Non-destructive evaluation of localised heat damage occurring in carbon composites using thermography and thermal diffusivity measurement

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A B S T R A C T
Carbon fibre reinforced polymer (CFRP) composites are now a common occurrence in the aerospace sector especially with their introduction into the aero-engine. With their ever-increasing use in harsh and extreme environments, it is important to understand their behaviour and performance when exposed to such working conditions. This paper presents a novel approach to understand the thermal degradation mechanism of CFRP composites based on the measurement of thermal diffusivity through non-destructive testing (NDT) when exposed to heat in a localised area. The study explored the suitability of pulsed thermography in detecting the physical damage caused due to localised heat exposure together with its ability to identify chemical change that occurs during the initial stages of exposure. The results showed the ability of thermography in detecting discolouration damage through the use of diffusivity measurements acquired by reconstructing the thermography dataset.

1. Introduction

The technological advancements in the area of advanced manufacturing over the last century have been a major driver for the introduction of advanced materials in the aerospace and automotive sectors. The aerospace industry has always been regarding the need for structural materials that are less dense, high in strength and are impact – abrasion – corrosion resistant. Carbon fibre reinforced polymers (CFRP) parts have now become the front runner due to their improved strength to weight ratio [1,2]. However, these composite materials are relatively new and their material properties are not fully defined. In fact, the earliest evidence of the introduction of CFRP in civil aviation applications came in the 70’s with steady progress to specific application: such as primary airframe structure, in the late 90’s [3]. Though these materials provide a better alternative to traditional metal counterparts, a number of limitations ranging from damage detection and characterisation for damage such as delaminations, disbands, barely visible impact damage (BVID), water ingress and their repairability in service continue to be a challenge to the industry [4]. It must be understood that, the main reason for the uncertainty in the behaviour of the laminate with defects and damage under load is that fact that they are heterogeneous and anisotropic in nature and thus differ from their metallic counterparts.

1.1. Background

Literature indicated that the previous attempts to investigate damage created due to heat exposure was mainly in the 90's with more recent papers focussing on non-destructive evaluation (NDE) methods [5,6]. One of the major focuses of this paper is looking at the degradation process that occurs during the exposure of extreme temperature in a localised area and the laminates’ response to such conditions. Research indicated that most of these studies look at uniform heat damage investigation with very limited information on the behaviour of CFRP when exposed locally [7–9]. Heat damage typically could occur as a result of fire or during service operations where they can be exposed to hot gasses, lightning strike, jet engine efflux during landing and exhaust from the jet engine [5,10]. Most of these degradations occur in localised areas and could go undetected. Thus, it is pivotal to understand and characterise such damage to prevent any catastrophic failure.

1.2. Heat damage

The damage created due to high temperature exposure on a composite material is referred to as heat damage and is a result of irreversible thermodynamic change that results in a physical and or chemical change. Studies have indicated that there are
two major forms of damage which include incipient and physical damage [5,6]. Physical damage is characterised by various forms of cracks, disbonds, delaminations and blisters. They are created when the exposure temperature exceeds the materials glass transition temperature ($T_g$) [5]. On the other hand, incipient heat damage may not show any characteristic physical features that can be visually detected as they are created at a lower temperature exposure [10].

The damage creation is dependent on the selected heat source, the rate and amount of energy applied and the mode of application; conduction, convection or radiation [5,9,10]. In order to characterise damage, it is important to understand the impact of various factors such as material properties, impact of environment, the degradation process and the characteristic features of the damage created.

The performance of composites is primarily dependent on the material constituents such as the matrix composition, the type of resin and lay-up where the manufacturing processes determine the material characteristics of the laminate. Though the resin is non-conducting in nature, it can be understood that heat propagates through the fibres, them being conductive and thus their orientation and volume fraction control the thermal properties of the laminate, especially the carbon – graphene based composites [11,12]. Optimum curing is necessary as either under or over curing could result in a large variation in the laminates thermal property [13]. Studies indicate that moisture absorption over time is a major factor that contributes to environmental degradation where it alters the laminates durability leading to change in mechanical, chemical and thermo-physical properties [14]. With the presence of moisture, plasticisation can occur when exposed to extreme temperatures leading to permanent deformation. In addition to moisture, ultraviolet or UV degradation, foreign object contamination (chemical and biological), impact damage and fatigue are other environmental factors that affect the properties of CFRP laminates [14,15].

1.2.1. Thermal degradation process

There is significant evidence that the degradation attacks the matrix first, it being the most sensitive and reactive to heat and then the fibres [16–18]. On extended heat exposure, the resin starts degrading resulting in material oxidation and or pyrolysis which includes processes ranging from gas decomposition through to charring of the laminate [17].

