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Temperature driven failure of carbon epoxy composites – A quantitative full-field study

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ABSTRACT

Aerospace composites are exposed to low temperatures that induce high levels of stress within the material. This is sufficient to induce fractures and eventually delaminations and failure. Thus, understanding how these temperature induced translaminar fractures can be reduced is an important area of research.

This work investigates cross-ply unidirectional (UD) and woven (W) carbon fibre laminates with MTM46 epoxy to assess how the cure schedule (low temperature, LTC and high temperature, HTC) effect temperature driven fractures. A novel digital image correlation technique was applied to determine in-situ fracture progression versus temperature. Thermal techniques investigated the degree of cure, resin plasticity, thermal expansion and beta transition effects.

The cure schedule for carbon epoxy laminates has a marked effect on quantity of manufacturing induced fractures and the temperature at which temperature induced internal fracture occurs. This work has demonstrated that a lower temperature cure is more robust against temperature driven fracture despite having a larger coefficient of thermal expansion (CTE) and similar levels of plasticity. Low temperatures induce high internal stresses but the residual stress resulting from high temperature cure is of greater concern.

DIC is an excellent method to determine onset and progression of translaminar fracture as well as the behaviour of composite materials subject to temperature effects. This work is of great benefit when considering the design of CFRP structures subject to low temperature loading, furthermore the data can be used to more accurately model this phenomena in future.

1. Introduction

Unidirectional (UD) prepreg carbon fibre reinforced polymers (CFRP) possess superior in plane mechanical properties to woven systems and offer large weight savings in mass critical sectors such as aerospace. UD prepregs are inherently anisotropic possessing fibre dominated longitudinal strengths of greater than 2000 MPa but comparatively poor resin dominated transverse strengths of approximately 30 MPa [1]. This, in combination with the widely differing longitudinal (-0.3×10^{-6} °C^{-1}) and transverse (30×10^{-6} °C^{-1}) linear coefficient of thermal expansion (CTE) [2] leads to residual stress within the laminate which can surpass the transverse strength of the ply leading to transverse crack formation [3–6].

These cracks reduce the modulus of the laminate [4,7], act as stress concentrators, provide a pathway to freeze thaw cracking [8,9]. Consequently, the management of residual stress in cross ply laminates is critical in aerospace CFRP components which operate at −56.5 °C at an altitude of 12 km [10]. Potential solutions include the use of off axis fibres laminated to introduce compliance and manage out of plane loading. Another option is the use of toughening agents within the epoxy matrix to limit the residual stress within the laminate [4] and increase the thermal stability [11,12], however these do not fully prevent microcracking. Other researchers have demonstrated that a reduction in the degree of cure (α) for an epoxy system has been shown to increase fracture toughness from approximately 1 to 1.25 K_I/K^o_I by reducing the α

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from 100 to 60% [13]. Within a composite system the optimum epoxy degree of cure has been shown to depend on the mechanical property being measured [14,15]. For a Fiberite T300/976 carbon epoxy system the 0° tensile modulus varied from 172.4, 179.3, 182.7 to 175.8 MPa with a α of 60, 70, 80 and 90% respectively [14]. Furthermore, reducing degree of cure has been shown to significantly lower residual stress within the laminate [6,16].

Epoxy systems possess a low temperature energy dissipation mechanism, typically over the range −120 to 20 °C [17,18] which are known as a beta transition. These beta transitions correspond to the reduction in molecular mobility at low temperatures and have been shown to play a role in the toughness of the epoxy system [17,19]. There is no evidence of a link between beta transition and fracture toughness although it is known to cause a reduction in plasticity and is therefore suggested to have an effect on low temperature behaviour. This work will investigate the low temperature behaviour of a commercial epoxy and assess if it exhibits any changes in resin plasticity with respect to temperature. It will also assess if there is a link between the temperature at which composite fracture is induced and the reduction in molecular mobility of the epoxy system at sub ambient temperatures.

Fractures in CFRP have been analysed using a wide array of techniques such as optical microscopy [6,20–22], acoustic emission [5], speckle pattern interferometry [23,24] and in-situ microscopy [3,7], digital image correlation (DIC) [25] and X-ray tomography [20,25] to name a few. Thermally induced fractures in CFRP are either a post fracture analysis, which enables a quantification of fractures or in situ fracture analysis gathering fracture temperatures and/or fracture quantification. Optical microscopy [6,20–22] and X-ray tomography [20] have been used as post fracture techniques for analysis of thermally stressed laminates. In situ methods for low temperature loading of samples have included optical microscopy [3,26,27] acoustic emission [28] and dynamic mechanical analysis [29]. These low temperature in situ techniques offer information of the fracture temperature and in the case of optical microscopy can offer quantification of cracks, but they are analytically intensive and produce limited information on the fracture progression. DIC is a simpler route to gathering rich data on the development of laminate cracks. DIC analyses the changes in sequential images of a textured surface during deformation to produce a surface strain field. This enables time based visualisation and quantification of crack formation. It has been used by many researchers to measure the crack length and opening displacement profile in a range of materials [30].

