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Stiffening effect of fatigue and creep loading in unidirectional flax fibre/epoxy composites

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Abstract. A study related to the fatigue behaviour of natural fibre composites was conducted to expand their industrial applications. Unidirectional flax/epoxy composites were fabricated, and their fatigue and creep behaviour was investigated by applying a range of different testing conditions. Stiffness was found to increase during fatigue loading, and this was accompanied by accumulation of residual strain. By arranging the results in a stiffness-strain diagram, a linear trend was established with a positive slope representing the stiffening effect. A similar linear trend was obtained for creep loading, however, with a larger stiffening effect. For fatigue loading, the stiffening effect was found to be changed by the applied cycle frequency. The combined findings in the study suggest that stiffness change in the composites is reflecting a balance between stiffening due to residual strain, and softening due to fatigue damaging.

1. Introduction

Replacing synthetic fibres, such as glass fibres and carbon fibres, with plant-based fibres, widely denoted as natural fibres, is an effective way to realize materials carbon neutrality because plants absorb CO₂ during their growth. In addition, natural fibres are known to have mechanical properties comparable to glass fibres. Therefore, a large number of research studies related to natural fibres and their composites have been performed to evaluate materials characteristics, and to maximize materials performance.

Recent industrial use of natural fibre composites is mostly limited to non-structural components, such as body panels of automobiles made from non-woven random mats of natural fibres. One of the reasons why industrial applications have been limited so far is the lack of knowledge related to the long-term reliability of natural fibre composites. Especially fatigue behaviour should be clarified to expand their application to load-bearing structural components. A common observation in studies of fatigue behaviour of natural fibre composites is stiffness increase during fatigue loading [1,2], while conventional composites based on glass and carbon fibres are known to show stiffness decrease, which typically is assumed to be due to damage progression [3-5]. Liang et al. [2] presented that a natural fibre composite based on a cross-ply flax fabric showed continuous increase of stiffness during almost all of the fatigue life, while a glass fibre composites with an identical fibre configuration exhibited significant decrease of stiffness. Baley [6] reported that cyclic tensile loading of single natural fibres also showed



stiffness increase. Nevertheless, the underlying mechanisms for this stiffness increasing behaviour remain unclear [1].

In the present study, it is hypothesised that the stiffness change observed in natural fibre composites during fatigue loading is the result of two counteracting mechanisms taking place: stiffening (i.e. stiffness increase) and softening (i.e. stiffness decrease). This is investigated by performing tension-tension fatigue tests of unidirectional natural fibre composites, and by focusing on the stiffness change and the concurrent change of residual strain. In addition, creep tests were performed to examine whether constant loading, i.e. a fatigue test without cyclic loading, induces a stiffening effect in the same way as fatigue loading does. This approach was used to separate the effects of the cyclic and constant loading components in fatigue loading.

2. Materials and methods

2.1. Fabrication of composite laminates and test specimens

Unidirectional natural fibre composites were fabricated from a flax yarn (Smeraldo, Nm 1/9.7, Linificio e Canapificio Nazionale SpA, Italy) and an epoxy resin (Araldite LY1564SP, epoxy; Araldite 3486, hardener; Huntsman, USA). The density of the flax fibres and the cured resin was measured to be 1.59 and 1.15 g/cm³, respectively. The same type of flax yarn has been used in a previous study of natural fibre composites, but with focus on static tensile properties [7].

For fabrication of unidirectional flax/epoxy composite laminates, 14 layers of flax yarn were wound on to an aluminium plate (470 mm x 400 mm) using a custom-made winding machine. The misalignment of yarns was calculated to be negligible ($\approx 0.1^\circ$). The plate with the wound yarn was packed in a vacuum bag. After a vacuum infusion process with the epoxy resin, the entire aluminium plate with the impregnated yarn was placed in a thermo-chamber for the curing process, 5 hours at 50°C. After demoulding, the composite laminates were post-cured at 80°C for 4.5 hours. The density, volume fraction of fibres, and porosity of the four fabricated laminates were measured to be 1.28 ± 0.02 g/cm³, $31.4 \pm 0.2\%$, and $0.3 \pm 0.3\%$ (mean \pm standard deviation), respectively. The laminates were cut into (i) butterfly-shaped test specimens (length 410 mm, R = 900 mm, gauge section 60 x 25 mm), a type of specimen geometry specially designed for tensile fatigue testing of composites [8], and (ii) rectangular test specimens (length 180 mm, gauge section 100 x 25 mm). Tapered glass fibre composite tabs were mounted on both types of test specimens.

