Fatigue behavior of 3-D orthogonal woven composite with silane modification

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Abstract
As key part in wind turbine, the blade is a vital component for energy transformation. Fiber reinforced composite has advantage of light weight, large specific strength and specific stiffness, which has been used in wind turbine blade. In order to avoid delamination of conventional laminate and debonding between fiber and epoxy, the silane modification composite specimen reinforced with 3-D orthogonal woven fabric/epoxy was prepared using vacuum assisted resin transfer molding (VARTM) for blade. As for fatigue experiment, the critical stress levels corresponding to micro-crack initiation and failure of composite were obtained by combining of acoustic emission technology and quasi-bending experiment. The fatigue behavior of 3-D orthogonal woven composite with silane modification was experimental evaluated based on the minimum and maximum stress levels. The fatigue behavior, such as S-N curve, stress-strain behavior and stiffness degradation behavior were analyzed. The result will provide the basis for investigation of bending behavior with nondestructive tests and set of stress level in bending fatigue experiment.

1. Introduction
As key part in wind turbine, the blade is a vital component for energy transformation. Fiber reinforced composite has advantage of light weight, large specific strength and specific stiffness, which is used in wind turbine blade. With increase of blade length and harsh service environment, the blade will bear the most static and dynamic loading [1], and the fiber reinforced composite with light weight and higher strength [2, 3] is usually used in blade. The multiaxial warp-knitted composite has specific strength, specific modulus and good fatigue performance, which is usually used as skin and main beam in blade. However, 3-D woven composite has better shear and bending behavior in comparison to multiaxial warp-knitted composite [4] due to the existence of Z-yarns in thickness of fabric. In comparison to 2D plain weave composite with same fiber volume fraction, the 3-D orthogonal woven composite exhibited improved mechanical properties including tensile strength, flexural strength, impact strength and inter-laminar shear strength (ILSS) [5]. In order to avoid the delamination, the 3-D orthogonal woven fabric is selected to fabricate the blade. The abbreviation ‘3DOWF’ and ‘3DOWC’ is used represent 3-D orthogonal woven fabric and 3-D orthogonal woven composite, respectively. The principle of 3DOWF is to bind straight warp and weft yarns together by using Z-yarns which run through the whole thickness of fabric. Since the existence of Z-yarns in the 3-D structures, the composite has advantages of delamination resistance and damage tolerance in comparison to 2D woven composite due to Z yarn [6–10].

In recent years, researchers investigated the fatigue behavior of composite reinforced with 3DOWF by means of experiment and finite element analysis. Jin et al [11, 12] simulated the tension-tension fatigue behavior with FEA and analyzed the damage process, stress distribution and crack propagation. Yao et al [13] conducted the bending fatigue behavior, residue strength and modulus, etc, of 3-D woven composite. As detailed in above reference, the stress level in fatigue process was defined as a specific ratio to ultimate quasi-static strength, which
is not scientific. In order to accurately assess the fatigue behavior, it is important to determine the critical stress level corresponding to micro-crack initiation and failure.

Since glass fiber is inorganic fiber and epoxy is organic substance, the adhesion force between fiber and epoxy resin is poor. As is well known, the failure of composite includes matrix crack, debonding between fiber and epoxy and fiber breakage. As far as the poor binding force between glass fiber and epoxy, more and more researchers attempt to improve the interfacial binding force by means of modification. The mechanical behavior of glass fiber composite could be increased by applying nanoclay modification [14, 15]. Jiang et al [6] modified the 3-D orthogonal woven composite with silane couple agent and improved tensile and bending behavior. The effect of nanoclay addition on fiber/epoxy adhesion was characterized by the single-fiber micro-bonding method [16]. The result showed that the addition of nanoclay formed a stronger fiber-matrix interface.

