Effects of environmental exposures on the barely visible impact damage of flax fibre reinforced biocomposite and its glass fibre hybrids

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Abstract

Exposure of biocomposites to various environmental conditions is a concern when used for structural and semi-structural outdoor applications. This study investigates the effect of different environmental exposures on the low-velocity impact damage behaviour of flax fibre reinforced epoxy composite and its glass/flax hybrids. Flax and flax/glass hybridised epoxy laminates were fabricated using the vacuum infusion technique. A drop-weight low-velocity impact tests were performed on composite laminates at 5 J of incident energy with sub-zero temperatures, i.e., at (-10°C and − 20°C) and room temperature (R.T.). In order to evaluate the induced damage at different temperatures, micro-computed tomography (μ-CT) and visual inspection techniques were employed. The experimental results show that at a sub-zero temperature of -20°C, the flax and hybrid glass/flax composites showed more brittle damage behaviour than at room temperature. Furthermore, these experimental results have shown that alternating hybrid glass/flax laminates exhibited noticeable transverse cracks at R.T. and − 10°C., with higher absorbed energy and maximum impact load-carrying ability arising from positive hybrid effects. Also, it is evidenced from the results that the hybrid approach can be a viable strategy for achieving improved impact performance of natural plant fibre reinforced composites (NPFRCs) when exposed to different environmental conditions.

1. Introduction

Biocomposites have made significant advances as green materials due to concerns relating to the environment and sustainability [1]. Since petroleum-based polymers can cause many adverse environmental effects, such as releases of toxic gases and vapours at burning and inadequate disposal, much of the research focuses on green biopolymeric materials and their use in green composites [2]. Bio-based composites such as plant fibre, for example, can be easily recycled after the end of their service lives without harming the environment [3]. Although bio-based composites reinforced with plant fibres offer many advantages, they also have some disadvantages, such as low moisture resistance and incompatibility between fibres and matrices [4]. However, several approaches have been developed to address these compatibility issues, such as physical and chemical treatment, hybridisation, and nanotechnology [5].

There has been increasing interest in using bio-based composites with natural plant fibres as potential replacements for traditional composite reinforcement, primarily E-glass, due to their low density, high specific properties, low cost of raw materials, and renewable nature [6]. A growing interest in environmentally friendly products has prompted researchers to research natural fibre based composite materials [7–12]. Besides, these researchers have studied and analysed natural fibres embedded in polymeric matrix materials in order to assess their suitability, competitiveness, and capabilities. For example, most automotive companies in the U.K., Europe and worldwide have done extensive research on implementing natural fibre plant reinforced composites (NPFRCs) into their products in making door linens, car interiors, and panels [13] driven by a light-weighting initiative to improve fuel efficiency and CO_2 reduction. Natural fibres derived from plants, such as kenaf, sisal, flax, jute, and hemp, have received considerable attention in producing composite materials attributed to their higher specific strength and stiffness compared to their conventional counterparts [14].

Hybrid composite materials are gaining popularity in the research field because they offer enhanced performances compared to non-hybrid single fibre-reinforced composite materials [15]. Through hybridisation, the composite material exhibits more distinct behaviour, thereby balancing the different properties of the fibres in the composite material and allowing the fibres to utilise their individual properties to meet the final application's requirements [16]. Hybrid composites may also be used for economic reasons since some synthetic materials can be replaced with less expensive natural plant fibres [17, 18]. However, several studies have shown that NPFRCs experience considerable degradation when exposed to humid or wet environments [19–22]. Some application areas, including construction, aerospace, nautical and automotive, may face some structural design issues due to the difficulty in determining the actual strength limits of aged materials [23]. The hybridisation of flax fibres with more robust and corrosion-resistant fibres, such as glass and carbon, can provide a possible solution to overcoming this problem and preventing premature joint failures [24, 25].

On the other hand, composites can fail in several ways and include barely visible impact-induced damage (BVID), significantly reducing the component's structural integrity [26]. A structural composite is likely to be exposed to low-velocity energy impacts during its lifetime [27]. If composite laminates are hit or dropped by a low-energy object, such as a tool used in assembly or maintenance, they will suffer severe cracks, fibre breaks or delaminations [28]. However, there are still unanswered concerns, particularly regarding the influence of environmental factors. Composite structures are exposed to various climates and temperatures during operation and may act more ductile or brittle, compromising their performance in service [29]. The majority of the research in the literature is on glass fibres [30–32] or carbon fibres [33–35]. According to Mathivanan et al. [36], impact damages were frequently undetected at low energy impact levels at room temperatures. Salehi-Khojin et al. [37] investigated the effect of temperature (-50°C to 120°C) on the impact characteristics of GFRP laminates. They observed that the laminates were rigid at sub-zero temperatures with high stiffness, resulting in small deflections. Icten et al. [29] also noticed similar results in glass/epoxy laminates. They found that GFRP laminates exposed to a low-velocity impact at low temperature (– 60°C to 20°C) had smaller damage regions and a more significant perforation threshold. More recently, Vinod and Sudev [38] examined the effect of cryogenic temperatures on polymer composites reinforced with jute and hemp fibres. According to the researchers, the composite's maximum impact strength was 8.935 kJ/m^2 at room temperature. Additionally, they found that the decline in temperature causes many small cracks to appear within the composite material, which results in the material becoming brittle and challenging to handle unexpected loads, reducing its toughness and impact strength. Therefore, it is essential to know the effects of temperature on impact resistance, particularly on NPFRCs, which have received little attention in the literature.