2.2. Static tensile tests

To establish the testing conditions for the fatigue and creep tests, static tensile tests were initially performed with the rectangular specimens using a hydraulic testing machine (Instron, UK). The displacement rate was 2 mm/min. Strain was measured by two extensometers (50 mm gauge length) attached to each side of the specimens.

2.3. Fatigue tests and creep tests

Fatigue tests were performed using an identical setup as used for the static tensile test. The applied fatigue testing mode was tension-tension, as indicated in Figure 1 (left), and with a constant stress ratio of $R = 0.1$. Different levels of stress amplitudes and frequencies were examined, as presented in Table 1. Although almost all fatigue tests were performed on the butterfly-shaped specimens, a few tests that were focusing only on the stiffening effect were done on the rectangular specimens. During the fatigue tests, the stress-strain hysteresis loops were recorded intermittently, and stiffness was determined as the slope of a linear regression line fitted to the loops (see Figure 4).

Creep tests were performed using an electronic testing machine (Instron, UK). Figure 1 (right) shows the general time course of the creep tests. For each test, two values of stiffness were determined from the slopes of linear regression lines fitted to the two loading phases (1st and 2nd ramp). Two stress levels ($\sigma_c = 100$ and 150 MPa) and several holding times ($t_h = 0$ to 1800 sec) were examined, as presented in

Table 2. The magnitude of the two stress levels were selected to be within the range of mean stress levels applied in the fatigue tests.

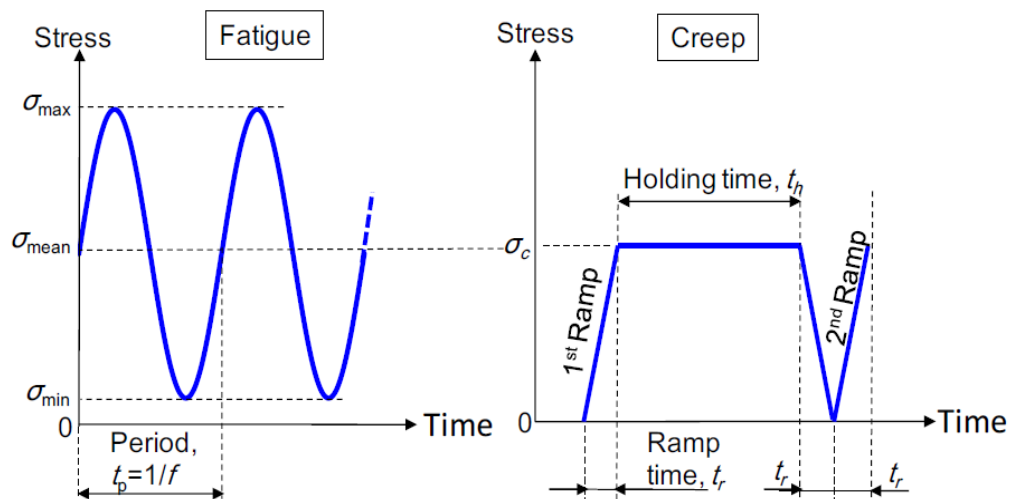


Figure 1. Time course for fatigue tests (left) and creep tests (right).

Table 1. Applied conditions for fatigue tests.

Specimen shape	Max. stress, σ_{\max} [MPa]	Mean stress, σ_{mean} [MPa]	Stress ratio, $R = \sigma_{\min}/\sigma_{\max}$	Frequency [Hz]
Butterfly	110	61	0.1	5
Butterfly	129	71	0.1	2
Butterfly	159	87	0.1	2
Butterfly	180	99	0.1	2
Butterfly	203	112	0.1	2
Butterfly	220	121	0.1	2
Butterfly	240	132	0.1	2
Rectangular	220	121	0.1	2
Rectangular	220	121	0.1	1
Rectangular	220	121	0.1	0.5
Rectangular	220	121	0.1	0.25

Table 2. Applied conditions for creep tests.

Stress level, σ_c [MPa]	Holding time, t_h [sec]	Ramp time, t_r [sec]
100	0	1
100	1	1
100	5	1
100	30	1
100	300	1
100	1800	1
100	0	0.3
100	0	0.5
150	0	1
150	1	1
150	5	1
150	30	1
150	300	1
150	1800	1
150	0	0.3
150	0	0.5

3. Results and discussion

3.1. Tensile properties

Based on the measured static tensile stress-strain curves of the unidirectional flax/epoxy composites, the stiffness and the ultimate tensile stress (UTS) were determined to be 18.5 ± 0.5 GPa and 282 ± 14 MPa (mean \pm standard deviation, $n = 5$), respectively. These properties are comparable to previously reported properties of unidirectional natural fibre composites with similar fibre contents of about 30 % [7, 9]. The stress-strain curves showed transition regions (or yielding regions) in the strain range 0.2 - 0.4%. This specific characteristic has been reported in several studies both for flax fibres and for flax fibre composites [6, 10], though the underlying mechanism is still unclear. In the present study, the applied strain levels in the fatigue and creep tests are all above 0.5%, and therefore, they exceed the transition region of the composites.

