

THE INFLUENCE OF PLASMA SURFACE TREATMENT ON THE FRACTURE TOUGHNESS OF PEEL PLY PREPARED BONDED COMPOSITE JOINTS

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Introduction

The increasing use of composite materials in various industries, such as aerospace, automotive and renewable energy generation, has driven a need for a greater understanding of the fracture behaviour of bonded composite joints.

An important prerequisite for the adhesive bonding of composites is the existence of a uniform surface free from contaminants and mould release agents. While there are several ways in which this may be achieved, the use of peel plies has emerged as the preferred choice for many industries due to the repeatable nature of the resulting surface, particularly in the highly regulated aerospace industry.

The use of peel plies can present some problems. It is possible that contamination from the peel ply can be transferred to the composite substrate and adversely affect the adhesive joint [1].

Plasma treatments have been shown to improve the fracture toughness of adhesively bonded composite joints [2] and can be used to remove contaminants, such as mould release agents, from the surface [3].

The aim of this work is to evaluate the influence of various peel ply treatments on the mode I fracture toughness of different aerospace grade bonded composite joints and to assess the subsequent benefits of employing an atmospheric pressure plasma (APP) surface treatment prior to adhesive bonding in each case.

Experimental

Materials & Manufacture of Bonded Specimens

A variety of aerospace grade materials were used in the present study. Two carbon-fibre/epoxy prepreps were used as substrates and will be referred to as S1 and S2. Two liquid shims (two part epoxy paste) were used as structural adhesives and will be denoted LS1 and LS2. Four commercially available peel plies, 2 dry (D1 & D2) and 2 wet (W1 & W2), were used to prepare the composite substrates prior to adhesive bonding. In total, 16 different composite joint systems were tested (2 substrates x 2 adhesives x 4 peel plies).

The composite laminates were manufactured in-house at University College Dublin using a press-clave and vacuum bagging procedure. The press-clave was heated up to 180 °C over 2 hrs and then held at 180 °C for 2 hrs under a

constant pressure of 80 psi for both composite substrates as per the manufacturer's guidelines. Once cured, specimens were cut to a size of 25mm x 150mm using a diamond grinding disc. S1 and S2 substrates were approximately 2mm and 1.7mm thick respectively. The peel ply was removed just before application of the adhesive. Bondline thickness was controlled at 0.25mm using metal spacers at either end of the specimen and also glass beads sprinkled along the joint. The joints were cured in accordance with the manufacturer's guidelines in an air-circulated oven. A special curing jig was used to ensure alignment of the substrates.

Double Cantilever Beam Test

The mode I fracture toughness, G_{IC} , of the composite joint systems was evaluated using the double cantilever beam (DCB) test. These were performed in accordance with BS7991 [4]. The propagation values of G_{IC} were calculated using a corrected beam theory method as shown in Equation (1):

$$G_{IC} = \frac{3P\delta}{2B(a + |\Delta_I|)} \cdot \frac{F}{N}, \quad (1)$$

where P is the applied load, δ the crosshead displacement, B the width of the specimen, Δ_I the crack length correction term, F the large displacement correction factor and N the load block correction factor. Three repeats were performed for each joint system.

Atmospheric Pressure Plasma Treatments

Peel ply prepared composite substrates were treated using an APP system called Labline™ [5] manufactured by Dow Corning. The Labline incorporates a dedicated reel-to-reel web handling system that passes through two vertical electrodes over which a dielectric barrier discharge He/O₂ plasma is formed. The 300mm x 320mm electrodes consist of a conductive liquid housed in a dielectric perimeter. The samples are mounted onto a poly(ethylene terephthalate) support web with double-sided adhesive tape and passed through the plasma at a constant speed of ≈ 1.5 m/min. One pass through the electrodes corresponds to a treatment time of approximately 25 seconds.

Water Contact Angle Measurements

The plasma treated substrates were characterized using water contact angle (WCA) measurements on a Data

Physics OCA 20 system. Water drops of size 1ul were placed on the surfaces using syringes. The contact angle was measured using the accompanying software.

Scanning Electron Microscopy

Micrographs of the fracture surface were taken using a Hitachi TM-1000 tabletop scanning electron microscope (SEM). Samples were cut from the fractured DCB specimen and gold coated prior to examination.

