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1. Title: **Water absorption and low-energy impact and their role in the failure of ±45° carbon fibre composites**

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Abstract

Defects in polymer composites associated with in-service damage induced by impact and those produced by environmental degradation due moisture absorption can have a significant influence on the mechanical properties and failure modes of the structures. The present study evaluates the flexural characteristics and failure modes of $\pm 45°$ carbon/epoxy laminates under different conditions. The flexural strength and stiffness of these composites were inspected before and after subjecting to water absorption, drying, re-drying and low energy impact. It was observed that both absorbed water and low energy impact caused significant deformations and fractures which drastically reduced the load carrying ability of the $\pm 45°$ carbon/epoxy laminates. Re-drying slightly improved the properties and reversed the effects of water.

Key words:

Carbon fibre composites, Water absorption, Impact, Flexural properties, Failure mode.

1. Introduction

Composite materials generally are the most advanced and adaptable engineering materials known to man, and the growth in the use of polymers and fibre/polymer composites is due to their outstanding mechanical properties, unique flexibility in design capabilities and ease of fabrication.

One of the significant considerations in designing composite laminates and especially in aircraft structures is impact which can reduce the strength of the material by 50 % [1]. The general issues are impact damage resistance and impact damage tolerance [2]. The impact resistance and tolerance of composite structures have been reported in the literature [3-7]. It has been reported that catastrophic failure can occur when loaded panels are subjected to impact. This phenomenon has been proved by the investigation of impact resistance and tolerance of a range of tensile and compressive preloaded structures [5-9].

Fibre orientation angle is one of the important parameters that affect the impact properties and performance of composites. It was observed by some researchers that the failure under the impact of composites with fibre angles between $(0° < \theta < 90°)$ is mainly brittle inter-fibre cleavage mode with slight or no indication of interlaminar delamination. On the other hand, the failure modes of composites with fibres angle of $(\theta = 0°)$ were fibre fracture, interfacial splitting and layer-to-layer delamination [10-11].

During the service of different engineering structures such as aircraft and vehicles structures, carbon fibre reinforced polymer composites might be subjected to accidental impact damage at high strain rate. These impact damages, which are a major concern, lead to reductions in stiffness and strength, especially under hot/wet environments [12].

The velocity of the impact is commonly divided into low and high velocity impacts. The mechanism of the damage in polymer composites in both cases is complicated and normally involves delamination and intra-laminar damages which in both cases depends on the component's thickness. [13]

Low energy or velocity impact leads to matrix cracking, inter-laminar damage, delamination and debonding between fibres and matrix which might be left undetected [14-16]. While high velocity impacts lead to fragmentation or perforation, which can be detected during maintenance inspections [13]. Low velocity impact may occur from various sources such as tool falling during manufacturing or maintenance operations, hail, debris on the track,

and bird collision. The reduction in the strength of composites can reach up to 30% due to low velocity impact damages. [17]

These undetected damages on the impact surface or BVID (Barely Visible Impact Damage) cannot be generally seen by visual inspection and lead normally to a significant reduction in strength and stiffness [18]. Hot/wet environments normally lead to hygrothermal effects such as plasticization, hydrolysis, interface debonding and micro-cracking [19-23]. These hygrothermal effects and impact damage generally can affect the overall performance of CFRP such as stiffness and strength [24-28].

The invisible damage from low velocity impacts can have a significant effect on the residual strength of composite materials, which is a critical design aspect. Therefore, conventional safety factors have to be applied to the ultimate strength of composite materials. [17]

Generally, it can be very difficult to investigate the mechanical reaction of composite materials under impact loads because the failure occurs simultaneously [29]. Low velocity impact damages are commonly detected by non-destructive ultrasound technique. This technique is a slow, expensive and technically difficult to apply on large parts. Therefore, effective numerical techniques and tools are used to predict the impact damage tolerance of composite materials for both excellent performance and safe design [30]. Numerical modelling techniques can be easily used to investigate the effect of impact damage on the overall mechanical behaviour of simple and complex structures and can predict delamination growth and fibre and matrix failure [13]. Numerical investigations significantly reduced the number of the experimental tests with a considerable reduction in time and cost. [13]

Many researchers using numerical and experimental investigation techniques have studied the influence of impact damages on composite materials. The behaviour of stiffened composite panels was numerically studied, using non-linear explicit FEM analyses, under different impact energy levels. An excellent match between the experimental values and numerical predictions was obtained in terms of impact force, displacement and energy [29, 30]. In another study, the onset and the evolution of low velocity impact damages in Carbon/Epoxy composites were numerically investigated. A detailed finite element model was created to simulate the inter-laminar and intra-laminar damage in composite structures. The model used special purpose-elements (cohesive elements) to predict delamination growth, while Hashin criteria was used to simulate by fibre and matrix failure, by using. For validation, numerical values were compared with experimental results at impact energy values of 6 J, 10 J and 13 J.

Model predictions was found to be in a good agreement with the experimental data. Furthermore, the model allowed predicting the growth of delamination and the failure of fibre/matrix bonding [17]. In this study, a numerical investigation on the impact behaviour of composite materials was carried out under several impact conditions in terms of impact position and energy. Numerical model was used to study the intra-laminar and inter-laminar damage behaviour in a localised area, which in turn gives a better understanding of the effect of impact on the behaviour of the overall structure. The match between numerical and experimental results was excellent, and the authors believe that the use of these numerical techniques can save time and cost. [13]

There have been many reviews and publications [2, 31-39] that address the hygrothermal effects on the mechanical properties of polymer composite materials. These studies serve as a good source for understanding these influences on these materials. In this context, Sala [26] found that the stiffness and strength of carbon fibre/ epoxy composites were reduced by 35 % after being subjected to liquid absorption and low velocity impact. Sala [26] also stated that the behaviour of [+45°/−45°]s materials is a matrix dominated behaviour. It was also noted by Kumar [36] that water uptake increased the fracture strain for ±45° laminates although their tensile strength was reduced. Moreover, the elastic modulus of impact damaged carbon fibre/epoxy laminates was noticed to be controlled mainly by the fibre breakage amount [2].

However, the use of carbon fibre / epoxy composites in critical engineering applications needs more investigation since the mechanical performance is not well examined in active and changing environmental situations [40]. Therefore, a need exists for an assessment of the mechanical performance of such potentially promising materials under the influence of changing environments and loading speed. In the current study, interactions between the water uptake and low energy impact were investigated in carbon fibre/ epoxy laminates with [-45°/+45°]s lay-up. The aim was to understand the combined effect of these factors on the flexural strength and stiffness and failure modes of these laminates. The flexural strength and stiffness of [-45°/+45°]s laminates were evaluated by means of a three-point bending test.

2. Materials

The composite investigated in this research is a $[+45°/-45°]_s$ carbon fibre reinforced epoxy laminate. The carbon fibre/977-2 composite material uses Toho Tenax HTS fibres, which are high performance carbon fibres with a density of 1.77 g/cm^3 [41]. CYCOM 977-2 is a 177℃ curing toughened epoxy resin with (126-138℃) dry and (104℃) wet service capability. The density of 977-2 epoxy resin is 1.31g/cm^3. This epoxy is formulated for autoclave or press moulding. CYCOM 977-2 epoxy has an excellent impact resistance and it has a shelf life of 12 months at (-18℃) and 42 days at (22℃) [41]. 977-2/HTS composite laminate was manufactured from pre-preg into sheets generally of 2 mm thickness with a ±45° (symmetrical), Figure (1), lay-up and cured in an autoclave. The volume fraction of the composites had been measured as 57% for the ±45° composites. Table (1) give the typical properties of 977-2 composite laminates provided by Cytec [42]. Rectangular specimens of nominal dimensions (60 mm x 15 mm x 2 mm) ±0.2mm were used.
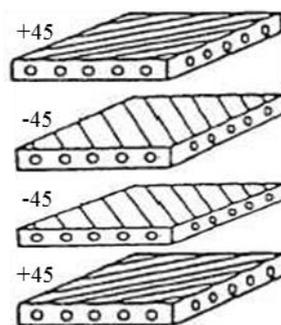
Figure 1. Symmetrical ±45° lay-up of the specimens

