HYGROTHERMAL FATIGUE OF STRUCTURAL BIOCOMPOSITES: PATHWAYS TO DAMAGE ANALYSIS AND LIFETIME PREDICTION

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Abstract: Biobased fibre reinforced polymer (FRP) composites, consisting of natural lignocellulosic fibres such as flax or hemp, are great alternatives to synthetic fibres to mitigate the environmental impact of high-performance engineering structures. Natural fibres such as flax have damping and specific mechanical properties suitable to potentially replace glass fibres in FRP composites. However, the hygroscopicity in natural fibres raises durability questions for structures subjected to seasonal environmental changes such as wind turbine blades. Existing research on flax FRPs describes on one hand damages related to hygrothermal ageing and on the other hand damage related to fatigue but the interaction of hygrothermal effect with mechanical ageing such as fatigue is not yet well understood.

A concept is proposed to relate the hygrothermal fatigue behaviour of biobased FRP composites to their fatigue behaviour in standard laboratory air conditions, using damage analysis comprised of permanent strains and stiffness variation measurements, as well as visual macro and micro damage inspection, to enable the development of a mechanism-based lifetime prediction model.

Keywords: Flax fibre, FRP, Biocomposites, Fatigue, Hygrothermal ageing

1 INTRODUCTION

The development of glass fibre reinforced polymer composites (GFRPs) has allowed for the design of ambitious large-scale engineering structures, thanks to the high specific mechanical properties of this material. With wind turbine blades in service for multiple decades, GFRPs have demonstrated adequate durability when exposed to environments with variable temperature and moisture combined with mechanical fatigue. However, those decades of operations have exposed problems with disposal and recycling of GFRPs at their end of life. Glass fibres are difficult to recycle, not suitable for incineration, except in cement kilns, and economic incentives often lead to landfilling solutions that are detrimental to the environment [1].

Recent research in the field of structural biobased FRPs has demonstrated that natural fibres such as flax are appropriate for the replacement of glass fibre in certain applications [2]–[4] enabling a reduction of environmental impact. However, to guarantee lower environmental impact, biobased FRPs must have sufficient durability, in this context fatigue life, which can be affected by hygrothermal conditions as natural fibres are hygroscopic. The impact of hygrothermal effects on the fatigue life of biobased FRPs is not known yet but multiple researchers have demonstrated that exposure to a high-humidity environment can create internal cracks and have a negative impact on the quasi-static mechanical properties of flax FRPs [5]–[8].

Hence this study investigates the relation between conventional fatigue tests performed on pristine specimens in standard lab environmental conditions (approximately 20°C, 50% relative humidity) and fatigue tests representative of in-service conditions by controlling hygrothermal conditions before and during fatigue testing. To that aim, the mechanism of hygrothermal effects combined with mechanical loading is studied by analysis of the damage captured mechanically; permanent strain, change of stiffness, and visually; change of colour, macro cracks, micro cracks. This paper presents an experimental methodology based on the current state of knowledge as well as preliminary testing on flax FRPs, and discusses the challenges of testing numerous time dependant parameters of different nature.

2 HYGROTHERMAL AND FATIGUE DAMAGE IN FRP

2.1 Structure of glass vs. flax FRPs

Contrarily to glass fibres which are uniform and isotropic, the internal structure of flax fibres is known to be anisotropic and non-uniform. The fibre of 10 to 30 µm, in diameter, extracted from the stem and separated from the bundle is referred to as elementary flax fibre in this paper (Figure 1). The elementary fibre itself is composed of cell walls made of cellulose microfibrils providing strength and stiffness mostly along the main axis of the fibre [9]. Flax FRP composites may contain elementary fibres, fibre bundles, or both, depending on the flax fabric specific manufacturing process.

