Development of high performance damage tolerant, damage indicating hybrid composites

Final report of the research programme no. PD 121121 supported by the Hungarian National Research, Development and Innovation Office (NKFIH)

Dr. Gergely Czél

research fellow czel@pt.bme.hu Department of Polymer Engineering Budapest University of Technology and Economics Műegyetem rkp. 3, H-1111 Budapest, Hungary

Introduction

The failure of conventional structural materials (steel and other metals, alloys) has a favourable ductile character. They can undergo large deformation before their final failure, and a given part of this deformation is permanent providing a warning of overload after unloading. High performance composites has been in the spotlight for the last few decades because of their outstanding specific strength (strength/density ratio), but their fundamental limitation is sudden brittle failure. Composites only withstand a few percent of strain before the usually catastrophic failure and they do not provide a detectable sign before final rupture. Ductile or pseudo-ductile behaviour and overload indication capability could enable the high volume application (e.g. transportation and construction industries) and safe operation of composite components. Damage and impact resistant composites can help exploiting the full potential of this unique material family. The materials of high-tech applications may finally penetrate into everyday products after half century.

Hybridisation of reinforcement materials (i.e. fibre types) is capable of improving the damage tolerance of the constituent composites. Thin (30-50 µm) pre-impregnated (prepreg) composite sheets were introduced to the market recently, which made it possible to mimic the ductile character of high strength metals in unidirectional (UD) glass/carbon interlayer (layer-by-layer) hybrid composites under tension. This so called pseudo-ductility refers to the stress-strain response of the new material, not the mechanisms behind, as no yielding and plastic deformation is present in composites made of fibres and thermoset resins, both being brittle. Damage tolerance can however be introduced at a higher structural level by designing the ply-level architecture of the composite to allow for favourable damage mechanisms such as fragmentation and stable delamination of the plies before final failure. Two types of UD pseudo-ductile interlayer hybrid composites were designed and developed recently: one with continuous fibres, where pseudo-ductility is provided by the mismatch in the failure strain of the constituent fibres [1],[2]. The other pseudo-ductile hybrid material consisting of a discontinuous carbon fibre layer sandwiched between continuous glass fibre reinforced layers have a uniquely designed structure with cuts in the carbon layer at pre-defined positions promoting the controlled and stable delamination of the carbon and glass layers before any fibre fracture [3]. Figure 1. shows the pseudo-ductile stress-strain curve of a thin-ply continuous interlayer hybrid composite featuring a pseudo-yield point, a wide plateau at around 1000 MPa stress and a second rising part before final failure. The stain between the pseudo-yield point and the final failure is called the pseudo ductile strain. This is up to 2% in the example and provides a significant safety margin against catastrophic failure which clearly indicates the potential for excellent damage tolerance in practice. Analytical and numerical models were also developed to predict the mechanical behaviour of thin-ply hybrid composites and initiate the preparation of a comprehensive design framework for the new materials [4]-[6].

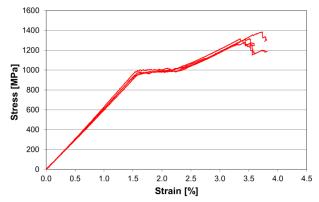


Figure 1. Pseudo-ductile tensile stress-strain response of continuous thin-ply glass/carbon fibre hybrid composite specimens [5]

The overall aim of the research programme was to characterise the previously developed pseudoductile composites under some of the key mechanical loading and environmental conditions. Further steps in the development of the new materials are the improvement of their mechanical performance by modification of the layer interfaces and the investigation of their damage indication capability.

More specific key challenges of the research programme are summarised below:

- Development and tensile characterisation of pseudo-ductile hybrid composites providing high strength in more than one directions
- Investigation of the compression performance of the previously developed (UD) pseudoductile hybrid composites
- Study on the damage sensing functionality of thin-ply hybrid composites
- Study of the effect of temperature and moisture to the damage initiation strain and damage modes of UD pseudo-ductile hybrid composites
- Investigation of the effect of layer interface modification approaches on the performance of damage tolerant hybrid composites
- Exploratory study of the self-healing potential of damage tolerant thin-ply hybrid composites
- Attempt for small scale feasibility studies of the developed and characterised new materials for suitable applications potentially in collaboration with industrial partners