Table 1. Typical properties of 977-2 composite laminates

| Mechanical Properties	Room Temperature
0° Tensile Properties	
Strength, ksi (MPa)	390 (2690)
Modulus, msi (GPa)	24.0 (165)
0° Compression Properties	
Strength, ksi (MPa)	230 (1580)
Modulus, msi (GPa)	22.0 (152)
Quasi Compression After Impact	
(1500 in-lb/in impact)	
Strength, ksi (MPa)	38 (262)
Quasi Open Hole Compression	
Strength, ksi (MPa)	45 (310)
Quasi Open Hole Tensile	
Strength, ksi (MPa)	65 (448)

3. Testing

3.1. Drop-weight impact test

Impact testing was performed at room temperature by using a drop weight impact rig with hemispherical steel nose with a diameter of 12.7 mm. The impact rig, Figure (2), is designed with a maximum height of 1.2 m, corresponding to an impact velocity of 4.85 m/s. The support fixture for the impact specimens consisted of a top and a bottom plate, each with a 19.3 mm circular opening. The impact samples were sandwiched between the two plates using four tie-down bolts near and around the opening. Different heights and loads have been used to produce four different low impact velocities/energies. Five specimens were tested at each of the four cases given in Table (2). Figure (3) shows samples geometry and location of the impact point.
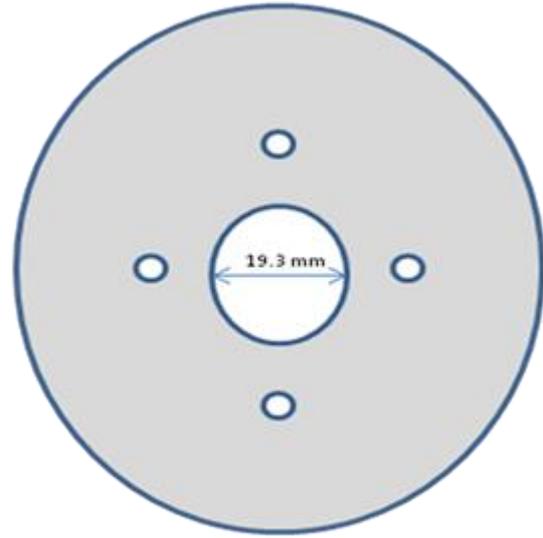
Figure 2. a) Free weight impact rig, and b) Impact specimen support frame

Table 2. Heights, Loads and Energy used for the impact tests

Case	Height, cm	Load, N	Energy, J
1	10	10	1.3
2	20	10	2.3
3	10	20	2.6
4	15	20	3.4
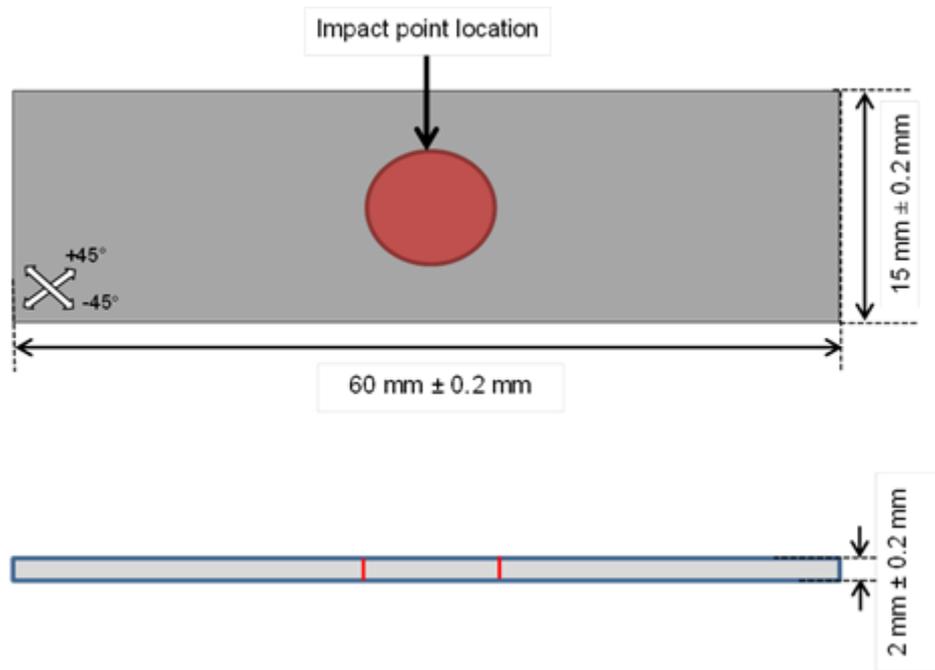
Figure 3. Specimen's geometry and location of impact point

3.2. Water uptake measurements

Conditioning was carried out at 23°C in water for 1000 hours. Samples were split into three groups according to their treatment prior to conditioning. First group of specimens was conditioned from the as-received state. A second group was subjected to two different impact energies before being immersed in water to study the influence of the impact damage on water absorption of these laminates, Table (3).

Table 3. Conditioned specimens

Sample case	Height, cm	Load, N	Number of samples
Un-impacted(As received)	-	-	3
Impacted at 1.3J	10	10	3
Impacted at 2.6 J	10	20	3

3.3. Three-point bending tests

The tests were conducted to evaluate the residual flexural strength of the ±45° laminates before and after being subjected to moisture absorption, drying, re-drying and impact. The three-point bending load, Figure (4), was applied at a constant cross-head rate of 10 mm/min on the impacted side of the specimens and perpendicularly to the fibres.
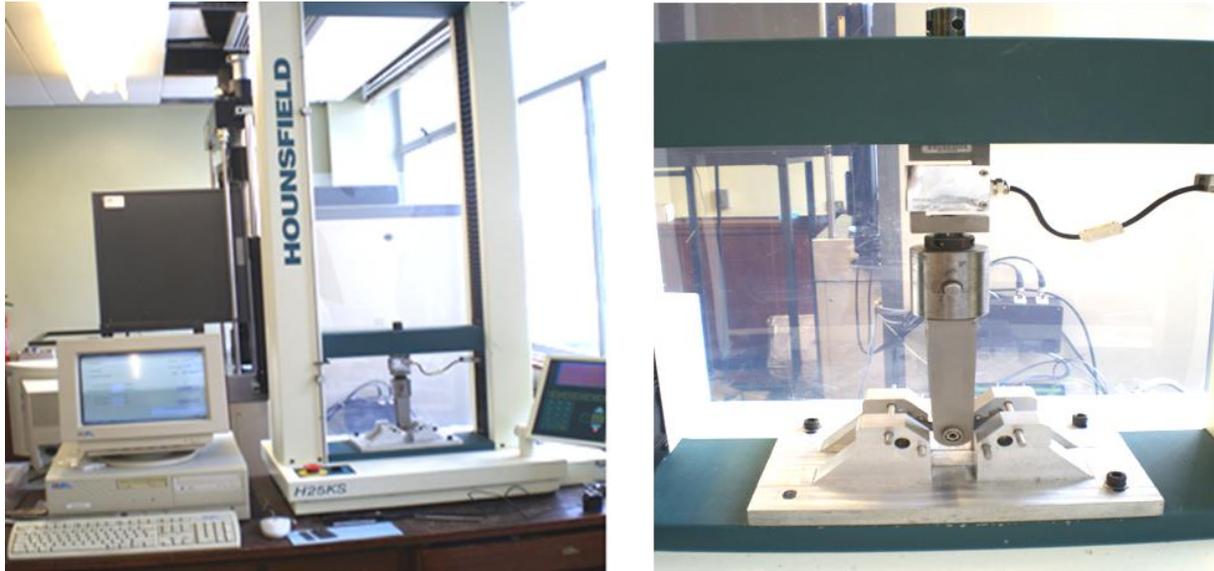
**Figure 4.** Hounsfield- three-point bending testing machine

3.4. Microscopy examination

The fractured specimens were investigated after the bending test using a digital optical microscope. The optical microscopy evaluation has been utilised to show the failure mode in the specimens before and after being subjected to water immersion and low energy impact tests.

