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Characterization of triboluminescent enhanced discontinuous glass–fiber composite beams for micro-damage detection and fracture assessment

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ABSTRACT

This work reports the micro-emissions of triboluminescent (TL) concentrated composites and their evaluation at the onset of damage and crack propagation. Unreinforced vinyl ester resin and discontinuous glass–fiber reinforced non-prismatic beams were fabricated incorporating 10 wt% concentration of a highly triboluminescent material (ZnS:Mn). Triboluminescent observations were seen in both two- and three-phase composite systems throughout the failure loading-cycle. Results indicate emissions occur at various intensities corresponding to initial notch-length and imminent micro-matrix fracture. The fracturing or deformation energy was estimated by an experimental method of the *J*-integral analysis [1], where a lower threshold for excitation was found to be approximately less than $0.5\ \mathrm{J\,m^{-2}}$, below its respective critical composite fracture energy (~ 3 and $7\ \mathrm{J\,m^{-2}}$). Initiation of micro-cracks was observed for reinforced samples and were subjected to three-point bend tests in lieu of the multiple signatures of the transient signal response.

1. Introduction

Composite material systems offer robust mechanical properties and are widely used for structural applications in the aerospace, marine, defense and transportation industries. The problematic internal failure of composite systems makes it imperative to develop means for detecting failure. Damage monitoring for engineering structures is advancing towards exploration into various research disciplines utilizing technologies aimed at providing novelty in detection capabilities [2]. For instance, exploits of the triboluminescent (TL) phenomenon have paved the way for intrinsic damage sensing in macro-fiber-reinforced composites (FRCs) [3].

Triboluminescent smart materials are physical action-based luminescent phenomenon initiated by mechanical duress in excess of 1 MPa [4,5]. TL is reported to occur during duress of a material compound by elastic-, plastic-, or fracto-disturbances [6–8]. The compound known as zinc-sulfide manganese (ZnS:Mn) has been actively studied for its efficient triboluminescent production [9,10]. The mechanism behind luminescence is derived from mechanical excitation upon accruing crystal defects during new surface creation [7,8].

Prior to the previous decade, research had focused on novel synthesis of triboluminescent materials and chemical synthesis of nanomaterials [11]. Experimentation with triboluminescence has been studied at a moderate pace over the past decade. Recently, research has focused on the viability of damage monitoring applications amongst composite usage, however, an application was not fully realized until studied by Sage and Bourhill [12]. Xu et al. [9] and Hollerman et al. [10] applied a homogenized resin mixture to fabricate films for optical measurements. Bourhill et al. [3] used hand-layup procedures to manufacture composites for damage sensing in glass and carbon FRCs. The synthesized material form of TL phosphor crystals allow for homogenization in polymer matrices [13,14] and other configurations [15], thus enabling the use of liquid composite molding processes (i.e. VARTM, DFD, etc.) for its manufacturing [16].

The mechanics of composite materials is different from other modern materials. Consequently, their fracture mechanics are of high interest in practice. During composite testing and in service, crack propagation occurs due to matrix cracking, debonding, and fiber breakage [17,18]. These instances of damage result from internal energy releases. The *J*-integral proposed by Rice [19], based on the approach by Eshelby [20], is a mathematical approach to characterizing the stress concentration surrounding a crack-tip. The *J*-integral is a standard fracture mechanics test for computing the

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fracture toughness of materials. It reveals the strain energy release rate proffered by Griffith [21]. The experimental method to formulating the *J*-integral follows the procedures laid out by Landes and Begley [1,22].

In-view of micromechanics and fracture mechanics, the failure mechanisms in discontinuous short fiber and continuous composites are commensurate with several modes of propagation. Indeed, most literature whether in continuous or discontinuous cases support initial and dominant failure modes consisting of matrix cracking [23]. Fiber–matrix debonding and delamination usually occur thereafter. Matrix cracking can be assumed dominate, if a lower weight and volume fraction of fiber exist. Nguyen [24] describes two main damage mechanisms related to short fiber composites as matrix cracking and fiber–matrix debonding leading to fiber pull out. In his simulations of short fiber composites under tension, non-linear behavior was observed as a result of matrix cracking. The macroscopic response was a resultant of damage accumulations that lead towards initialization and propagation of cracking. Fiber and matrix properties, aspect ratio, volume fraction and orientation distribution of short fibers were said to strongly influence the mechanical results. Meraghni et al. [25] classified damage progression as a sequential event corresponding to the strain level that process events leading from matrix cracking to fiber pullout and then fiber breakage. This was evident with an experimental approach adopted utilizing AE signals.

