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## Role of inclusion stiffness and interfacial strength on dynamic matrix crack growth: An experimental study

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#### ABSTRACT

Experimental simulations of dynamic crack growth past inclusions of two different elastic moduli, stiff (glass) and compliant (polyurethane) relative to the matrix (epoxy), are carried out in a 2D setting. Full-field surface deformations are mapped in the crack-inclusion vicinity optically. The crack growth behavior as a function of inclusion-matrix interfacial strength and the inclusion location relative to the crack is studied under stress-wave loading conditions. An ultra high-speed rotating mirror-type digital camera is used to record random speckle patterns in the crack-inclusion vicinity to quantify in-plane displacement fields. The crack-tip deformation histories from the time of impact until complete fracture are mapped and fracture parameters are extracted. The crack front is arrested by the symmetrically located compliant inclusion for about half the duration needed for complete fracture event. The dynamically propagating crack is attracted and trapped by the weakly bonded inclusion interface for both stiff and compliant symmetrically located inclusion cases, whereas it is deflected away by the strongly bonded stiff inclusion and attracted by strongly bonded compliant inclusion when located eccentrically. The crack is arrested by a strongly bonded compliant inclusion for a significant fraction of the total dynamic event and is longer than the one for the weakly bonded counterpart. The compliant inclusion cases show higher fracture toughness than the stiff inclusion cases. Measured crack-tip mode-mixities correlate well with the observed crack attraction and repulsion mechanisms. Macroscopic examination of fracture surfaces reveals much higher surface roughness and ruggedness after crack-inclusion interaction for compliant inclusion than the stiff one. Implications of these observations on the dynamic fracture behavior of micron size A-glass and polyamide (PA6) particle filled epoxy is demonstrated. Filled-epoxy with 3% V<sub>f</sub> of PA6 filler is shown to produce the same dynamic fracture toughness enhancement as the one due to  $10\% V_{\rm f}$  glass.

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#### 1. Introduction

Fracture-resistant, lightweight, toughened materials are desirable in a wide variety of engineering applications. In the past few decades, there has been a great deal of interest in improving fracture toughness of polymers by adding either stiff or compliant filler particles into the matrix. Early works in this regard have been reported by Kinloch et al. (1983a,b, 1994) on deformation and fracture behavior of rubber-toughened epoxies. They performed microstructural and fracture studies on unmodified and CTBN rubber modified epoxies and proposed that localized cavitation at the particle-matrix interface, plastic shear yielding in the matrix and crack-tip blunting are the main sources of energy dissipation and increased toughness in the rubber-modified epoxy. Geisler and Kelley (1994) used rubbery and rigid fillers as well as a combination of both types to improve fracture toughness of epoxy resins and found that both rubbery and rigid particle-filled epoxies showed higher fracture energies than the neat epoxy. In addition, cured resins prepared with an optimum loading of both rubbery and rigid particles resulted in greater fracture energies than those from rubbery or rigid particles alone. The crack front impedance is said to have toughened the rigid particle composites and localized plastic deformation ahead of the crack front to have contributed in the hybrid composites. Hussain et al. (1996) investigated fracture behavior of particle-filled epoxy composites by varying TiO<sub>2</sub> filler volume fraction and particle size (20 nm and 1  $\mu$ m) and found that the micron size particles led to higher fracture toughness with increasing volume fraction than the nanoparticles. The work of Tirosh et al. (1995) focused on detailed stress analysis around compliant rubber inclusion particles in a brittle epoxy matrix and a brittle inclusion (styrene-acrylonitrile copolymer, SAN) in a compliant polycarbonate matrix. They found that the tensile strength of a brittle matrix with a soft inclusion continuously degraded as the inclusion size increased whereas the tensile strength of a soft

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matrix reinforced with stiff inclusions could be maximized with respect to the matrix properties using optimum particle size. Kitey and Tippur (2005a) examined the role of particle size and fillermatrix adhesion strength on dynamic fracture behavior of glassfilled epoxies. Their work showed that both weakly and strongly bonded filler particles enhanced the steady-state fracture toughness of the composite during crack growth compared to the unfilled epoxy. More interestingly, they observed an optimum particle size for fracture toughness enhancement and noted that weakly bonded filler improved fracture toughness more than the strongly bonded filler during dynamic fracture. The crack front blunting as well as crack front twisting were said to be the dominant toughening mechanisms.

