Out-of-autoclave scarf repair of interlayer toughened carbon fibre composites using double vacuum debulking of patch

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Abstract

Interlayer particle toughened carbon fibre composites play an integral role in the lightweight design of primary aerospace structures. We investigate an out-of-autoclave method using double vacuum debulking (DVD) to perform in-situ soft patch repairs. Utilizing the DVD process decreases the porosity of the co-cured film adhesive and patch from 4.7% to 0.4%, thereby increasing the flexural and interlaminar shear strength of 1D repair laminates by 30% to levels equal to autoclave cured laminates. In contrast, the higher void content did not significantly affect straight (2D) and round (3D) scarf repair strengths. 3D repairs showed significantly improved strength recovery compared to 2D repairs due to the stress shedding in the hoop direction. Finally, DVD process parameters may be optimized to reduce repair time by increasing the temperature and ramp rates while reducing the soak times, with no detrimental effects on porosity or strength observed.

Keywords: A: Carbon fibres; B: porosity; E: Joints/Joining; E: Out of autoclave processing

1 Introduction

Composite materials, and more specifically carbon fibre-reinforced polymer (CFRP) composites, are increasingly being used in a wide range of industry sectors. Of particular interest to the engineering community is their use in large, load-bearing structures such as aircraft fuselage, wings [1], automobile [2] and storage tanks [3] to improve fuel efficiency via lightweight solutions. While CFRPs have high specific strength, stiffness and superior creep performance, the maintenance and repair of these structures is relatively complex and labor-intensive due to the unfamiliarity and

inability to adapt simple metallic repair technologies to these new materials [4]. Large composite structures, such as the Boeing 787 fuselage assembly of barrel sections, cannot be broken down to smaller parts for repair in a conventionally sized autoclave [5]. In addition, many industry sectors that operate in the maintenance, repair and overhaul (MRO) sector do not have access to on-site autoclaves.

The requirements of the MRO sector necessitate the development of reliable out-of-autoclave processes capable of achieving high quality, low porosity repairs directly on the composite structure. The carbon fibre prepreg materials used on the Boeing 787 [6, 7] and Airbus A350XWB [8, 9, 10] are toughened by interlayer particles to provide improved compression after impact resistance. Furthermore, these prepregs are manufactured by hot melt impregnation method [11, 12] with little or no residual solvents in the final product. Despite the low volatile content, these materials have unacceptable porosity levels when cured using conventional vacuum bagging lay-ups [13]. The problem is further compounded by the need to use the same material for the patch and the parent, which means that partially-impregnated prepreg systems specifically designed for out-of-autoclave (OOA) curing or repair processes cannot be used.

Porosity can have highly detrimental effects on the mechanical properties of composite materials. For example, a 1% increase in void content can cause a reduction of 7% in intralaminar shear strength due to promoting crack initiation and/or propagation [14]. Tang et al. [15] tested composites with a range of porosities and found that above a void content of 4%, the interlaminar shear strength decreases at an increasing rate. For scarf joints, Préau and Hubert found that every 1% of bondline void content in a 2D composite scarf repair contributes to a decrease of 4.5% in tensile strength recovery [13].

The double vacuum debulking (DVD) process was developed in the 80s and was recently shown to be a promising approach for OOA curing by several authors as the composite porosity is reduced significantly [16, 17, 18, 19]. The DVD process has shown good performance for several different types of resins, each with differing characteristics [20, 21]. The main mechanism of the DVD process is the debulking process at vacuum conditions without compaction, which increases the transverse volatile flow. This is achieved by applying a second vacuum via a rigid chamber on top of the lower bagging lay-up. The process was adopted by Boeing as the required soft patch curing method for primary composite structures on 787 aircraft for repair and structural modifications [22, 23].

Less information has been published about the influence of the DVD process on the integrity of the repair and its bondline, requiring additional considerations such as size effects, joint geometry and co-curing with the adhesive film layer. Void formation during vacuum bag curing of the film adhesives can also lead to premature bond failure [24]. Pearce et al. found that volatiles of organic solvents such as methyl ethyl ketone (MEK) and water absorbed by the adhesive or from manufacturing are the most prominent cause in epoxy film adhesives [25]. Chester and Roberts noted that the type of adherend can also have a significant effect on the void content in the adhesive, as aluminium surfaces can readily adsorb moisture [26]. Several authors also formulated strategies to reduce bondline porosity through either partial curing (B-staging), perforation [13] or the addition of air evacuation layers [24].

This paper evaluates the repair process of a high performance interlayer toughened CFRP composite using the DVD process. This CFRP is qualified for primary composite structures on Boeing aircraft due to its high compression after impact resistance [6, 7], but limited research has been published about its performance during out-of-autoclave

processing and repair [27]. Here, the influence of residual volatiles, repair geometry and adhesive co-curing on the porosity location and content are investigated by microscopy and thermal analysis. The effect of porosity on the laminate's mechanical properties and repair strength was then examined in scarf lap joints, 1D, 2D, and 3D repair configurations by experimental testing. Repair strength at the coupon level and full scale round scarf repairs were compared to the parent and open-hole tension strengths as benchmarks, highlighting the high strength retention of void-free repairs. Additionally, we outline strategies to improve the efficiency of the currently time-consuming DVD process in this work. These findings contribute to the increasing requirement for manufacturing high quality lightweight primary composite structures at low cost and performing in-service repairs and potential aircraft structure modifications.

2 Experimental Methodology

2.1 Material and manufacturing details

The carbon fibre laminates were manufactured from T830H/3900-2D carbon/epoxy plain weave 6K woven prepreg plies (FM6673G, Torayca) with an 8 ply quasi-isotropic lay-up $[(-45/+45)/(90/0)/(+45/-45)/(0/90)]_s$, resulting in a laminate thickness of 1.8 mm. The parent material was cured in an autoclave in accordance with the manufacturer's recommendations (177°C, 580 kPa, 150 min), resulting in a stiffness of 44.5 ± 0.8 GPa and a material strength of 657.8 ± 19.1 MPa. The repair patch and repair specimens were OOA cured using the same cure cycle and standard vacuum bagging lay-up. The adhesive used was a Henkel PL7000 film adhesive with a nominal thickness of 200 µm and a recommended curing temperature of 177°C. Its modulus was measured as

2.5 ± 0.1 GPa and its shear strength is specified as 28 MPa by the manufacturer based on shear tests with composite substrates [28].

For OOA curing, the term “hotbonding cured” refers to laminates and repairs that were cured without any DVD patch preparation, with the same thermal cure cycle as the autoclave. For “DVD cured” laminates, the patch was pre-cured under elevated temperature and double vacuum as shown in Figure 1. The DVD box forms a vacuum half-chamber when seated on top of the lower bag lay-up. Both the DVD box and 1 inch thick base are constructed of aluminium alloy 7075-T6. After the DVD box was sealed, a vacuum in the DVD box was applied to 1 inHg below that of the lower vacuum bag. The temperature was then increased at a rate of 1°C/min using a heat blanket. As per repair specifications for the given CFRP material, the DVD box was vented after dwelling at the DVD process temperature of 104°C for 30 min and held for a further 30 min at temperature to allow for full consolidation of the prepreg plies. Following this pre-cure step to remove prepreg volatiles, the patch was transferred to the panel, placed on top of the film adhesive and cured with the same thermal cure cycle and standard vacuum bagging lay-up as used for the hotbonding processes. It should be noted that the patch remained under controlled atmospheric conditions at all times prior and during to transfer to the repair panel as stipulated in the repair specifications to avoid re-absorption of moisture.

2.2 Curing characteristics of prepreg material and film adhesive

Curing studies were undertaken for the prepreg and film adhesive materials to optimize the DVD process temperature. Differential scanning calorimetry (DSC) was used to investigate the glass transition temperature and curing behaviour. A TA Q100 DSC was used with a temperature range from -80°C to 300°C and operated under N_2

atmosphere. A heating rate of 2°C/min was used to remain consistent with the actual cure cycle.

Thermal Gravimetric Analysis–Mass Spectrometry (TG-MS, Netzsch TG 209 F1 coupled with QMS 403 C Aëolos) was used to study the volatiles emitted from the prepreg and film adhesive materials in-situ during the pre-cure and final cure stage at a temperature range of 23°C to 200°C with a ramping rate of 10°C/min under nitrogen purge of 20 mL/min. This method allows for a highly sensitive determination of volatile compositions up to 1 atomic unit resolution at varied temperatures.

A rotational rheometer (Anton-Paar MCR301) was used to study the cure progression of the film adhesive and prepreg by observing the change in viscosity as temperature was increased. An oscillation mode was used with 25 mm diameter aluminium parallel plates at a frequency of 1 Hz and amplitude of 1% strain. The gap was determined based on the height of the samples by keeping the normal force below 10 N.

2.3 Composite repair geometries

Composite repair specimens of various configurations were examined to elucidate the porosity contribution factors. The “patch only” specimens were carbon fibre laminates of 240 mm x 240 mm in size. Two types of patch specimens were produced: (1) 8 ply quasi-isotropic lay-up and (2) the “1D repair” specimen with an additional single layer of co-cured adhesive at the bottom of the prepreg patch, see Figure 2(a). The latter design allowed for separate study of adhesive layer effects on repair quality for OOA curing. To evaluate the composite properties as a function of the curing process, tensile, flexural and interlaminar shear strength tests were conducted on the hotbonded and DVD cured laminates in accordance with ASTM D3039 [29], D790 [30] and D6272 [31], respectively. Six samples were tested for each test series. The results

were benchmarked against the open-hole tensile strength of the autoclave cured prepreg system, in accordance with ASTM D5766 [32].