4. Results and Discussion

4.1. Water absorption

Water uptake tests were carried out for 6 weeks at room temperature for un-impacted specimens and specimens impacted at 1.3 J and 2.6 J energies prior to the test. Figure (5) shows the averaged water contents of un-impacted and impacted specimens after 6 weeks of immersion. The specimens did not reach the equilibrium moisture content after 6 weeks of immersion at 23 °C due to the short term water uptake test and the complexity of the diffusion paths of the ±45° laminates. It can be seen in Figure (5) that moisture content increased with increasing impact energy, where the moisture content after 6 weeks was about 0.25%, 0.48 and 0.58% for un-impacted, 1.3 J impacted and 2.6 J impacted specimens, respectively. Sala [26] reported similar moisture absorption behaviour where the moisture content increased with increasing the impact energy.
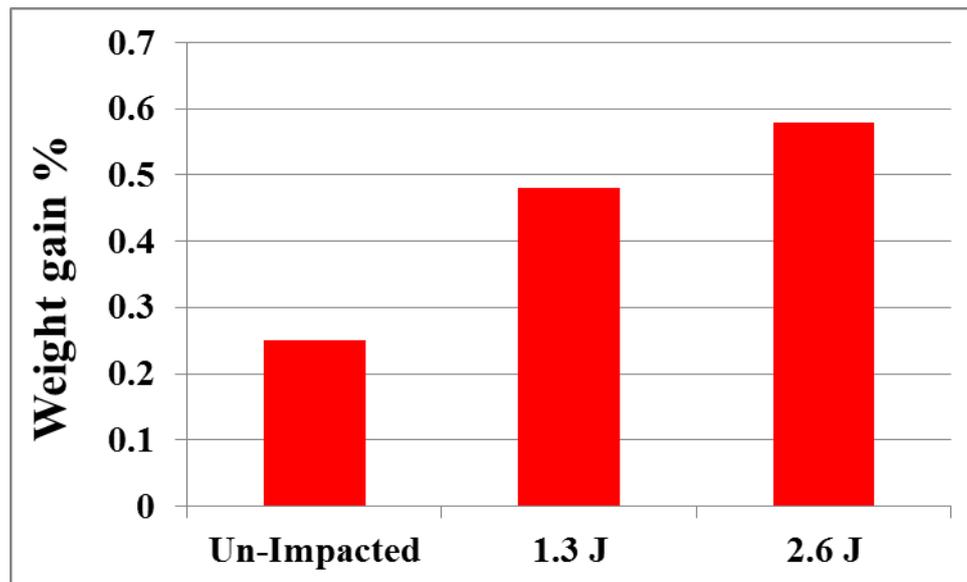
Figure 5. Water content of the un-impacted and impacted ±45° CF/EP laminates

These values, given in Figure (6), show a higher effective diffusion coefficient in the impacted samples. This could not be quantified as the samples had not reached a steady state so the saturation water content was unknown.
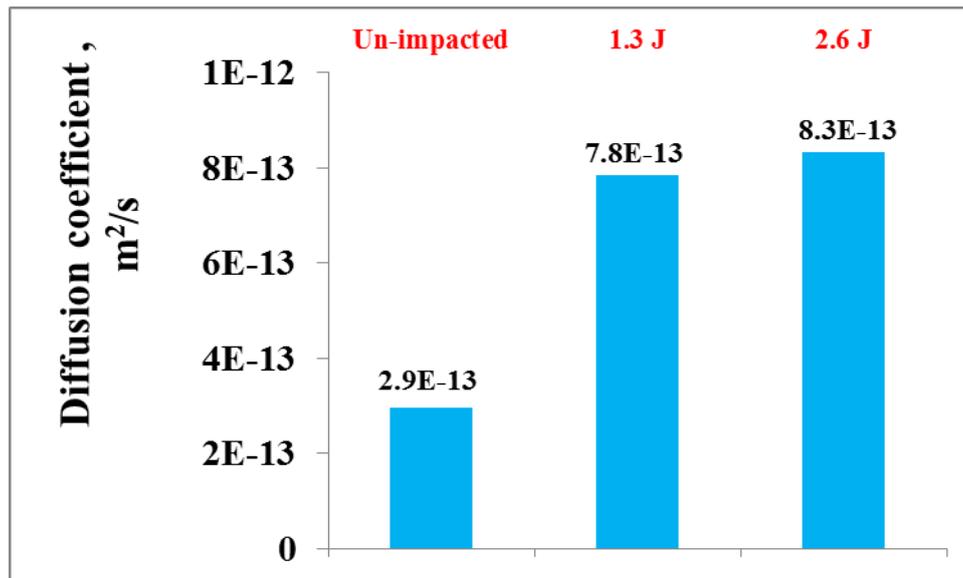
Figure 6. Averaged diffusion coefficients of un-impacted and impacted specimens after 1000 hours immersion in water at room temperature

The higher diffusion rate for the impacted samples, shown in Figure (6), reinforced the high values of moisture contents seen in the impacted samples during the first immersion. This can be attributed to the inter-laminar cracks induced initially by low energy impact. These damage and cracks at 20 N were higher than at 10 N allowing faster diffusion through the specimens. There may also have been an effect from the breakage of the bonds between resin and fibres resulting from the impact, which enhanced the rate of water diffusion by forming additional diffusion paths.

4.2. Low energy impact

Figure (7) shows some digital microscopy images taken after impact tests for the wet and dry samples. Impact damage area increased with increasing impact energy for wet samples while the damaged area varied for dry samples. It can also be noted that impact areas are actually larger for wet samples than dry ones when the height is 10 cm and larger for dry than wet when the height is greater. The propagation of damage mode of dry specimens is usually more catastrophic than wet specimens [14]. This is because wet samples can absorb impact energy and inhibit the formation of delamination due to the presence of interfacial damage in wet specimens before impact. Imielinska [25] reported similar behaviour where impact damage area was slightly less extensive in wet samples. Generally, it is expected that not all

