

TENSILE FATIGUE PERFORMANCE OF CARBON-CARBON HYBRID QUASI-ISOTROPIC LAMINATE

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Abstract: *This experimental study has been focused on the tensile-tensile fatigue performance of a carbon-carbon fiber hybrid quasi-isotropic laminate. The laminate contained two unidirectional carbon fiber prepregs with the same epoxy matrix. One prepreg had carbon fibers T800 for the high-elongation layer (HE), the other prepreg had carbon fibers DIALEAD for the low-elongation layer (LE). The sub-laminate stacking sequence was [HE/LE/HE]. Sub-laminates have been piled to get a quasi-isotropic layup [0/45/90/-45]_s. The displacement (strain) controlled tension-tension tests were along the 0° fiber direction. Preliminary quasi-static tests provided the pseudo-ductile behavior considered for the fatigue loading levels. The evolution of the fatigue damage was macroscopically analyzed by the stiffness degradation, and microscopically by X-ray micro-CT observations. As main conclusions, the composite retains its load-carrying ability in the pseudo-ductile regime. The evolution of the fatigue damage involved fracture of 0° LE plies, transverse cracks in ±45° plies and delamination of 0°/+45° and ±45°/90° interfaces.*

Keywords: Carbon-carbon hybrid laminates; Pseudo-ductility; Fatigue; Damage.

1. Introduction

One of the main advantages of fiber reinforced composites is the design freedom, which can be better exploited using fiber-hybridization (coupling two or more fiber types). Fiber-hybrids reinforced plastics received particular attention due to the synergetic (or 'hybrid') effects [1,2], which can provide, among other features, the pseudo-ductility, namely a pseudo-ductile response by the combination of brittle composites [3]. Pseudo-ductility is often studied in quasi-static tension and the damage mechanisms underneath are reasonably well-understood [4,5].

Although cyclic loading is often the main condition on composite components in several industrial applications [6], the current knowledge on the fatigue behavior of fiber-hybrid composites is limited. Focusing the attention on tension-tension fatigue loading, being frequently adopted in experimental investigations as well as in the present study, the few available researches have been mainly dedicated to unidirectional hybrid laminates, coupling: carbon and glass fibers (e.g. [7]), carbon and basalt fibers (e.g. [8]) and all carbon fibers (e.g. [9]). The fatigue performance of hybrid laminates with other stacking sequences was barely investigated (see e.g. [10]). This is the aim of the present study, dedicated to an all-carbon interlayer hybrid quasi-isotropic laminate.

The hybrid laminate contained standard thickness prepregs, same matrix, reinforced with low elongation (LE) and high-elongation (HE) carbon fibers. The sub-laminates [HE/LE/HE] were stacked to have a quasi-isotropic layup [0/45/90/-45]_s.

The tension-tension strain-controlled cyclic loadings allowed to gain several insights, mainly related to: the fatigue life of the hybrid laminate for a wide range of maximum strain levels in the quasi-static tensile pseudo-ductile range and beyond it; the evolution of the fatigue damage by the stiffness retention as macroscopic metric; the damage observation by X-ray micro-CT imparted after selected number of cycles. The final goal of the study was the correlation of the macroscopic stiffness degradation and the evolution of the fatigue damage modes.

2. Carbon-carbon hybrid quasi-isotropic laminate

The laminate has been manufactured with two unidirectional carbon fiber prepregs supplied by Composite Materials Italy (CIT) – Toray Group, which had both the epoxy matrix ER450. The prepregs nominal fiber volume fraction was 55–56% (datasheet). The high contrast between the low-elongation (LE) and high-elongation (HE) composite stiffness and strain-to-failure (see fiber properties in Table 1) provides a pseudo-ductile plateau in quasi-static tension of UD hybrid laminates [11], which was related to the critical energy release rates [12].

One prepreg contained TORAY TORAYCA® T800 carbon fibers (in the following T) for the HE layer (thickness of 0.11 mm), the other prepreg was made of Mitsubishi Chemical DIALEAD™ K63720 carbon fibers (in the following D) for the LE layer (thickness of 0.22 mm). The considered laminate consisted of a set of sub-laminates. The sub-laminate had one ply of the D prepreg sandwiched between two plies of T [T/D/T]. Sub-laminates were stacked to create a quasi-isotropic layup [0/45/90/-45]_s. The laminates were then autoclave-cured for 2 hours at a temperature of 135 °C and a pressure of 4.5 bar. No post curing or conditioning was applied.

Table 1: Carbon fiber properties (manufacturer's data sheets).

