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Impact damage and CAI strength of a woven CFRP material with fire retardant properties

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Abstract. This paper presents the interrogation of low velocity impact and compression after impact test results on a woven fibre composite having a fire retardant, syntactic core, two phase epoxy matrix. The results of the study were to be utilized in a decision making process regarding the appropriateness of the material usage in question for a certain aerospace application. The epoxy matrix of the material system had dispersed black-pigmented particles with flame-retarding properties. Impact tests were performed at five impact energy levels. Two different laminate layup configurations were tested. Visual and C-Scan inspection were conducted, in order to observe the extent of the damage in the composite material. Compression tests were performed to study the residual strength after impact. Analytical formulation correlations with the test results presented opportunities for quantifying the interfacial fracture toughness resistance. Micro-graphs of the specimen's cross section were also produced in an effort to observe the fractured sections and characterise the various fracture mechanisms involved. The results exploitation in terms of design decision making are presented.

Keywords: A. Particle reinforcement; B. Impact behaviour; B. Fracture toughness; B. High temperature properties

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1. Introduction

Aerospace structural development had always been driven by new materials that are being developed for performance and function. The material characterization presented in this article was motivated by the consideration of applying a special woven fibre composite material system to a conceptual aircraft vehicle, due to its peculiar fire retardant matrix characteristics. The composite under investigation was to be utilized in a location, where its fire retardant properties presented an opportunity for fulfilling the airworthiness bottle-neck design specifications. Apart from the fire self-extinguishing character that had to be demonstrated for the certification, strength, stiffness and damage tolerance requirements of the material had to be met, therefore assessed. The response of this new material system, due to its peculiar syntactic core matrix, to low velocity impact and compression after impact residual strength was the subject of the below presented investigation. Following, in the literature review section, a short summary of important research findings that are relevant to our investigation are presented. The intention is to draw the boundaries of the technological domain of our work. In section three our research input is exhibited and in section four we presented our contribution which lies in the proposed method of manipulating the results that helped with our design decision making process.

2. Literature review

Woven carbon fibre reinforced plastics (CFRP) have a better drape ability and are able to be morphed into complex double curvature shapes more effectively than conventional aerospace unidirectional (UD) material systems [1]. Although overall laminate stiffness and strength are somewhat lower for woven comparing to UD laminates [2], the former offer greater flexibility for producing highly complex shapes and present opportunities for lowering the manufacturing cost [1]. The material fabric under investigation is shown in Fig.1a while the micro-graph in Fig.1b, depicts a section through the cured laminate. The mechanical properties of the material as provided by the manufacturer [3, 4] were inferior in terms of lamina strength and stiffness (0^o tensile strength approximately at 292 MPa, 0^o tensile stiffness approximately at 38 GPa) as opposed to the more widely used aerospace woven materials [2]. The design decision favoured this material system on the basis of its fire retarding and flame self-extinguishing properties. The inherent inferiority of the material system in terms of laminate strength and stiffness was addressed and overcame in the design process by employing slightly thicker laminated structural components.

The airworthiness design specifications for this vehicle were to follow similar guidelines to [5]. Under those specifications, structural strength and stiffness requirements were met. Damage tolerance had to be demonstrated as well; therefore within the current study the response to low velocity impact loading and compression after impact (CAI) strength of representative test articles of the structural parts were investigated. The major concern during the investigation was the response of the two phase pigmented epoxy matrix material and the synergy of it with the woven carbon fibre weave in order to provide with an acceptable resistance level to impact loading and with adequate strength under compression had an impact event occurred.

On the impact behaviour of unidirectional versus woven CFRP materials

The impact damage imprint of low velocity impact onto woven CFRP laminates via the various damage mechanisms employed to absorb the impact and the effect of these damages upon the structural life of the material [6-8], produce a more favourable result than the one caused upon similar fibre and matrix UD material systems [9-12].

Low velocity impact damage and post-impact strength in composites have been investigated extensively during the last 40 years, especially for the aerospace grade carbon fibre epoxy composites [13-17]. The majority of the experimental research for the predictive capability of resistance to impact damage, damage extends and residual strength after impact was mainly focused and formulated around UD laminate composite materials [18-21]. For the unidirectional composites the damage phenomena and mechanism are well understood and models based on the strength degradation and fracture mechanics have been developed for predicting the damage initiation and propagation.

Analytical prediction of impact damage and post impact performance of woven composite laminated structures is a more difficult task to perform than for UD materials. Fracture mechanisms and failure sequences are documented from observations [6-8] but parametric analytic formulations for predicting the impact performance have not attained yet the maturity level of the unidirectional ones. Impact performance indicators for the laminates tested herein will be presented in the format of experimental observations. Current research effort in terms of prediction is mainly on the improvement of the numerical model efficiency and accuracy in order to develop computer based tools for material selection in structural design. Up-to-date numerical computations consolidate the composite material mechanical and failure properties of either a UD or a woven layer into the properties of a three dimensional finite element generating a mesoscale representation of the laminate. The computational capacity needed to capture the microstructural woven pattern and the assorted individual damage mechanisms during an explicit numerical event is not widely available as of yet.

On the matrix material and inter-laminar interface importance

It was anticipated early during the study that the fire-retardant particles dispersed into the matrix would affect the laminate impact performance. Impact and post impact phenomena are dominated by the inter-laminar fracture toughness properties of the matrix material [22]. Many authors have addressed the issue of assessing and even enhancing the fracture toughness response to impact loading and the subsequent resistance to CAI. For example by using different matrix thermosetting or thermoplastic materials [23] or by applying veils which are other layered materials within the laminates [24, 25] or even by applying metallic materials in the form of titanium pins in the transverse direction [26]. The major concern in our study was the fracture toughness properties of the two phase epoxy material matrix with the interspersed pigments.

On the fracture toughness of woven CFRP materials

Amongst the many material properties and loading parameters influencing the impact damage response of a CFRP laminate, Mode-II fracture toughness (G_{IIC}) plays a fundamental role especially in the process of delamination progression under Mode-II inter-laminar shear. The other important material parameter that influences mostly the CAI strength is Mode-I fracture toughness (G_{IC}) since the delamination progression within layers under compression resembles a crack opening Mode-I fracture process.