plies will fail at the same load since the strength of a ply is a function of its orientation. Therefore, the failure of plies occurs consecutively in the increasing order of strength.
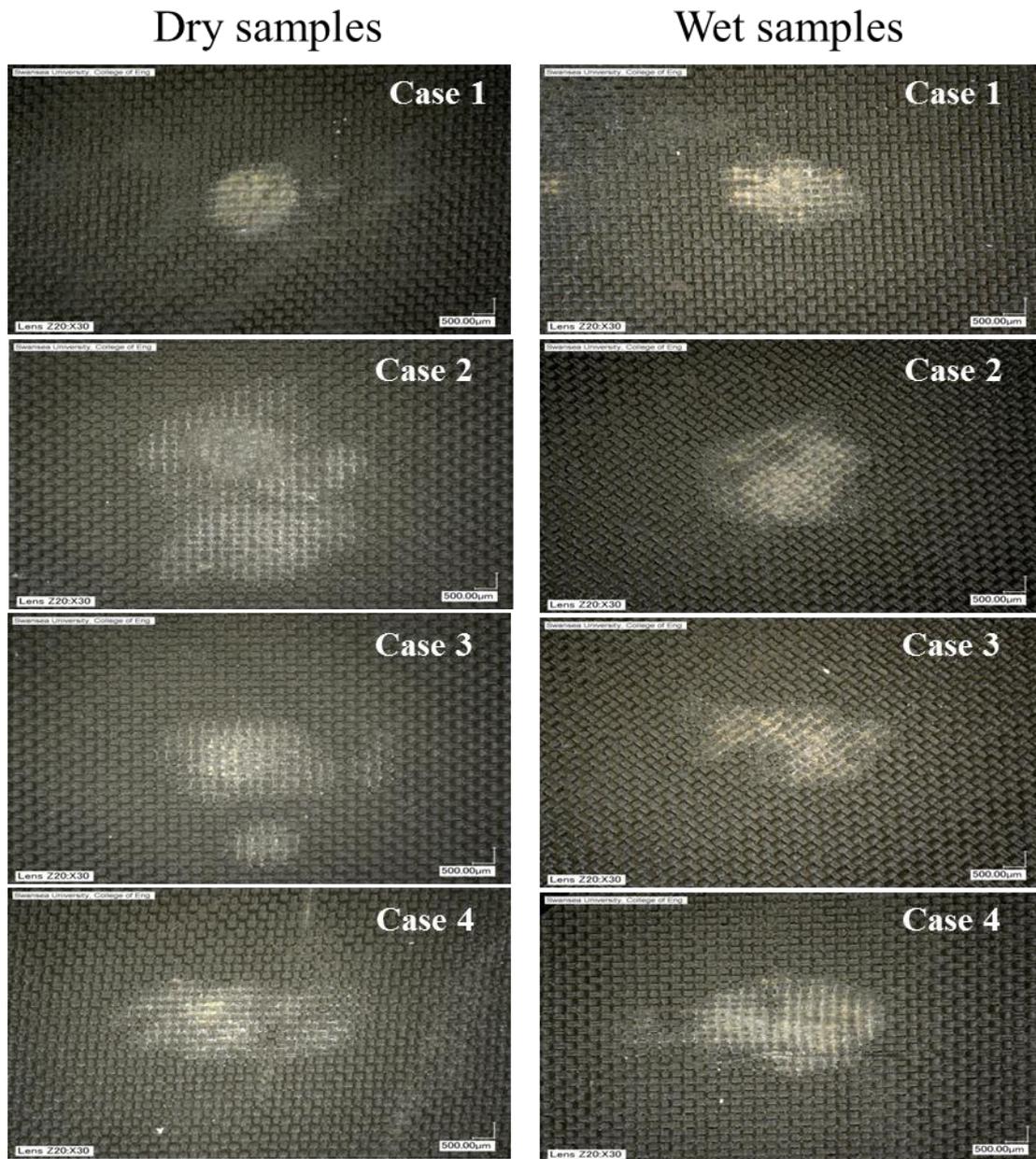
1. (10 cm, 10 N), 2. (20 cm, 10 N), 3. (10 cm, 20 N), 4. (15 cm, 20 N)

Figure 7. Digital optical microscopy images showing the damaged areas of wet and dried specimens subjected to impact tests

4.3.Strain to failure

Specimens were subjected to bending under simply supported beam conditions until complete failure. The exerted loads and displacements were recorded once per second and subsequently converted to engineering stress and strain values.

The averaged strain values (ε) at (σmax) for each of the conditions and impact cases given in Table (1) and (2) are given in Figure (8). The displacement of the wet samples is seen to be higher than that for the other samples and different conditions and cases. This may be attributed to matrix plasticization and softening of the wet material. Imielinska [25] also attributed the bigger deflection of wet samples than dry samples to matrix plasticisation. No significant reduction in the strain was found in the other cases.
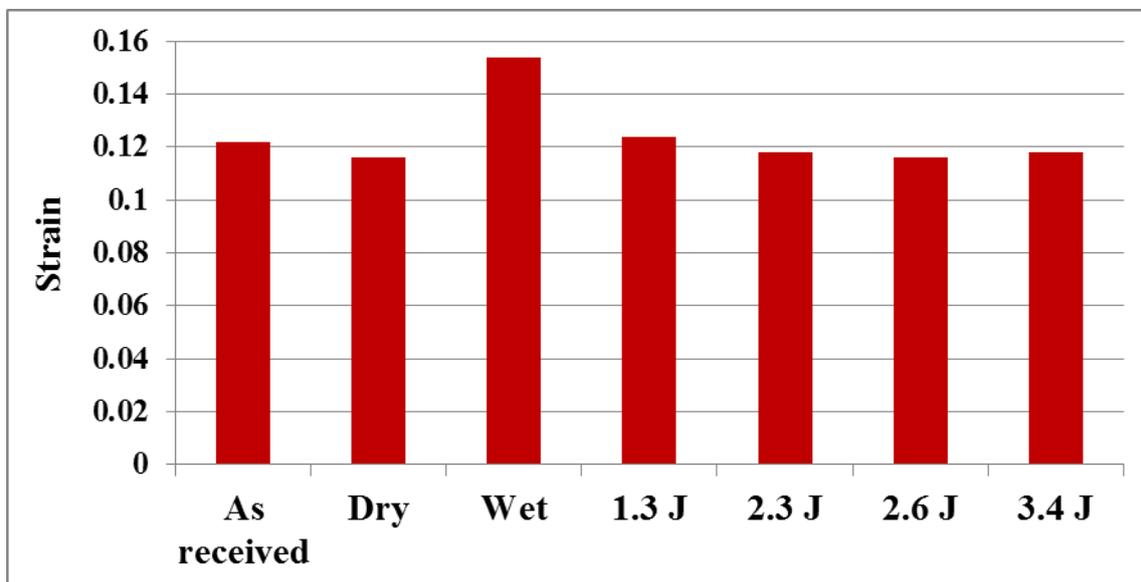
Figure 8. The average strain (ε) at (σ_{max}) of all cases and conditions

4.4. Flexural strength and modulus

Figure (9) shows the ultimate flexural strength σ_{max} and flexural stiffness (modulus) for different cases and conditions. The same pattern can be seen from the graph for both flexural strength and stiffness. The maximum load and stiffness of the wet specimens are slightly lower than that of as-received and dry specimens, while the strength and stiffness dropped drastically to a lower value as the energy impact increased, due to the damage caused by impacting the specimens.
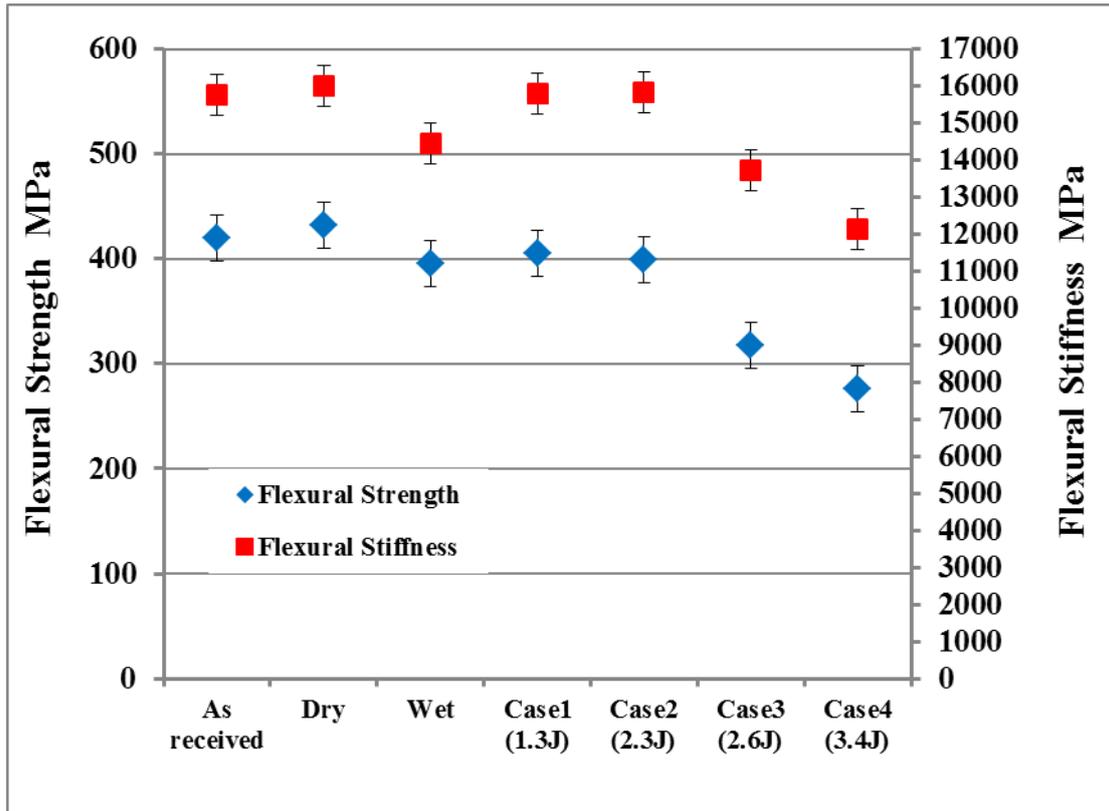
Figure 9. Ultimate flexural strength and flexural stiffness (modulus) of all cases and conditions