3.2. Fatigue behaviour

Figure 2 shows the typical fatigue failure mode of the composites. At first, a localized damage region appeared at the specimen edge within the gauge section. After another several hundreds of cycles, a splitting crack propagated from the initial damage region to the start of the tab, presumably due to the increase of shear stresses along the fibres. Finally, the decrease of the load carrying area induced bulk failure at the start of the tab. It can be noted that due to the stochastic nature of the fatigue failure mode, it is inevitable that the recorded maximum number of cycles at failure (N) will show some degree of scatter, even for identical materials and test conditions.

Based on the results of the fatigue tests, the obtained S-N diagram is shown in Figure 3, together with the results of the static tensile tests. Generally, S-N data points of composites tested with a constant stress ratio (i.e. R-value) are known to be well fitted using an empirical power law relation, $S = C \cdot N^{-1/m}$, where S is maximum stress, and, C and m are fitting coefficients. As shown in Figure 3, the coefficient m , which determines the slope of the S-N curve, is 9.7 for the unidirectional flax/epoxy composites. This is similar to the m -value of about 10 seen for unidirectional glass/epoxy composites [11].

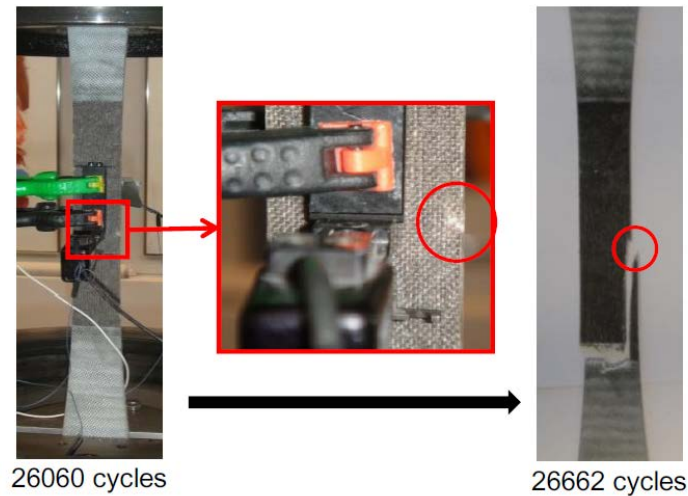


Figure 2. Typical fatigue failure mode observed for the unidirectional flax/epoxy composites. The red circles indicate the position of the visible initial damage region.

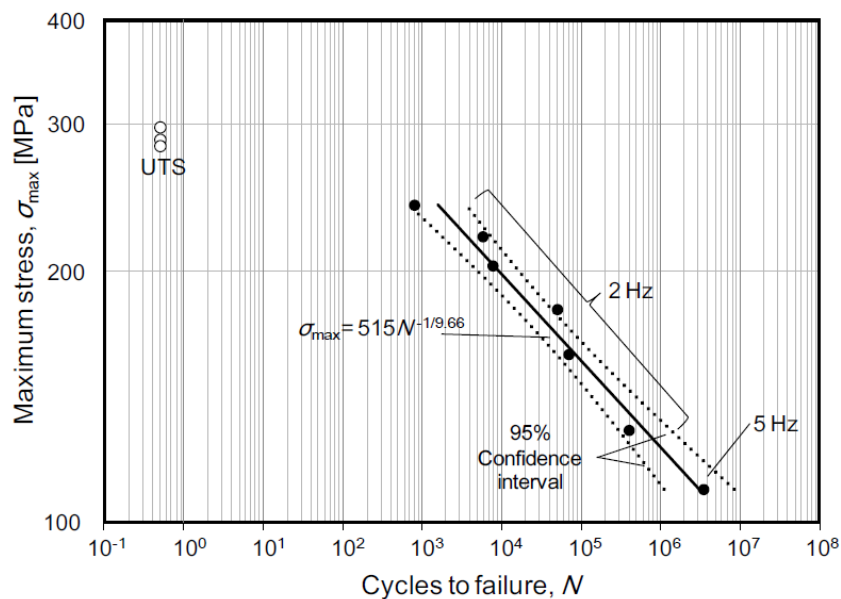


Figure 3. The logS - logN fatigue diagram of the unidirectional flax/epoxy composites, together with the results of the static tensile tests.

