



| | |
|-------------------------------------|--|
| Title | Characterisation of the fracture energy and toughening mechanisms of a nano-toughened epoxy adhesive |
| Authors(s) | McAuliffe, David, Karac, Aleksandar, Murphy, Neal, et al. |
| Publication date | 2011-09 |
| Publication information | McAuliffe, David, Aleksandar Karac, Neal Murphy, and et al. "Characterisation of the Fracture Energy and Toughening Mechanisms of a Nano-Toughened Epoxy Adhesive." Trans Tech Publications, September 2011. https://doi.org/10.4028/www.scientific.net/KEM.488-489.573 . |
| Publisher | Trans Tech Publications |
| Item record/more information | http://hdl.handle.net/10197/4691 |
| Publisher's version (DOI) | 10.4028/www.scientific.net/KEM.488-489.573 |

Downloaded 2026-05-01 23:40:30

The UCD community has made this article openly available. Please share how this access benefits you. Your story matters! (@ucd_oa)



© Some rights reserved. For more information

Characterisation of the Fracture Energy and Toughening Mechanisms of a Nano-toughened Epoxy Adhesive

D. Mc Auliffe^a, A. Karač, N. Murphy and A. Ivanković

University College Dublin, Dublin, Ireland

^adavid.mcauliffe@ucd.ie

Keywords: Nano-particles, adhesive joints, toughening mechanisms

Abstract. In this study the adhesive joint fracture behaviour of a nano-toughened epoxy adhesive was investigated. Two experimental test methods were used; (i) the standard tapered double cantilever beam (TDCB) test to measure the mode I adhesive joint fracture energy, GIC, as a function of bond gap thickness and (ii) a circumferentially deep notched tensile test to determine the cohesive strength of the adhesive for a range of constraint levels. It was found that the fracture energy of the adhesive followed the well-known bond gap thickness dependency [1]. SEM analysis of the TDCB fracture surfaces revealed significant plastic void growth. Finally, numerical modelling of the experimental tests suggested that most of the fracture energy was dissipated via highly localised plasticity in the fracture process zone ahead of the crack tip.

Introduction

Structural epoxy adhesives are commonly used to join materials placed in load bearing applications. Adhesive bonding has many advantages over conventional joining techniques such as the elimination of stress concentrations, improved rigidity and the possibility of joining dissimilar materials. These adhesives are typically based on a thermoset epoxy matrix. An epoxy matrix possesses a relatively high stiffness, but is prone to crack initiation and propagation. This limitation has been overcome by the addition of dispersed second phase of particles, which can provide a significant increase in toughness [2]. The processes responsible for this increase in toughness are still not clear with different mechanisms reported depending on the type, size and concentration of particle added. The aim of this study is to determine the fracture toughening mechanisms that occur during mode I fracture of a core shell rubber (CSR) nano-toughened epoxy adhesive.

Experimental Materials and Procedure

Two experimental test methods were employed to examine the adhesive joint fracture behaviour of a nano-toughened epoxy adhesive. All tests were conducted at low-loading rates and ambient temperature.

Adhesive. The adhesive used in this study is an experimental grade, single part, nano-toughened epoxy adhesive produced by Henkel. It contains two grades of CSR nano-particles of diameters 50 nm and 200 nm, which occupy 16% and 22% of the adhesive volume respectively. A CSR particle consists of a rubber core surrounded by a thin layer of stiff material. All bulk and adhesive joint samples were cured at 180 C for 90 minutes. The basic mechanical properties of the adhesive and other substrates used are shown in table 1.

Table 1. Basic mechanical properties of materials used.

| | E [GPa] | σ_{yield} [Mpa] | UTS [MPa] |
|------------|---------|-------------------------------|-----------|
| Adhesive | 1.76 | 31.4 | 45 |
| Al 2014 | 72.4 | 425 | 475 |
| mild steel | 200 | 165 | - |

TDCB test. The standard Tapered Double Cantilever Beam (TDCB) [3] test was employed to measure the mode I fracture energy G_{IC} , [J/m^2] of the adhesive as a function of adhesive layer thickness. The test configuration, shown in Fig 1(a), consists of two contoured beams separated by an adhesive layer. The profile of the beam is such that the change in compliance with crack length, is linear, described by a geometry constant, m .

