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# **MECHANICAL EVALUATION OF APC-2 POLY ETHER ETHER KETONE ADHERENDS JOINED USING POLY (ETHER IMIDE) FOR REPAIR**

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## **ABSTRACT**

To fully take advantage of thermoplastic composites (TPCs) on aircraft structures and to be able to certify them, it is key to establish a methodology for repair. One strategy being explored is by joining a repair patch with the Thermabond™ process. This process consists of co-consolidating an amorphous resin layer of poly ether(imide) (PEI) onto a patch made out of carbon fibre (CF) APC-2 poly ether ether ketone (PEEK) at the processing temperatures of PEEK. By using an amorphous polymer layer, it is possible to perform the repair above the glass transition temperature ( $T_g$ ) of PEI but below the melting temperature ( $T_m$ ) of the parent PEEK structure. While it is possible to directly co-consolidate a layer of PEI onto the CF/PEEK patch at the processing temperatures of PEEK, this process may not be feasible for the structure to be repaired as this component cannot be placed directly into an oven or an autoclave. In addition, application of pressure to attain high consolidation and high-quality joints remains a challenge for repair of TPCs. Typically, pressures as high as 0.8 MPa are required when welding TPCs.

The objective of this paper is to evaluate the performance of the Thermabond™ process via single lap shear tests when only one or both of the CF/PEEK adherends has the layer of PEI co-consolidated prior to the joining operation. The joining operation is performed at 300 °C for 30 minutes at two different pressures: at 0.1 MPa to simulate a repair under vacuum bag only (VBO) conditions, and at 0.8 MPa using a press. The mechanical performance and quality of the joint are evaluated with each condition via microscopy. When only the top substrate had the layer of PEI co-consolidated onto it and joined at 0.1 MPa, the apparent lap shear strength was only 7 % from the baseline value. Similar results were obtained for the specimens joined using the press at 0.8 MPa, obtaining an apparent shear strength of only 9 % from the baseline value. The failed specimens that had the layer of PEI co-consolidated onto the substrate on both top and bottom adherends showed consistent cohesive failure, whereas adhesive failure was observed in the cases where PEI was co-consolidated on only the top adherend prior to the joining operation. This work highlights the importance of the co-consolidation step prior to joining to ensure the required performance can be attained when using the Thermabond™ process.

## **1 INTRODUCTION**

Thermoplastic composites (TPCs) are being increasingly used in multiple sectors where advanced processes to manufacture complex parts are required such as in aerospace, automotive and urban air mobility applications, as

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observed in recent trends [1, 2]. To fully take advantage of TPCs on aircraft structures and to be able to certify them, it is key to establish a methodology for repair. When these structures are damaged, they are often removed and replaced. However, the supply of spare parts is often limited to very few suppliers around the world, which can lead to very long lead times and high replacement costs. While there are repair methodologies for thermoset matrix composite materials, the path for TPCs is not yet clear. Viable repair techniques could potentially make the entire product life cycle more sustainable and less expensive as opposed to replacing these TPC parts. Thermoplastic welding which was primarily introduced as a method to join TPC structures, can be adapted for repair as a patch can be joined to the damaged structure to bring it back to its ultimate or limit load. In thermoplastic welding, temperature is increased, reducing the viscosity to allow the movement of the polymeric chains across the welding interface. By applying pressure, close contact between adjacent substrates is created (process also referred to as intimate contact). After intimate contact has been created, healing (i.e., inter-diffusion of the polymer chains) follows, until a full weld is attained [3, 4].

As for every thermoplastic welding process, the three critical parameters are temperature, pressure and time. Smiley et al. [5] generated process maps relating these three key parameters with single lap shear specimens that were joined using the Thermabond™ process. This process consisted of co-consolidating an amorphous resin layer of poly ether(imide) (PEI) onto an adherend made out of carbon fibre (CF) APC-2 (i.e., poly ether ether ketone (PEEK) as the thermoplastic polymer) at the processing temperatures of PEEK. This process allows to then conduct the joining operation above the glass transition temperature ( $T_g$ ) of PEI but below the melting temperature ( $T_m$ ) of PEEK. The adherends for these specimens consisted of 16-ply, [0/45/90/-45]<sub>2s</sub>, CF APC-2 PEEK laminates with a layer of PEI co-consolidated onto the substrate. An overlap of 12.5 mm was selected for the single-lap shear specimens. When conducting the joining operation at a pressure of 0.1 MPa, the highest apparent single lap shear strength with a value of 35 MPa was obtained at a minimum temperature of 300 °C while keeping a dwell of 30 minutes. An increase in strength of 15% was observed when applying a pressure of 0.8 MPa. Pressures higher than 0.8 MPa were shown to be detrimental to the performance of the joint due to excess flow, leading to reductions in the bond-line [6].