It is recognized that the fracture toughness values required for the engineering investigation of delamination propagation in CFRP laminated structures, although matrix dominated [22], they depend on number of other factors such as the type of fibres, fibre volume fraction, manufacturing process, interphase regions between the matrix and the fibre and many more. This being the reason why fracture toughness values are interrogated by testing composite layered specimen and not by using methods that test purely matrix materials. The engineering/scientific community has been successful so far in generating reliable testing procedures to quantify inter-laminar fracture toughness for unidirectional composites under Mode-I [27] and Mode-II [28]. These methods, when employed within the limitations specified, are capable of producing repeatable results with a small scatter. Unfortunately, when woven fabrics are tested to the above specifications, due to the peculiarity provided by the woven fibre architecture to the split surface morphology, run-arrest type of propagation is experienced most of the times rather than slow stable crack propagation [27, 28]. Run-arrest type of crack propagation, induce dynamic effects and the test standards do not address these implications [27, 28]. Other peculiarities that could be experienced while testing woven CFRP materials are the branching of the delamination away from the mid-plane through matrix cracks in off axis plies and the varying toughness measurements due to encountering richer or poorer pocket areas of resin. All these implications generate a much greater scatter in the fracture toughness test results [29-31].

The current standards of fracture toughness testing methods in Mode-I and Mode-II crack opening, assume unidirectional test specimens, thus test results characterize the fracture toughness in the 0/0 inter-laminar interface. Although the above mentioned testing procedures have been applied to other type of specimens with various interface arrangements [31], it can be argued that reliable and widely acceptable testing methods are not available as of today for measuring the toughness values of for example for the 0/45 inter-laminar interface fracture toughness [29].

The final complication of this study was that the woven CFRP material system contained pigments of another substance interspersed within the epoxy matrix. The matrix was practically a two phase substance and delamination was expected wander about in between the matrix phase where cohesive type of failure within the epoxy would be mixed with an adhesive type of failure between the matrix and the pigments.

Summarizing

- Woven CFRP laminates do not exhibit the strength and the stiffness values of UD laminates of a similar fibre-matrix system but they are more damage tolerant in terms of impact loading damage imprint which results in a smaller decrease in the residual compression after impact strength.
- The computational capacity needed to solve finite element explicit numerical simulations to capture the micro-scale failure mechanisms during impact and post

impact events is enormous. Numerical predictive solutions of that kind are not available in the public domain yet.

- Amongst the important material properties influencing the impact and CAI processes are the Mode-I and Mode-II fracture toughness values. These are highly depended from the matrix material. Specific testing procedures for measuring those values for woven fabrics and at various angle ply directions do not exist. Tests for other than unidirectional laminates along the major fibre direction are conducted by slightly violating the region of validated applicability of the existing unidirectional testing methods. During the study an approximate value of Mode-II fracture toughness of the material system was proposed and derived indirectly by using the analytic formulation in [18].
- The main objective of this research was to present the impact damage characteristics and the compression after impact strength of a conceptually applied, fire retardant woven composite laminate.

3. Experimental methods

3.1 Material

VTS243FR/CF3500 [3, 4] is a partially impregnated pre-preg woven composite material manufactured by Cytec. The material system is made of two plies. VTS243FR is a black-pigmented, flame-retarding, epoxy syntactic-core ply. CF3500 is a high strength (12k) woven carbon fibre ply, with a fabric density of 380 g/m^2, twilled in 2 x 2 weave style, Fig.1a. The two plies were expected to infuse into one another during the curing process. The system is capable of initial cure temperatures between 65°C and 150°C. Following post-cure, a glass transition temperature of at least 160°C can be achieved [32]. VTS243FR is self-extinguish when tested to ISO3795/FMVS302 [3].

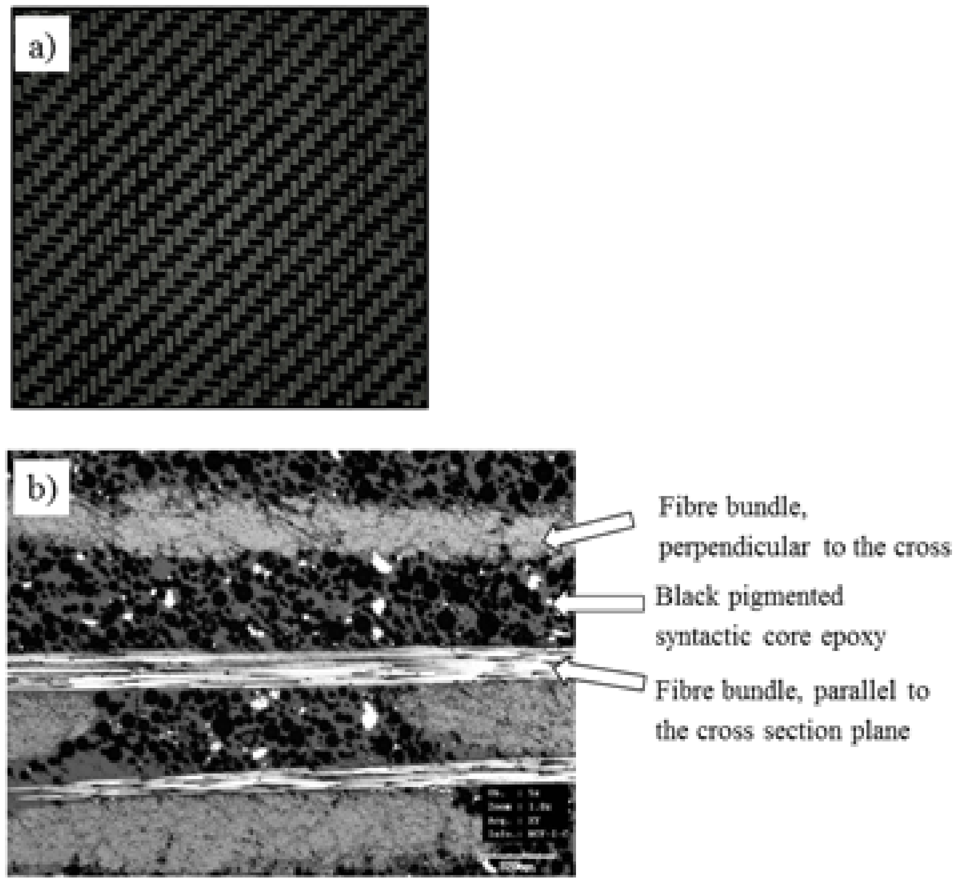


Figure1: a) The 2 x 2 twill weaving pattern of CF3500 woven carbon fibre ply. b) Microscopic image of cross section of cured VTS243FR/CF3500 composite; image scale shown on bottom right: 320 mm

Mechanical properties of cured laminate are lower than that of similar woven composites used in the aerospace industry (0^{o} tensile strength approximately at 292 MPa, 0^{o} tensile stiffness approximately at 38 GPa). Cured ply thickness is about 0.79mm and the density is 1.74kg/m^{3}, [3, 4].

3.2 Specimen

One of the objectives of this study was to investigate the effect of different layup on the damage resistance. Two stacking sequences were fabricated, i.e. a quasi-isotropic layup [+/-, 0/90, -/+, 90/0]s denoted as configuration C1, and [+/-, 0/90, 90/0, 0/90]s, configuration C2. Five specimens for each configuration were produced, 10 specimens overall for impact and CAI testing. The nominal thickness of cured laminate was 6.5 mm.