Drying the as-received specimens at 70℃ for 6 weeks was observed to increase the flexural strength of the laminates by 3% due to the reduction of the amount of the absorbed water existed in the material and enhancement of the fibre/matrix bonding. Drying samples also normally leads to further curing which was noticed to lead to slight increase in the flexural stiffness and to slight reduction in the strain as shown in Figure (8) and (9) respectively. Wet conditioning of the specimens induced strong matrix plasticization and reduced the flexural strength and modulus of the material by 6 and 10 %, respectively. Sala [26] also found out that water uptake induced matrix plasticisation in [−45°/+45°]s laminates, and this was responsible for large reductions in strength and stiffness, in both tension and compression.

The reduction of the properties was slightly more in the wet samples compared to specimens impacted at low energy 1.3J and 2.3J, Figure (10). Increasing impact energies were found to decrease the strength by 3-35 % and the stiffness by 1-24% depending on the force and height of the striker. The reduction is seen to be much lower at 1.3J compared to

the high deterioration at 3.4J. This supports the outcome from Figure (7) which shows that increasing the force applied at different heights increases delamination and crack propagation, which led to this reduction in the strength and stiffness. This internal damage commonly decreases the properties of the material leading to a reduction in their service life.
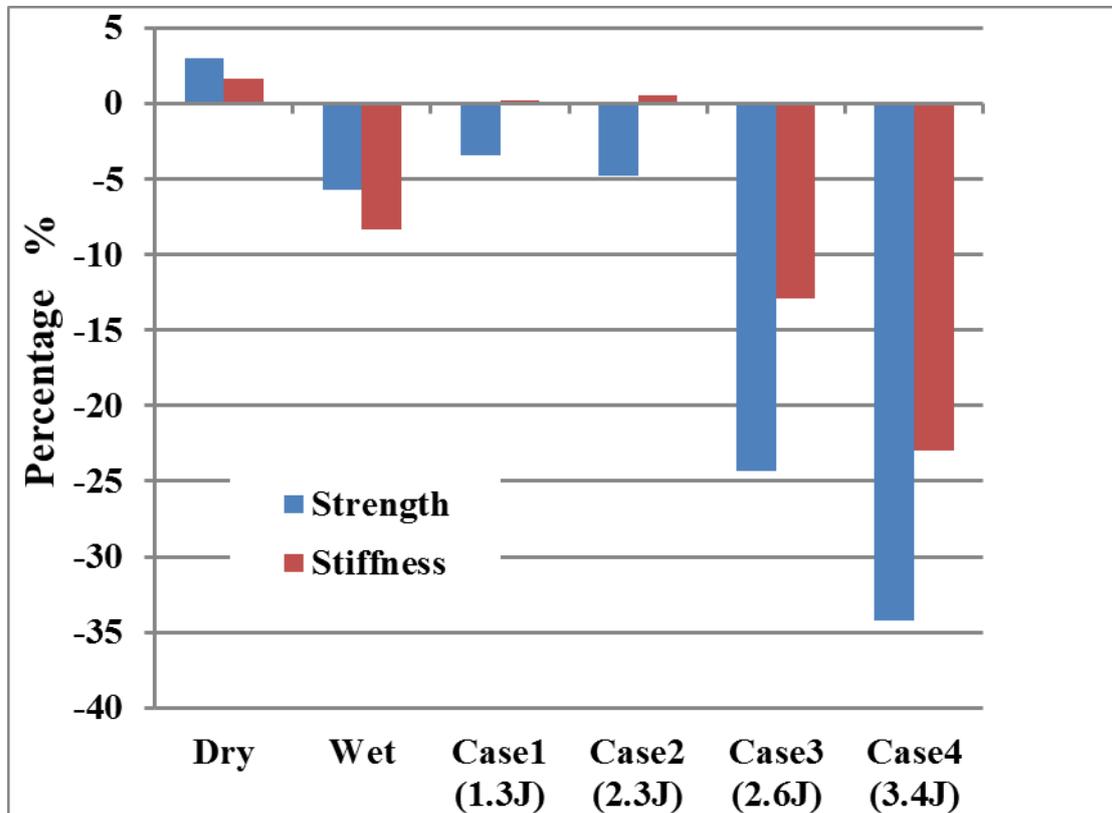
Figure 10. Percentages of reductions in both flexural strength and stiffness

4.5.Evaluation of the combined effect of various conditions

Moreover, more investigation was carried out on a number of samples to study the effect of combining water absorption, drying, re-drying and impact on the overall flexural properties of the samples. Table 4 gives information about the conditions and number of samples used in this part of the investigation.

Table 4. Number of samples used in the impact study

Condition	Number of samples			
	Impact case 1	Impact case 2	Impact case 3	Impact case 4
Impact /Wet	5	5	5	5
Wet/Impact	5	5	5	5
Wet/Impact/Dry	5	5	5	5
Dry/Impact	5	5	5	5

Some samples were impacted and then immersed in water (Impact /Wet) followed by a three-point bending test. Another set of samples was immersed in water for 6 weeks followed by impact at four different energies (Wet/Impact) and then tested. Another group of (Wet/Impact) samples was dried at room temperature and then tested to study the possibility of eliminating or reversing the effect of water on the properties. The final group of samples were dried at 70˚C, Impacted at all cases followed by bending test. Figures (11), (12) and (13) illustrate the differences between all cases at different conditions for flexural strength, stiffness and strain respectively.