Since composite material will release strain energy and transient elastic wave when it is exposed to load, which is named after AE (Acoustic emission). The AE source of composite include matrix crack, debonding between fiber and matrix, delamination and fiber breakage, which results in variation of AE count and energy. The critical stress can be obtained by analyzing the AE signal. Unnthorsson et al [17] considered that the failure of carbon reinforced composite in cyclic loading could be characterized by AE technology. AE technology was used to detect the defects and density of crack in carbon fiber reinforced concrete composite [18, 19]. Baran et al [20] detected the micro-crack initiation in carbon fiber reinforced polymer composites which subjected to shear stresses with acoustic emission (AE). The author considered that the acoustic emission historic index is the most effective AE parameter in detecting damage initiation. The AE count and energy were used to determined damage mode of 3-D woven composite during fatigue process [11–21]. Based on above studies, we can conclude that the AE technology is an effective method of detection of composite damage.

In this paper, the composite blade reinforced with 3DOWF and epoxy with silane modification is prepared using vacuum assisted resin transfer molding. The critical stress levels corresponding to micro-crack initiation and failure of composite are obtained by combining of acoustic emission technology and quasi-bending experiment. Fatigue behavior of 3DOWC, i.e. S-N curve, stress-strain behavior and stiffness degradation are experimental evaluated based on different stress levels.

2. Materials and composite fabrications

2.1. Material

The 3DOWF perform for this study is produced by Changzhou Topweaving New Material Tech. CO., Ltd. (Changzhou, China). The perform consists of three warp and four weft yarn layers, as shown in figure 1. The structural parameters of 3-D orthogonal woven fabric is shown in Table 1.

The epoxy resin and curing agent are E-2511–1A and 2511–1BT, and supplied by Shangwei (Tianjin) wind power material Co., Ltd. Silane is A-1387, a commercial product, supplied by Foshan Daoning Chemical Co., Ltd. (China).

2.2. Composite preparation

Three layers of 3DOWF was stacked as reinforcement. The solution composed of 100 parts mass of epoxy resin (E-2511–1A) and 30 parts mass of curing agent (2511–1BT) was selected as matrix. The silane A-1387 content at
1.2wt% of epoxy hardener mixture was dispersed in the mixture. The 3DOWC was manufactured by means of Vacuum Assisted Resin Transfer Molding [22].

The fiber volume fraction \( V_f \) of the 3DOWC measured using the matrix burn-off test is 53.13% according to standard of ASTM D3171–15.

3. Mechanical tests

3.1. Quasi-static three-point bending and AE tests

The quasi-static three-point bending tests were performed on WDW-30 materials tester at the speed of 3 mm min\(^{-1}\) according to the ASTM D 790 standards. Two AE sensors were attached on the surface of specimen, and the spacing between AE sensors was 130 mm, as shown in figure 2. The damage and crack progression of specimen during bending process were detected by AE event counts and event energy. In order to ensure the sensitivity and eliminate external noise as much as possible, the threshold of AE test was set to 40 dB.

The ratio of specimen length to depth is 16. The size of the 3DOWC specimens for three-point bending tests is 200 mm (length) × 20 mm (width) × 7 mm (thickness).

3.2. Bending fatigue test

Bending fatigue behavior of the 3DOWC was tested at different stress levels with MTS Landmark fatigue tester. The stress ratio of specimen during bending fatigue test was set to 0.1 and the frequency was set to 4 Hz. The span of specimen was 112 mm according to the standard of ASTM D790.

4. Results and discussions

4.1. Three-point bending tests and AE tests

The AE count and energy were selected for characterizing the deformation of composite from crack initiation to failure. The curve of bending stress, event count, and event energy versus strain were used to determine the critical stress at micro-crack initiation and failure. The first inflection point in curve of event count versus strain is the moment of crack initiation, the stress at this point corresponds to micro-crack. The maximum stress corresponding to failure is also determined with same method. The variations of mechanical properties of composite in 0° and 90° directions were recorded with AE technology, as shown in figures 3 and 4.

