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Surface preparation methods for thermoplastic composite welding via the Thermabond™ process

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1. Abstract

Thermoplastic composites (TPCs) are increasingly used in multiple industries. To support their widespread implementation, this study investigates a repair methodology using TPC welding. A key challenge of using this approach is achieving patch joining without melting or degrading the parent structure. A solution is proposed via the Thermabond™ process to join APC-2 carbon fibre (CF) poly ether ether ketone (PEEK) laminates using an amorphous layer of poly (ether-imide) (PEI) resin film. Welding is conducted at the PEI processing temperatures (290°C to 330°C, below the PEEK melting temperature), provided that a PEI layer is co-consolidated onto the CF/PEEK substrate surface during initial laminate manufacture. In a repair scenario, however, the layer of PEI cannot be co-consolidated onto the damaged structure using conventional processing methods (e.g., hot press or an autoclave). Thus, the objective of this work is to evaluate surface preparation strategies to introduce a PEI layer onto CF/PEEK laminates. PEI is dissolved in dichlorometane (DCM) at 10 wt% and applied on surfaces that are cleaned, abraded using Scotch-Brite™ (SB), and etched using Tetra-Etch® (TE). The specimens are joined at 0.1 MPa and at 0.8 MPa using a high-temperature oven and a hot press, respectively. These specimens are tested in single lap shear (SLS) configuration to evaluate their performance.

Baseline specimens (i.e., PEI co-consolidated on both top and bottom substrates) achieved apparent SLS strengths of 23 MPa (joined at 0.1 MPa) and 32 MPa (joined at 0.8 MPa). Specimens where the bottom substrate was abraded, etched, and coated with the DCM PEI solution achieved up to 49% of the baseline SLS strength when joined at 0.1 MPa approximately 40 hours after etching (11.2 MPa). In contrast, specimens joined at 0.8 MPa after 190 hours attained only 17% of the baseline strength (5.3 MPa), despite the higher joining pressure. These findings suggest the presence of an effective processing time window for joining after etching (between 25 and 180 hours), beyond which surface activation diminishes.

2. Introduction

Thermoplastic composites (TPCs) are attracting growing attention in multiple industries, including in aerospace [1-6]. While these materials are being increasingly used and integrated into aircraft structures [7-13], their full adoption remains limited in part by the absence of established repair methodologies. Conventional repair methods used for thermoset polymer matrix composites are inadequate as mechanical fastening adds weight to the structure and introduces stress concentrations, while adhesive bonding is challenging due to the low free surface energies and the required surface treatments of TPCs. Thermoplastic welding, initially developed to join and assemble TPCs, is also an enabling technology for TPC repair. However, its adaptation for field repair introduces unique challenges that have not been fully addressed, and thus it remains limited. Thermoplastic welding requires precise control of the interface temperature and high consolidation pressures to ensure high-quality welded parts. In addition, the challenge of scale-up of laboratory setups to production environments has not yet been fully resolved for thermoplastic welding itself. Beyond these general welding challenges, repair-specific constraints introduce additional complexity including limited accessibility in service environments and the general unavailability of suitable portable equipment.

One potential solution to address these challenges is by using TPC welding with the Thermabond™ process [14-16]. This process uses a poly ether(imide) (PEI) polymer interlayer co-consolidated onto each carbon fibre (CF) poly ether ether ketone (PEEK) substrate at the PEEK processing temperatures, enabling welding above the glass transition temperature (T_g) of PEI but below the melting temperature (T_m) of PEEK. Heimerdinger et al. [16] used resistance welding with the Thermabond™ process to perform a repair of a damaged structure, co-consolidating a PEI interlayer onto the repair patch and the heating element in an autoclave at 385°C with pressures ranging from 0.1 MPa to 0.2 MPa. The surface of the component to be repaired was first primed and etched with Tetra-Etch® (TE) prior to the application of PEI dissolved in dichloromethane or methylene chloride (DCM). TE is a fluorocarbon etchant that reacts with the TPC surface, activating it and providing free radicals to promote adhesion with PEI. In their work, however, details pertaining to the surface preparation methodology were not reported. Therefore, the objective of this work was to evaluate surface preparation strategies to enable joining of CF/PEEK substrates using the Thermabond™ process. The substrates were cleaned and treated (i.e., abraded and etched), followed by the introduction of PEI by spreading a DCM PEI solution. The specimens were then joined at 0.1 MPa using a high-temperature oven or at 0.8 MPa using a hot press, and evaluated by single lap shear (SLS) testing. Contact angles were measured to evaluate the effect of abrasion and etching, as well as the time sensitivity of the surface preparation method, correlating the results to the mechanical performance.