Sasayama et al. [26] experimented with fundamental simulations involving microstructural damage propagation in aligned discontinuous fiber composites. The evolution of matrix cracking can be found to originate around the fibers due to impeding sites of stress concentrations. As these events take place around local fiber–matrix interface during loading cycle, the combinatorial stress starts to decrease. With continued stress, cracks will effectively link together and the load-carrying capacity of the composite will eventually be lost. In most cases, this probably signifies interfacial degradation in addition to the matrix microcracking. Interfacial degradation has been found to lead to microcrack propagation and trailing debonding paths in random structures [27].

Review of Iosepescu shear test reveal damage described as longitudinal matrix cracks are common occurrence in 0° specimens [28]. A 90° specimen orientation will display brittle failure with a subsequent load-drop-off ending in ultimate failure. Contrastingly, 0° specimens exhibit minor load drop-offs throughout the failure cycle until ultimate rupture. The behavior of the 0° shear samples is consistent with the results of the random orientation of fiber whiskers in this work, and can be attributed to small load drop/deflections resultant from notch root cracking.

In this study, we utilize the *J*-integral procedure to ascertain the strain energy induced in single-notch specimens to deduce the lower bound for excitation for multi-phase composite systems of discontinuous fibers in elastic–plastic type deformation. This study investigated the feasibility of using triboluminescent ZnS:Mn phosphors concentrated in vinyl ester resin/chopped glass fiber (6.4 mm length) for damage monitoring of two- and three-phase polymer composites under flexural loading. The behavior of unreinforced composites is strikingly different from reinforced TL concentrated specimens. The added reinforcement introduced toughness and plasticity therefore changing the failure behavior, and thus leading to unique observation of TL in the composite system. Micrographs of the two and three-phase composites reiterate the role of the matrix dominated interactions.

2. Experimental work

2.1. Material selection and fabrication

The composite material matrix employed in this study is the thermosetting ARMORSTAR IVEXC410 vinyl ester (VER) blended infusion resin from Cook Composites & Polymer (CCP). The ZnS:Mn (GL25/N-U1) phosphors were purchased from Phosphor Technology Ltd. (United Kingdom) with ranges of particle size from 2 to 25 μm. ZnS:Mn particles were directly added into VER before coupling with initiator amount. The particle phosphors were thoroughly mixed until agglomerations were well dispersed. The chopped glass–fiber whiskers used in fabricating the reinforced samples were purchased from Fiber Glast Development Corp. with diameter and lengths of, 13 μm and 6.4 mm, respectively.

Molds were made with a siloxane polymer for fabrication and ease of demolding of single-edge notched beams (SENBs) as depicted in Fig. 1. The procedure calls for a varied notch approach to deciphering the threshold energy of a propagating crack, which is an inherent material property. A notch was not included by post machining as traditionally observed. Thus, the composites were cast in an open mold as to not excite the virgin TL properties. A number of varied notches were used in this study. The variable notch lengths were 0.5, 1.5, 2.5, 3.5 and 4.5 mm. Two-phase and three-phase beams were cast according to the methods presented above to fabricate un-reinforced TL/VER and reinforced TL/VER/GF composites, respectively. The specimens were cast with nominal dimensions $55 \times 10 \times 5\ mm^3$ and poured under room temperature conditions and left to cure overnight. Samples held constant at a concentrated 10 wt% of ZnS:Mn filler; the volume fraction of GF is approximately 3 wt% to produce a uniform contact area.

Fig. 1(a) depicts the flexural test setup, whilst an illustration of the short beam sample is given in Fig. 1(b). The effective length of the sample is clearly defined as the available material length along the vertical fault-line. The effective and notch lengths are identified in the magnified illustration as e_L and n_L, respectively. In this study, the notch or pre-notch length is the difference of the standard sample thickness (5 mm).

2.2. Material testing

The physical setup is shown in Fig. 1(a), where a standard three-point bend fixture (Shimadzu Corp.) was adapted to the MTS insight system. Note, a fiber volume fraction greater than 5% was not exceeded due to loading capacity of the MTS 1 kN Insight mechanical testing system. Load–displacement plots were recorded as a function of time. The additional fixture was amended by the base blocking mount and the locking bar as depicted. As the flexural load was applied, a Hamamatsu H10722 series photomultiplier tube (PMT) was utilized to monitor the excitation emissions during loading. Data acquisition was through a NI USB-6210 DAQ (National Instruments) device connected to a mobile PC, where light emission was recorded over time. Scanning Electron Microscopy (SEM) with a JEOL JSL-7401 microscope was used to perform fractography analysis on the samples.

3. Results and discussion

The results of the *J*-integral method for triboluminescent concentrated composites reveal the bounds of the strain-to-TL for unreinforced and reinforced samples via crack opening energy. For simplicity, the material component systems are denoted two-and three-phase composite systems consisting of (1) filler, (2) resin matrices and/or (3) short-fibrils. Temporal differences between the two cases of un-reinforced and reinforced samples were identified