THE EFFECTS OF PRE-BOND MOISTURE AND ENVIRONMENTAL AGING ON THE FRACTURE TOUGHNESS OF BONDED COMPOSITE REPAIRS

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ABSTRACT

The mechanical performance of bonded composite repairs depends on the moisture content of the adherents. After bonding, the operating conditions and environmental aging also play a fundamental role in the joint durability and strength and they should be investigated to understand the long term repair behaviour. An experimental investigation has been carried out on the effect of pre-bond moisture on the fracture toughness (Mode I) and failure behaviour of repair joints bonded with an adhesive film. Pre-cured substrates were subjected to different conditioning (in a climatic chamber in a humid environment) and drying procedures monitoring the final moisture content (high / medium / low) prior to bond. In a second curing step, they were joined to the repair materials using an out of autoclave process with only vacuum pressure. The co-bonded specimens were tested in different conditions (room temperature/ambient, cold/ambient and room temperature/wet) in order to determine the fracture toughness under Mode I loading, as an indication of the bonding quality and strength. Fractographic inspection of the fractured surfaces was conducted to investigate the failure modes. The response to pre-bond moisture was found to be modulated by the reinforcement, the environmental history of the pre-cured adherent and the post-bond environmental aging. This knowledge is essential for the proper definition of the repair process and for the substantiation of the repair.

1 INTRODUCTION

Bonded repairs of composite structures in civil aircrafts are often performed in field conditions, which introduces severe restrictions in materials processing as compared to those used in a manufacturing plant (out of autoclave cure, moisture control of environment and components, drying methods, etc.). One very frequent repair process involves producing a joint between a pre-cured adherent (substrate) and the uncured repair patch by means of an adhesive film, being both cured simultaneously under vacuum pressure. The mechanical performance and durability of the resulting joint is highly dependent on the surface state of the adherents prior to the bonding process (surface preparation, presence of chemical substances, moisture content, etc.). Therefore, there is a clear interest to define repair procedures that could guarantee a proper adhesion between the substrate and the patch. In particular, the moisture content of the adherent prior to bonding is of high concern. Its effect on the joint performance should be clarified in order to define an acceptable moisture threshold and to find a suitable procedure to dry the adherents before bonding. After bonding, the operating conditions and environmental aging also play a fundamental role in the joint durability and strength and they should be investigated to understand the long term repair behaviour.

In this study, one carbon epoxy unidirectional repair prepreg and one adhesive film were used to produce co-bonded joints with several substrates. The substrates (also carbon epoxy unidirectional prepregs) were cured in autoclave and then submitted to different drying steps, conditioning (in a climatic chamber in a humid environment) and drying procedures monitoring the weight gain/loss and the final moisture content prior to bond. In a second curing step, they were joined to the repair materials using an out of autoclave process with only vacuum pressure. The co-bonded specimens were tested in different conditions (room temperature/ambient, cold/ambient and room temperature/wet) in order to determine the fracture toughness under Mode I loading and the failure modes.

2 EXPERIMENTAL

The repair system consisted of one carbon epoxy unidirectional repair prepreg and one adhesive film, both intended for out of autoclave cure. Four different adherents (also carbon epoxy unidirectional prepregs) were used.

The pre-cured panels were submitted to several drying, conditioning and drying cycles monitoring the weight gain/loss and the final moisture content (high, medium and low) prior to bond. Surface preparation prior to bond was done by sanding and cleaning with pure water followed by a water break test. The co-cured bonded joints were processed according to the sketch shown in Fig. 1: plies of the un-cured repair prepreg and the repair adhesive film were placed on top of an already pre-cured panel and cured under vacuum.

The co-bonded specimens were kept prior to test in ambient laboratory conditions wrapped in plastic bags or introduced in a climatic chamber for accelerated aging until equilibrium. Mechanical tests were performed at room temperature and at -55ºC.

The Mode I fracture toughness tests on double cantilever beam (DCB) specimens were performed according to AIRBUS test method AITM1-0053 [1]. The test was performed by means of a 100 kN MTS Insight test machine with a 1 kN load cell. In this test method, the Mode I fracture toughness, GIc, is calculated from the area integral under the load-displacement curve and the crack length increment.

