International Journal of Adhesion & Adhesives 49 (2014) 80–89

Contents lists available at ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

Influence of pre-bond moisture in the adherents on the fracture toughness of bonded joints for composite repairs

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ARTICLE INFO

Article history:
Accepted 9 October 2013
Available online 17 December 2013

Keywords:
Composite repairs
Adhesive joints
Moisture
Fracture toughness

ABSTRACT

The mechanical performance of composite components repaired with bonded patches depends on the moisture content of the adherents. There is a need to define drying procedures which guarantee the quality of the bonded joint. An experimental investigation has been carried out on the effect of pre-bond moisture on the fracture toughness (G_{IC}) and failure behavior of repair joints bonded with adhesive films. Substrates were conditioned by immersion in distilled water at 70 °C for 336 h and subsequently dried for 1 h or 24 h to achieve different moisture contents before bonding. Specimens of bonded joints were tested under mode I loading at room temperature. The fracture toughness (G_{IC}) decreased with increasing pre-bond moisture level in the substrates. Fractographic inspection revealed an enhancement of the cohesive mode of failure as the pre-bond moisture decreased. Extending the duration of the drying operation of the substrate with pre-bond moisture, caused an improvement in the fracture toughness of the joint, although not a full recovery of the reference values obtained in absence of pre-bond moisture.
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1. Introduction

Composite structures in service experience environmental and mechanical threats. The main environmental threats are related to the effect of temperature and moisture absorption which can affect the strength of composite structures and reduce their lifetime. Therefore, moisture absorption should be taken into account during the design of a structure. In composite bonded joints, as in those used for repairs, the amount of moisture uptake in the adherents, which might have an influence on the final performance of the joint, depends on a number of factors, e.g. specific adherent material used, exposure conditions (temperature, humidity), exposure time and adherent thickness [1].

In spite of the fact that structural design procedures take into account the detrimental effect of moisture on the mechanical properties of CFRP (carbon fiber reinforced polymers), its effect on repairs to damaged structures is unclear. In previously published works, it has been claimed that the presence of absorbed moisture in the adherent may be regarded as essentially the same as pre-bond moisture absorbed by the fresh adhesive, since it will diffuse from the laminate into the adhesive during the heat and cure cycle [2]. Few studies have reported on the pre-bond moisture effect on the mechanical properties of the adhesive joints, although most of them found that the presence of moisture in the composite lead to the reduction in joint strength [2–6]. Sage and Parker [4,7], attributed the decrease in G_{IC} to voiding, plasticization of the adhesive and reduction in interfacial adhesion. Drying the composite substrates and curing the bonded joints were found to prevent the occurrence of voids [3] but, for higher moisture content, drying at high temperatures induced blistering [8]. However, a low level of moisture in the joints (below 0.5%) had little effect on the strength of adhesively bonded repairs, so it was concluded that complete drying is not essential to attain the desirable fracture toughness [9]. Some unexpected results for certain adhesive joints have been published, claiming that as the moisture level increased the bond strength, to some extent, increased [3,6,10–13]. Matrix ductility [12–14], phase separation of the toughening rubber phase [3], or increase in fiber bridging [15], are the possible reasons for this increase in G_{IC}. For certain adhesives, there is no evidence of significant changes occurring when increasing pre-bond moisture content up to a certain level, above which G_{IC} decreased [5,9,10,13,16,17]. There is no single theory or model with sufficient experimental support to explain the generalized relation between pre-bond moisture and the mechanical properties of joints.

Many authors [4–6] reported that the mechanisms of failure alter as the level of pre-bond moisture changes. Robson et al. [9] did not find any change in failure mode with increasing pre-bond moisture, while Parker [3] observed a different failure mode in an adhesive. Inspection of the fractured surface revealed that the dry

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0143-7496/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.ijadhadh.2013.12.007

joints failed cohesively in the adhesive layer and that the crack path moved towards the interphase after conditioning [6,18]. This suggests that the joints between adherents with low levels of moisture can lead to a cohesive failure of the adhesive, but the mode of failure contribution can vary depending on the adhesive used. On the other hand, fiber–matrix interface failure leads to extensive fiber bridging, which increases the energy needed to grow the crack, thus enhancing the apparent fracture toughness [19]. The level of moisture content in the joints has an effect on the fracture toughness as well as on the failure mode. A proper selection of the adhesive material and drying procedures to assure a low level of moisture would positively influence the performance of adhesive joints.

The focus of this research is to characterize the effect of pre-bond moisture on the Mode-I interlaminar fracture behavior of composite repairs by using Double Cantilever Beam specimens. To attain the pre-bond moisture, the adherents were immersed in water at 70 °C for 336 h, as it was known that, for the material considered in this study, this procedure gives a quick moisture uptake with final moisture content close to equilibrium. Then, two drying processes of 1 h and 24 h were used before curing the bonded joints. Scanning Electron Microscopy (SEM) and Optical Microscopy studies were performed to understand the failure mechanisms.

