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Tensile Response of Adhesively Bonded Composite-to-composite Single-lap Joints in the Presence of Bond Deficiency

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Abstract

This paper studies the quasi-static tensile response of adhesively bonded composite-to-composite single-lap joints in the presence of weak and kissing bonds, as an attempt for characterisation of bond deficiencies likely to occur in polymer composite bonded repair. Cytec FM®94 adhesive film (0.25mm nominal thickness) was used for all joints to bond two 2mm-thickness carbon fibre polymer composite laminates manufactured from unidirectional Hexcel M21/T800S pre-pregs. Peel-ply surface treatment was used for all joints. The bonds were deteriorated via five methods: pre-curing the centre of bond area prior to the cure of the bond edges, increasing the curing temperature rate, reducing the curing time, and embedding PTFE films over the centre of the bond. For the last method, the studies were carried out by embedding PTFE films on one and two sides of the adhesive film. The bond deterioration was followed by non-destructive inspections using ultrasound C-scanning. The ultimate failure load of the joints with defected bonds (i.e. weak and kissing bonds) was measured and compared to that of the joints with no defect (i.e. good bonds). It was found that rapid curing and short-time curing reduces more than 50% of the load carrying capacity of the single-lap joins in tension while the joints with weak bonds introduced by pre-curing of a large area of the bond (>60%) can take up more than 65% of the ultimate load of the joint with good bond. Also, optical microscopy of the bond surfaces after failure showed changes in failure type for the rapid and short-time cure, strongly correlated with their significant failure load reduction.

Keywords: Single-lap joint; Weak bond; Degree of cure; Composite repair; Rapid cure

1. Introduction

The global shift from bolted to bonded lightweight composite joints has been well-known as an environmentally friendly dictated regulation in critical transport structures e.g. in aerospace industry [1-4]. Furthermore in the 2015-£46bn aircraft Maintenance, Repair and Overhaul (MRO) sector [5], the globally growing market for polymer composites has necessitated the use of intense rapid bonded repair such as use of easy-to-apply adhesive films, accelerated induction heating [6] and atmospheric plasma treatment for more elevated surface energy [7]. However, no matter how the adherend surfaces are cleaned or etched so as to prepare for bonding, the underlying science behind the effect of curing trend on the bond integrity is lagging behind. The research in knowledge-based repair should also have the certification rules (e.g. those in the FAA’s certification for Repair and Alternations to Composite and Bonded Aircrafts [8]) at its very core before being accredited by industry, attracting the MRO market.

The quality of the repaired structure in strong relation with the variabilities caused by process parameters (e.g. cure time, temperature and heating rate) introduces an urgent need for controllable cure. But any variation in curing method can potentially behave as a defect introducer to the bond integrity. Though a bond defect (e.g. weak or kissing bonds) can be introduced by contaminations or voids during the bonding process, and is challenging to detect [9], zero-thickness bond deficiencies caused by improper cure is more challenging to analyse, inspect and control.
The current ongoing research at The Enhanced Composites and Structures Centre in Cranfield University provides a comparative study for the effect of bond deficiency, introduced by a number of curing methods and using PTFE films, on the quasi-static response of adhesively bonded composite-to-composite single-lap joints. The research also provides initial examination of the capability of C-scanning technique for detection of such defects.

2. Materials and experiments

2.1. Joint materials, geometry and manufacture

A 2mm-thickness carbon fibre-reinforced composite panel was manufactured from aerospace grade unidirectional Hexply® M21/T800S prepreg using manual lay-up and autoclave cure, having the stacking sequence of [0° 90° 45° -45°]. The joint laps were then extracted from the laminate using abrasive water jet technology (dimensions are given in Figure 1(a)).

The joints figure were bonded as advised by ASTM D5868 [10]. Cytec FM10 94 modified epoxy adhesive film, designed for high temperature and moisture resistant bonding applications, was applied to the 25mm×25mm overlap region of one adherend. The surface preparation for all laminates was obtained by use of peal ply, to provide a textured surface, which was removed from the laminate just prior to application of adhesive to provide a contaminant free surface. Note that the nominal thickness of the bond was 0.25 mm which is smaller than that recommended [10] (0.76 mm). Curing of the standard joints was performed in a heating oven at 120°C, using a ramp rate of 2°C/min from ambient, and holding at 120°C±5°C for 40 minutes. This was in accordance with the adhesive specifications, and sufficient to reach the bond maximum strength. The constant pressure of 0.28 MPa was uniformly applied using a rig with mechanical fasteners and clamping plates.

