

Effect of prepreg storage humidity on the mixed-mode fracture toughness of a co-cured composite joint

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ABSTRACT

The present work investigated the effect of the level of prepreg moisture content on the mixed-mode fracture toughness of a co-cured composite joint. It was found that moisture was stored in the prepreg as either free or bound water. It was also shown that the prepreg stores moisture from high humidity environments as free water, while the level of bound water remains unaffected. The excessive moisture was shown to plasticise the adhesive, lowering the glass transition temperature. The fracture toughness decreased under mode I and mode II loading as the humidity level was increased. There was a significant increase in mixed-mode toughness under low humidity conditions. Although the mixed-mode toughness reduced with increasing levels of humidity, the values never fell below that of the joints fabricated using the as-received materials.

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1. Introduction

High humidity environments are among the most hostile experienced by an adhesive joint. The resulting moisture uptake can have a pronounced effect on the behaviour of the adhesive during fracture. Water can be introduced into the joint in a number of ways. It can be present in the composite substrate or prepreg prior to curing or co-curing [1]. If proper precautions are not met, condensation can form on the prepreg or adhesive when taken out of cold storage. Finally, it may enter the joint during in-service conditions [2,3].

The role of moisture in composite joints is only recently being understood. In a series of tests to determine the effect of substrate material and test geometry on the mode I fracture toughness of adhesively bonded joints, Bell and Kinloch [4] found that the double cantilever beam (DCB) joints prepared with the composite substrates exhibited a much lower fracture toughness than those bonded with metallic substrates. These differences were initially believed to be due to the different levels of constraint in the various joint systems. In a later investigation [5], it was revealed that the differences were in fact caused by moisture stored in the composite substrate that was then released during the curing of the adhesive. These results were also published as part of a European Structural Integrity Society – Technical Committee 4 (ESIS-TC4) round-robin in the standardisation of adhesive fracture testing [6]. The same authors then undertook a more extensive investiga-

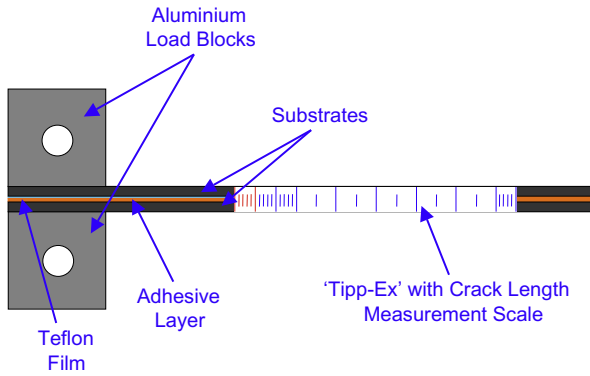
tion of the effect of pre-bond moisture in secondary bonded composite joints [1].

Once moisture has entered the joint, the effects can be quite complex and difficult to analyse. Small amounts of moisture, less than 1%, can drastically reduce the fracture toughness of some adhesive joint systems [1,5] while other systems can actually experience a slight increase in fracture toughness [2] that some attribute to extra energy being absorbed due to plasticisation of the adhesive [7]. It has also been shown that certain adhesives are relatively unaffected by small amounts of moisture, particularly film adhesives [1,8,9].

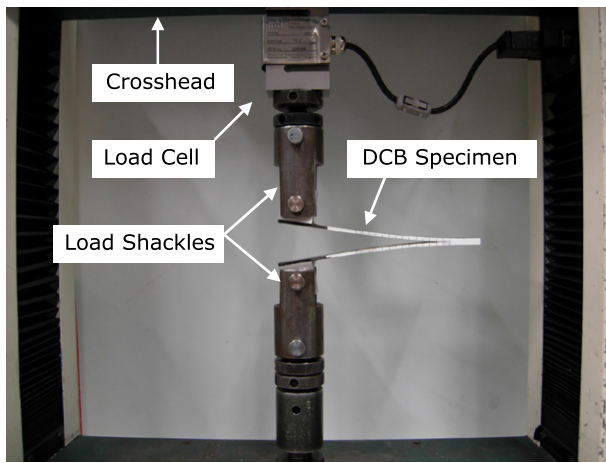
A considerable amount of research has been published on the nature of water in epoxies that are used as neat resins, adhesives and in polymer composites. Zhou and Lucas [10,11] showed that water molecules bind with epoxy resins through hydrogen bonding. The authors further classified the absorbed water into two types; type I 'free' water and type II 'bound' water. The difference lies in the complexity of the hydrogen bonds. Type I bound water only forms a single hydrogen bond with the epoxy resin. Type I free water disrupts the initial interchain Van der Waals forces and hydrogen bonds resulting in increased chain segment mobility. On the other hand, type II bound water forms multiple hydrogen bonds with the polymer network. Therefore, type I free water acts as a plasticizer and decreases the glass transition temperature, T_g , [11] while type II bound water acts as a secondary crosslinked network and can increase T_g [11,12]. Berry et al. [12] reported a similar effect of absorbed moisture in a rubber modified epoxy. In that work, small amounts of moisture were found to increase the glass transition temperature due to the formation of strong dipole–dipole interactions. However, further levels of absorption decreased

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(a) DCB illustration.



