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Evolution of mechanical properties of flexible epoxy adhesives under cyclic loading and its effects on composite hybrid bolted/bonded joint design

Gyu-Hyeong Lim, Kobye Bodjona, Karthik Prasanna Raju, Sean Fielding, Valentin Romanov, Larry Lessard*

Composite and Structures Laboratory, Department of Mechanical Engineering, McGill University, Macdonald Room 270, 817 Sherbrooke Street West, Montreal, Quebec H3A 0C3, Canada

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A B S T R A C T

Though aircraft joints conventionally employ either mechanical fastening or adhesive bonding, the hybrid bolted/bonded joint has recently appeared as an alternative with possible superior performance. A key aspect of the hybrid joint design is the load-sharing between the bolts and the bond, and performance can benefit from using a flexible adhesive. While the static properties of flexible epoxy adhesives are covered in the literature, the properties under cyclic loading are not fully understood. This study investigates the mechanical properties of flexible epoxy adhesives under cyclic loading and the corresponding effect on composite hybrid joints. This study is twofold. First, cyclic tensile tests on the bulk adhesive investigate the evolution of the adhesive stress/strain. The results show that the modulus and yield stress progressively decrease due to the accumulation of plastic deformation. As the load cycles continue, the stress/strain response converges limiting this accumulation. Subsequently, cyclic tension-tension tests are performed on hybrid joints. Attributed to the aforementioned behaviour of the adhesive properties, the bolt load-sharing is observed to progressively increase until a convergence is reached. This paper provides the understanding on the evolution of the mechanical properties of flexible adhesives under cyclic loading and further confirms their potential in hybrid joint applications.

1. Introduction

For the past decades, two major techniques of joining composite structural components have been conventionally employed in the aerospace industry: mechanical fastening [1–4] and adhesive bonding [3,5–7]. These techniques have their advantages and disadvantages, which led to the idea of combining the two joining mechanisms: hybrid bolted/bonded joints, employing both bonding and fastening in a single joint. This combined joint type, referred to as a "hybrid joint" further in the text, has revealed its potential to be a superior joint type over either bonding or fastening alone. Hybrid joints showed an improvement in the fatigue life [8,9] over that of fastened and bonded joints. Bolts in hybrid joints were shown to suppress the crack propagation in a joint once the adhesive failed [10]. However, only a marginal strength improvement was reported in hybrid joints with the strong and stiff adhesives that are conventionally used for bonded joints.

Interestingly enough, the key to joint strength improvement is to use a less stiff adhesive. Studying hybrid joints with adhesives of different moduli, Kweon et al. [11] reports that joint strength with a stiff adhesive is mainly controlled by the corresponding bonded joint's strength, whereas joints with the softer adhesive attained strengths significantly higher than that of the bonded joints. It was concluded that the design of hybrid joints becomes effective if the mechanical fastening is stronger than the adhesive bonding. Evidently, an efficient hybrid joint design can be attained only by proper load sharing between the bond and the bolt. This is in agreement with the experimental study by Kelly [12] where the bolt loads were measured while testing hybrid joints with stiff and flexible adhesives. Unlike joints with the stiff adhesive, where only a negligibly small load through bolts was observed, the joints with the flexible polyurethane adhesive showed the bolt load sharing value (= bolt load/total load) as high as 42%. Later experimental studies by Bodjona et al. [13] showed that bolt load transfer could be up to 38% higher for the hybrid joints with a flexible epoxy adhesive.

The enhanced load sharing is responsible for improved hybrid joint performance. Studying joints with a flexible polyurethane adhesive, Kelly [7] reported hybrid joints' strength to be 25% and 33% greater than that of bonded and bolted joints, respectively. In addition, a fatigue resistance improvement was achieved by using the flexible adhesive, while only negligibly affected in case of the stiff adhesive.

* Corresponding author.
E-mail address: Larry.Lessard@mcgill.ca (L. Lessard).

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Follow-up quasi-static tests by Bodjona and Lessard [14] revealed up to 12% higher ultimate failure strength of hybrid joints with flexible epoxy adhesive compared to those with stiff adhesive. As the first failure in hybrid joints normally occurs in the adhesive, the improved strength might be attributed not only to the improved bolt load transfer, but also to the capacity of the flexible adhesive to reduce adhesive peak stresses [15–17]. Another feature of the flexible adhesive is the distinct yielding behaviour [18,19], which facilitates the bolt load sharing in hybrid joints. Employing a bilinear elastic/plastic material model, numerical sensitivity tests [20] identified the adhesive yield strength to be the most relevant parameter for effective load sharing in hybrid joints. While yielding benefits the load sharing, it also causes permanent deformation in the adhesive [18]. Hence, under the subsequent loadings, the adhesive behaviour is expected to be different from the initial response.

In the aerospace industry, structural joints are not designed for one-time load application, but rather are expected to be loaded numerous times during their operation lives. A number of the authors discuss the evolution of epoxy polymer properties under cyclic loading [21,22]. In these studies, the accumulation of plastic deformation was observed, even though the stress/strain response eventually converged. It was confirmed to have insignificantly detrimental effect on the fatigue life of the epoxy polymers [22]. However, the aforementioned studies investigated stiff epoxy polymers having a limited tested strain range compared to the one achievable by flexible adhesives. To date, the evolution of the flexible adhesive properties remains unclear as studies on this subject virtually do not exist in literature.