In the figures (11),(12) and (13); the label at the column for example (Wet-Case 1) means that samples were subjected first to water absorption followed by impact at first case (1.3J). A similar interpretation is applied for all labels at all columns.
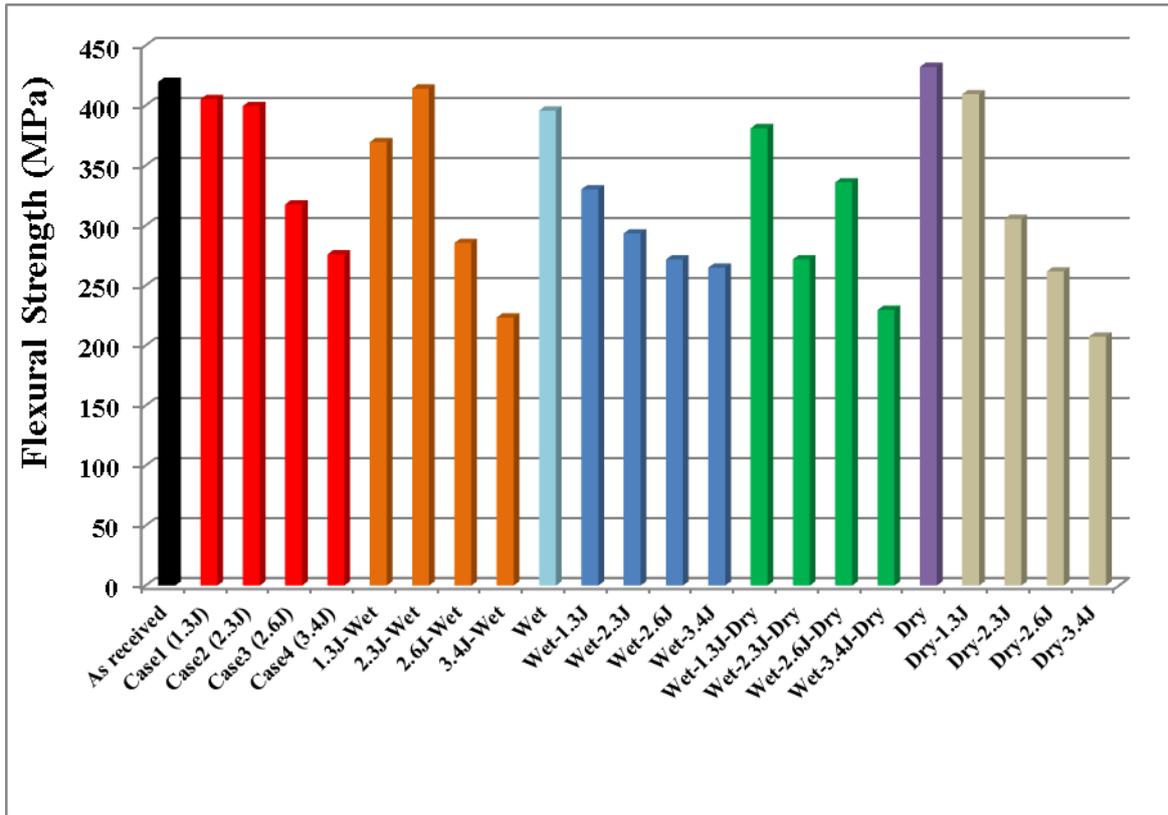
**Figure 11.** Strength of specimens at all impact cases and different conditions
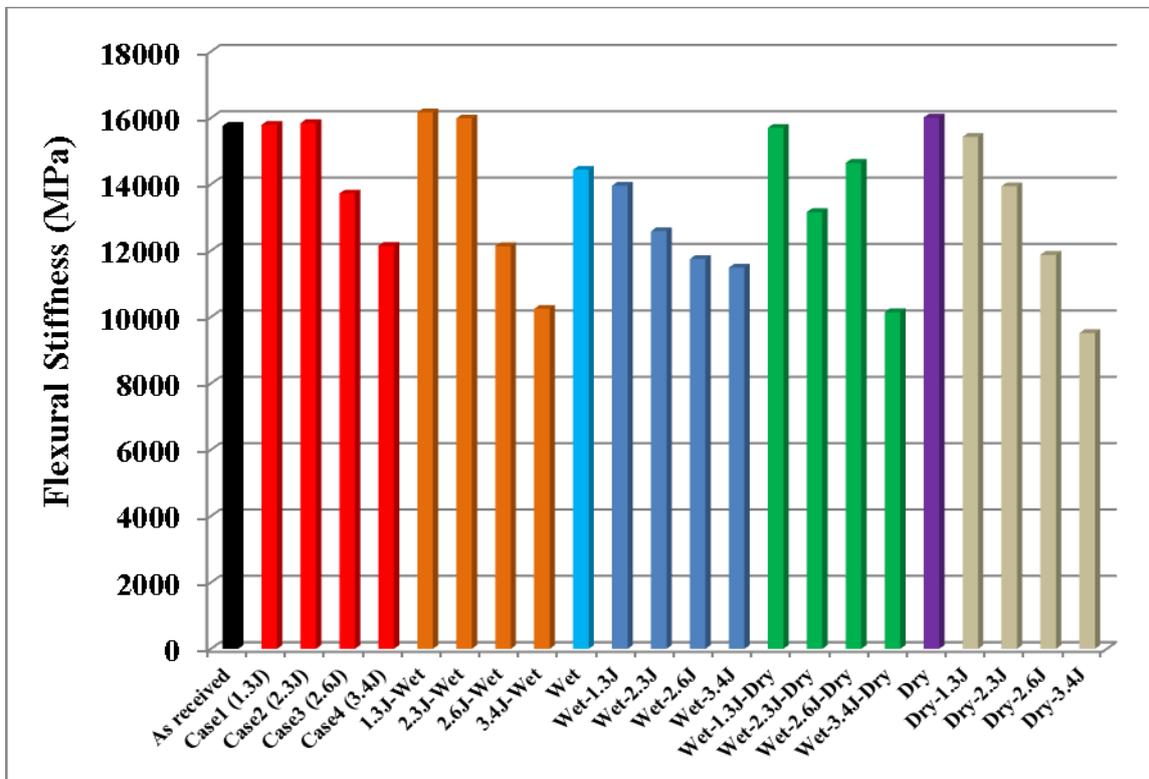
Figure 12. Stiffness of specimens at all impact cases and different conditions
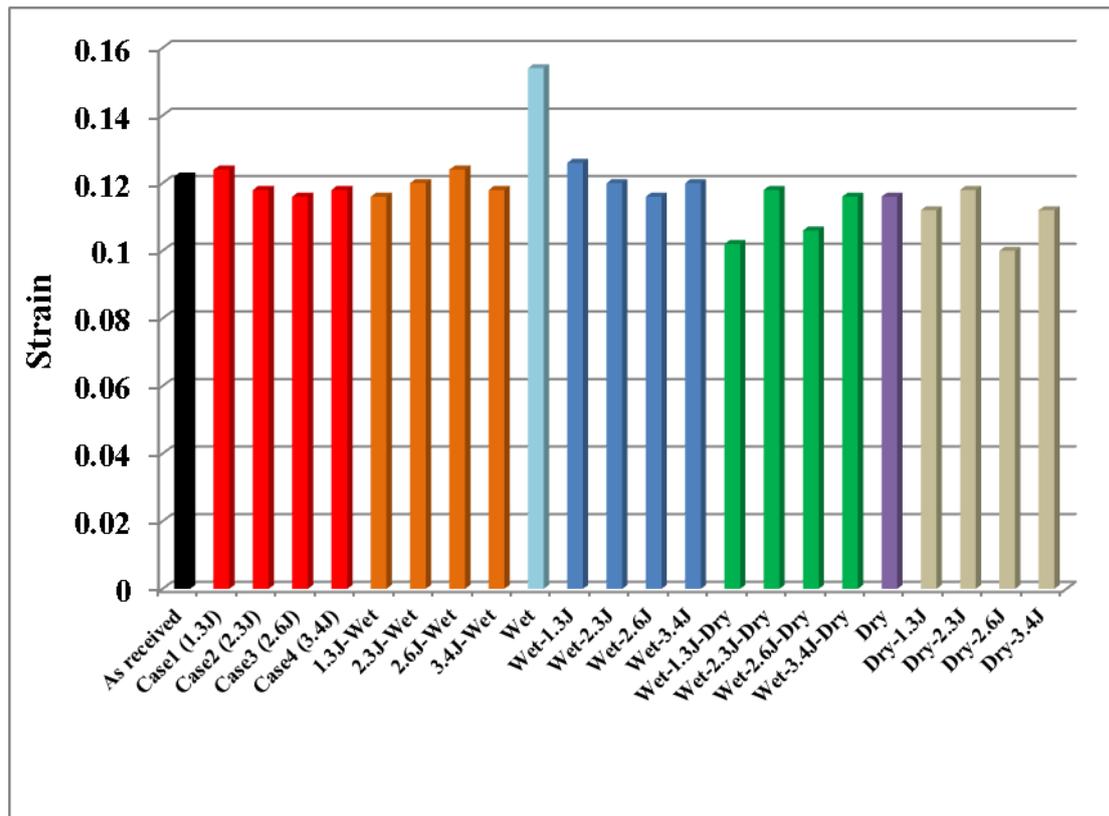
Figure 13. Strain of specimens at all impact cases and different conditions

It has to be mentioned that in all cases, the variation between samples at same condition can be due to the variation in the fibre volume fraction. The flexural properties of samples were investigated at the following combined conditions:

4.5.1. As-received / Impact (Four Cases)

Comparing the flexural properties of as received samples impacted at four cases with as received samples shows that the strength and stiffness decreased slightly with increasing impact energy but still better compared to impacted wet and impacted dry samples. The strain was noticed to increase slightly with energy at 1.3 J but decreased with increasing energy up to 2.6 J which after the strain started to increase again at 3.4 J.