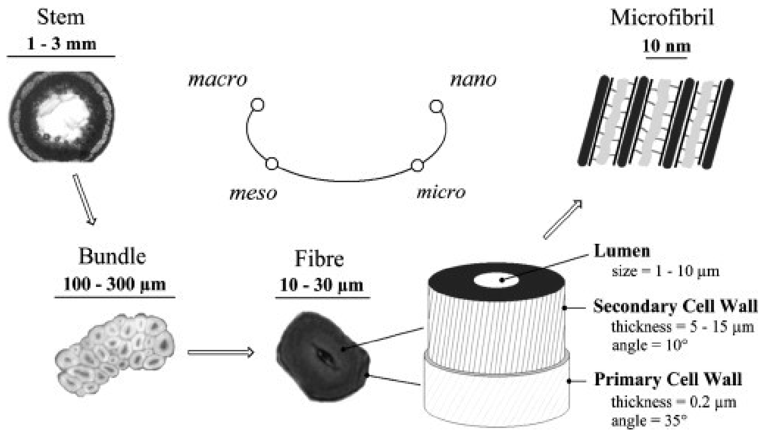

Figure 1: *Multi-scale hierarchical structure of flax fibres* [9].

2.2 Hygrothermally induced damage in glass vs. flax FRPs

When placed in an environment with high or low humidity FRP composites absorb or respectively desorb moisture by diffusion of water in the polymer matrix. In the case of glass FRP (GFRP), the fibres act as barrier to moisture diffusion [10] while in flax FRP (FFRP) the hygroscopic nature of the fibres makes them act as channels driving moisture diffusion [6] and actually absorb considerably more water in the fibres than in the matrix.. It results that FFRP composites exchange significantly more water with the environment than GFRP while exposed to the same conditions.

The main consequence of moisture absorption in FRP composites is a non-uniform change of dimensions creating internal stresses and eventually leading to cracking. Flax fibres have been demonstrated to quickly absorb/desorb moisture [11], leading to swelling/shrinkkage respectively [8], and when embedded in a polymer matrix, damage the fibre-matrix interface (Figure 2). Damage due to hygrothermal effects has also been observed in GFRPs [12] but with damage concentrated at the interface of matrix rich-fibre rich areas (Figure 3) rather than around every fibre as is the case for FFRP.

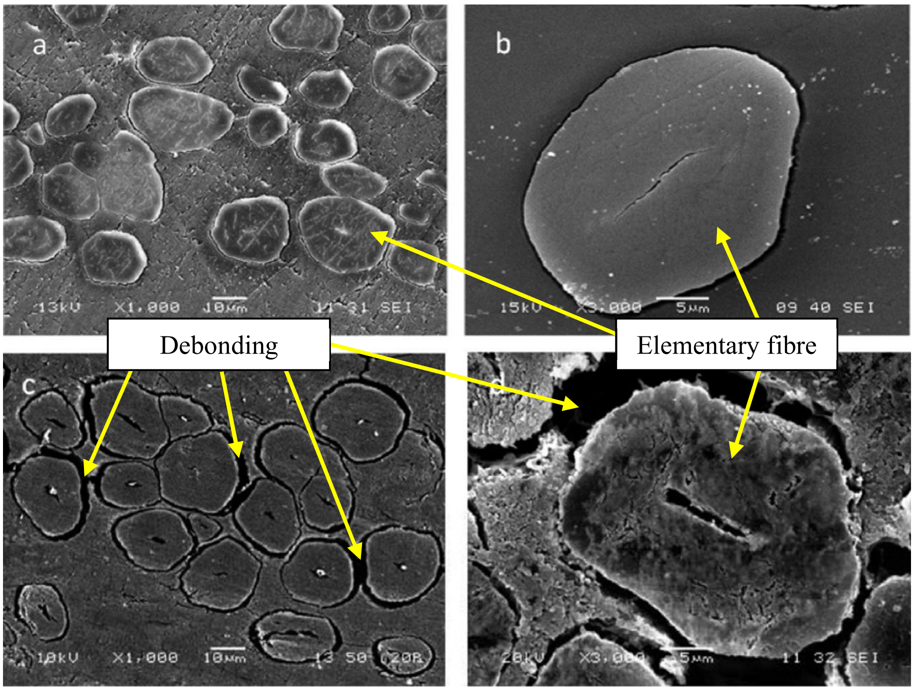

Figure 2: *Hygrothermal damage in flax FRP* [13].