Chemical degradation is mainly resin dominated and occurs before the formation of any physical damage such as cracks, blisters and delamination. Exposure to high temperatures results in depolymerisation, random chain scission, side group elimination and carbonisation of the composite. The initial exposure oxidises the matrix and can be characterised by a visible surface discolouration and is a result of cross-linking process that contributes to the release of volatile gases. Extended heat exposure further leads to charring as a result of resin pyrolysis [19,20]. It was reported that mass variation characterisation through Thermogravimetric Analysis recorded a mass loss due to volatilisation of the monomer confirming the loss of overall mass due to chemical degradation [21]. On the other hand, the physical irreversible damage is a result of rapid change through the glass transition temperature due to thermodynamic inequilibrium. It should be understood that thermal conductivity of the material plays a major role in the heat diffusion process especially during solidification and leads to change in the material density of the laminate creating free volume, residual mechanical stress and excess energy in the matrix [22].

1.2.2. Damage detection and characterisation using NDT

NDT techniques are those methods that determine the health of the component without causing further damage to the part being inspected [23]. As discussed, in the case of heat damage, NDT techniques such as thermography, ultrasonic testing and spectroscopy exist which provide an understanding of the damage occurring as a result of extreme temperature exposure [10]. Pulsed thermography has proven advantage of fast inspection with specific advantage of detecting defects and damage occurring in CFRP composites [24,25]. Studies also indicated that thermography and ultrasonic testing are not sensitive enough to detect incipient heat damage where there is minimal or no physical damage that is easily detectable with this technique with the diffuse reflectance infrared Fourier transform spectroscopy or DRIFT being the only other technique capable of monitoring chemical changes [5,10]. Where techniques like Fourier transform infrared spectroscopy (FTIR) are capable of detecting chemical damage, the ready and robust nature of pulsed thermography with additional suitability aspects such as large area ‘in-field’ inspection scenarios make it a more attractive alternative technology. The present study thus establishes the applicability of pulsed thermography in detecting discolouration damage through the use of surface diffusion measurements reconstructed from the acquired thermal data.

2. Methods & materials

This paper presents the research taken up to identify, classify and understand the characteristics of heat damage occurring in composites on a local area and presenting not just a damage creation method, but the establishment of a competitive technology to detect and establish incipient damage. For the scope of this study, a parametric experimental study was introduced to obtain repeatable and reliable results at the damage creation, damage detection and analysis stages. The local heat exposure was initially characterised by selecting a temperature range, based on the laminates response to the glass transition temperature of 195 °C and identified through numerous preliminary tests [26]. Based on the selected temperature range, multiple sets of experimentation were carried out to determine the effect of different exposure temperatures on degradation process over time. The following subsections clearly define the experimental specifications and plan.

2.1. Sample manufacture

For this study, HexPly M21/T800H unidirectional carbon-epoxy prepreg was chosen due to its good tensile strength of 2860 MPa and a high glass transition temperature of 195 °C [26]. The laminates were manufactured in a traditional autoclaving process to a quasi-isotropic layup to acquire 30 samples of $150 \times 100 \times 4$ mm. The sample thickness of $4$ mm was selected based on the requirements for the short beam test as per the standard ISO 14130.

2.2. The experimental plan

Composite laminates post manufacture were carefully marked for repeatability and weighed to capture the weight after manufacture. They were then inspected for any surface inconsistencies and the same was recorded together with a pulsed thermography inspection of both sides of the sample. They were then exposed to localised heat using a hot air heat gun to achieve various levels of damage. The samples were subjected to visual inspection, pulsed thermography and was followed by ultrasonic immersion test to confirm damage. It should be noted that there was at least one ‘as-manufactured’ sample as a reference for all tests and 3 repeat samples with the same level of degradation were used for each test to achieve repeatability. Based on preliminary tests the following test routines were established;
Experiment 1: Set exposure temperature of 386.2 °C (Medium Heat Exposure) and exposure time of 2, 5 and 30 min respectively (Time to initial crack: 5 min).

Experiment 2: Varying exposure temperatures of 340.8 °C (Low Heat Exposure) for 220 min and 473.2 °C (High Heat Exposure) for 0.25 min, based on time to initial crack.

For ease of understanding the following nomenclature of samples will be used for the entirety of the paper (see Table 1). It should be noted that the exposure temperatures indicated above were measured using a calibrated infrared radiometer due to the uncertainty associated with the indicated temperature on the hot air gun. The random error was also calculated and averaged across all thirty exposures, and the exposure temperature was recorded from the reflected temperature on the heat exposed area of the laminate at the time of exposure.

