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International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

Mechanical bulk properties and fracture toughness of composite-to-composite joints of an elastomer-toughened ethyl cyanoacrylate adhesive

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ARTICLE INFO

Article history:

Accepted 27 February 2016

Available online 4 March 2016

Keywords:

Cyanoacrylate

Toughened adhesive

Composites

Fracture toughness

Mechanical properties

ABSTRACT

The bulk mechanical and fracture characterisation of an elastomer-toughened ethyl cyanoacrylate adhesive were investigated using thin film and standard double cantilever beam specimens, respectively. It is shown that these properties are strongly dependent on the prevailing curing conditions. These in turn are influenced by factors such as surface chemistry, curing time, the use of initiators, and the concentration of stabilizing acids in the system which have a profound effect on the polymerisation reaction. The curing conditions in turn have been shown to greatly influence the phase separation of the elastomeric toughening agent and the effectiveness of its associated toughening mechanism.

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1. Introduction

The first patent dealing with the preparation of alkyl cyanoacrylate monomers was lodged by Ardis [1] in 1949. Their performance as a one-part adhesive, which cured at room temperature without the addition of an initiator or curing agents was recognised by Coover and Shearer [2] eight years later. Today, cyanoacrylate (CA) adhesives are used to bond a wide variety of substrates, including metals, ceramics, plastics, rubber, wood, and biological tissue [3], in addition to applications such as the fabrication of hollow submicron and microspheres for drug delivery as described by Makuta et al [4]. The polymerisation reaction of CAs is initiated by free radicals or nucleophiles, i.e., neutral bases or ionic initiators such as the water molecule, and is stabilized through the addition of a strong acid and free radical inhibitors as described in [5] by Coover et al. The moisture present on the bonding surface is sufficient to initiate curing within a few seconds. When cured, CA adhesives are hard, clear solids with good tensile shear strengths but are brittle with poor peel properties, low thermal resistance, and low gap filling ability. To improve the mechanical properties, rubber toughening agents or other improving fillers are often used. CA adhesive compositions containing toughening agents such as acrylic rubbers, polyester polymer, methacrylate-butadiene-styrene (MBS), acrylonitrile-butadiene-styrene (ABS), methacrylate-acrylonitrile-butadiene-

styrene (MABS) core-shell copolymers, poly (vinyl chloride-co-vinyl acetate) (PVCVA), and ethylene-methyl acrylate copolymers (VAMAC) have demonstrated improved impact toughness, peel and tensile shear strength properties and improved thermal resistance of the adhesive bonds. These developments have been described in a series of patents over the past three decades, including [6–11]. In [12], Leblanc discusses the reinforcement of elastomeric networks by fillers such as carbon black which typically results in an increase in modulus, hardness, tensile strength, and resistance to fatigue and cracking of the elastomeric network. The average particle size of the filler is the most important parameter in reinforcement. Particles of 10–100 nm have a reinforcement action upon the rubber network while particles larger than 1–100 μm have a degrading effect. Although many authors have investigated the kinetics of phase-separation of other elastomer-toughened adhesives, no such investigation has yet been carried out for toughened CAs. Pepper et al. [13,14] have previously investigated the polymerisation of CAs using initiators. These studies showed that the presence of strong acids in the adhesive caused an inhibition period prior to polymerisation (effectively providing an increased shelf life for the product) while the presence of weak acids caused a retardation of the polymerisation reaction itself.

In this research the mechanical and fracture characterisation of a carbon black filled, elastomer-toughened, ethyl cyanoacrylate (ETECA) adhesive for composite-to-composite joints are investigated. To the authors' knowledge, such a comprehensive investigation has not been previously reported in the literature. The use of composite materials is increasing rapidly in many industries.

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Their inherently high strength to weight ratio, ease of manufacturing and their ability to be reinforced in multiple directions has made them an attractive alternative to traditional structural materials. Although these adhesives are not intended for primary structural applications, their potential use in composite bonding applications under ambient temperature, indoor conditions, remains to be fully developed. In addition, recent investigations exploring the application of cyanoacrylate adhesives in the development of self-healing polymer composites have been promising as shown, for example, by Fifo et al. [15].

