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The fracture behaviour of nanostructure added adhesives under ambient temperature and thermal cyclic conditions

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ABSTRACT

In this study, the fracture behavior of nanocomposite adhesives produced by adding nanostructure in to the adhesives were investigated using Double Cantilever Beam (DCB) test under ambient temperature and thermal cycle conditions. Adhesively bonded DCB joints were produced using DP460 toughened adhesive type and DP125 flexible adhesive type as the adhesives; AA2024-T3 aluminum alloy was used as the adherend, and 1 wt.% Graphene-COOH, Carbon Nanotube-COOH and Fullerene C60 were used as the added nanostructures. As a result, when the experimental fracture energy was examined, the nanocomposite adhesives obtained by adding nanostructure were found to have increased the fracture energy of the joint. In the joints bonded with the flexible DP125 adhesive that were subjected to thermal cycles, the addition of Graphene-COOH into the adhesive increases the fracture energy of the joint by 55%, the addition of Fullerene increases the fracture energy of the joint by 135%. Also, it was observed that there is a significant difference between the displacements that were obtained directly from the test machines stroke and measured via video extensometer giving the crack opening between the top and bottom adherends during the DCB test. This situation significantly effects the correct calculation of the fracture energy of adhesive.

1. Introduction

In recent years, adhesively bonded joints are frequently used the aviation and automotive industry. Therefore, published literature shows numerous studies that have been conducted to increase the load-carrying capacities of adhesively bonded joints. The majority of these works have been aimed at increasing the load-carrying capacity of the joint by changing the joint geometry. However, studies carried out in recent years have been based on increasing the load-carrying capacity of the joint by adding nanostructures into an adhesive [1–13].

It is important to do experimental and numerical analyses of adhesively bonded joints, and look at the compatibility of the experimental and numerical data. In published literature, there are several methods used in the numerical analysis of adhesively bonded joints. However, the Cohesive Zone Model (CZM)—which is considered the most suitable—has been widely used for numerical analysis of adhesively bonded joints. In published literature, there are many studies conducting numerical analyses using CZM [14–19]. The CZM parameters of an adhesive must be determined to perform numerical analyses of the CZM. There are several different methods for determining CZM parameters (Mode I-Double Cantilever Beam, Mode-II End Notch Flexure and Mixed Mode-Four Point Bend). One of the methods to determine CZM parameters is the Mode I-Double Cantilever Beam (DCB) test. In published literature, there are few studies that measure the fracture energy, as well as the displacement (opening between the lower and upper adherend) values corresponding to the maximum stress and failure of the adhesive, by conducting DCB tests of the adhesively bonded joint [20–27]. Some of them are summarized below.

In a study by Khoramishad et al. [28], the influence of graphene oxide nano-platelets on fracture energy was investigated experimentally and numerically in adhesively bonded joints. According to the result of this study, adding 3 wt.% graphene oxide nano-platelets into the adhesive increases the fracture energy of the link by about 69%.

In a study done by Kim et al. [29], an adhesive was reinforced with fiber, and its DCB test was conducted. A substantial increase was observed in the fracture energy of the fiber reinforced adhesive, and the amount of the increase varied with respect to the amount of fiber used

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Nomenclature

DCB	Double Cantilever Beam
G_{IC}	the tensile critical strain energy release rate
CBT	Corrected Beam Theory
STM	Standard Test Method (for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joints)
P	applied load, N
δ	displacement at the point the load was applied, in mm
B	specimen width, in mm
a	crack length (from the point where the load was applied), in mm
Δ	crack length correction for crack tip rotation and deflection (Δ is a function of C and a, and can be found experimentally)
C	compliance of the specimen, δ/P
P_{max}	load to start crack, N
E	tensile modulus of adherend, MPa
B	specimen width, mm
h	thickness of adherend, mm
K_n	normal cohesive stiffness $\frac{T_n^{max}}{\delta_n^*}$
T_n^{max}	maximum normal cohesive traction σ_{max}
δ_n^*	normal displacement jump at maximum normal cohesive tractioncohesive traction
δ_n^c	normal displacement jump at the completion of debonding
δ_n^{max}	maximum normal displacement jump attained in deformation history
D_n	damage parameter associated with Mode I dominated bilinear cohesive law.

in the adhesive.

There are several theories used to calculate the fracture energy of an adhesive by using the DCB test. Mohammadreza et al. [30] used the Modified Beam Theory (MBT) and Compliance Calibration Method (CCM) to calculate the fracture energy of the adhesive, comparing the results with 2D and 3D numerical analyses. According to the experimental and numerical analysis results, it was found that 3D analysis was more consistent.

