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FATIGUE BEHAVIOUR OF UNI-DIRECTIONAL FLAX FIBRE/EPOXY COMPOSITES

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ABSTRACT

A study related to the fatigue behaviour of natural fibre-reinforced composites was conducted to expand their range of product applications. A uni-directional flax-epoxy composite was fabricated and several conditions of tension-tension fatigue tests were performed. During fatigue testing, the composite showed an increase of stiffness, a typical observation for natural fibre-reinforced composites, and this was found to be accompanied by accumulation of residual strain. A clear linear relationship was found between the stiffening effect and the residual strain. In addition, it was revealed that the fatigue behaviour was clearly influenced by the frequency of cyclic loading. Lower frequencies induced more significant stiffening and shorter fatigue life. These results suggest that fatigue damaging is progressing simultaneously with the stiffening effect in natural fibre-reinforced composites, and it is therefore important to involve creep damaging to the failure criteria for these composites.

1 INTRODUCTION

Replacing synthetic fibres, such as glass fibres (GFs) and carbon fibres (CFs), with plant-based fibres (natural fibres, NFs) can be an effective way to realize carbon neutral materials because plants absorb a huge amount of CO_2 during their growth. NFs are also known to have mechanical properties comparable to glass fibres. Therefore, many research works related to NFs and natural fibre-based composites (NFCs) have been performed to clarify the mechanism behind the material characteristics, and to maximize the material performance.

However, recent industrial use of NFCs is mostly limited to non-structural components such as body panels of automobile from non-woven random mat of NFs. One of the reasons why industrial applications have been limited so far is the lack of knowledge related to their long-term reliability. Especially, fatigue mechanisms should be clarified to expand the application to load-bearing structural components. Although there are only a few studies related to the fatigue of NFCs, a common observation is stiffness increasing during fatigue test of NFCs [1, 2], while composites based on artificial fibres such as carbon fibres and glass fibres are known to show stiffness decreasing due to damage progression [3, 4]. Liang et al. [1] indicated that a NFC from a bi-axial flax fabric with yarns in 0° direction showed continuous increase of stiffness during almost all of the fatigue life while a GF-based composite (GFC) with an identical fibre configuration exhibited significant decrease of stiffness. Baley [5] reported that cyclic tensile loading of a single natural fibre also showed the stiffening phenomenon. Nevertheless, it still remains unclear whether the stiffening effect and the damaging,

where the latter one will result in stiffness decreasing, are progressing in parallel or sequentially. The answer to this question is very important to the fatigue design of products from NFCs and the structural health monitoring of them. The fatigue mechanism of this specific effect in NFCs should be understood in order to know and control the damage accumulation during the long-term use of NFCs.

Therefore, in this study, we performed tension-tension fatigue tests of uni-directional (UD) NFCs and detailed analyses on the results. The fatigue behaviour was evaluated by focusing on the stiffening effect and the concurrent change of residual strain.

2 MATERIALS AND METHODS

2.1 FABRICATION OF LAMINATE AND SPECIMEN

As a model material, we fabricated a uni-directional (UD)-NFC from a twisted flax yarn (Smeraldo, Nm 1/9.7, Linificio e Canapificio Nazionale SpA, Italy) and an epoxy resin (Araldite LY1564SP (epoxy) and Araldite 3486 (hardner), Huntsman, USA). The mechanical characteristics of the yarn and the cured resin are indicated in Table 1. Firstly, 14 layers of the yarn was wound on an aluminium plate (470 mm x 400 mm) using a custom-made winding machine. The misalignment of yarns was calculated to be only 0.061° and it was considered to be small enough to be neglected. After the winding process, the plate with the wound yarn was packed in a vacuum bag together with aid materials for the vacuum infusion moulding.

Then, the yarn assembly was impregnated by the resin using the negative pressure until the outlet tube was filled with resin. The entire aluminium plate with the impregnated yarn assembly was placed in a thermo-chamber for the curing process. After 5 hours of curing at 50°C, the composite laminate plates were de-moulded and put into the thermo-chamber again for the post-curing at 80°C for 4.5 hours. Figure 1 shows a cross sectional view of a laminate captured by X-ray computed tomography (Zeiss, Germany). The volume fraction of fibres (V_j), porosity (V_p) and the density of 4 fabricated laminates were measured to be 31.4 ± 0.2% (Mean±SD, n=4), 0.3 ± 0.3% (Mean±SD, n=4) and 1.28 ± 0.0 g/cm³ (Mean±SD, n=4), respectively. The laminates were cut into (i) butterfly-shaped specimens (Figure 2-left); a specimen geometry which has been specially designed for tensile fatigue testing of composites, and (ii) rectangular-shaped specimens (Figure 2-right). GFC-tabs were mounted on both types of test specimens.

	Linear density	Density	Tensile Modulus
Flax yarn [6]	88.9±2.7 g/1000m	1.59 g/cm^3	67-71 GPa
Epoxy resin	-	1.15 g/cm^3	2.9 GPa

Table 1: Mechanical characteristics of flax yarn and epoxy resin used in this study.