A special fatigue behaviour observed for unidirectional flax/epoxy composites, not seen for conventional types of composites, is the change in stiffness during fatigue testing. Figure 4 shows examples of the recorded stress-strain hysteresis loops during two fatigue tests with mean stress levels of 61 and 121 MPa (corresponding to maximum stress levels of 110 and 220 MPa). For both stress levels, the slope of the loops at halfway of fatigue life is increased compared with the slope at the start of the fatigue test. For the high mean stress level of 121 MPa, this trend is continued towards the end of fatigue life, whereas for the low mean stress level of 61 MPa, the slope is decreased at the end of fatigue life. For both stress levels, the position of the loops is consistently shifted to the right during the fatigue testing. The starting strain value for each loop is denoted *residual strain*. The two examples in Figure 4 show that the composites become stiffer during a major part of their fatigue life, while at the same time, the residual strain is increased.

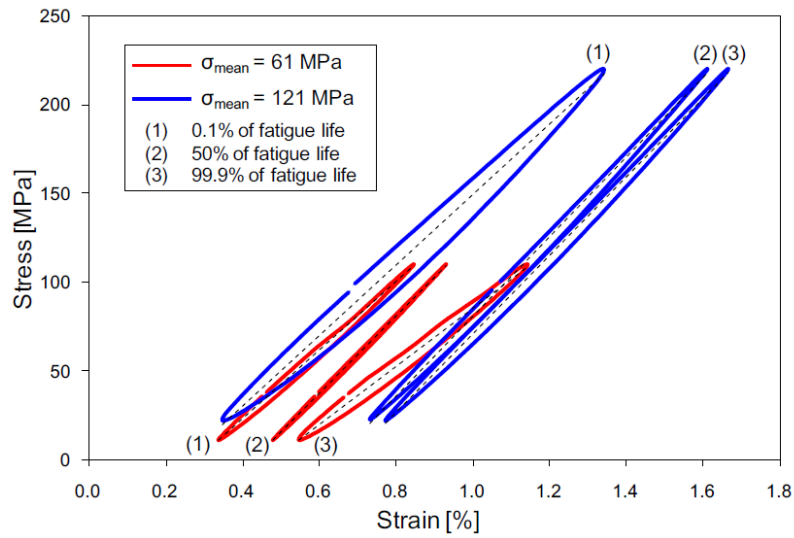


Figure 4. Examples of hysteresis loops recorded during two fatigue tests with mean stress levels of 61 and 121 MPa. Dotted lines show linear regression lines of each loop to determine stiffness.

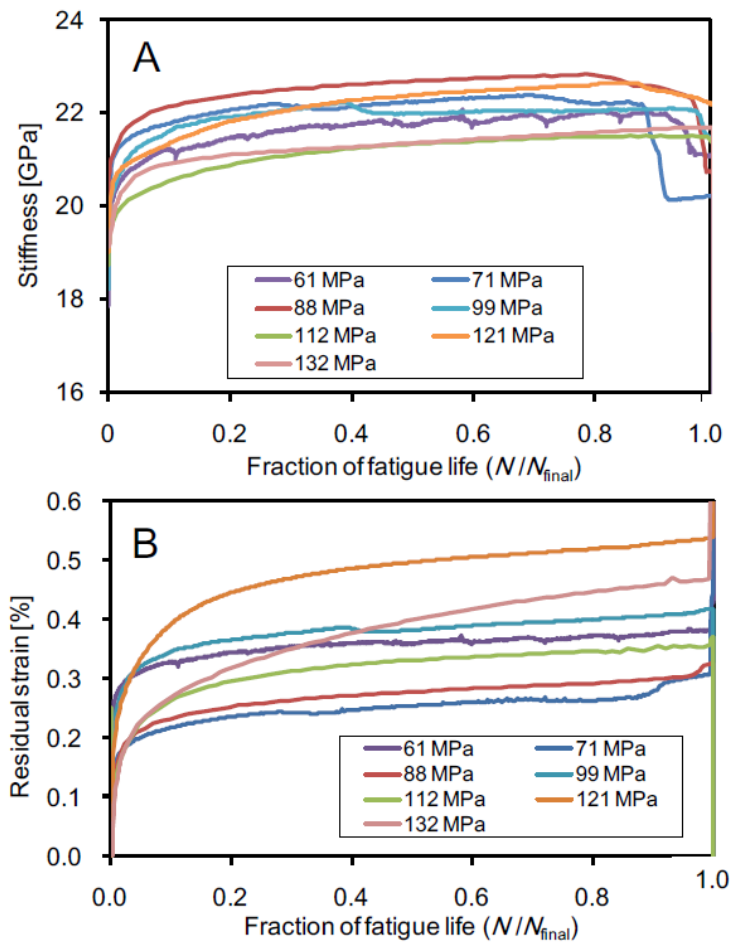


Figure 5. Stiffness (A) and residual strain (B) as a function of fraction of fatigue life. Results are shown for fatigue tests with different mean stress levels.