2.3. Experimental setup

In order to maintain repeated presentation of the part, the rig was setup as seen in Fig. 1. The setup consisted of a heat gun with electronic temperature controller; two custom made holders set on the stand, the height of which was set using a Vernier height gauge and with an accuracy of ±0.02 mm. The holder was designed to match the precise location of the sample and maintain repeatability. For the selection of a suitable heat source a range of heat sources were explored during the research and included systems that used laser, infrared and resistive heating methods. With an approach to represent an in-field scenario, a hot air gun was finalised as the heat source.

2.4. Non-Destructive testing techniques

The following non-destructive evaluation techniques were used to detect and quantify damage

- Visual inspection using a digital camera and an optical microscope.

### Table 1

<table>
<thead>
<tr>
<th>Name</th>
<th>Heat exposure (°C)</th>
<th>Exposure time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>No heat exposure</td>
<td></td>
</tr>
<tr>
<td>M2</td>
<td>386.2</td>
<td>2</td>
</tr>
<tr>
<td>M5</td>
<td>386.2</td>
<td>5</td>
</tr>
<tr>
<td>M30</td>
<td>340.8</td>
<td>30</td>
</tr>
<tr>
<td>L220</td>
<td>340.8</td>
<td>220</td>
</tr>
<tr>
<td>H1</td>
<td>473.2</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Pulsed thermography inspection was performed using ThermoScopell® which included two flash lamps (25KJ nominal), a computer control unit and a cooled, quantum detector based infrared radiometer, FLIR SC7600MB, with a maximal resolution of 640 × 512 pixels. The flash pulse length of 10 ms (ms) was set to achieve the required rise in surface temperature corresponding to a 2K (nominal) surface energy exposure over an area of 250 × 200 mm [24]. All data was captured at an acquisition framerate of 25 Hz over a time of 20 s (500 frames in total).

- The ultrasonic testing (UT) was conducted for damage validation using a pulse-echo double transmission immersion system that used a Techsonic 5 MHz probe with a diameter of 5in. The water path was set to 75 mm to the reference reflector glass plate with a resolution of 1 × 1 mm.

2.5. Thermal diffusivity measurement

To better reveal the change of material property due to the exposed heat, this paper proposes to use the surface thermal diffusivity map estimated from the inspection data obtained using pulsed thermography. In pulsed thermographic inspection, a short and high energy light pulse is projected onto the sample surface using flash lamps. Heat conduction then takes place from the heated surface to the interior of the sample, leading to a continuous decrease of the surface temperature. An infrared camera controlled by a PC captures the time-dependent response of the sample’s surface temperature. The time when the temperature deviation occurs is used to estimate the thickness (if thermal diffusivity is known) or the thermal diffusivity (if the thickness is known) of local materials. The surface temperature due to the back-wall at depth L for a homogeneous plate is given by [27]

\[
T(0, t) = \frac{Q}{\sqrt{\pi \rho c k t}} \left[ 1 + 2 \sum_{n=1}^{\infty} R^n \exp \left( -\frac{n^2 L^2}{2kL} \right) \right]
\]

(1)

where \(T(0, t)\) is the temperature variation of the surface at time \(t\), \(Q\) is the pulse energy, \(\rho\) is the material density, \(c\) is the heat capacity, \(k\) is the thermal conductivity of the material, \(R\) is the thermal reflectivity of the air gap interface, and \(x\) is the thermal diffusivity.

A linear relation in the logarithmic domain with slope –0.5 as Eq. (2) exists for time and temperature if both sides of Eq. (1) are applied by the logarithmic operation [28].

\[
\ln[\ln(T(t))] = \ln \left[ \frac{Q}{\sqrt{\pi \rho c k t}} \right] - 0.5 \ln(t)
\]

(2)

The temperature response of any change in thermal material property from structure, damage or defect will derivate from the linear response. Shepard [29] proposed a Thermal Signal Reconstruction (TSR) technique to reduce temporal noise using a high order polynomial model to fit the temperature cooling curve. The model can be written as

\[
\ln[T(t)] = \sum_{i=0}^{N} a_i [\ln(t)]^i
\]

(3)

where \(T(t)\) is the surface temperature at time \(t\), \(N\) is the model order, and \(a_i\) are coefficients to be estimated. Once the unknown coefficients \(a_i\) are estimated by the least square method, the temperature behaviour can be reconstructed to replace the raw data. The first and second derivatives of \(\ln[T(t)]\) with respect to \(\ln(t)\) can be calculated from the estimated coefficients directly, expressed as

\[
\frac{d[\ln[T(t)]]}{d[\ln(t)]} = \sum_{i=1}^{N} a_i i [\ln(t)]^{i-1}
\]

