DAMAGE-TOLERANT NACRE-INSPIRED CFRP

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Abstract

In this paper, a carbon/epoxy composite with nacre-inspired micro-structure is designed, synthesised and tested. A nacre-like discontinuous micro-structure of interlocking tiles is designed via original analytical and numerical models, and then laser-engraved in the laminate plies. Firstly, three-point bending (3PB) tests are carried out on the nacre-like carbon/epoxy composite, demonstrating its capability in deflecting cracks and avoiding localised failure in the specimen, with some amount of damage diffusion. Subsequently, the laminate interfaces are toughened by film-casting thin (0.3 μ m) patches of poly(lactic acid) (PLA), in order to promote a more extensive pull-out of tiles and increase the damage-diffusion capability of the material. Finally, continuous layers of glass-fibre/epoxy, similar to the thick protein interlayers that separate layers of ceramic tiles in real nacre, are introduced in the laminate, in order to act as crack stoppers in case of unstable crack propagation in the micro-structure.

1. Introduction

Carbon-Fibre Reinforced Polymers (CFRP) are being increasingly used in the aerospace and automotive industry, due to their high strength-to-weight and stiffness-to-weight ratios, with respect to steel and aluminium alloys. However, their inherent brittleness is still a significant limitation to further extending the use of these composite materials.

In recent years, biomimetics has emerged as a promising strategy to develop new composites with enhanced properties [1], by replicating the toughening mechanisms commonly found in natural composites. Among the latter, nacre is one that provides a remarkable balance between strength and toughness. Nacre, found in the inner layer of many seashells, has a discontinuous 'brick-and-mortar' micro-structure (Figure 1), with hard aragonite (ceramic) interlocking tiles embedded in a soft protein matrix. With this micro-structure, nacre is up to 30 times tougher than the ceramic phase it is made up of (and which accounts for 95% of the overall weight). Among the toughening mechanisms of nacre, crack deflection, crack bridging and damage diffusion are the prominent ones, leading reasearchers to reproduce them in synthetic composites using a variety of materials [2].

Additionally, nacre features a hierarchical structure [3], with mesolayers of aragonite tiles (~300 μ m in thickness) separated by organic interlayers of protein, as shown in Figure 1. These softer interlayers, thicker than the biopolymer film that glues tiles in mesolayers (~20 μ m vs ~20 nm) serve as crack-arrest features, trapping cracks before they spread to the next nacre mesolayer.

In this paper, a CFRP composite with nacre-inspired micro-structure is designed and prototyped, in order to exploit its potential for crack deflection and damage diffusion, and to investigate the role of continuous interlayers for crack arrest.



Figure 1. Micro-structure of nacre, with detail of nacre mesolayers (layers of aragonite tiles) separated by thicker organic interlayers. Adapted from Ref. [1].

2. Design of micro-structure

A nacre-like carbon/epoxy laminate is designed as shown in Figure 2(a). In each prepreg ply, the carbon fibres are cut in order to reproduce an array of interlocking tiles that overlap by half of their length. The tile geometry, shown in Figure 2(b), is defined with suitably developed models [4], aimed at ensuring that tiles do not break during pull-out and at maximising the pull-out energy dissipation.

3. Experimental

3.1 Material

The laminate is manufactured using a Skyflex carbon/epoxy thin-ply prepreg, with a thickness of $22 \mu m$ [5]. The tiled pattern is laser-cut in the prepreg plies prior to layup, as shown in Figure 3.



Figure 2. (a) Tiled micro-structure of the laminate and (b) detail of the tile geometry [4].





3.2 Specimen preparation

Using the laser-cutting method described in §3.1, the nacre-like CFRP laminate is manufactured to reproduce the layup sketched in Figure 2(a). Specimens are prepared for three-point bending (3PB) tests, to be carried out in the SEM. Different types of samples are manufactured, as detailed below.

3.2.1 Nacre-like CFRP with interface texturing

The first group of specimens are aimed at assessing the damage-diffusion capability of the nacre-like material. In addition to the tiled specimen with pure epoxy interfaces (specimen E) shown in Figure 2(a), two more specimens are prepared, with an interface toughened by addition of poly(lactic acid) (PLA) via film-casting [6]: one with a full film of PLA (specimen FF), and one with a pattern of discontinuous fractal patches of PLA deposited on the ply interfaces (specimen FP) [7]. These designs are shown in Figure 4, and lead to a film thickness of \sim 3 µm and \sim 0.3 µm for FF and FP, respectively.



