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ENHANCING FATIGUE PERFORMANCE OF STRUCTURAL BIOCOMPOSITES BY PRE-STRAINING AND PRE-CREEPING METHODS

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Abstract

Synthetic fibre-reinforced polymer composites (FRPs) have long been favored in structural engineering for their exceptional mechanical properties. However, their environmental impact due to energy intensive manufacturing, and disposal has prompted exploration into sustainable biobased alternatives such as flax FRP composites (FFRPs). While using flax fibres as composite reinforcement has lightness and damping benefits from a structural point of view, it also introduces design challenges as the complex microstructure of flax fibres and flax FRPs induces a more viscoelastic fatigue response. Therefore, pre-straining and pre-creeping are proposed in this study as simple methods to improve fatigue performance by taking advantage of alignment mechanisms intrinsic to flax fibre and yarn microstructure. An experimental campaign was conducted on [0/90/0]_s flax FRP laminates (hence, predominantly UD) to compare the tension-tension fatigue performance of reference specimens to pre-strained and pre-creeped specimens. It was observed that pre-straining and especially pre-creeping are effective at improving FFRPs fatigue performance with significant increase in fatigue life, increase in dynamic modulus, and decrease in accumulation of deformation (ratcheting).

1. Introduction

In the pursuit of peak performance in structural engineering, synthetic fibre-reinforced polymer composites (FRPs) have emerged as promising materials in multiple contexts, offering simultaneously high specific stiffness, and high specific strength along with design freedom. Currently, glass and carbon fibres dominate reinforcement of FRP composites for structural applications. While offering attractive mechanical properties, using synthetic fibres in large quantities is harmful to the environment as the production of those fibres consumes a large amount of energy and deplete non-renewable resources. Additionally, disposing of or incinerating synthetic FRP materials at the end of their lifespan causes environmental harm, and recycling them poses challenges. Consequently, numerous researchers and industries are exploring biobased FRPs as an alternative to synthetic FRPs where strong natural fibres such as flax are used as reinforcement instead of synthetic fibres, particularly glass fibres.

Elementary flax fibres have a complex microstructure mainly composed of cellulosic microfibrils [1] and those microfibrils are not naturally aligned with the axis of the flax fibres [2]. Coincidentally, cyclic

mechanical testing on single elementary fibres reveals a non-linear and viscoelastic behaviour with a peculiar increase of modulus [3], [4] suggesting that an alignment of the microfibrils occurs during loading. Direct observations of microfibrils angles during mechanical loading revealed that an alignment indeed occurs, however, the change of microfibrils angle is small and therefore unlikely to have a significant impact on the composite properties. Adding to the complexity of understanding the mechanics of flax FRP composites (FFRPs), flax fibres preforms utilized for manufacturing are often made of yarns in which a degree of twist is applied on the elementary flax fibres. This twist angle of the elementary fibre with respect to the yarn axis and the loading axis of the composite may again lead to alignment mechanisms upon loading but at a different scale due to unravelling of the twist [5].

Regardless of the exact mechanisms behind the viscoelasticity of flax fibres and its composites, this material must meet safety and serviceability requirements to be utilized in structural applications. In particular for fatigue applications, the fatigue life, stiffness (modulus) degradation, and accumulation of permanent deformation (ratcheting) must be within limits for structural design. Currently available results of FFRPs fatigue tests show that in predominantly UD laminates ratcheting is significantly larger than in glass FRP composites and that the modulus increases during fatigue while it decreases in glass FRPs [6].

In this paper we present two novel and yet simple methods aiming at improving the fatigue performance of FFRP composites by taking advantage of their intrinsic viscoelasticity and related alignment mechanisms within their intricate multi-scale microstructure. To demonstrate the effects of those two methods, namely pre-straining and pre-creeping, a fatigue experimental campaign was performed on $[0/90/0]_s$ FFRP laminates. Pre-strained and pre-creeped specimens were subjected to the same force-controlled tension-tension fatigue as reference specimens to measure the effects of the proposed methods. Results show that both methods are beneficial for fatigue performance, pre-creeping in particular.