(4)
\[
\frac{d^2 \ln \left[ T(t) \right]}{dt^2} = \sum_{i=2}^{N} a_i \cdot (i - 1) \cdot \left[ \ln(t) \right]^{-2}
\]

Shepard [29] proposed a Log Second Derivative (LSD) method to estimate the depth of defect or thickness of sample by

\[
L = \sqrt{t_{LSD} \cdot \pi \cdot \alpha}
\]

where \(t_{LSD}\) is the peak time of the second derivative if the thermal diffusivity \(\alpha\) is known. If the thickness of the sample is known, the thermal diffusivity can be estimated by [30]

\[
\alpha = \frac{t^2}{t_{LSD} \cdot \pi}
\]

3. Results

As described in the methodology section, various tests were conducted before and after heat exposure. The following are the results obtained using various NDT and mechanical tests.

3.1. Visual inspection

As described previously, the samples were exposed to two different sets of experiments. The set one had a set medium temperature of 386.2 °C with exposure durations set to 2, 5 and 30 min and set two was exposed to low and high heat conditions. The visual and microscopic indications obtained from set one are presented in Fig. 2.

All samples were subjected to pulsed thermographic inspection before and after heat exposure as mentioned in the methodology. The inspection was performed as described by Zhao et al. [25]. The following is the traditional RAW thermal image obtained for the medium heat 30-minute exposed laminate sampled at 0.28 s from flash.

Fig. 4 clearly picked up the damage caused due to heat exposure, which is also visible from a normalised line profile plot. However, it did not reveal the circular pattern as seen in the digital image (Fig. 2(c)). Also, the sample M2 showed discoulouration visually and did not show any thermal response to the discoulouration as presented by Fig. 5.

Fig. 6 shows the results of estimated surface diffusivity maps of the considered samples.

It can be inferred from Fig. 6 that the medium heat exposed laminates clearly picked up damage caused for the initial crack (at 5 min) and extended char (30 min), but did not show major change in terms of detecting the incipient damage characterised by visible discoulouration. Hence a statistical approach was adopted where histograms were plotted from multiple trials. Fig. 7 shows the results obtained from statistical analysis, where the histogram of estimated thermal diffusivity values is plotted. The ‘as-is’ laminate with no damage achieved an approximate Gaussian distribution confirming the thermal diffusivity of the laminate. As expected, this distribution changed owing to the presence of damage, with changes occurring even for the discoulouration damage. This is evidenced by higher sigma values (>0.0081 mm²/s) corresponding to the laminate heat exposure conditions. It was also observed that the sigma value increases with increase in exposure time (0.0101, 0.0112 and 0.0117 mm²/s), indicating a larger damage area evidenced by the deviation in the thermal diffusivity of the laminate.

Though neither the thermograms nor the diffusivity maps presented any change associated with discoulouration, the histogram plot readily confirmed the presence of surface anomaly. Assuming that non-exposed or virgin laminates exhibited a normal Gaussian distribution when plotted for thermal diffusivity, the distribution completely changed for the 5 min and the 30 min exposure conditions where significant change in both the peak and coverage area

Fig. 2. Visual inspection results for laminates exposed to medium heat exposure; Digital and microscopic images: (a) (d) M2, (b) (e) M5, and (c) (f) M30.

Fig. 3. Visual inspection results for laminates exposed to varying heat exposure; Digital and microscopic images: (a) (c) L220, (b) (d) H1.

temperature of 473.2 °C exceeding the glass transition temperature of 195 °C. Based on experimental trials, it can be reported that a temperature exposure less than 340 °C did not yield any physical damage for exposure time exceeding 480 min.

3.2. Pulsed thermography inspection

All samples were subjected to pulsed thermographic inspection before and after heat exposure as mentioned in the methodology. The inspection was performed as described by Zhao et al. [25]. The following is the traditional RAW thermal image obtained for the medium heat 30-minute exposed laminate sampled at 0.28 s from flash.
Fig. 4. RAW thermal image of the laminate M30 sampled at 0.28 s (left) and the normalised thermal line profile plot (right).

Fig. 5. RAW thermal image of the laminate exposed to medium heat for 2 min; and sampled at 0.28 s (left) and the normalised line profile plot (right).