ID	Fiber	Diameter [μm]	Elastic modulus [GPa]	Strength [MPa]	Failure strain [%]
T	TORAYCA® T800	5	294	5880	2
D	DIALEAD™ K63720	11	630	2620	0.4

3. Quasi-static and fatigue loading features

Preliminary quasi-static tensile tests were dedicated to the evaluation of the main mechanical features including the pseudo-ductile behavior. The results have been considered for the selection of the fatigue loading levels. The loading rate, along the 0° fiber direction, was set to 1 mm/min by an electromechanical machine INSTRON 5978. The tests were assisted by a digital camera to evaluate the full field strain on the specimen surface by the digital image correlation (DIC) technique (as in [11] for the hybrid UD counterpart). One surface of the prismatic specimens (dimensions 240 × 15 × ~3.7 mm³) was painted white and then speckled by black spray paint. The stress was evaluated, as for cyclic tests, using the cross-sectional area of the pristine specimens.

Prismatic specimens have been dedicated to tension-tension fatigue loading. The displacement (strain) controlled cyclic loading was along the 0° fiber direction, by a hydraulic machine MTS 319. The constant displacement amplitude was imposed using a sinusoidal wave-form with a

ratio $R = 0.1$ (ratio of the minimum to the maximum displacement in the cycle) and a frequency of 5 Hz. The strain was estimated by the displacement of the actuator, assuming as the base length the distance between end tabs (140 mm). Several maximum strain levels have been applied close and beyond the pseudo-yield strain ε_p level (Figure 1). One specimen for each strain level has been cyclically loaded up to complete failure or runout after 1 million cycles. Despite the limited number of tests, statistically meaningless, the strain-controlled loadings provided a clear insight into the hybrid laminate fatigue response.

4. Quasi-static tensile behaviour

The preliminary quasi-static tensile tests provided the pseudo-ductile tensile response of the hybrid quasi-isotropic laminate as shown by a typical stress-strain diagram in Figure 1. The three main region of the diagram represents: the linear behavior (first region) whose slope E_0 (72.8 ± 2.9 GPa) is a combination of the stiffness of the LE and HE layers; the pseudo-ductile plateau (second region) with an almost constant stress level, starting at the pseudo-yield strain ε_p ($0.41\% \pm 0.063$), close to the ultimate strain of the LE component, and ending at the strain level ε_{PY} ($1.15\% \pm 0.088$); the almost linear trend of the third region, whose slope E_1 (15.3 ± 3.1 GPa) depends on the stiffness of the intact 0° HE plies and the residual stiffness of the damaged LE ones.

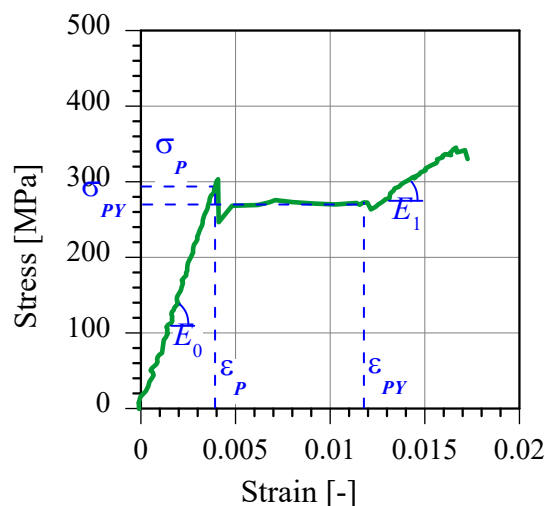


Figure 1. Typical quasi-static tensile pseudo-ductile stress-strain behavior.

5. Tensile-tensile fatigue behaviour

A broad range of maximum strain in the cycles has been considered to cover the quasi-static pseudo-ductility (second region) and post plateau linear behaviour (third region) of the hybrid laminate. Eight different maximum strain levels have been set in the range 0.46% - 1.7% (see Figure 2). The corresponding initial stress levels were close to σ_{PY} for strain levels in the second region and over it for the maximum strains in the third one (Figure 2).

All fatigue specimens did not fail after 1 million cycles, except the one subjected to the highest considered cyclic strain level (1.7%), which had a fatigue life of about 909 thousand cycles.

The peculiar fatigue response of the considered carbon-carbon hybrid quasi-isotropic laminate is observed overlapping the quasi-static stress-strain diagram, the fatigue strain levels and the initial and residual maximum fatigue stress.