2. Materials and methods

2.1. Materials

A carbon black filled, ethylene acrylic elastomer-toughened, ethyl CA adhesive supplied by Henkel Ireland was used in this research. The composition of the adhesive formulation cannot be disclosed for confidentiality reasons. The only differences between the adhesive batches employed in this research, denoted A, B, C, D, E, F, G and H, are the concentrations of weak and strong acids contained in each batch. The initiators, denoted Initiator 1 and Initiator 2 in this paper, are pyridine-based. Table 1 outlines a summary of the differences between the adhesive batches and the variables tested. The substrates employed in the fracture tests were carbon fibre reinforced epoxy laminates, manufactured from Hexcel Hexply woven prepreg. The woven fibre pattern is a 5H satin weave with a four-by-one interfacing where a filling yarn floats over four warp yarns and under one.

2.2. Bulk samples preparation

The mechanical properties of the bulk ETECA adhesive were investigated as a function of curing conditions for a variety of adhesive batches. Thin films of the bulk adhesive (in the absence of initiators) were cured in an open poly (tetrafluoroethylene) (PTFE) mould (exposed to air) with a machined depth of 0.45 mm, as shown in Fig. 1a. The adhesive, i.e., Batch A was left to cure in the open mould for different time intervals, i.e., 6, 12, and 55 days at room temperature (21 °C). Samples of a variety of adhesive batches were also cured for more than 3 months in the same PTFE mould to which a cover had been fitted. These batches are denoted Batch B, Batch C and Batch D. The difference between the batches is in the strong and weak acid content. The concentration of weak acid increases from Batch B to Batch D, as shown in Table 1.

In additional tests, two different pyridine-based initiators were used to cure the adhesive under different conditions. In a low-density polyethylene (PE-LD) container, 2 µl (microlitres) of Initiator 1 (10% v/v) were rapidly mixed with 1 ml of ETECA. A micropipette with a 10 µl polypropylene (PP) tip was used to mix Initiator 1 into the adhesive. The mixture was cured at room temperature between polyethylene (PE) substrates using 80 µm spacers. A mild steel weight was then placed on the test specimens to ensure a uniform gap. The same procedure was used to cure 1 ml of ETECA adhesive with 2 µl of Initiator 2 (50% v/v). The adhesive cured using Initiator 1 was solid in 1 day, while the sample cured using Initiator 2 was solid in 4 days. The samples cured using these initiators were then stored in the laboratory for 1 month to ensure that they were fully cured prior to testing. The batches used to cure the adhesive using initiators are denoted Batch E, Batch F, and Batch G. From Batch E to Batch G the concentration of weak acid increases, as shown in Table 1. Tensile test specimens were then cut from the adhesive bulk films using a standard dog-bone shaped punch, as shown in Fig. 1b and c.

Table 1
Summary of the adhesive batches used in this research and the variables tested.

Batch	A	B	C	D	E	F	G	H
Weak acid (ppm)	X	75%X	80%X	96%X	72%X	97%X	233%X	105%X
Strong acid (ppm)	Y	77%Y	91%Y	104%Y	70%Y	105%Y	176%Y	9%Y
Variables tested	<ul style="list-style-type: none"> • Bulk mechanical properties of ETECA adhesive as a function of curing period (cured in open PTFE mould); • Phase-separation on the fracture surface of the bulk samples cured for 55 days by Scanning Electron Microscopy (SEM). • Bulk mechanical properties of ETECA adhesive cured using initiators between poly (ethylene) substrates; • Fracture toughness; Phase-separation on the fracture surface of the bulk samples by SEM. • Fracture toughness as a function of curing period and surface treatment; • Phase-separation on the fracture surface of the composite joints for solvent-cleaned substrates by SEM. 							

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