A fluorescence optical microscope Leica DMR-XA was used to study the fracture surfaces in sections transversal to the crack propagation direction after the completion of the mechanical test. The samples were prepared by cutting pieces of the semi-specimens, including them in an epoxy matrix and then grinding and polishing. Fluorescence mode was used to enhance contrast between the matrix, the fibres and the adhesive.

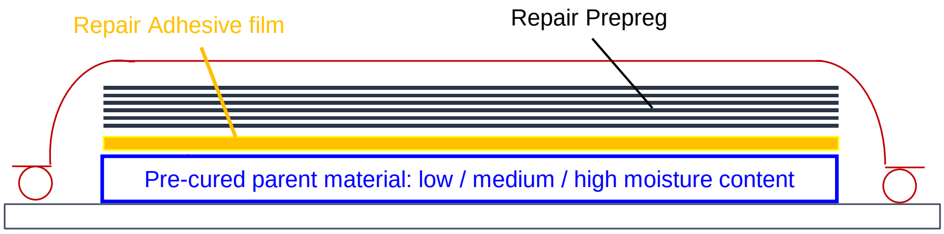


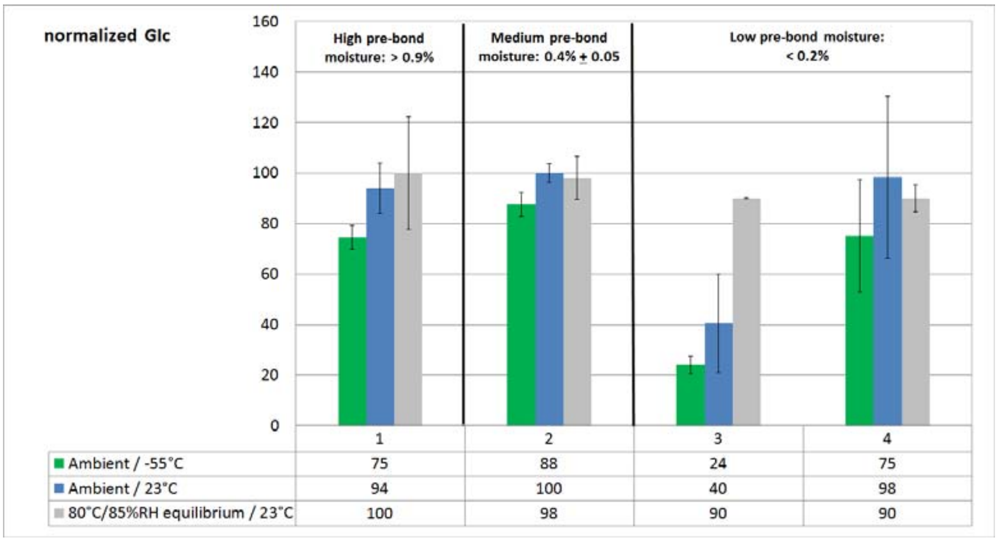
Figure 1: Arrangement of pre-cured adherent, repair adhesive film and repair prepreg during the co-bonding process.

3 RESULTS

3.1 Mode I Fracture toughness results

A representative set of results of Mode I fracture toughness determined in co-bonded specimens with high (> 0.9%), medium (0.4% ± 0.05) and low (< 0.2%) pre-bond moisture content is shown in Fig. 2 for three test conditions, room temperature/ambient, -55ºC/ambient and room temperature/wet.

Values are presented normalized to the highest average value. The presence of high or medium pre-bond moisture produced high GIc values at all tested conditions. With low pre-bond moisture two types of responses were found in the specimens stored at ambient conditions prior to test (room temperature/ambient and -55ºC/ambient) depending on the procedure followed to reach the low moisture content in the pre-cured adherent prior to bond. When the pre-cured panel was submitted to several long drying/conditioning/drying cycles up to low moisture content, the GIc values of the co-bonded specimens were low (specimens labelled 3 in Fig. 2), while if the pre-cured panel was only dried (specimens labelled 4 in Fig. 2), the GIc values were high. But in both cases, when the co-bonded specimens were tested at room temperature after being saturated with moisture in a climatic chamber the difference did not appear anymore and both types had a high GIc value. The path followed to reach high or medium pre-bond moisture in the pre-cured panels did not show any influence in the GIc values.



	1	2	3	4
Ambient / -55°C	75	88	24	75
Ambient / 23°C	94	100	40	98
80°C/85%RH equilibrium / 23°C	100	98	90	90

Figure 2: Fracture toughness of co-bonded specimens prepared with high, medium and low pre-bond moisture.