2. MATERIALS AND METHODS

2.1 Composite laminates

In this study, the FFRP laminate employed is a 2.8 mm thick coupon with a narrow central cross-section and a wider cross-section towards the grips, as illustrated in figure 1, to prevent failure at the clamps' edges during fatigue testing which was observed in preliminary testing with rectangular coupons. Strain measurements are obtained using an extensometer in the narrow cross-section. The FFRP laminate comprises six layers of Bcomp quasi-unidirectional flax mat Amplitex, with an aerial weight of 280 g/m², stacked in a $[0/90/0]_s$ lay-up configuration. The choice of this lay-up configuration was driven by the aim to examine the mechanical behavior of flax fibers under loading along their primary axis, necessitating the inclusion of 0° layers. Although a strictly unidirectional lay-up was initially considered, preliminary testing showed susceptibility to fatigue-induced splitting in such specimens. Consequently, 90° layers were introduced to mitigate the occurrence of splitting phenomena.

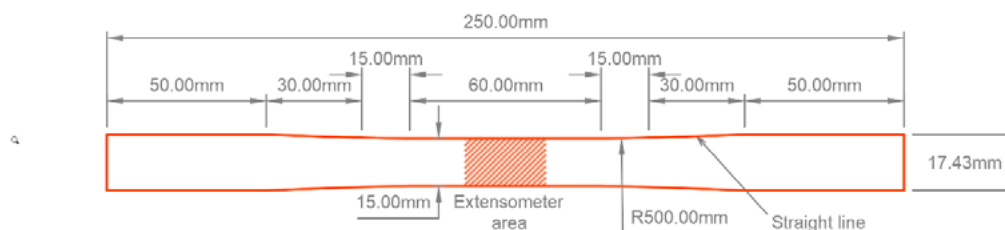


Figure 1. Specimen geometry.

FFRP plates were manufactured using a vacuum-assisted resin infusion process using the Swancor 2511-1 resin epoxy system. The infusion was followed by an 18-hour post-cure in an autoclave set at 70°C and 7 bars of pressure. Subsequently, the plates underwent cutting into test coupons utilizing a waterjet cutter. To ensure the completion of resin curing, a differential scanning calorimetry (DSC) analysis was conducted after the manufacturing process. This involved subjecting the specimens to three heating/cooling cycles between 0°C and 150°C. The midpoint of the stepwise change in the heat flow versus temperature curve in the first and third thermograms were measured to ascertain that the glass transition temperature (T_g) did not deviate by more than 3°C. Optical microscopy was employed to qualitatively assess the microstructure of pristine specimens, ensuring the absence of cracks or manufacturing defects/voids significant enough to impact fatigue damage. The fibre volume fraction was estimated at 42% based on calculations considering the density of the fiber mats, epoxy resin, and composites.

2.2 Pre-straining and pre-creeping protocols

For the experimental campaign, specimens were placed in an MTS 60 kN fatigue machine equipped with hydraulic grips (figure 2). An extensometer was attached to the specimens with rubber bands to measure the strain. As testing was not performed in an environmentally controlled environment, a thermocouple was installed to capture air temperature at proximity of the specimen and another thermocouple was installed on the specimen to measure its surface temperature.

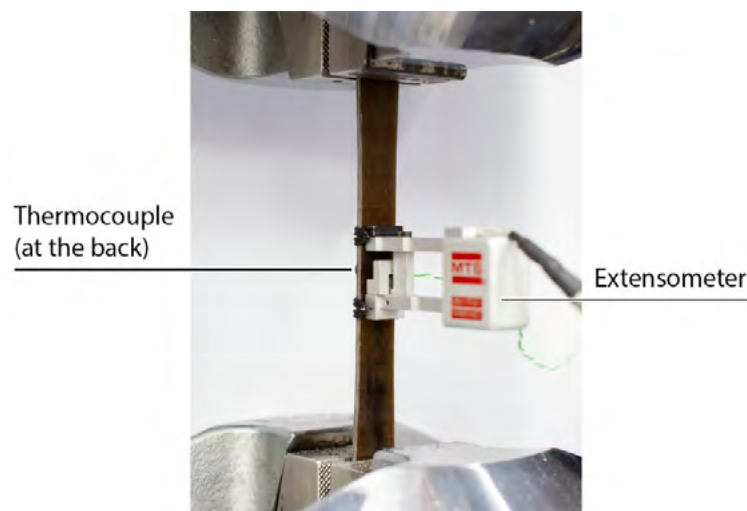


Figure 2. Tensile testing setup.