The presence of limited research on the influence of temperature on the low-velocity impact response of natural plant fibre reinforced composites was emphasised in the earlier part. It was also observed that there were limited studies related to the hybrid composites on the influence of low temperatures on the BVID. To address this gap, the present work aims to provide a deeper understanding of hybridisation's effect on the low-velocity impact damage of composite laminates, influenced by different environmental conditions, in terms of impact performance and damage mechanisms. Therefore, this study investigates the low-velocity impact behaviour of flax and flax/glass -hybrid epoxy composite laminates with ± 45 ^{o} fibre orientations tested under low temperatures (-10°C and – 20°C) and room temperature (R.T.) using a drop-weight impact testing system. In addition, visual inspection and micro-computed tomography (μ-C.T.) are used to characterize the damage mechanisms evolved during the falling weight impact event.

2. Materials And Methods

2.1 Materials

In this study, epoxy Evopreg EPC300-F150U was used as reinforcement material, and the hardener 956 was used for the curing process. The laminates were cured at a temperature of 90°C at a rate of 2°C/min, followed by 60 minutes of dwell at 120°C. Laminated materials were then cooled to a temperature above 30°C and demoulded. A constant pressure of 3.5 bar was maintained during the curing process [39].

2.2. Fabrication of composite laminates

In this study, four types of composite laminates were fabricated using Evopreg EPC300-F150U epoxy prepregs with Flax (F), glass (G), and hybrid flax/glass reinforcements using the vacuum infusion technique. The reinforcement was woven [± 45] biaxial stitched configuration of 6.5 g/m^2 and fibre volume fraction of 45% supplied by Coventive Composites, United Kingdom. Four different types of composite laminates were fabricated and are illustrated in Fig. 1: (a) a flax fibre reinforced epoxy composite laminate (Lam F) with six layers of flax (FFFFFF), with a thickness of 5.14 mm; (b) a glass fibre reinforced epoxy composite laminate (Lam-G)with six layers of glass (FFFFFF)with a thickness of 2.60 mm; (c) a hybrid flax/glass fibre reinforced composite laminate with the glass-reinforced layers on the outer surfaces (GFFFG), with a thickness of 4.2 mm; (d) a hybrid flax/glass-reinforced composite laminate with alternating glass and flax reinforced layers (GFGFGFG) with a thickness of 2.52 mm. The fibre volume fraction and thickness of flax, glass and hybrid flax/glass-reinforced epoxy laminates are summarised in Table 1. Throughout the study, the primary focus was to maintain the same number of layers for flax fibre, glass fibre, and hybrid GFFFFG reinforced laminates. However, hybrid GFGFGFG laminates are made up of seven layers because the glass layer on the top and bottom surfaces of the laminate was of primary importance, considering the impact properties of the material. Despite the different thicknesses of the final laminate, the effect of the number of layers was most important due to the structure of the flax and glass fabrics.

Table 1
Fibre weight percentage and thickness of flax, glass and hybrid flax/glass laminates

Composite laminates	Thickness (mm)	Wt. percentage %
Flax	5.14	43.64
Glass	2.60	54.42
Hybrid GFFFFG	4.2	46.6
Hybrid GFGFGFG	2.52	49.9

2.2. Impact machine setup and test parameters

A drop-tower impact system from Instron CEAST 9350 was used for the impact test (Instron Corporation, Norwood, Massachusetts, U.S.A.), as shown in Fig. 2 (a). A hemispherical impactor of 20 mm in diameter and 1.85 kg of mass was used in the tests, along with a circular holder with an unsupported inner area of 40 mm. A square plate of 70 mm x 70 mm samples was clamped to the support at a clamping force of approximately 2.8 kN through a pneumatic clamping plate shown in Fig. 2 (c) with the same inner diameter as the support. Data acquisition software (CEAST Visual IMPACT) was used during the impact tests to control the pendulums, set up test parameters, manage the impact parameters and record the impact force and energy absorption data. The impact energy of 5 J and three operating temperatures, i.e., − 10°C, − 20°C and room temperature (R.T.), were considered. The drop weight tower was equipped with an environmental chamber shown in Fig. 2 (b) with heating and cooling connectors to analyse the effect of temperature on the impact resistance and damage tolerance of flax, glass and hybrid flax/glass-reinforced epoxy composites. Liquid nitrogen was used as a refrigerant. A feedback loop guarantees that liquid nitrogen is delivered to maintain a consistent temperature in the chamber within ±1°C as the temperature is monitored by CEAST software. In order to acquire uniform temperature profiles throughout the specimens, samples were preconditioned for 1 hour at test temperatures in a cooling chamber.