Heimerdinger et al. [7] used resistance heating with the Thermabond™ process and conducted a demonstration of repair. An amorphous resin layer of PEI was co-consolidated onto a patch made out of CF APC-2 PEEK in an autoclave with 0.2 MPa of pressure at 385 °C for 30 minutes. The surface of the PEEK parent structure was etched and primed as it was not possible to directly co-consolidate a layer of PEI onto it. The repair patch was then joined to the structure at 150 to 165 °C for 60 minutes (to remove entrapped moisture) and then between 285 and 330 °C for 30 minutes. The outcome of this process was a repaired structure that was later subjected to 115% of the design ultimate load where the repair patch was not affected. While the authors in this work highlighted the steps followed to repair the structure, there were not enough details available in terms of the preparation of the CF/PEEK parent structure and it is not clear that PEI was fully co-consolidated through the adherend with their method. Therefore, the objective of this work is to evaluate the effect of the PEI layer being co-consolidated prior to the process and its relevance to the Thermabond™ process. This objective is achieved by evaluating the mechanical performance in the single lap shear configuration when having a layer of PEI co-consolidated on only one and on both of the adherends at low and high-pressure conditions (i.e., 0.1 MPa and 0.8 MPa, respectively), which are representative of typical repair and welding scenarios.

## 2 METHODOLOGY

To study the importance of co-consolidating the PEI resin film to the PEEK substrate and to show the detriment of not co-consolidating this layer prior to the joining operation, four scenarios were evaluated, as summarized in Table 1. The first and second cases (i.e., the baselines) involved co-consolidating a layer of SABIC PEI ULTEM 1000 on both top and bottom Solvay APC-2 CF/PEEK adherends prior to the joining operation at both low and high-pressure conditions (i.e., 0.1 MPa and 0.8 MPa). The third and fourth cases had a layer of PEI co-consolidated on the top adherend only for both low and high-pressure conditions.

Table 1. Test matrix to evaluate the effect of different pressures and having one or both adherends with co-consolidated (cc) PEI, and to evaluate the effect of surface preparation

Case	Pressure (MPa)	PEI cc on both adherends
1	0.1	✓
2	0.8	✓
3	0.1	
4	0.8	

A schematic of the four cases is shown in Figure 1. Two additional layers of ULTEM 1000 were added between the adherends in every case.

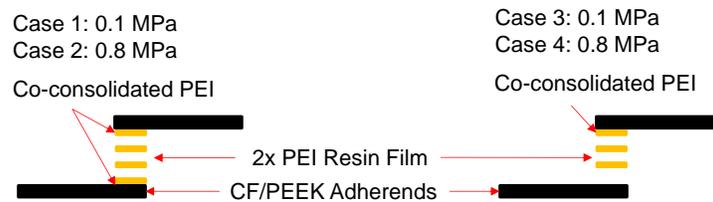


Figure 1: Schematic of configurations evaluated in this study

### 2.1 Composite laminate processing

APC-2 AS4 CF/PEEK 16-ply  $[0/90]_{4s}$  laminates 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 laminates had a strip of PEI (0.127 mm thick) co-consolidated at the centre of the laminate (PEI was only required at the centre 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. 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.

### 2.2 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 single lap shear specimens), as shown in the schematic in Figure 3.

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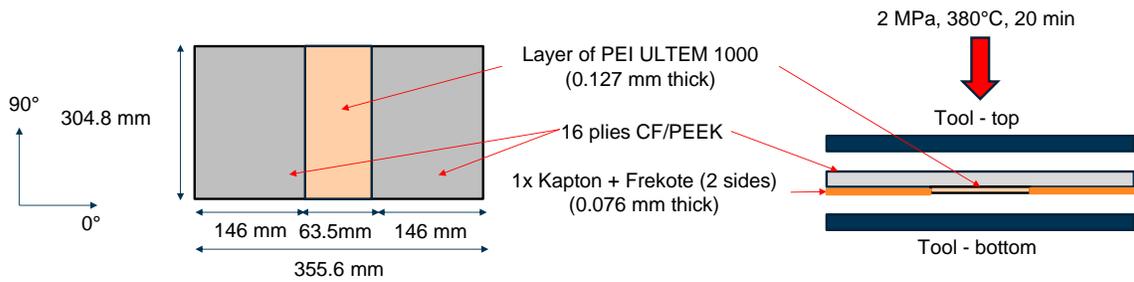


Figure 2: Schematic showing the setup and dimensions used to process the laminates used for this study. The Kapton polyimide film and the PEI resin film were placed below the material stack