The material was supplied in a roll form and was stored at -18°C. It was important to thaw the material to room temperature before kitting process takes place for condensation reasons. Thawing process took place overnight at room temperature before the role's packaging bag was opened.

The semi pre-preg was cut into square 340 x 340 mm pieces required for the fabrication of the test specimens. The panels were cured under constant pressure of 627 kPa at elevated temperature of 100 °C for 135 minutes. The temperature increase ramp rate was 0.5 °C per minute and the cooling down rate 1.5 °C per minute. The panels were subsequently post-cured in a pre-heated autoclave for 1 hour at 180 °C to fully develop the material's glass transition temperature. The ramp rate of post curing temperature increase was 0.3 °C per minute and the cooling down rate was 3 °C per minute until 60 °C. After curing, specimens of 100 x 150 mm were cut out of each panel. This dimension is the ASTM standard for impact and compression after impact tests [33, 34].

3.3 Test facilities and procedures

Low velocity impact

The impact test procedure adhered to the guidelines [33]. Prior to impact testing, visual and ultrasonic C-Scan observations were made to ensure that no physical damages or delamination were present. Impact test was performed by using the Rosand Instrumented Falling Weight Impact Tester. The striker used for the impact test was blunt with a hemispherical tip. The total mass of the drop weight was 2.2 kg for all the tests. Time histories of the impact force, velocity, acceleration, deflection and absorbed energy were measured and recorded by a computer controlled processor. Five specimens were tested from each configuration at the impact energy levels of 8, 15, 25, 35 and 50 J. Impacted specimens were inspected by ultrasound C-scanning to measure the delamination shape is according to ASTM D7136 [33].

Compression-after-impact (CAI)

The compression test set up was originally designed by Boeing and was later adopted by ASTM D7137 [34]. The machine used was an Avery 600 kN. Compression loading was induced at a constant head displacement rate of 0.1

mm/min. The load was applied onto the specimens until ultimate failure. The machine was stopped immediately after the specimen failure to allow for the retention of the distortion just before / at failure.

4. Experimental results and discussion

4.1 Impact test

The main focus of this study was to quantify the damage tolerance extends of the fire retardant CFRP material. The synergy of the woven fabric and the matrix was of great importance to the study. Judging from the material mechanical properties published by the manufacturer [3, 4], slightly thicker specimens were designed to counterbalance the slightly inferior mechanical properties benchmarked against other material system candidates. Some of the thickness effects for a different material system were captured in [35]. Amongst the results discussed in [35], a higher peak force is expected for thicker laminates, smaller transverse displacement, increased damage tolerance and shear failure under CAI.

Figure 2 presents images of ultrasonically detected delamination damage for the five C1 and five C2 configuration specimens along the various impact energy levels. The maximum damage diameter and area were defined according to [33]. Configuration C1 had bigger damage areas than those of C2, although the maximum diameter was similar at each energy level. The results were used to construct Fig.5. It was evident that bigger damage was incurred into the quasi-isotropic layup C1 for the same amount of impact energy.

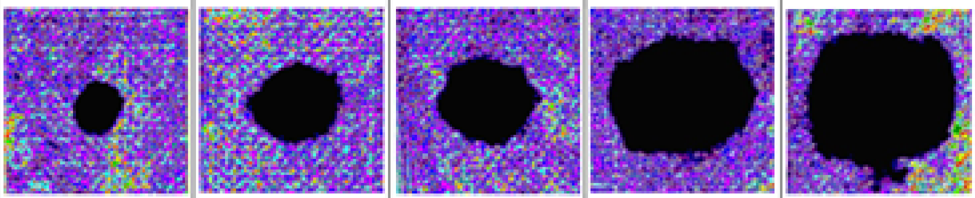
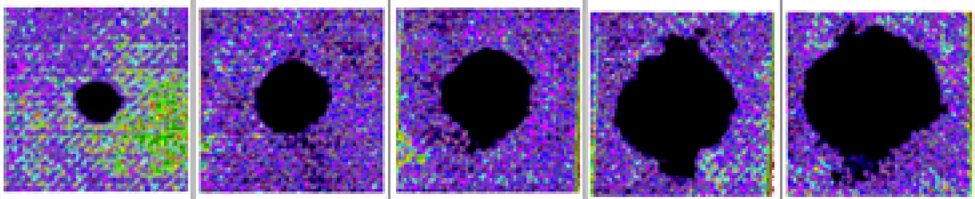
Impact Energy (J)	8	15	25	35	50
Configuration C1 [+/-, 0/90, -/+, 90/0]s					
Max Diameter (mm)	30	57	59	85	89
Area (mm^2)	688 (circular)	2122 (oval)	2346 (oval)	4504 (oval)	5767 (oval)
Configuration C2 [+/-, 0/90, 90/0, 0/90]s					
Max Diameter (mm)	29	45	56	79	87
Area (mm^2)	550 (oval)	1523 (oval)	2028 (diamond)	3904 (diamond)	4288 (diamond)

Figure 2: Images of ultrasound detected delamination area for the 10 impacted specimens of two configurations (C1, C2) at various impact energy levels

Impact force versus time histories is shown in Fig.3. Figure 3a depicts the comparison of the two configurations at four impact energies, indicating that C1 and C2 had virtually the same dynamic response at each energy level. Since the response obtained was very similar, only C1 configuration is further presented in Fig. 3b that depicts all impact energy levels tested in one plot. The quasi-isotropic C1 configuration is stiffer than C2 in terms of transverse deflection. This result was also evident from the stepper initial rise of impact force response versus time shown in Fig.3a. Similarly in Fig.4a, the maximum impact force attained from the C1 configuration is somewhat larger at least for the impact levels of 8 and 15 J. Thus the stiffer in terms of transversal deflection quasi-isotropic layup, resist the impact loading more and a bigger damage was inflicted onto it. Figure 3 also shows that generally the two layup configurations responded similarly apart from the 15 J impact case. At that impact energy level, configuration C2 exhibited a distinctly more compliant character, also captured in Fig.4a.

