Influence of thermal exposure and carbon fibre orientation on the post-fire tensile behaviour of CFRP laminates

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Abstract

The effect of carbon fibre orientation on the post-fire tensile behaviour of carbon fibre-reinforced polymer (CFRP) laminates is investigated in this study. CFRP specimens produced using unique carbon fibre orientations, unidirectional, bidirectional and multidirectional denoted S1, S2 and S3, respectively, are compared before and after exposure to thermal exposure. This study has practical usefulness as CFRP laminates containing these types of carbon fibre orientations are often utilised adjacent or close to fuel storage in aircraft that may present a fire hazard. The study's results showed that the S1 specimens exhibited the highest resistance to tensile failure before and after thermal exposure, whereas the S3 specimens exhibited the lowest resistance to tensile failure before and after thermal exposure. Furthermore, the data has shown that after thermal exposure, the tensile properties of the S1, S2 and S3 CFRP specimens reduced by 35%, 51% and 52%, respectively, compared to before thermal exposure. This loss in tensile properties can be attributed to the pyrolysis of the epoxy matrix and subsequent loss of interfacial bond strength, as the irradiance intensity used in the study was purposely chosen to represent the heat from a fire due to a small fuel leak in an aircraft resulting in temperatures lower than those required to oxidise the carbon fibres. Post-fire imagery has also shown that all the specimens in their undamaged state exhibit brittle failure; however, after thermal exposure, all CFRP specimens exhibit explosive delamination failure.

Keywords: Fire testing, tensile testing, heat flux, fire safety, post-fire properties, carbon fibre reinforced polymer

1 Introduction

Carbon fibre reinforced polymer (CFRP) laminates have been used in aircraft structures for over 50 years [1]. Currently, they are the most common fibre reinforced polymer (FRP) material used to construct new aircraft [2]. In past applications, the use of CFRP laminates in aircraft structures has remained limited to non-load-bearing structural elements such as interior sections and cockpit controls [1]. In recent years, however, due to the development of more durable resin matrices and increasingly robust and stiffer carbon fibres, CFRP laminates have, been utilised in highly hazardous structural locations, such as main spar sections and near to or adjoining fuel tanks [1], [3] that are often load-bearing and placed under tensile stress.

As an engineered material, the manufacture of CFRP laminates can vary. Most importantly, the ability to customise the carbon fibre orientation means their mechanical properties can be optimised to suit the requirements of the application or load [4]. Therefore, the fibre reinforcement of CFRP laminates may take different forms. A few examples of common carbon fibre orientations used to construct main spar sections that are often under tensile stress include unidirectional [0°], quasi-isotropic [0°, 90°] and cross-plied quasi-isotropic [0°, ±45°, 90°] [5].

Nevertheless, despite CFRP laminate's favourable specific strength and stiffness properties relative to traditional aviation materials such as metallic alloys, CFRP laminates are highly combustible and present poor tensile performance following a fire [6] due to the pyrolysis of the matrix resin, loss of interfacial bond strength and oxidation of the carbon fibres. This behaviour means that in the case of an in-flight or post-crash fire mishap, CFRP laminates can ignite and burn uncontrollably, and even if quickly extinguished,

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the tensile performance may be reduced, and the material properties may be lower than their original values [7].

Due to this behaviour, the design variable of the carbon fibre orientation is important because carbon fibres burn at a higher temperature than the resin matrix and may remain intact and able to transfer load after a fire. This behaviour means that the influence of the carbon fibre orientation on a CFRP laminate's tensile response following a fire is critically important as some carbon fibre orientations may be able to withstand load for longer than others. Because of this, this study aims to investigate the post-fire tensile behaviour of CFRP specimens containing three unique carbon fibre orientations that are utilised adjacent to or close to fuel storage tanks onboard aircraft. The results generated from the study will allow a better understanding of fire's impact on the loss of tensile properties in terms of load-displacement relationship, stress-strain relationship and failure modes.

1.1 Background

When fire heats a CFRP laminate, the temperature of the solid phase nearest the heated surface will increase. As the temperature rises, a physico-chemical process known as glass transition will begin causing the matrix (usually an epoxy matrix in aircraft structures [1]) to physically transition from a glassy state into a rubbery state. The temperature at which the effects of the glass transition are most observed is known as the glass transition temperature (T_g). The T_g can vary between 70°C and 150°C [6] for epoxy depending on the additives of the matrix, such as plasticisers and flame retardants and the method of measurement. Mechanically, the effects of the glass transition can significantly reduce the strength and stiffness properties of the matrix and lead to a reduction in load distribution between the matrix and carbon fibres due to a loss of interfacial bond strength.

If the temperature increases above the T_g, a second physico-chemical process known as pyrolysis (thermal decomposition), specifically the pyrolysis of the matrix, will typically begin between temperatures of 250°C and 450°C [8] depending on the properties of the matrix causing an intermediate char layer to develop. Mechanically, the effects of the pyrolysis of the matrix can significantly impact the strength and load transferring capacity [9] due to the loss of the matrix and negligible strength of the remaining char. However, the high threshold temperature for the carbon fibres may allow them to carry some load in the tensile loading direction at temperatures up to 900°C in some cases [10]. However, at high temperatures (above 600°C), when the carbon fibres are exposed directly to the heat source, the carbon fibre reinforcement may begin oxidising [6]. Heterogeneous oxidation of the CFRP laminate results in surface damage to the carbon fibres causing them to thin. Mechanically this process can result in a significant loss in those properties dominated by the fibres, for example, the tensile strength.

