caused by slippage between fibre and frame and/or between frame and grip.

Indirect strain measurement is therefore not advised to determine the fibre's modulus and strain to failure since slippage is difficult to detect during or after the test. Hence, the results produced with this method when slippage occurs should be considered invalid, even when the standard deviation on the method would be small. Direct strain measurement based on digital image

correlation eliminates the effect of slippage because the deformation of the fibre is registered optically. Using this direct method, the measured mean stiffness is highly accurate. The measured stiffness of the above mentioned fibres was not statistically different from the effective fibre stiffness. However, the spread on the result was increased compared to the indirect measurement methods. Indeed, two-dimensional optical strain measurement is sensitive to lighting conditions and out-of-plane fibre motion. The latter problem could possibly be eliminated by moving towards stereo-2D optical strain measurement, leaving direct strain measurement as the preferred method in determining the fibre tensile modulus.

Direct strain measurement offers the advantage of reducing the number of tests compared to indirect techniques, all requiring tests at different gauge lengths. This creates a significant opportunity for the evaluation of natural fibre properties where material scarcity and extraction difficulties often prevent the use of an indirect method.

Acknowledgements

The authors would like to acknowledge the support of the Agency for Innovation and Entrepreneurship Flanders via the Strategic Initiative Materials in Flanders in the framework of the SIM-ICON FlaxPreComp project (IWT 140149). Secondly the authors thank the Impulse Fund KU Leuven (BOF/IMP14/034).

References

- [1] Summerscales J, Le Duigou A, Baley C. A checklist for the description of plant/vegetal/bast natural fibre reinforced composites. ICCM 20th International Conference on Composite Materials. Copenhagen2015.
- [2] Haag K, Mussig J. Scatter in tensile properties of flax fibre bundles: influence of determination and calculation of the cross-sectional area. *Journal of Materials Science*. 2016;51(17):7907-17.
- [3] Shah DU, Nag RK, Clifford MJ. Why do we observe significant differences between measured and 'back-calculated' properties of natural fibres? *Cellulose*. 2016;23(3):1481-90.

- [4] Alix S, Lebrun L, Marais S, Philippe E, Bourmaud A, Baley C, et al. Pectinase treatments on technical fibres of flax: Effects on water sorption and mechanical properties. *Carbohydrate Polymers*. 2012;87(1):177-85.
- [5] Kim JH, Heckert NA, Leigh SD, Kobayashi H, McDonough WG, Rice KD, et al. Effects of fiber gripping methods on the single fiber tensile test: I. Non-parametric statistical analysis. *Journal of Materials Science*. 2013;48(10):3623-37.
- [6] Kim JH, Heckert NA, Mates SP, Seppala JE, McDonough WG, Davis CS, et al. Effect of fiber gripping method on the single fiber tensile test: II. Comparison of fiber gripping materials and loading rates. *Journal of Materials Science*. 2015;50(5):2049-60.
- [7] Thomason JL, Carruthers J, Kelly J, Johnson G. Fibre cross-section determination and variability in sisal and flax and its effects on fibre performance characterisation. *Composites Science and Technology*. 2011;71(7):1008-15.
- [8] ASTM C1557-14, Standard Test Method for Tensile Strength and Young's Modulus of Fibers, ASTM International, West Conshohocken, PA, 2014, www.astm.org
- [9] Defoirdt N, Biswas S, De Vriese L, Tran LQN, Van Acker J, Ahsan Q, et al. Assessment of the tensile properties of coir, bamboo and jute fibre. *Composites Part a-Applied Science and Manufacturing*. 2010;41(5):588-95.
- [10] Fuentes CA, Willekens P, Hendrikx N, Lemmens B, Claeys J, Crouchs J, et al. Microstructure and mechanical properties of hemp technical fibres for composite applications by micro computed tomography and digital image correlation. *ECCM17 17th European Conference on Composite Materials*. Munich, Germany 2016.
- [11] Callens MG, Gorbatiikh L, Verpoest I. Ductile steel fibre composites with brittle and ductile matrices. *Composites Part A: Applied Science and Manufacturing*. 2014;61:235-44.
- [12] Trujillo E. Polymer composite materials based on bamboo fibers. In: Leuven K, editor. *Leuven 2014*.
- [13] Levenberg K. A Method for the Solution of Certain Non-Linear Problems in Least Squares. *Quarterly of Applied Mathematics*. 1944;2:164-8.
- [14] Marquardt DW. An Algorithm for Least-Squares Estimation of Nonlinear Parameters. *SIAM Journal on Applied Mathematics*. 1963;11(2):431-41.
- [15] Pomerantsev AL. Confidence intervals for nonlinear regression extrapolation. *Chemometrics and Intelligent Laboratory Systems*. 1999;49(1):41-8.
- [16] Osorio L, Trujillo E, Van Vuure AW, Verpoest I. Morphological aspects and mechanical properties of single bamboo fibers and flexural characterization of bamboo/epoxy composites. *Journal of Reinforced Plastics and Composites*. 2011;30(5):396-408.
- [17] Bos HL, Van den Oever MJA, Peters O. Tensile and compressive properties of flax fibres for natural fibre reinforced composites. *Journal of Materials Science*. 2002;37(8):1683-92.
- [18] Lefeuvre A, Bourmaud A, Lebrun L, Morvan C, Baley C. A study of the yearly reproducibility of flax fiber tensile properties. *Industrial Crops and Products*. 2013;50:400-7.
- [19] Mehdikhani M, Aravand M, Sabuncuoglu B, Callens MG, Lomov SV, Gorbatiikh L. Full-field strain measurements at the micro-scale in fiber-reinforced composites using digital image correlation. *Composite Structures*. 2016;140:192-201.
- [20] Rao KMM, Rao KM. Extraction and tensile properties of natural fibers: Vakka, date and bamboo. *Composite Structures*. 2007;77(3):288-95.
- [21] Okubo K, Fujii T, Yamamoto Y. Development of bamboo-based polymer composites and their mechanical properties. *Composites Part a-Applied Science and Manufacturing*. 2004;35(3):377-83.
- [22] Wittevrongel L, Badaloni M, Balcaen R, Lava P, Debruyne D. Evaluation of Methodologies for Compensation of Out of Plane Motions in a 2D Digital Image Correlation Setup. *Strain*. 2015;51(5):357-69.