For the performed fatigue tests with the different mean stress levels, Figure 5 shows the determined stiffness and residual strain as a function of the fraction of fatigue life (N/N_{final}). For both stiffness and residual strain, it is difficult to see a common trend with respect to the mean stress level. However, as shown in Figure 6, if the relative change in stiffness ($(E-E_0)/E_0 = \Delta E/E_0$, where E and E_0 are the current and the initial stiffness, respectively) is plotted as a function of the residual strain, the curves for the different mean stress levels share a common linear trend. Such a diagram is hereafter referred to as a *stiffness-strain diagram*. The established correlation shown in Figure 6 suggests that stiffening of the composites is induced during accumulation of residual strain. The stiffening effect is represented by the slope of the linear trend line.

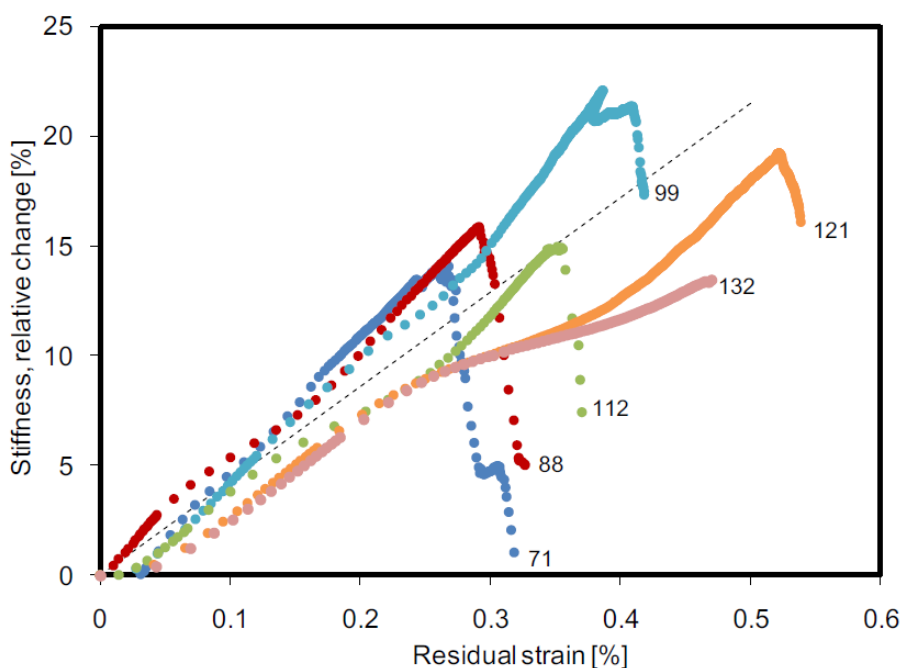


Figure 6. Stiffness-strain diagram for fatigue. Relationship between relative change in stiffness and residual strain. Numbers at the curves indicate the applied mean stress levels. Dotted black line is a linear regression line for all stress levels.

3.3. Creep behaviour

In the results of the fatigue tests presented above, a stiffening effect was clearly observed. If this stiffening effect is induced by the residual strain, a similar effect should be expectedly be observed in creep tests, which correspond to fatigue tests with no stress amplitude, i.e. the applied creep stress can be assumed equivalent to the mean fatigue stress (see Figure 1). In Figure 7, an example of results of a creep test with an applied stress of 150 MPa is shown. As can be observed in Figure 7A, the onset strain value is increased above zero for the 2nd ramp, which shows that residual strain is generated. In addition, as can be observed in Figure 7B, the slope of the 2nd ramp is steeper than the slope of the 1st ramp. This is indicating a stiffening of the composites as an effect of the constant loading. Analogous to the fatigue test, the relative change in stiffness ($(E_2-E_1)/E_1 = \Delta E/E_1$, where E_1 and E_2 are stiffnesses obtained from the 1st and the 2nd ramps, respectively) can be calculated.