Figure 1: Cross-sectional view of fabricated flax yarn/epoxy composite laminate. Scale bar is 200 µm.



Figure 2: Schematic drawings of the test specimens used in this study. Left: Butterfly-shaped specimen, Right: Rectangular specimen

2.2 STATIC TENSILE TESTS

To determine the testing conditions for the fatigue tests, initial static tensile tests were performed with the rectangular-shaped specimens using a hydraulic testing machine (Instron, USA). Figure 3 shows a photograph of the tensile testing setup. Displacement rate was set at 2 mm/min. Tensile strain was defined as the average value of the measured strains by two extensometers with a 50 mm gauge length (Instron, USA) attached to each side of the specimens. Strain was recorded together with the exerted load and the applied displacement of the actuator.



Figure 3: Photograph of the setup for the initial tensile tests of composites using rectangular specimens.

2.3 FATIGUE TESTS

Fatigue tests were performed using an identical setup to the static tensile tests as indicated above. All fatigue tests were done in a stress controlled mode, and the stress ratio ($R = \sigma_{\min}/\sigma_{\max}$) was fixed to 0.1 (tension-tension mode). The applied stress level (σ_{\max}), the testing frequency (*f*) and the type of specimens are summarized in Table 2. During fatigue testing, stress-strain hysteresis loops were recorded intermittently. To obtain the S-N (σ_{\max} -number of cycles to failure) curve of the composite, linear regression was performed on the data pairs of log*N* - log*S*.

Specimen ID (Plate NoSpecimen No.)	Specimen type	Maximum stress, σ_{max} [MPa]	Frequency [Hz]
DV01-03	Butterfly	110	5
DV02-05	Butterfly	129	2
DV02-02	Butterfly	159	2
DV01-02	Butterfly	180	2
DV02-03	Butterfly	203	2
DV01-04	Butterfly	220	2
DV01-06	Butterfly	240	2
DV04-09	Rectangular	220	2
DV04-10	Rectangular	220	1
DV04-11	Rectangular	220	0.5
DV04-12	Rectangular	220	0.25

Table 2: Specimens and conditions for the fatigue tests.

3 RESULTS AND DISCUSSION

3.1 Tensile properties

Figure 4 shows stress-strain (S-S) curves for the UD-NFC composite obtained from the static tensile tests. As also reported in several previous reports [7, 8], the S-S curve exhibited transition points of the slope at about 0.2 to 0.3% of tensile strain. The S-S curve of the resin used in the composite is almost straight in this strain region (data is not shown), and therefore, this specific behaviour of the composite is expected to come from the characteristic of the fibres. From the S-S curves, the tensile stiffness (linear regression between strain 0.05 and 0.25%), and the ultimate tensile stress (UTS; maximum tensile stress) were measured to be 18.5 ± 0.5 GPa (mean±SD, n=5) and 282 ± 14 MPa (mean±SD, n=5), respectively.



Figure 4: Stress-strain curves for the UD flax-epoxy composite obtained from 5 tensile tests.

3.2 Fatigue properties

Figure 5 shows photographs of a butterfly specimen during a fatigue test. At first, a localized damage appeared on the edge of the gauge area. After another several hundreds of cycles, a splitting

crack propagated from the initial damage point to the start of the tab. Finally, the decrease of load carrying area induces bulk failure at the start of the tab. Figure 6 shows the *S*-*N* diagram of the UD flax-epoxy composite obtained by using the butterfly-shaped specimens. The fatigue data were well fitted to the linear model on the log*S*-log*N* plot (the Basquin's law), which also is used for conventional composites reinforced by glass or carbon fibres. A previous study on UD sisal/epoxy and polyester composites indicated that linear regressions on the *S*-log*N* plot was able to include results of the static tensile tests [2]. However, in the present study, as can be seen in Figure 6, it was not possible to cover both data of fatigue strength and data of static strength by simplified models, such as the linear models in the log*S*-log*N* plot and the *S*-log*N* plot.



Figure 5: An example of the fatigue failure observed in the UD flax-epoxy composite ($\sigma_{max} = 220$ MPa). The red circle indicates the position of the initial visible damage.



Figure 6: The logS-logN diagram of the UD flax-epoxy composite

Figure 7 shows examples of the hysteresis loops recorded during fatigue tests with two different stress levels. As reported in several previous studies, the slopes of the loops are becoming steeper until about the half point of fatigue life [1, 2]. Concurrently with the stiffening effect, the loops are shifted to the right in the stress-strain plot. This behaviour of increased residual strain is believed to be induced by creep deformation due to the constant component of the tension-tension fatigue loading and/or plastic deformation due to the repetition of the cyclic loading. The change of the stiffness and the residual strain during the fatigue tests are indicated in Figure 8A and B, respectively. For both parameters, it was difficult to find any common trend of change with respect to the stress level or the failure criteria. However, as indicated in Figure 9, when the increase of stiffness was plotted as a function of the residual strain (hereafter referred to as the stiffening-strain diagram), a clear correlation was found. Although the variation is becoming larger for the higher strain region, the correlation is almost linear until around 0.3% strain. This result might indicate that the stiffening effect is induced by accumulation of the residual strain, like the strain-hardening effect, which is a well-known phenomenon for metals.