Loading prior to fatigue for the reference, pre-strained, and pre-creeped specimens are described in figure 3. All the loading and unloading were performed at a rate of 2 mm/min except for the pre-creeping plateau during which a constant force corresponding to the maximum fatigue load was maintained until a strain of 1.6% was reached.

The maximum stress reached during pre-straining corresponds to 80% of the material ultimate tensile strength (UTS). The start of fatigue testing, at the mean fatigue stress, is indicated by a star for each case in figure 3.

2.3 Fatigue testing protocol

Tension-tension fatigue tests were carried out under force control mode, employing a loading ratio (R) of 0.1 and a frequency of 5 Hz. The maximum fatigue load was set at 133 MPa corresponding to 60% of the UTS determined by quasi-static tensile testing at a loading rate of 2 mm/min. In the fatigue testing,

the data pulling was set such that 50 data points were recorded every cycle for the first 500 cycles and then 1 cycle was recorded every 1'000 cycles.

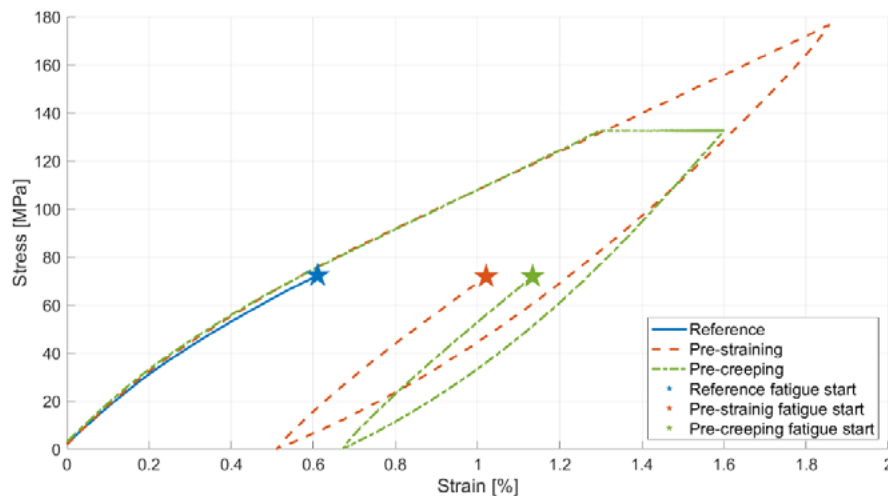


Figure 3. Depiction of pre-straining, pre-creeping and quasi-static ramp protocols before fatigue.

3. Results and discussion

Tests results were obtained with 3 repetitions for each fatigue case studied; reference, pre-straining, and pre-creeping symbolized by different colors. As the machine did not instantly reach the targeted fatigue force, all the analysis take the 10th cycle as reference from which the minimum and maximum force remained constant.

3.1. Effect of pre-straining and pre-creep on the evolution of the dynamic modulus during fatigue

The dynamic modulus is defined here as the slope of the line connecting the top and bottom points of the hysteresis loop in a stress-strain graph. Figure 4 shows that the dynamic modulus as a general tendency to increase during fatigue but pre-straining and especially pre-creeping mitigate this increase in dynamic modulus. Furthermore, the dynamic modulus of pre-creeped specimens is significantly higher than the dynamic modulus of reference specimens and this throughout the entire fatigue life.