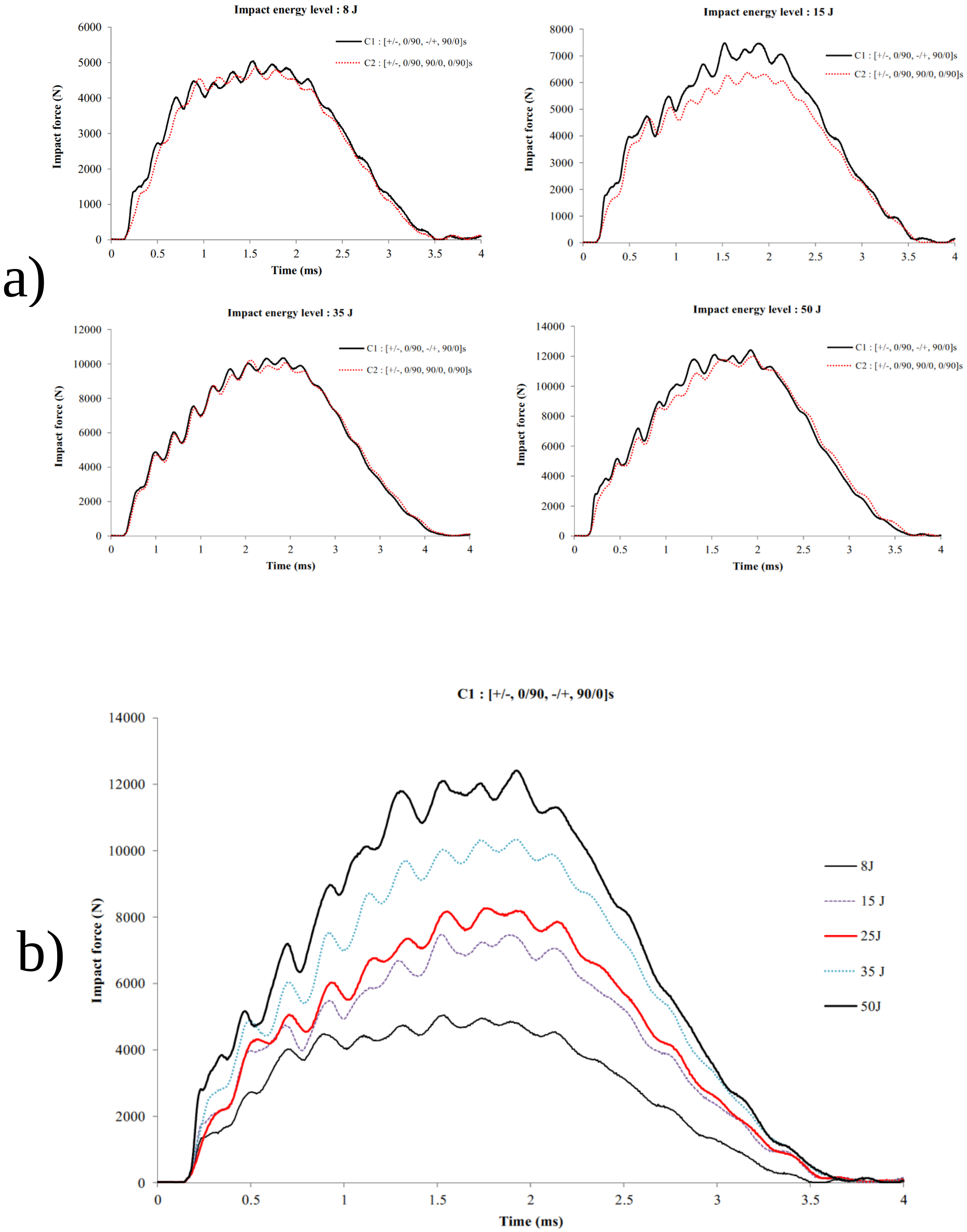
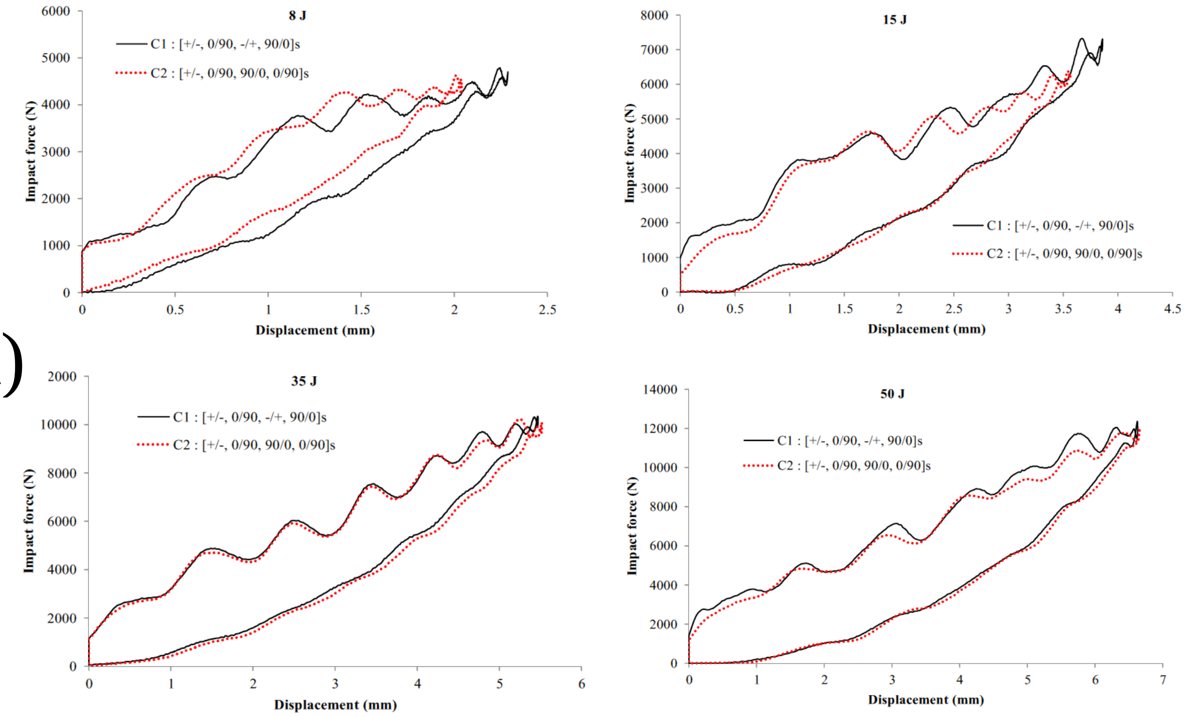


Figure 3: a) Impact force versus time histories for the two layup configurations at four impact energy levels: 8 J, 15 J, 35 J and 50 J. b) Impact force versus time for configuration C1 at various impact levels

a)



b)

