CHARACTERISATION OF THE EFFECT OF THROUGH-THICKNESS REINFORCEMENT ON IMPACT TOLERANCE AND OUT-OF-PLANE TENSILE PROPERTIES OF COMPOSITE MATERIALS

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Abstract

The effectiveness of through-thickness reinforcement at improving impact performance and out-ofplane tensile strength of fibre reinforced composites has been investigated. Two types of carbon fibre/benzoxazine composites were manufactured and characterised; one comprising a threedimensional (3D) orthogonal fabric preform and the other a two-dimensional (2D) bidirectional 0°/90° non-crimp fabric. The materials were subjected to 6 J/mm of impact energy and damage areas were measured for damage resistance by a combination of penetrant-enhanced x-radiography and image processing. The impacted samples were thereafter tested to failure in compression to determine their residual compressive strength as a measure of damage tolerance. Lastly, a non-standard test method was employed to quantify tensile strength in the out-of-plane direction. The 3D-woven composite was observed to have higher damage resistance and damage tolerance. Furthermore, as expected, preliminary out-of-plane tensile tests showed a marked increase in tensile strength for the 3D-woven composite.

1. Introduction

Traditional laminated polymer composites have gained significant interest and widespread usage in structural applications across many sectors. This is because they have been found to possess exceptional characteristics over conventional materials such as high specific strength and stiffness properties and excellent resistance to aggressive chemicals and environments. Composites for aerospace and other high-performance applications are generally fabricated from individual layers of unidirectional fibres built up in a ply-by-ply manner, where each ply may be strategically oriented so as to tailor the material's load-bearing characteristics to its end-use.

Despite having excellent in-plane properties, laminates exhibit low interlaminar strength and are highly susceptible to damage following out-of-plane loading. This is predominantly due to the absence of reinforcement in the out-of-plane direction; loads acting in this direction are borne solely by the fibre-matrix interface which is relatively weak and as such, is easily damaged. Additionally, the inherent heterogeneity and anisotropy of laminated structures result in a mismatch of bending characteristics between individual plies; as such, matrix cracks may initiate and subsequently propagate through the thickness of an impacted component in a radial manner. Delamination (i.e., separation of adjacent plies) develops concurrently with matrix damage. At locations where the tensile stresses along reinforcing fibres exceed their tensile strength, they fail in fracture [1].

Delamination is life-limiting and causes a dramatic loss of compressive strength and structural integrity of the material as it causes a redistribution of applied loads and gives rise to zones of high stress concentrations within undamaged plies which in turn initiate delamination with continued

loading [2, 3]. This is a major concern for the design of composites for use as primary structural components because for low energy impacts, the damage may not be easily detected by routine visual inspections. However, the component is often extensively damaged beneath the surface and may potentially worsen to catastrophic failure if retained in service. As such, strategies to improve impact damage resistance and interlaminar properties have been the subject of extensive research over the last number of decades.

Most impact toughening methods involve the modification of resins and also the incorporation of thermoplastic film inter-layers prior to composite fabrication. These techniques have been reported to effectively improve impact resistance of laminates, however, the associated cost and increased cycle time can restrict their widespread application (particularly for large composite structures) [4].

Advances in weaving technology have allowed for the development of enhanced preforms with three characteristic yarn sets (i.e., warp, weft and z-binder). Three-dimensional weaves (3DW) are available in an array of architectures and the binder path and penetration depth may be tuned to further tailor the material to end-use. Idealised representations of some common fabric architectures are shown in Figure 1; these are the layer-to-layer angle interlock and the through-thickness orthogonal.



Figure 1: Idealised fabric architecture of 3D woven fabric; (a) Layer-to-Layer angle interlock (b) and Through-thickness orthogonal.

The purpose of this research is to investigate the effect of through-thickness binders within 3DW on impact damage resistance and out-of-plane tensile behaviour of composite materials. Damage resistance will be evaluated by assessing the extent of damage induced by a medium level impact event and damage tolerance (the ability of a material with a given damage state to retain its structural integrity) will be investigated as a three-part study. A non-standard test has been reported to allow for the determination of out-of-plane tensile strength [5]; this test will be adapted to comparatively quantify tensile strength in the z-direction for the through-thickness reinforced composite and a conventional laminated composite.