Results and Discussion

DCB Test Results

The results for the mode I fracture toughness of joints bonded with LS1 are shown in Figure 1. All joints resulted in interfacial failure. Note that there is no presented value for the propagation fracture toughness of substrate S2 prepared with peel ply W1 as the joint exhibited stick-slip fracture. Only one sample using substrate S2 and W2 peel ply resulted in stable interfacial failure. The other two samples resulted in interfacial/cohesive stick-slip failure. Initiation and arrest values for the joints exhibiting stick-slip failure can be found in Figure 2.

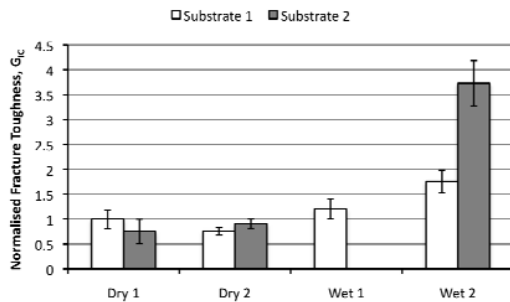


Figure 1: Normalised mode I fracture toughness of joint systems bonded with LS1.

Figure 3 presents propagation values of G_{IC} bonded with adhesive LS2. All joints prepared with the dry peel plies, regardless of substrate material, resulted in interfacial failure while those prepared with the wet peel plies gave cohesive failure, hence the similar values for fracture toughness.

SEM Analysis of Fracture Surfaces

One of the joint systems that exhibited interfacial failure was examined under SEM. The micrograph is shown in Figure 4 and shows a region where the crack propagated at the interface between the peel ply treated substrate and the adhesive layer. The imprint left on the substrate by the peel ply can be clearly seen. It appears that the adhesive does not bond to the imprint region but rather to the area between the tows of the peel ply weave.

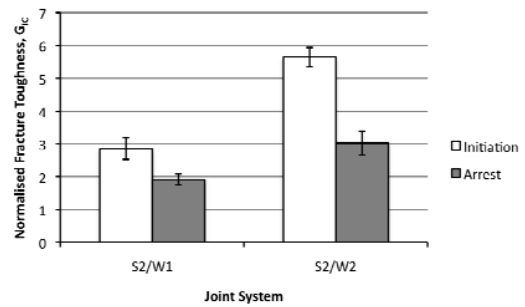


Figure 2: Initiation and arrest values for joint systems manufactured from S1, prepared with 2 wet peel plies and bonded using LS1.

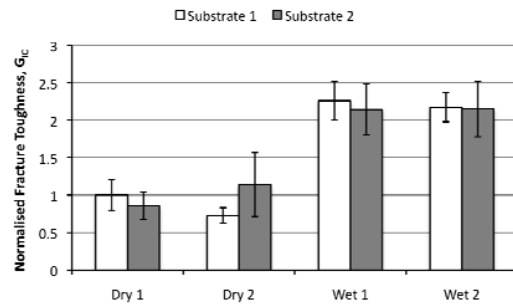


Figure 3: Normalised mode I fracture toughness of joints bonded with LS2.

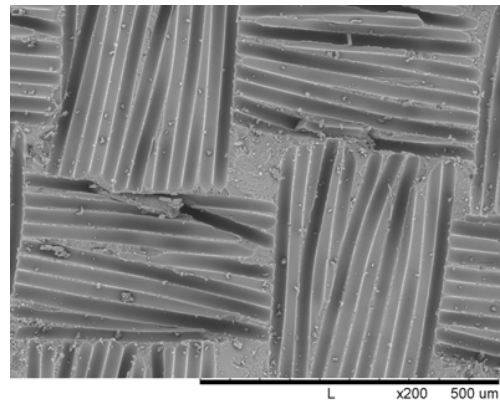


Figure 4: SEM image of substrate S1, prepared with peel ply W2 and bonded with LS1.

APP Treated Composite Joints

Two joint systems that exhibited interfacial failure were selected for plasma treatment. The systems were:

- Substrate S1 prepared with peel ply W2 and bonded with LS1. The samples were treated in a He/O₂ plasma at 1250W for 1 pass.
- Substrate S1 prepared with peel ply D2 and bonded with LS2. The samples were treated in a He/O₂ plasma at 1250W for 1, 3, 5 & 10 passes.