Scarf lap joints were used to evaluate the strength of a repair without the effect of a poor quality patch, effectively representing a hard patch repair (Figure 2(b)). The “2D repair” specimens are simplified test coupons for a soft patch composite repair, as shown in Figure 2(c, d). A scarf angle of 2˚ was machined by milling for the scarf lap joint and 2D repair. The 2D repair specimens were manufactured with a 25 mm gap between the parent substrates, representing an initial damage size of 25 mm, see Figure 2(d). Glass fibre end tabs of 3 mm thickness were bonded to the grip areas for the scarf lap joints and 2D repairs to reduce failure at the grips. Six samples were tested for each repair scenario.

The “3D repair” differs from the “2D repair” as patches are scarfed in a round geometry, as shown in Figure 2(e, f). The 3D repair tensile test specimens were 650 mm x 270 mm in size, where the width tapers down to 250 mm at the repair area in the centre. The tapering parameters were optimized via linear finite element analysis (Abaqus 6.14) to minimize edge stress concentrations by increasing the radius and length of the thinner section. A central hole of 25 mm diameter was cut to represent the initial damage. A computer numerical control (CNC) milling process was used to scarf the circular 3D repair patch area at 2˚ to result in minimal fibre breakage around the inner hole. The average roughness of the scarf machined surface was 2 µm, which is approximately equivalent to that achievable using a standard hand tool used in industry. Carbon fibre end tabs were used for the 3D repair tensile test specimens to allow for even stress transfer between the drilled bolt holes. Only two test samples were tested per configuration to reduce test cost.

3 Results and Discussion

3.1 Optimum DVD process conditions

Composite DVD repair includes two separate processes: 1) repair patch preparation, and 2) on-site patching and full curing. In the repair patch preparation process, the prepreg plies will experience a pre-cure process. The pre-cure temperature and exposure time should be sufficient to decrease viscosity such that volatiles can be easily removed under vacuum conditions. Additionally, it is desirable that the pre-cured repair patch maintains a glass transition temperature lower than room temperature to maintain its ability to conform to the potentially curved contour of the damaged composite surfaces.

The influence of pre-cure temperature during the repair patch preparation process on cure content and glass transition temperature was evaluated systematically as shown in Figure 3. The pre-cure time was fixed at 60 min, and the temperature was varied from 104°C to 134°C. The glass transition temperature and cure content shows an exponential increase with pre-cure temperature.

Loosely-bound volatiles (water, gases) are known to cause voids to form during the curing process [33, 25]. The weight loss of the prepreg and film adhesive during heating from room temperature to 200°C is shown in Figure 4(a). Both prepreg and film adhesive show a fast weight loss of approximately 0.175% from room temperature to 90°C. Above 90°C, the prepreg mass remains stable, whereas the film adhesive exhibits a different weight loss profile. Its mass continues to decrease, followed by an accelerated weight loss at 140°C.

Further details about this weight loss profile can be deduced from the TG-MS investigations. Analyses of volatiles emitted from the prepreg and film adhesive

indicate that the major component in the volatiles released is water (H_2O) at temperatures below 100°C. No residual traces of solvents such as acetone could be detected in either the prepreg or film adhesive, as shown in Figure 4(b) and (c). Maximum water release is found at 75°C and 68°C for the prepreg material and film adhesive, respectively, as shown in Figure 4(c). In order to maximize volatile loss while maintaining minimum cure, pre-cure temperatures of 104°C and 114°C are hence confirmed as lower and upper bound temperatures suitable for the DVD process for the prepreg material.

A further peak was observed at 140°C in the TG-MS experiments for the film adhesive as part of the on-going curing reaction within the material. TG-MS scans further confirm the major volatile components are H_2O and CO_2 at this temperature range, both of which might have been chemically absorbed from the atmosphere previously by the amine groups of the hardeners [34]. Desorption will take place at elevated temperatures, depending on the chemical bonding strength with the amine groups. It is of concern that this released H_2O and CO_2 may be trapped in both adhesive film and repair patch, thus forming additional porosities.

The viscosity measurements in Figure 5 show that it is not possible to pre-treat the film adhesive at temperatures around 140°C to remove these volatiles prior to repair as the material begins to gel and harden at approximately 100˚C, whereas the patch starts at a significantly higher temperature of ~150˚C. Potential volatile removal for the adhesive film is therefore limited to temperatures below 90°C, which is insufficient and therefore not further considered in this work.

3.2 Influence of curing process and geometry on porosity content

Polished cross-sections of the patch only laminates cured by autoclave, hotbonding and DVD processes are shown in Figure 6. As expected, the autoclave cured laminates show no sign of porosity as seen in Figure 6(a), hence demonstrating good resin retention with minimal surface porosity and full volatile depletion. The interlayer toughening particles are clearly visible and exhibit good adhesion to the matrix and sufficient resin penetration between the particles. The hotbonding cured laminates, as shown in Figure 6(b), were measured to have a void content of 0.5 ± 0.1%. Voids were observed to be located in both the resin-rich zones and inter-fibre regions. Size and shape of porosities are significantly different depending on location. The voids that are located in the resin-rich zones are larger and mostly spherical and reduce the interlayer particle/matrix adhesion. The voids within the inter-fibre regions are mostly smaller and elongated along the fibres. The DVD cured laminates were shown to be able to achieve autoclave quality in terms of porosity content as shown in Figure 6(c). A void content of 0.2 ± 0.1% was measured from the optical micrographs. Furthermore, Figure 6(c) indicates that the DVD process has no negative effects on the interlayer particles, as there was no evidence of debonding or voids in this region.

The effect of the film adhesive on the porosity of the patch was examined using the 1D repair specimens. Typical micrographs are shown in Figure 7. We note that the hotbonding cured 1D repair laminate as shown in Figure 7(a) exhibits much higher porosity compared to the patch only laminate (4.7% versus 0.5%). On the other hand, the DVD cured 1D repair laminate as shown in Figure 7(b) does not show any significant increases in void content in comparison (0.4% versus 0.2%). This indicates that the additional porosity content is not caused by volatiles released from within the adhesive film at higher temperatures, but through the effect of limiting volatile escape from the prepreg patch. We recall that the onset of solidification happens much earlier

for the film adhesive, hence effectively creating an impenetrable barrier layer beneath the patch for the temperature region of lowest viscosity for the prepreg material, hence leading to premature void entrapment. In a similar context Préau and Hubert showed that utilizing a perforated film adhesive can reduce repair porosity [13].

We also confirmed the detrimental role of the film adhesive with regard to porosity for the 3D repair specimens. An overview of the representative porosity results for both patch and adhesive as a function of repair location can be found in Figure 8. As this specimen has an angled patch geometry, the porosity was analysed in sections as indicated in the figure, with at least three randomly selected locations evaluated for each data point. Firstly, it can be seen that the porosity content is again consistently high for hotbonding cure within the repair patch and mostly independent of section location. The average porosity content in the 3D repair agrees well with those measured from the 1D repair specimens with an average of 3.9 ± 1.2% in the patch and 1.6 ± 1.5% in the adhesive for the hotbonding cured 3D repairs. In comparison, the DVD cured 3D repairs are almost void-free, with an average porosity of 0.1 ± 0.1% in both the patch and adhesive. The decrease in porosity in the adhesive by applying the DVD process on the patch again gives evidence to the argument that prepreg volatiles become trapped in the adhesive film layer. It is also noted from the 1D versus 3D repair comparison that the fully cured composite structure underneath the repair patch does not contribute to further porosity increase.

Additionally, it can be noted from the through-thickness optical micrographs that the porosity distribution across the thickness of the patch is not necessarily uniform. The analysis of the hotbonding cured 3D repair across the entire thickness at locations 3 (near the tip of the scarf) and 4 (centre hole) is shown in Figure 9(a). At location 3, it was found that the region closest to the adhesive exhibits the highest porosity, which

gradually decreases towards the upper surface of the patch (shortest path to the vacuum source). Meanwhile, the centre of the repair (location 4) contains fewer voids on average, as shown in Figure 8, and the porosity distribution is fairly uniform across the thickness. The 1D repair laminate with the film adhesive at the lower surface has a different porosity distribution, where the highest porosity was found in the lower and centre part as shown in Figure 9(b). The difference in porosity distribution is the result of the restricted volatile escape path due to the presence of cured CFRP and adhesive film. In contrast, standard hotbonding curing for laminates without adhesive presence yields a fairly uniform void distribution of only 0.5%. 1D repair laminates are therefore identified as more representative designs to mimic repair porosity content as the maximum porosities in the 1D and 3D repairs of 8% are of similar order of magnitude. As some of the mechanical laminate properties are highly sensitive to porosity, it is important to achieve a similar void content to ensure that the property degradation results are representative.