The goal of present experimental study is to provide a better qualitative understanding on the changes in the properties of flexible adhesives under cyclic loads. The study is twofold. First, the flexible epoxy adhesive in bulk form is tested in order to qualitatively investigate the evolution of its mechanical properties under cyclic loads. The second part of the study is to examine the effect of the evolving adhesive properties on the *in-situ* response of hybrid joints.

2. Methodology

2.1. Material selection

The selected representative epoxy adhesive is EA9361 (commercially supplied by Henkel), which is specifically designed for aerospace applications. Its elastic modulus is 723 MPa at room temperature of 25 °C [23], which is two orders of magnitude lower than the modulus of typical metallic or carbon-fiber composite substrates. The adhesive's viscosity is 100 Pa·s. EA9361 is a two-part adhesive, with a mix ratio of 1A:1.4B by weight. For the hybrid joints tests, Cycom 5320 carbon fiber reinforced polymer (CFRP) prepreg tape is used to manufacture the adherends. The properties of Cycom 5320 tape are tabulated in Table 1.

2.2. Bulk adhesive test

2.2.1. Sample preparation

The tensile test for the bulk adhesive employs specimen type I of the standard ASTM D638 [24]. The methodology to produce specimens with EA9361 is adopted from Lim et al. [18]. A Thinky Mixer ARE-310 operating at 2.200 rpm mixes the two parts of the adhesive. The mixed adhesive is then compression moulded by a hot press: the adhesive is cured at 82 °C for an hour as recommended by the adhesive's data sheet [23]. During the curing, the press applies a hydrostatic pressure of 2 MPa. The cured plate is then cut and machined into specimen shape. The gage sections of the specimens are painted for the strain measurement by digital image correlation (DIC). The steps of the specimen preparation are illustrated in Fig. 1. Four specimens to be tested are prepared. For each of these four specimens, the manufacturing process achieves an average thickness of 3.30 ± 0.3 mm.

Table 1
Properties of Cycom 5320 tape.

E_{11} [GPa]	$E_{22} = E_{33}$ [GPa]	$G_{12} = G_{13}$ [GPa]	G_{23} [GPa]	$\nu_{12} = \nu_{13}$	ν_{23}	t_p [mm]
141	9.7	5.1	3.4	0.33	0.44	0.25

2.2.2. Preliminary sensitivity test

Viscoelastic material properties can significantly affect the material response under cyclic loading. To better understand the effect of the adhesive's viscoelasticity, sensitivity tests are performed with different values of test speeds prior to testing the adhesive and hybrid joints under cyclical loading. These preliminary tests are performed within a range of head speeds from 0.1 mm/min to 10 mm/min. The head is displaced from 0 mm to 1 mm. It is ensured that no yielding occurs in the tested displacement range, based on the tensile test results from Lim et al. [18]. No strain measurement by DIC is performed; the results of this sensitivity test are expressed by force vs cross-head displacement.

2.2.3. Cyclical loading

The cyclical loading for bulk adhesive tests is applied in the tension-tension regime. The upper limit of the cyclic loading is defined by displacement, and the lower limit is defined by force. The upper displacement limit is selected to be 26 mm, which corresponds to approximately 25% true strain. This value is determined by separate experiments initially performed to tune the final experiments. This also corresponds to half of the ultimate failure strain [18]. The lower limit is defined by force, and its value is set at 10 N. Due to the high viscosity of EA9361, the speed of the specimen's shrinkage is slower than the head speed during the unloading. As the machine's head travels downward during the unloading, it starts to compress the material once the pre-built tensile stress in the material is fully released. Therefore, in order to avoid compressive stress in the specimen and possible subsequent buckling, the lower limit is set at 10 N, which is just above zero force.

The specimen is cyclically loaded until convergence is detected. In order to reduce the viscous effects, the head speed is set to 1 mm/min for both loading and unloading, which is five times slower than the ASTM D638 recommendation. No further reduction is taken in order to complete the testing in a reasonable time span. Once convergence is detected, the specimen is unclamped and left to shrink until it becomes stress-free. Then, the specimen is re-loaded in tension, at the same head speed as in the cyclic part, until failure.

2.2.4. Experimental set-up

The specimens are loaded by an Insight 5kN MTS machine. The MTS machine measures the force with an error level of ± 0.5 N. Simultaneously, the camera, a PointGrey Flea 5MP, takes images of the speckled gage section. The DIC technique is used for the strain measurement. The force is measured at 1 Hz, and the images are captured at 0.2 Hz. All four specimens are tested at room temperature of 20 °C.

2.3. Hybrid joint test

2.3.1. Specimen preparation

In order to prepare the hybrid joints, for which the dimensions are illustrated by Fig. 2a, a composite plate is first produced with Cycom 5320 unidirectional material. The lay-up sequence is [45/0/−45/90] 4s. Then, bonded joints are manufactured with a bonding jig designed by Bodjona [14]. The precisely machined jig and shims enable control of the adherend alignment, overlap length and the adhesive thickness of 0.5 mm. The process starts with sandblasting of the substrate bonding surfaces. EA9361 is applied at the locations of the overlap and the doublers. The bottom substrates, shims and doublers are placed on the jig (Fig. 2b-1). The top shims are placed on the adherend, and solder wires of 0.508 mm (0.02″ inches) diameter are placed on the bottom