2.3. Damage characterisation

A Nikon (Xtec) XTH225 X-ray micro-computed tomography (µ-CT) was used to conduct micro-CT scans of the impacted samples. The CT scanner uses a 225kV micro-focus source with a rotating target option. The resolution was adjusted at 164 µm in the current study to capture the whole composite plate as it rotated 360 degrees inside the machine, yielding 2400 projections. The scanned plates were scanned at a maximum resolution of 824x 824x824 pixels. [40]. Using Volume Graphics V.G. Studios Max version 2.0, the projections were processed to create the 3D images.

3. Results And Discussion

3.1. Force-displacement characteristics

The impact test data were normalised by a thickness scaling rule to compare the impact behaviour of the flax, glass, hybrid GFFFFG and hybrid GFGFGFG laminates. The normalised force-displacement curves representative of each composite type for 5 J impact energy for all temperatures examined are shown in Fig. 3. It is clear that the maximum load and impact rigidity increase as the temperature decreases. In all cases, the striker rebounded, and the samples did not perforate. The flax, glass (without hybridisation), GFFFFG and GFGFGFG (hybrid) composite samples remained intact. The striker rebounds on the impact surface, indicating closed-loop force-displacement curves, exhibiting residual elastic behaviour in contrast to the three graphs of R.T., -10°C and – 20°C, the slope increases when the temperature decreases. Force-displacement curves with greater slope indicate stiffer samples showing greater load-carrying capability. In this case, the glass fibre without hybridisation and hybrid GFGFGFG reinforced epoxy laminates showed a maximum force of 1225 N/mm and 1151.68 N/mm at -10°C. As a result, the sample stiffens in the out-of-plane direction as the temperature decreases. However, a remarkable difference was observed at -20°C, where the maximum force of hybrid GFGFGFG was less than that of R.T.; perhaps with sub-zero temperatures, there is more of a drop in the penetration threshold. In Table 2, it is clear that the maximum displacement decreases with decreasing temperature in contrast to R.T. and – 20°C, indicating that the material is less elastic at sub-zero temperatures. Also, Kim. et al. [41] observed similar results in graphite/epoxy composites, where they reported that fibres become more brittle at cryogenic temperatures, which contributes to composites' greater stiffness.

Table 2
Key impact properties results for the investigated composites at three temperatures

Composite laminates	(R.T.)			-10°C			-20°C		
	Max normalised force	Max absorbed energy	Max displacement	Max normalised force	Max absorbed energy	Max displacement	Max normalised force	Max absorbed energy	Max displacement
	[N/mm]	[J]	[mm]	[N/mm]	[J]	[mm]	[N/mm]	[J]	[mm]
Flax	673.7	5.439	18.133	671.7	5.385	14.596	664.2	5.421	15.7
Glass	1218	5.466	19.013	1220	5.45	16.704	1205	5.438	16.3
Hybrid GFFFFG	779.7	5.449	18.468	831.8	5.471	15.549	802.9	5.414	15.53
Hybrid GFGFGFG	1150.3	5.514	16.963	1151.6	5.453	16.801	1143.61	5.435	16.91

3.2. Normalised force-time traces

Figure 4 depicts the normalised force-time histories curve results of flax, glass and hybrid flax/glass-reinforced epoxy composites subjected to R.T., -10°C and − 20°C for an energy level of 5 J. Under − 20°C, glass-epoxy laminates showed the highest contact force, followed by the hybrid GFGFGFG epoxy laminates. As shown in Fig. 4 (a) – (c), it is evident that the shapes of curves for flax, glass, and hybrid flax/glass laminate specimens tested at different temperatures exhibit similar profiles. In addition, the force starts linearly from 0 N and increases a non-linear curve up to 1200 N and then increases until the maximum force is reached. The time for impact on these specimens was 15 msec, and the maximum force was recorded at around 11 msec. However, in Fig. 4 (a), flax- epoxy reinforced composite without hybridisation at R.T. showed a slightly different pattern: force increased non-linearly up to 700 N, followed by a non-linear pattern when it reached its maximum level, and then, gradually, the force decreased linearly, finally reaching zero. Also, a similar trend was observed by Kumar et al. [28] for the hemp-basalt/epoxy composites under ambient temperatures.

It is also observed that the impactor did not puncture any samples at all temperatures except for flax fibre reinforced composites, where delaminations and matrix cracking were observed. For an energy level of 5 J, the most significant peak force and shortest contact time developed at − 10°C and − 20°C. This is due to the drop in temperature; the stiffness of the sample increases, which results in a higher contact force developed at the same energy level.

3.3. Absorbed energy – time traces

A graph of absorbed energy versus time is shown in Fig. 5. The maximum energy absorbed is higher than the total energy, which is 5 J, because the striker is rebounded in all the laminates. Based on the three curves, Fig. 5 (a) – (c), there are no visible differences in the energy absorption results as the curves for all samples seem similar. However, in R.T.'s case, the flax laminates absorb a large amount of energy, increasing impact energy, primarily caused by internal damage developing a small dent on the front surface. In addition, the peak force increased as the energy level increased up to the negligible amount of perforations, which was determined by impact energy. The results for maximum normalised force and absorbed energy are reported in Table 2, where it can be observed that maximum absorbed energy is higher at room temperature (R.T.). The difference in penetration behaviour is due to the stiffening of the material at low temperatures and indicates that the hybrid GFGFGFG performed better under dynamic loads.