This work presents a novel in-situ approach for analysing thermally induced crack formation in CFRP laminates via the application of DIC. The data rich nature of the DIC technique enables formation of the crack opening displacement (COD) via a custom Matlab script, providing a detailed analysis of translaminar fracture and delamination in UD and woven CFRP samples. The work will utilise the results of this novel application to analyse the effects of degree of cure on the laminates resilience against thermally induced fracture.

2. Experimental

2.1. Materials and manufacture

Samples were manufactured using two pre-impregnated fabrics: a 660 gsm unidirectional (UD) with Pyrofil™ HR40 (Misubishi Rayon Carbon) fibres and medium temperature moulding MTM46 (Cytec ltd) resin and a 660 gsm 2 × 2 twill with HTA40 (Toho Tenax) fibres also with MTM46. Samples were cured with either a high temperature cure (HTC), maximum 180 °C or a low temperature cure (LTC), maximum 120 °C as defined in Table 1 [31]. The LTC cure represents a part cured from low temperature tooling in which a cooling step is required to remove the tool before final post-cure whilst, the HTC represents a more traditional high temperature tooling typically used in aerospace components. 400 mm × 400 mm panels were laid up on a 770NC Frekote (Henkel, Germany) mould released aluminium sheet. Debulking was performed every four plies for 15 min at room temperature (20 °C) to aid consolidation. Samples were then cured under vacuum using either the LTC or HTC schedule.

Table 1
Low and high temperature cure cycles.

Low temperature cure (LTC)	High temperature cure (HTC)
Ramp at 2 °C/min to 80 °C	Ramp at 2 °C/min to 135 °C
80 °C Dwell for 300 min	135 °C Dwell for 90 min
Ramp at 2 °C/min to 20 °C	Ramp at 0.3 °C/min to 180 °C
Ramp at 2 °C/min to 80 °C	180 °C dwell for 60 min
Ramp at 0.3 °C/min to 120 °C	Ramp at 3 °C/min to 20 °C
120 °C dwell for 120 min	
Ramp at 3 °C/min to 20 °C	

Two layups were used for cryogenic fracture analysis, one composed solely of UD fibres and the other a typical aerospace layup designed to be robust against temperature driven fracture with a UD central core surrounded by woven material. A UD layup of $[90_3, 0]_s$ was chosen as previous work has shown it to be particularly susceptible to temperature driven fracture [3]. The UD samples were designated UD-LTC and UD-HTC with a cured laminate thickness of 5.2 ± 0.1 mm. The woven/UD layup used the following schedule [±45 W, 0/90 W, 0,90 W, 0 UD, 0/90 W, 0/90 W, ±45 W] where W corresponds with woven plies. The resulting cured laminates were designated W-LTC and W-HTC with a cured laminate thickness of 5.1 ± 0.1 mm.

For thermal expansion measurements, panels of 10 off 0° plies measuring 100 mm × 100 mm of the UD and woven MTM46 were laminated using the same procedure as above. Each panel was cured using the LTC and HTC schedule and designated UD HTC CTE, UD LTC CTE, WHTC CTE and WLTC CTE laminate.

2.2. Differential scanning calorimetry (DSC)

Degree of cure (α) was determined using a differential scanning calorimeter (DSC1 - Mettler Toledo Ltd, UK) equipped with an automatic sample changer. Samples of uncured MTM46 HR40 prepreg, UD LTC and UD HTC panels were powdered using a cryomill (Retsch, Germany) to produce a homogeneous powder. 5–10 mg of each sample were sealed in 40 μL aluminium pans with a pierced lid. Samples were isothermally held for 2 h at 250 °C until the samples had fully cured. The enthalpy of cure was determined via integration of the normalised heat flow with respect to time over the first hour using Origin (OriginLabs Corp., USA). α was calculated using the formula below [32]:

$$\alpha = \frac{(\Delta H_t - \Delta H)}{\Delta H_t} \times 100 \quad (1)$$

Where ΔH_t is the total enthalpy of reaction in $J.g^{-1}$ from the uncured prepreg, whilst ΔH is the enthalpy of reaction.

2.3. Coefficient of thermal expansion (CTE)

Samples were cut from the UD HTC CTE, UD LTC CTE, WHTC CTE and WLTC CTE panels using a diamond-coated blade along the fibre axis into 5 mm × 5 mm x 5 mm cubes with a linear precision saw (Isomet 5000–Buehler, Ltd). Two samples from each panel were polished using 1200 grit carbide paper followed by a 9 μm and 6 μm