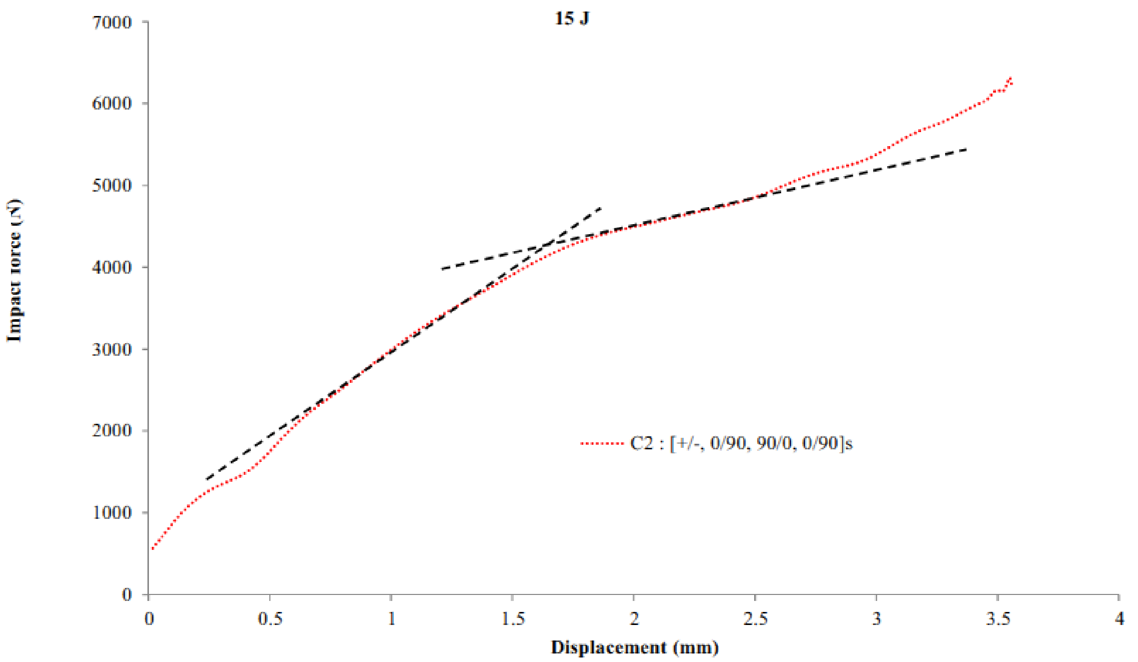


Figure 4: a) Impact force versus impactor displacement for the two layup configurations at four impact energy levels. b) Smoothened impact force versus impactor displacement for identifying the critical impact force

An interesting parameter to be investigated during the impact events is the first load drop in the impact force versus time graphs [22]. This first peak point in the graph indicates damage initiation. In our study, even with filtered impact force versus time results a clear picture providing with the first load drop was not able to be produced. Instead, following the suggestions in [19], the impact force versus deflection diagram was further processed by removing the high frequency components from it. The result of the filtered image is shown in Fig.4b. The change in the tangency indicated the change in the laminate stiffness along the transverse direction, which in turn implied the initiation of damage. The first load drop was found to be approximately at 4.2 – 4.5 kN for both layup configurations. This load is often called as the threshold impact force for delamination onset or the critical impact force and is denoted as P_{crit} [20].

As mentioned earlier, Mode-II fracture toughness (G_{IIC}) is an important parameter, amongst many others, for assessing the resistance to impact damage especially the damage initiation. With woven CFRP materials the derivation of G_{IIC} values from tests is a rather tedious task if not impossible to perform. For UD materials, there is a widely accepted analytical formulation which relates the critical threshold values of P_{cr} to G_{IIC} [18] and is shown below (eq.1):

$$P_{cr} = \sqrt{\frac{8\pi^2 E t^3 G_{IIC}}{9(1-\nu^2)}} \qquad (1)$$

In the above equation, E and v are the equivalent Young's modulus and Poisson's ratio of the quasi-isotropic laminate and t is the thickness of the laminate. Reference [22] suggested for equation (1) to be inversely applied in order to estimate G_{IIC} from the values of P_{cr}. It is also suggested that acceptable results were obtained for G_{IIC} values in the case of UD materials related to actual test results. The value of P_{cr} which depends purely on the matrix material system [22] was observed in Figure 4b to be in the vicinity of 4.2 kN. Following a similar approach and disregarding the rest of the complications of the woven architecture along with the two phase matrix system, an equivalent bulk mode II fracture toughness G_{IIC} was calculated in the range of 300 J/m^2. That result apparently came close to the values presented in [22] for other UD material systems tested which had similar P_{cr} critical threshold values. It needs to be reminded that this bulk fracture toughness quantification, takes into account all the microstructural behaviour that promote or retard mode fracture, meaning the effect of the pigments and the effect of the woven surface architecture. In [29], it is shown that higher G_{IIC} values are expected for a woven CFRP material system as opposed to a UD of the same material properties for the fibres and matrix. Thus for the material in our study the Mode-II inter-laminar fracture toughness G_{IIC}, resembled more the values exhibited by UD epoxy material systems. The decrease in the expected G_{IIC} can be partly attributed to the two phase epoxy matrix.

Since the first load drop occurred at approximately 4.2 kN, damage in the form of delamination exist for all laminates even at the impact level of 8 J. For the higher impact levels as shown in Fig.4a, the response is more or less the same and most probably other damage modes are present besides delamination. Similarly for the 8 J experiments both configurations responded similarly. The only graph which presented some difference was the one at 15 J level. That can be translated as an indication of triggering the shifting from certain damage modes to include others as well, possibly fibre breakage that occurred for configuration C2 but not for C1.

Figure 5 shows the delamination area versus impact energy. Under the same impact energy, the C2 configuration had smaller damage area than that of C1, especially at the higher impact energies of 35-50 J.

Delamination area versus peak impact force is shown in Fig.6. The two configurations had virtually the same response, except at the higher impact force range of 10-12 kN, in which C2 had approximately 20% smaller damage area.