2 Experimental Program

2.1 Material and fabrication

Three CFRP laminates were produced for this study. The first laminate contained unidirectional fibres [0°] denoted 'S1', the second contained bidirectional fibres [0°, 90°] denoted 'S2', and the third containing multidirectional fibres [0°, ±45°, 90°] denoted 'S3'. A graphical illustration showing the carbon fibre orientations of each CFRP laminate created for this study is shown in Figure 1. Each laminate comprised *seven* plies bonded using epoxy matrix (diglycidyl ether bisphenol A) in combination with a hardener (curing agent). The carbon fibres were supplied by *Hexcel Composites GmbH*, *Germany*, and the epoxy matrix and hardener were supplied by *Easycomposites Ltd*, *UK* and applied using a 70:30 resin-to-hardener ratio. The seven-ply sheets' nominal average thickness (calculated from five measurements of each sheet taken from different locations) was 5±0.8mm. The fibre-to-resin volume ratio was 52%, 55% and 57% for the S1, S2 and S3 CFRP laminates, respectively and contained 100%, 50% and 33% of the fibre orientated in the longitudinal loading direction, respectively. The fibre-to-resin volume ratio of each laminate was determined using standard burn-off tests following ASTM standards D3171–22, Procedure G [11].

The method of manufacture for each CFRP laminated material was identical, which involved bonding the plies together using a hand (wet) lay-up technique. After the uncured laminates were produced, they were left to cure at room temperature for 24 hours. After curing at room temperature, specimens were produced

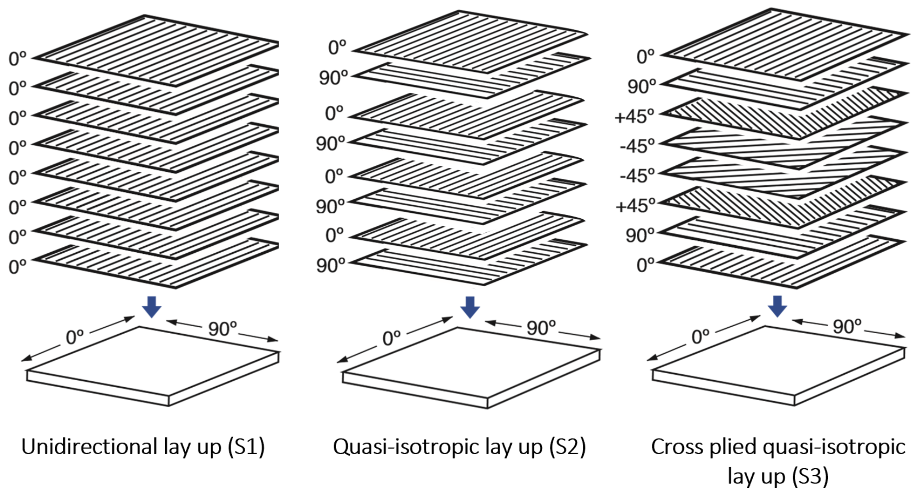

Figure 1: An illustration showing the carbon fibre orientations used in this study.

Table 1: Details about the CFRP laminates composition.

Laminate ID [-]	Laminate thickness [mm]	fibre-to-resin volume ratio [%]	Number of plies [-]	Carbon fibre orientation [-]
S1	5	52	7	$[0,0,0,0,0\ldots]_s$
S2	5	57	7	$[0,90,0,90,0\ldots]_s$
S3	5	55	7	$[0/+45/90/-45/0\ldots]_s$

from the CFRP laminates using a water-cooled diamond wheel saw. All specimens were then dried at 20°C for 12 hours in a gravity convection oven to remove water, after which they were visually inspected for surface defects to confirm they were free from any external delamination, cracks or significant voids.

2.2 Testing specimens

In total, 18 testing specimens were produced (6 from each laminate). The dimensions of each CFRP specimen were identical and measured 250mm × 30mm. In order to reduce gripping damage to the specimens and prevent mechanical failure outside the fire gauge region during the post-fire tensile tests, small end (bonding) tabs measuring 50mm × 30mm were carefully attached to each end of the specimens using an epoxy based adhesive (Sikadur 330). The end (bonding) tabs were produced from cross-ply glass-fibre/resin laminate with the fibres at [±45°] to the specimen axis. The tab thickness was 2mm, with a tab angle of [90°] (i.e. not tapered). The dimensions and geometry of tensile specimens are shown in Figure 2, whereas details about the CFRP laminates composition are given in Table 1.

2.3 Fire exposure tests

The experimental set-up of the fire tests involved using radiant heating from a cone calorimeter to reproduce the effect of fire. By using this heating method, the heat flux and the heating conditions are controlled and repeatable and represent an idealised fire condition (i.e., it is stable and involves a continuous fire with no convective heat transfer from the heat source to the specimen). In addition, this fire-exposure test method allows the area subjected to heating to be well-defined and controlled. Therefore, a heat flux gauge can calibrate the cone calorimeter and control the specimen surface's heat flux.