3.3 Influence of porosity on laminate and repair properties

The influence of the porosity on the laminate mechanical properties is summarized in Table 1. Literature generally reports that stiffness values are less affected by porosity content than strength values [35, 36], which is also the case for the presented results here. Additionally, matrix dominated strength values are expected to reduce more severely than fibre-dominated strength for the obvious reason of void accumulation in the resin-rich zones as described earlier.

For tensile stiffness, all measurement outcomes are similar, with the 1D repair specimens showing the largest reduction in stiffness with around 5%. For tensile

strength, it can be seen that the tensile strength is reduced by 12% for both the hotbonding cured patch and 1D repair laminates with high porosity contents. While the strength loss is similar despite the different void content, the standard variation is much lower for the 1D repair laminates with higher void content, leading to consistently lower results. Strength reduction occurs as large voids create stress concentrations and can initiate and propagate cracks. Zhu et al. showed cracks originating from voids greater than 400 µm in size for off-axis plies, hence reducing the tensile strength for woven CFRP laminates [35].

The four point bend test introduces a uniform bending moment under the upper rollers and produces the highest tensile stress on the lower surface of the specimen. During testing, more than one area of failure is often observed on this lower surface, highlighting the uniform stress distribution over a longer distance with this test set-up compared to the sensitivity of the tensile test to stress concentrations. Firstly, it can be seen that the flexural modulus is not significantly affected by the porosity levels, which agrees well with the findings for the tensile modulus and literature [35]. For hotbonding cured laminates, a strength reduction of 9% is observed. The higher porosity of the hotbonding cured 1D repair laminates reduces the flexural strength by 18%, which is higher than the tensile strength reduction. It is noted that visible failure occurs on the tensile side for all specimens, and that the void distribution is non-uniform and highest in the lower part and centre for the 1D repair specimens. All specimens were tested with the film adhesive on the bottom surface, hence placing the zone of highest porosity closer to the tensile failure initiation zone. By applying the DVD pre-cure, the flexural strength is recovered and even exceeding the benchmark autoclave property in the case of the 1D repair. This result might be caused by the additional adhesive layer at the lower surface, which was not removed prior to testing.

The interlaminar shear strength is the most sensitive test with regard to porosity content and location. The patch only laminates show little decrease in interlaminar shear strength, whereas the hotbonding cured 1D repair laminates show the lowest strength (-24%) as their porosity is highest, especially towards the centre of the laminate thickness. The levels of interlaminar shear strength loss reported here are in good agreement with previously reported literature values due to increased void content and are generally attributed to the decrease in the cross-sectional load-bearing area due to distributed voids and initiation of failure from individual voids if their size is sufficiently large [37] [38]. The strength decrease was fully recovered by applying the DVD patch preparation step as evident by the results for the DVD cured 1D repair laminates.

3.4 Influence of porosity on 2D and 3D repair strength

The repair strength results are summarized in Figure 10, together with a comparison with the open-hole, scarf lap joint and the parent material strengths. Sudden catastrophic failure was observed for all tests, and localized strain gauge readings and global digital image correlation (DIC) measurements were not able to indicate any locations of non-uniform strain increase prior to failure.

The open-hole tensile strength results show that the quasi-isotropic CFRP laminates are moderately notch-sensitive and fall within the expected normalized strength band of 32% (notch-sensitive) and 83% (notch-insensitive) [39]. The tested normalized strength of 50% agrees with the observed catastrophic failure development perpendicular to the loading direction. Extensive ductile failure development is not encountered, and the stress-strain curves remain linear up to failure. The normalized

strength is in good agreement with quasi-isotropic results for non-woven CFRP composites as reported by O'Higgins et al [39].

In all 2D cases, failure originated from within the overlap region based on the fracture patterns shown in Figure 11. The scarf lap joint failure originates at the scarf tip, then propagates primarily through the parent material as shown in Figure 11(a) with little evidence of failure along the adhesive bond line. It is known that 2D scarf joints with through-thickness fibre lay-up angle variations show a tensile strength significantly below the parent material even for very small scarf angles [40] [41] [42] [43]. This is attributed to stress concentrations in the adhesive and around the tips of the 0° ply laminates terminating at the bond line. It should be noted that a maximum scarf lap strength of 50-70% compared to parent strength has been reported in these earlier works which is in good agreement with our results; however, exact strength retention values depend on fibre lay-up angle, stacking sequence and stiffness properties.

For the 2D scarf repair specimens, a further 5% strength reduction compared to the 2D scarf lap joint is determined. In this case the failure was found to be a mix of cohesive failure along the film adhesive and failure in the composite patch and parent as shown in Figure 11(b) and (c). No significant differences were observed between the hotbonding cured and DVD cured 2D repairs, as expected given the similarity in the strength results. This indicates that the additional strength reduction effect is mostly due to resin-rich pockets and potential residual stress development in the composite repair patch.

The 3D repair tensile tests confirm similar trends and failure modes for the hotbonding and DVD cured repairs. Brittle fracture occurred at the maximum load, which resulted in catastrophic failure with numerous amounts of crack branching for both the patch

and parent laminate as shown in Figure 12. Both 2D and 3D repairs exceed the open-hole tensile strength benchmark as required.

The failure stress for the 3D repairs in comparison to the 2D repairs is significantly higher and in fact only 10% below the parent material strength. It was previously reported that the 2D scarf repair may underestimate the load-carrying ability of 3D scarf repairs under tension [43]. The 2D stress distribution assumes that the bondline will yield uniformly across the joint width, which is not the case under uniaxial loading for 3D repairs. Based on 3D FE modelling, the regions of highest adhesive and laminate tip stresses are located along the loading direction [44], and these stresses are very similar to the stress distribution in the 2D scarf joint. Following first yield, the load can therefore be shed and redistributed in the hoop direction (perpendicular to the idealized joint). Similarly, stress concentrations around the laminate ply tips are localized rather than extending across the whole joint width. It is postulated that the high toughness interlayer of the investigated prepreg material [6, 7] may also be beneficial for the stress redistribution process.

Lastly, while failure mode and relative strength measurements revealed that the average patch porosity of up to 4.5% is not a major factor affecting static tensile 3D repair strength, this does not indicate that these porosity levels are acceptable for composite repair processes. In fact, the hotbonding cured joints fail OEM guide lines for acceptable repair. A high porosity content also makes structures more susceptible to environmental degradation and reduces fatigue life [45] [46] [47]. These effects were not investigated in the current work.

3.5 Optimized DVD process

Repair processes need to be conducted quickly to minimize aircraft downtime. As such, OEM suggested DVD processing parameters were investigated in order to reduce the cycle time. Here, a systematic study was conducted using three alternative DVD cure cycles with improved productivity as shown in Table 2. The DVD process can be analysed as a four step procedure: (a) heating up, (b) holding with reduced compaction, (c) holding with compaction, (d) cool down. The heating rate is typically very low to prevent temperature overshoot during final cure. However, here it was found that the ramp rate can be increased with minimal impact due to the relatively low process temperatures of the DVD process and the controlled laboratory environment enabling uniform heating. The second step is the main volatile depletion stage and this can be shortened by increasing the temperature. The third step consolidates the patch with applied atmospheric pressure to remove entrapped air but was found to provide minimal impact and the time period can again be reduced significantly. Finally, the cool down step can be shortened by improving heat transfer out of the patch, which also assists to reduce the cure content.

All improved DVD processes led to significantly reduced process times and resulted in essentially void-free laminates as summarised in Table 3. Four point bend, short beam shear tests and 3D repair tests confirmed the consistently high quality of the patch and repair.

4 Conclusions

The high quality repair of an interlayer toughened carbon fibre laminate in an out-of-autoclave scenario was demonstrated using the double vacuum debulking (DVD) process. The porosity contributing factors were identified and correlated against

mechanical properties. A more time-efficient DVD cure cycle is suggested based on this study.

Carbon fibre panels manufactured by a standard vacuum bagging lay-up produced fairly low porosity laminates with a void content of 0.5%. However, the addition of a single layer of film adhesive to the bottom of the stack caused significant porosity to build up, where a maximum of 4.7% was measured. This increase in porosity contributed to a reduction in patch properties, where the flexural strength and interlaminar shear strength decreased by 18% and 24%, respectively. Thermogravimetric-mass spectroscopy (TG-MS) studies on the prepreg and adhesive showed that there was a significant quantity of water released during the cure cycle which contributes to this porosity. This was further exacerbated by the relatively higher cure rate of the film adhesive, restricting the volatile flow paths at an early stage of the cure and causing entrapment of volatiles.

The parameters for DVD processing were obtained by systematic differential scanning calorimetry (DSC) studies. By pre-curing the prepreg material prior to final cure using the DVD method, the void content reduced and the mechanical properties of the patch material were sufficiently recovered. The porosity did not have a negative effect on the repair strength in 2D or 3D configurations, where stress concentrations in the adhesive and around laminate tips are shown to be the most dominant factor. In addition, the process efficiency can be further improved by increasing the ramp rates and reducing the hold times of each DVD step, without detrimental effects on the porosity or mechanical properties.

The experimental results shown in this study provide strong evidence for the employment of an out-of-autoclave methodology for repairing composite primary

structures. The work shows that next generation carbon fibre materials in critical aerospace structures can be maintained and repaired to a high quality using conventionally available tools.