(a) Interface with full film of PLA (FF)

(b) Interface with fractal patches of PLA (FP)

3.2.2 Nacre-like CFRP with continuous GF interlayers

The second group of specimens manufactured and tested in this work is instead aimed at mimicking

Figure 4. Nacre-like CFRP laminate with interfaces toughened by film-casting: (a) full film of PLA and (b) discontinuous fractal patches of PLA [7].

the hierarchical micro-structure of nacre [1], whereby mesolayers of interlocking tiles are separated by soft continuous interlayers of organic protein (Figure 1). In order to simulate the role of softer interlayers for crack arrest in nacre-like CFRP, we use glass-fibre/epoxy (GF) prepreg (HexPly F155, Hexcel [8]), interleaved within the CF nacre-like layup as shown in Figure 5(a). Three layup types are considered (1M, 2M, 4M – where 'M' indicates the number of CF mesolayers), as in Figure 5(b): for all of them, the outer GF skin is intended to trigger unstable crack propagation in the nacre-like mesolayers, whereas the internal GF interlayers are intended to act as crack stoppers.



Figure 5. (a) Micro-structure obtained by alternating mesolayers of nacre-like CF plies with GF interlayers, and (b) architecture of the three layup types designed and tested.

3.2 Test method

Both sets of specimens are tested in 3PB in the SEM, using a micro-tester with a 20 mm span, a loading rate of 0.1 mm/min and a load/displacement acquisition time of 200 ms. The tests are run discontinuously, stopping the motor at equal load intervals in order to allow for SEM image capturing.

4. Results and discussion

4.1 Nacre-like CFRP with interface texturing: damage diffusion

Figure 6 includes SEM images of failure of specimens E, FF and FP in 3PB, showing that for the three types of interface failure starts in the outer tensile region and propagates in a stable fashion. Specimen E is able to achieve large crack deflection and crack bridging, with some limited amount of damage diffusion, as visible in Figure 6(b). The latter is substantially greater for the PLA-textured specimens (FF and FP, Figure 6(d,f)).

Specimen FP exhibits the most diffuse pull-out of tiles, with damage spreading in large volumes of the material. This is likely related to the increased ductility of the interlaminar region (+80% Mode I and +12% Mode II interlaminar toughness compared with the pure epoxy interface [6]) and to the heterogeneity of the interface, which has been shown to provide a significant toughening effect [7,9].



(a) specimen E



(c) specimen FF



(e) specimen FP



4.2 Nacre-like CFRP with continuous GF interlayers: crack-arresting mechanisms

For all of the three specimens with GF interlayers (Figure 5(b)), during initial loading, the sliding of tiles is obstructed by the contiguous GF skin, leading to the formation of micro-cracks in the carbon-fibre layers. Failure then starts with tensile failure of the outer GF skin, which then triggers unstable crack propagation in the contiguous nacre-like mesolayer, as shown in Figure 7.

For layup 1M, which has a single mesolayer of CF nacre with no GF interlayers, the unstable crack propagation is characterised by extensive fracture of the CF tiles, with very limited pull-outs (Figure 7(b)). Layup 2M, with its internal GF layer, provides an obstacle to the unstable crack propagation, as detailed in Figure 7(d).

Layup 4M instead, with three GF interlayers, provides multiple crack-arrest features to the unstable



(b) specimen E, detail of failure region



(d) specimen FF, detail of failure region



(f) specimen FP, detail of failure region

crack propagation. Furthermore, as shown in Figure 7(f), the multiple CF/GF transitions determine a progressive change in failure mode of the nacre-like tiles, from brittle tile fracture in the outermost mesolayer to a more ductile tile pull-out in the inner mesolayers.



(a) specimen 1M



(c) specimen 2M



(e) specimen 4M



(b) specimen 1M, detail of failure region



(d) specimen 2M, detail of failure region



(f) specimen 4M, detail of failure region

Figure 7. SEM images showing failure of specimens 1M, 2M and 4M under 3PB loading.

5. Conclusions

The work presented herein demonstrated that nacre-inspired micro-structures can be successfully synthesised in CFRP, leading to failure mechanisms characterised by large crack deflection and damage diffusion. In particular, it can be concluded that:

• a manufacturing technique has been developed to laser-cut µm-sized nacre-like interlocking

tiles in carbon/epoxy prepreg;

- the designed nacre-like CFRP succeeds in deflecting cracks and spreading damage under bending loading, with the damage-diffusion capability being significantly enhanced by adding a thin $(0.3 \ \mu\text{m})$ texture of fractal patches of PLA deposited via film-casting;
- the hierarchical micro-structure of nacre has been reproduced in CFRP by interleaving tiled CF mesolayers with continuous GF/epoxy prepreg interlayers, thus simulating the thick organic interlayers that in natural nacre separate mesolayers of ceramic tiles;
- such GF interlayers succed in arresting catastrophic crack propagation in the nacre-like mesolayers, additionally promoting a transition in failure mode from brittle tile fracture to damage-tolerant tile pull-out.

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