3. Methodology

To evaluate the effectiveness of chemical etching on the mechanical performance in shear, eight test cases were investigated as summarized in Table 1. The schematic for each case is shown in Figure 1. For this study, Scotch-Brite™ (SB) was used for surface abrasion and TE was used to

chemically etch and activate the CF/PEEK substrate surfaces prior to joining. Sheets of Ultem 1000 PEI with a thickness of 0.250 mm were cut into small squares of approximately 20 mm by 20 mm. The PEI was then weighted to target concentrations ranging from 10 wt% up to 80 wt%. Subsequently, PEI was mixed into DCM (Fisher Scientific) within closed glass jars and left for approximately one hour, while shaking the mix occasionally. A 10 wt% of PEI solution in DCM was selected, as it was the highest concentration that achieved complete dissolution.

Table 1: Test matrix to evaluate the effect of different pressures and surface treatment, treating the surface by abrading using Scotch-Brite™ (SB) and etching with Tetra-Etch® (TE), followed by application of and DCM PEI 10 wt% solution. PEI was co-consolidated (cc) on one or both substrates

Test case	Pressure [MPa]	PEI cc (bottom)	PEI cc (both sides)	SB + TE + PEI 10 wt% (bottom)	SB + TE + PEI 10 wt% (both sides)
1	0.1				
2				✓	
3					✓
4			✓		
5	0.8				
6				✓	
7					✓
8			✓		

In every test case, there was a layer of PEI co-consolidated (identified as “cc” in Table 1) on the top substrate to represent the repair patch, which can be prepared in advance and thus PEI may be co-consolidated using an autoclave or a hot press. Cases 1 to 4 were joined at 0.1 MPa using a high-temperature oven. Case 1 involved co-consolidating PEI only onto the top substrate to assess the performance penalty of not having PEI on both substrates prior to joining. Case 2 had the bottom substrate abraded using SB, etched with TE, and coated with the DCM PEI 10 wt% solution. Case 3 had both of the substrates abraded, etched, and coated with the PEI solution following the similar procedure as for Case 2. Finally, Case 4 had PEI co-consolidated on both substrates, and it was considered as the baseline for the low-pressure condition. Cases 5 to 8 had similar configurations as Cases 1 to 4, with the difference that they were joined at 0.8 MPa using a hot press. Case 8 was identified as the baseline for the high-pressure condition. The two pressure conditions are representative of current repair pressures for thermoset composites and of welding pressures, respectively.

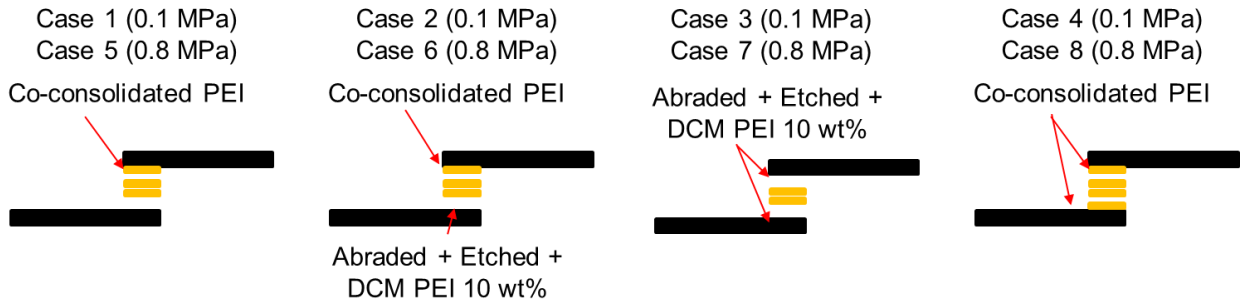

Figure 1: Test cases evaluated in this work to study the mechanical performance and effectiveness of surface treatment