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References

[1] K. Lu, “The future of metals,” *Science,* vol. 328, no. 5976, pp. 319-320, 2010.

[2] H. Adam, “Carbon fibre in automotive applications,” *Materials & Design,* vol. 18, no. 4, pp. 349-355, 1997.

[3] L. Schlapbach and A. Züttel, “Hydrogen-storage materials for mobile applications,” *Nature,* vol. 414, no. 6861, pp. 353-358, 2001.

[4] G. Gardiner, “Primary structure repair: the quest of quality,” *High-Performance Composites,* vol. 19, no. 6, pp. 48-54, 2011.

[5] G. Marsh, “Airbus takes on Boeing with reinforced plastic A350 XWB,” *Reinforced Plastics,* vol. 51, no. 11, pp. 26-27, 2007.

[6] N. Odagiri, H. Kishi and M. Yamashita, “Development of TORAYCA prepreg P2302 carbon fiber reinforced plastic for aircraft primary structural materials,” *Advanced Composite Materials,* vol. 5, no. 3, pp. 249-254, 1996.

[7] N. Odagiri, S. Suzue, H. Kishi, T. Nakae and A. Matsusaki, “Fiber reinforced composite materials having resin particle inter-layer zones”. United States of America Patent 5,028,478, 1991.

[8] S. C. Garcea, M. N. Mavrogordato, A. E. Scott, I. Sinclair and S. M. Spearing, “Fatigue micromechanism characterisation in carbon fibre reinforced polymers using synchrotron radiation computed tomography,” *Composites Science and Technology,* vol. 99, pp. 23-30, 2014.

[9] S. C. Garcea, I. Sinclair and S. M. Spearing, “In situ synchrotron tomographic evaluation of the effect of toughening strategies on fatigue micromechanisms in carbon fibre reinforced polymers,” *Composites Science and Technology,* vol. 109, pp. 32-39, 2015.

[10] C. Hunt, J. Kratz and I. K. Partridge, “Cure path dependency of mode I fracture toughness in thermoplastic particle interleaf toughened prepreg laminates,” *Composites Part A: Applied Science and Manufacturing,* vol. 87, pp. 109-114, 2016.

[11] F. C. Campbell, Manufacturing processes for advanced composites, Oxford: Elsevier, 2003.

[12] B. C. Kim, P. M. Weaver and K. Potter, “Manufacturing characteristics of the continuous tow shearing method for manufacturing of variable angle tow composites,” *Composites Part A: Applied Science and Manufacturing,* vol. 61, pp. 141-151, 2014.

[13] M. Préau and P. Hubert, “Processing of co-bonded scarf repairs: Void reduction strategies and influence on strength recovery,” *Composites Part A: Applied Science and Manufacturing,* vol. 84, pp. 236-245, 2016.

[14] E. J. Barbero, Introduction to composite materials design, Boca Raton: CRC Press, 2010.

[15] J. M. Tang, W. I. Lee and G. S. Springer, “Effects of cure pressure on resin flow, voids, and mechanical properties,” *Journal of Composite Materials,* vol. 21, no. 5, pp. 421-440, 1987.

[16] T. H. Hou, J. M. Bai and J. M. Baughman, “Processing and properties of a phenolic composite system,” *Journal of Reinforced Plastics and Composites,* vol. 25, no. 5, pp. 495-502, 2006.

[17] J. C. Dominguez and B. Madsen, “Development of new biomass-based furan/glass composites manufactured by the double-vacuum-bag technique,” *Journal of Composite Materials,* vol. 49, no. 24, pp. 2993-3003, 2015.

[18] L. Alam Khan, A. H. Mahmood, S. Ahmed and R. J. Day, “Effect of double vacuum bagging (DVB) in quickstep processing on the properties of 977-2A carbon/epoxy composites,” *Polymer Composites,* vol. 34, no. 6, pp. 942-952, 2013.

[19] G. R. Sherwin, “Non-autoclave processing of advanced composite repairs,” *International Journal of Adhesion and Adhesives,* vol. 19, no. 2-3, pp. 155-159, 1999.

[20] L. B. Buckley, R. E. Trabocco and E. L. Rosenzweig, “Report No. NADC-83084-60,” Naval Air Warfare Center, Warminster, PA, 1983.

[21] A. N. Rider, A. A. Baker, C. H. Wang and G. Smith, “ An enhanced vacuum cure technique for on-aircraft repair of carbon-bismaleimide composites,” *Applied Composite Materials,* vol. 18, no. 3, pp. 231-251, 2011.

[22] R. L. Keller and J. F. Spalding, “Process development protocol and vacuum bag process for carbon-epoxy prepreg production”. United States of America Patent 7,935,200, 2011.

[23] M. W. Evens, K. E. Nelson, J. F. Spalding Jr, J. D. Chanes, J. P. Baldwin and P. S. Rutherford, “Systems and methods for on-aircraft composite repair using double vacuum debulking”. United States of America Patent 8,986,479, 2015.

[24] A. N. Rider, C. H. Wang and P. Chang, “Bonded repairs for carbon/BMI composite at high operating temperatures,” *Composites Part A: Applied Science and Manufacturing,* vol. 41, no. 7, pp. 902-912, 2010.

[25] P. J. Pearce, D. R. Arnott, A. Camilleri, M. R. Kindermann, G. I. Mathys and A. R. Wilson, “Cause and effect of void formation during vacuum bag curing of epoxy film adhesives,” *Journal of Adhesion Science and Technology,* vol. 12, no. 6, pp. 567-584, 1998.

[26] R. J. Chester and J. D. Roberts, “Void minimization in adhesive joints,” *International Journal of Adhesion and Adhesives,* vol. 9, no. 3, pp. 129-138, 1989.

[27] E. Kutscha, P. Vahey, M. Belcher, P. VanVoast, W. Grace, K. Blohowiak, F. Palmieri and J. Connell, “Contamination and surface preparation effects on composite bonding,” in *SAMPE 2017*, Seattle, WA, United States, 2017.

[28] Henkel, “Loctite EA 7000 Aero Epoxy Film Adhesive Technical Process Bulletin”.

[29] ASTM D3039 / D3039M-17, “Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials,” ASTM International, West Conshohocken, PA, 2017, www.astm.org.

[30] ASTM D790-17, “Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials,” ASTM International, West Conshohocken, PA, 2017, www.astm.org.

[31] ASTM D6272-17, “Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending,” ASTM International, West Conshohocken, PA, 2017, www.astm.org.

[32] ASTM D5766 / D5766M-11, “Standard Test Method for Open-Hole Tensile Strength of Polymer Matrix Composite Laminates,” ASTM International, West Conshohocken, PA, 2011, www.astm.org.

[33] L. K. Grunenfelder and S. R. Nutt, “Void formation in composite prepregs–Effect of dissolved moisture,” *Composites Science and Technology,* vol. 70, no. 16, pp. 2304-2309, 2010.

[34] G. T. Rochelle, “Amine scrubbing for CO2 capture,” *Science,* vol. 325, no. 5948, pp. 1652-1654, 2009.

[35] H. Zhu, B. Wu, D. Li, D. Zhang and Y. Chen, “Influence of voids on the tensile performance of carbon/epoxy fabric laminates,” *Journal of Material Science Technology,* vol. 27, no. 1, pp. 69-73, 2011.

[36] A. G. Stamapoulos, K. Tserpes, P. Prucha and D. Vavrik, “Evaluation of porosity effects on the mechanical properties of carbon fiber-reinforced plastic unidirectional laminates by X-ray computed tomography and mechanical testing,” *Journal of Composite Materials,* vol. 50, no. 15, pp. 2087-2098, 2016.

[37] M. R. Wisnom, T. Reynolds and N. Gwilliam, “Reduction in interlaminar shear strength by discrete and distributed voids,” *Composites Science and Technology,* vol. 56, pp. 93-101, 1996.

[38] M. L. Costa, S. F. M. Almeida and M. C. Rezende, “Critical void content for polymer composite laminates,” *AIAA Journal,* vol. 43, no. 6, pp. 1336-1341, 2005.

[39] R. M. O'Higgins, M. A. McCarthy and C. T. McCarthy, “Comparison of open hole tension characteristics of high strength glass,” *Composite Science and Technology,* vol. 68, pp. 2770-2778, 2008.

[40] D. W. Adkins and R. B. Pipes, “End effects in scarf joints,” *Composites Science and Technology,* vol. 22, pp. 209-221, 1985.

[41] S. B. Kumar, I. Sridhar, S. Sivashankar, S. O. Osiyemi and A. Bag, “Tensile failure of adhesively bonded CFRP composite scarf joints,” *Materials Science and Engineering B,* vol. 132, pp. 113-120, 2006.

[42] R. D. S. G. Campilho, M. F. S. F. de Moura, A. M. G. Pinto, J. J. L. Morais and J. J. M. S. Domingues, “Modelling the tensile fracture behaviour of CFRP scarf repairs,” *Composites: Part B,* vol. 40, pp. 149-157, 2009.

[43] C. H. Wang and A. J. Gunnion, “On the design methodology of scarf repairs to composite laminates,” *Composites Science and Technology,* vol. 68, pp. 35-46, 2008.