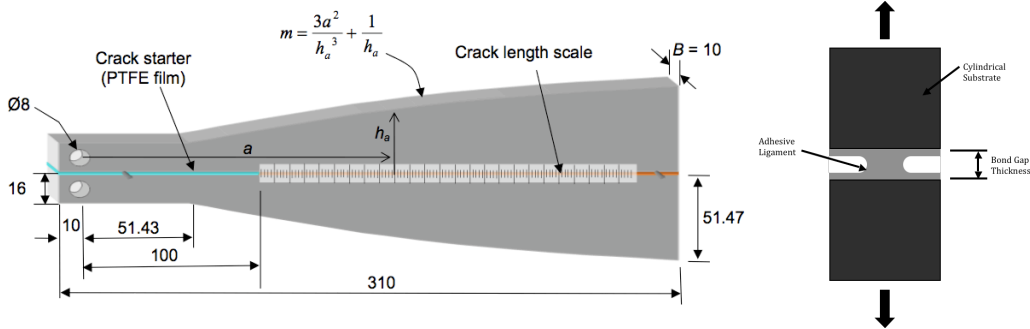


Fig 1. Schematic diagrams of (a) the TDCB geometry and (b) the CDNT geometry.

To prevent plastic deformation of the beams during testing, a high yield strength Aluminium alloy (Al 2014) was chosen as the substrate material. TDCB joints were produced with bond gap thicknesses ranging from 0.2mm to 2.5mm. A groove was machined along both sides of the adhesive layer for specimens with a bond gap thickness greater than 0.4 mm, this was to ensure pure mode I failure. Testing was carried out with a standard tensile testing machine at a constant crosshead displacement rate of 0.5mm/min. During the test, the load, crosshead displacement and crack length, were recorded. From these measurements, G_{IC} was calculated for each bond gap thickness, according to the corrected beam theory solution [3].

CDNT test. A new test method, the circumferentially deep notched tensile (CDNT) test has been developed to measure the cohesive strength of an adhesive in terms of constraint, the ratio of hydrostatic stress to equivalent von mises stress (σ_{Hyd}/σ_{eq}). This is especially useful to experimentally calibrate numerical cohesive zone models. The test involves bonding two cylindrical substrates at a given bond thickness. A notch is then machined circumferentially into the adhesive layer. Appropriate selection of the bond gap thickness, notch geometry, and substrate material produces the desired level of constraint across the remaining adhesive ligament. A typical specimen is shown schematically in Fig. 1 (b). Samples with bond gap thicknesses of 1.0mm and 1.6mm were manufactured with ligament to bulk area ratios of 25% and 50%, thus four specimen geometries each with a notch root radius of 0.3mm. The specimens were loaded in tension and the displacement and load were recorded.

Microscopy. During the fracture process, the CSR particles debond from the epoxy matrix, followed by plastic growth of the resultant void. A microscopy study of the TDCB fracture surfaces was carried out to determine the dilatational void strain, ϵ_f , prior to fracture for each bond gap thickness. Fractured TDCB specimens were cut to provide sections containing the first 10mm of crack growth. These sections were then gold sputter coated and viewed under SEM. Images were taken along the centreline of the specimen in the direction of crack propagation, The radii of the voids remaining from debonding of the larger ~ 200 nm particles were measured for each bond gap thickness. The average fracture strain, ϵ_f was then computed where $\epsilon_f = u_r/r_0$, and u_r is the radial displacement of the void and r_0 is the initial radius of the nano-particle, i.e. 100 nm in this case.

Experimental results

Steady state cohesive crack growth occurred for all TDCB tests. It was found that G_{IC} steadily increased from 3400 J/m^2 at 0.2 mm bond gap thickness up to 5600 J/m^2 at 1.6 mm beyond which G_{IC} reduced slightly as shown in Fig 2. The CDNT tests resulted in cohesive failure across the adhesive ligament for all test geometries. It was found that all specimens failed at a ligament stress of $52 \text{ MPa} \pm 2\%$. From the microscopy study it was also found that the ε_f increased with fracture energy (Fig. 2).