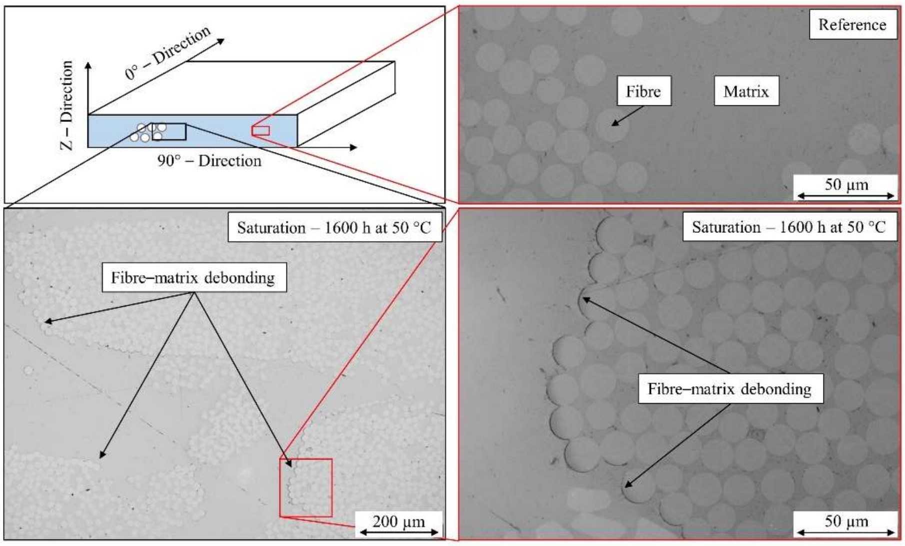

Figure 3: *Hygrothermal damage in glass FRP* [12].

2.3 Fatigue induced damage in glass vs. flax FRPs

Damage in FRPs due to mechanical loading is dependent on the type of loading (tension, compression, bending), fibre orientation, and loading axis. Mahboob et al. [14] have shown that for FFRP with 0°, 0/90°, and ±45° lay-up subjected to tension-tension fatigue debonding develops inside and/or around fibre bundles with progression as matrix cracks towards the end of life (Figure 4). The same authors showed that fatigue damage in GFRPs is also dominated by debonding with cracks mostly appearing at the fibre-matrix interface and being connected by cracks across matrix rich areas. (Figure 5).

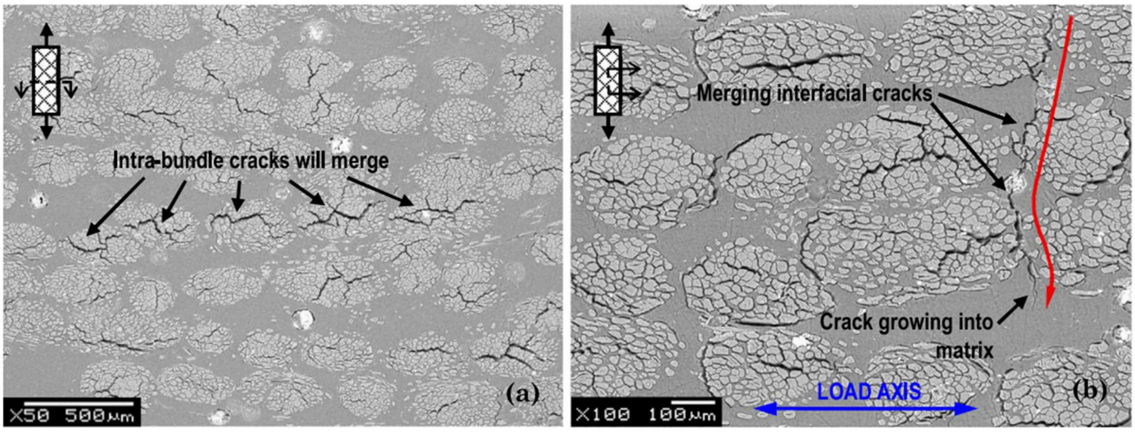

Figure 4: *Fatigue damage in flax FRP* [14].