Also, the hybridisation of flax fibre reinforced composites with the glass-reinforced layers on the outer surfaces (GFFFFG) produces the highest impact tolerance than other laminates. It is mainly because the flax fibre as an inner layer absorbs more energy absorption than the glass fibre with transverse cracks and a small dent on the front side. Also, hybrid GFGFGFG composites achieve maximum energy absorption at room temperature compared to other laminates. These results proved that plant fibre composites hybridised with synthetic fibres would improve the impact resistance and damage tolerance when subjected to low-velocity impact and exposed to different environmental conditions.

3.5. Impact induced damage progression of flax, glass and flax/glass hybrid epoxy composites

Impact-induced damage and failure mechanisms visually observed on the samples' impacted front and rear faces for all temperatures are illustrated in Fig. 6. faces. On the front surface of all the samples, the impacted sides revealed no significant damage, rather only a small dent, which did not appear to be dependent on impact energy levels. However, for the flax laminates under − 10°C, -20°C and R.T., the fibre fracture and matrix crack transverse to the fibres are seen on the rear surface. A minor dent in the form of a conical shape was seen on the back face of the glass, hybrid laminates GFFFFG and GFGFGFG samples. This is due to the fibre failure, and multiple cracks were found on the rear face of the impacted composites. Although visual inspection detected internal failure among fibre layers, other techniques, such as micro-computed tomography (μ-CT), are used to determine failures among fibre layers.

3.6. Micro-computed tomography (μ-CT) analysis

In order to provide insight into the damage modes of the flax, glass and hybrid flax/glass-reinforced epoxy laminates, all samples were examined by X-ray micro-computed tomography; representative images are shown in Figs. 7 and 8. As shown in Fig. 8, for R.T. -10°C and − 20°C, the extent of damage on the flax composites on the back face increased with increasing impact energy. Recent papers have highlighted various damage patterns in flax reinforced laminates affected with a combination of masses and impactor geometry identical to the one employed in the current study [43, 44]. In addition, flax laminates exhibit conventional "pine tree"[45] damage patterns (refer to Fig. 7) characterised by matrix cracks caused by shear, bending and extensive delamination (refer to Fig. 8). However, the damage was barely detectable in glass, GFFFFG, and GFGFGFG laminates and the damage had a conical shape, with an in-plane damage area that increased from the impact surface to the rear one. This damage pattern is commonly found in thick laminates [46]. Also, very noticeable transverse cracks were found in GFGFGFG laminates for R.T., -10°C and − 20°C, proving that hybrid composites are the viable strategy for improving the impact performance of NFRCSs.

In addition, the hybrid GFFFFG laminates under – 20°C noticed delamination due to the presence of flax fibre and the damage caused by the matrix cracking, fibre failure and debonding. However, these cracks are joined as energy increases, resulting in delaminations and fibre fractures, which cause the lower plies to debond [47]. A similar trend has been observed in the current work for hybrid GFGFGFG laminates, where small delamination was found due to the presence of flax fibre while the temperature remained constant. Hybridisation of glass fibre into sustainable flax fibre is a unique approach which has been employed in recent years in order to enhance the mechanical properties of natural plant fibre reinforced polymer composites in which a synergic effect of both reinforcements is exploited [48–51].

4. Conclusions

Many existing works are related to the impact properties of glass and carbon fibre reinforced composites under cryogenic and sub-zero temperatures. Nevertheless, there are limited reports available related to the impact properties of biocomposites at sub-zero temperatures. This study investigated the barely visible impact damage (BVID) behaviour of flax, glass and flax/glass hybrid composites under room temperature (R.T.) and sub-zero temperatures (− 10°C and − 20°C) at 5 J. The following conclusions are reached after a thorough investigation of the impact behaviours of composite samples at room and sub-zero temperatures.

- It is evident that the maximum load and impact toughness increases with a decrease in temperature.
- The hybridisation of flax fibre reinforced composites with the glass-reinforced layers on the outer surfaces (GFFFFG) produces the highest impact tolerance (positive hybrid effects) than other laminates at 20°C. It is mainly because the flax fibre as an inner layer absorbs more energy absorption than the glass fibre with transverse cracks and a small dent on the front side.
- Also, hybrid GFGFGFG laminates showed very noticeable transverse cracks at R.T. and − 20°C., resulting in improved energy absorption. Therefore, considering all these results, it is clear that plant fibre, such as flax fibre reinforced composites hybridised with synthetic fibres, achieved the highest impact resistance and damage tolerance when subjected to low-velocity impact behaviour exposed to different environmental conditions.

Declarations

Declaration of conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author contribution: MAHA conducted the experimental work, analysis of data, wrote the main manuscript, HND, ZZ, AB and RBZ provided the supervision and overall guidance along with editing and analysing the data, CL helped in experimental work and characterisation,

AAS and SEB provided help in editing, CJ helped in data extraction and experimental work.