Three specimens from the S1, S2 and S3 CFRP laminates had a 100mm × 30mm midspan section exposed to

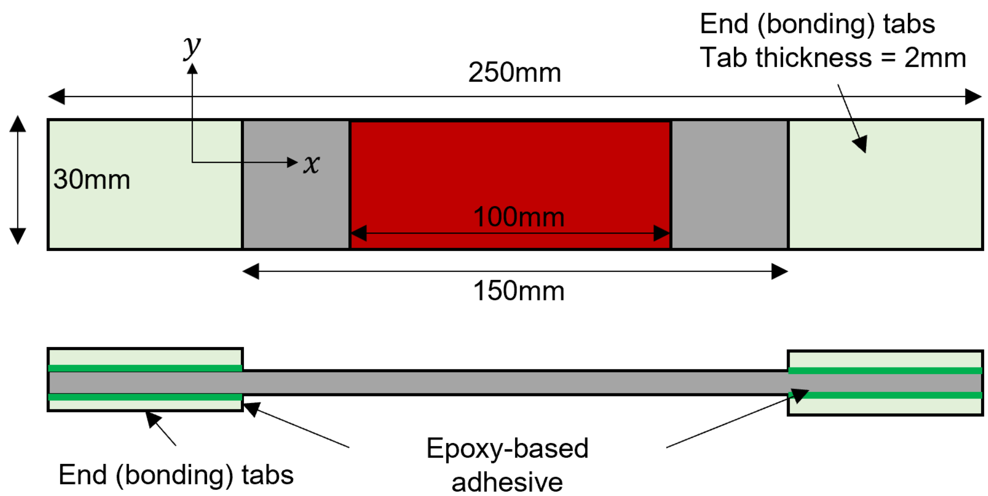

Figure 2: Dimensions and geometry of tensile specimens.

40kW/m^2, leaving the remainder of the material undamaged using ceramic insulation. This exposed area of the CFRP specimens is shaded red in Figure 2. The heat flux was chosen as it is structurally meaningful in that it is linked to the pyrolysis of the epoxy matrix but should not cause damage to the carbon fibre reinforcement, as would be the case during an aircraft fire. This choice of heat flux also has a practical significance as heat from a fire due to a fuel leak on board an aircraft is unlikely to oxidise and therefore damage the carbon fibres completely at the very early stages when it is still small and its spread is limited. The fire exposure time was 3 minutes to represent the response time objective for the UK Rescue and Fire Fighting Service from the time-of-call to when the first responders are in a position to produce 50% of their required discharge rate [12]. After the fire exposure tests using the cone calorimeter, the CFRP specimens were left to cool overnight and had their insulation removed from above and below the exposed midspan section and then mechanically tested. Undamaged specimens were chosen for comparison as it represents the state of specimens exposed to no prior fire. A photograph of the cone calorimeter and set-up used to expose the specimens to thermal exposure is shown in Figure 3.
The pyrolysis of the epoxy matrix and carbon fibre oxidation temperatures of the CFRP specimens were determined using thermogravimetric analysis. This technique involved heating small crucibles filled with milled CFRP at a heating rate of 2.5°C/min from 25°C to 900°C whilst in an air atmosphere using a flow rate of 50 ml/min.

2.4 Post-fire tensile tests

Post-fire tensile mechanical properties were obtained using an industry-standard universal testing machine (UTM) using a loading rate (cross-head speed) of 10mm/min. Each post-fire test was carried out under identical conditions (i.e. room temperature, airflow). Tensile specimens were loaded in the longitudinal x direction using wedge action grips attached to the bonding tabs. The undamaged specimens were initially tested until failure in orders S1, S2 and then S3. This procedure was followed by the fire-damaged CFRP specimens in orders S1, S2 and then S3. The tests were stopped when mechanical failure of the specimens, indicated by a significant drop in load and a runaway displacement, was recorded. A photograph showing the test set-up for the post-fire tests is shown in Figure 4.

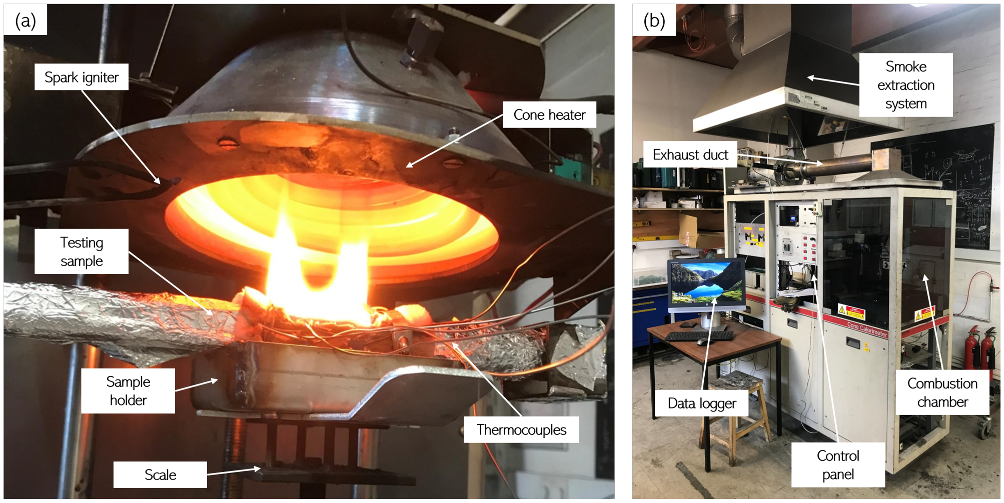

Figure 3: Experimental fire apparatus, showing: (a) a CFRP specimen within the combustion chamber during a fire test and (b) a photograph of a cone calorimeter.