- [23] Tay CJ, Quan C, Huang YH, Fu Y. Digital image correlation for whole field out-of-plane displacement measurement using a single camera. *Optics Communications*. 2005;251(1-3):23-36.
- [24] Badaloni M. Optimization and design of tests for material characterization using simulated experiments. In: KU Leuven FoES, editor. 2016.
- [25] Pan B, Lu Z, Xie H. Mean intensity gradient: An effective global parameter for quality assessment of the speckle patterns used in digital image correlation. *Optics and Lasers in Engineering*. 2010;48(4):469-77.
- [26] Hua T, Xie HM, Wang SM, Hu ZX, Chen PW, Zhang QM. Evaluation of the quality of a speckle pattern in the digital image correlation method by mean subset fluctuation. *Optics and Laser Technology*. 2011;43(1):9-13.
- [27] Pan B, Qian KM, Xie HM, Asundi A. Two-dimensional digital image correlation for in-plane displacement and strain measurement: a review. *Measurement Science and Technology*. 2009;20(6).

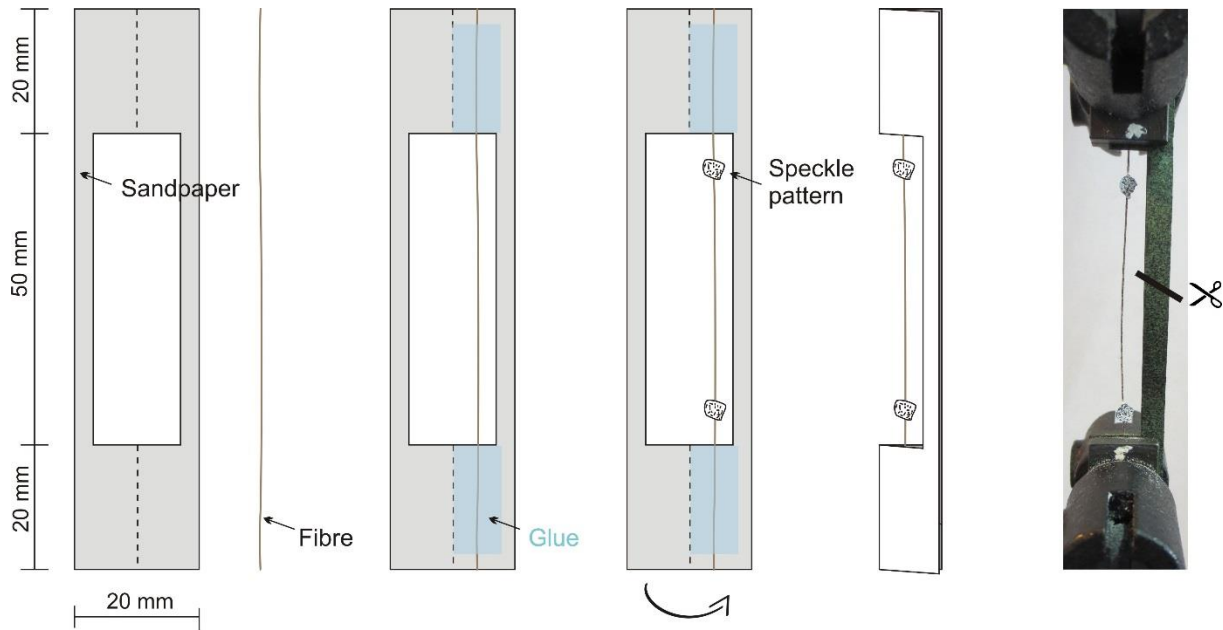


Figure 1: Sandpaper frames and fiber preparation for the fibre tests. The frame is folded to increase fibre gripping during the test. Indirect strain measurement does not require optical flags to be attached to the fibre whereas direct strain measurement relies on these optical flags to measure the strain.

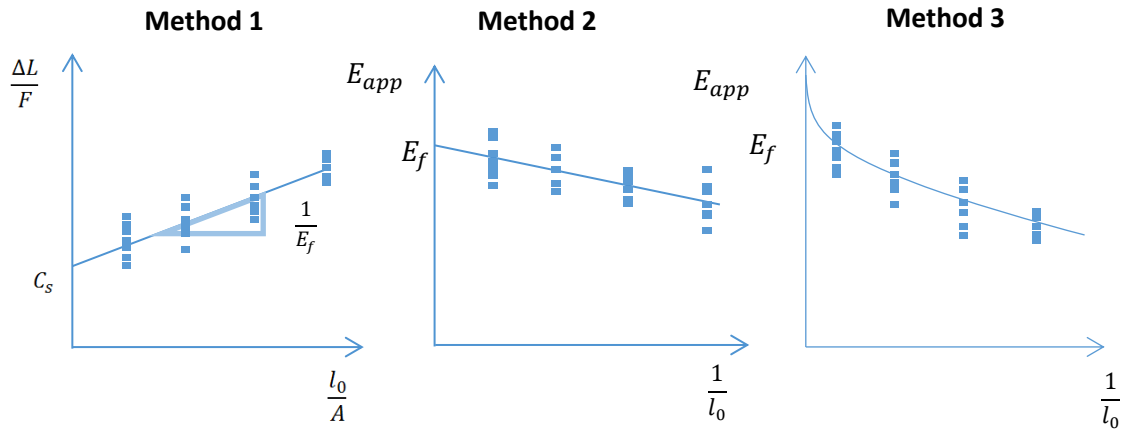


Figure 2: Schematic representation of the indirect methods to determine fibre modulus.

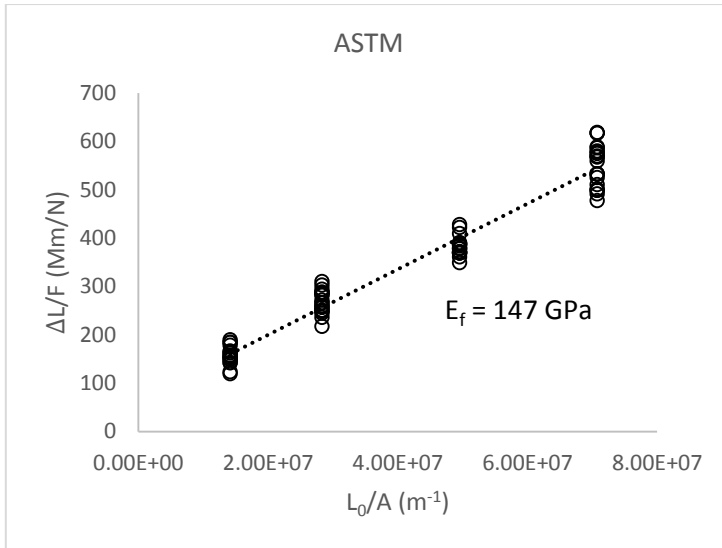


Figure 3: Linear regression of the measured data via method 1 for the determination of the fibre modulus.