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Figures

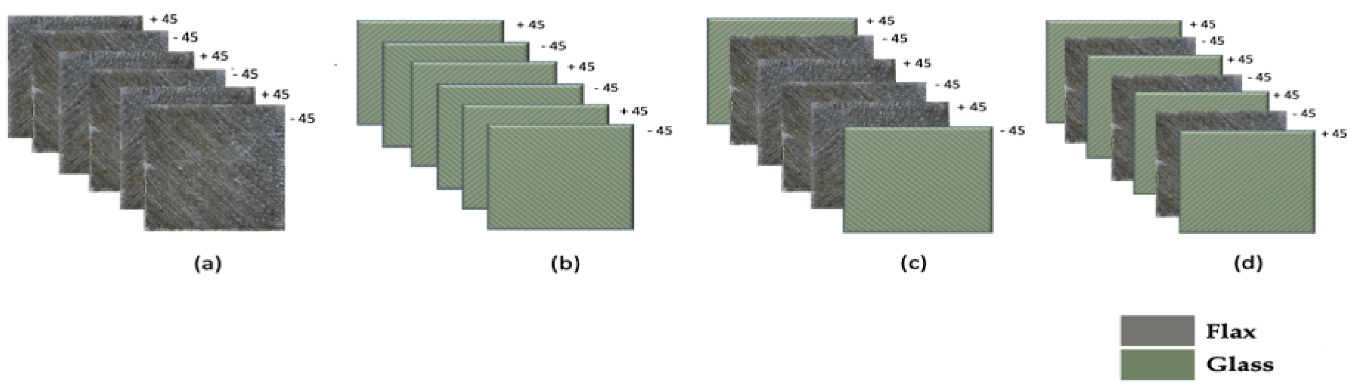

Figure 1

Fabricated composite laminates with fibre orientation [±45] biaxial (a) F- flax FFFFFF laminates; (b) G- glass laminates GGGGGG; (c) Hybrid GFFFFG laminates; (d) Hybrid GFGFGFG laminates.

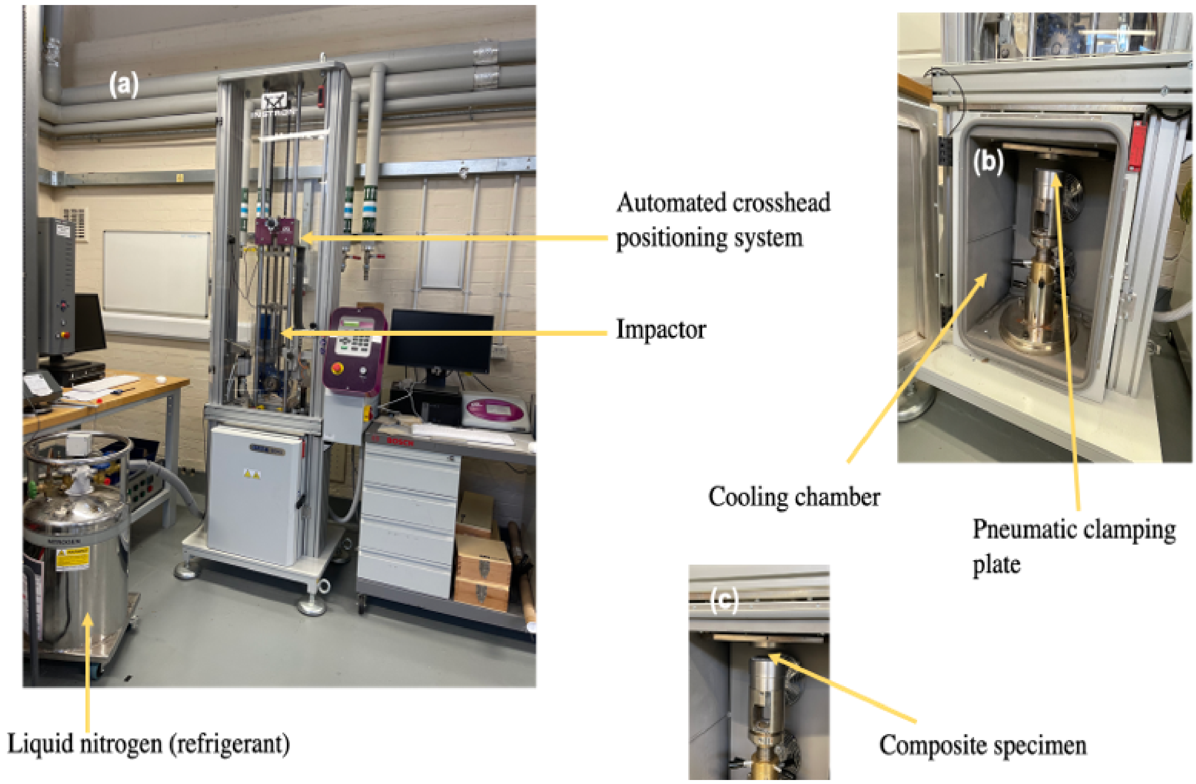

Figure 2

(a) CEAST 9350 drop-tower impact system; (b) cooling chamber with pneumatic clamping plate; (c) composite specimen.