2. Materials and Methods

As part of the current investigation, two types of carbon fibre composites were manufactured. These were a 3DW-reinforced benzoxazine composite and a two-dimensional non-crimp fabric (2DNCF)-reinforced benzoxazine composite. The 3DW material was supplied by Axis Composites Ltd., Belfast, as a single-ply orthogonal carbon preform with 3% binder content (6k) and equal proportions of warp and weft (12k). The 2DNCF preform was assembled in house using 12 plies of bidirectional 0/90 NCF carbon (12k Tenax[®] EHTS40 F13 800tex) supplied by Saertex GmbH; this material was supplied with one surface coated by a powder binder and PES stitching. The areal weights were 5280 gsm (3DW) and 559 gsm (2DNCF). A single-component aerospace grade benzoxazine resin was used for the matrix in both architectures; Loctite[®] BZ9130 AERO (Henkel AG & Co., KGaA) has been specially formulated for out-of-autoclave processing of composites for high-service temperature applications.

2.1. Test panel fabrication

The test panels were manufactured using the EADS-patented Vacuum Assisted Process (VAP[®]) technique. As shown in Figure 2, the process involves the use of a VAP[®] membrane (C2003, Trans-Textil GmbH) as a gas-permeable barrier, preventing the outflow of resin whilst allowing for a continuous evacuation of the internal infusion chamber. This process allows for optimised wet-out of the preform during the infusion process as trapped air and gas molecules are readily evacuated over the entire part during infiltration and cure resulting in reduced void content and improved end-product quality.



Figure 2: Schematic of the VAP[®] membrane-assisted infusion method.

For each infusion, resin was degassed in accordance with the manufacturer's recommendations (110°C at 850 mbar absolute; 1.5-2 hours) in a vacuum oven (Memmert VO400). Once degassed, the resin was transferred to a convection oven; the oven, inlet tube and tool were heated and maintained at the infusion temperature (120°C) for the duration of the infusion. Temperatures on all infusion equipment were controlled and monitored by means of proportional-internal-derivative (PID) controllers. For each panel, the preform was placed on the metallic tool and bagged up as shown in Figure 2. The degassed resin was drawn into the dry fibre bed under vacuum through the heated inlet tube. Due to the presence of breather over the VAP[®] membrane, visual flow front monitoring was not possible; as a result, the out-flow of resin from the heated reservoir was monitored as an indication of flow front progression. Upon completion, the inlet tube was clamped and the reservoir was isolated. The tool was raised to 185°C for 2 hours under vacuum to cure the panel.

The manufactured composite panels were subsequently post-cured (232°C for 1 hour). The nominal thicknesses of the 3DW and 2DNCF composite panels were 5.4 mm and 6.4 mm, respectively. Based on these thicknesses, areal weights and the rule of mixtures, the fibre volume fractions (V_f) were estimated to be 55% for the 3DW composite and 57% for the 2DNCF composite.

2.2. Mechanical Characterisation

2.2.1. Drop-Weight Impact and Damage Assessment

For impact testing, test coupons measuring 100 mm x 150 mm were extracted from each panel and tested in accordance with ASTM D7136 [6]. In order to allow for direct comparisons of results from this test, impact energies were determined for each batch of coupons based on thickness, where 6 J per mm of thickness was considered to induce medium level impact damage. The test rig comprised a free-falling cross-head fitted with a piezoelectric force sensor (22 kN capacity model no.: QFG201; Coopers Instruments, USA) and a striker with a hemispherical tip. A data acquisition system (System 6000; Vishay Precision Group, Inc.) was used in conjunction with the force sensor to capture force data during impacts.

Subsequent to the drop-weight impacts, the coupons were evaluated for damage resistance by means of a penetrant-enhanced x-radiography technique (PEXR). For all assessments, a HP Faxitron x-ray scanner (Hewlett Packard Model 4385D) was used in conjunction with an EZ240 digital imager (NTB elektronische Geraete GmbH). A low voltage of 40 kV and 3.5 mA current was used with diiodomethane as the penetrant of choice. Resulting images were processed and assessed using the ImageJ software (developer: National Institutes of Health).

2.2.1. Compressive properties and Damage Tolerance

In-plane compressive strengths were determined from untabled pristine samples by combine loading compression (CLC) in accordance with Procedure A, ASTM D6641 [7]. A torque of 3 Nm was applied to all clamping bolts. Five samples (13 mm x 140 mm) of each material were loaded to failure at a test speed of 1.3 mm/min on a Zwick/Roell hydraulic materials testing machine with a load capacity of 100 kN. These compressive strengths serve as baseline values for damage tolerance evaluations.

Residual compressive strengths were also deteremined in accordance with ASTM D7137 [8] for compression after impact (CAI) testing. Impact damaged samples (five 2DNCF and four 3DW) were installed into a CAI test fixture, afterwhich, a torque of 7Nm was applied on all clamping bolts. Each sample was loaded at a stroke rate of 1.25 mm/min the same test machine as the CLC specimens. Residual compressive strengths and reductions in compressive strengths were taken into consideration for the assessment of damage tolerance.