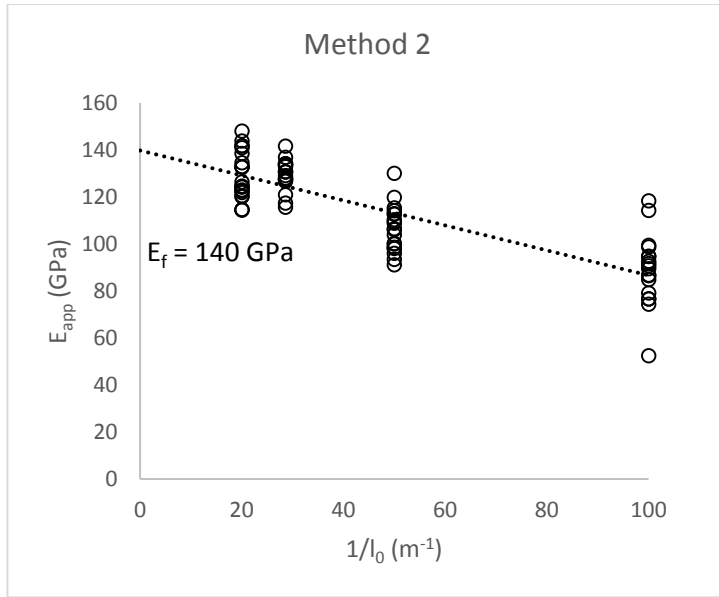


Figure 4: Linear regression and extrapolation of the measured data to infinite gauge length ($\frac{1}{l_0}=0$) via method 2 for the determination of the fibre modulus.

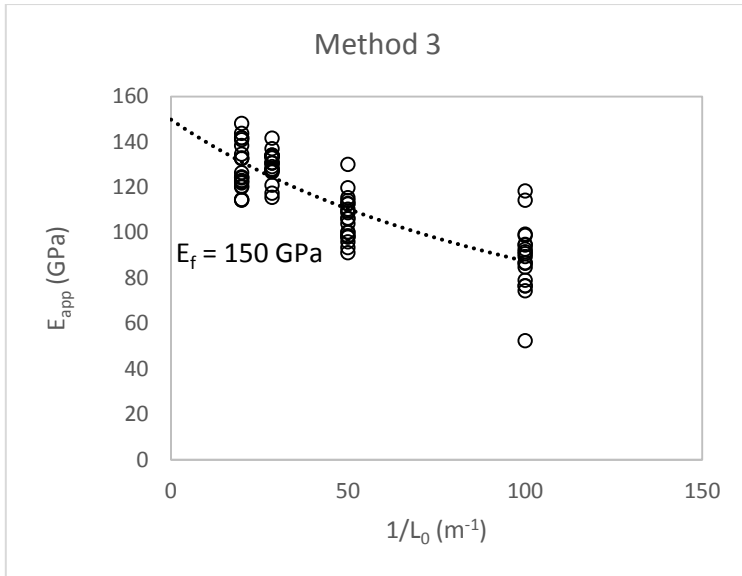


Figure 5: Rational extrapolation of the measured data via the method 3 for the determination of the fibre modulus.