[44] M. Ridha, V. B. C. Tan and T. E. Tay, “Traction-separation laws for progressive failure of bonded scarf repair of composite panel,” *Composite Structures,* vol. 93, pp. 1239-1245, 2011.

[45] A. Y. Zhang, D. H. Li, D. X. Zhang, H. B. Lu, H. Y. Xiao and J. Jia, “Qualitative separation of the effect of voids on the static mechanical properties of hygrothermally conditioned carbon/epxoy composites,” *Express Polymer Letters,* vol. 5, no. 8, pp. 708-716, 2011.

[46] J. S. Yoo, V. H. Truong, M. Park, J. H. Choi and J. H. Kweon, “Parametric study on static and fatigue strength recovery of scarf-patch-repaired composite laminates,” *Composite Structures,* vol. 140, pp. 417-432, 2016.

[47] S. Sisodia, E. K. Gamstedt, F. Edgren and J. Varna, “Effects of voids on quasi-static and tension fatigue behaviour of carbon-fibre composite laminates,” *Journal of Composite Materials,* vol. 49, no. 17, pp. 2137-2148, 2015.

Tables

Table 1: Porosity and mechanical properties of patch and 1D repair laminates

Process		Porosity (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Flexural modulus (GPa)	Flexural strength (MPa)	Interlaminar shear strength (MPa)
Patch only	Hotbonding	0.5 ± 0.1	44 ± 2	580 ± 47	34 ± 1	681 ± 27	65 ± 3
	DVD	0.2 ± 0.1	45 ± 2	615 ± 39	37 ± 1	745 ± 10	63 ± 2
	Autoclave	0.0 ± 0.0	45 ± 1	658 ± 19	40 ± 2	746 ± 29	67 ± 2
1D repair*	Hotbonding	4.7 ± 0.6	42 ± 2	578 ± 26	38 ± 3	615 ± 54	51 ± 2
	DVD	0.4 ± 0.3	44 ± 1	593 ± 54	41 ± 1	806 ± 51	66 ± 2

*Thickness of adhesive film not considered for property calculations

Table 2: Improved DVD process parameters

Process	Heating rate (°C/min)	Soak temperature (°C)	Holding time (min)	Compaction time (min)
Original	1.7	104	30	30
Improved DVD 1	1.7	104	15	5
Improved DVD 2	1.7	114	15	5
Improved DVD 3	5.5	114	15	5

Table 3: Porosity and mechanical properties of improved DVD cured 1D repair laminates

Process	Porosity (%)	Flexural modulus (GPa)	Flexural strength (MPa)	Interlaminar shear strength (MPa)	3D repair strength (MPa)
Original	0.4 ± 0.3	41 ± 1	806 ± 51	66 ± 2	570 ± 5
Improved DVD 1	0.3 ± 0.2	41 ± 1	763 ± 26	66 ± 2	-
Improved DVD 2	0.4 ± 0.3	40 ± 1	785 ± 31	67 ± 2	-
Improved DVD 3	0.4 ± 0.2	42 ± 3	782 ± 26	68 ± 2	592

Figure 1: Double vacuum debulking (DVD) set up.

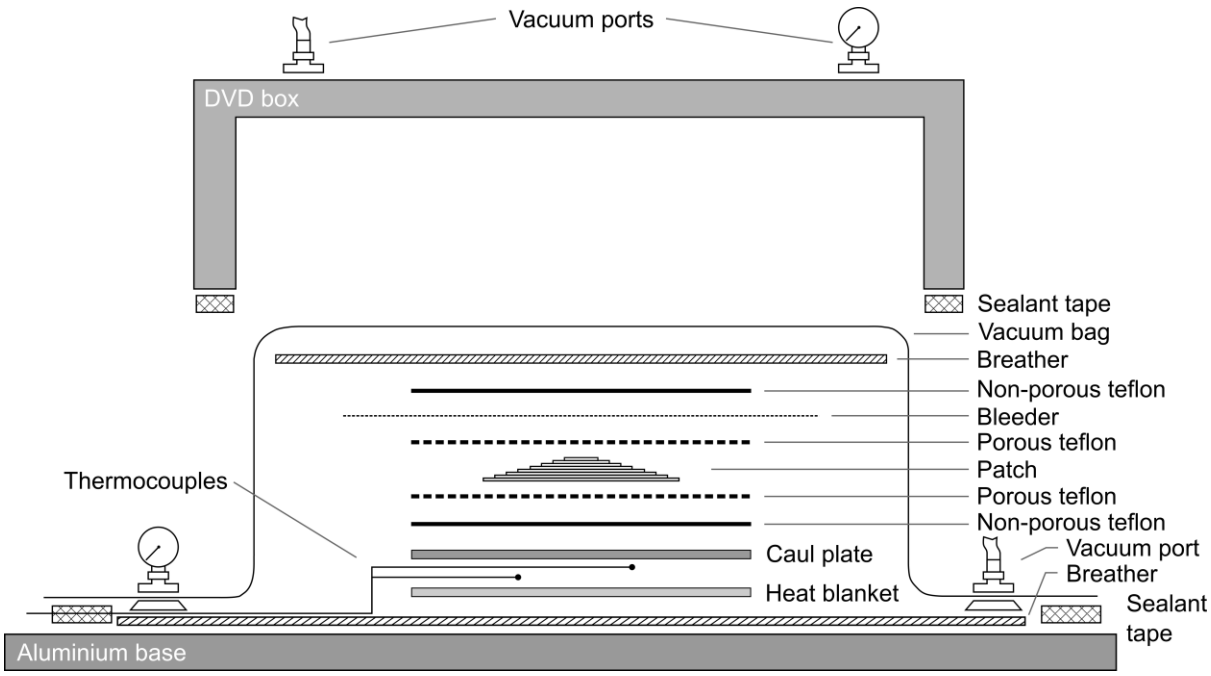


Figure 2: Repair test specimen configurations: (a) 1D repair (b) Scarf lap joint, (c) 2D repair (top view), (d) 2D/3D repair (side view), (e) 3D repair (top view), and (f) 3D repair test specimen

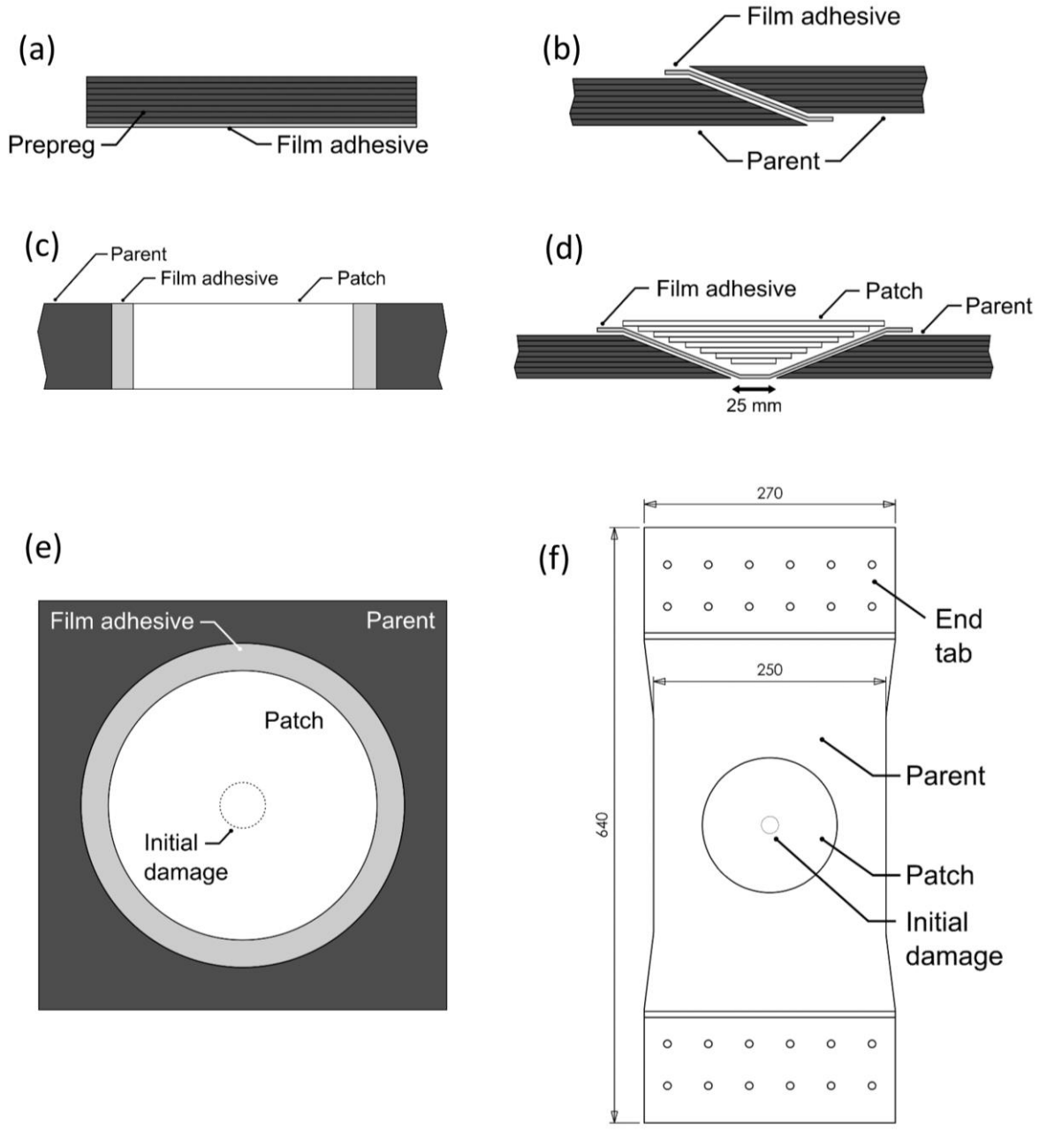


Figure 3: Cure content and glass transition temperature of pre-cured prepreg.