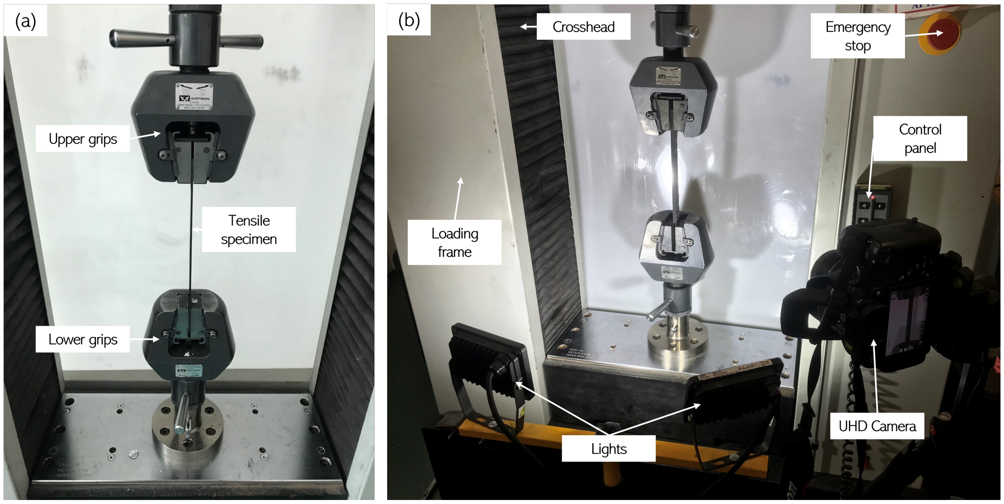

Figure 4: Method for obtaining the post-fire tensile properties, showing: (a) the tensile grips and (b) the UTM apparatus.

3 Test results and discussion

3.1 Temperature evolution and thermal degradation

Figure 5 shows the CFRP specimens' temperature evolution during cone calorimeter tests. Thermocouples were nominally placed at 1mm, 2.5mm and 5mm depths from the exposed surface of the CFRP specimens to monitor the temperatures. However, the temperature measurement accuracy depends on the thermocouple's contact with the walls of the pilot holes, particularly on the fire-exposed surface where the matrix thermal degradation tends to remove the outer surface, exposing the thermocouple to the thermal wave. The thermocouples were therefore secured to the specimens using 1.5mm steel wire to limit this effect.
The result shows that the maximum temperatures of the specimens at x=1mm were 389°C, 312°C and 287°C for the S1, S2 and S3 specimens, respectively. At x=2.5mm, the maximum temperatures were 123°C, 136°C and 141°C for the S1, S2 and S3 specimens, respectively. At x=5mm, the maximum temperature was 78°C, 82°C and 73°C for the S1, S2 and S3 specimens, respectively. This data shows that as the fibre orientation changes from unidirectional to bidirectional and finally to multidirectional, the temperatures at x=1mm decrease. Furthermore, these temperatures show that the epoxy matrix has pyrolysed at the exposed surface for all the specimens following the fire test; however, below the exposed surface, the temperatures remain below the pyrolysis temperature, and therefore the epoxy matrix can be assumed undamaged. The contrast between the through-thickness temperatures of the specimens can be attributed to the low transverse thermal conductivity of the carbon fibres and subsequent thermal decomposition of the matrix, which does not occur uniformly due to the formation of an intermediate char layer that reduces heat transfer.
Furthermore, this behaviour can also be attributed to the fact that from a thermal conductivity standpoint, the S2 and S3 carbon fibre orientation will behave like insulating layers, slowing heat penetration within the laminates. These results suggest that the temperatures of the CFRP specimens depend on the carbon fibre orientation and the specific thermal degradation through the thickness due to the pyrolysis of the epoxy matrix contributing to insulating the lower layers of the composite (unexposed surface) through the formation of an insulating char layer. Mechanically, these temperatures mean a loss of interfacial bond strength and matrix strength has occurred at the exposed surface leading to a reduction in tensile load-bearing capacity in this zone; however, this is not the case at the middle and unexposed surfaces where the matrix is undamaged, and the interfacial bond between the fibred and matrix is good.

3.2 Post-fire tensile tests

Figures 6, 9, 7 and 8 show the results obtained from the post-fire tensile tests on the CFRP specimens. Each curve represents the average data from three repeat tests under identical conditions (airflow, room temperature). The results are tabulated in Table 2 and show that the tensile properties, failure time and displacement at failure of the CFRP specimens reduce after thermal exposure, with the S2 and S3 reducing by more than the S1 CFRP specimens.

3.2.1 Influence of thermal exposure and carbon fibre orientation on tensile behaviour

Figures 6, 7, 8 and 9 shows the tensile behaviour of the CFRP specimens before and after thermal exposure. Each curve represents the average taken from three specimens. Figures 6, 7, 8 shows the load-displacement relationship of the undamaged and damaged CFRP specimens whereas Figure 9 shows the stress-strain relationship of the undamaged and damaged CFRP specimens, respectively.

Influence of thermal exposure

As expected, the data in these Figures show that the CFRP specimens' tensile properties decrease after thermal exposure. Quantitatively the loss of tensile properties after thermal exposure amounts to a 37%, 52% and 54% decrease in tensile load-bearing capacity for the S1, S2 and S3 CFRP specimens, respectively. This behaviour shows that the S2 and S3 CFRP specimens exhibit a larger loss in tensile load-bearing properties than the S1 CFRP specimens. This behaviour can be attributed to the carbon fibre orientation because the carbon fibres are inert at the heat flux severity used and therefore remain undamaged and able to transfer load. Furthermore, the S2 and S3 CFRP specimens rely heavily on the interfacial bond strength between