Six categories of single-lap joints with and without deficiencies were manufactured, three specimens per category, as listed in Table 1, and described below:

1. Good bonds (G) prepared according to the FM10 94 specification
2. Weak bonds (WP) manufactured by:
   - pre-curing a 20mm×20mm square region of the centre of the 25mm×25mm adhesive area before bonding, on one adherend only,
   - adding adhesive at the outer region (2.5mm border at perimeter),
   - clamp and cure the joint
3. Weak bonds(WR) introduced by rapid heating with temperature rate of 4°C/min
4. Weak bonds(WT) introduced by reducing the curing time from 40min to 10min (i.e. 75% cure time reduction after reaching 120°C)
5. Kissing bonds (KS) introduced by embedding one 20mm×20mm 0.1mm-thickness PTFE layer over the centre of the overlap region offset to one side of the bond
6. Kissing bonds (KD) introduced by embedding two 20mm×20mm 0.1mm-thickness PTFE layers over the centre of the overlap region attached to each joint adherend

Figure 1(b) schematically shows five modified categories with the dashed line representing the weak bonds. The PTFE films (categories KS and KD) were embedded at centre not the bond run-outs, to avoid pre-cracking. The WP category was then introduced to the centre to provide comparisons with the KS and KD cases.

Single-lap bonded joints experience stress concentrations at the free ends (run-outs) of the overlap region. As the centre of the overlap is approached, the load stress (shear and peel) significantly reduce, i.e. the centre of the bond is ineffective at transferring load [11, 12]. This stress variation along the bond is the reason that the authors decided to affect a large central portion of the bond relative to the size of the overlap, and enter the high stress region without inserting pre-cracks at the free ends (20mm×20mm region was deteriorated at the centre of 25mm×25mm overlap region, thus 64% deteriorated).

![Figure 1](image-url)

**Figure 1.** (a) Bonded joint geometry (all dimensions are given in mm); (b) schematic of weak bond categories (dashed lines represent regions where bond was weakened).

| Table 1. Bond categories in single-lap joints (pressure = 0.28MPa) |
|-----------------------------|-----------------------------|-----------------------------|
| **Category** | **Deficiency method** | **Label** | **Cure condition** |
| Good bond | - | G1,2,3 | 120°C, 2°C/min |
| Weak bond | Bond centre pre-cure (20mm×20mm) | WP1,2,3 | 120°C, 2°C/min |
| Weak bond | Rapid curing | WR1,2,3 | 120°C, 4°C/min |
| Weak bond | 75% reduced cure time | WT1,2,3 | 120°C, 2°C/min |
| Kissing bond | Single-side PTFE bond (20mm×20mm) | KS1,2,3 | 120°C, 2°C/min |
| Kissing bond | Double-side PTFE bond (20mm×20mm) | KD1,2,3 | 120°C, 2°C/min |

2.2. Test setup

The test scenario to investigate the quasi-static response of the bonded single-lap joints is a uniaxial tensile test carried out in accordance with ASTM D5868. Testing was carried out
using a uniaxial tensile test frame fitted with a 30kN load cell, wedge-action grips and a crosshead speed of 1mm/min.

2.3. Non-destructive inspection

Non-destructive inspection (NDI) of the joints was carried out in collaboration with the Through-life Engineering Services Centre, using ultrasound technique by means of an immersion C-scan facility with the gain of 18-23dB. This is known to be one of the most widely used techniques to inspect bonded joints as a simple and effective means [13] that has proved reliable for aerospace applications. The speed of scanning was set at approximately $30\text{mm/sec}$ and an ultrasound frequency of 5MHz (Sonatest v64 system).

2.4. Optical microscopy

Optical images of the bond area after joint ultimate failure were captured using optical microscopy. The microscopy was conducted on a Prizm image acquisition system. Images were then taken at points of interest in each category.

3. Results and discussion

3.1. Failure load

Consistent data were obtained for each bond category. Figure 2. The KD (double-side PTFE) bond provided the most reproducible data, and WR (rapid cure) bond provided the largest spread in failure loads. The weak joint with the precured bond (WP) represents an incomplete or partial curing scenario in actual bonded repair applications and is of greatest interest. The results in Figure 2 show that despite curing the centre prior to bonding the composite adherends, the joint has been able to sustain 67% of the failure load of the good bond (no defects). Whilst rapid curing using the temperature rate of $4^\circ\text{C/min}$, twice the specified rate, produced a bond which only reached 50% of the standard joint's failure load. This is approximately the same response as the joint with a double-side PTFE. Among all joint categories, the bonded joint with the reduced cure time exhibited with the greatest reduction (79%) in the failure load.