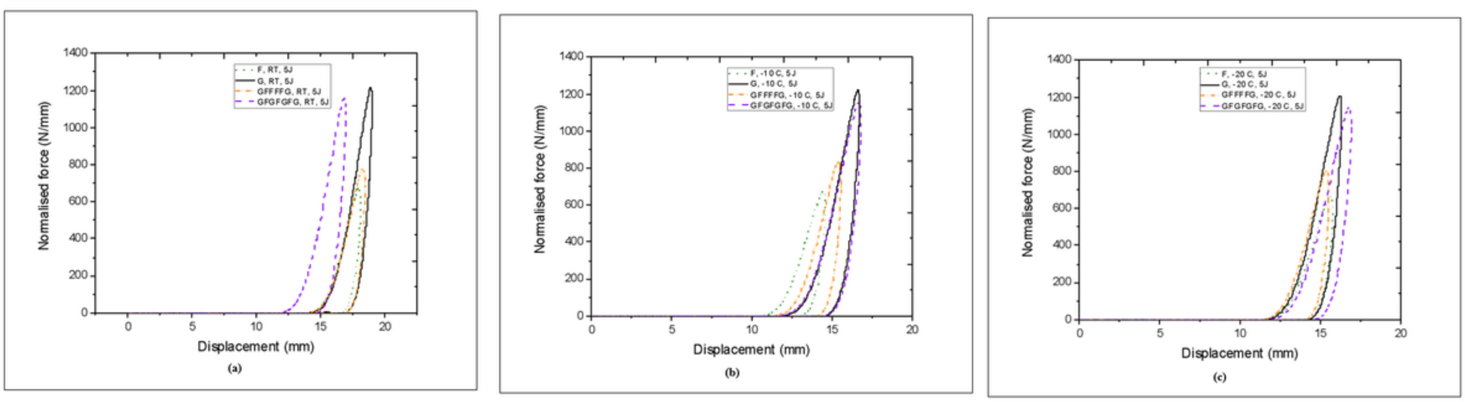

Figure 3

Normalised force vs displacement for 5 J impact energy for (a) Room-temperature (R.T.); (b) -10 ˚C; (c) -20 ˚C.

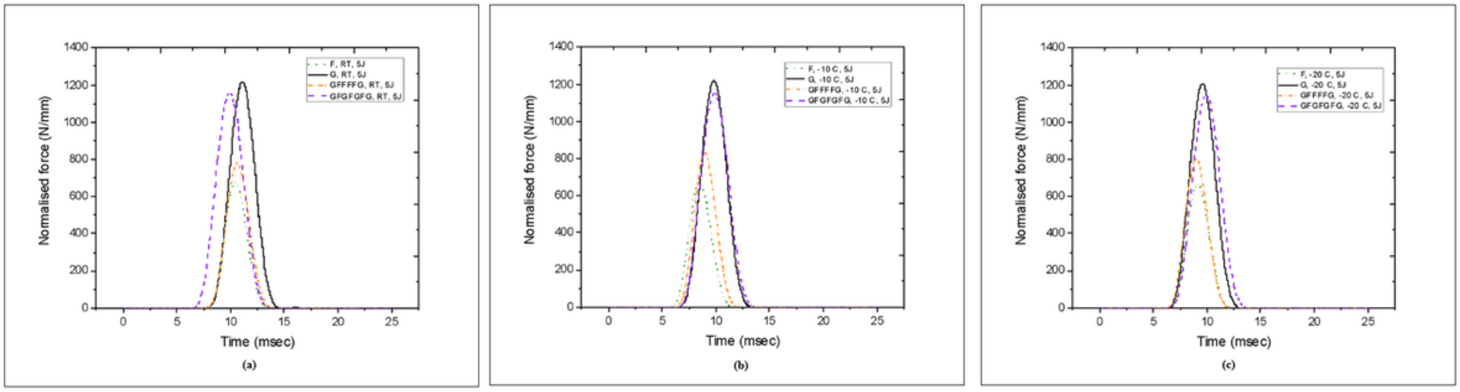

Figure 4

Normalised force vs time for 5 J impact energy for (a) Room-temperature (R.T.); (b) -10 ˚C; (c) -20 ˚C