2.2.1. Out-of-Plane Tensile Strength

Planar composites manufactured by means of infusion techniques are often thin, shell-like components and the determination of out-of-plane tensile (OPT) properties proves challenging due to the relative difficulty faced in transmitting loads in the thickness direction. A non-standard test method was adopted from published literature to investigate the through-thickness tensile strength.

Coupons were machined from small (L=20 mm) square pieces and loaded in compression to generate a tensile stress state using a two-part fixture; critical dimensions of the coupons and the test fixture are shown in Figure 3. A length-to-width ratio of 2:1 was selected such that the gauge area measured 100 mm². Steel sections were adhesively bonded to the upper and lower surfaces of each coupon to prevent undue bending.



Figure 3: (a) Drawing showing characteristic dimensions of the tensile coupons; (b) steel sections were adhesively bonded to the upper and lower surfaces of the machined coupons to prevent bending; (c) schematic showing loading configuration.

3. Results and Discussion

3.1 Compressive & Impact Performance

The results of all tests conducted as part of this study will be presented in this section. A summary of some of these results may be found in Table 1. As previously discussed, the materials in this study were subjected to drop-weight impacts at energies levels in accordance with their thickness; a normalised impact energy level of 6 J/mm was used to facilitate comparison of results. Furthermore, compressive strengths presented in this section are normalised by the fibre volume fraction of each material type.

Table 1: Summary of results for CLC, impact tests, CAI and PEXR damage evaluation.

	Thickness	Fibre volume fraction	Impact energy	Damage area	Compressive Strength	Residual Compressive Strength
	(mm)	-	(J)	(%)	(MPa)	(MPa)
3DW	5.4	0.55	30	18.3±3.7	580.6±131.8	133.6±8.2
2DNCF	6.4	0.57	36	31.3±8.4	604.9 ± 47.6	107.3±10.9

Upon completion of drop-weight impacts, the specimens were visually inspected to determine what damage modes were present; impact side dents were observed on all 2DNCF samples, with combined splits/delamination on the reverse side. The 3DW specimens had splits/cracks on both sides. No perforation was observed and thus, the internal damage states of the scanned coupons were not masked. Delamination was observed to be the most dominant internal damage mode as revealed by PEXR radiographs, these are presented in Figure 4.



Figure 4: PEXR Micrographs showing internal impact damage modes.

The 3DW material was found to have lower internal damage than the 2DNCF composite. The average damage area (expressed as a percentage of the total surface area of the plate) for the 2DNCF and 3DW materials were 31.3 and 18.3, respectively. As a significant amount of energy is absorbed causing binder fibre fracture and transverse matrix cracking within 3DW, interlaminar delamination is effectively contained within the vicinity of the z-binders, this results in a smaller and more

concentrated damage area than observed in the 2DNCF samples. In addition, the load-time traces show that the 2DNCF undergoes a significantly higher extent of delamination upon impact. This is evidenced by the sudden drop in load as shown in Figure 5 for this architecture.



Figure 5: Representative impact load histories of the 2DNCF and 3DW composites.

Damage resistance (impact energy divided by the unit damage area) was found to be 7730 J/m² for the 3DW and 6450 J/m² for the 2DNCF architecture.

To effectively quantify the deterioration of compressive properties, both pristine and damaged samples were tested to failure in CLC and CAI tests, respectively, to determine compressive strengths and residual compressive strengths. The 3DW composite was 4% lower in undamaged compressive strength. This is largely due to tow misalignment in binder yarn crimp and slight distortion of both weft and warp yarns within the its preform as a result of the weaving process. This is in agreement with findings in literature [9, 10, 11]. As is evident in Figure 6, severe damage occurred in the vicinity of the through-thickness reinforcement and exposed a large extent of matrix damage. These matrix rich areas exist at binding sites where large distortions and gaps exist between weft and warp yarns.



Figure 6: Image showing the failure in the vicinity of a binder in a 3DW CLC specimen.

The compressive residual strength of the 3DW material was 25% higher than that of the 2D laminate, this indicates that the 3DW material is not only more damage resistant, but also possesses superior damage tolerance properties.

3.2 Out-of-plane Tensile Properties

The results presented in this section are preliminary and are part of on-going research. All samples were visually inspected after testing to determine their respective modes of failure. All 2DNCF coupons failed by separation along the plane perpendicular to the loading direction. With the exception of two coupons, complete separation was not observed for the 3DW material. All 3DW coupons exhibited a more complex failure mode with the binder remaining intact. All samples were observed to have varying degrees of partial separation/debonding and the formation of matrix cracks in the vicinity of z-binders. For coupons with edge-located binders, the binders were found to become severely displaced and plastically deformed. Images of failed coupons for the 3DW architecture are shown in Figure 7.