4.5.2. Wet / Impact ((Four Cases)

Clear pattern was observed in this case where strength and stiffness of samples decreased with increasing impact energy. It can be seen from Figure (11) and (12) that this reduction at four cases of impact is small compared to the significant reduction seen in impacted as

received and dry specimens at the four impact cases. For the strain, the behaviour is similar to the pattern seen for as received samples. All these can be due to the plasticization effect of water, which reduced the properties but improved the elastic behaviour.

4.5.3. Dry /Impact (Four Cases)

Similar behaviour and pattern were seen in this case to the wet/impacted samples case where a reduction in both strength and stiffness was noticed with increasing impact energy. But the reduction is significant between the impact energies in this case compared to wet/impacted samples. That can be due to the elimination of the small amount of water existed in the as received samples. Plueddemann [43] reported that water is necessary to support fibre-matrix bonding. No pattern was seen for the strain in this case but the reduction in strain is very small between impact energies.

4.5.4. Impact (Four Cases) / Wet

No clear pattern was seen in this case for both strength and stiffness but the values here are slightly better than values of wet/Impacted samples. This can be attributed the higher amount of water diffused into impacted samples and thus a slight improvement in the elastic behaviour. This confirms somehow in both cases that more amount of water in the sample to a certain extent maintain the properties by improving softening the material and makes it more flexible to absorb more energy, as well as improving the bonding between fibres and epoxy at the interface. Immersing the samples in water after being impacted at low impact energy (1.3J and 2.3J) was found to have slight or no significant effect on the strength and stiffness compared to samples being impacted as received. A significant reduction in properties was noticed with increasing the impact energy. The strain of the samples, in this case, showed a pattern where strain decreased first at 1.3 J and then started to increase again up to 2.6 J followed by a drop at 3.4 J. This behaviour or pattern is on the contrary to the behaviour seen for wet/impacted samples, Figure (13).

4.5.5. Wet / Impact (Four Cases) / Re-Dry

Wet/impacted samples were dried to find the possibility of reversing the water effect. No obvious pattern was obtained in this case with increasing impact energy but a dependency on

impact velocity can be noticed. At low velocity impact (at it was found that drying samples 10 cm height) both strength and stiffness increased significantly compared to wet/impacted samples and the difference between values at this velocity was seen to be small. At higher velocities the difference between values was also noticed to be small and the strength and stiffness were noticed to be close to the values of wet/impacted samples. The dependency on velocity can also be seen for the strain values with slight more reduction at low velocities compared to higher velocities. Also, the strain at low velocities showed a significant reduction compared to wet/impacted samples. This also confirms the effect of water on the flexibility of the material. It can be concluded in this case that the dependency on the impact velocity is more noticeable than the impact energy and this needs further investigation. It can also be said here that the effect of water can be slightly reversed at energies lower than 3.4 J, since at this energy water believed to induce more damage due to the high concertation of the cracks.

4.6. Stored energy capability

The maximum stored energy during the bend test in the material in deformation defines the energy stored in the material after impact and immersion. The area under the stress-strain curve represents the stored energy in the material under loading [44]. The values of the energy stored by each specimen are plotted in Figure (14). Wet samples which tested directly under bending load showed the highest stored energy which is a result of the plasticization due to the presence of water. Specimens impacted at 3.4J at all conditions showed the lowest energy absorption capability due to the damage induced in samples as cracks and delamination. Water absorbed after impact didn't have a significant effect on the stored energy under bending loads. The sensitivity to impact energy was again reduced by the presence of water before impact, but the energy stored under bending reduced compared to Impacted/wet samples. Samples dried prior to impact also showed lower stored energy compared to those impacted as received. Re-drying the specimens slightly improved the stored energy performance at low velocity impact (10cm height). From the Figure (14), the results of Impact/Wet, Wet/Impacted and Wet/Impact/Dry samples show that the 1.3J and 2.6J impact energies do not do too much to the samples. This is the lower height, larger mass, so lower velocity. This suggests that there is a lower velocity threshold when the samples are wet. The stored energy in the materials under bend test after impact and water absorption shows a dependency on the velocity more than energy which needs further investigation.
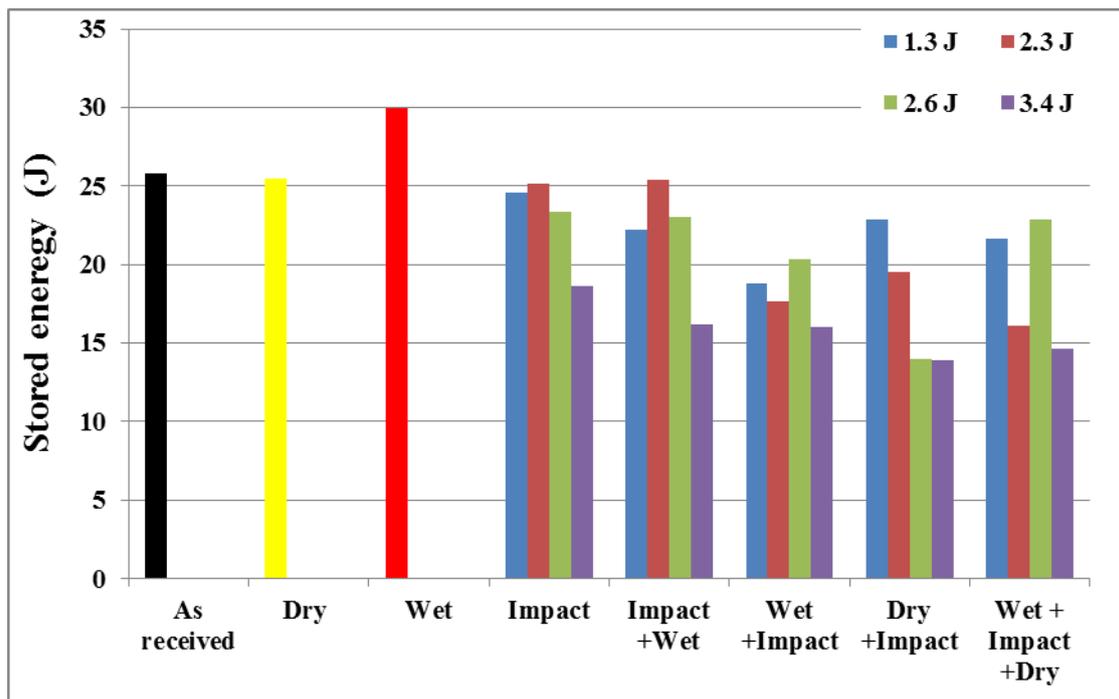
Figure 14. Stored energies during bend test for all conditions

5. Failure Modes

Specimens were examined under a digital optical microscope for accurate determination of failure modes and failure mechanisms such as fibre fracture, matrix cracking, delamination, etc. Figure (15) shows all the failure modes of specimens loaded in three-point bending.