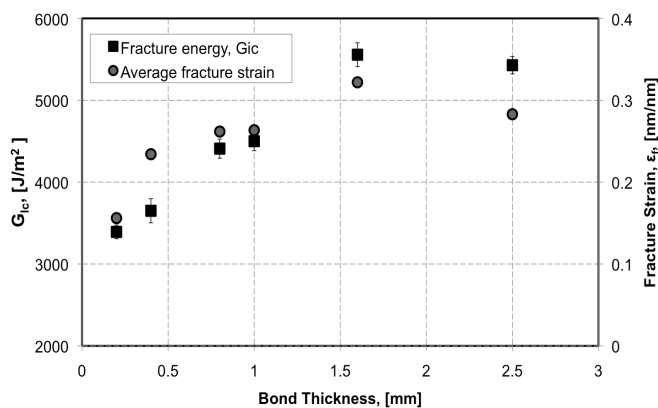


Fig 2. Fracture energy G_{IC} , [J/m^2] and void dilatational strain, ε_f as a function of bond gap thickness.

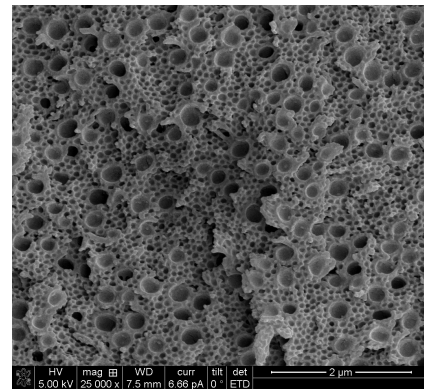


Fig 3. Typical SEM image of TDCB fracture surface with two distinct void size distributions.

Numerical modelling

Numerical simulations of the CDNT and TDCB tests were performed with the OpenFoam Finite Volume software package (version 1.4). A non-linear elastic plastic material model including conventional incremental J2 plasticity governed adhesive behaviour while all substrates were treated as being linear elastic. The fracture process was incorporated via a Dugdale cohesive zone model (CZM) specified by the cohesive strength, σ_{\max} and the separation energy, G_0 . A cohesive strength of 52 MPa , based on the experimental CDNT tests, was selected for all simulations.

Analysis of the CDNT tests illustrated that the average level of constraint across the adhesive ligament prior to fracture was quite insensitive to the value of G_0 specified. Variations in G_0 only affected the extension to failure once the maximum stress was reached. A G_0 value of 2800 J/m^2 was used to obtain reasonable agreement with the experimental data (Fig. 4). The average constraint across the adhesive ligament prior to fracture was between 1.5 and 2.0 for the different test geometries. These values correspond to the range of constraints experienced during TDCB fracture.

Simulations of the TDCB tests were completed for bond thicknesses of 0.2 , 0.4 , 1.6 and 2.5 mm . To reproduce the experimental results it was necessary for G_0 to be between 90% and 95% of the experimentally measured fracture energy. The remaining fracture energy was dissipated through a small diffuse plastic zone ahead of the crack tip at the free surface of the adhesive layer.

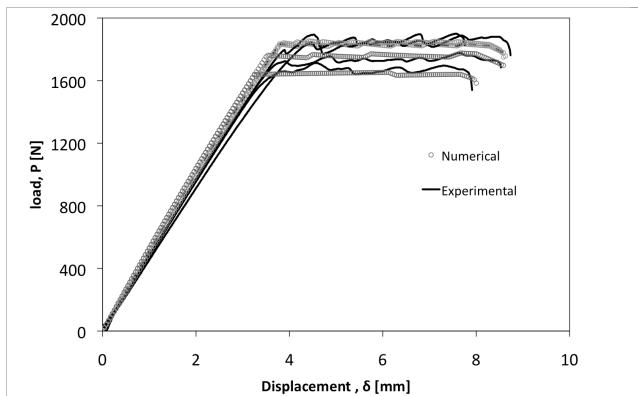


Fig 4. Comparison of experimental and numerical load displacement traces, for TDCB specimens