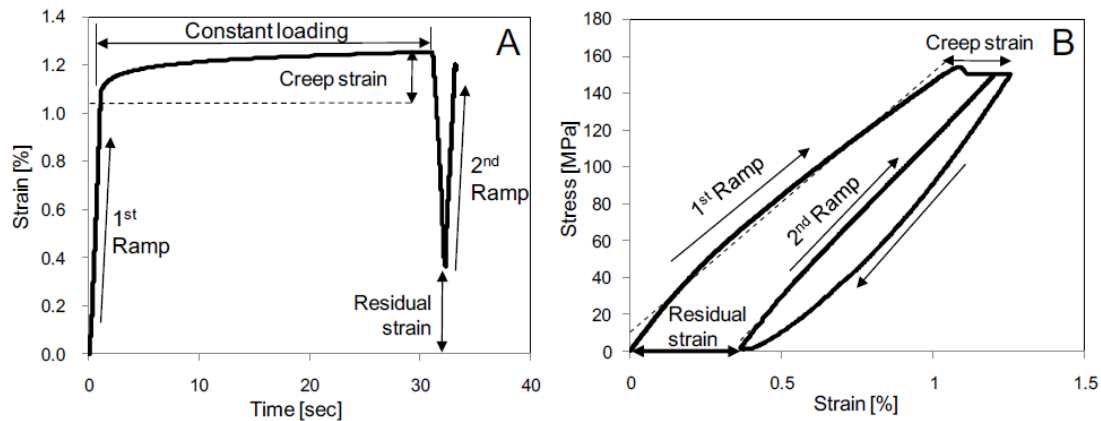


Figure 7. An example of results of a creep test with a stress level of 150 MPa, and a holding time of 30 sec. A: Strain-time, B: Stress-strain.

For the applied two stress levels in the creep tests (100 and 150 MPa), Figure 8 shows the determined relative change in stiffness and the residual strain as a function of the holding time. It is clearly indicated that the effect of holding time on stiffness and residual strain is similar. Both properties are increased rapidly in the range of short holding times, and the rate of increase is reduced in the range of long holding times. In addition, as expected, the increase of the stress level leads to an increase of both the stiffness and the residual strain.

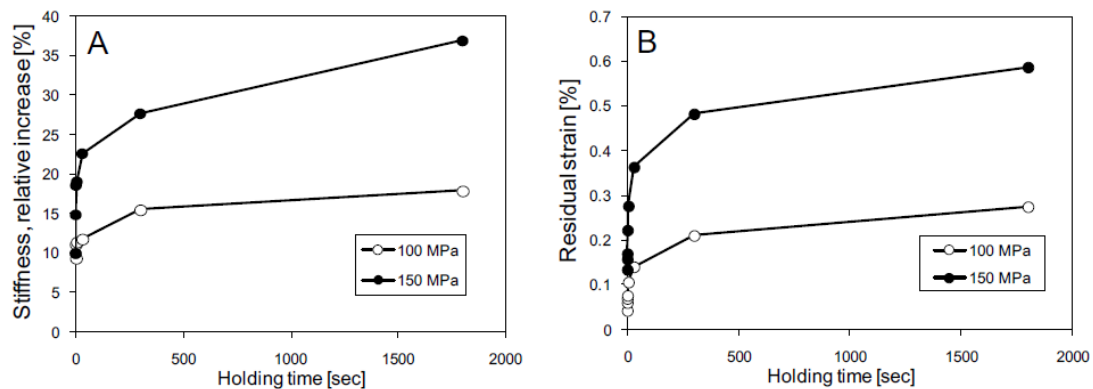


Figure 8. Relative change in stiffness (A) and residual strain (B) as a function of the holding time in the creep tests.

Based on the results from the creep tests, Figure 9 shows the stiffness-strain diagram, i.e. the relative change in stiffness as a function of residual strain. The results from the two stress levels share a common linear trend, as was also observed for the fatigue tests. Thus, it is clearly demonstrated that the stiffening effect does not require the repeated loading-unloading cycles of the fatigue tests.

3.4. Fatigue and creep behaviour

Figure 10 is a co-plot of the results of the fatigue tests and the creep tests in the same stiffness-strain diagram. As can be observed, the slope of the linear trend line is clearly lower for the fatigue tests, than for the creep tests. This indicates the existence of a concurrent mechanism in the fatigue test that is counteracting the stiffening effect. This mechanism is likely to be the damage induced by the repeated loading in the fatigue tests, which is the typically stated reason for the softening seen for conventional composites, such as glass fibre composites [3, 4]. In the case of the flax/epoxy composites in the present study, it is indicated that stiffening proceeds simultaneously with softening, and this explains why the

fatigue tests result in a lower slope of the linear trend line compared to the creep tests. Thus, the slope in the stiffness-strain diagram is reflecting the balance between stiffening related to residual strain, and softening related to fatigue damaging.

Next, the balance between stiffening and softening will be studied by changing the applied loading frequency in the fatigue test.

