EFFECT OF THE MANUFACTURING PROCESS ON THE INTERLAMINAR FRACTURE TOUGHNESS OF 2/2 TWILL WEAVE FABRIC CARBON/EPOXY COMPOSITES

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ABSTRACT

A 2/2 twill weave fabric carbon fibre reinforced epoxy matrix composite MTM56/CF0300 was used to investigate the effect of different manufacturing processes on the interlaminar fracture toughness. Double cantilever beam tests were performed on composites manufactured by hot press, autoclave and ‘Quickstep’ processes. The ‘Quickstep’ process was recently developed in Perth, Western Australia for the manufacture of advanced composite components. The values of the mode I critical strain energy release rate ($G_{IC}$) were compared and the results showed that the composite specimens manufactured by the autoclave and the ‘Quickstep’ process had much higher interlaminar fracture toughness than the specimen produced by the hot press. When compared to specimens manufactured by the hot press, the interlaminar fracture toughness values of the Quickstep and autoclave samples were 38% and 49% higher respectively. The ‘Quickstep’ process produced composite specimens that had comparable interlaminar fracture toughness to autoclave manufactured composites. Scanning electron microscopy (SEM) was employed to study the topography of the mode I interlaminar fracture surface and dynamic mechanical analysis (DMA) was performed to investigate the fibre/matrix interphase. SEM micrography and DMA spectra indicated that autoclave and ‘Quickstep’ produced composites with stronger fibre/matrix adhesion than hot press.

1. INTRODUCTION

The use of textile fabric reinforced composites in the aerospace, automotive and marine industries has increased substantially in the past two decades. Woven fabric composites have gained great attention due to their superior reinforcing properties, ease of handling and well established textile technologies[1]. Predicting the mechanical behaviour of these woven fabric composites is essential for structural design. Interlaminar fracture or delamination is one of the most important failure modes limiting the application of fibre reinforced composites. It may be caused by low velocity impact, manufacturing defects, lightning, and even bird strikes, but can incur a dramatic loss of residual compression strength of up to 60%[2]. The double cantilever beam (DCB) test is widely used to measure the interlaminar fracture toughness of mode I crack deformation not only for unidirectional composites but also for textile composites[3, 4]. Dynamic mechanical analysis (DMA) has attracted increasing attention, because it provides a sensitive and qualitative detection of the interphase region[5].

“Quickstep” is a fabrication process invented by Quickstep Technologies Pty Ltd in the 1990s and jointly developed by CSIRO (Commonwealth Scientific and Industrial Research Organization) Molecular Science for the manufacture of advanced composite components from thermoset or thermoplastic prepregs as well as wet resin/dry fibre. The process is based on a fluid-filled, balanced pressure, heated floating mould technology for the curing, partial curing and joining of composite materials.

2. EXPERIMENTAL PROCEDURES

The material used in this study was 2/2 twill weave carbon/epoxy prepreg (MTM56/CF0300) which was obtained from the Advanced Composites Group Ltd. It is based on a 120°C curing epoxy matrix resin designed for component manufacture. The prepreg was cut into sheets with dimensions of 210mm×300mm and were laid up as laminates with 16 plies. A 15 µm thick aluminium foil was treated with a release agent (Frekote 44NC) and was inserted between the centre layers prior to processing to provide the initial mid-plane delamination crack. Three manufacturing processes were employed to fabricate the composite laminates, these were hot press, autoclave and ‘Quickstep’. Each apparatus used is shown in Figure 1.

The CFRP laminates were cured under the conditions shown in Figure 2. The cure cycles for hot press and autoclave were chosen according to the manufacturer recommendations from the material data sheet of ACG MTM56. The cure cycle for Quickstep was modified to utilise the faster ramp rate of the heat transfer fluid in the ‘Quickstep’ equipment. For the cure of the MTM56/CF0300 prepreg, the ‘Quickstep’ took 27 minutes, the hot press took 56 minutes and the autoclave took 115 minutes. The ‘Quickstep’ cure cycle consumed 77% less time than the autoclave cure.
cycle and 52% less time than hot press. Except for vacuum, the autoclave also required inert gas pressure of 620kPa (see Figure 2c) to complete a cure which increases the cost of this process relative to the other two processes.

Laminates were cut into DCB specimens measuring 175mm ×20mm ×4mm via diamond-coated saw. The cutting surfaces of individual specimen were then ground to the required dimensions using a grinding machine to minimize the possible damage on the specimen edges caused by the cutting process. Aluminium blocks measuring 25mm ×20mm ×3mm with a loading hole of 6mm in diameter were bonded on the pre-cracked end to allow unrestrained rotation for DCB test.