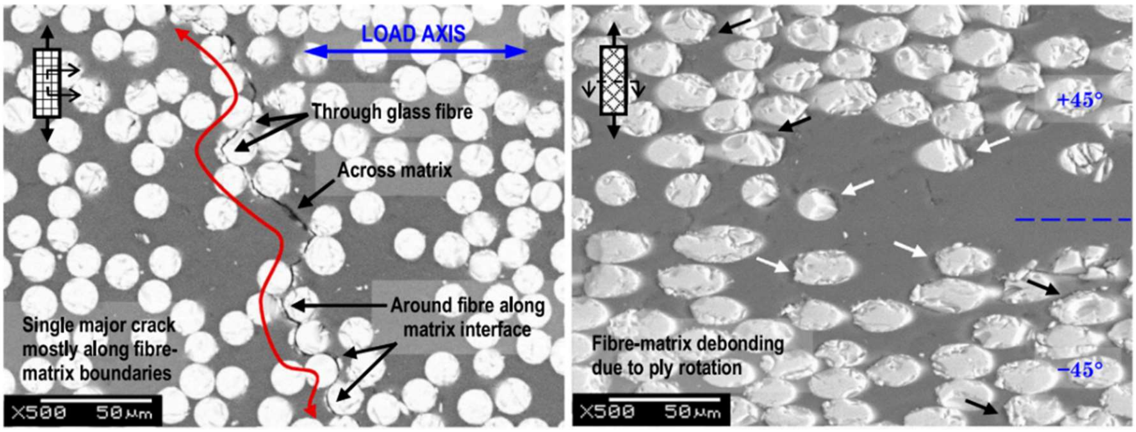

Figure 5: *Fatigue damage in glass FRP* [14].

2.4 Hypothesis on hygrothermal and fatigue interaction in damage evolution

Based on the above observations of flax FRP the hypothesis is made that the hygrothermally-induced damage will interact with the fatigue-induced damage and reduce the overal fatigue life of the composite without changing the damage mechanism. The fatigue life is expected to be reduced because fatigue cracks often grow from the fibre matrix interface and thus, if this interface is already damaged by hygrothermal ageing, opening the cracks requires less energy, so less cycles to failure. The deterioration of mechanical properties, permanent strain and elasticity modulus reduction, are expected to follow the same pattern (Figure 8) because fatigue cracks are expected to follow the same path whether they grow inside a pristine or hygrothermally damaged specimen. In summary, the observed and expected similarities of hygrothermal and fatigue damage mechanisms should allow for hygrothermal fatigue prediction with correction factors not only for the number of cycles to failure but also for permanent deformation and stiffness.

While hygrothermal effects can interact with fatigue by creating damage prior to mechanical loading as described above, or already in the early cycles of loading, hygrothermal conditions (in-service conditions) might also affect damage by changing the mechanical response, e.g. stiffness is lower for a fatigue test performed in a 50°C and 90% relative humidity environment (with the specimen at equilibrium with the environment) compared to stiffness when testing at standard conditions. Literature about the effect of in-service conditions on the mechanical properties of flax FRPs is very scarce, however based on tensile testing of dry and wet flax fibre yarns [15] and preliminary quasi-static tensile testing of FFRPs, it is expected that increase of both temperature and moisture of the specimen will result in decrease in stiffness and decrease in fatigue life.