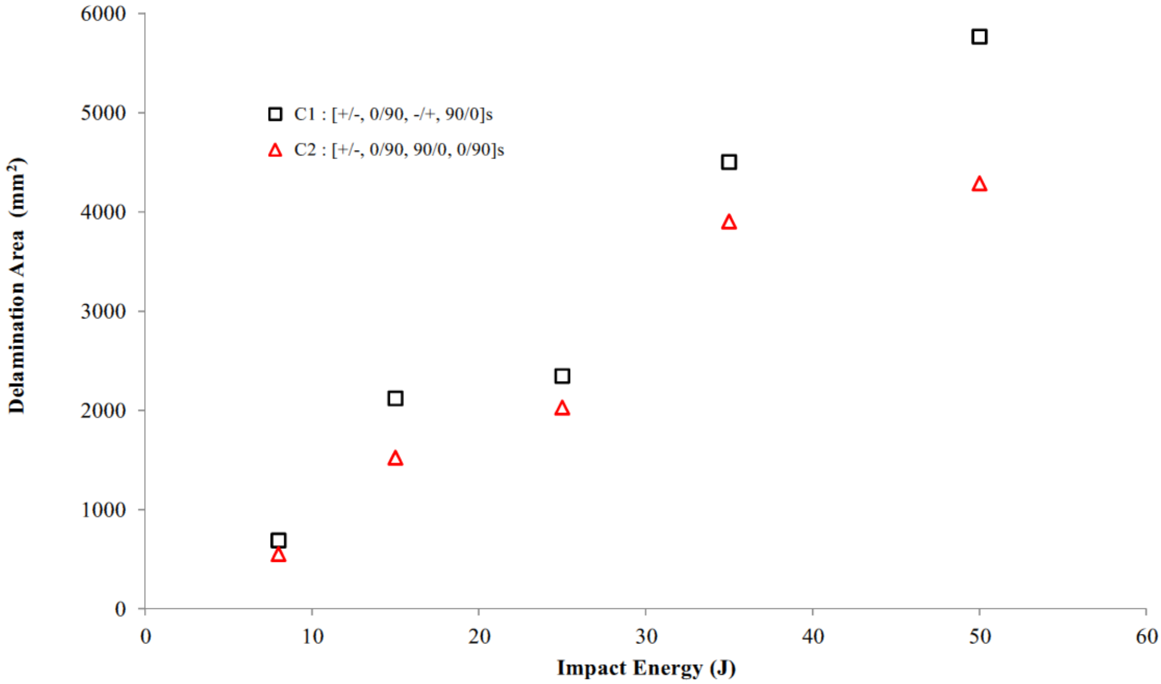


Figure 5: Delamination area vs. impact energy for all specimens

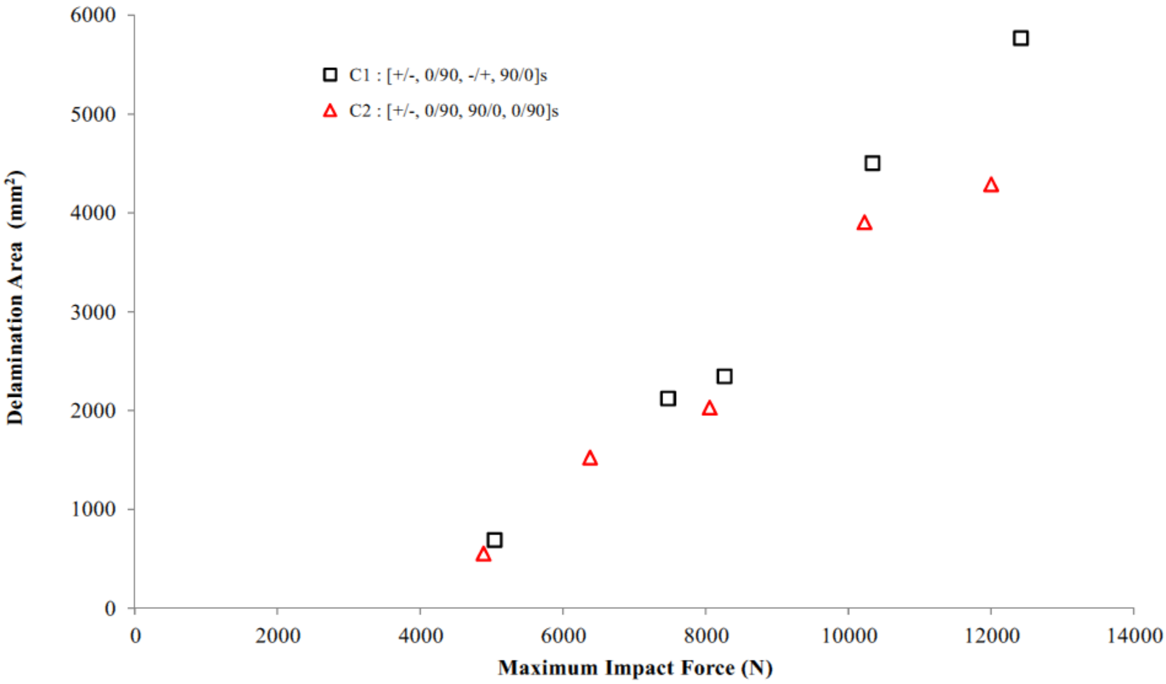


Figure 6: Delamination area vs. maximum impact force for all specimens

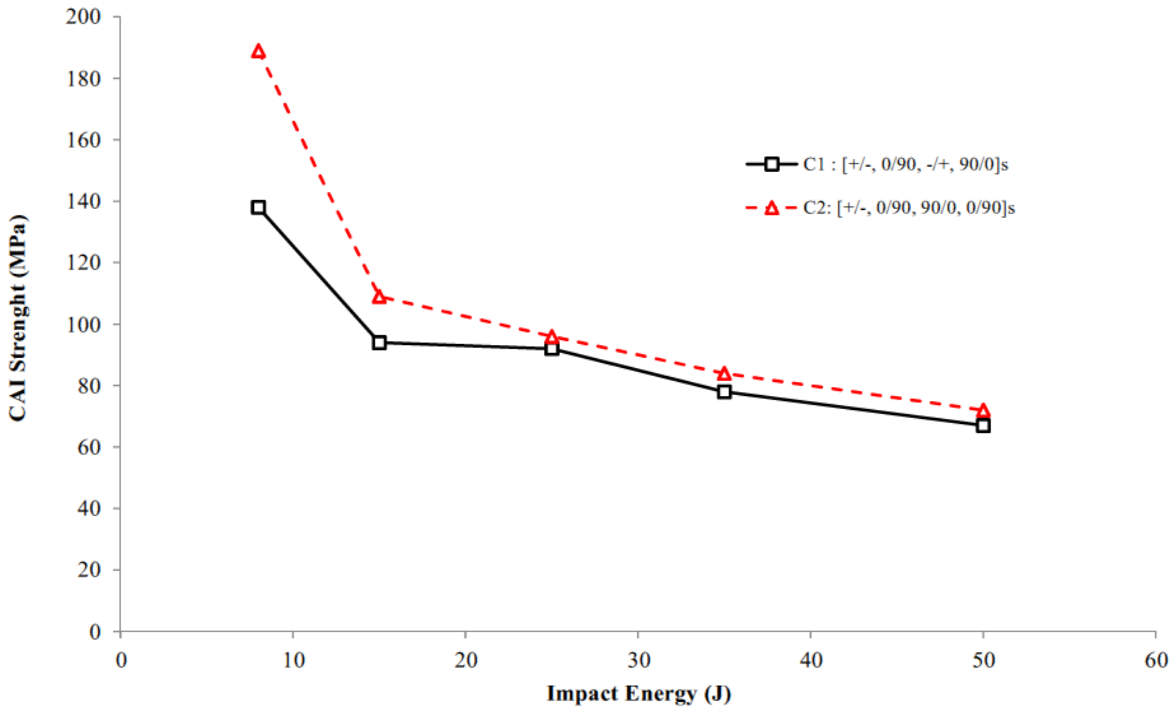


Figure 7: CAI strength vs. impact energy for two layup configurations

.2 Microscopic observation

After the impact events, microscopic pictures were taken to inspect the cross-section of impact damaged specimens. Microscopic samples of 10 x 30 mm size were cut off around the impact zone and potted into resin pool of 35 mm diameter and allowed to be hardened and self-cured overnight. Polishing was performed initially by a manual grinder machine, and followed by an automatic grinder. Two of the most representative pictures are shown in Fig.8.