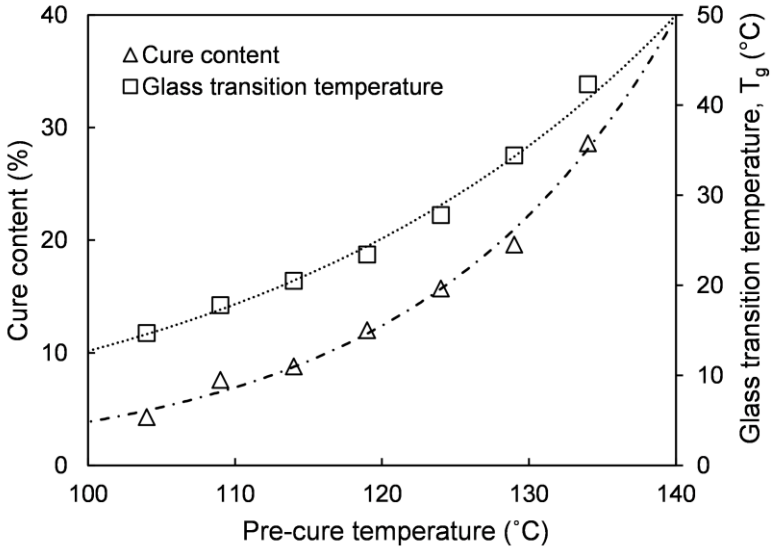
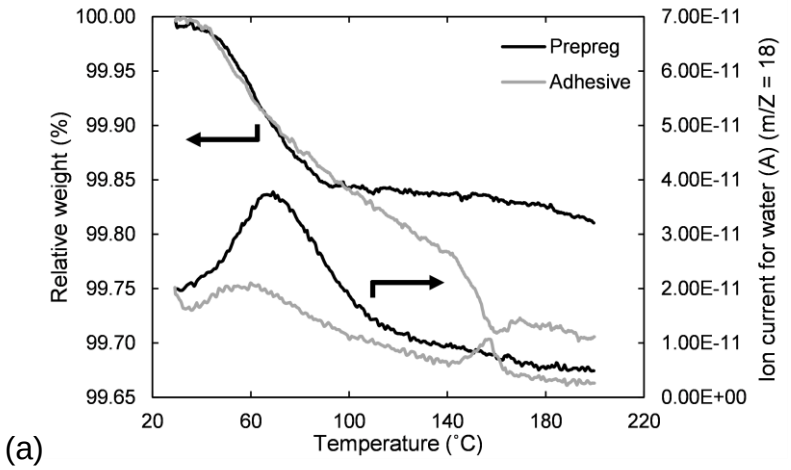
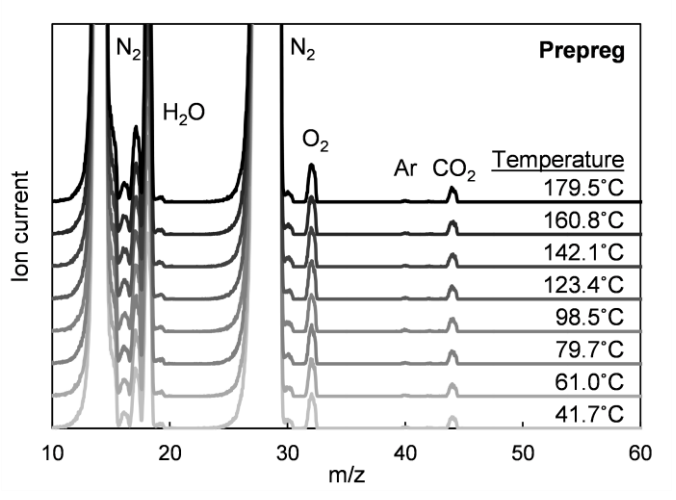


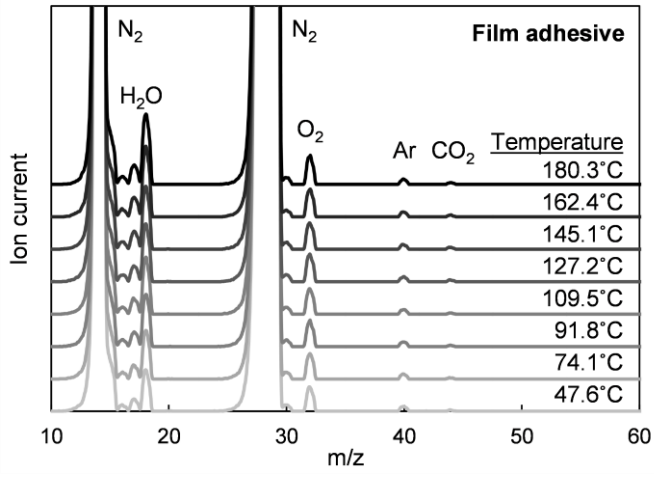
Figure 4: Temperature scan plots from mass spectroscopy with (a) relative weight loss and water release profile from prepreg and film adhesive during heating phase, (b) mass spectroscopy scan of prepreg material with temperature ramp, and (c) mass spectroscopy scan of film adhesive with temperature ramp.



(a)



(b)



(c)

Figure 5: Comparison of cure progression of carbon fibre prepreg and film adhesive in terms of viscosity changes.

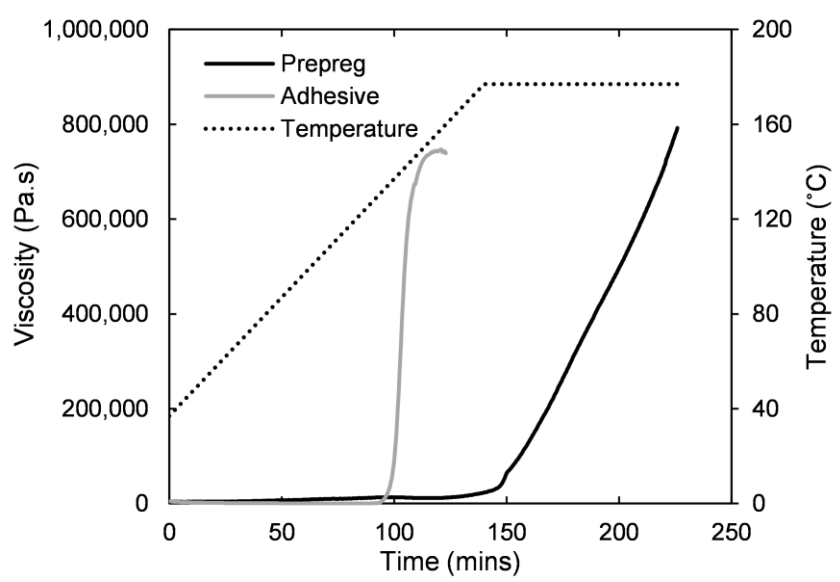
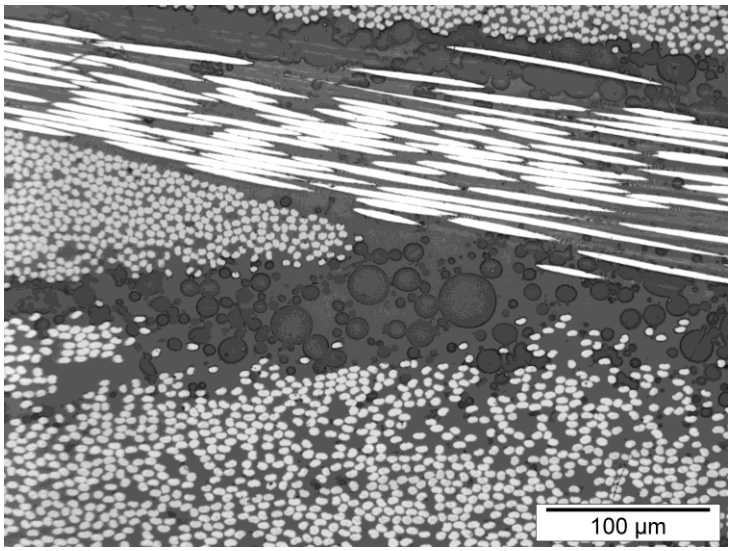
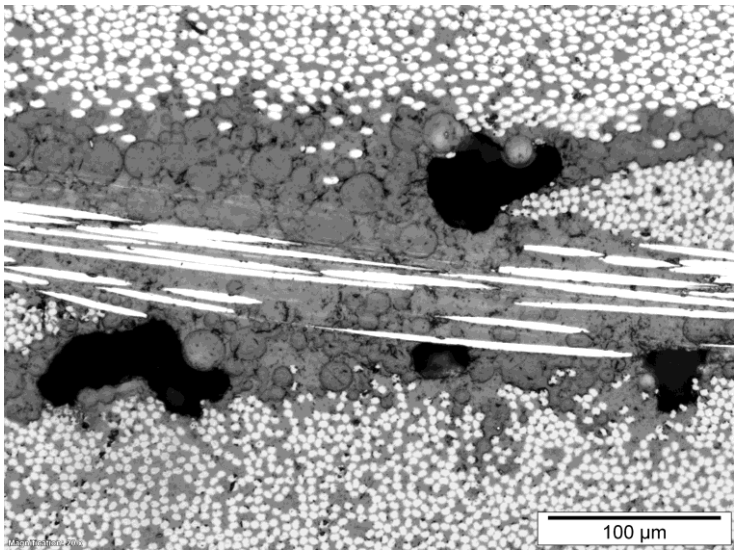


Figure 6: Optical micrographs of composite laminates for (a) autoclave, (b) hotbonding, and (c) DVD processing.