3.1 Visual and microscopic inspection of fracture surface

First the analysis of the specimens tested at ambient conditions, either at room temperature/ambient or at -55ºC/ambient, is presented.

The failure surfaces of the specimens were inspected visually and recorded by macrophotography. In Fig. 3, the starter crack formed by the Teflon insert was at the left hand end of the specimens, with the crack growth running from left to right. The adherents (the pre-cured substrate and the co-bonded repair prepreg) are identified in the Figure. Two specimens are shown: (a) with medium pre-bond moisture and (b) with low pre-bond moisture, tested at ambient conditions (room temperature/ambient and -55ºC/ambient, respectively) and with normalized GIc values of 78 and 28, respectively. The specimen with medium pre-bond moisture is representative of the specimens labelled 1 in Fig. 2. The specimen with low pre-bond moisture is representative of the specimens labelled 3 in Fig. 2. It is from one set of six specimens which pre-cured substrate had been submitted to long drying/conditioning/drying cycles up to low moisture content.

In the fractured surfaces of the specimen with medium pre-bond moisture (specimen (a) in Fig. 3) the adhesive cannot be seen but only the carbon composite surfaces. The inspection by fluorescence microscopy of the fracture surfaces in sections transversal to the crack propagation (see Fig. 3) revealed that the adhesive had kept well bonded to the pre-cured substrate and had pulled out the co-bonded adherent. Actually, the crack grew in the co-bonded adherent (the repair prepreg) through the lamina with a considerable amount of fibre bridging. The same type of failure was consistently found in the remaining specimens of this set and also in all sets with high/medium pre-bond moisture.

Neither can the adhesive be seen in the specimen with low pre-bond moisture (specimen (b) in Fig. 3). But in this case, the fluorescence microscopy revealed that the adhesive had kept well bonded to the co-bonded adherent (the repair prepreg) and had pulled out the pre-cured substrate. This type of failure was the one regularly found in the sets which pre-cured substrate had been submitted prior to bond to long drying/conditioning/drying cycles (specimens labelled 3 in Fig. 2). In some specimens of this type adhesion failure was also found.

(a)
Medium pre-bond moisture
(specimen type: specimens labelled 2 in Fig. 2)
Tested at room temperature/ambient
Normalized GIc = 78

(b)
Low pre-bond moisture
(specimen type: specimens labelled 3 in Fig. 2)
Tested at -55ºC/ambient
Normalized GIc = 28