As shown in figures 3 and 4, the AE count and energy gradually increase at initially and steeply increase at a certain point, which indicates that crack is initiated. The cracks are propagated with increase of bending load.
shows that the stress levels at the initial crack is 50%, and the maximum stress level corresponding to failure is 90%. The representative stress levels of three damage stages are 50%, 70%, and 90%, which are indicated on the figure.

The distribution of three stage is shown in table 2. It shows that the deformation time of composite are composed of three stages, which relates with three stress levels, i.e. crack initiation, fracture and failure of composite.

4.2. Bending fatigue test
Bending fatigue test of composite were conducted at different stress level, which is defined as the ratio of the applied maximum stress $S_{\text{max}}$ in one cycle to the ultimate static bending stress $\sigma_{\text{ult}}$ of the composite specimen. It can be founded that stress levels are 50% and 90% by combining quasi-bending experiment and AE detection. As for loading level of 50%, the micro-cracks are initiated. However, the maximum loading level is 90%, and the composite is failed. In order to evaluate the fatigue performance from micro-crack initiation to failure, the tests were performed under five stress levels, i.e., 50%, 60%, 70%, 80% and 90%.

Figure 3. The stress and AE parameters versus time for composite specimens in 0 direction.

(a) Count and cumulative count

(b) Energy and cumulative energy
4.2.1. S-N curve

Fatigue life (S-N) of the 3DOWC specimens in 0° and 90° direction are presented in figure 5. It shows that the number of cycles to failure of composite with silane modification has much more than that of pristine [23].

With respect to the Weibull distribution, the S-N curve of fiber reinforced composite could be fitted with equation (1).

\[
S = 1 + a \times \left( \exp \left( -\left( \frac{\log(N + 1)}{b} \right)^c \right) - 1 \right)
\]  

(1)

Where, \( S \) is stress level, \( N \) is number of cycles to failure, \( a, b \) and \( c \) are constants.

As shown in figure 5, the S-N curves were fitted with equation (1). The constants are shown in table 3. The obtained equation can be used to predict fatigue life of composite.

Fitting aptness is assessed by comparing the coefficient estimation \( R^2 \). The higher the coefficient (near to unity), the better the model will be more fitted to experimental results.

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Figure 4. The stress and AE parameters versus time for composite specimens in 90° direction.

Table 2. Time distribution in three stages.

| Stage | Direction | First stage/s | Second stage/s | Third stage/s |
|-------|-----------|---------------|----------------|---------------|
|       | 0°        | 0–148.50      | 148.50–174.10  | 174.10–550.40 |
|       | 90°       | 0–145.02      | 145.02–182.48  | 182.48–505.60 |

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4.2.2. Stress-strain curves

In this part, the bending stress ($\sigma$) and strain ($\varepsilon$) were calculated according to equations (2) and (3). Then, the bending modulus was evaluated based on initial elastic part of bending stress and strain curve using equation (4) [22].

$$\sigma = \frac{3F.L}{2bh^2}$$  \hspace{1cm} (2)

$$\varepsilon = \frac{6Sh}{L^2}$$  \hspace{1cm} (3)

$$E = \frac{\Delta F.L^3}{\Delta S.Abh^5}$$  \hspace{1cm} (4)

Where, $F$ is the load applied at the central part of coupon (the maximum bending load $F$), $\Delta F$ is the loading increment of $F$, $\Delta S$ is the central deflection increment. $L$, $b$ and $h$ are the span between two supporting rolls, width and thickness of sample, respectively.

The stress-strain curves of the 3DOWC with silane modification in $0^\circ$ and $90^\circ$ directions at stress level of 70% are shown in figure 6. The stress-strain curves show hysteresis loops during the bending fatigue process.
With increase of loading, the strain is increased and number of hysteresis loop is also increased, the damage is accumulated to failure.

4.2.3. Stiffness degradation curve

In order to characterize cumulative damage process of 3DOWC under bending fatigue loading, the ratio of stiffness in each fatigue cycle to initial modulus versus Number of Cycles of the 3DOWC under the stress level of 70% is shown in Figure 7.