3.2. Optical microscopy images

All joints were observed to fail at the bond interface. The images from each adherend contact faces (A and B) of the bond of one representative joint from each category (except KD) are shown in Figure 3. Bright and dark regions represent the adhesive and the surface of the composite respectively. The images for the bonds incorporating PTFE film (KS and KD) were very similar and only images for the KS joint are presented. Any cohesive failure behavior of the adhesive was minor, with most joints exhibiting adhesive failure at the adherend film adhesive interface. This was evident from the microscopic images in Figure 3 where the adhesive has been observed bonded on one adherend only and peeled from the other. The only exception was the bonded joint cured in a time shorter than the others (WT), where a combination of interface and cohesive failure was observed. This is expected as in a

![Figure 2. Comparison of failure loads in joints with different bond quality](image-url)

![Figure 3. Microscopic images of the bonded joints after failure](image-url)
shorter curing time, the bulk of the epoxy adhesive has not reached to its ultimate strength, and tends to deform with greater ductility and lower strength. The observed sudden drop in load level occurring at the ultimate failure point is also an indication of the dominant interfacial failure.

The adhesive films contain carrier cloth which, based on our observations, did not fail (see e.g. WT(B) in Figure 3), and thus is believed to be directing damage toward the interface. The bond thickness was relatively low (0.25mm) compared to the Standard [10] recommendation (0.76mm). This difference should reduce the joint eccentricity but the reduced thickness is expected to experience higher peel strain from geometry effects.

The images, figure 3, are consistent with the failure load results, figure 2. The precured bond (WP) and the kissing bonds with PTFE films (KS) failed approximately at a same load and their failure mechanism appears to be similar, Figure 3. It should be noted that due to the unsymmetric bond area in the WP and KS categories, unsymmetric secondary bending is introduced, i.e. the composite adherend at the weak bond side undergoes higher bending strain.

Also of interest is the bond cured by rapid heating (WR). As seen, the morphology of the failuresurface is different from the others. It has been observed that higher ramp rate could cause increased voids and increased variation in degree of cure due to temperature gradients throughout the bonded joint [14]. This significantly affects the bond and structural integrity. This category with the one with reduced curing time (WT) has both shown the lowest failure loads in Figure 2.

3.3. NDI images

The C-scanned images of the joints’ overlap region is shown in Figure 4, carried out using the parameters given in section 2.3 (the outer dashed line represents the overlap bonded region for all joints). Note that the C-scan images represents attenuation of a probe signal through the two composite adherends and the adhesive (bond). However as the significant difference is expected to arise from the adhesive (bond), and not in the adherends, the difference seen in the colour contours is referred to as defects. Taking the good bond with green/yellow contours as the reference for a bond with no defect, apparent differences observed for the rest of the joints are a sign of bond deficiencies. The WR and WT joints have also shown a clear mismatch and difference in the whole overlap region of the bond, compared to the image of the good bonded joint. This is in agreement with the results in Figure 2 where WR and WT had the most significant drop in failure load compared to the good bond. Clearly for the joint with reduced cure time, the C-scan is able to qualitatively capture the significant mismatch in the adhesion and bulk properties of the pre-maturely cured bond. Moreover, the main outcome from the figure is that the ultrasound technique clearly highlights the bond deterioration at the centre of the overlap region for WP (pre-cured bond) and KS (single-PTFE bond). This central region captured by NDI, shown by the arrows in the figure, represents the 20mm×20mm disbond intentionally introduced at the centre of the overlap for WP and KS joints. Surprisingly, the disbond region is not observed for the KD joint which has two PTFEs at either side of the bond. Understanding this requires further investigation.

![Figure 4](image4.png)

Figure 4. C-scan images of the bonded joints before test (outer dashed line represents the overlap bonded region of the joints (25mm×25mm) while inner dashed lines with arrows represents 20mm×20mm disbond at the centre of the overlap for WP and KS joints)

4. Conclusions

The current research provided a comparative study of adhesively bonded joints with bond deterioration. A number of techniques were used to introduce ‘defects’ to the central bond area of the composite-to-composite single-lap joints. Three main curing strategies were selected to prepare such ‘defects’. It was found that a bonded joint with any defect, non-standard cure or contaminant, has a reduced strength. The ‘kissing bond’ defect was found to cause the lowest ‘strength reduction’ (categories WP and KS with strength reduction of 33% and 40%). Rapid cure was found to cause a 50% reduction in failure load. The effect of curing time was also quantified, and found that, in terms of a curing parameter, it has the most significant effect on the bond mechanical response, also observed using NDI. The NDI ultrasound technique was also capable of capturing the 20mm×20mm central disbond region of the bond for WP and KS joints.

The study on the failure load in relation to the curing parameters showed that control of the cure process is as important as ensuring good surface preparation (to avoid kissing bond) and absence of contaminants when producing adhesive joints.
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