The damage pattern created by the first loading cycle is hypothesized to contain different damage modes according to the strain level. The lower strain levels close to ε_p imparted fracture of the 0° LE plies, and transverse cracks in the $\pm 45^\circ$ and 90° plies. Those damage modes have been coupled to different stages of delamination for the strain levels up to ε_{PY} . The fatigue strain levels closer to ε_{PY} developed almost complete delamination leading to weak interactions between the damaged LE plies and the HE ones. This initial damage scenario evolved by further cyclic loading mainly with initiation and propagation of delamination. The damage pattern evolution for strain levels in the pseudo-ductile plateau is reflected on the residual fatigue maximum stress, depicted by green circles in Figure 2. Those residual stress levels fall on a line whose slope is almost that of the quasi-static tensile post plateau region (E_1). It demonstrates that the load at this stage of the fatigue life is mainly carried by the 0° HE plies.

The initial damage patterns of the fatigue strain levels higher than the quasi-static ε_{PY} (>1.2% in Figure 2) resulted in weak coupling of the damaged LE plies and the HE plies. Therefore, the cyclic load carrying capacity was mainly transferred to the 0° HE plies. Then, the further gradual damage imparted to the 0° HE plies during cyclic loading led to the reduction of the residual stress with increased strain level (Figure 2).

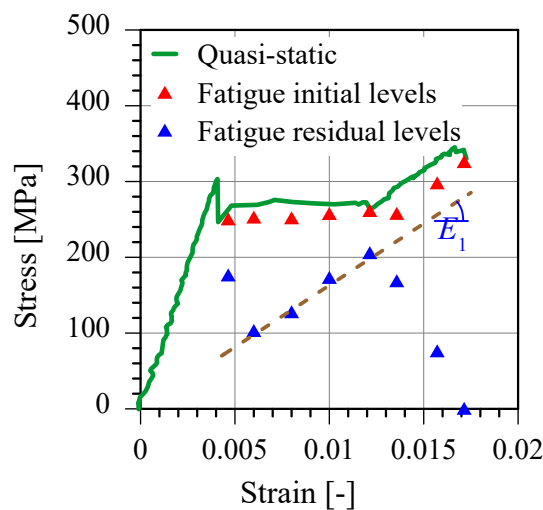


Figure 2. Initial and residual fatigue stress/strain levels compared to typical quasi-static stress-strain behavior.

The damage evolution can be macroscopically estimated considering the stiffness retention as a metric [7,8]. Figure 3 details the stiffness retention with the number of cycles for one strain level very close to the quasi-static ε_p , one strain level almost at mid of the pseudo-ductile plateau and one in the quasi-static post plateau linear region. They highlighted a considerable initial drop, limited to about 10% of the runout threshold. For the remaining cyclic loading, the stiffness kept almost a constant retention whose magnitude depends on the strain level. The initial drop is related to the mentioned fatigue damage development. The initial reduction of stiffness, for the lower strain level close to ε_p , could be connected to the onset of delamination and increase of transverse cracks in the $\pm 45^\circ$ and 90° plies, while the further continuous slight reduction could

be due to the slow propagation of the delamination at different interfaces. The initial stiffness drop of the strain level within the pseudo-ductile plateau could be the effect of the development of the intra- and inter-sub-laminates delamination, which led to a residual stress belonging to the quasi-static linear behavior of slope E_1 (Figure 2).

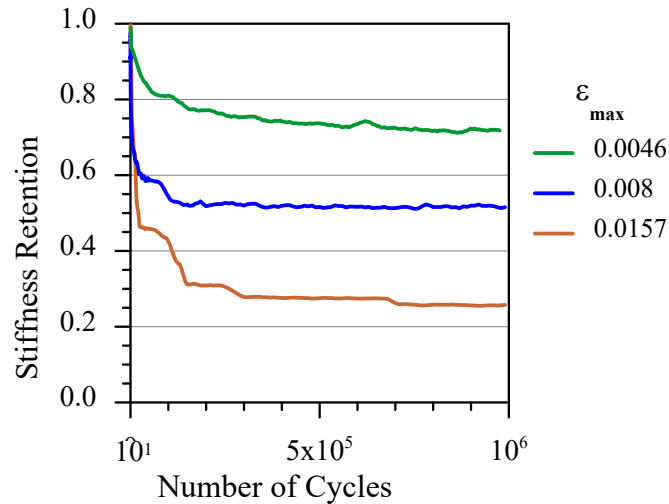


Figure 3. Fatigue tests: stiffness retention evolution during cyclic loading, for three levels of max strain (ϵ_{max}) in the cycle.