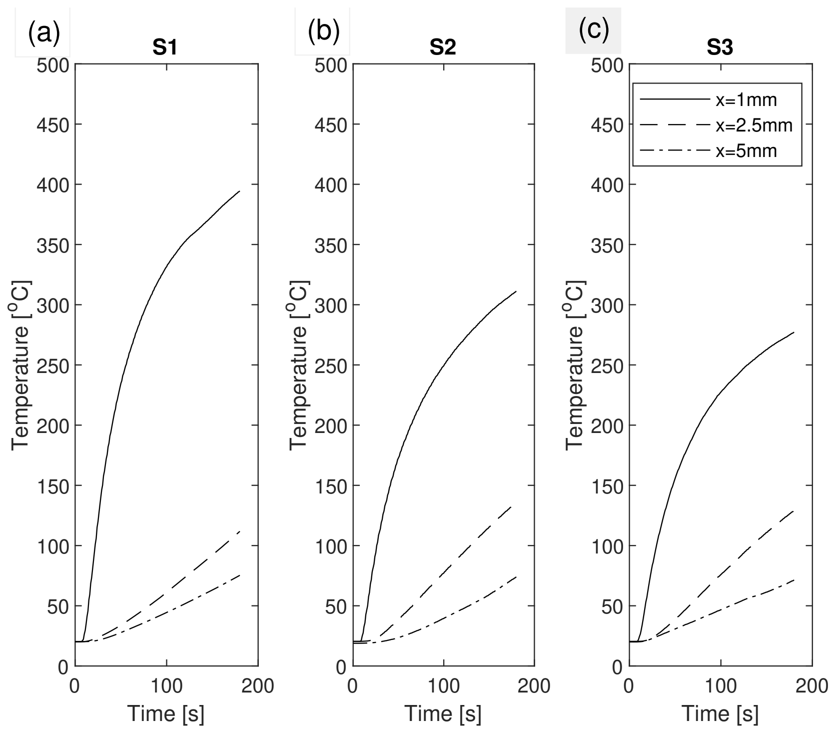

Figure 5: Temperature distribution for the CFRP specimens.

Table 2: Experimental results by CFRP specimen.

Specimen ID [-]	Max. displacement [mm]	Failure load [kN]	Ult. tensile strength [Pa]	Ult. tensile strain [-]	Failure time [s]
S1-Undamaged	10.61	94.49	629.93	0.038	73
S1-Undamaged	10.28	101.10	674.03	0.041	58
S1-Undamaged	11.74	85.98	573.24	0.034	53
S1-40	9.87	61.62	391.08	0.038	68
S1-40	9.57	58.54	371.52	0.036	58
S1-40	9.81	60.39	383.26	0.037	47
S2-Undamaged	10.82	68.11	420.46	0.038	62
S2-Undamaged	10.28	64.76	399.44	0.036	66
S2-Undamaged	10.57	66.60	410.79	0.035	77
S2-40	8.16	33.80	225.33	0.032	44
S2-40	7.59	31.43	209.56	0.030	47
S2-40	7.83	32.44	216.32	0.031	60
S3-Undamaged	12.07	61.62	391.08	0.038	48
S3-Undamaged	11.87	60.39	383.26	0.037	49
S3-Undamaged	9.15	56.08	355.88	0.034	60
S3-40	8.33	29.87	199.19	0.033	42
S3-40	7.61	27.18	181.26	0.030	43
S3-40	7.45	26.59	177.28	0.029	44

the carbon fibres and epoxy matrix to carry and transfer load because more fibre reinforcement is out-of-plane to the principal loading direction. Hence when the epoxy matrix pyrolyses, the tensile properties significantly reduce. This behaviour, therefore, shows that the epoxy matrix has a critical role within the composite in supporting the load and redistributing stresses between fibres, particularly in bidirectional and multidirectional CFRP composites and also highlights that the tensile deformation response of quasi-isotropic laminates is dependent on the carbon fibre orientation. Furthermore, Table 2 shows that maximum displacement at failure and failure time decreases after thermal exposure and reveals that the carbon fibres can continue to support an applied load (albeit with a significant loss in tensile properties) after the epoxy matrix phase has thermally decomposed due to the retained strength of the load-bearing carbon fibres.

Influence of carbon fibre orientation

Before thermal exposure, the S1 specimens have the highest tensile properties and longest failure time, whereas the S3 specimens have the lowest tensile properties and the shortest failure time, and the S2 specimens are in the middle. This behaviour is also the case after thermal exposure. Quantitatively, the data shows that the specimens exhibited a mean (from three repeat tests) tensile failure load before thermal exposure of 94±8kN, 67±2kN and 60±3kN for the S1, S2 and S3 CFRP specimens, respectively. Whereas after thermal exposure, the mean failure load of the S1, S2 and S3 CFRP specimens were 60±2kN, 33±1kN and 28±2kN, respectively. This decrease in mean failure load shows that the carbon fibre orientation is important to retain load-bearing ability as it shows that unidirectional fibre orientations retain a larger quantity of their tensile properties than bidirectional and multidirectional fibre orientations. This behaviour can be attributed to the longitudinal in-plane linear deformation response of the CFRP specimens to a tensile load when placed under stress and the fact that the carbon fibres can still transfer and redistribute the load after this severity of thermal exposure. In contrast, the epoxy matrix cannot transfer and redistribute the load because it has decomposed, meaning the S2 and S3 CFRP specimens rely heavily on the epoxy and interfacial bond of the epoxy and carbon fibres and therefore lose more tensile properties than the S1 CFRP specimens.