3.1 Composite laminate processing

Sixteen ply APC-2 AS4 CF/PEEK laminates with stacking sequence of $[0/90]_{4s}$ were processed using compression moulding with a Wabash, 150-ton hot press at 380°C at 2 MPa for 20 minutes. Similar processing conditions were used for the laminates that had PEI co-consolidated on the surface. A schematic of the setup used to process the laminates is shown in Figure 2. Half of the processed laminates had a strip of PEI (0.127 mm thick) co-consolidated at the centre (PEI was only required at this location to join the laminates in a single lap configuration). Two layers of 0.076 mm thick Kapton® polyimide film were placed on each side of the PEI film to act as "shims". Both the Kapton® polyimide layer and the PEI resin film were placed on the tool to prevent excessive movement of these layers and to ensure consistency during the process.

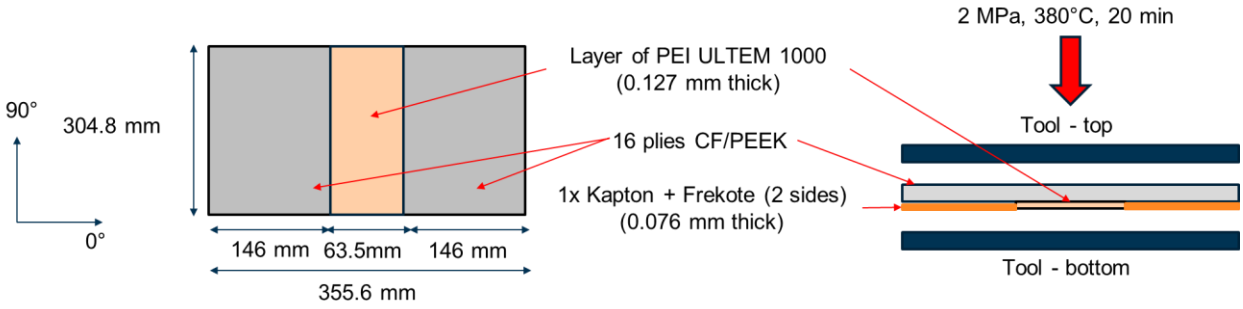

Figure 2: Schematic showing the setup and dimensions used to process the laminates used for this study. The Kapton® polyimide film and the PEI resin film were placed below the material stack

3.2 Surface preparation

The surface preparation procedure for CF/PEEK substrates was developed by extracting limited details reported in the literature [16] and adapted to ensure safe handling and repeatability. The surface was cleaned with methyl ethyl kethone (MEK) and abraded using Scotch-Brite™ pads with a pneumatic grinder. Then, the specimens were rinsed with isopropanol alcohol and dried. Etching was performed with TE for 10 minutes, followed by rinsing with hot water and cleaning with acetone to remove residual crusts. Three coats of the DCM PEI 10 wt% solution were subsequently applied, after which the specimens were left under a fume hood for at least 12 hours (as instructed by the supplier GracoRoberts). This procedure produced a chemically activated CF/PEEK surface suitable for joining using the Thermabond ™ process.

3.3 Contact angle measurements

Contact angle measurements were conducted on the surface of etched substrates to evaluate the effectiveness of the etchant and potential time sensitivity effects, as per Table 2. The specimens used for the contact angle measurements had dimensions of approximately 30 mm by 30 mm. Low surface energy thermoplastics such as CF/PEEK exhibit high contact angles, ranging from 78° to 83° [17, 18], making bonding challenging. Etching with a solution like TE chemically activates the inert surface, thus improving adhesion. A CF/PEEK specimen without any surface preparation was evaluated and considered as the baseline for these measurements. Abraded and non-abraded specimens were identified as SB and XSB, respectively, allowing the effect of abrasion on the contact angle to be assessed. Measurements were taken after different time intervals for the specimens that were abraded and etched, as indicated in the Table 2.