(a)



(b)



(c)

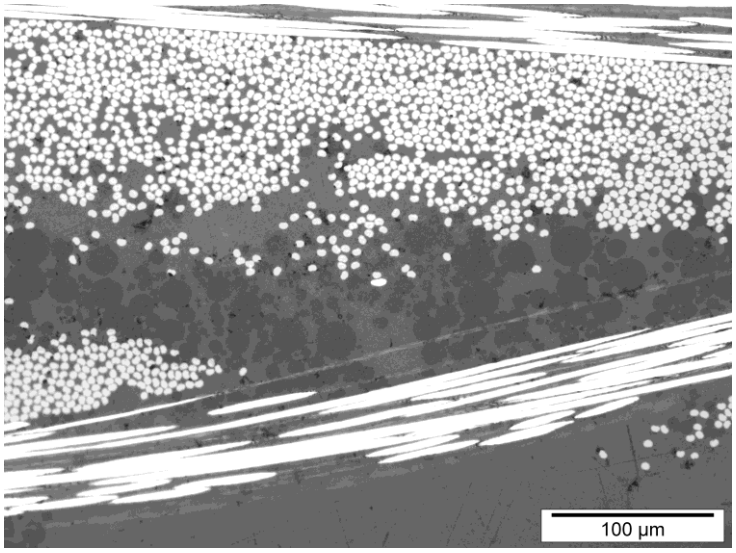
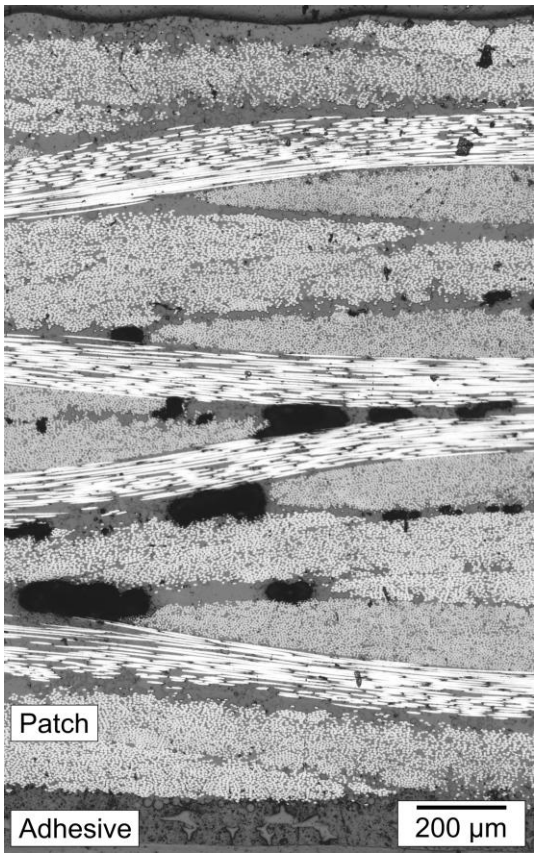


Figure 7: Optical micrographs of 1D repair patches for (a) hotbonding and (b) DVD processing.

(a) 

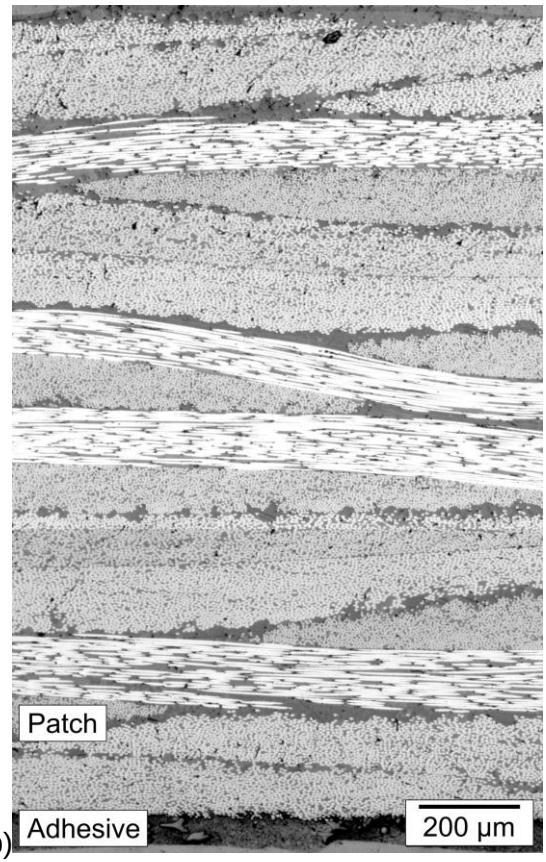
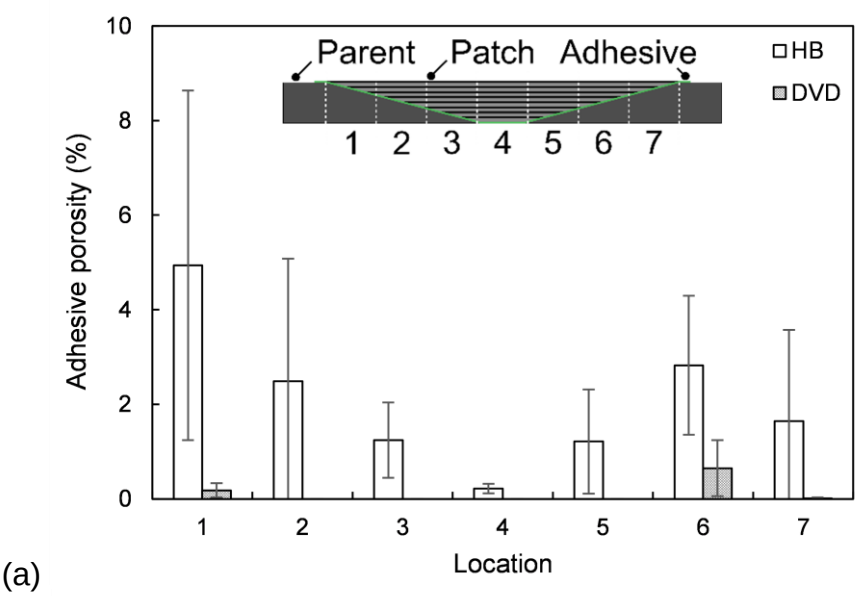
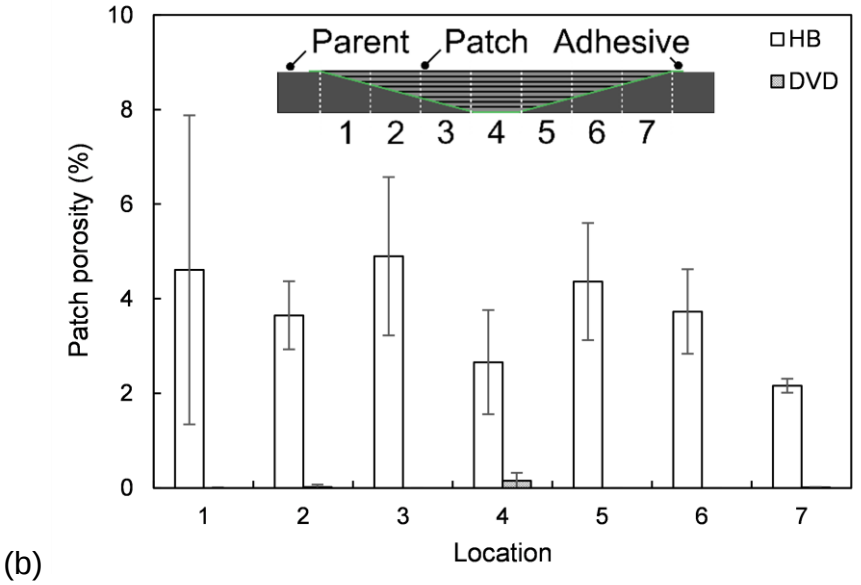
(b) 

Figure 8: (a) patch and (b) adhesive porosity in 3D repair.