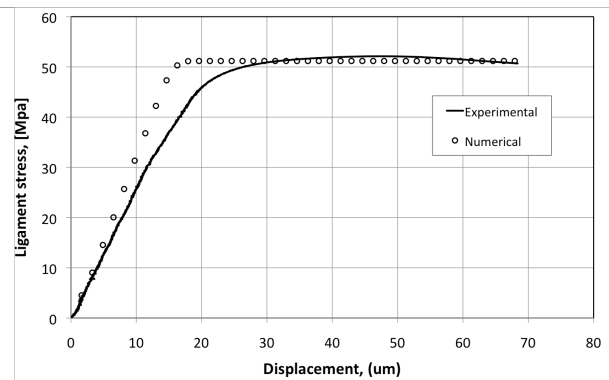


Fig 5. Typical CDNT load displacement trace and numerical simulation

Conclusions and Future Work

Two experimental test methods were used to examine the fracture behaviour of a nano-toughened structural epoxy adhesive. Under mode I testing the adhesive demonstrated the classical bond gap thickness dependency, whereby, the fracture energy increased steadily with bond gap thickness from 3400 J/m^2 to a maximum of 5600 J/m^2 after which the fracture energy reduced slightly. A CDNT test was developed to measure the cohesive strength of the adhesive for a range of constraint levels. It was found for the geometries tested, that the cohesive strength remained constant at 52 MPa. Also, the shape of the force displacement curves suggest that a constant stress damage model is a suitable representation of the traction separation law for this adhesive. A Dugdale cohesive zone model based on the experimental findings was implemented in numerical simulations to reproduce the experimental data. The cohesive strength was 52 MPa for all simulations but the separation energy, G_0 , was varied to reproduce the experimental tests. In the TDCB simulations the effects of the selected cohesive parameters became clear. The defined ratio of σ_{\max} to yield strength of 1.65 prevented a significant diffuse plastic zone from developing before crack advance. Therefore, most of the fracture energy was dissipated in the cohesive zone. This suggests toughening is achieved via intense localised plasticity mechanisms in the fracture process zone ahead of the crack tip. Scanning electron microscopy of the TDCB fracture surfaces revealed that significant void growth occurred, up to 35% dilatational strain. Also, the extent of void growth prior to fracture increased with fracture energy. This indicates that the energy dissipated in the fracture process zone isn't constant but varies with fracture energy. Further work is ongoing to characterise the fracture process zone, in particular to determine the depth and extent of void growth below the fracture surface. Also, the presence of shear bands will be investigated by use of polarised light microscopy. Bulk mechanical tests are being performed on the epoxy matrix alone to determine its deformation behaviour for a range of constraint levels. Furthermore, a partner research project is examining energy dissipation mechanisms by micromechanical modelling of the fracture process zone. This experimental and numerical description of the process zone should allow the energy dissipated in the FPZ and the associated mechanisms to be fully quantified.

Acknowledgements

The authors wish to acknowledge the support provided by the Irish Research Council for Science Engineering and Technology (IRCSET) and Henkel Ireland. The assistance provided by the surface engineering group in UCD is also greatly appreciated.

References

- [1] Hunston, D. L., Kinloch, A. J. and Wang, S. S. (1989) 'Micromechanics of Fracture in Structural Adhesive Bonds', *The Journal of Adhesion*, 28:2, 103 – 114
- [2] Sultan, J. N., McGarry, F. (1973) 'Effect of rubber particle size on deformation mechanisms in glassy epoxy', *Polymer Engineering and Science*, 13:1, 29-34
- [3] British Standard Institution. BS 7991 Determination of the mode I adhesive fracture energy, GIC, of structural adhesives using the double cantilever beam (DCB) and tapered double cantilever beam (TDCB) specimens, 2001.