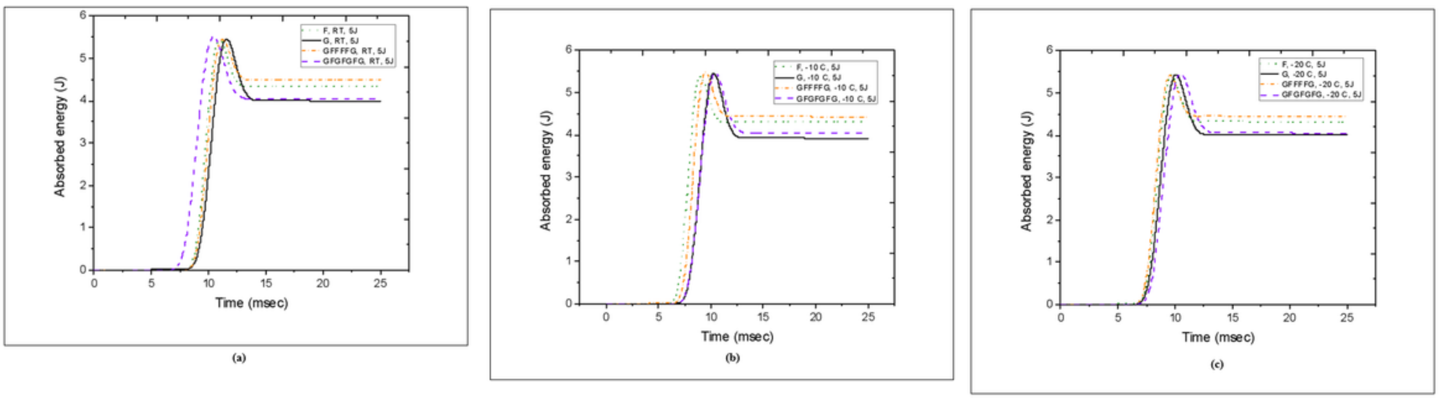

Figure 5

Absorbed energy vs time traces for 5 J impact energy for (a) Room-temperature (R.T.); (b) -10 ° C; (c) -20 ° C

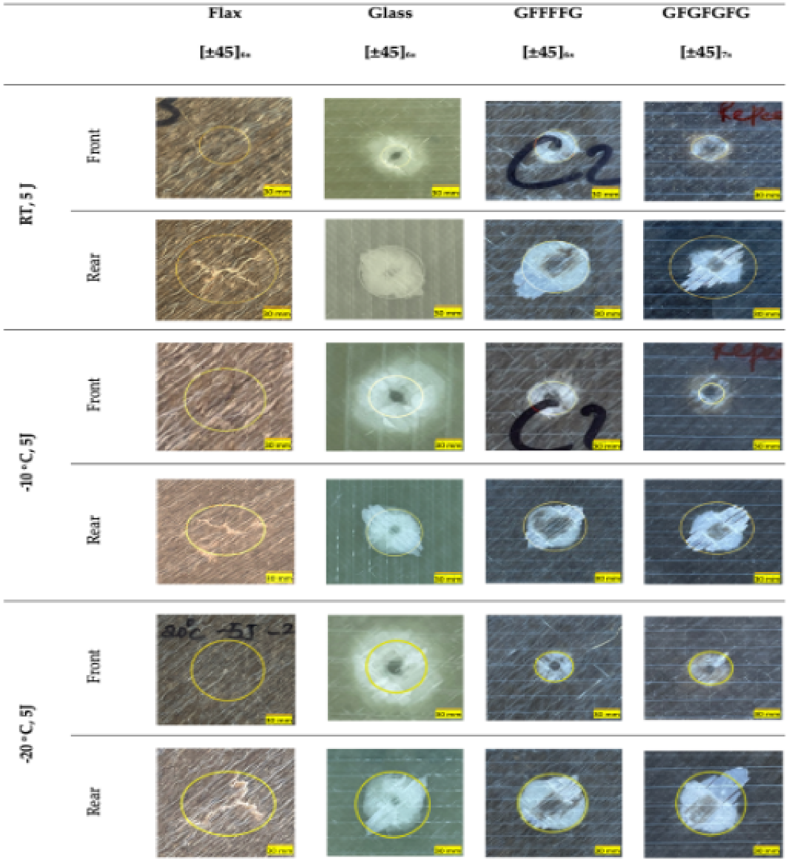

Figure 6

Damage progression of font and rear faces of flax, glass, and flax/glass hybrid composites under R.T., -10 ˚C and -20 ˚C at 5 J.