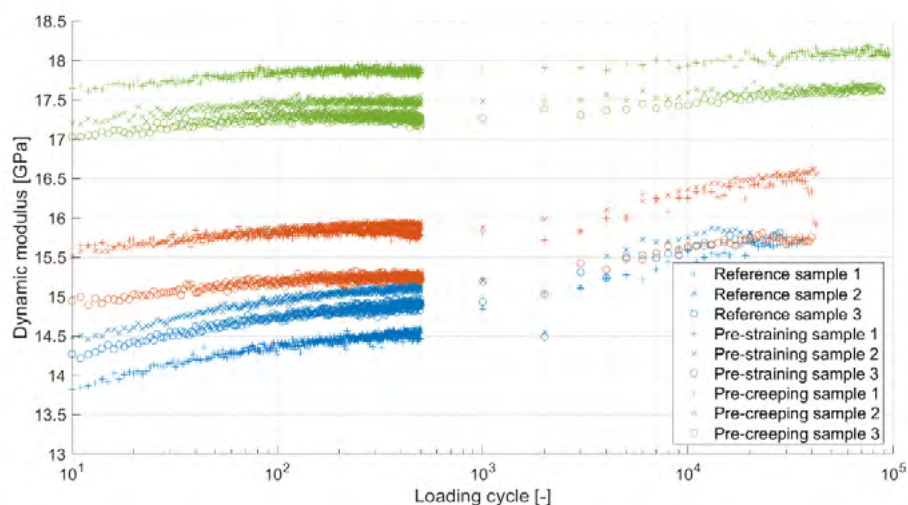


Figure 4. Effect of pre-straining and pre-creeping on dynamic modulus.

3.2. Effect of pre-straining and pre-creep on ratcheting

Ratcheting refers to an the incremental plastic strain accumulation during fatigue. Therefore, ratcheting is generally undesirable for structural applications as it creates significant deformation over the long-term. Here ratcheting is computed by measuring the shift in strain at the bottom of each hysteresis loop relative to the strain at loading cycle 10. Figure 5 shows that the ratcheting in the reference fatigue testing can be mitigated by the application of pre-straining. Furthermore, pre-creeping almost completely eliminates ratcheting and tends to a perfectly elastic behaviour with ratcheting values remaining below 0.05% strain for the entirety of the fatigue life.

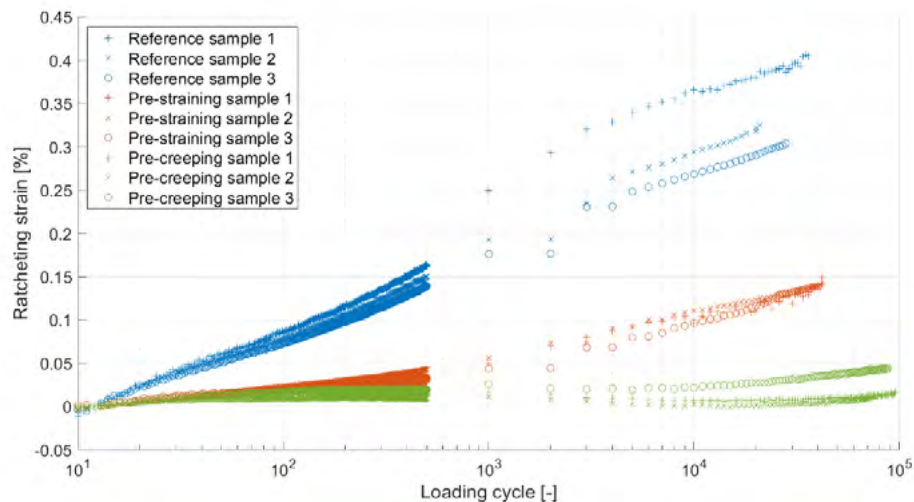


Figure 5. Mitigation of ratcheting with pre-straining and pre-creeping.

3.3. Ratio of inelastic work over total work

The ratio of inelastic work over total work is computed for individual loading cycles by dividing the area inside of the hysteresis loop by the area inside of the hysteresis loop plus the area below the hysteresis loop [7]. Figure 6 shows that this work ratio is decreasing during the entirety of the fatigue life indicating that most of the fatigue damage accumulation occurs towards the beginning of the fatigue life. The ratio is reduced by applying pre-straining or pre-creeping suggesting that less damage is accumulated at each loading cycle.