The data also shows that the magnitude of the CFRP specimen's linear load-bearing response, characterised by its stiffness, is lowest for the S3 CFRP specimens and highest for the S1 CFRP specimens. This behaviour is because carbon fibres are stiff and do not elongate much under stress. In contrast, the epoxy matrix does elongate because it is ductile (relative to the carbon fibres), hence the S1 specimens, containing 100% of the carbon fibres orientated in the load-bearing [0°] direction, support the stress linearly using all the carbon fibres. On the other hand, however, the S2 and S3 CFRP specimens, where only 50% and 33% of the fibres are orientated in the load-bearing [0°] direction, respectively, demonstrate less stiffness but with a greater displacement before failure and can resist failure at lower loads. This behaviour shows that more carbon fibres orientated in the in-plane loading direction mean the CFRP specimens exhibit more linear-elastic behaviour and less plastic behaviour before tensile failure due to more of the load being taken by the (relatively) weaker epoxy matrix both before and after thermal exposure. Therefore, the behaviour presents an increasing ductility and less strain before failure. This behaviour makes the carbon fibre orientation critically important because it shows that in tension, the carbon fibres dominate the load-bearing response of CFRP laminates due to the matrix being relatively weaker and more ductile and having a lower pyrolysis temperature than the carbon fibres.

Furthermore, based on the data in Table 2, the maximum displacement at failure before thermal exposure is similar between fibre orientations (approximately 10mm); however, after thermal exposure, the measurement fluctuates with the S2 and S3 CFRP specimens exhibiting a shorter displacement at failure than the S1 CFRP specimens.

3.3 Failure modes

Figure 10 shows images detailing the tensile failure modes of the undamaged CFRP specimens at failure. In general, the failure mode of all the undamaged CFRP specimens can be described as being caused by a rapid build-up of localised stress that occurred disproportionately over a small region at the midpoint leading to all the fibres in this zone fracturing. This area was where the nominal stress against nominal strain was at a

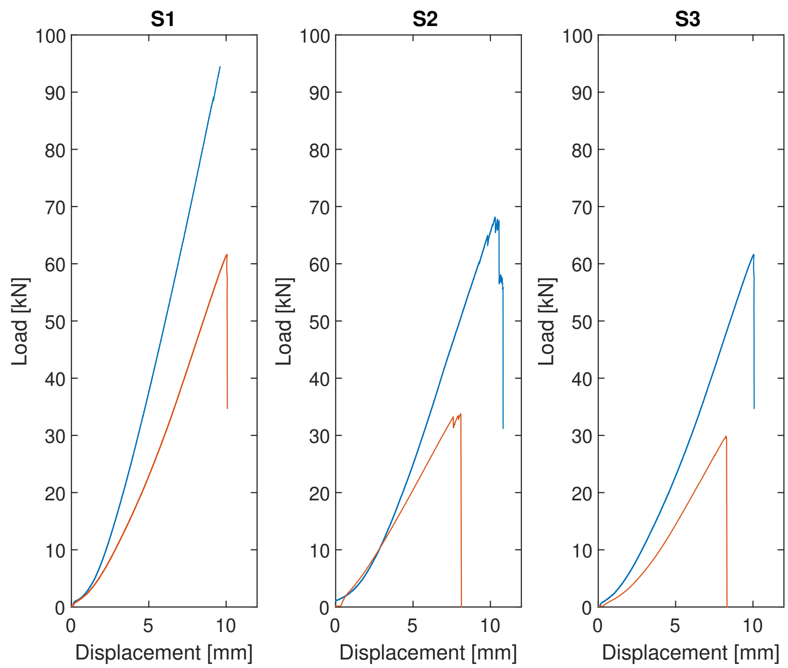

Figure 6: Comparison showing the difference in the load-displacement relationship between the undamaged and thermally exposed CFRP specimens. Each curve represents the average taken from three specimens.

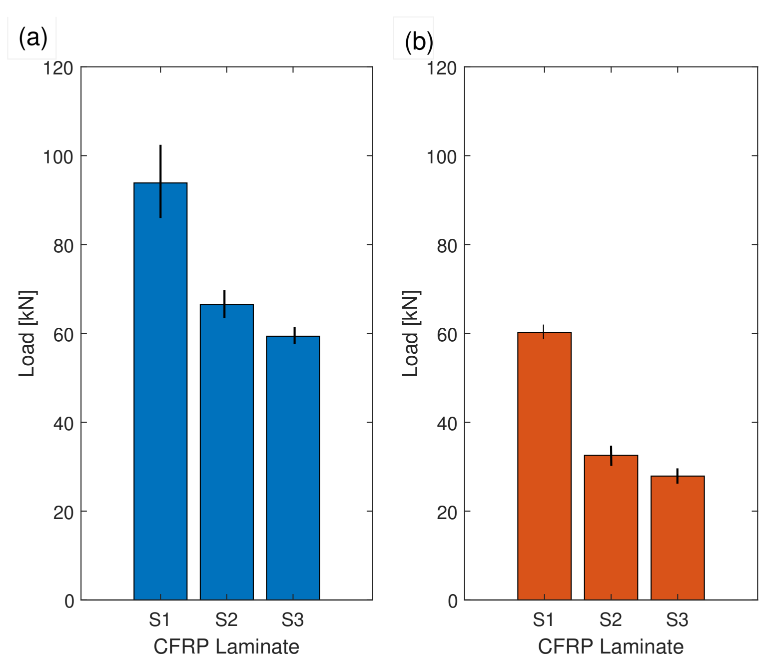

Figure 7: Influence of thermal exposure on the tensile properties of CFRP specimens showing data for the (a) undamaged and (b) damaged specimens.