2. Experimental

In order to determine the effect of pre-bond moisture on the fracture behavior of bonded composites joints, an experimental study was carried out with two different adhesive films, denoted as F1 and F2. One of the adherents (a $[0, 90]_n$ plain weave carbon fabric epoxy prepreg) was previously cured in an autoclave at 180 °C and 700 kPa of applied pressure. The CFRP substrates were immersed in distilled water at a constant temperature of 70 °C for 336 ± 12 h to promote moisture uptake. After that, the substrates were dried in an air circulated oven at a constant temperature of 80 °C, for 1 h and for 24h. The drying for 24 ± 0.5 h at 80 °C was known to reduce the moisture content in this material notably, although not down to zero or near zero moisture. Reducing the moisture content to near zero would take a much longer time, unacceptable for in-service repair conditions. The weight measurements of two adherent material coupons were used to calculate the moisture uptake and loss (%, weight) during the substrate conditioning steps prior to the bonding operation. The coupons were periodically removed from the chamber, wiped with tissue paper and then weighed. Weighing was performed on an analytical balance with an accuracy of 0.1 mg.

Surface preparation of the tool side of the pre-cured substrates was done immediately after drying and consisted of sanding and cleaning with a solvent, followed by a water break test and then, a brief drying using a hot air gun for at least 1 min at an air temperature of 70 °C maximum. The bonded joints with the pre-cured panel, the adhesive films (F1 and F2) and the fresh repair prepreg plies (plain weave carbon fabric epoxy prepreg) were cured at 120 °C under vacuum pressure simply to reproduce those "in-field" conditions normally used in repair procedures. Curing process time and temperature were selected according to the manufacturer's indications. A Teflon insert was placed between the adhesive film and the precured adherent so that an initial pre-crack of 60 mm was obtained. The dimensions of the panels were 350×300 mm^2. The specimens for Double Cantilever Beam fracture toughness tests, according to ISO15024 [20], were 25 mm in width and 150 mm in total length. The nominal thickness of panels was 3 mm. Pre-cured panels were inspected by c-scan. The bonded joints were also inspected by C-scan to check that no flaws (zones with porosity or non-bonded areas) were present in the bond line.

The adhesive thickness in each panel was measured by means of optical microscopy. The edges of all specimens were abraded with an emery paper. Specimens were wiped with alcohol to remove any dust particle before observation. Fluorescence Optical Microscope (Leica brand, DMR-XA) was used to distinguish the resins in the pre-cured and the co-bonded adherents from the adhesive film (Fig. 1).

All specimens were observed at a $10\times$ magnification using a blue filter (Band pass filter BP 450–490 nm) under a fluorescence light radiation. 20 single optical images were taken for each sample. "Canon Stitch" software was used to obtain a mosaic image by composing all the single images of each specimen. Quartz software was used to measure the adhesive thickness in each mosaic image. The adhesive thickness of the joints measured at the thinnest zones was around 90 μm. Maximum adhesive thickness was found to be around 195 μm.

A universal testing machine, MTS Insight with a 1 kN load cell, at a crosshead rate of 5 mm/min was used for the DCB tests. Tests were performed at room temperature (RT) in a laboratory controlled environment (23 ± 2 °C, 50 ± 5% RH) at the University of Girona (ISO 17025 accredited). Batches of six specimens were tested at each condition. The crack length during propagation was monitored optically at the specimen's edge by means of a long distance microscope Questar QM100. Values of load, displacement, and crack length were measured simultaneously for crack

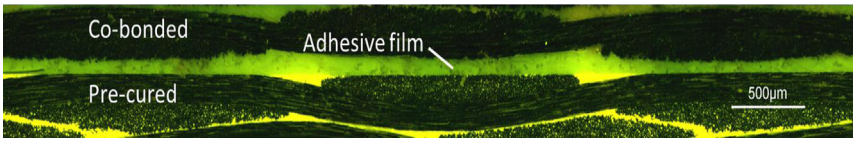

Fig. 1. Fluorescence optical inspection of the edge of the specimen from which the adhesive thickness was measured.

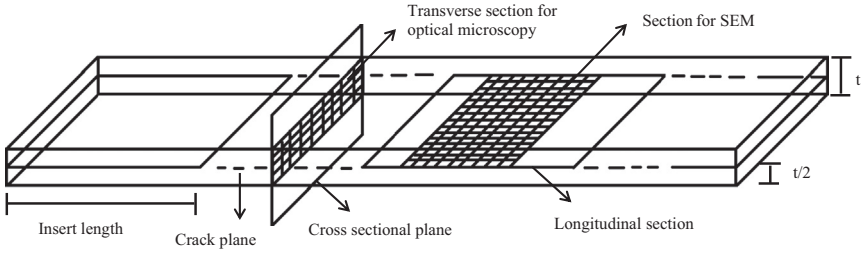

Fig. 2. Configuration of DCB specimen and extraction of samples for microscopy inspections.

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