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Effect of rate and temperature on the mechanical properties of epoxy BADGE reinforced with carbon nanotubes

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ABSTRACT

The purpose of this work is to investigate the behavior of the epoxy diglycidyl ether of bisphenol A (BADGE) reinforced with carbon nanotubes when tested in tensile and flexural modes at different cross-head rates and temperatures. Tensile and flexural tests were performed under three temperatures (25 °C, 50 °C and 75 °C), varying the cross-head rate (0.5 mm/min, 5.0 mm/min and 50 mm/min) for tensile and (0.2 mm/min, 2.0 mm/min and 20 mm/min) for flexural at each designated temperature. Also, the glass transition temperature was observed, which helped understand the change in the behavior of the material during tests. An analytical mathematical model was proposed to predict the tensile and flexural strength and the model results were compared with the experimental results showing great accuracy.

1. Introduction

Maintenance of pipelines and pressure vessels represent a great cost to industry. It is essential to do it to prevent failures that would harm people and the environment. This kind of accident brings is a high cost to companies, besides that the image of the company would be denigrated. The industrial metallic structures are under great load and severe environment that leads to fissure, since the region where the fissure starts is a stress concentration area. This is more concerning in chemical and petrochemical industry, where the steel structures and pipelines to transport oil and gas are always in contact with corrosive fluids and salt water [1]. To prevent these issues and lower the cost of preventive maintenance, many industries have used polymer adhesives to repair some damage or to coat pipelines and vessels. Polymer adhesives are becoming more present on rehabilitation, protective coatings and reparability of metallic structural elements [2]. In fact, polymers have been used in many different fields of industry, like aerospace, automotive and petrochemical, because of its low cost, low weight and ease of manufacture [3,4].

Welded structures are the most fragile ones and have shown unwilling, since the weld process causes metallurgical transformations and concentration of residual stresses. Besides, from time to time the weld joints show crack, due to the aggressive and corrosive environment they are exposed [5]. A “repair weld” can repair a cracked weld bead, but it aggravates the residual stress and other imperfections [4].

In order to override this method, petrochemical industries have used adhesive polymers and their composites to repair welded structures. Fiber reinforced polymer composites are shown to be very effective because of high strength and stiffness and good corrosion resistance [1]. The combination of matrix phase’s properties and fiber’s properties of a composite can be designed as desire, since the environment and work condition of each structure may vary.

Currently, the epoxy resin is widely used as adhesives and as matrix composites due to its easiness of shaping and its excellent adhesive, mechanical and thermal properties [6]. Since mechanical properties of carbon fiber are engineered, being the most resistant reinforcing fiber, and at room temperature, it is not affected by humidity or aggressive environment like acids [7], its usage as fiber phase on composites has increased and the necessity to know better its interaction with resin matrix is needed [8]. Mechanical properties of composites are determined by both fiber and matrix phases. Not only the properties of its single material have great influence, but also the size and disposition of the fiber, i.e. the interface fiber/matrix affects the properties of the final composite [9].

Until today there are few studies and information available about the effects of strain rate and temperature on polymer composites. The stiffness and strength of composite materials normally drop with temperature rise, and decrease even more when the temperature exceeds the glass transition temperature [10,11]. It is reported by researchers that as the strain rate goes higher, the ultimate strength increase [12–15].

In this present paper, the effect of cross-head rate and temperature

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on the behavior of Bisphenol A diglycidyl ether (BADGE) epoxy reinforced with multiwalled carbon nanotubes is analyzed through tensile and flexural tests. The experimental results show the influence of temperature and rate on the strength and Young Modulus of the material. It was chosen three temperatures (25 °C, 50 °C and 75 °C) and for tensile tests three cross-head rates (0.5 mm/min, 5.0 mm/min and 50.0 mm/min). For the flexural tests, it was chosen three different cross-head rates (0.2 mm/min, 2.0 mm/min and 20.0 mm/min). An analytical expression for those parameters as functions of cross-head rate and temperature are proposed to be used as prediction models.

2. Material and method

2.1. Materials

The epoxy polymer used as matrix phase is a Bisphenol A diglycidyl ether (BADGE), DERTM 331 from Dow Química S.A. According to the polymer manufacturer, DER 331 is the most used liquid epoxy resins, since its applications are wide: adhesives, automotive coatings and casting. It is also used as a standard for composites and other variations. Morell [16] presents that BADGE Young's modulus and glass transition temperature are 3.8 GPa and 116 °C, respectively. Table 1 presents the DER® 331 epoxy matrix according to the manufacturer.

Baytubes® C150P are macro-sized agglomerates of multi-walled carbon-nanotubes (MWCNT) with a chemical purity of more than 95% carbon, without detectable free amorphous carbon and cobalt content below 1%. The particle size of the bulk material ranges from 0.1 to 3 nm and it has a mean nanotube length of 770 nm. The bulk density ranges from 120 to 270 kg/m^3. Baytubes® with a concentration of 5% in weight were dispersed in the BADGE polymer matrix in order to produce MWCNT composites.