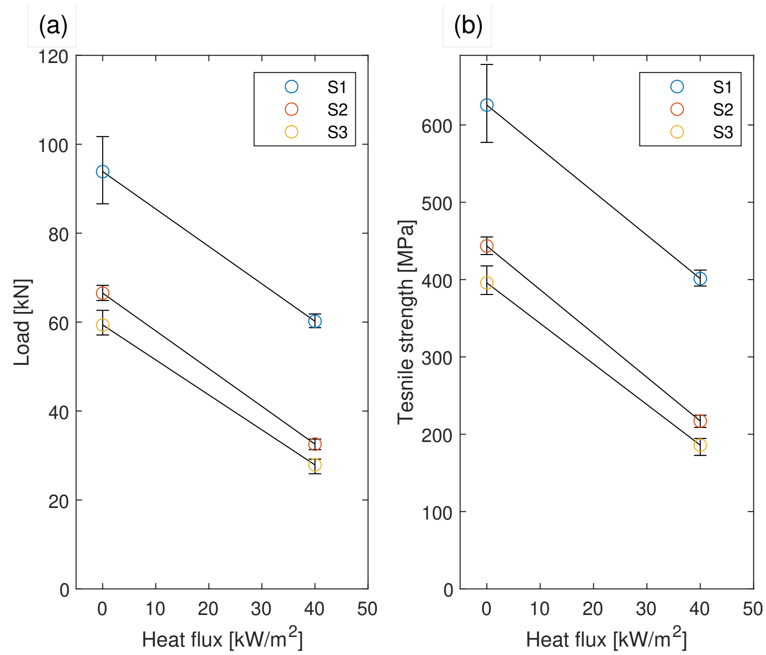

Figure 8: Evolution of (a) ultimate failure load and (b) tensile strength as a function of heat flux depending on fibre orientation.

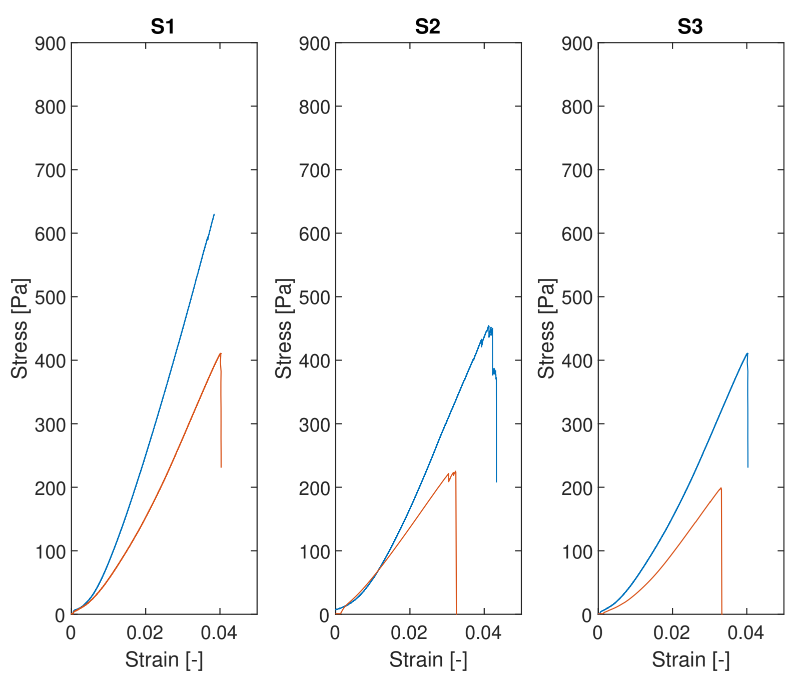

Figure 9: Comparison showing the difference in the stress-strain relationship between the undamaged and thermally exposed CFRP specimens. Each curve represents the average taken from three specimens.

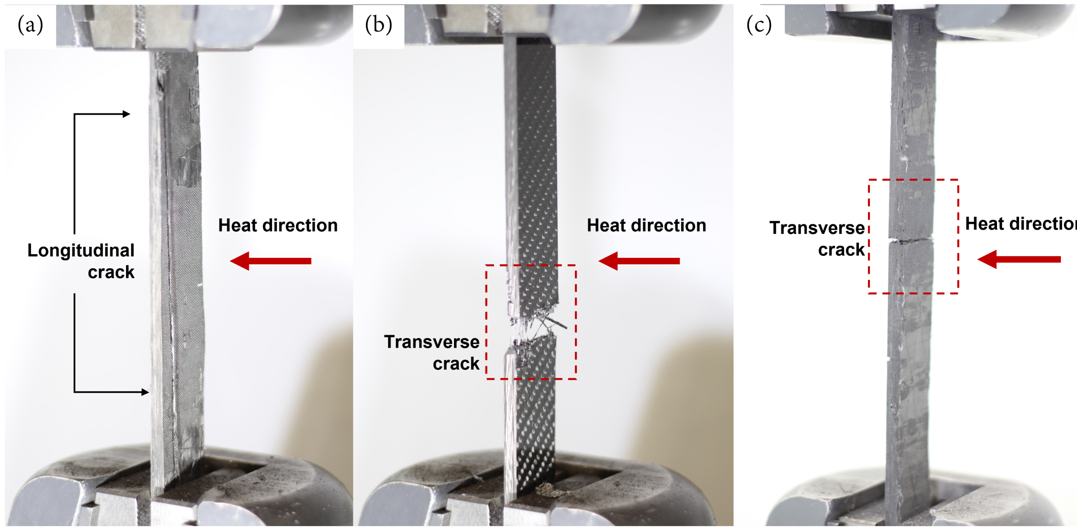

Figure 10: Photographs showing the tensile failure mode of the undamaged (a) S1, (b) S2 and (c) S3 CFRP specimens.

maximum and resulted in a brittle [0°] plane longitudinal crack for the S1 CFRP specimens and a brittle [90°] plane transverse crack for the S2 and S3 CFRP specimens.