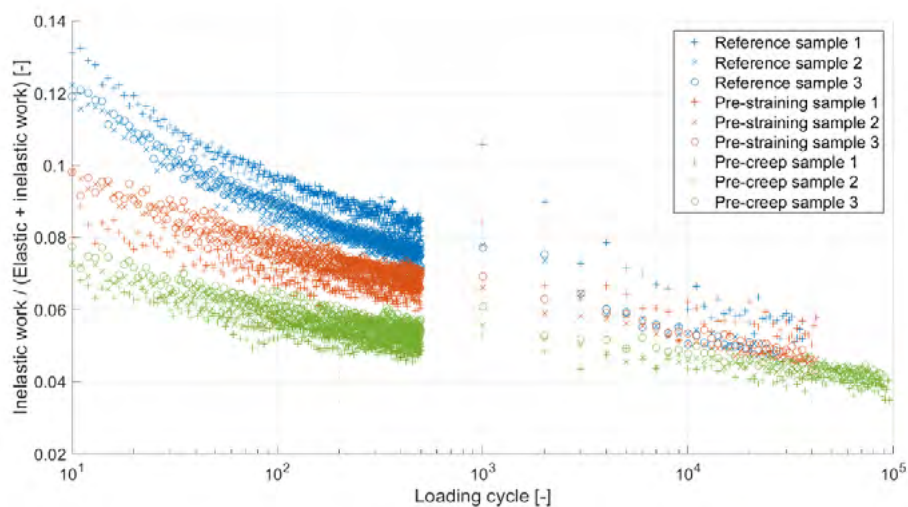


Figure 6. Effect of pre-straining and pre-creeping on the ratio of inelastic work over total work. The ratio is computed for individual loading cycles.

3.4 Effect of pre-straining and pre-creeping on fatigue life

Fatigue life results in figure 7 show that the pre-straining and pre-creeping methods have a significant impact on fatigue life. Pre-straining increases fatigue life by a factor of 1.3 compared to the reference specimens and remarkably pre-creeping increases the average fatigue life by a factor of 3. The comparatively large effect of pre-creeping compared to pre-straining highlights the importance of loading rate and viscous effects in FFRP composites. Given an alignment hypothesis, pre-creeping has a surprisingly significant impact, especially considering that the maximum strain achieved during pre-creeping is less than that reached during pre-straining.

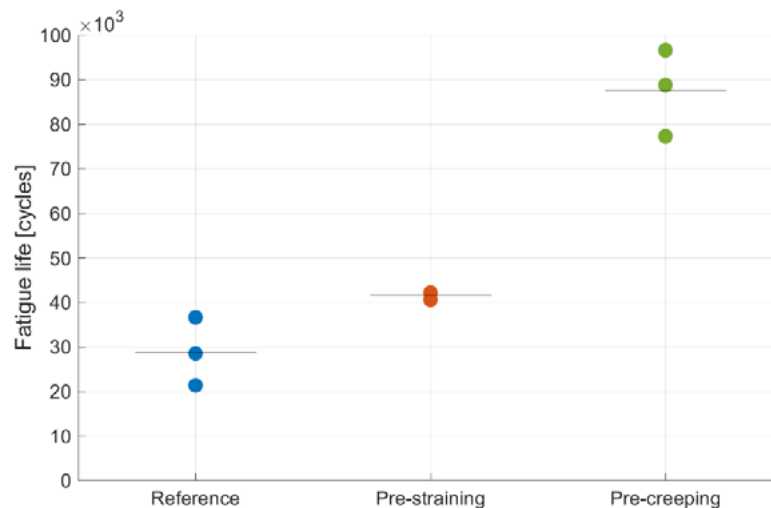


Figure 7. Effect of pre-straining and pre-creeping on the fatigue life .