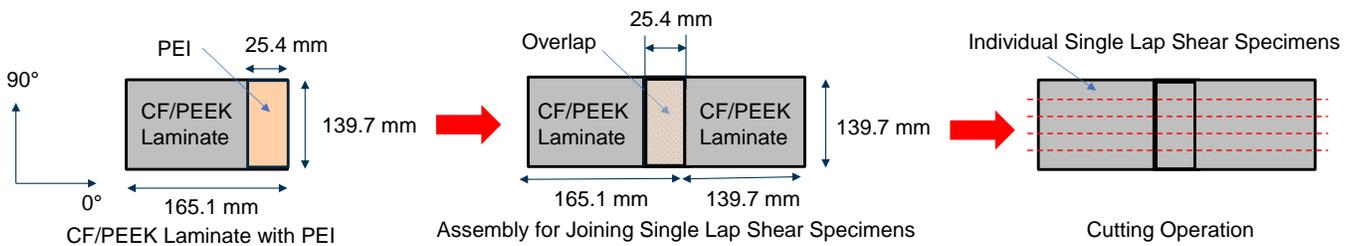


Figure 3: Schematic showing the preparation of the specimens used for the single-lap shear specimens

All laminates were dried at 150 °C for one hour (following similar drying conditions as per Heimerdinger et al. [7]) prior to joining and two additional layers of PEI resin film with a thickness of 0.127 mm were introduced between the adherends 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 60 kW thermoforming 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 the Wabash 150-ton hot-press. Metallic shims wrapped in Kapton polyimide film were used to increase the surface area of the assembly to be joined to attain the desired pressure. For both low and high-pressure cases, temperature was increased to 300 °C (below the  $T_m$  of PEEK of 343 °C) and kept for 30 minutes, which was within the recommended processing window for the Thermabond™ process to achieve healing. The assemblies were cut to obtain five specimens per case using a water-cooled diamond saw with 2 mm thick blade at 4200 RPM machined. 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.

### 3 RESULTS AND DISCUSSION

Figure 4 shows the mechanical results obtained. To highlight the effect of co-consolidating the layer of PEI onto the substrate and to compare the results at the different pressures, the results were normalized with respect to the maximum apparent lap shear strength values (obtained at the tested pressures). At the low pressure of 0.1 MPa, the specimens that had the PEI layer co-consolidated only on the top adherend displayed a significant reduction in strength, attaining only 7 % of the baseline strength (i.e., the strength of the specimens that had PEI co-consolidated on both top and bottom adherends). Similarly, only 9 % of the baseline strength was achieved for the specimens that were joined at the high-pressure of 0.8 MPa. From these results, it was clear that to join the adherends at a temperature below than the melting temperature of PEEK (i.e., at 300 °C which is below  $T_m = 343$  °C), it is critical to have PEI co-consolidated on both adherends. The failure modes of the specimens were observed using a stereo-

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microscope, Carl Zeiss Smartzoom 5 at 1.6X magnification, as shown in Figure 5. Adhesive failure was observed for both low and high-pressure conditions where there was no PEI previously co-consolidated on the surface of the bottom adherend (Figure 5, b and d). On the other hand, there was cohesive failure observed when there was PEI co-consolidated on both the top and bottom adherends (Figure 5, a and c). There were small bubbles observed on the surface of the failed specimens that were joined at the low-pressure of 0.1 MPa (Figure 5, a and b). On the other hand, the specimens that were processed at the high-pressure of 0.8 MPa (Figure 5, c and d) displayed evidence of irregular shape voids. The bubbles may be an indication that there was moisture present in PEI and thus by applying a higher pressure, these air bubbles collapsed, creating these irregular shaped voids. It has been reported that PEI can be susceptible to moisture [9]. The drying cycle used in this work followed the recommendations previously cited by Heimerdinger et al. [7], however these micrographs suggest additional time may be required to ensure the material is fully dried.

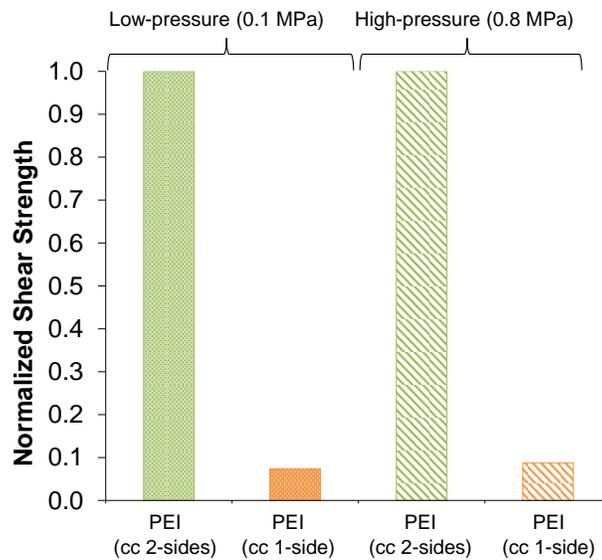


Figure 4: Apparent single lap shear strength of the different configurations at low- and high-pressure conditions