The MWCNTs were dispersed in the polymer matrix at high speed with grinding mill. After mixing, the reinforced matrix was mixed the curing agent (OUDRACure® LC 5603) with a 100:43 ratio. The dispersion of nanotubes in the epoxy matrix was observed on a Scanning Electron Microscopy. Fig. 1 presents the SEM image of the MWCNT composite at 8300× magnification.

2.2. Methods

Tensile and 3-point bending tests were performed based on variable cross-head rates. Tensile tests started at a cross-head rate of 0.5 mm/min and then changed 5 mm/min, and lastly to 50 mm/min. The 3-point bending tests were also performed varying the cross-head rate starting at 0.2 mm/min, then changed to 2 mm/min and finally 20 mm/min were used. The cross-head rates were chosen based on ASTM D638 [17] for the tensile tests and ASTM D790 [18] for the flexural tests. Electro-mechanical sensors were used to control the longitudinal strain in the active zone of the test specimens. Additionally MWCNT composites tests were performed at 3 different constant isothermal temperatures of 25 °C, 50 °C and 75 °C using a thermostatic chamber attached to a Shimadzu® AG-X universal testing machine [19]. With this procedure, MWCNT composite was evaluated and was fully designed with fewer tests. Using a traditional design of experiment matrix, it was necessary to have at least 9 batches of MWCNT composite. With this methodology, the same information can be obtained from only 3 batches.

Table 1
Properties of DER® 331 matrix.

Viscosity at 25 °C (mPa s)	11,000–14,000
Density at 25 °C (g/ml)	1.16
Heat Deflection Temperature (°C)	111
Flexural Strength (MPa)	96
Flexural Modulus (GPa)	3.0
Tensile Strength (MPa)	79
Maximum elongation (%)	4.4

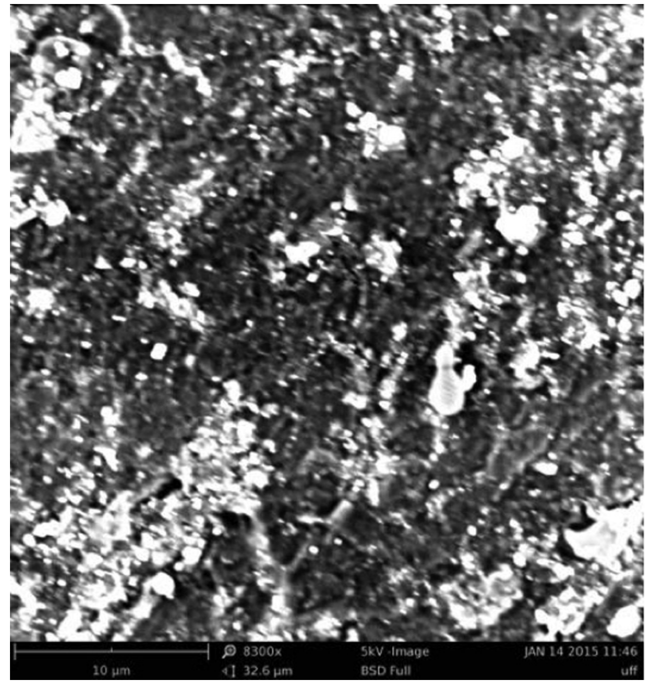

Fig. 1. SEM image of MWCNT dispersed in the BADGE matrix.

To observe the glass transition temperature Tg and the melting point a differential scanning calorimetric test (DSC) was performed on a DSC F3-MAIA Netzsch under a nitrogen atmosphere. The samples were heated at a rate of 10 °C/min from 30 °C to 500 °C. Standards indicate that polymer should be used at a maximum working temperature of Tg – 30 °C [20].

3. Results and discussion

3.1. DSC

The differential scanning calorimetric test (DSC) of the MWCNT composite is displayed in Fig. 2.

The second descent of the curve represents the glass transition temperature of 61.4 °C, which explains some of the ductile behavior of the material during the tensile and flexural tests. The working temperature of the composite should be between 20 °C and 50 °C, and this is in accordance with the experiment, where until 50 °C the material showed a more linear behavior.

The melting point *Tm* is around 150 °C, where it happens an exothermic reaction. During the endothermic reaction, the decomposition

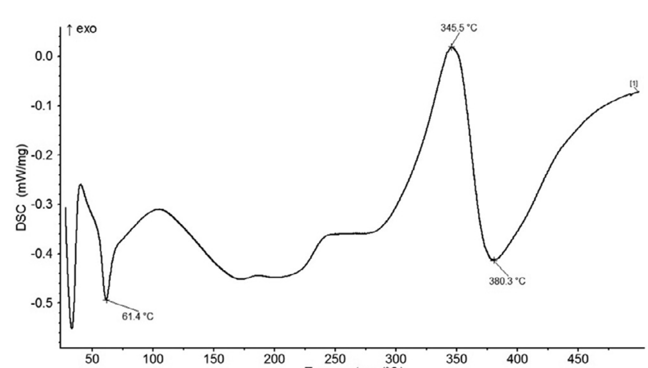

Fig. 2. DSC experiment results of the MWCNT composite.