Fibre wrinkling is believed to be the initial failure in the outermost ply under compression, followed by the progress of the failure in the form of delamination, matrix cracking, and buckling in the middle layers. Finally, the ultimate failure of the specimen was noticed to be due to the failure of the outer tensile layer (fibre breakage). Sjoren [2] also found out in his work on carbon fibre composites that the main failure mode was fibre breakage. A horizontal split can be seen for all samples under bending followed by vertical cracking and then catastrophic breakage of the fibres. This behaviour was observed for all various conditions in spite of the difference of the degree of specimen's deterioration as shown in Figure (15). The delamination and the transverse cracks of the as received (AR), dry and wet specimens are

noticed to be pretty similar and slightly less severe compared to the impacted samples at all impact cases. Increasing the impact energy increases the defects produced by low energy impact which in turn lead to intensive deterioration as seen in case 4. Due to the complexity of the failure modes and the stress distribution, it is quite difficult to interpret the inter-laminar failures.
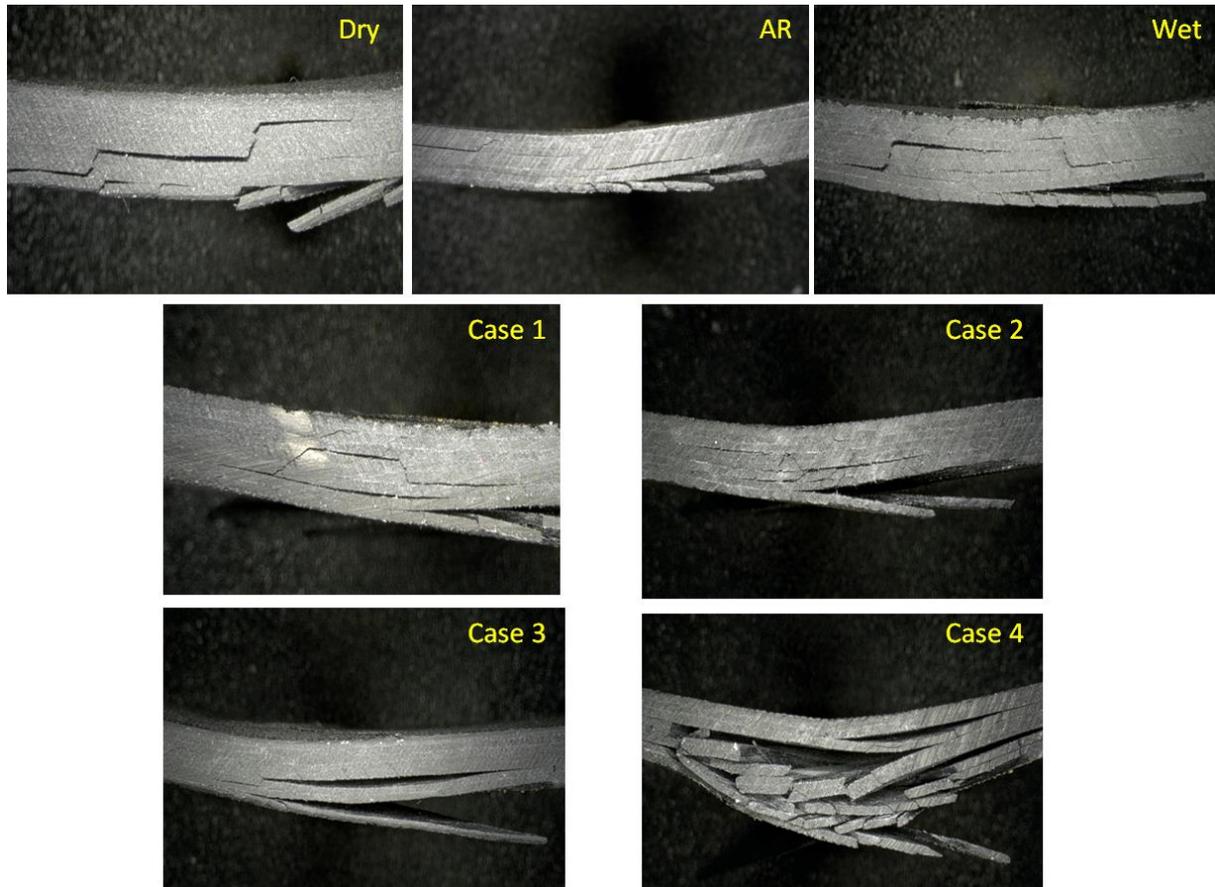
Figure 15. Optical microscopy images after bending tests for all conditions and cases

Drying the specimens after subjecting to water absorption and impact did not change the failure modes; on the contrary, the deterioration at higher energy was worse after drying such as seen in case 4 in Figure (16). That suggests that removing water did not improve the re-bonding.
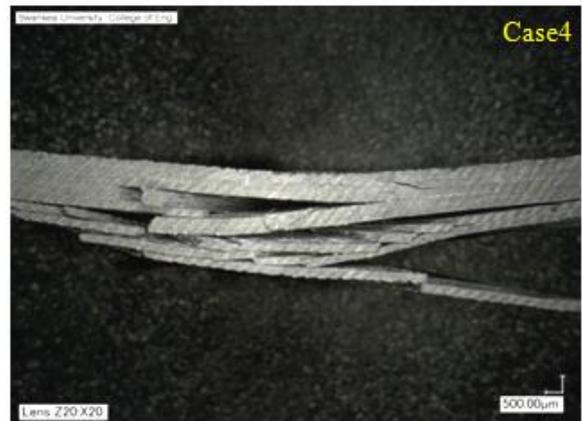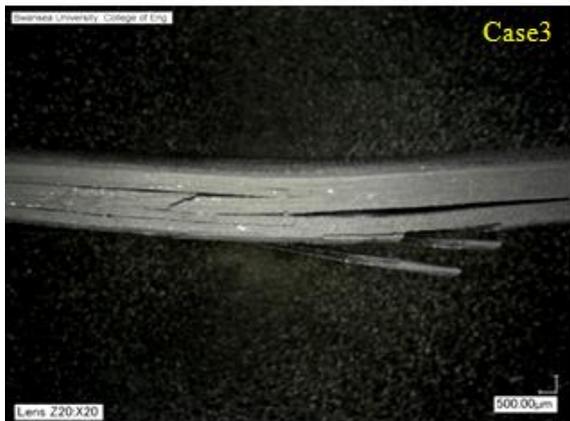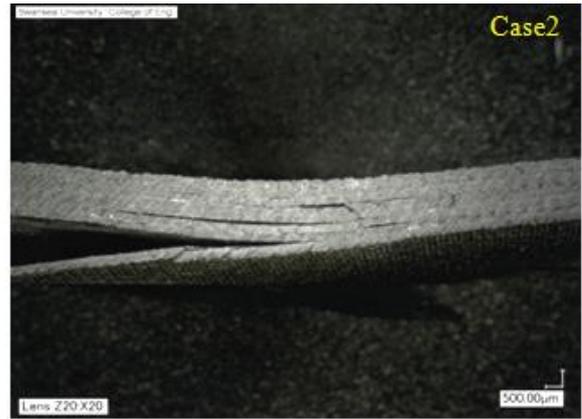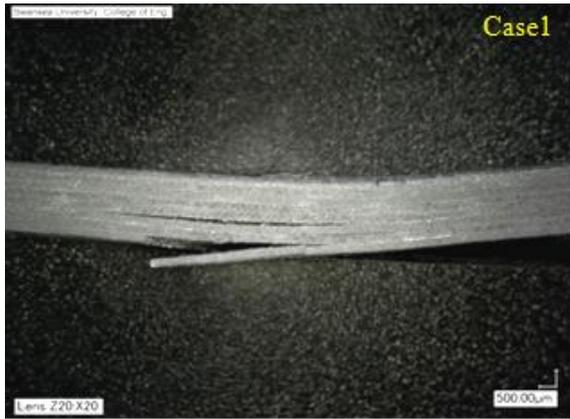
Figure 16. Optical microscopy images after bending test of Wet/Impact/Re-dry specimens

6. Conclusion

$\pm 45°$ Carbon/epoxy laminates were extensively damaged by low energy impact since these laminates responded poorly to impact due to their brittle nature. Moisture content increased with increasing impact energy, and increasing impact energy enhanced the rate of water desorption. Resin plasticization is always a result of water absorption, which can lead to a reduction in the performance of laminates. The experimental results and microscopic images suggested that absorbed water significantly changes the strength, stiffness and fracture mode of the composites but slightly reduces the sensitivity to impact. Removing the absorbed moisture after impact was found to slightly improve strength, stiffness and absorbed energy at energy lower than 3.4J. Drying the samples before impact was found to reduce all properties. The stored energy in the materials was found to be dependent on the impact velocity more than impact energy which needs further investigation. The severity of the failure of the materials in the form of matrix cracking and delamination increased with moisture and impact energy.

7. References

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