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Tensile strength of two-part epoxy paste adhesives: Influence of mixing technique and micro-void formation

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ABSTRACT

Two-part epoxy paste adhesives are frequently used to bond metals and composite materials in many structural applications. After mixing two reactive parts (by weight or volume ratio), adhesive paste is applied to the substrate surfaces and cured at elevated temperatures. Air-entrapment during mixing and/or application process often produces micro-voids in the adhesive bondlines and influences the strength of the bonded joints. In this work, void formation was investigated using two adhesive mixing techniques: (a) dual-cartridge and static-mixer with a dispenser and (b) hand-mix. Flat adhesive sheets were cured by mixing a two-part epoxy adhesive, and bulk specimens with notches were cut using CNC-machining. Using X-ray microtomography scans, the micro-voids were detected and material porosity was evaluated. Furthermore, tensile tests were performed on the specimens and two-dimensional digital image correlation (2D DIC) was employed to analyse the surface strain concentrations near the notches. The fracture surfaces were examined using optical and scanning electron microscopy. The results indicated that mixing technique influences the formation of micro-voids and thus the tensile strength of two-part epoxy paste adhesives.

1. Introduction

Adhesive usage is gaining momentum in structural applications for assembling both primary and secondary structural components. In the aircraft industry [1], the main use of structural adhesives is in bonding internal structural elements (*e.g.* stringers) to skins in fuselage, wing, aileron, *etc.* However, for the optimal performance of adhesive joints, the bonding process must overcome two major issues: (a) reliable adhesive application (*e.g.* mixing process and application of two-part paste adhesives) and (b) defect-free bondlines (*e.g.* kissing bonds and micro-voids). Micro-voids are frequently seen in adhesive bonds cured at elevated temperatures. Though the average size of these micro-voids is small ($<100\ \mu m$), the effective cross-sectional area of the bond reduces significantly with increase in void density and thus influences the structural performance. Void formation is often connected to: (a) air-entrapment during adhesive mixing and/or application, (b) vaporisation of the moisture absorbed by substrate surfaces (or by carrier in film adhesives) prior to bonding, (c) the type of substrate surface preparation, etc. [2–5].

The structural strength of bonded joints depends on the mechanical properties of substrate material, adhesive material and substrate–adhesive interface. To characterise the mechanical properties of adhesives, experimental tests are performed on either bulk specimens or bonded joints (*i.e.* in-situ form). It is often argued that the constraint-effect (bondline being constrained by two substrates) may influence the mechanical properties and thus the properties obtained through bulk material testing may not represent the in-situ properties. However, some research studies [6–9] showed that the mechanical behaviour of the bulk adhesive material is similar to those in bonded joints.

In this paper, a two-part paste adhesive was considered to investigate the influence of micro-voids on the tensile strength. Bulk specimens were manufactured using two different mixing techniques—to achieve two different porosity levels in the bulk material. The key objectives were: (a) to perform tensile tests on double-notched specimens and evaluate the influence of micro-voids on the tensile strength of two-part epoxy paste adhesives, (b) to monitor surface strains near the notches using 2D DIC for failure strains, (c) to evaluate the porosity levels in dispenser-mix and hand-mix specimens using X-ray microtomography and (d) to examine fracture surfaces using optical and scanning electron microscopy and identify failure mechanisms. The study indicated that adhesive micro-voids adversely affect the failure stresses and strains.

2. Materials and manufacturing

2.1. Two-part epoxy adhesive

A two-part epoxy paste adhesive, EA 9380.05 from Henkel Adhesives, was used to manufacture bulk specimens by curing thin

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sheets. It is a toughened structural adhesive with glass particles to improve fracture properties. This adhesive was supplied in both dual cartridge and tin forms. For the current study, dispenser-mix and hand-mix techniques were employed to manufacture bulk adhesive sheets. The process used for dispenser-mix is shown in Fig. 1. The dispenser was loaded with a dual cartridge and a static mixer, and compressed air was pumped into the system to get a uniform adhesive paste from the nozzle of the static mixer. For hand-mix adhesive, the two parts were weighed and mixed in a bowl using a rod/spatula until a uniform colour is obtained.

2.2. Adhesive application and curing

To manufacture bulk adhesive sheets two PTFE sheets were used to sandwich the adhesive paste, and then two aluminium plates were used to apply pressure on the PTFE-adhesive sandwich system. For the dispenser-mix, the adhesive was applied on to one of the PTFE sheets by beading the paste adhesive from the nozzle. On the other hand, for the hand-mix case, adhesive paste was applied in the middle of the PTFE sheet and then spread using a spatula (by moving the spatula in only one direction). Once the adhesive was applied, the PTFE-adhesive sandwich system was enclosed using the aluminium plates. The thickness of the adhesive sheet was controlled by inserting brass shims (1 mm) on the four edges (located using a high temperature tape) of the PTFE sheets. The adhesive was then cured in a hot drape former (HDF2 from Laminating Technology) for 4 h at 80 °C under 10 kPA pressure.