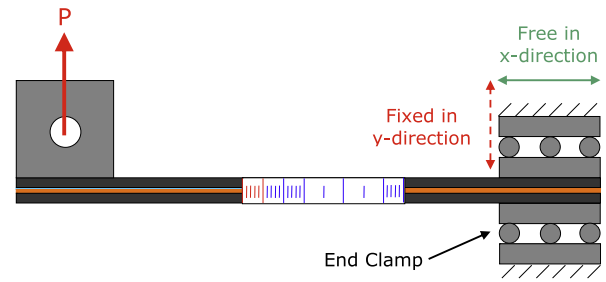
(b) DCB photo.

Fig. 1. Illustrations and photographs of DCB experimental setups showing loading directions and fixtures used during testing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

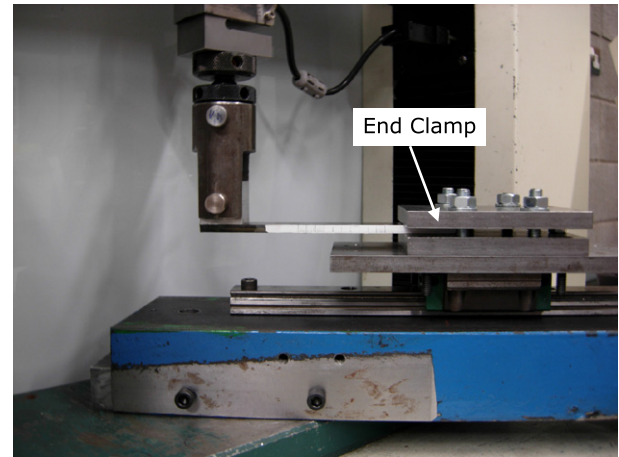
T_g . Others have estimated the amount of residual bound water that remains in epoxy resins after drying, having previously been stored in a hot/wet environment, as being approximately 0.12% [13]. In order to remove this remaining water, an activation energy of the order of 6 kcal/mol is required. The authors noted that this is very close to the activation energy of a hydrogen bond.

Referring back to the work of Zhou and Lucas [10], the authors noted that the water from hot/wet environments was typically absorbed by the epoxy as free water. The level of type II bound water only increased significantly when the samples were stored for very long times at elevated temperatures in the hot/wet environments. The apparent reluctance of the water to be stored as bound water within the polymer network may be due to the higher activation energy needed to form the required hydrogen bonds. It is possible that the bound water was introduced into the epoxy resin during the manufacturing process. Polymer matrices used in the manufacture of composite materials consist of epoxy resins, thermoplastic tougheners and curing agents. These constituent components are typically mixed at elevated temperatures. The high temperature could provide enough energy to form multiple hydrogen bonds between the polymer network and any water present. While free water can always be removed by drying the polymer, in order to prevent the absorption of bound water, every stage of the manufacturing process would have to be strictly controlled.

As mentioned previously, type I free water acts to plasticise the adhesive. However, this absorbed moisture can also displace the molecular chains and swell the polymer, leading to chemical com-

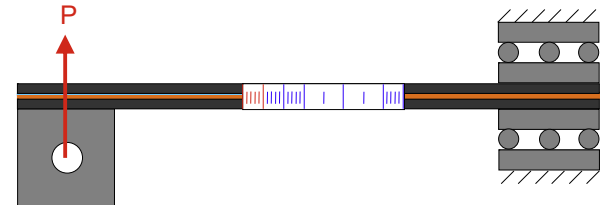


(a) FRMM illustration.

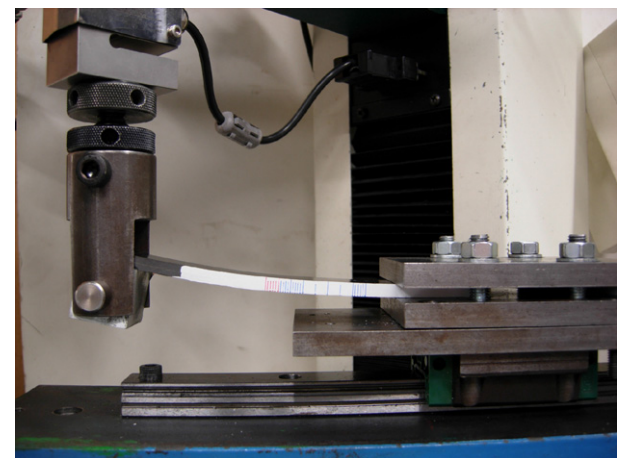


(b) FRMM photo.

Fig. 2. Illustrations and photographs of FRMM experimental setups showing loading directions and fixtures used during testing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



(a) ELS illustration.



(b) ELS photo.

Fig. 3. Illustrations and photographs of ELS experimental setups showing loading directions and fixtures used during testing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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