Results

After a literature survey and search for new raw materials, the tensile response of UD carbon/carbon hybrid composites comprising ultra-high modulus (UHM), high modulus (HM), and intermediate modulus (IM) carbon/epoxy layers were investigated in preparation for the more complicated multi-directional carbon/carbon hybrid laminate design. Favourable pseudo-ductile failure behaviour with linear-plateau-linear style stress-strain responses was demonstrated with progressive damage accumulation due to low strain material fragmentation and stable delamination instead of sudden, catastrophic fracture typical of conventional UD composites. All five material combinations exhibited exceptionally high initial moduli and a wide stress plateau with further increase in stress both of which can be exploited as a warning of accumulating damage before final failure. Our findings were published in an international scientific journal paper [7].

Multi-directional interlayer hybrid composites of ultra-high modulus (UHM) and intermediate modulus (IM) carbon/epoxy were designed and characterised both under un-notched and notched conditions based on the previous study. The motivation for this key task was to trigger a break-through in the exploitation of the ductility mechanisms demonstrated in our UD hybrid composites, as multi-directional pseudo-ductile laminates can be suitable for a wide range of applications not being limited to uniaxial load scenarios. The selected material pairs for this study had the highest initial modulus and the widest plateau of all the previously tested UD carbon/carbon hybrids. The design concept of the multi-directional laminates was that a UD three layer hybrid unit can be used as a sub-laminate and rotated to various directions according to a quasi-isotropic (QI) lay-up sequence. Both tested QI un-notched configurations with slightly different constituents exhibited favourable linear-plateau style pseudo-ductile failure mode due to fragmentation of the low strain material in the 0° sub-laminate. Reduced notch sensitivity similar to the ductile net-section behaviour of metals was achieved in both hybrid laminates due to local damage (see fig. 1.) and induced load re-distribution around the notches, for both open holes and sharp notches. The detailed results can be found in an international scientific journal paper [8].

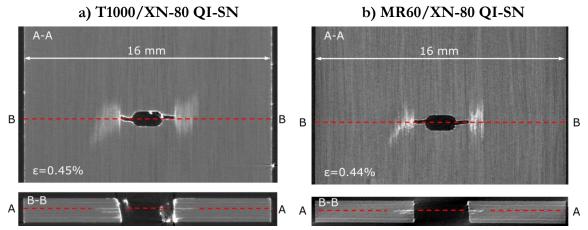


Figure 1. Full width X-ray CT scans of different type notched specimens after tests interrupted before any stress-drop at the overall strains indicated on the scans. Top view scans show the damage in one of the 0° sublaminates. [8]

To widen the scope of the successful pseudo-ductile unidirectional glass/carbon hybrid materials, simple structures were developed to provide the material with high strength and stiffness in two perpendicular directions. These structures are a lot less complicated than the quasi-isotropic plates, but they provide more balanced mechanical properties than UD materials. In the most successful configurations dry, biaxial, high modulus carbon spread-tow tape fabrics were combined with unidirectional S-glass prepreg plies laid up in 0 and 90 degree orientation. This material provided very similar type stable, progressive failure and stress-strain response to those of the UD hybrids, but in two different directions. An MSc thesis was written based on the results of this project.

Previously, around 3 mm thick unidirectional asymmetric glass/carbon-epoxy hybrid specimens were designed and the compressive damage and failure mechanisms of three layer glass/thin carbon hybrid blocks were investigated in four point bending test setup. This new test setup was developed to allow for damage and failure type observations, which are extremely challenging in case of the conventional direct compression test setup where the specimens typically fail catastrophically. The strains were monitored by a novel optical measurement procedure based on the tracked positions of five markers on the edge of the bending specimens and the radii of

circular arcs fitted onto the marker positions. A new failure mechanism was observed which has not been reported in the international scientific literature yet: Some of the investigated thin carbon/epoxy prepregs (i.e. the high and ultra-high modulus ones) have shown stable fragmentation distributed within the volume of the layer. This type of failure has only been shown under tensile loading so far. The typical failure under compression is the global shear instability usually initiated by the micro-buckling of the fibres. The reason for the new failure type was probably the supporting effect of the glass/epoxy layers of higher failure strain adjacent to the thin carbon/epoxy plies. Compressive failure strains significantly higher than those on the manufacturer's datasheets were obtained for all tested carbon/epoxy prepregs. The new observations suggests, that it may be possible to exploit the fragmentation as a key ductility mechanism in compression as well as tension dominated load scenarios and in the variety of corresponding applications. The results are published in an international scientific journal paper [9].