Figure 7: Hysteresis loops during fatigue tests exemplified by the stress levels of $\sigma_{max} = 110$ MPa and 220 MPa. Dotted lines indicate the linear regressions for each loop.



Figure 8: Change of stiffness and residual strain during fatigue tests at different maximum stress levels. A: Change of stiffness. Vertical axis is defined as the differential increase of stiffness divided by the initial stiffness. B: Change of residual strain. The residual strain was defined as the differential increase of the minimum strain in each hysteresis loop.

If the magnitude of the stiffening effect is mainly modulated by the residual strain, which assumingly is dominated by creep strain, the change of the stiffness should be affected by the temporal pattern of the fatigue loading. Figure 10 shows the frequency-dependence of the fatigue behaviour of the UD flax/epoxy composite. At first, it can be observed that the stiffening effect is higher in the lower frequencies (Figure 10A). This result may suggest that the stiffening effect is determined mainly by the duration of loading, not by the number of cyclic loadings. In addition, interestingly, the slope in the stiffness-strain diagram (Figure 10B) is steeper in the lower frequencies. This result is reasonable if the cyclic component of fatigue loading induced the softening due to the fatigue damaging simultaneously with the stiffening effect. Therefore, although significant decrease of stiffness cannot be observed in the tension-tension fatigue test, it is believed that fatigue damaging is progressing from the beginning.

Although this study revealed a macroscopic response of the UD-NFC, a micro-mechanical mechanism of the fatigue behaviour still remains unclear. A previous study indicated that the stiffening effect can be implicated as a result of alignment of the cellulose microfibrils in the cell wall of natural fibres [9]. However, as indicated in Figure 10A, specimens tested in lower frequencies showed more rapid increase of the stiffness, and exhibited shorter fatigue life. Thus, there is a possibility that the stiffening effect is a sign of creep damaging, and both the creep damaging and fatigue damaging should be taken into account for the failure criteria of NFC exposed to fatigue loading.



Figure 9: Relationship between the stiffening effect and the residual strain. (The stiffening-strain diagram)

Figure 10: Results of fatigue tests performed at different frequencies. σ_{max} was fixed to 220 MPa for all tests. A: Relative increase of stiffness vs. number of fatigue cycles, B: The stiffening-strain diagram.

4 CONCLUSIONS

In the present study, several conditions of fatigue tests were performed on a uni-directional flaxepoxy composite. The following conclusions have been obtained.

(a) Increase of stiffness (the stiffening effect) was correlated to an increase of the residual strain.

(b) Fatigue tests performed with lower frequencies induced faster stiffening and shorter fatigue life.

These findings suggest that the stiffening effect in NFCs is induced by creep deformation, and its magnitude is affected by fatigue damage due to alternate loading.

REFERENCES

- [1] S. Liang, P. B. Gning and L. Guillaumat, A comparative study of fatigue bahavior of flax/epoxy and glass/epoxy composites, *Composites Science and Technology*, **72**, 2012, pp. 535-543.
- [2] A. N. Towo, M. P. Ansell, Fatigue of sisal fibre reinforced composites: Constant-life diagrams and hysteresis loop capture, *Composites Science and Technology*, **68**, 2008, pp. 915-924.
- [3] B. Liu, L. B. Lessard, Fatigue and damage-tolerance analysis of composite laminates: Stiffness loss, damage-modelling, and life prediction, *Composites Science and Technology*, **51**, 1994, pp. 43-51.
- [4] J. Zangenberg, P. Brøndsted, J. J. Gillespie, Fatigue damage propagation in unidirectional glass fibre reinforced composites made of a non-crimp fabric, *Journal of Composite Materials*, **48**, 2014, pp. 2711-2727.
- [5] C. Baley, Analysis of the flax fibres tensile bahavior and analysis of the tensile stiffness increase, *Composites: Part A*, **33**, 2002, pp. 939-948.
- [6] S. Mehmood and B. Madsen, Properties and performance of flax yarn/thermoplastic polyester composites, *Journal of Reinforced Plastics and Composites*, **31**, 2012, pp. 1746-1757.
- [7] B. Madsen, P. Hoffmeyer, H. Lilholt, Hemp yarn reinforced composites II. Tensile properties, *Composites: Part A*, **38**, 2007, pp. 2204-2215.
- [8] A. H. Abdullah, A. Khalina, A. Ali, Effects of fibre volume fraction on unidirectional kenaf/epoxy composites: the transition region, *Polymer-Plastics Technology and Engineering*, 50, 2011, pp. 1362–1366.
- [9] L. Kohler H. C. Spatz, Micromechanics of plant tissues beyond the linear-elastic range, *Planta*, 215, 2002, pp. 33-40.