Fig. 6. Surface diffusivity maps reconstructed for an area of 100 × 100 pixels using the LSD method for the laminate exposed to medium heat for (a) S1, (b) M2, (c) M5 and (d) M30.
were detected with additional tailing for higher diffusivity values exceeding that of 0.48 mm²/s as indicated by the virgin plate. It can be established from Fig. 7 that the laminates exposed at a medium heat for a 2 min exposure shifted the peak and the coverage area (also evidenced by the increase in sigma value to 0.0101 mm²/s) and thus it does not follow the Gaussian distribution any more. Thus, pulsed thermography clearly picked up discolouration through statistical analysis.

Again, the immersion UT results confirmed the presence of both the initial and extended cracks but failed to detect the discolouration damage other than presenting a very minor amplitude change near the exposed area as seen from Fig. 8(a) above.

A similar analysis of the laminates exposed to low and high temperature exposure conditions clearly picked up damage on the laminates (Fig. 9). The diffusion map for the high heat sample represented data similar to the medium heat exposure laminates. This is due to the fact that the crack formed instantaneously within the first minute (0.25 min) indicating that the high stress to the resin fibre matrix caused the crack to appear straightaway, leaving limited or no time to undergo the extended degradation process of discolouration and charring. However, the low heat 220 min exposure caused severe localised damage as confirmed by low extended peak from the histograms. This result was found to be in line with the original literature that the degree and the type of damage formed is related with both exposure temperature (L220, H1) and time (M2, M5 & M30).

It was observed that both physical and surface breaking damage were identified by both pulsed thermography and UT. Additionally, the data presented new insights that pulsed thermography revealed discolouration or incipient damage through statistical analysis. It should be understood that this chemical damage has only been picked up either visually or through FTIR as found in the literature [31]. Whilst the strength of the laminate in response to physical damage can be measured, it is a huge challenge to identify discolouration or incipient damage on a large structure,

4. Discussion

The main aim of this study was to understand the thermal degradation process when a CFRP laminate was exposed to heat on a local area. A majority of the research, as indicated in the introductory sections, has been in the area of uniform heat exposure where the laminates were fully exposed to set temperatures. This work presents the findings of an alternate approach to understand the degradation mechanism especially when the heat exposure is localised. All experiments undertaken confirm the CFRP laminate’s degradation process as found in the literature, with supporting evidences presented through NDT measurements. The degradation process is in line with the original literature that the degree and the type of damage formed is related with both exposure temperature (L220, H1) and time (M2, M5 & M30).

It was observed that both physical and surface breaking damage were identified by both pulsed thermography and UT. Additionally, the data presented new insights that pulsed thermography revealed discolouration or incipient damage through statistical analysis. It should be understood that this chemical damage has only been picked up either visually or through FTIR as found in the literature [31]. Whilst the strength of the laminate in response to physical damage can be measured, it is a huge challenge to identify discolouration or incipient damage on a large structure,
let alone the ability to establish its loss in performance. This paper thus puts forth a pattern recognition method that not just identifies chemical damage, but also provides a benchmark solution that can be applied to large scale structures. The work presented is only an initial effort to promote the applicability of pulsed thermography to detect chemical or compositional change occurring in composites exposed to harsh environmental conditions, and more in-depth study is needed to improve the serviceability of such advanced structures.

The statistical analysis assumed a Gaussian distribution as a reference to understand the ideal behaviour of the ‘as-is’ laminate. The change in distributions as seen in Figs. 7 and 9 show that the distribution has changed which can be evidenced not just by the fitting but also the sigma values of the histograms.

It has been widely acknowledged that the accuracy of any measurement is dependent on the uncertainties associated with the measurement activity itself. Effort has been taken up to assess process level uncertainties associated with thermography under a system of a system context that identifies various uncertainties that can affect the measurement [32]. This work only included the overall measurement of thermal diffusivity under the context of damage identification with multiple trials being used to achieve repeatability. Future work is envisaged to accurately measure various levels of incipient damage with an incorporated uncertainty model.

5. Conclusion

Studies indicated that the thermal degradation process is dependent on the material, environmental condition and the heat source. It can be concluded that thermal degradation process is resin dominated around the glass transition temperature whose characteristics could be classified into three levels of damage as presented in the results sections. This study confirmed not just the thermal degradation process occurring in CFRP laminates, but also proposed a novel detection method which combined the use of both traditional material property measurement; in our case the thermal diffusivity measurement and the box standard statistical approach for damage assessment. This approach has enabled the transformation of a visual qualitative data with a firm quantitative outcome. Further, this paper advances the applicability of pulsed thermography to detect damage purely based on thermal diffusion characteristics of the material and thus removing the technique’s dependence on physical damage features.

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