More recently the compressive behaviour of glass/carbon hybrid materials was analysed further and experimental work was executed to understand the damage development and mechanisms in the carbon layer and at the glass/carbon layer interface. Four point bending specimens with carbon layers close to their top surface were deformed in a purpose-designed steel frame (see fig. 2.).

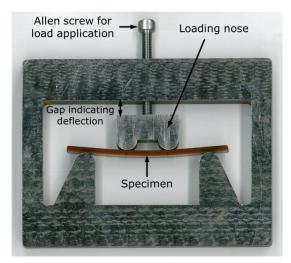


Figure 2. Four point bending frame for microscopy under deformation [10]

The frame was put under optical and scanning electron microscope to analyse the damage modes and the micro-deformations in the hybrid material under compressive deformation. It was confirmed from edge-view mosaic scanning electron micrographs covering an approx. 4 mm long section of the specimen that the high modulus carbon layer was fragmented (see fig. 3.). Close-up images of carbon layer fractures at un-deformed and deformed state revealed that local delaminations developed at the tips of the inclined through thickness cracks of the carbon layer, where the glass/carbon layer interface was in tension (opening mode). Optical micrographs confirmed that the fragments were also sliding along the inclined fracture surfaces when significant deformation was applied to the specimens. The observed micro-deformations were summarised on an explanatory schematic. The results were presented at the 21st International Conference on Composite Materials (ICCM 21) in Xian, China, the leading scientific forum of the field. A full paper was also published in the conference proceeding. [10]

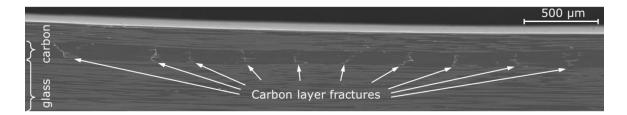


Figure 3. Carbon layer fractures on an edge view scanning electron micrograph [10]

The efforts to characterise the new pseudo-ductile interlayer hybrid materials under compressive loading was continued with direct compression tests of glass-carbon hybrid specimens with different carbon fibres and various carbon-to-glass ratios. Favourable pseudo-ductility was obtained in compression for the lower carbon-glass ratios with high modulus carbon fibres. This is a very important result which demonstrates that the new materials are suitable for reversed loading, not just tension. The results were presented at the prestigious Technical Conference of the American Society for Composites in 2018 September. A full paper is published in the proceeding of the conference [11] and an international journal paper is in preparation.

An experimental campaign was completed to assess the visual strain overload indicating capability of carbon/glass hybrid composites due to the translucent glass layer which can reveal carbon layer fractures and interfacial damage. The results demonstrated that it is possible to use a retrofitted thin sensor patch of about 10x50 mm to indicate if a component (eg. a flat carbon/epoxy composite rod) was deformed beyond a pre-defined strain in tension. The key design parameters and the most important factors affecting the accuracy of the sensors were identified. The technology was demonstrated on a bike handlebar (see fig. 4). The results were published in an international journal paper [12].

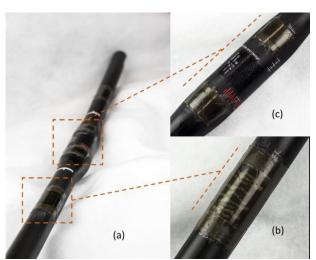


Figure 4. Bike handlebar fitted with hybrid composite overload sensors: (a) MTB Racing flat handlebar, retrofitted with (b) long single layer (c) short single layer sensors [12]

The previously demonstrated hybrid composite visual overload sensor was applied at a larger scale not only as a retrofitted patch on tensile test specimens but as a multifunctional structural sensing layer on a 600x300 mm pseudo-ductile sandwich panel as well as on a full scale longboard (up to 1 m long). These components are large enough to represent small-scale feasibility studies with realistic load scenarios. The structural sensing layer of the sandwich panel successfully gave warning of bending overload (see fig. 5.) at the beginning of a benign, progressive damage accumulation process well before the final failure of the sandwich beam

specimens. The feasibility studies demonstrated that the developed materials and overload sensing technology can be applied to safety critical components of medium size. An MSc thesis was written based on the results of this project.