Microscopic images revealed that the failure mechanism for impact energy levels below 15 J is mainly due to the internal delamination and matrix cracking; an example of low impact energy is illustrated by in Fig.8a for the 8 J impact. When the impact energy was beyond 15 J, more damage modes were observed which confirms the transition region captured in Fig.4a, at least for configuration C2. An example the highest impact energy of 50 J is shown in Fig. 8b showing delamination, matrix cracking, and also significant portion of fibre breakage.

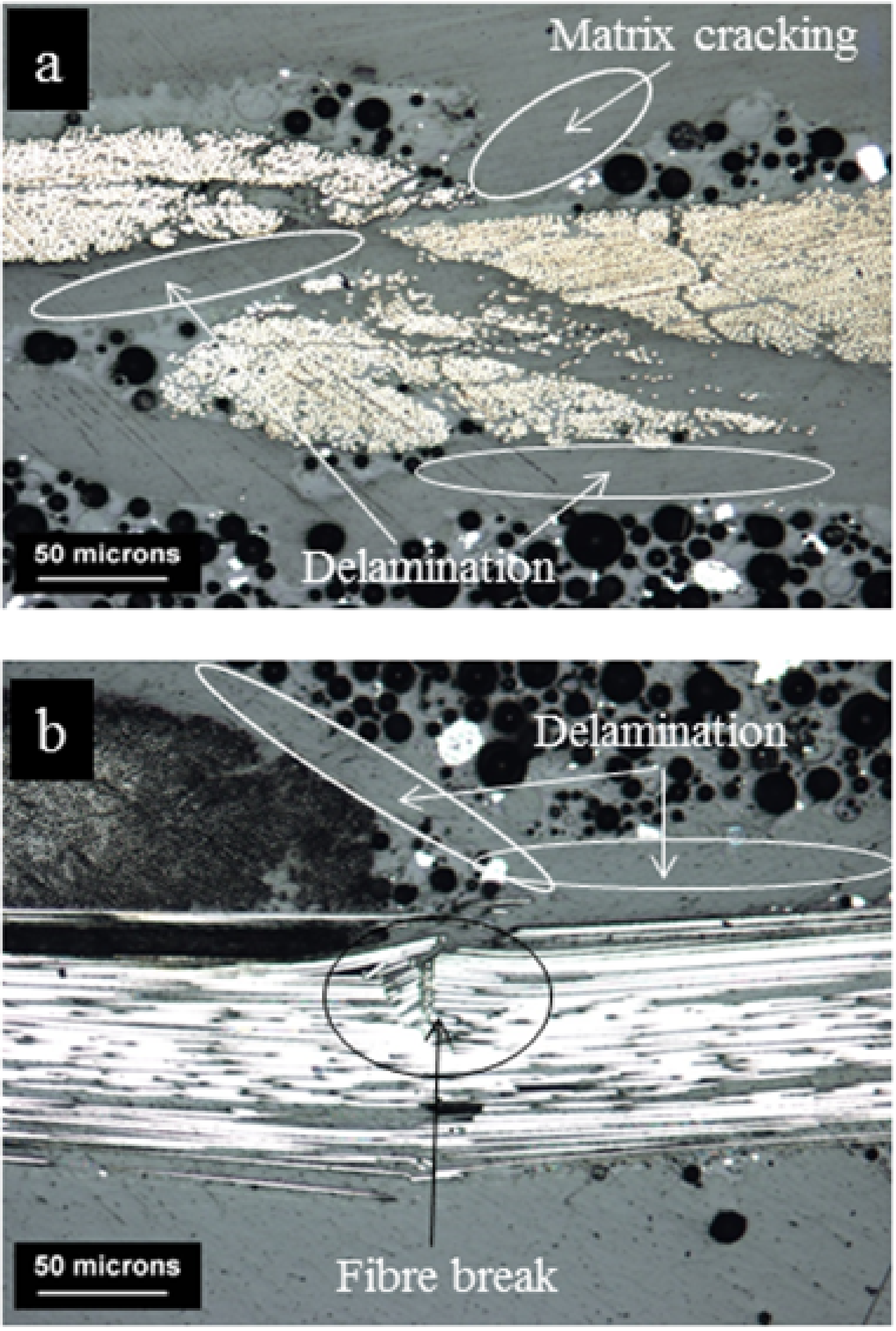


Figure 8: Microscopic photo of the C1 specimen: a) 8 J impact is mainly delamination and matrix cracking. Damage location shown is near the specimen mid thickness. b) 50 J impact revealing multiple damage modes of delamination, matrix cracking, and fibre fracture. Location shown is near the back face of the specimen. Note: grey background is the potting resin

4.3 Compression-after-impact

Figure 7 shows the CAI strength vs. impact energy for the two lay-up configurations. For impacts below 15 J the C1 configuration had lower CAI strength because it had suffered larger impact damage (Fig.5). However, beyond the 20-25 J mark, the CAI strength values of the two configurations were virtually the same despite the C1 specimens having had much larger impact damage area at higher impact energies of 35 J and 50 J (Fig. 5). This sign indicated the change of damage/failure mode under the compressive load for higher impact energy discussed in the previous section in the light of microscopic inspections. The strength of the C2 configuration was expected to be greater along the 0/90 plys since more fibres are aligned along these directions. Performing a rough 10% rule Hart-Smith strength estimation, C2 configuration could potentially exhibit 1.33 times higher strength than configuration C1 under tensile loading. Therefore effect on the decrease in the CAI strength if assumed normalized to the actual un-notched laminate strength is more severe for the C2 configuration.

The impactor head punctured barely visible impact type of damage (BVID) on the laminates at energy levels of 8 and 15 J. Above15 J, the damage was fairly visible (VID).