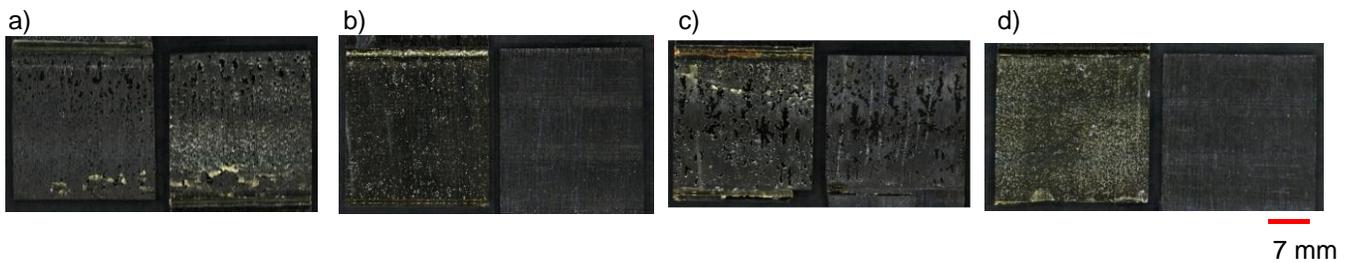


Figure 5: Failure mode of the specimens joined at 0.1 MPa where PEI was co-consolidated on both adherends (a), only on the top adherend (b); and at 0.8 MPa where PEI was co-consolidated on both adherend (c), and only on the top adherend (d)

## 4 CONCLUSION

Composite laminates made out of APC-2 CF/PEEK were prepared to evaluate the effect of having a layer of PEI co-consolidated onto the substrate prior to the joining operation. While it was possible to join the adherends at a temperature of 300 °C which is well below the melting temperatures of PEEK, the performance attained at the two pressures (i.e., 0.1 MPa and 0.8 MPa) was highly dependent on the presence of the PEI layer. Only 7 to 9% of the overall strength was attained when having the layer of PEI co-consolidated only on one of the adherends compared to the baseline (i.e., where PEI was co-consolidated on both adherends). These results highlight the importance to co-consolidate the layer of PEI at the processing temperatures of PEEK prior to the joining operation if the Thermabond™ process is to be used. This work leads the way to investigate ways to introduce the PEI layer onto the parent laminate to ensure a repair with the required mechanical performance can be attained.

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## 6 REFERENCES

1. Gardiner, G. *Thermoplastic composite demonstrators - EU roadmap for future airframes*. Processes 2018; Available from: <https://www.compositesworld.com/articles/thermoplastic-composite-demonstrators-eu-roadmap-for-future-airframes->.
2. Gardiner, G. *Carbon fiber will enable air taxi eVTOLs*. Urban Air Mobility 2019 [cited 2021 21-Sept]; Available from: <https://www.compositesworld.com/articles/carbon-fiber-will-enable-air-taxi-evtols>.
3. Wool, R.P. and K.M. O'Connor, *A theory of crack healing in polymers*. Journal of Applied Physics, 1981. **52**(5953).
4. Gennes, P.G.d., *Reptation of a polymer chain in the presence of fixed obstacles*. The Journal of Chemical Physics, 1971. **55**(572).
5. Smiley, A.J., et al., *Dual polymer bonding of thermoplastic composite structures*. Polymer Engineering & Science, 1991. **31**(7): p. 526-532.
6. Ageorges, C., L. Ye, and M. Hou, *Experimental Investigation of the Resistance Welding of Thermoplastic-Matrix Composites. Part II: optimum Processing Window and Mechanical Performance*. Composites Science and Technology, 2000. **60**: p. 1191-1202.
7. Heimerdinger, M.W., *Repair technology for thermoplastic aircraft structures*, in *79th Meeting of the AGARD Structures and Materials Panel on "Composite Repair of Military Aircraft Structures"*. 1994, Northrop Grumman Corporation.
8. International, A., *ASTM D1002-10: Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)*. 2019, ASTM International.
9. Botelho, E.C., et al., *Environmental effects on thermal properties of PEI/Glass fiber composite materials*. Journal of Aerospace Technology and Management, 2013. **5**(2): p. 241-254.