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# Effect of filler shape, volume fraction and loading rate on dynamic fracture behavior of glass-filled epoxy

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The effect of filler shape and filler volume fraction on the dynamic fracture behavior of particulate polymer composites (PPC) has been studied. Mode-I dynamic fracture experiments were carried out on pre-notched glass-filled epoxy. An experimental setup comprising of a gas-gun and a long-bar was used to deliver one-point impact loading to unconstrained specimens. Pulse shapers were utilized to control the loading rate during impact loading. The dynamic crack initiation and propagation events were captured using high-speed photography (~300,000 frames per second). Digital Image Correlation (DIC) method was utilized to measure in-plane displacement fields around the crack-tip and extract fracture parameters including stress intensity factor histories to examine the filler shape, volume fraction and loading rate effects. The results showed a pronounced improvement in crack initiation toughness for rod-shaped fillers producing ~145% increase over unfilled epoxy at 15% $V_f$ with flakes and spherical fillers showing ~97% and ~67% improvement, respectively. For all three different volume fractions – 5%, 10%, and 15% – considered, the rod-shaped fillers produced the highest crack initiation toughness as well as post-initiation stress intensity factors followed by flakes and spheres, respectively. A linear relationship between crack initiation toughness and log of filler aspect ratio was also recorded. In addition, for 10% $V_f$ rod-shaped filler case, the effect of loading rate on dynamic fracture behavior has been examined. The loading rate study showed ~113% and ~50% increase in crack initiation toughness for the lowest and the highest loading rate cases, respectively, compared to that of neat epoxy.

## 1. Introduction

Particle-filled polymer composites (PPC) have been widely used in various engineering fields due to their excellent mechanical properties, chemical resistance, and electrical insulation. More importantly, they are also relatively easy to process at low costs and the overall properties can be tailored by choosing the filler and/or its volume fraction in the composite. Unlike traditional fiber reinforced composites, simplicity of PPC in terms of macroscopic isotropy is another aspect which often makes them quite desirable for mechanical design. Therefore, understanding the role of filler concentration, filler size and shape, and filler interfacial strength with the polymer matrix on the macromechanical properties such as stiffness, strength and toughness of the resulting PPC is critical.

Polymers are normally modified by adding inorganic-particulate fillers such as alumina, mica or silica, to name a few [1–9]. Song et al. studied the particle shape effects on the fracture and ductility of spherical and an irregularly shaped particle-reinforced Al-6061 composite containing 20% $Al_2O_3$ by volume under quasi-static tensile loading [10]. The spherical particles produced a slightly lower yield strength and work hardening rate but considerably higher ductility than the irregular particle counterpart. Their finite element analysis results indicate that the distinction between the failure modes for these two composites can be attributed to the differences in the development of internal stresses and strains within the composite due to particle shape. Nakamura et al. examined the effect of particle size on the static fracture behavior of epoxy filled with different size (ranging from 6 to 42 μm) spherical silica particles [11]. They observed increase in both energy release rate and fracture toughness with particle size. Fractography showed a relatively smooth fracture surface with small particles (6 μm) and a rough surface with large particles (caused by crack deflections around large particles). Wu analytically studied the effect of inclusion shape on the elastic modulus of two phase solids [12]. Their disk-shaped inclusions showed the maximum enhancement in elastic modulus compared to needles and spheres. The effect of particle size (4.5–62 μm) on the elastic modulus of epoxy/spherical glass particle composites were examined by

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Spanoudakis et al. [13]. At lower volume fractions ($V_f$) (10–18%), the modulus was nearly independent of particle size. For higher $V_f$ (30–46%), there was a slight decrease in modulus with increasing particle size. In a similar study, the effect of particle size on the modulus of epoxy/spherical and irregularly-shaped silica composites have been explored [14] for a size range of 2–30 μm and the modulus was observed to remain constant with particle size. Kitey and Tippur studied the role of particle size in the dynamic fracture behavior of glass-filled epoxy using optical interferrometry and high-speed photography [15]. Spherical particles (ranging from 7 to 200 μm) were used in their work to reinforce epoxy at a constant 10% $V_f$. The elastic characteristics were unaffected by the filler size, whereas fracture toughness increased with size from 7 to 35 μm and then decreased from 35 to 200 μm.