Figures 5 and 6 show the WCA versus time post treatment for the two substrates. The WCA was drastically reduced even after only one pass for both treated substrates. There

was some hydrophobic recovery after treatment especially for shorter treatment times. However, the WCA did not return to its original value after almost 3 weeks post treatment. It should be noted that the joints prepared in this work were bonded within a few hours after treatment.

The S1/W2 substrates were treated and bonded as outlined above using LS1. All 3 repeat specimens resulted in cohesive stick-slip failure after treatment as opposed to interfacial/cohesive stable/stick-slip failure before treatment. Fracture toughness values are presented in Figure 7.

The S1/D2 substrates bonded with LS2 gave interfacial failure before treatment. After plasma treatment there was approximately a two-fold increase in fracture toughness as can be seen in Figure 6 for all treatment times. More importantly, the locus of failure changed from interfacial to cohesive.

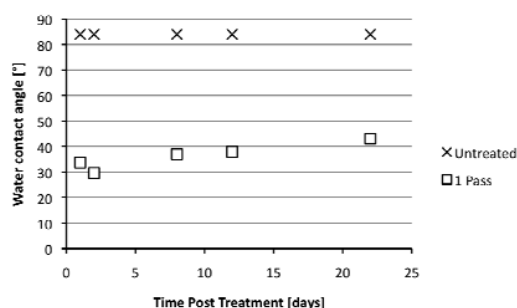


Figure 5: WCA versus time post treatment for substrate S1 prepared with peel ply W2.

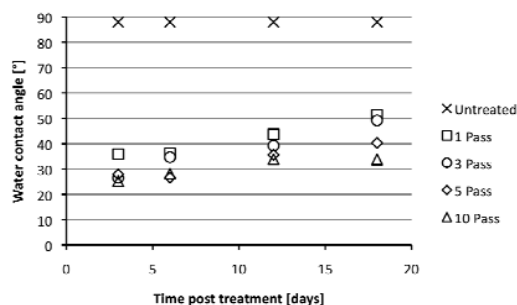


Figure 6: WCA versus time post treatment for substrate S1 prepared with peel ply D2.

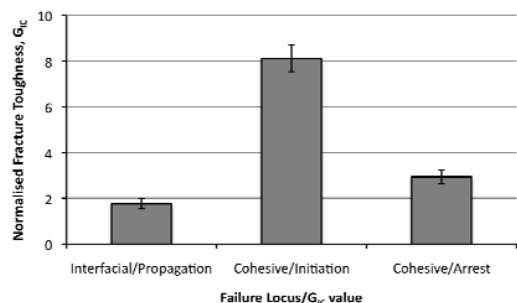


Figure 7: Fracture toughness values for treated and untreated S1/W2 substrates bonded with LS1.

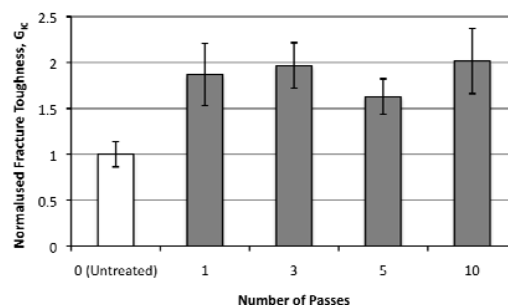


Figure 8: Normalised fracture toughness of S1 substrates prepared with D2 peel ply and bonded with LS2 subjected to APP treatment.

Conclusions & Future Work

The present work has shown that the fracture behaviour of adhesively bonded composite joint systems is highly dependent on the substrate, peel ply surface treatment and adhesive used. It is likely the contaminants left behind after peel ply removal are the cause of the interfacial failure in certain joint systems [1]. The exact nature of this contamination will be investigated in the future using x-ray photoelectron spectroscopy.

An APP treatment was shown to be extremely effective in improving the fracture toughness of the two joint systems that were investigated. It was observed that the locus of failure changed from interfacial to cohesive failure with a corresponding increase in fracture toughness.

Acknowledgements

The authors would like to gratefully acknowledge the financial support of Enterprise Ireland. The materials supplied by Bombardier Aerospace and Henkel Loctite Ltd. are also gratefully acknowledged.

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