Double cantilever beam tests were performed in accordance with the protocol of the European Structural Integrity Society [6]. The crack opening displacement (COD) was measured by crosshead displacement at a crosshead speed of 1mm/min on a LLOYD LR30K instrument. Five specimens were tested from each processing system. The interlaminar fracture toughness was determined in terms of the mode I critical strain energy release rate, $G_{IC}$, using the corrected beam theory derived by Hashemi et al.[7] (see appendix)

3. RESULTS AND DISCUSSION

The MTM56/CF0300 composites manufactured by three different processes exhibited different mode I interlaminar fracture toughness. The ‘Quickstep’ composites had 92% interlaminar fracture toughness of the autoclave composites. The autoclave composite was 49% higher and the ‘Quickstep’ composite was 38% higher than composites produced by hot press.

Typical load P versus COD δ curves of woven composites for three processes are shown in Figure 3. The P-δ response of 2/2 twill weave composite exhibited sudden crack advances. The curves showed several critical points with sudden load drops corresponding to the crack propagation. The critical loads and the crack lengths were taken at the onset of the crack propagation which causes a load drop. Three composite systems showed different propagation behavior. ‘MTM56-A’, ‘MTM56-Q’ and ‘MTM56-H’ represent the composites manufactured by autoclave, ‘Quickstep’ and hot press respectively. The MTM56-A
and the MTM56-Q needed higher load than the MTM56-H to produce the same COD which means the former were stronger than the latter. The MTM56-A experienced almost 30mm longer COD than the MTM56-Q and the MTM56-H when specimens broke in half. The time taken to complete the full scale crack propagation for the MTM56-A was 37% longer than that for the MTM56-H but 39% longer than the MTM56-Q. More load drops were found in the MTM56-A and the MTM56-Q than the MTM56-H.

Figure 4 shows the mean values of $G_{IC}$ and their standard errors for three composite systems. The scatter of $G_{IC}$ values of the MTM56-A in the typical R-curves was the highest, followed by the MTM56-Q and then the MTM56-H. The $G_{IC\text{-max}}$ of the MTM56-A was 49% higher and the $G_{IC\text{-max}}$ of the MTM56-Q was 38% higher than that of the MTM56-H respectively. The MTM56-Q had 92% of the value of the $G_{IC\text{-max}}$ of the MTM56-A. The standard errors of the MTM56-A and the MTM56-Q had intersection which means statistically they can have the same $G_{IC\text{-max}}$.

It is noted that the difference in mode I critical energy release rate $G_{IC}$ resulting from the different manufacturing processes was pronounced. In order to characterise the fracture surface of DCB specimens, scanning electron microscope was employed. Figure 5 presents the comparison of the SEM micrographs of mode I interlaminar fracture surface for three composite systems. The microtopography of three composites was quite different. When delamination starts, the crack propagates along a path where the resistance is lowest [3]. When the fibre/matrix bond is stronger than the resin around the fibre, the fracture occurs in the resin and leaves a skin of matrix over the fibres [8]. That a large amount of smooth fibres and fibre imprints stayed in epoxy resin indicated an interfacial failure of the MTM56-H where there was a weak adhesion between fibre and matrix. The crack propagated along the fibre matrix contact area. It is also easy to see that some spacing between fibre and matrix existed which was the result of poor adhesion and massive interfacial failure. By comparison, the MTM56-Q was resin matrix failure dominated. Extensive matrix deformation occurred before delamination and a large amount of resin stuck to the surface of carbon fibre indicating a strong bond between fibre and matrix.
Figure 5: SEM micrographs of mode I interlaminar fracture surface for three composite systems (the crack propagation from bottom to top, left is the fracture surface, right is the enlarged fibre surface)
  a) the hot press DCB specimen, MTM56-H  b) the 'Quickstep' DCB specimen, MTM56-Q
  c) the autoclave DCB specimen, MTM56-A
From the MTM56-A fracture surface, it can be seen that most of the fibre surface was covered by compressed conchoidal resin not only recording the high pressure performance during the consolidation process but also designating the strong bond between fibre and matrix. The enlarged images of fibre surface for three composite systems exhibit distinct adhesion of matrix to fibre. Shuttle-like matrix maintained between fibres perpendicularly to the fibre surface on the fracture surface of the MTM56-H, matrix resin ‘extruded’ from fibre surface with abundant riverbed markings in Figure 5 b) right for the MTM56-Q, and imbricate resin adhered tightly to fibre surface on the mode I fracture surface of the MTM56-A. The difference in fibre matrix adhesion for three composite systems is an interesting phenomenon and this is the subject of further investigation.