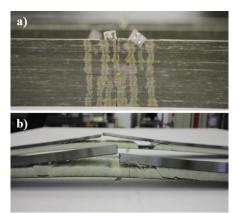


Figure 5. a) activated overload sensor, b) post mortem image of the sandwich beam specimens

A separate project to assess the fatigue response of pseudo-ductile UD glass/carbon hybrids was completed by testing up to 20 specimens in tension with a stress ratio of 0.1. The key conclusions are the following: The non-damaged new materials can be loaded up to the 80% of their pseudo-yield stress without fatigue damage accumulation. The pre-damaged specimens also fail gradually after delamination growth during thousands of cycles. The damage accumulation in the specimens can be monitored by the optical assessment of the delamination area between the carbon and glass layers (see fig. 6). The results were published in an international journal paper [13].

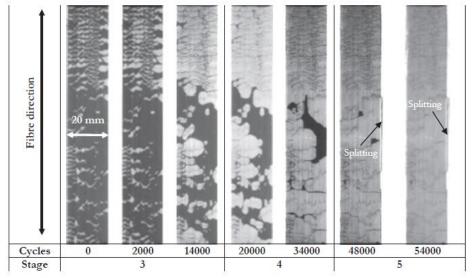


Fig. 6. Typical fatigue damage development of an overloaded UD thin-ply hybrid composite cycled at 70% of the damage initiation stress [13]

The effect of different environmental conditions on the mechanical properties is a key topic when it comes to introducing new materials to safety-critical applications. Therefore a comprehensive test campaign was executed to characterise the new pseudo-ductile glass/carbon interlayer hybrid composites at different temperatures (-50, 25 and 80°C) and to explore the effect of moisture on the failure strain and ductility of the materials. It was found that the temperature has minor effect on the fibre fragmentation dominated pseudo-yield strain of the

hybrids, however up to 60% difference was observed in the mode II interlaminar fracture toughness values measured at the two extreme temperatures. This proves that the effect of temperature on the ply fragmentation mechanism is negligible, but the material is sensitive to the change of interlaminar toughness with temperature, which has to be considered during the component design phase. The results of the moisture sensitivity study indicated that long-term immersion of the specimens in 60°C deionised water has detrimental effect to the strength of the specimens, therefore a less aggressive ageing procedure was implemented at 60°C with 90% relative humidity. It was concluded, that wet conditioning changed the damage mode of the hybrid specimens from pseudo-ductile to catastrophic failure, because the glass/epoxy layers lost strength due to exposure to humidity. Wet conditioning decreased the mode II fracture toughness at all test temperatures. The results of the environmental effect study were presented at ECCM18 and ICCM 22 conferences [14,15].

A promising project was initiated towards the end of the programme to improve the mechanical performance of the developed glass/carbon interlayer hybrid composites by increasing the mode II fracture toughness of the layer interfaces. This way the proportion of the carbon can be increased in the hybrid generating higher initial modulus without the risk of delamination at the first fracture of the carbon layer. Different materials (epoxy resin film, electrospun PAN nanofibers, PS and ABS films) were used to toughen the layer interfaces. So far the very thin nanofibre mats showed the best results improving the mode II fracture toughness of the layer interfaces by up to 30% without significantly increasing the overall thickness of the specimens. We are still looking for even more suitable thermoplastic films, which may also be used for repairing the specimens after damage accumulation. A BSc thesis was prepared based on the results of this project.

An exploratory study was initiated recently to assess the possibility to add repairability as a new function to pseudo-ductile hybrid composites. So far only one configuration was tested with a 35 μ m ABS film between the glass/epoxy and the periodically cut carbon/epoxy layer, but the bonding of the interlayer film was insufficient for good load transfer between the glass and carbon fibre reinforced composite layers. However the repairing cycle when the ABS film was re-melted and bonded again to the composite layers seemed to be feasible as the delaminations were no longer visible. These promising preliminary results provide a solid basis for further investigation with more suitable thermoplastic films.