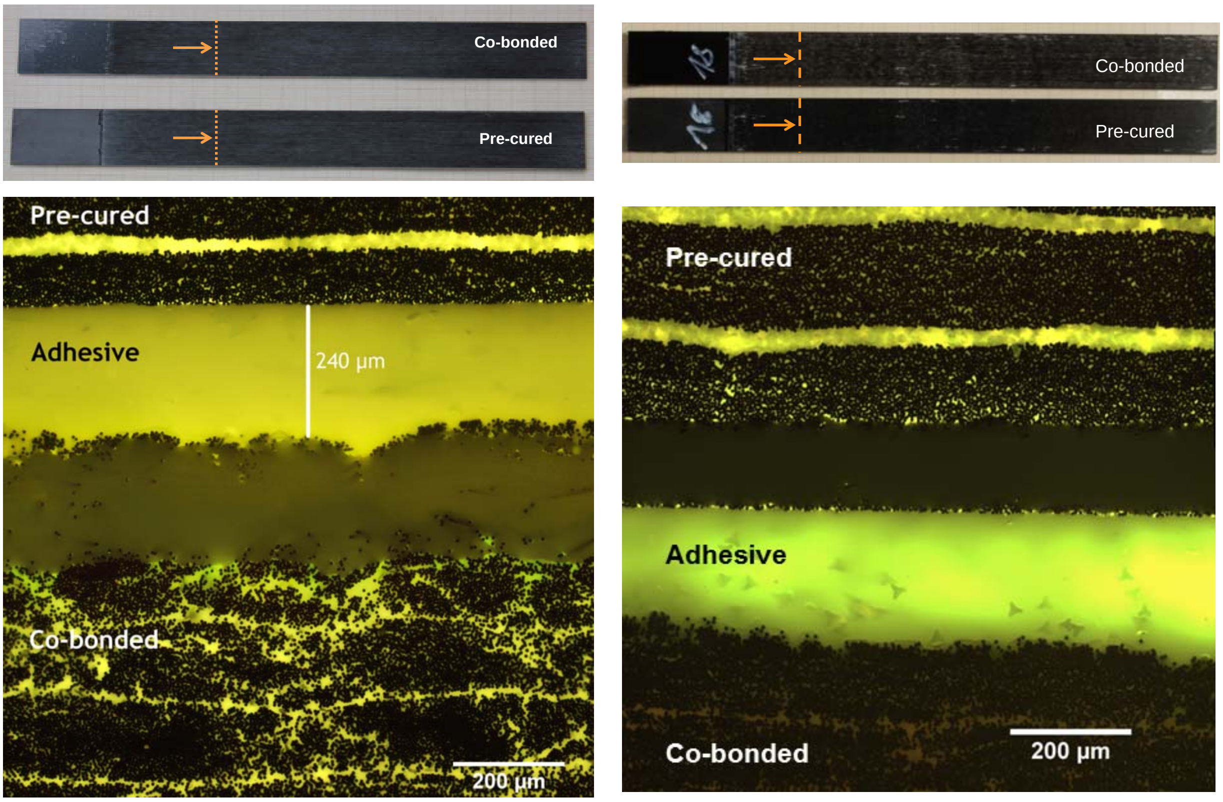


Figure 3: Fractured surfaces and optical micrographs of the transverse cross sections of tested co-bonded specimens with (a) medium pre-bond moisture tested at room temperature/ambient and (b) low pre-bond moisture, tested at -55ºC/ambient

If the pre-cured substrate had been only dried shortly up to low moisture content prior to bond the co-bonded specimens (labelled as 4, in Fig. 2) tested at ambient conditions presented a mix of failure modes. In Fig. 4 (a), the initiation and propagation zones of two specimens of this type, tested at room temperature/ambient, are shown: there is a mix of failure in the co-bonded adherent and failure in the pre-cured substrate as well as small zones with cohesive failure. The presence of failure in the co-bonded adherent contributed to increase the fracture toughness as the crack grew, as indicated above, in the lamina with fibre bridging. The presence of some spots with cohesive failure also contributed to increase the GIc value: the normalized GIc values were 81 and 98.

The effect of post-bond moisture on the failure mode of the co-bonded specimens was also analysed and it is summarized here below.

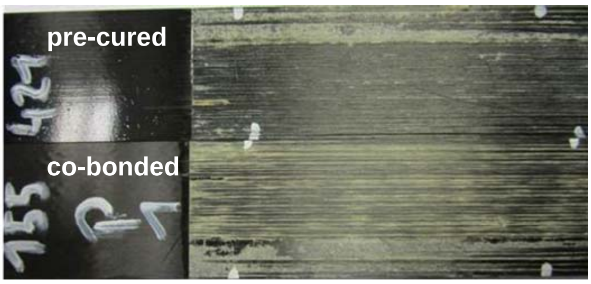
The sets with high or medium pre-bond moisture did not change their behaviour when they were tested after having been saturated with moisture in a climatic chamber. The fracture surfaces were of the same types of those seen for the specimens tested at ambient conditions (see Fig. 3 (a)): the adhesive remained bonded to the pre-cured substrate and pulled out the co-bonded adherent (the repair prepreg).

Fig. 4 (b) shows the initiation and propagation zones in the fracture surfaces of two specimens of co-bonded sets tested at room temperature after having been saturated with moisture in a climatic chamber, i.e., with post-bond moisture. The pre-cured substrate in these sets had been submitted to several drying/conditioning/drying cycles and had residual moisture content prior to bond circa 0. That is, they are of the same type as the specimens labelled 3 in Fig. 2. They presented mixed failure modes, predominantly in the co-bonded adherent and in some cases with a small amount of failure in the pre-cured adherent and/or cohesive failure. The post-bond moisture increased remarkably the fracture toughness (normalized GIc values of 90 and 85), and changed the failure mode. The same behaviour with regards to the effect of post-bond moisture was observed in specimens labelled 4 in Fig. 4, those which pre-cured substrate had been dried shortly prior to bond.