3 EXPERIMENTAL METHODS

3.1 Specimens and test setup

FFRP plates with a symmetrical 0/90° lay-up are manufactured using a vacuum assisted epoxy resin infusion process followed by an autoclave post-cure. The rectangular specimens designed for tensile testing are cut from the plate using waterjet cutting. The angle of the cut is either set at 0° to obtain 0/90° specimens, or at 45° to obtain specimens with a ±45° lay-up. GFRP specimens are manufactured following the same process to serve as benchmark for FFRP.

For hygrothermal ageing or conditioning, specimens are placed in a climate chamber with controlled relative humidity and temperature. The temperature of the climate chamber is set at 50°C to increase the rate of moisture diffusion. During ageing or conditioning intermittent weight, width, and thickness measurements of the specimens are performed. The end of ageing or conditioning is determined by equilibrium with the environment based on weight meaurements.

For mechanical testing, a servo-hydraulic tensile testing machine with hydraulic grips is used. High-density paper tabs are glued to the specimens to increase friction in the grips. A climate chamber is installed around the grip and specimen area to control the temperature and relative humidity of the environment during testing. Stress and strain are measured using respectively the tensile testing machine force sensor and an extensometer attached to the specimen (Figure 6).

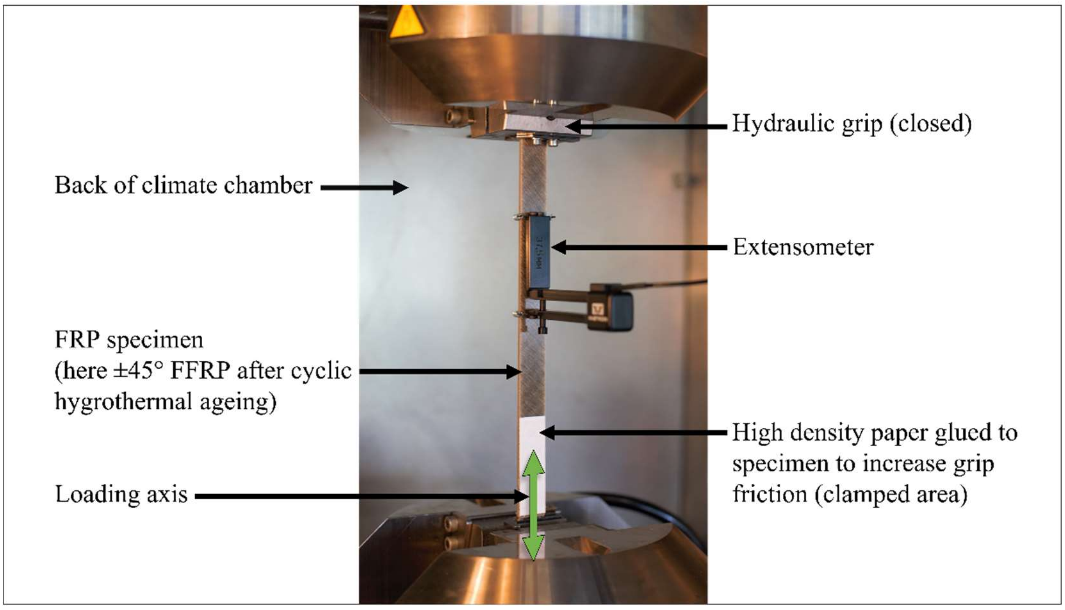

Figure 6: *Picture of mechanical test setup.*

3.2 Visual evaluation of damage

Visual inspection of specimens is performed at macro and micro scale to explain the mechanisms responsible for stability or variation of mechanical properties. Inspections are performed on the pristine specimen, on the specimen after fatigue, after hygrothermal ageing, and after sequential hygrothermal ageing and fatigue. Pictures of specimens after preliminary testing are shown in Figure 7 where macro damage is visible. After hygrothermal ageing and fatigue, damage appears as a global change of color of the specimen (3) and as strips of colour change parallel to the fibres (2) respectively. Analysis of damage at the micro-scale is performed by optical and SEM (scanning electron microscope) observations on cross-sections cut from the specimens after hygrothermal and fatigue testing.