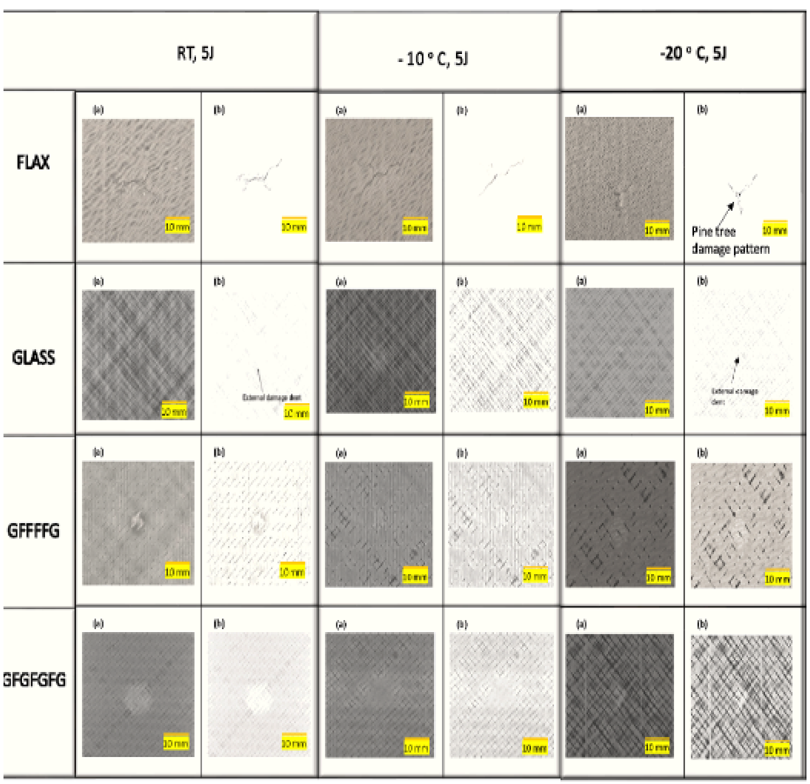

Figure 7

Micro-computed tomography (μ-CT) scans of flax, glass, and flax/glass hybrid composites under R.T., -10 ˚C and – 20 ˚C at 5 J. (a) the rear surface of the impact damage; (b) Skeletonised image of the crack

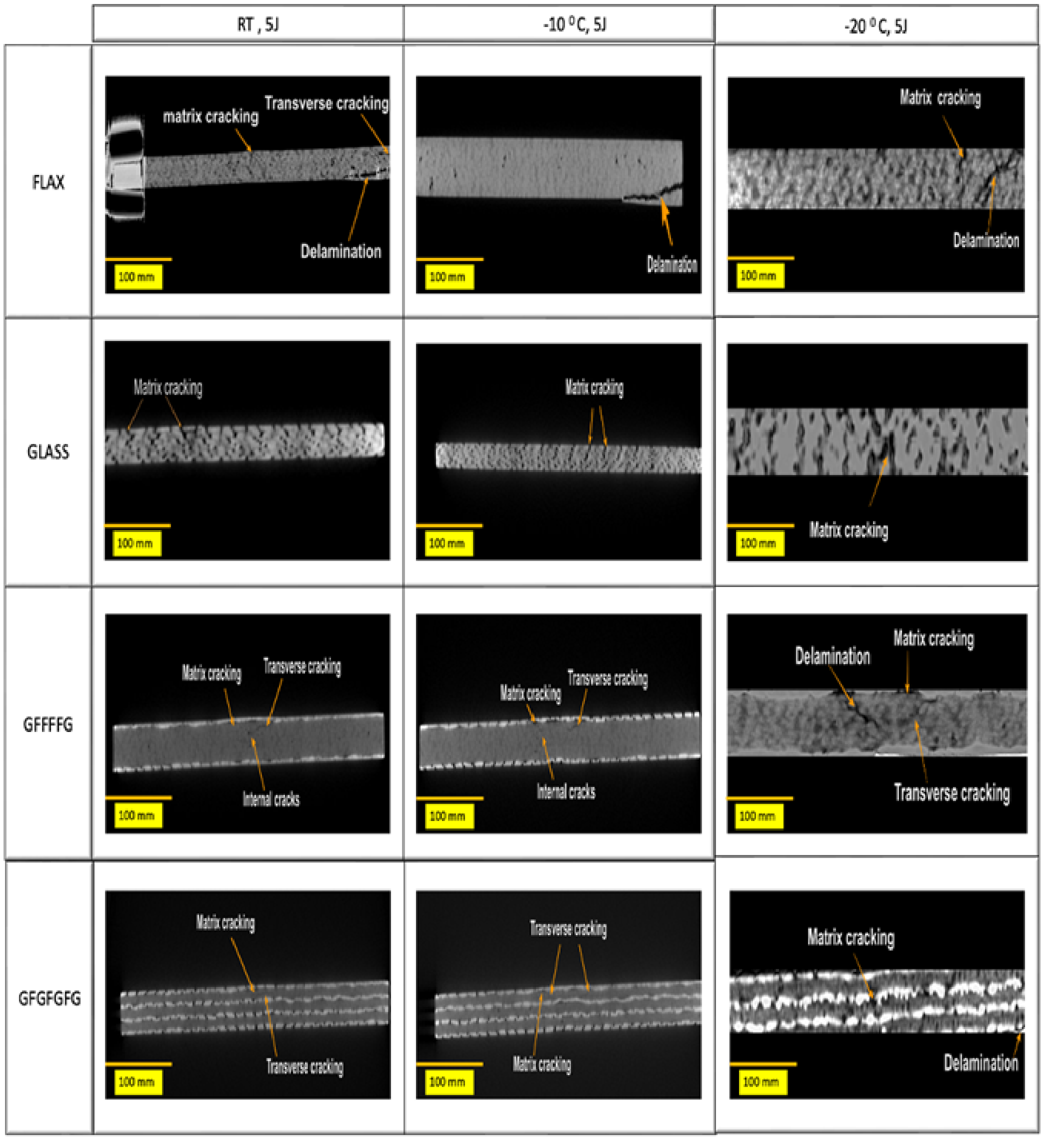

Figure 8

Micro-computed tomography (μ-CT) scans of the laminate thickness of flax, glass, and flax/glass hybrid composites under R.T., -10 ˚C and -20 ˚C at 5 J