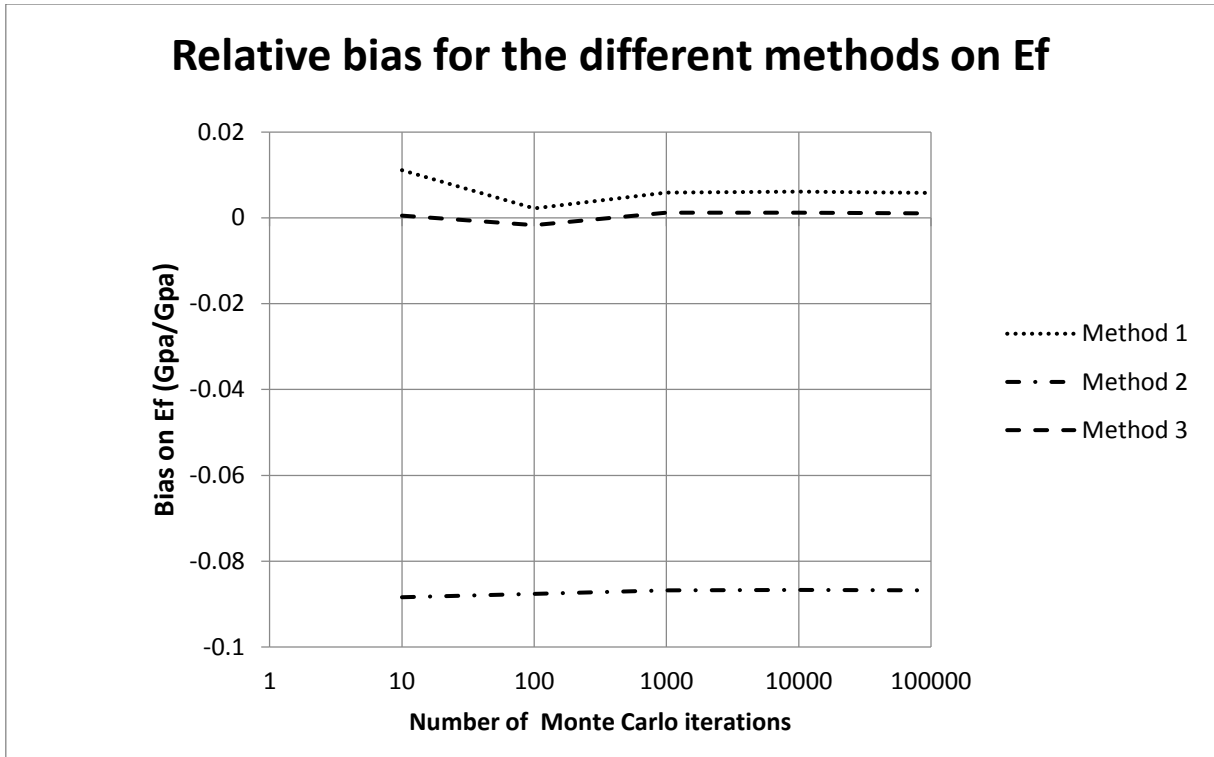


Figure 6: The evolution of the bias on E_f in relation to the number of iterations in the Monte Carlo simulation for the different methods.

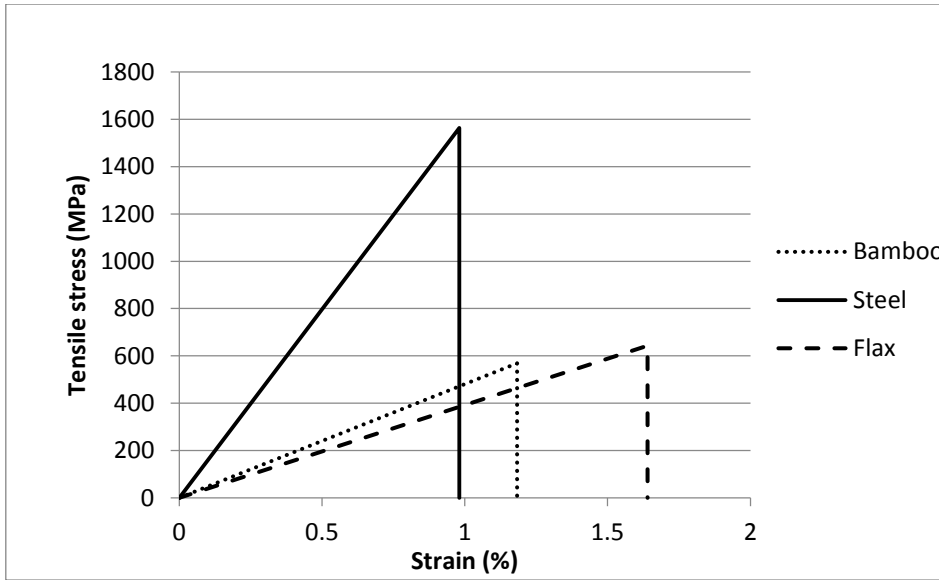


Figure 7: Typical stress-strain curves for the three fibres considered: bamboo, steel, flax.

Table 1: Mechanical properties of flax and bamboo technical fibres.

| Young's modulus (GPa) | Strength (MPa) | Failure strain (%) | Comment | Ref. |
|-----------------------|---------------------------------------|---------------------------------------|--|------|
| Flax | | | | |
| 31 ± 12 | 305 ± 120 | 1.3 ± 0.4 | Gauge 75mm, 1mm/min, green flax | [4] |
| 32 ± 12 | 310 ± 120 | 1.1 ± 0.4 | Gauge 75mm, 1mm/min, retted flax | [4] |
| Bamboo | | | | |
| 33.37 | 639 ± 175 - 813 ± 94 ^a | 2.0 ± 0.6 - 2.9 ± 0.7 ^a | Gauge 5,15,25,35, 1mm/min, steam explosion | [9] |
| 43 | 775 ± 103 - 860 ± 119 ^a | 1.7 ± 0.2 - 1.9 ± 0.3 ^a | Gauge 5,10,25,40, 1mm/min, mechanical extraction | [16] |
| 35.91 | 503 | 1.4 | Mechanical extraction | [20] |
| 19.67 | 341 | 1.73 | Chemical extraction | [20] |
| 35.9 ± 13.1 | 441 ± 220 | 1.3 | 1mm/min, steam explosion | [21] |