Figure 7: Images of failed 3DW coupons with all observed failure modes presented.

Strengths were determined for the 3DW and 2DNCF materials based on a cross sectional area of 100 mm²; these were 7.02 ± 2.74 MPa and 4.59 ± 2.57 MPa, respectively.

The surfaces were inspected after separation to reveal fibre fracture, fibre pull-out and matrix cracks. The tensile stresses generated in the remainder of the 3DW coupons did not exceed the tensile strength of the binder yarns; however, various failure mechanisms were present upon inspection. Delamination cracks initiated and progressed across the coupons but were arrested by the through-thickness yarns.

4. Conclusions

This study was conducted to investigate the effect of through-thickness reinforcement on impact performance and out-of-plane tensile strength of fibre reinforced composites. Two different fabric architectures were evaluated using a number of test methods: CLC, drop-weight impact, CAI, PEXR and OPT testing. Impact damage areas were measured to determine the damage resistance of both materials. It was found that the presence of through-thickness binders within 3D woven composites effectively increases damage resistance following out-of-plane impact events. The percentage damage areas for the 3DW and 2DNCF materials were 18 and 31, respectively.

The compressive strength of the through-thickness reinforced composite was 4% lower than that of the laminate. This is most likely due to the degree of crimp and misalignment in all reinforcing yarns in the 3DW composite. The residual compressive strength of the 3DW material was found to be 25%

higher, this is indicative of superior damage tolerance. The out-of-plane tensile strength of the 3DW material was found to be 53% than that of the 2DNCF.

The incorporation of through-thickness yarn components within 3D woven reinforcements may effectively improve impact performance and out-of-plane tensile strength. As has been reported by several authors and as evidenced by the CLC tests, these improvements come at a cost as a result of lower in-plane performance.

Further investigations of the out-of-plane test method as reported in this paper are on-going with the aim of attaining an acceptable degree of repeatability and thus, yielding more conclusive observations.

References

- [1] A. D. Resnyansky, "The Impact Response of Composite Materials Involved in Helicopter Vulnerability Assessment Literature Review Part 2," Defence Science and Technology Organisation, Edingurgh South Australia, 2006.
- [2] V. Alveraz, C. Bernal and P. Fortini, "The influence of matrix chemical structure on the mode I and II interlaminar fracture toughness of glass-fiber/epoxy composites," *Polymer Composites*, vol. 24, no. 1, pp. 140-148, 2003.
- [3] D. Kreculj and B. Rašuo, "Review of Impact Damages Modelling in Laminated Composite Aircraft Structures," *Tehnički vjesnik*, vol. 20, no. 3, pp. 485-495, 2013.
- [4] L. Tong, A. P. Mouritz and M. K. Bannister, 3D Fibre Reinforced Polymer Composites, 1st ed., Oxford: Elsevier, 2002.
- [5] R. Gerlach, C. R. Siviour, J. Wiegand and N. Petrinic, "In-plane and through-thickness properties, failure modes, damage and delamination in 3D woven carbon fibre composites subjected to impact loading," *Composite Science and Technology*, vol. 72, no. 3, pp. 387-411, 2012.
- [6] ASTM, Standard Test Method for Measuring the Damage Resistance of a Fibre-Reinforced Polymer Matrix Composite to a Drop-Weight Impact Event, West Conshohocken, PA: ASTM International, 2007.
- [7] ASTM, Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture, West Conshohocken, PA: ASTM International, 2009.
- [8] ASTM, Standard Test Method for Compressive Residual Strength Properties of Damaged Polymer Matrix Composite Plates, West Conshohocken, PA: ASTM International, 2007.
- [9] P. Potluri, P. Hogg, M. Arshad, D. Jetavat and P. Jamshidi, "Influence of Fibre Architecture on Impact Damage Tolerance in 3D Woven Composites," *Applied Composite Materials*, vol. 19, pp. 799-812, 2012.
- [10] F. Stig and S. Hallström, "Influence of crimp on 3D-woven fibre reinforced composites," *Composite Structures*, vol. 95, pp. 114-122, 2013.
- [11] Y. Mahadik and S. R. Hallett, "Effect of Fabric Compaction and Yarn Waviness on 3D Woven Composite Compressive Properties," *Composites Part A*, vol. 42, no. 11, pp. 1592-1600, 2011.