References

- [1] Czél G., Wisnom M.R., Demonstration of pseudo-ductility in high performance glassepoxy composites by hybridisation with thin-ply carbon prepreg. Composites Part A: Applied Science and Manufacturing. 52 (2013);23-30.
- [2] Czél G., Meisam J., Wisnom M.R.: Design and characterisation of advanced pseudoductile unidirectional thin-ply carbon/epoxy–glass/epoxy hybrid composites. Composite Structures. 143 (2016);362-370.
- [3] Czél G., Meisam J., Wisnom M.R.: Demonstration of pseudo-ductility in unidirectional hybrid composites made of discontinuous carbon/epoxy and continuous glass/epoxy plies. Composites Part A: Applied Science and Manufacturing. 72 (2015);75-84.
- [4] Jalalvand M., Czél G., Wisnom M.R.: Numerical modelling of the damage modes in UD thin carbon/glass hybrid laminates. Composites Science and Technology. 94 (2014);39– 47.
- [5] Jalalvand M., Czél G., Wisnom M.R.: Damage analysis of pseudo-ductile thin-ply UD hybrid composites – A new analytical method. Composites Part A: Applied Science and Manufacturing. 69 (2015);83–93.

- [6] Jalalvand M., Czél G., Wisnom M.R.: Parametric study of failure mechanisms and optimal configurations of pseudo-ductile thin-ply UD hybrid composites. Composites Part A: Applied Science and Manufacturing. 74 (2015);123-131.
- [7] Czél G., Jalalvand M., Wisnom M. R., Czigány T.: Design and characterisation of high performance, pseudo-ductile all-carbon/epoxy unidirectional hybrid composites. Composites Part B: Engineering, 111 (2017);348-356.
- [8] Gergely Czél, Tamas Rev, Meisam Jalalvand, Mohamad Fotouhi, Marco L. Longana, Oliver J. Nixon-Pearson, Michael R. Wisnom: Pseudo-ductility and reduced notch sensitivity in multi-directional all-carbon/epoxy thin-ply hybrid composites. Composites Part A: Applied Science and Manufacturing. 104(2018);151-164.
- [9] Gergely Czél, Meisam Jalalvand, Michael R. Wisnom: Hybrid specimens eliminating stress concentrations in tensile and compressive testing of unidirectional composites. Composites Part A: Applied Science and Manufacturing. 91(2016);436–447.
- [10] Gergely Czél, Putu Suwarta, Meisam Jalalvand, Michael R. Wisnom: Investigation of the compression performance and failure mechanism of pseudo-ductile thin-ply hybrid composites. In the proceedings of ICCM 21 conference 20-25 August 2017, Xian, China. pp. 1-8.
- [11] Putu Suwarta, Gergely Czél, Mohamad Fotouhi, Jakub Rycerz, Michael R. Wisnom: Pseudo-ductility of unidirectional thin ply hybrid composites in longitudinal compression, Proceedings of the American Society for Composites— 33. Technical Conference, September 24-26, 2018. Seattle USA pp. 1-10.
- [12] Tamas Rev, Meisam Jalalvand, Jonathan Fuller, Michael R. Wisnom, Gergely Czél: A simple and robust approach for visual overload indication - UD thin-ply hybrid composite sensors. Composites Part A: Applied Science and Manufacturing. 121(2019);376–385.
- [13] Putu Suwarta, Mohamad Fotouhi, Gergely Czel, Marco Longana, Michael R. Wisnom: Fatigue behaviour of pseudo-ductile unidirectional thin-ply carbon/epoxy-glass/epoxy hybrid composites, Composite structures 224(2019);110996
- [14] Gergely Czél, Márton Bugár-Mészáros, Michael R. Wisnom: The effect of temperature on the pseudo-ductility of thin-ply hybrid composites, Proceedings of the 18th European Conference on Composite materials, ECCM18. Athens, Greece, 2018.06.24-.06.26. pp. 1-8.
- [15] Gergely Czél, Márton Bugár-Mészáros, Michael R. Wisnom: The combined effect of moisture and temperature on the pseudo-ductility of thin-ply hybrid composites. 22. International Conference on Composite Materials (ICCM22), Melbourne, Australia, 2019