Figure 9 shows the cross sections of failed specimens after CAI covering the full range of impact energies. Following observations were made:

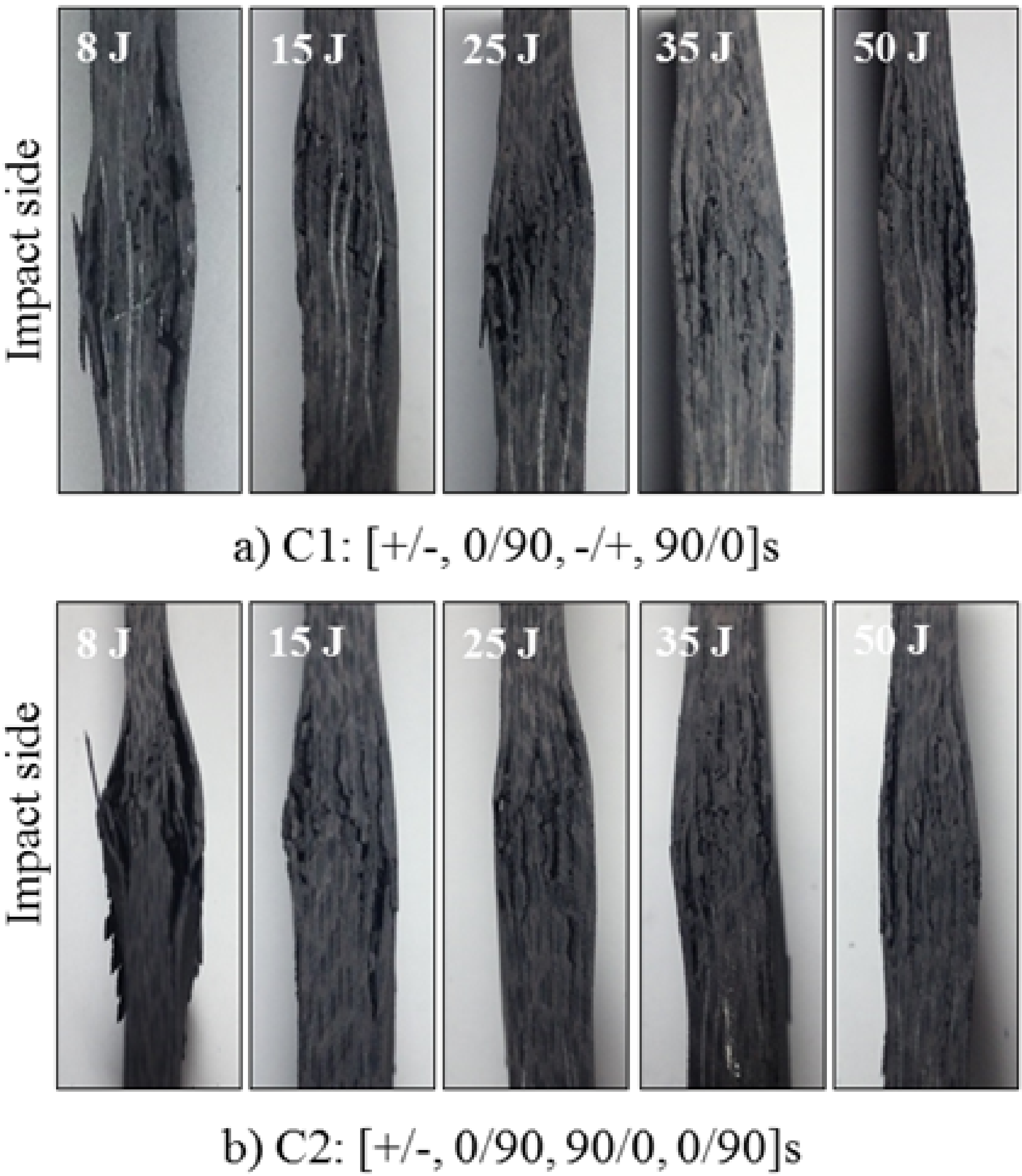


Figure 9: Photos of failed specimens after the CAI tests at various impact energy levels. “Pine tree” shaped fracture pattern clearly visible

- Since the 8 J impact caused the smallest damage area, specimens (both C1 and C2) failed at much higher compressive load in the CAI test comparing to the ones impacted at higher energy levels. The photos of the 8 J impact specimens depicted a clear outer ply mode I delamination and fibre crushing in the main core of the specimen due to the high compressive load.

- Configuration C2 exhibited the outer layer delamination at all impact energy levels, which indicated the weaker interface in terms of mode I fracture toughness for the inter-laminar region of adjacent plys having a 45^o shift in the orientation
- When the impact energy was greater than 8 J, fractured patterns in terms of cracked matrix under shear and broken fibres in a “pine tree” pattern were formed underneath the impactor head. These locations marked the CAI test failure initiation points.

Overall, The laminate CAI strength measured is smaller than most of the commonly used fibre CFRP materials employed currently in the airframe industry [38], where a rather general and rough estimate for impacted laminates with Visible Impact Damage (VID) can average from 200 to 250 MPa in terms of CAI strength levels.

4.4 Design decision

The outcome of the study indicates that C1 configuration was preferred over configuration C2. In general the two layups performed similarly at least above a certain impact energy level. Although the damage imprint was larger for C1, the ratio of the decrease in the residual CAI strength to the original un-notched strength was better. Also the quasi-isotropic arrangement can carry variable direction in-plane loading more efficiently. The reasons for the minor difference in impact and CAI response can be attributed partly to fracture toughness properties and partly to the residual thermal stresses arising from the mismatch of the Coefficient of Thermal Expansion (CTE). The more directional configuration C2 had lower curing induced residual stress in the matrix due to less mismatch of the CTE. The C1 quasi isotropic configuration had more inter-laminar regions interfacing +45/-45 to 0/90 layers. On the other hand, for the inter-laminar regions interfacing layers of the same orientation, fibre tows from one layer sit among the bundles of the adjacent layer, effect which greatly enhances the resistance in shear thus affects the mode II fracture toughness.

5. Conclusions

A new material system has been assessed on its resistance to low velocity impact and in terms of residual strength in post-impact compression. Based on the impact damage size and CAI strength, the test results indicated a design application window for the woven material system for the two selected layup configurations. Two different layup configurations of a woven carbon fibre composite with a fire retardant epoxy matrix were impacted at five energy levels. Impact damage size was measured by ultrasonic C-scan and the subsequent CAI strength was measured by compression load test until specimen failure.

The material system was more complex in microstructure as opposed to a unidirectional one, taking into account the pigmented epoxy matrix and the woven interlaminar surface architecture. Nevertheless, by the use of the manipulated force-displacement diagrams along with the critical load formula originally conceived for the unidirectional materials, a plausible quantification of “equivalent bulk Mode II fracture toughness” can be assumed.

The results obtained indicate the usage limitations for this material system, specifically for the two layup configurations tested. The material may be used in certain applications where a major driver for materials selection for the structural location under consideration would be exposure to flame.

Relating the CAI strength measured by testing to the most commonly used materials in the airframe industry [38], the CFRP material system presented here in would ideally be best utilized in non-critical, non-primarily loaded structural components, whose probable failure during service will not result in the loss of the aircraft.

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