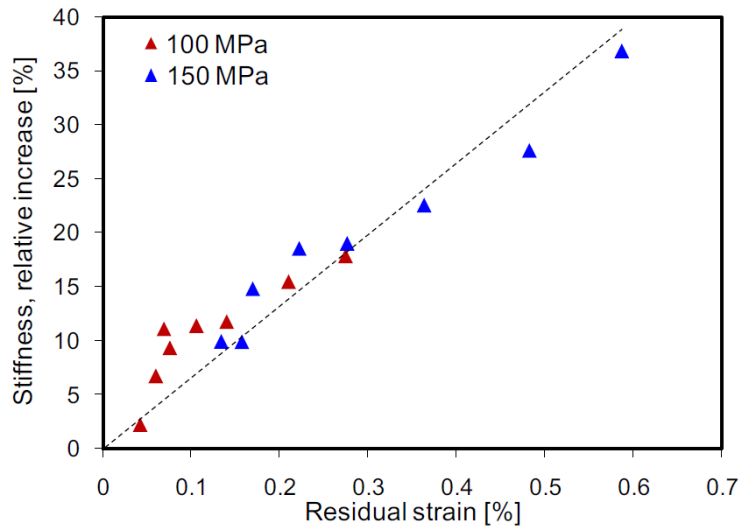


Figure 9. Stiffness-strain diagram for creep. Relationship between relative change in stiffness and residual strain. Dotted black line is a linear regression line for all data points.

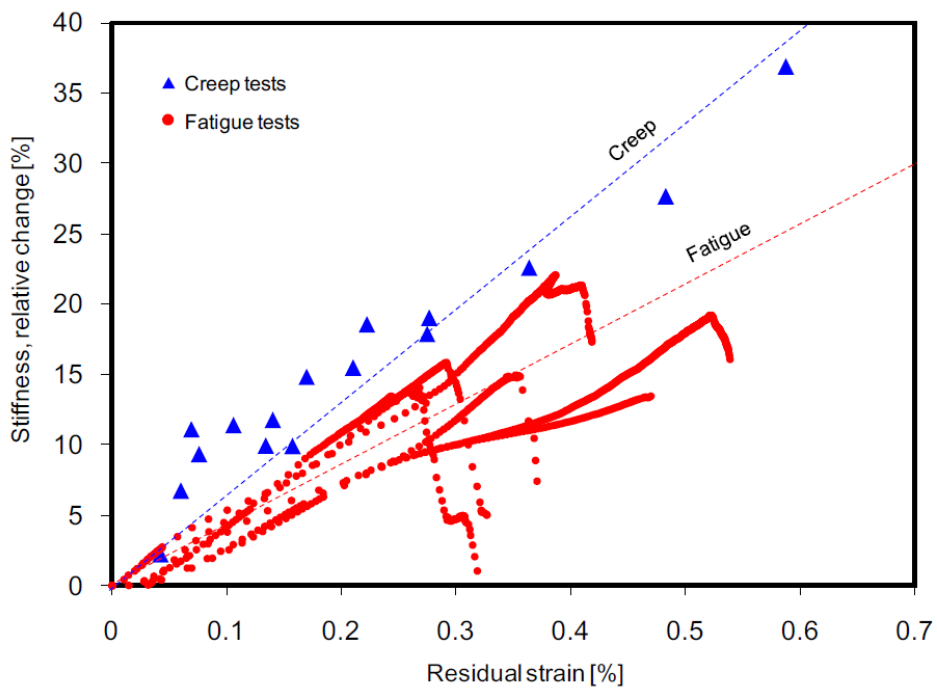


Figure 10. Stiffness-strain diagram for fatigue and creep.

3.5. Effect of loading frequency on fatigue behaviour

Additional fatigue tests were performed with a mean stress level of 121 MPa, and with loading frequencies of 0.25, 0.5, 1 and 2 Hz (see Table 1). In these tests, the constant stress component is the same, which means that the induced residual strain per unit of time can be assumed to be the same. The number of cycles per unit of time is however different between the tests, and this is assumed to lead to a difference in the amount of induced damage. Therefore, the balance between stiffening and softening is expected to be affected by the loading frequency, e.g. a lower frequency is expected to lead to a larger resulting stiffening effect due to less damaging. Figure 11 presents the results of these additional fatigue tests. As shown in Figure 11A, the relative change in stiffness proceeds faster for the lower frequencies, when the results are plotted as function of the number of fatigue cycles. Furthermore, as can be seen in the stiffness-strain diagram in Figure 11B, the slope of the curves is clearly steeper for the lower frequencies. Thus, as expected, the stiffening effect is getting larger when the cycle frequency is reduced. For a given residual strain induced by the constant stress component, the induced damage is lower for the fatigue tests with the lower frequency.