Figure 11 shows images detailing the tensile failure modes of the thermally exposed CFRP specimens. The failure of the thermally exposed specimens was largely characterised by fibre pull-out and debonding of the plies due to the pyrolysis of the epoxy matrix, which culminated in explosive delamination damage and failure. However, before failure, the failure mechanism was a stress concentration and the gradual release of energy. This gradual release of energy resulted in an audible loud 'pinging' sound as a fibre tow reached its maximum stress and failed in isolation. When a fibre tow reaches its maximum stress, the stress released is transferred to neighbouring fibres, and consequently, the stress in the fibres nearest to the break magnifies. Transferring the stress overload begins sequential overloading of the other fibre tows, albeit with a shorter duration scale between each audible 'ping'. The audible 'ping' occurred more frequently for the S1 specimens than for the S2 and S3 specimens and showed that carbon fibre remained undamaged.

4 Conclusions

This study has investigated the influence of thermal exposure and carbon fibre orientation on the post-fire tensile behaviour of CFRP specimens. Three CFRP laminates were produced, each containing a unique carbon fibre orientation commonly found in close proximity to fuel storage on commercial aircraft and exposed to thermal exposure using a cone calorimeter. The thermal exposure intensity was chosen as it is structurally meaningful in that it is linked to the pyrolysis of the epoxy matrix but should not cause damage to the carbon fibre reinforcement. This choice of heat flux also has a practical significance as heat from a fire due to a fuel leak on board an aircraft is unlikely to expose the carbon fibres to the heat required for oxidative decomposition. The time of exposure was chosen to coincide with the response time objective for the UK Rescue and Fire Fighting Service. In total, 18 CFRP specimens were produced, 6 from each of the CFRP laminates. This quantity meant that the S1, S2 and S3 specimens could be tested in triplicate initially in their undamaged state and then after thermal exposure to compare behaviour. Furthermore, the mechanical failure modes have been identified and discussed using macroscopic observations supported by high-definition imagery.

The results have shown that thermal exposure and fibre orientation influences the post-fire tensile behaviour of CFRP specimens. On average, the CFRP specimens exhibited a 46% loss of load-carrying capacity between their undamaged and post-fire state; this reduction was attributed to the pyrolysis of the epoxy matrix

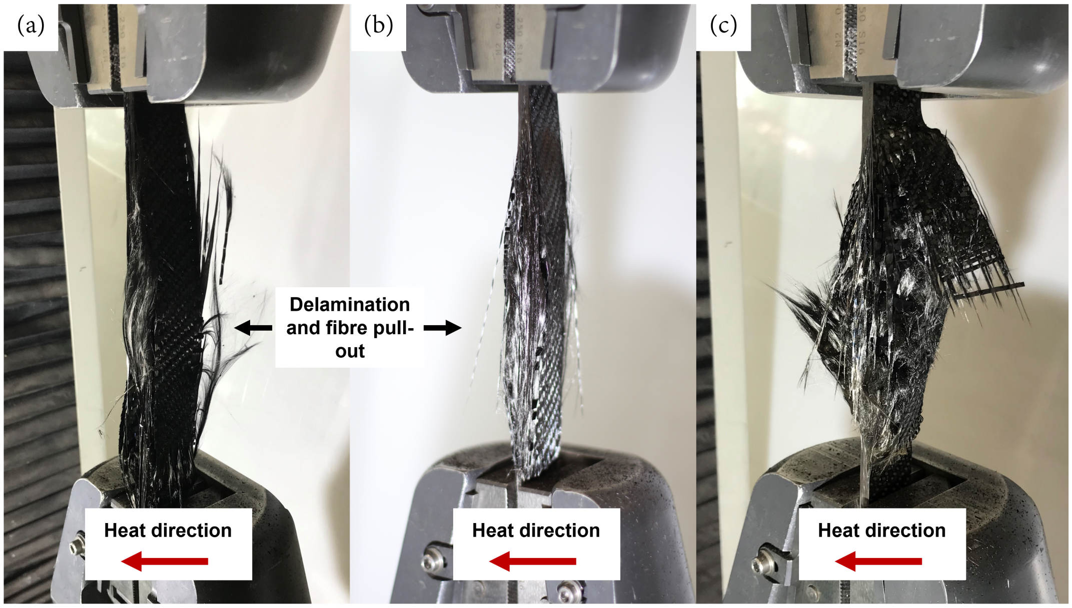

Figure 11: Photographs showing the tensile failure mode of the thermally exposed (a) S1, (b) S2 and (c) S3 CFRP specimens.

and subsequent loss of interfacial bond strength. The results have also shown that the S1 CFRP specimens present maximum performance before and after thermal exposure due to the unidirectional nature of the S1 CFRP specimens. At the same time, the S3 CFRP laminate shows the lowest performance before and after thermal exposure. This behaviour, therefore, elucidates that the epoxy matrix plays a less significant role in supporting and redistributing the tensile load across the fibres in unidirectional CFRP specimens after thermal exposure than it does for CFRP specimens containing bidirectional and multidirectional fibre orientations. The results also showed that the tensile failure modes are sensitive to thermal irradiation. In their undamaged state, failure modes of the CFRP specimens appear to be sudden and brittle, consisting of a large longitudinal (S1) or transverse (S2, S3) crack. However, after exposure, the failure can be characterised as explosive due to a large amount of stored energy released simultaneously, irrespective of the carbon fibre orientation.

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