(a)
Low pre-bond moisture
(specimen type: specimens labelled 4 in Fig. 2)
Tested at room temperature/ambient

Normalized GIc = 81

Normalized GIc = 98



(b)
Low pre-bond moisture
(specimen type: specimens labelled 3 in Fig. 2)
Tested at room temperature/wet

Normalized GIc = 98

Normalized GIc = 85

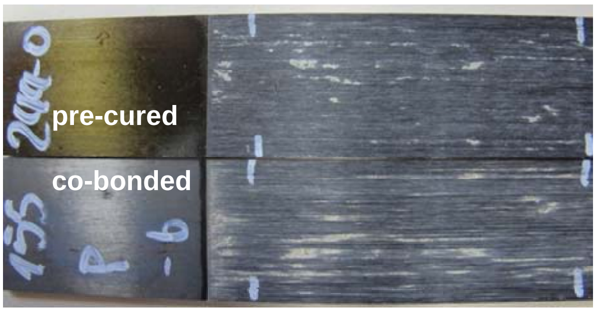


Figure 4: Fractured surfaces of tested co-bonded specimens with (a) low pre-bond moisture, tested at room temperature/ambient and (b) low pre-bond moisture, tested at room temperature/wet

3 CONCLUSIONS

The effect of pre-bond moisture on the fracture toughness (Mode I) and failure behaviour of repair joints bonded with an adhesive film was investigated. Pre-cured substrates (carbon epoxy unidirectional prepregs) were subjected to different drying steps followed by conditioning (in a climatic chamber in a humid environment) and drying procedures monitoring the final moisture content (high / medium / low) prior to bond. In a second curing step, they were joined to the repair materials (also a carbon epoxy unidirectional repair prepreg and an adhesive film) using an out of autoclave process with only vacuum pressure. The co-bonded specimens were tested in different conditions (room temperature/ambient, cold/ambient and room temperature/wet) in order to determine the effect of the operating conditions and environmental aging in the joint durability and strength. Visual and microscopic inspections of the fractured surfaces were conducted to investigate the failure modes.

The path followed to get a low pre-bond moisture in the pre-cured substrate influenced the behaviour of the co-bonded specimens when tested in ambient conditions: low GIc values and failure in the pre-cured adherent (and occasionally adhesion failure) when this had been submitted to several drying/conditioning/drying cycles; high GIc values and mixed failure modes with predominance of failure in the co-bonded adherent when the pre-cured substrate had also low pre-bond moisture but it had been only shortly dried.

However, the post-bond moisture made irrelevant the history of the pre-cured substrate increasing remarkably the fracture toughness and changing the failure mode. The specimens presented mixed failure modes, predominantly in the co-bonded adherent and in some cases with a small amount of failure in the pre-cured adherent and/or cohesive failure.

The presence of high or medium pre-bond moisture with or without post-bond moisture also drove the failure consistently to the co-bonded adherent (repair prepreg). The interphase more affected by the presence of pre-bond moisture was the interphase between the adhesive and the co-bonded adherent (the “wet-wet” interface during the bonding process) indicating that moisture diffusion from the pre-cured adherent, through the adhesive, during curing plays a crucial role. This effect of the pre-bond moisture, driving the failure to the wet-wet interface, had been previously reported for repair co-bonded joints with adhesive films and a plain weave carbon fabric epoxy prepreg [2]. But if the pre-bond moisture produced a reduction of the GIc values for those systems involving a co-bonded carbon fabric prepreg, this was not the case for the material studied here, a carbon unidirectional tape. The GIc values were consistently high and repetitive due to the fact that the crack grew in the lamina with remarkable amount of fibre bridging. The same effect has been found linked to the post-bond moisture. The role of moisture in the enhancement of fibre bridging in fracture toughness Mode I tests has been previously described [3].

Additional parameters were taken into account in this study: the influence of the human factor in the preparation of specimens (three manufacture shops produced the co-bonded panels), of the substrate (four pre-cured parent materials were tested) and of key repair process parameters (the cure cycle, the repair material grade, the effect of autoclave positive pressure versus only vacuum pressure and the extended cure cycle tolerances (+/- 10ºC) were investigated). Results confirmed in all cases the behaviour described above.

For the material combinations investigated in this study the response to pre-bond moisture has been found to be modulated by the reinforcement, the environmental history of the pre-cured adherent and the post-bond environmental aging. This knowledge is essential for the proper definition of the repair process and for the substantiation of the repair.

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