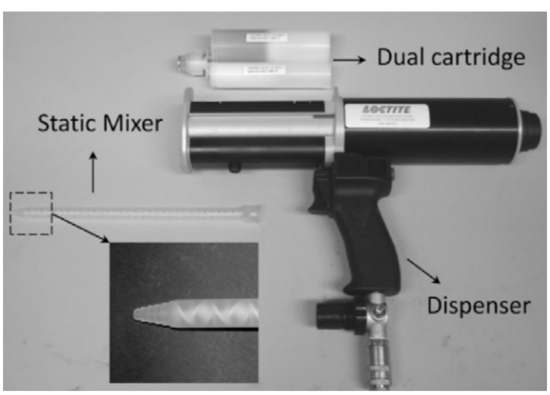

Fig. 1. Dispenser-mix using dual-cartridge and static-mixer.

2.3. Bulk specimens

Bulk specimens were manufactured by cutting the cured adhesive sheets using a CNC machine. The dimensional details (in mm) of the bulk specimens are shown in Fig. 2a. The thickness of the specimens, t, is 1 mm. To induce strain concentration and fracture in the middle of the specimen, a circular notch with 5 mm radius was cut on both edges. The specimen geometry (with two circular notches) is used to monitor strain concentrations at failure using the 2D digital image correlation technique. However, the notch geometry used (which is not a sharp notch) introduces negligible transverse stresses near the notch tip and thus the failure is predominantly governed by the axial stresses. The edge quality obtained from CNC-cutting is shown is Fig. 2b. Two circular holes (4 mm radius) were also drilled at the top and bottom for applying a tensile load using dowel pins to eliminate issues related to specimen alignment. To avoid failure at these pin holes, aluminium shims (1 mm) with holes are bonded with Loctite super glue on both sides.

3. Experimental work

3.1. Test setup

Tensile tests were conducted on bulk specimens in a Tinius-Olsen mechanical test machine with a 1000 N capacity load cell. The specimens were tested at three different crosshead displacement rates. Dowel pins were employed to accurately align the specimens and also to transfer the crosshead displacement. For each combination of mixing technique and displacement rate 3 tests were conducted, and axial load and crosshead displacement data were obtained. However, to accurately monitor the failure behaviour, one instrumented test was performed for each combination of mixing technique and displacement rate using a 2D DIC [10].

3.2. Digital image correlation

To experimentally evaluate the tangential-surface strains (*e.g.* axial strain, ε_{yy}), non-contact techniques such as 2D DIC gained popularity for material testing in recent times [11,12]. In the current study, the 2D DIC system was used to assess the surface strain evolution at the notches with increasing loads. To perform 2D DIC on the bulk specimens: (a) a random surface

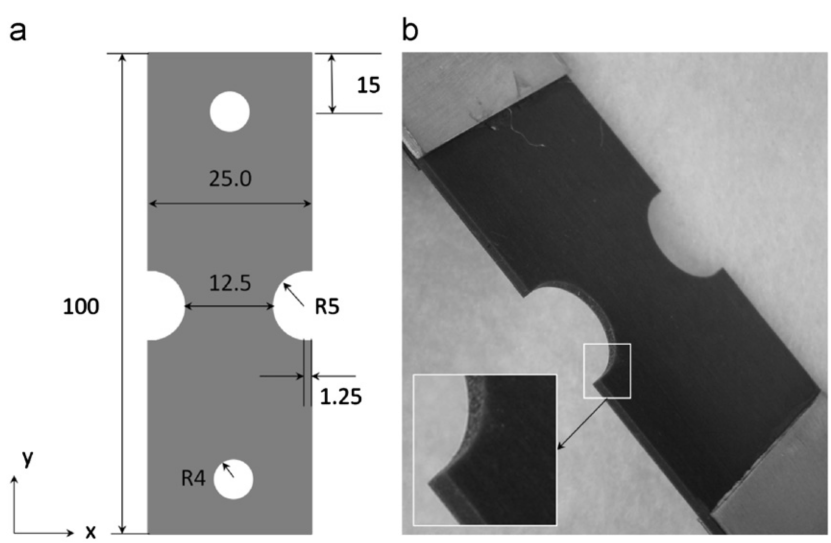

Fig. 2. Bulk specimen: (a) dimensions (in mm) and (b) the edge quality from CNC-cutting.