6. X-ray micro-CT fatigue damage observations

The evolution of the fatigue damage scenario was observed, to some extent, by X-ray micro-computed tomography (micro-CT). The cyclic loading with the lowest strain level of two specimens has been interrupted after 100 thousand and 1 million cycles (runout). The latter is here described for the sake of brevity. The central volume of the specimens was scanned by a TESCAN UniTOM XL, setting the acquisition parameters: tube potential 40 kV, power 15 W, source-object distance 30 mm, source-detector distance 800 mm, exposure 330 ms, 3000 projections. The resulting voxel size was 5.6 μm .

The damage scenario after 1 million cycles of maximum strain 0.46% is shown in Figure 4. At the initial fatigue stage, several cracks in the -45° central sub-laminates (see white ellipses in Figure 4) and onset of delamination at the $+45^\circ/90^\circ$ interface were pinpointed, which mainly affected the initial loss of stiffness. The former damage mode evolved as delamination at the $-45^\circ/90^\circ$ interfaces, while the latter enlarged covering almost half-width (see orange ellipses in Figure 4). A more extended delamination was generated at the two external 0° sub-laminates, namely: intra-sub-laminate at the LE/HE interface (yellow ellipses) and inter-sub-laminate delamination at the $0^\circ/+45^\circ$ interfaces (brown ellipses). The partial extension of the delamination through the width shows that the load was carried by the still connected plies and sub-laminates, as demonstrated by the residual stress of this strain level in Figure 2. This residual stress level is far from the linear segment representing the quasi-static tensile post plateau behavior. It shows that the LE and HE plies in the 0° sub-laminates still have a partial collaboration and the load is transferred to the 0° sub-laminates by the $\pm 45^\circ$ ones.

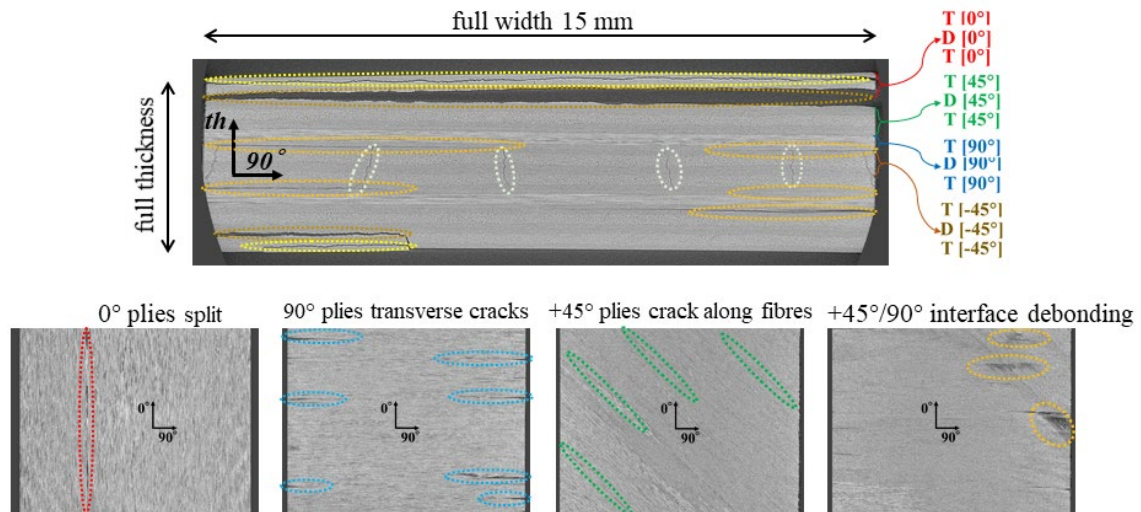


Figure 4. Fatigue tests, maximum strain $\varepsilon_{max}=0.46\%$: X-ray micro-CT observations of the damage, after 1 million cycles, at center of the specimen.

7. Conclusions

This study was devoted to a specific aspect of the mechanical behavior of hybrid laminates, namely fatigue performance. The experimental study focused on the fatigue behavior of a carbon-carbon hybrid quasi-isotropic laminate, which has not been covered in the literature, to the authors' knowledge.

The tension-tension strain-controlled cyclic loading was designed considering the preliminary measurements of the tensile quasi-static pseudo-ductile behavior of the hybrid laminate.

The main achievement of this study was the connection between the macroscopic stiffness evolution, as a metric for the overall damage, and the damage modes observation by X-ray micro-CT. The damage scenario imparted during the early stage of the cyclic lower strain level, involved fracture of 0° LE plies, transverse cracks in $\pm 45^\circ$ plies and onset of delamination at $+45^\circ/90^\circ$ interface, which were responsible of the initial drop of the stiffness retention. Further cyclic loading pointed out the very slow stiffness reduction which was motivated by the evolution of those damage modes as enlargement of the delamination at: the $\pm 45^\circ/90^\circ$ interfaces, the 0° intra-sub-laminate LE/HE interfaces and the inter-sub-laminate 0°/ $+45^\circ$ interfaces.