From the FTIR (Fourier transform infrared spectroscopy) spectra (Figure 6) of three cured composite systems, we can find that three processes had the same degree of cure. Figure 7 presents the dynamic mechanical analysis (DMA) spectra for three composite systems. The typical results, including storage modulus, E’ and the ratio of storage and loss moduli, tan δ as a function of temperature were recorded. The glass transition temperature, T_g (the temperature at the maximum peak on the energy dissipation, tan δ curve) for MTM56-H, MTM56-A and MTM56-Q composites were 115°C, 123°C and 132°C respectively. The MTM56-A exhibited initial storage modulus as 240GPa which was higher than the MTM56-H (22GPa) and the MTM56-Q (20GPa). After 99°C, E’ of the MTM56-Q became higher than the MTM56-H and kept this tendency until the end. Gerard et al [9] concluded that the T_g and storage modulus are lower for composites with poor interphase adhesion than those with strong interfacial bonding due to the increased mobility of polymer chains in poor adhesion composites. As a result, the MTM56-A and the MTM56-Q are believed to be the composites with stronger adhesion between fibre and matrix. It is noticeable that the MTM56-Q had the highest glass transition temperature.

Ko [10] found that with the improvement of interfaces in carbon epoxy composites the damping decreases and Chua [11] noticed that the damping at T_g decreases in proportion to the increase of the interfacial shear and transverse flexural strength for glass fibre polyester composites. The damping at T_g for three composite systems was different, following the descending sequence of MTM56-H, MTM56-Q and MTM56-A. The area under the tan δ curves has been calculated and the result shows the MTM56-H and the MTM56-Q had 9.0% and 7.2% bigger area than the MTM56-A respectively. Accordingly, the MTM56-H and the MTM56-Q were more dissipative than the MTM56-A composite.

4. CONCLUSIONS

The effect of the manufacturing processes on the interlaminar fracture toughness of carbon epoxy composite MTM56/CF0300 was investigated by the DCB test. It was found that the MTM56-Q composite had 92% of the interlaminar fracture toughness of the MTM56-A composite. Both of them were stronger than the hot press-made composite in terms of mode I interlaminar fracture toughness. The MTM56-A was 49% higher and the MTM56-Q was 38% higher than the MTM56-H.

These results indicate that manufacturing process plays an important role on the interlaminar fracture toughness. The MTM56-A and the MTM56-Q exhibited a matrix dominated failure mode; however, the MTM56-H showed an interface failure due to the poor adhesion between fibre and matrix. The MTM56-Q had a higher glass transition temperature than the other two which resulted from the decreased mobility of polymer chains in strong adhesion composite. The MTM56-H and the MTM56-Q were shown to be more dissipative than the MTM56-A. The difference in fibre matrix adhesion for three composite systems is subject to further investigation.

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![Figure 6: FTIR spectra of three composite systems after curing.](image-url)
large displacements, $G_C$ can be corrected by including a correction parameter, $F$.

$$F = 1 - \frac{3}{10} \left( \frac{\delta}{a} \right)^2 - \frac{3}{2} \left( \frac{\delta}{a_0} \right)^2$$

(2)

$N$ in Equation (1) is used to account for the stiffening of the specimen by the blocks.

$$N = 1 - \frac{9}{8} \left[ 1 - \left( \frac{L}{a} \right)^3 \right] - \frac{9}{35} \left( \frac{\delta}{a} \right)^2$$

(3)

where $t$ is the distance from the centre of the loading pin to the mid-plane of the specimen arm to which the block is attached and $L'$ is the half-length of the loading block.

REFERENCES


APPENDIX

The following equation was used to calculate the mode I critical strain energy release rate [6]:

$$G_C = \frac{3P_2\delta_C}{2b(a + \Delta)}$$

(1)

where $P_2$ and $\delta_C$ are critical loading and crack opening displacement respectively, $b$ is the specimen width, $a$ is the crack length and $\Delta$ is the crack length correction factor which is determined to be the X-axis intercept of the plot of the cube root of compliance $C(=\delta/P)$ versus the crack length $a$. When the specimen shows