Table 2: Test matrix for the contact angle measurements

	CF/PEEK	**Abraded (SB)**	**Not-abraded (XSB) and etched**	**Abraded (SB) and etched**				
No. of hours	**0**	**0**	**0**	**1**	**0**	**1**	**17**	**2**	**180**

The sessile drop method was employed, where a motorized precision syringe deposited a drop of water of 2 µL on a flat CF/PEEK substrate that was laying on a platform [19]. A high-resolution camera captured the cross-section of the droplet. The equipment used was contact angle goniometer from Dataphysics, Model TBU goE. At least five measurements were taken for each condition. Once the 2 µL water droplet was deposited on the substrate (setting deposition time to one second), five seconds were allocated to allow for the droplet to reach equilibrium with the surface. After five seconds, the contact angle was measured.

3.4 Joining operation and preparation for testing

To perform the joining operation, the laminates that had the co-consolidated layer of PEI at the centre were cut, ensuring the layer of PEI was 25.4 mm wide (i.e., the overlap for the SLS specimens), as shown in the schematic in Figure 3.

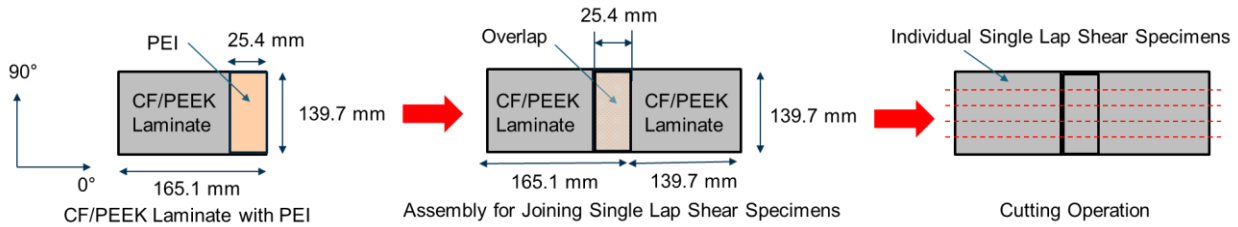

Figure 3: Schematic showing the preparation of the specimens used for the single-lap shear specimens

All laminates that had PEI co-consolidated at the surface were dried at 150°C for one hour (following similar drying conditions as per Heimerdinger et al. [16]) prior to joining. Two additional layers of PEI resin film with a thickness of 0.127 mm were introduced between the substrates to increase the resin content (as shown in Figure 2). To join the assemblies in the low-pressure condition (i.e., 0.1 MPa), a high-temperature oven with eight individual heating-controlled zones was used. An envelope vacuum bag was used over the assembly. Breather was placed on the sides to pull vacuum. The joining operation at high pressure (i.e., 0.8 MPa) was conducted using an Accudyne 15-ton hot press. For both low and high-pressure cases, temperature was increased to 300°C (below the T_m of PEEK of 343°C) and maintained for 30 minutes [15]. The assemblies were cut to obtain five specimens per case using a water-cooled diamond saw with a 2 mm thick blade at 4200 RPM. An MTS 810 machine was used with a 50 kN load cell and ASTM D1002 [8] was followed to evaluate the apparent shear strength of the specimens.

4. Results

The mechanical testing and contact angle measurements results are presented below.

4.1 Mechanical testing results

Figure 4 shows the mechanical testing results of the specimens joined under vacuum using the high-temperature oven at 0.1 MPa, and at 0.8 MPa using the hot press. At the joining pressure of 0.1 MPa, the baseline configuration with PEI co-consolidated onto both CF/PEEK substrates (Case 4) attained apparent SLS strengths of 22.7 MPa, which represents 72% of the strength obtained with the assemblies joined at 0.8 MPa in Case 8 (highlighting the effect of the joining pressure on the mechanical performance). When PEI was not present on the bottom substrate (Case 1), strength values of 1.7 MPa were attained (7% compared to the baseline in Case 4). However, a significant improvement was observed in the treated specimens (Case 3), attaining an apparent SLS strength of 11.2 MPa (49% of the baseline strength at the low-pressure condition in Case 4). These specimens were processed one day after etching (i.e., the fastest time attainable using the equipment available) and was properly stored in a sealed bag. Finally, at the low-pressure condition, the specimens that had both substrates treated prior to joining (Case 3) attained an apparent strength of 6.4 MPa (only 28% of the baseline strength in Case 4).