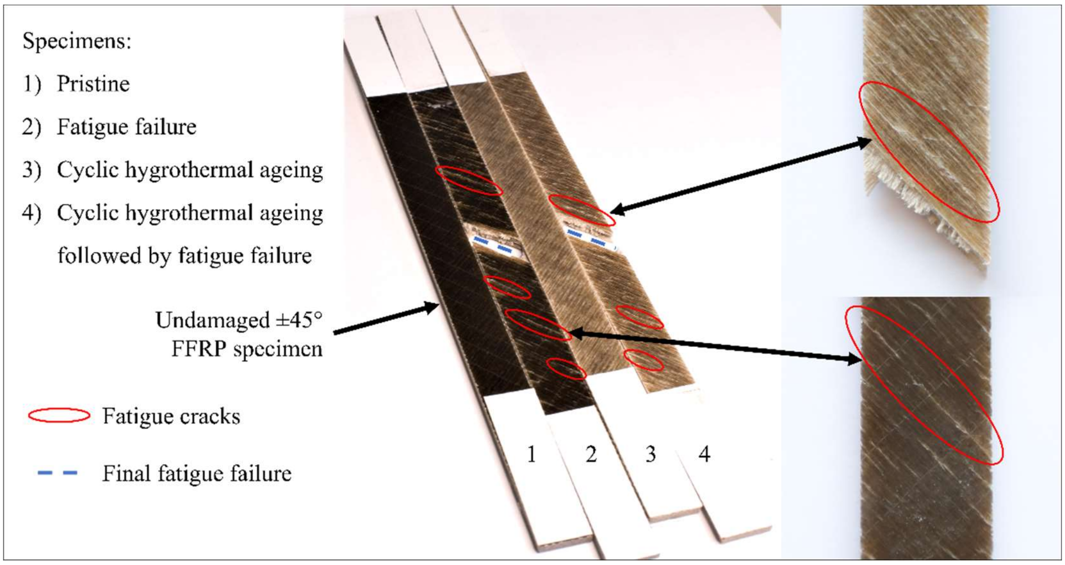

Figure 7: *Illustration of specimens color change and surface cracks.*

3.3 Mechanical evaluation of damage

Strain and stress are recorded during fatigue to track permanent strains and change in the elastic modulus during fatigue testing. Those measurements are used to compare the damage mechanism during fatigue in-between different hygrothermal ageing or hygrothermal conditions and will be related to the visual observations of damage.

Preliminary fatigue tests on ±45° FFRP lay-up in force-control with a maximum stress of 0.5, 0.6, and 0.7 UTS (ultimate tensile strength) comparing pristine specimens to hygrothermally aged specimens all showed a reduction of fatigue life due to hygrothermal damage. Figure 8 shows one of those tests with plotting of selected hysteresis curves. The overlap of the aged and unaged hysteresis curves suggests similar damage mechanisms happen despite damage growing about two times faster in the hygrothermally aged specimens.