The variation of bending modulus during fatigue test is calculated according to secant modulus and normalization method. The stiffness is represented by bending modulus, which can be calculated by equation (4). $E$ is the ratio between loading increment and central deflection increment in definite hysteresis loop; $E_0$ is bending elastic modulus of the composite in the first number of cycle. The ratio of $E$ to $E_0$ is used to indicate the variation of stiffness, as shown in Figure 7.

Figure 7 shows that the parameter of $E/E_0$ is decreased with increase of fatigue cycles. According to Figure 7, there are three stages. At stage I, the rapid decline of stiffness is attributed to matrix damage. At stage II, the variation of stiffness is gradually decreased with increase of cycles, and the damage is attributed to debonding between fiber and matrix, part of fiber breakage and etc. However, it shows dramatic decrease in III stage, which is attributed to fiber breakage at moment. The variation of cycles in every stage is shown in Table 4.

Table 4 shows that the second stage accounts for 94.23% of whole stiffness degradation process. At this stage, the main damages are debonding between fiber and matrix, part of fiber breakage and etc. We can obtain that the fatigue behavior of composite with silane modification is improved by increasing binding strength in comparison to result [23].
5. Conclusion

In this paper, the composite blade reinforced with 3DOWF and epoxy was prepared using vacuum assisted resin transfer molding. The silane was added to solution of resin and curing agent to improve the bonding strength. The AE technology was used to detect the crack imitation and fracture failure during quasi-bending test. The critical stress levels corresponding to micro-crack initiation and failure of composite are 50% and 90% by combining of acoustic emission technology and quasi-bending experiment. The fatigue behavior of 3DOWC in 0° and 90° directions were experimental evaluated based on five stress levels, i.e., 50%, 60%, 70%, 80% and 90%. The fatigue behavior were analyzed with respect to the S-N curves, the stress-strain curves and the stiffness degradation curves. The fatigue results show that the composite with silane modification has great number of cycles to failure than pristine one. The stiffness degradation curves also show that the second stage of silane modification composite accounts for more proportion than that of pristine one due to improvement of bonding strength between fiber and matrix.

Figure 7. The variation curve of stiffness (stress level: 70%) (a) in 0° direction (b) in 90° direction.

Table 4. The variation of cycles in three stages.

| Direction | First stage (%) | Second stage (%) | Third stage (%) |
|-----------|----------------|-----------------|----------------|
| 0°        | 1.18           | 94.23           | 4.59           |
| 90°       | 0.72           | 95.26           | 4.03           |