In contrast, the configurations that had PEI co-consolidated on both top and bottom substrates and joined at the high-pressure condition (Case 8), attained apparent SLS strength values of 31.6°MPa, consistent with previously reported values for the Thermabond™ process [14-16]. Similar to the low-pressure condition, not having PEI co-consolidated on the bottom substrate (Case 5), resulted in low apparent SLS strength, with values of only 1.5 MPa (5% compared to the baseline in Case 8). The low performance in both Case 1 and 5 highlighted the importance of having PEI on both substrates being joined using the Thermabond™ process, as a significant strength detriment was observed regardless of the pressure. The specimens where the bottom substrate was treated (i.e., abraded and etched) and had the DCM PEI 10 wt% applied subsequently (Case 6), attained an apparent SLS strength of 5.3 MPa (17% of the baseline strength in Case 8). Finally, similar performance of 4.7 MPa was achieved by the specimens that were abraded, etched, and had the

DCM PEI 10 wt% solution applied on both substrates (Case 7), attaining 15% of the baseline strength.

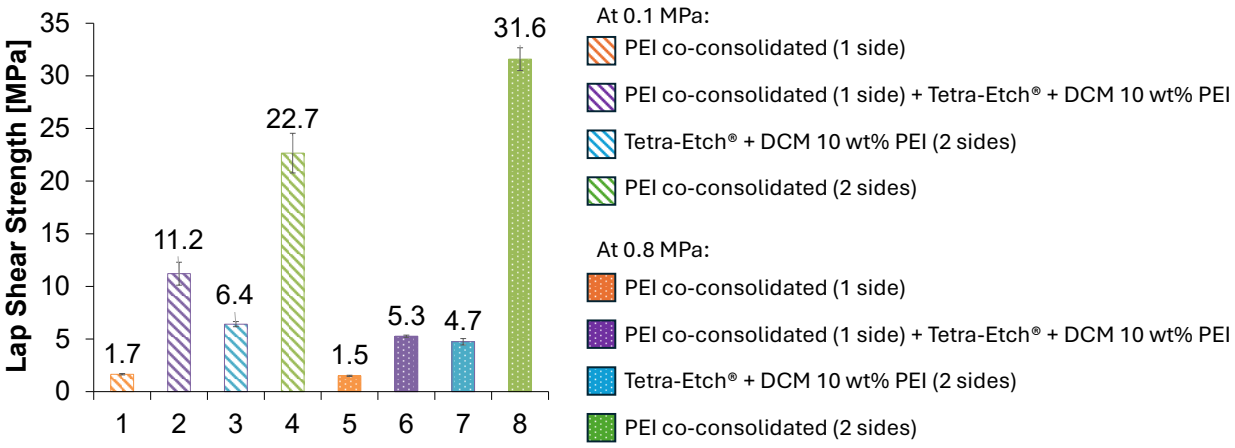

Figure 4: Single lap shear testing results comparing the effect of the etchant. TE indicated specimens etched using Tetra-Etch®; cc indicated specimens that had the layer of PEI co-consolidated on the surface at the processing temperatures of PEEK

4.2 Contact angle measurement results

The contact angle of the untreated CF/PEEK specimen was first measured and compared with specimens that were either abraded (SB), not abraded (XSB) prior to etching, with measurements taken after 0 and 1 hour; and with abraded and etched specimens measured after 0 to 180 hours. These results are summarized in Figure 5.

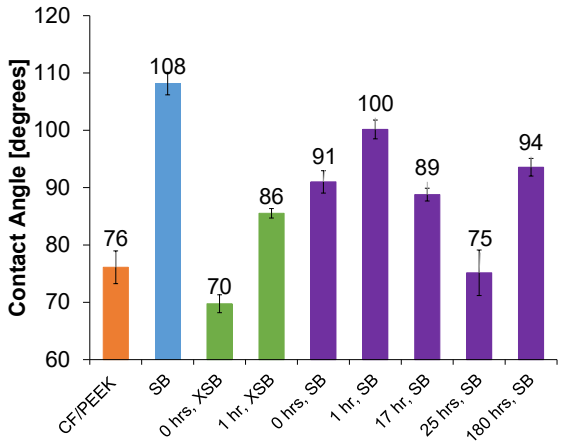

Figure 5: Contact angle measurements on specimens that were abraded and un-etched, not-abraded and etched, and abraded and etched and evaluated after 0-180 hours. These results were compared to baseline (i.e., CF/PEEK)