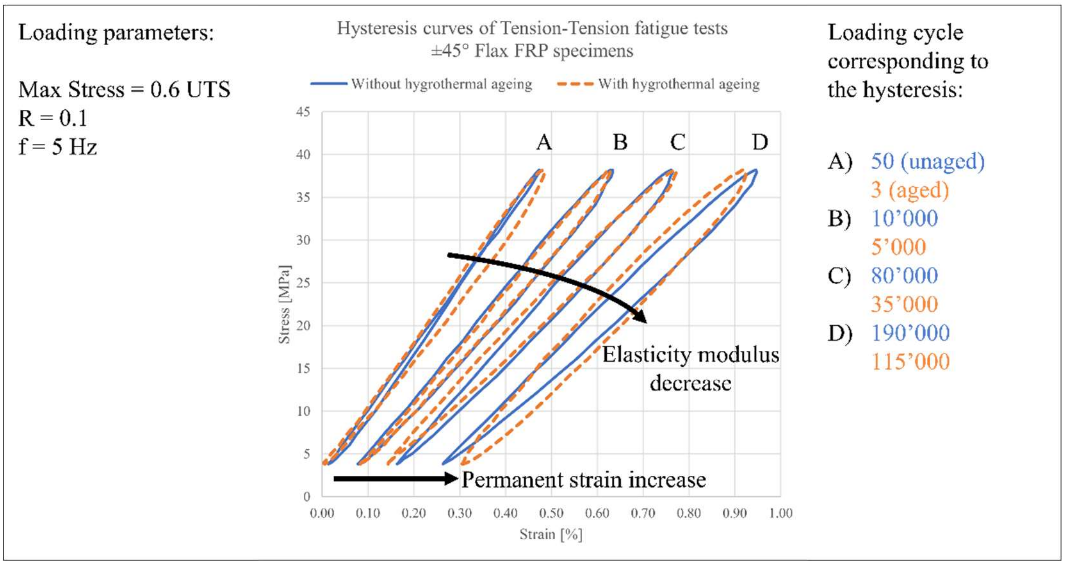

Figure 8: *Illustration of hysteresis overlap in fatigue ±45° flax FRP.*

4 DISCUSSION ON PARAMETERS, TIME, AND FATIGUE PREDICTION

Designing experiments to understand and predict the combined effects of fatigue loading and hygrothermal conditions is challenging due to the large number of variables involved. Conventional fatigue testing of FRPs for generating S-N curves can already be experimentally intensive, as factors such as peak load, fiber lay-up, loading ratio, and testing frequency may all influence the results. Adding hygrothermal conditions further increases the complexity, as temperature and moisture create numerous possible permutations of parameters.

To manage this complexity, the loading ratio and testing frequency are set to a fixed value based on existing literature, and two fiber lay-ups, 0/90° and ±45°, are selected to test extreme angles between the fibers and load. Quasi-static tests at the extreme expected in-service temperature and humidity levels are conducted to identify the hygrothermal conditions that have the most impact on mechanical performance. These conditions will then be used in fatigue testing.

Fatigue testing, especially combined with hygrothermal effects, requires careful consideration of time. To speed up testing, the testing frequency is commonly increased to a rate of multiple cycles per second well above the in-service loading rate which results in the testing duration of a single specimen usually from a few hours to about a week. However, hygrothermal cycles are dependent on moisture diffusion which can only be accelerated to a limited extent with the increase of temperature with one cycle being in the scale of multiple weeks. Therefore, testing with synchronisation of hygrothermal and mechanical effects representative of in-service conditions is impractical. To address this time-scale disparity, different specimens are mechanically tested in different constant hygrothermal conditions. Those test results will then be combined to predict the long-term effects considering the changes in hygrothermal conditions.

The preliminary results show that the types of damage in fatigue with or without hygrothermal ageing and the mechanical behaviour are similar but accelerated in case of hygrothermal effects. Based on these results, we aim to express hygrothermal fatigue life based on fatigue life in standard conditions combined with a reduction or knock-down factor, as could be done with the results of Figure 8.

5 CONCLUSIONS

The present study illustrates that the current knowledge on biobased FRP composites under combined mechanical fatigue and hygrothermal effects is insufficient to predict fatigue for materials exposed to in-service conditions for applications such as wind turbine blades.

Based on knowledge from the literature, damage growth at the fibre-matrix interface and inside fibre bundles has been identified as the critical failure mechanism driven both by hygrothermal ageing and mechanical fatigue. Following this observation experimental methods are proposed to investigate the evolution of damage both with a visual and mechanical assessment. Preliminary results are presented showing the similarities of damage due to cyclic hygrothermal ageing and cyclic mechanical loading (fatigue).

Finally, the challenges of the proposed experimental methods due to the high number of variables as well as time constraints and how fatigue life prediction of biobased FRP composites is approached are discussed.

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