3.5. Effects of temperature

Analysis of the recorded temperature during fatigue reveals that the air temperature in proximity to the specimen remained relatively constant at $22^{\circ}\text{C} \pm 2$. However, the surface temperature of the specimen increased by up to 10°C between the first and the 2'000th loading cycle (figure 8) likely due to the 5 Hz testing frequency and associated dissipation of energy. Then, temperature slowly decreased during fatigue until final failure at which point the surface temperature was about 3°C lower than at cycle 2'000.

Assuming that the stiffness of FFRPs is sensitive to temperature [8], the temperature profile in figure 8 can be used to explain the different dynamic modulus trends at the beginning and end of life in figure 4. Furthermore, at cycle 2'000 the temperature peaks indicating the equilibrium between the heat generated by the specimen deformation and the heat of the specimen dissipated to its environment due to the temperature gradient. Since the temperature decreases after 2'000 cycles, it suggests that either less heat is generated by the specimen deformation or more heat is dissipated to the environment. The decrease of the inelastic work/total work ratio observed in figure 6 is in agreement with the hypothesis that less heat is generated by the specimen deformation of every next cycle.

Additionally, the uncontrolled air temperature affected the repeatability of pre-creeping even with a $\pm 2^{\circ}\text{C}$ variation. Since the end of pre-creeping was defined with a strain limit, the ambient temperature variation from run to run affected the repeatability of the pre-creeping duration by affecting the stiffness of the FFRP composite [8]. Combined with sample to sample variation it resulted in the shortest pre-creeping lasting 5 hours and the longest 15 hours.

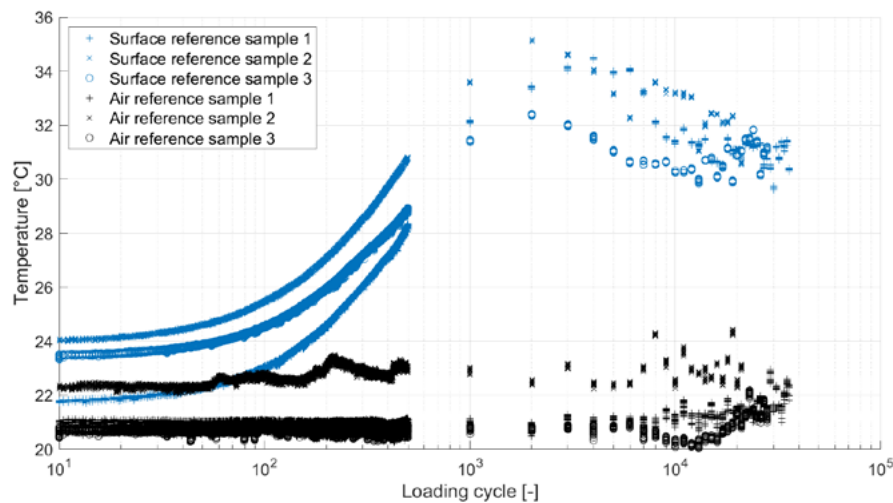


Figure 8. Comparison of the air temperature and the specimen surface temperature during fatigue

4. Conclusions

In this study, we presented a novel way of improving fatigue performance of structural flax fibre reinforced polymer composites. To this aim, we delved into the effectiveness of pre-straining and pre-creeping as methods for improving the fatigue performance of predominantly UD biobased FRP by taking advantage of the alignment mechanisms intrinsic to flax FRP composites. The main conclusion is that both methods, pre-straining and pre-creeping, have beneficial effects on the tension-tension fatigue performance of flax FRP composites with pre-creeping being the most effective.

The comparison of results of pre-strained and pre-creeped specimens highlighted the FFRPs mechanical response time dependency or viscoelasticity. Indeed, across all measured metrics - fatigue life, dynamic modulus, ratcheting - pre-creeping exerted a larger effect than pre-straining despite pre-straining inducing a higher maximum strain prior to fatigue. This implies that the loading rate plays a crucial role in any alignment mechanism occurring within flax FRP composites. By extension, the choice of the testing frequency is also susceptible to affects the fatigue response due to viscoelasticity. Additionally, flax FRPs were found to be sensitive to the temperature elevation generated by the 5 Hz testing frequency.

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