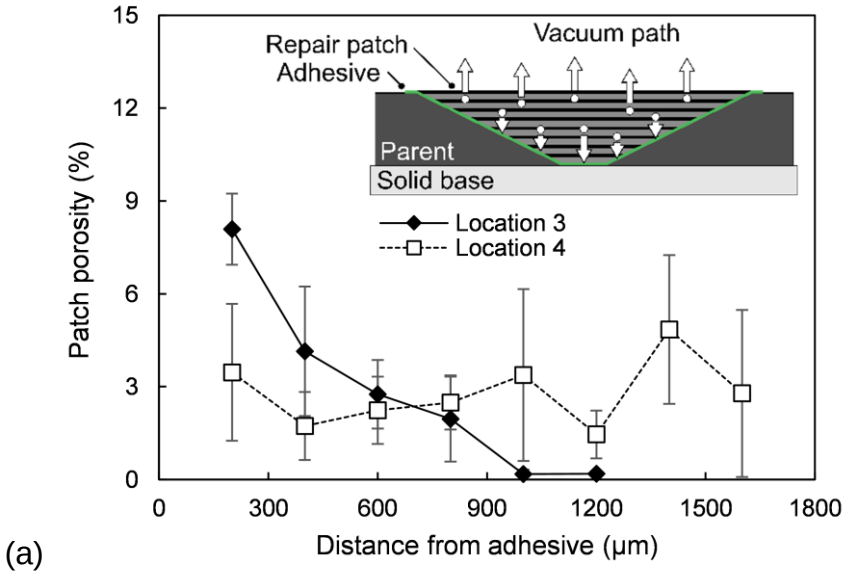


(a)

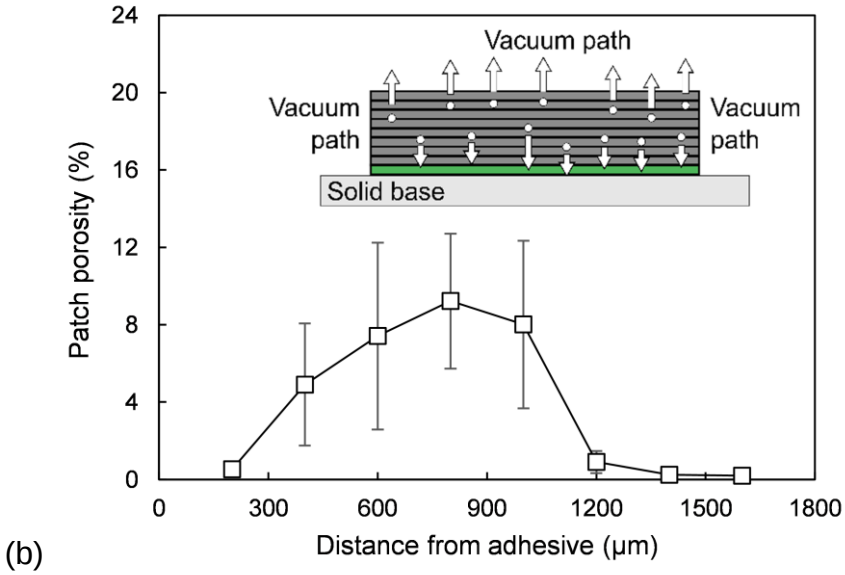


(b)

Figure 9: Porosity analysis of patch versus distance from adhesive layer for repair with hotbonding curing for (a) 3D repair and (b) 1D repair. The total laminate thickness is 1.8mm.



(a)



(b)

Figure 10: Strength comparison of open-hole tension, 2D repair, scarf lap, 3D repair, and parent material. Repair strength exceeds open-hole tensile strength benchmark.

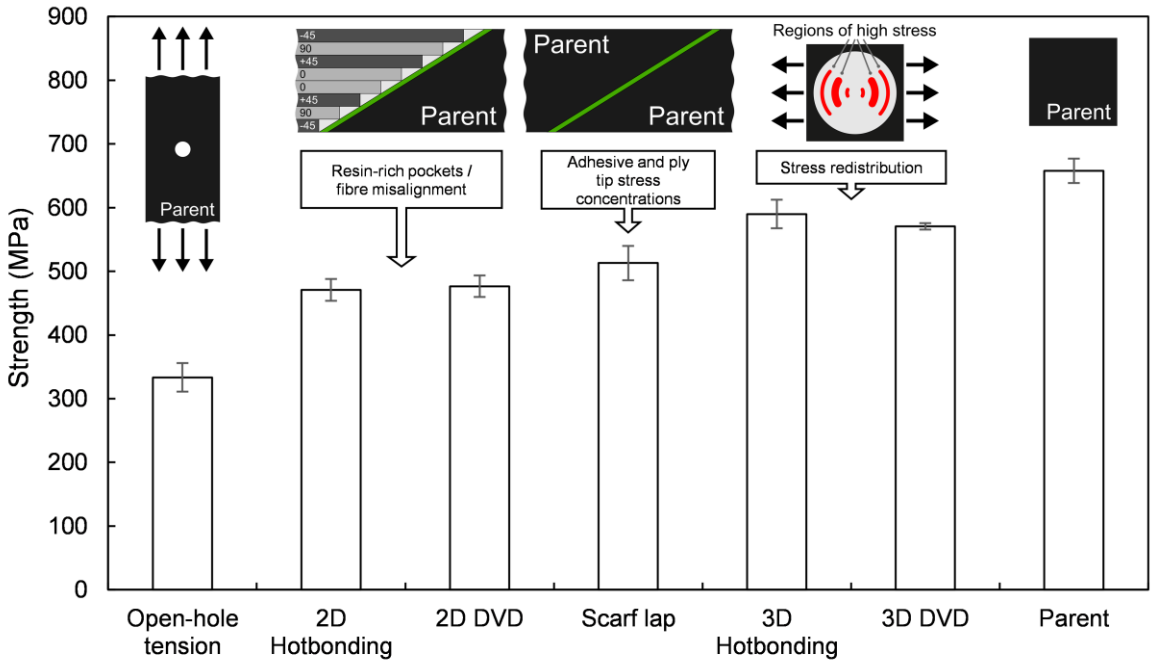


Figure 11: Failure locations of (a) 2D scarf lap and scarf repair joints with (b) hotbonding process and (c) DVD process.

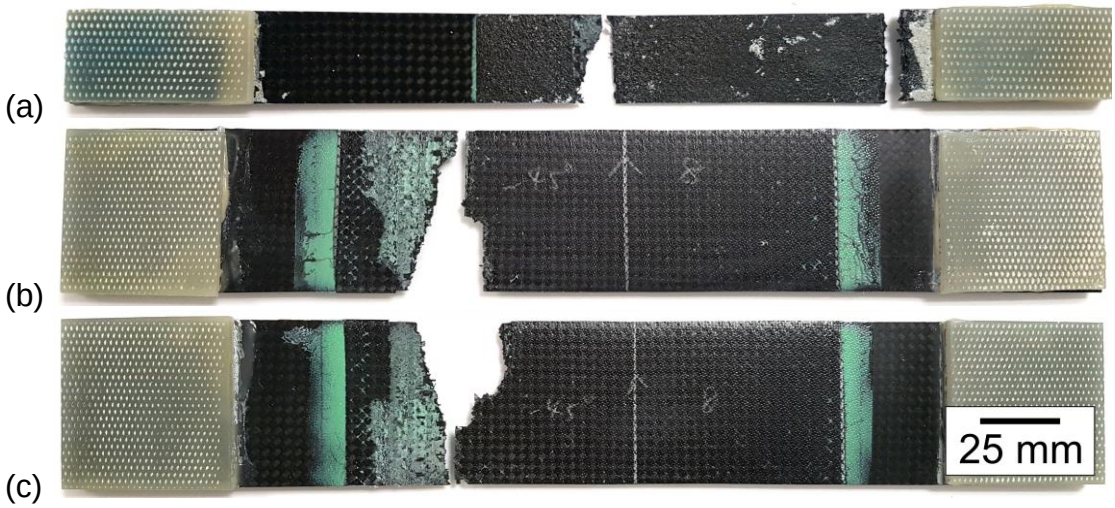
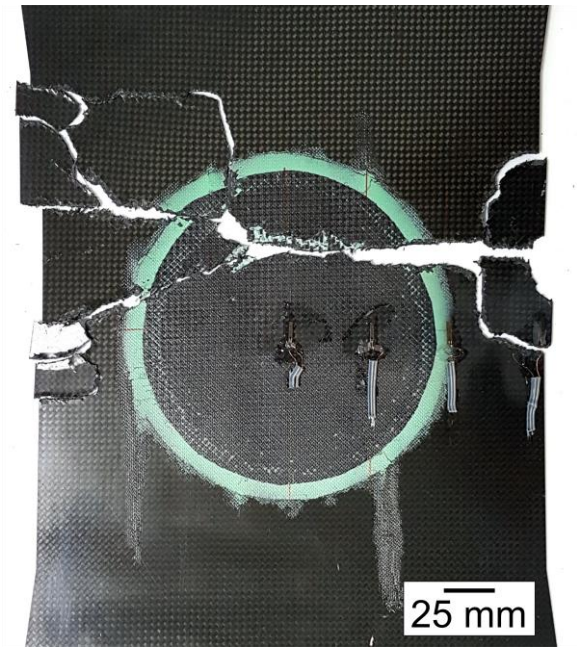


Figure 12: Representative failure patterns of 3D scarf repairs for (a) hotbonding and (b) DVD process.

(a) 

(b)