The aforementioned works mainly address the effect of micron size particles (rigid and/or rubbery) on fracture behavior of particulate composites. A few investigations also report fracture studies with macro size fillers of spherical and cylindrical shapes and mostly have been performed under quasi-static loading conditions. Further, most reported studies are analytical (Atkinson, 1972; Cheeseman and Santare, 2000; Erdogan et al., 1974; Kushch et al., 2010; Mantic, 2009; Tamate, 1968) or numerical (Bush, 1998; Eroshkin and Tsukrov, 2005; Kitey et al., 2006; Mogilevskaya and Crouch, 2002, 2004; Savalia et al., 2008) in nature and a rather limited number of experimental works exist. The current work aims to gain a basic understanding on the fracture behavior of particle filler polymers by experimentally investigating interactions between a dynamically growing matrix crack and a stationary stiff or compliant inclusion. One of the very early experimental efforts in this regard dates back to the photoelastic investigation of crack-inclusions under quasi-static loading conditions by O'Toole and Santare (1990). In another quasi-static investigation, Savalia and Tippur (2007) performed an experimental-numerical analysis of crack-inclusion interactions using moiré interferometry and finite element modeling. Among the very few dynamic experimental works in this area, Kitey and Tippur (2008) investigated the dynamic crack growth behavior in the vicinity of an isolated stiff inclusion using coherent gradient sensing (CGS) in conjugation with high-speed photography. CGS being a surface slope detection method, they faced difficulties in analyzing the interferograms satisfactorily when the crack tip was in the vicinity of the inclusion as fringes localized near crack-inclusion interface. In view of this, the authors recently (Jajam and Tippur, 2011) conducted an experimental study of dynamic crack growth past a stiff inclusion by measuring more readily interpretable full-field surface displacements before and after crack-inclusion interaction using a 2D digital image correlation (DIC) technique coupled with high-speed imaging. They also interpreted their measurements using interfacial crack-tip fields when the crack was situated at the crack-inclusion interface. Their work revealed a spike in effective stress intensity factor values and mode-mixity behaviors were consistent with crack attraction and deflection mechanisms. Additionally, the weakly bonded inclusion specimens showed higher fracture surface roughness compared to the strongly bonded ones. This work builds on the previous investigation by the authors and examines the role of elastic mismatch between the matrix and inclusion as well as the interfacial strength between the two during dynamic fracture. The objective of the present work is to perform an optical investigation of dynamic crack growth past stiff and compliant inclusions as a function of inclusion-matrix interfacial strength and the inclusion location relative to the crack under stress wave loading conditions using full-field optical metrology and highspeed photography.

Following this introduction, the basic concept and the approach of the optical methodology used in this work are briefly described. Next, the details of specimen preparation and geometry followed by experimental setup and testing procedure are provided. Subsequently a description of experimental observations and results in terms of contours of displacements and various fracture parameters such as crack velocity histories, effective stress intensity factor histories, mode-mixity behaviors are presented. The differences in fracture surface morphologies are discussed next. The implications of the current study are then demonstrated by studying the dynamic fracture toughness of particulate composites with stiff (glass) and ductile (PA6) fillers. Finally, the major conclusions of this work are reported.

#### 2. Experimental approach

The 2D DIC method was used to monitor decorated random speckle patterns on a specimen surface during crack growth near embedded inclusions. The gray scale of these image patterns was recorded before and after deformation. The images from the deformed and undeformed sets were paired and analyzed using speckle/image correlation approach. Conceptually, a sub-image in an undeformed image was chosen and its location in the corresponding deformed image was identified and the local displacements of this sub-image were quantified. In this study, an approach developed at Auburn (Kirugulige et al., 2007; Kirugulige and Tippur, 2009; Lee et al., 2009) on a MATLAB<sup>™</sup> platform, was used to estimate in-plane surface displacement components. In the first step, displacements were estimated by performing a 2D cross-correlation operation of gray scales in the Fourier domain and the peak of the correlation function detected to a sub-pixel accuracy using bicubic interpolation. This process was repeated for the entire image to obtain full-field in-plane displacements. In the second step, an iterative approach based on nonlinear least-square minimization was used to minimize the 2D cross-correlation function in the spatial domain in order to refine the previously computed displacements. Further details regarding experimental setup and testing procedure are presented in the ensuing sections.

#### 3. Experimental details

#### 3.1. Sample preparation and geometry

A low viscosity epoxy system (Epo-Thin<sup>™</sup> from Beuhler, Inc., USA) consisting of Bisphenol-A resin and an amine-based hardener in the ratio of 100:39 was employed as the matrix material. This epoxy system offers low shrinkage and relatively long duration room temperature curing characteristics. Prior to pouring the mixture into the mold, a cylindrical inclusion of diameter, d = 4 mm and length equal to the specimen thickness (8.6 mm), was positioned at the center of the mold as shown in Fig. 1a. In this study, inclusions of two different elastic moduli, stiff and compliant relative to the matrix, were used. The former was a borosilicate glass inclusion whereas the latter was a polyurethane inclusion. The physical and measured elastic properties of the matrix and inclusion materials are listed in Table 1. A weak inclusion-matrix interface was created by wiping a thin layer of lubricant on both stiff and compliant inclusion cases. In order to achieve a strong inclusion-matrix adhesion, the glass inclusion was treated with amine-based silane ( $\gamma$ -aminopropyltrimethoxysilane) whereas the polyurethane inclusion surface was roughened using a #1000 grit abrasive paper. It should be noted here that the silane is suitable for enhancing organic to inorganic material interface strength and not suitable for organic to organic material interfaces. Hence, the polyurethane inclusion was not silane treated but roughening of the inclusion surface was found to create a strong interface between polyurethane and epoxy (as demonstrated in later sections). To avoid residual stresses, the material was cured slowly at room

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