^aweakest test length – strongest test length

Table 2: Results of fibre tensile tests on steel fibres using the different methods to determine the fibre Young's modulus.

| | E (GPa) | 95% confidence interval | |
|----------|---------|-------------------------|-----|
| Method 1 | 147 ± 4 | 141 | 155 |
| Method 2 | 140 ± 2 | 135 | 144 |
| Method 3 | 150 ± 4 | 142 | 157 |

Table 3: Input parameters for the Monte Carlo simulation.

| | | | | |
|------------------------------------|-----------|---------|---------|---------|
| E_f (GPa) | 193 | | | |
| A (m ²) | 7 E-10 | | | |
| C_s (m/N) | 64.5 E-06 | | | |
| Number of samples per gauge length | 17 | | | |
| l_0 (mm) | 10 | 20 | 35 | 50 |
| Average $\frac{F}{\Delta L}$ (N/m) | 6288.34 | 3755.95 | 2618.14 | 1823.81 |
| Stdev $\frac{F}{\Delta L}$ (N/m) | 1054.08 | 342.51 | 127.07 | 144.54 |

Table 4: RMSE of the different methods after 10 000 Monte Carlo iterations.

| N=10000 | Method 1 | Method 2 | Method 3 |
|---------|----------|----------|----------|
| RMSE | 0.0344 | 0.0880 | 0.0303 |
| RB | 0.0061 | -0.0867 | 0.0012 |

Table 5: Tensile modulus, strength and strain to failure of the fibres using direct strain measurement

| Fibre | E modulus (GPa) | Tensile strength (MPa) | Strain to failure (%) |
|--------|-----------------|------------------------|-----------------------|
| Steel | 187 ± 12 | 1564 ± 141 | 0.98 ± 0.09 |
| Bamboo | 52 ± 7 | 658 ± 135 | 1.33 ± 0.20 |
| Flax | 40 ± 11 | 643 ± 247 | 1.64 ± 0.32 |

Table 6: Risks and mitigation during fibre testing of selected physical parameters

| Physical parameter | Risk | Mitigation |
|-----------------------|--|---|
| Out-of-plane movement | When the fibre moves towards or away from the camera, the registered pixel movement is under- or overestimated, respectively [22]. | <ol style="list-style-type: none"> 1. Limit the out-of-plane movement of the fibre by choosing a ball joint connection of 1 grip. 2. Correct using a single camera by rotating or using a region of interest as described in [22, 23] 3. Eliminate by using a stereo 2D set-up with 2 cameras to measure the out-of-plane movement [24]. |
| Out-of-plane movement | The optical flags can be considered as 2D flags. Rotation of the flags may occur during the test. | Prevent twisting the fibre during sample preparation. |
| Lighting conditions | The lighting conditions change during the test. | <ol style="list-style-type: none"> 1. Use a light source with a constant and diffuse illuminance [25, 26]. Increasing the illuminance |

| | | |
|---------------|--|---|
| | | <p>leads to a lower exposure time which decreases noise.</p> <p>2. If lightning changes cannot be prevented, use a robust correlation algorithm [27].</p> |
| Optical flags | <p>The attachment of the optical flags can give rise to stress concentrations during fibre loading, especially when the material used to create the optical flag has a high stiffness compared to the fibre. The measured stiffness would be largely unaltered but the fibre may fail prematurely, hence the apparent fibre strength and strain to failure is lower.</p> | <p>To prevent stress concentrations, the flag material stiffness should be as low as possible.</p> <p>When attaching flags to fibres that have high strains to failure the flags may detach or crack during the test.</p> <p>To avoid this, use a flag material with a high failure strain.</p> |
| Gauge length | <p>The length in pixels of the fibre's gauge length is insufficient, lowering the accuracy of the correlation.</p> | <p>Although DIC measurements have sub-pixel resolution it is advisable that the gauge length in the images is longer than 50 pixels.</p> |