The measurements and observations of the fatigue damage modes evolution in the studied carbon-carbon hybrid quasi-isotropic laminate are not exhaustive and need further analyses. Nevertheless, the detailed understandings highlighted the effect of pseudo-ductility on the tensile cyclic loading response and are a valuable contribution to the knowledge on the fatigue behavior of hybrid fiber-reinforced plastics, which is needed in a wide range of advanced industrial applications.

Acknowledgements

The work of S. B. Sapozhnikov was supported by the Russian Science Foundation (project No. 18-19-00377) and partially by KU Leuven. The authors are grateful to the Toray Group for providing prepreg materials and for supporting the Toray Chair at KU Leuven, held by S.V. Lomov. The FWO large infrastructure I013518N project is acknowledged for their financial support of the X-ray infrastructure and the KU Leuven XCT Core facility is acknowledged for the 3D image acquisition and quantitative post-processing tools.

8. References

- [1] Swolfs Y, Gorbatiikh L, Verpoest I. Fibre hybridisation in polymer composites: A review. *Compos Part Appl Sci Manuf* 2014;67:181–200. <https://doi.org/10.1016/j.compositesa.2014.08.027>.
- [2] Swolfs Y, Verpoest I, Gorbatiikh L. Recent advances in fibre-hybrid composites: materials selection, opportunities and applications. *Int Mater Rev* 2019;64:181–215. <https://doi.org/10.1080/09506608.2018.1467365>.
- [3] Czél G, Wisnom MR. Demonstration of pseudo-ductility in high performance glass/epoxy composites by hybridisation with thin-ply carbon prepreg. *Compos Part Appl Sci Manuf* 2013;52:23–30. <https://doi.org/10.1016/j.compositesa.2013.04.006>.
- [4] Jalalvand M, Czél G, Wisnom MR. Damage analysis of pseudo-ductile thin-ply UD hybrid composites – A new analytical method. *Compos Part Appl Sci Manuf* 2015;69:83–93. <https://doi.org/10.1016/j.compositesa.2014.11.006>.
- [5] Mesquita F, Swolfs Y, Lomov SV, Gorbatiikh L. Ply fragmentation in unidirectional hybrid composites linked to stochastic fibre behaviour: A dual-scale model. *Compos Sci Technol* 2019;181:107702. <https://doi.org/10.1016/j.compscitech.2019.107702>.
- [6] Swolfs Y. Perspective for Fibre-Hybrid Composites in Wind Energy Applications. *Materials* 2017;10:1281. <https://doi.org/10.3390/ma10111281>.
- [7] Suwarta P, Fotouhi M, Czél G, Longana M, Wisnom MR. Fatigue behaviour of pseudo-ductile unidirectional thin-ply carbon/epoxy-glass/epoxy hybrid composites. *Compos Struct* 2019;224:110996. <https://doi.org/10.1016/j.compstruct.2019.110996>.
- [8] Wu Z, Wang X, Iwashita K, Sasaki T, Hamaguchi Y. Tensile fatigue behaviour of FRP and hybrid FRP sheets. *Compos Part B Eng* 2010;41:396–402. <https://doi.org/10.1016/j.compositesb.2010.02.001>.
- [9] Ribeiro F, Sena-Cruz J, Vassilopoulos AP. Tension-tension fatigue behavior of hybrid glass/carbon and carbon/carbon composites. *Int J Fatigue* 2021;146:106143. <https://doi.org/10.1016/j.ijfatigue.2021.106143>.
- [10] Wu X, Fuller JD, Wisnom MR. An investigation into fatigue behaviour and damage progression in pseudo-ductile thin-ply angle-ply laminates. *Compos Part Appl Sci Manuf* 2021;149:106518. <https://doi.org/10.1016/j.compositesa.2021.106518>.
- [11] Sapozhnikov SB, Swolfs Y, Lomov SV. Pseudo-ductile unidirectional high modulus/high strength carbon fibre hybrids using conventional ply thickness prepreps. *Compos Part B Eng* 2020;198:108213. <https://doi.org/10.1016/j.compositesb.2020.108213>.
- [12] Sapozhnikov SB, Swolfs Y, Lomov SV. Mode I and II interlaminar critical energy release rates in all-carbon interlayer unidirectional fibre-hybrids based on ultrahigh-modulus and high-strength fibres. *Compos Struct* 2020;236:111886. <https://doi.org/10.1016/j.compstruct.2020.111886>.