In the study by Shah [12] of comparable unidirectional natural fibre composites, fatigue testing was performed using a loading frequency of 10 Hz, and a negligible change of the stiffness was found. Qualitatively speaking, the results by Shah [12] are supporting the findings in the present study, i.e. an increase in loading frequency is leading to a decrease in the stiffening effect (Fig 11B).

Figure 11A shows that the specimens tested by the lower frequencies exhibit shorter fatigue life, although the fatigue damage per cycle should be same, i.e. it should be independent of the loading frequency. Interestingly, it can be observed that the final level of the relative change in stiffness is almost the same (about 16 %) for all frequencies. It is therefore indicated that the failure criterion for unidirectional natural fibre composites is not only governed by the number of loading cycles, i.e. by the magnitude of accumulated damage, but also the magnitude of the stiffening must be taken into account.

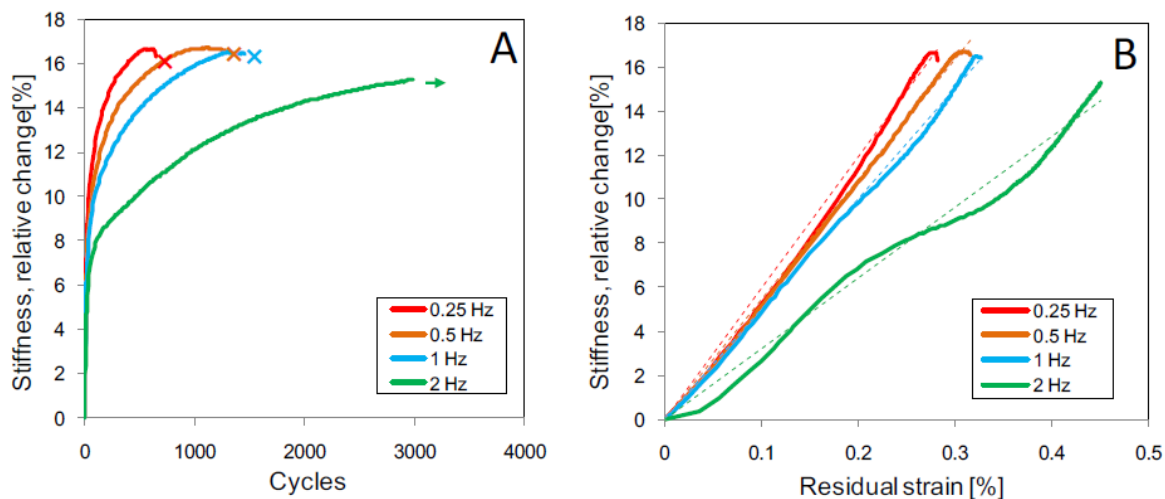


Figure 11. Results of fatigue tests performed with a mean stress level of 121 MPa and with various loading frequencies. Broken lines in B are linear regression lines for each curve.

3.6. Underlying mechanisms for stiffening and softening

In the present study, mechanical tests have been performed to quantify and analyse the mechanical response of unidirectional natural fibre composites to fatigue and creep loading. This has been done, however, without knowing the exact nature of the underlying mechanisms. In preliminary investigations by the authors, X-ray microtomography were used to observe the microstructure of the unidirectional flax fibre/epoxy specimens before and after testing, however, no clear differences were found (data not shown). In the case of natural fibres, Kohler and Spatz [13] reported a lowering of the cellulose

microfibril angle during the initial phase of loading until the yielding region, where after the angle remained almost constant. Upon unloading, a residual deformation of the fibres remained. In the case of natural fibre yarns, as used in the present study, it must be considered that the twisting angle of the fibres in the yarn becomes lowered during fatigue and creep tests. These changes on the two structural levels of fibres and yarns support a stiffening effect in the composites.

4. Conclusions

In the present study, fatigue and creep tests under variable conditions were performed on unidirectional flax/epoxy composites. The results were analysed, and the following findings were obtained.

- a) Increase of stiffness was correlated to increase of residual strain in both fatigue and creep tests. By arranging the results in a stiffness-strain diagram, a linear trend was established with a positive slope representing the stiffening effect.
- b) Creep tests induced a larger stiffening effect than fatigue tests.
- c) Fatigue tests performed with lower frequencies induced a larger stiffening effect.

Based on these findings, it is suggested that stiffening proceeds simultaneously with softening in the composites. The resulting stiffness change is reflecting the balance between stiffening due to residual strain, and softening due to fatigue damaging. Further work is needed to elucidate the underlying mechanisms.

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