The surface of the CF/PEEK laminate (untreated specimen and considered as the baseline for these measurements) had a contact angle of 76° which was below the reported angles of 78° to 83° [17, 18]. The specimens that were abraded had a contact angle of 108°, which represented an increase of 32°. This significant increase in the contact angle was likely due to the presence of

grooves and ridges which inhibited the water droplet from spreading out on the surface, thus resulting in a larger contact angle (not desirable to promote adhesion). The specimen labelled 0 hours XSB evaluated the effect of the etchant alone, showing a reduction of the contact angle from 76° down to 70° compared to the untreated specimen. When measured immediately after treatment (i.e., abrasion with SB followed by etching with TE), the contact angle increased from 76° to 91°. Similarly, an increase of the contact angle from 86 to 100° was observed with the specimens that were and were not abraded (identified as SB and XSB, respectively) prior to etching and measured after one hour. These results indicated that eching CF/PEEK reduced the contact angle, while the abrasion step appeared to offset this effect.

To assess the time sensitivity of the etchant, the contact angle was measured as a function of the elapsed time after surface preparation. For the specimens without abrasion (XSB), the contact angle increased from 70° to 86° (+19%) after just one hour. For abraded specimens, the contact angle increased from 91° to 100° (+9%), highlighting a smaller increase. However, for abraded and etched specimens, the contact angle decreased to 89° after 17 hours and to 75° after 25 hours, before increasing again to 94° after 180 hours. These results indicate a time-sensitive etching process and suggest a limited processing window, in agreement with the manufacturer's recommended 24 to 48 hours limit [20].

The delays between the overall surface preparation of the CF/PEEK substrates (i.e., cleaning, abrasion, etching, and the application of the 10 wt% PEI solution in DCM) and the joining operation were analyzed and correlated with the mechanical tests results to evaluate the time sensitivity on the mechanical performance. These results are summarized in Table 3.

Table 3: Results summarizing the time sensitivity of the process

Test case	Pressure [MPa]	Lap shear strength [MPa]	% from baseline strength	Time period between surface treatment and processing
1	0.1	1.7	7	N/A
2		11.2	49	~40
3		6.4	28	~44
4		22.7	100	N/A
5	0.8	1.5	5	N/A
6		5.3	17	~190
7		4.7	15	~111
8		31.6	100	N/A

Specimens processed after approximately 40 hours (Case 2 and 3) attained 28% to 49% of the baseline strength (Case 4). In contrast, the cases processed after longer delays (after 111 hours) attained only 15%, despite of the high joining pressure. These results suggested that the surface treatment was time sensitive and that the effectiveness of the etchant decreases with increasing delays between surface preparation and final joining of the substrates.

5. Conclusion

In conclusion, CF/PEEK laminates specimens were manufactured to evaluate a surface preparation method to introduce PEI to the substrate via a combination of abrasion, etching and applying dissolved PEI in DCM at 10 wt%. The specimens were joined at 0.1 MPa and 0.8 MPa and evaluated in SLS. Baseline specimens, in which PEI was co-consolidated onto both substrates, attained apparent SLS strengths of 22.7 MPa (joined at 0.1 MPa) and 31.6 MPa (joined at 0.8 MPa), where the low-pressure condition represented a reduction of 28% from the high-pressure condition. Contact angle measurements further showed that the surface treatment was time sensitive. For the specimens that were abraded and etched, the contact angle increased from 91° to 100° after 1 hour, then decreased to 89° after 17 hours and to 75° after 25 hours before increasing again at longer times. This trend was consistent with the mechanical results for which the specimens joined approximately 40 hours after surface preparation attained 49% of the baseline strength, whereas those joined after 190 hours reached only 17%, despite being processed at the higher pressure. Overall, abrasion of the surface increased the contact angle, reducing the wettability of PEEK, which is undesirable. Although chemical etching enabled surface activation of CF/PEEK for using the Thermabond™ process for joining, its strong sensitivity to processing time limits its practical applicability for repair, where immediate processing may not be feasible. Alternative surface preparations such as plasma treatment should therefore be investigated.

6. Acknowledgements

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