Lopes et al. [31] conducted DCB tests for three different adhesives, and the fracture energies of each adhesive were obtained by using three different energy methods: the Compliance Calibration Method, the Corrected Beam Theory and the Compliance-Based Beam Method. Based on the results of the study, the DCB test was deemed appropriate for soft and hard adhesives, while the Tapered Double-Cantilever Beam (TDCB) test was more appropriate for very hard adhesives. Furthermore, Blackman et al. [32] investigated the fracture behavior of the structure of adhesive joints using the DCB test specimens at test rates of 1 m/s up to 15 m/s. DCB test rates changed the fracture energy of the adhesive.

Adhesively bonded joints are exposed to the thermal cycle like environment, warm and cold, it is crucial to investigate the thermal cycle performances. In this study, the fracture behavior of nanocomposite adhesives produced by adding nanostructure in to the adhesives were investigated using Double Cantilever Beam (DCB) test under ambient temperature and thermal cycle conditions. Adhesively bonded DCB joints were produced using DP460 toughened adhesive type (strain rate about 4.7%) and DP125 flexible adhesive type (strain rate about 78.5%) as the adhesives; AA2024-T3 aluminum alloy was used as the adherend, and 1 wt.% Graphene-COOH, Carbon Nanotube-COOH and Fullerene C60 were used as the added nanostructures. In the experiments while the crack growth is measured by using a high-speed video (HSV) camera, the displacements were measured by extensometer. The total thermal cycling operation comprises five cycles; for one cycle, the sample is held at 21 °C for 10 min/40 °C for 30 min/21 °C for 10 min and −50 °C for 30 min. The ambient temperature is set at 21 °C.

2. Experimental work

2.1. Materials

AA2024-T3 aluminum alloy—commonly used in aerospace, transportation and general engineering industries due to its superior mechanical and physical properties—was used in this study as an adherend.

For adhesion, two-part epoxy adhesives (produced by 3M Company, St. Paul, Minnesota, U.S.A.) DP460 toughened adhesive and DP125 flexible adhesive were used. For the nanostructure, 1 wt.% Graphene-COOH (thickness: 5–7 nm; diameter: 5 µm; surface area: 120–150 m^2/g), 1 wt.% Carbon Nanotube-COOH (diameter: 10–20 nm; length: 10–30 mm; purity: 95%; surface area: 200 m^2/g; 2 wt.% COOH content) and 1 wt.% Fullerene C60 (purity: 99%) were used. The material properties of the adhesives used in the experimental studies are shown in Table 1 [7].

Structural adhesives are subject to curing depending on the temperature and time. Curing conditions and composition rates of the adhesives are given in Table 2.

2.2. Fabrication of the Double Cantilever Beam (DCB) joints

The adherend material used in the *Double Cantilever Beam (DCB)* was AA2024-T3 aluminum alloy, and the DCB joint samples' geometry and dimensions are shown in Fig. 1. DCB joint samples' geometry and dimensions are currently standardized [33,34]. In this study, the crack length is 55 mm (the part from where the load is applied to the beginning of the adhesive) and the pre-crack is not found.

The most critical part in preparing adhesives reinforced with nanostructures is to homogeneously distribute the carbon nanostructures in the adhesive to prevent flocculation between nanostructures. In a study performed by Gültekin et al. [4], the standard deviation was decreased to 1–2% in the joints produced by using a new method. This new method was developed together with colleagues at the department of chemistry education. Considering this new method, both non-reinforced and nanostructure reinforced adhesives was prepared. A full discussion can be found elsewhere [7].

For the adhesively bonded joints to display their high performance, surface machining methods were applied to the adhesive (AA2024-T3 aluminum alloy) before bonding. To clean burrs and remove oil, grease and dirt resulting from cutting the specimens into the desired dimensions, specimens were first ground with 600 SiC sandpaper followed by 1000 SiC sandpaper to obtain a smooth surface. After grinding, specimens were washed under flowing water and kept in acetone for twenty minutes.

The mold shown in Fig. 2 was used to produce the DCB joint sample with precision. The adhesive thickness in all joint types is 0.16 mm. In order to obtain an adhesive layer thickness of 0.16 mm after curing,

Table 1
Material properties of the adhesives [7].

	DP 460	DP 125
E (MPa)	$1984^{\pm 43}$	$25.1^{\pm 2}$
ν	0.37	0.35
σ_t (MPa)	$38.4^{\pm 1.1}$	$12.7^{\pm 0.4}$
ε_t (%)	4.7	78.5

E: Young's modulus; ν: Poisson's ratio; σ_t: Ultimate tensile strength; ε_t: Ultimate tensile strain.