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References

[1] Malhotra P, Hyers R W, Manwell J F and McGowan J G 2012 A review and design study of blade testing systems for utility-scale wind turbines Renew. Sustain. Energy Rev. 16 284–92
[2] Lekou D1 and Philippidis T P 2009 PRE- and POST-THIN: a tool for the probabilistic design and analysis of composite rotor blade strength Wind Energy 12 676–91
[3] Mishnaevsky L 2012 Composite materials for wind energy applications: micromechanical modeling and future directions Comput. Mech. 50 195–207
[4] Bilisik K 2009 Multiaxial 3D woven preform and properties of multiaxial 3D woven and 3D orthogonal woven carbon/epoxy composites Journal of Reinforced Plastics & Composites 29 1173–86
[5] Nayak S Y, Heckadka S S, Sadanand R V, Bhuradwaj K, Pokharna H M and Sanjive A R 2017 2D woven/3D orthogonal woven non-crimp e-glass fabric as reinforcement in epoxy composites using vacuum assisted resin infusion molding J. Eng. Fibers Fabrics 12 12–9
[6] Jiang X, Ma Y Y and Gao X P 2018 Mechanical properties improvement of silane addition epoxy/3D orthogonal woven composite material The Journal of The Textile Institute 109 1341–7
[7] Carvelli V, Gramellini G, Lomonov S V, Bogdanovich A E, Mungalov D D and Verpoest I 2010 Fatigue behavior of non-crimp 3D orthogonal weave and multi-layer plain weave E-glass reinforced composites Compos. Sci. Technol. 70 2068–76
[8] Ivanov D S, Lomonov S V, Bogdanovich A E, Karahan M and Verpoest I 2009 A comparative study of tensile properties of non-crimp 3D orthogonal weave and multi-layer plain weave E-glass composites. Part 2: comprehensive experimental results Composites Part A: Applied Science and Manufacturing 40 1144–57
[9] Lomonov S V, Bogdanovich A E, Ivanov D S, Mungalov D, Karahan M and Verpoest I 2009 A comparative study of tensile properties of non-crimp 3D orthogonal weave and multi-layer plain weave E-glass composites. Part 1: materials, methods and principal results Composites Part A: Applied Science and Manufacturing 40 1134–43
[10] Walter T R, Subhash G, Sankar B V and Yen cf 2010 Monotonic and cyclic short beam shear response of 3D woven composites Composites Science & Technology. 70 2190–7
[11] Jin L M, Jin B C, Kar N, Nutt S, Sun B Z and Gu B H 2013 Tension-tension fatigue behavior of layer-to-layer 3D angle-interlock woven composites Mater. Chem. Phys. 140 183–90
[12] Jin L M, Yao Y, Yu Y M, Rotich G, Sun B Z and Gu B H 2014 Structural effects of three-dimensional angle-interlock woven composite undergoing bending cyclic loading Science China Physics, Mechanics and Astronomy 57 501–11
[13] Yao L, Rong Q, Shan Z D and Qiu Y P 2013 Static and bending fatigue properties of ultra-thick 3D orthogonal woven composites J. Compos. Mater. 47 569–77
[14] Wang C, Gao X P and Li Y G 2019 Mechanical properties improvement of nanoclay addition epoxy 3D orthogonal woven composite material Polym. Test. 82 160–70
[15] Withers G J, Yu Y, Khabashesku V N, Cerccone L, Hadjiej V G, Souza J M and Davis D C 2015 Improved mechanical properties of an epoxy glass-fiber composite reinforced with surface organomodified nanoclays Composites Part B-Engineering 72 175–82
[16] Dorigato A, Morandi S and Pegoretti A 2012 Effect of nanoclay addition on the fiber/matrix adhesion in epoxy/glass composites J. Compos. Mater. 46 1439–51
[17] Wehrens R, Runnarsson T P and Jonsson M T 2008 Acoustic emission based fatigue failure criterion for CFRP Int. J. Fatigue 30 11–20
[18] Choi W C and Yun H D 2015 Acoustic emission activity of CFRP-strengthened reinforced concrete beams after freeze–thaw cycling Cold Reg. Sci. Technol. 114 47–58
[19] Mikael J and Peter G 2000 Broad-band transient recording and characterization of acoustic emission events in composite laminates Compos. Sci. Technol. 60 2803–18
[20] Baran I J, Nowak M B, Chlopek J P and Konsztowic K J 2018 Acoustic emission from microcrack initiation in polymer matrix composites in short beam shear test J. Nondestruct. Eval. 37 3–10.
[21] Karahan M, Lomonov S V, Bogdanovich A E and Verpoest I 2011 Fatigue tensile behavior of carbon/epoxy composite reinforced with non-crimp 3D orthogonal woven fabric Compos. Sci. Technol. 71 1961–72
[22] Gao X P, Li D X and Wu W 2018 Experimental investigation of the tensile and bending behavior of multi-axial warp-knitted fabric composites Textile Res. J. 88 333–44
[23] Gao X P, Tao N N, Yang X R, Wang C and Xu F J 2019 Quasi-static three-point bending and fatigue behavior of 3D orthogonal woven composites Composites Part B-Engineering 159 173–83