The existing literature in this area suggests that the particle shape effect on fracture toughness for particulate composites is largely unexplored. Therefore, the focus of the present study is to understand the effect of filler shape and their volume fraction on fracture behavior of glass-filled epoxy composites, particularly under *dynamic* loading conditions. However, the failure of the PPC could initiate differently under different loading rates [2,16–21]. Hence, to bridge this gap, the loading rate effects, characterized by the rate of change of stress intensity factor, on dynamic fracture behavior of epoxy filled with 10% $V_f$ rod-shaped filler were also studied since this filler shape produced a large improvement in fracture toughness.

## 2. Material preparation

Glass fillers of similar density and size scale, but differing aspect ratios (flakes, rods, and spheres; see Table 1) were chosen to study their relative shape effects on the dynamic fracture mechanisms of PPC (Fig 1). None of the fillers used had their surface modified by wetting agents and this was guided by the earlier work by Kitey and Tippur [22] showing uncoated fillers produce better fracture characteristics under dynamic loading conditions.

The glass fillers were dispersed into a low-viscosity epoxy (Epo-Thin, from Beuhler Inc., USA; Bisphenol-A resin and Amine based hardener; densities 1130 kg/m$^3$ and 961 kg/m$^3$, respectively). To carry out the dynamic fracture study, glass-filled epoxy (containing 0% (neat epoxy), 5%, 10% and 15% glass filler by volume, respectively) sheets were cast. To ensure uniform dispersion, fillers were added into the epoxy resin and stirred using a stirrer and then degassed until the mixture appeared to be free from trapped air bubbles. Subsequently, stoichiometric proportion of hardener was added to the mixture and stirred until it gelled to avoid settlement of filler particles before pouring into the mold. Upon curing for a minimum of 7 days, the sheets were demolded and machined into rectangular specimens of dimensions 60 mm × 30 mm × 9 mm (Fig. 2(a)). An edge notch of 6 mm length was introduced at the mid-span of each specimen using a diamond impregnated circular saw and the notch tip was sharpened using a sharp razor blade. The uniformity of filler dispersion was confirmed subsequently using SEM images of fractured specimen surfaces. Fractographs at four different locations for 15% $V_f$ rod-shaped glass-filled epoxy are shown in Fig. 2(b). The 15% $V_f$ rod-shaped filler example is presented here as they tend to be most prone to agglomeration. However, as evident from the micrographs, agglomerations are largely absent.

## 3. Physical and elastic properties

Physical and elastic properties were measured for all PPC and are tabulated in Table 2. Ultrasonic transducers (for longitudinal wave: Panametrics #V129 RM, 10 MHz; for shear wave: Panametrics #V156 RM, 5 MHz) coupled with a signal analyzer and an oscilloscope were used to perform pulse-echo measurements to determine the longitudinal ($C_l$) and shear wave ($C_s$) speeds at discrete locations of the cast sheet. After measuring the material density ($\rho$), dynamic elastic modulus ($E_d$) and Poisson's ratio ($\upsilon_d$) were calculated from expression for $C_l$ and $C_s$, $C_l = \sqrt{\frac{E_d(1-\nu_d)}{\rho(1+\nu_d)(1-2\nu_d)}}$, $C_s = \sqrt{\frac{E_d}{2\rho(1+\nu_d)}}$. Thus measured physical and elastic properties are shown in Table 2. It should be noted that relative to neat epoxy, $\rho$, $C_l$, $C_s$ and $E_d$ of the composites with 5%, 10% and 15% $V_f$ of fillers show a monotonic increase whereas the shape of the filler seems to produce negligible variation at a constant $V_f$.

## 4. Experimental details

### 4.1. Dynamic tests

A schematic of the experimental setup used for dynamic fracture tests is shown in Fig. 3. The setup included a 1.83 m long, 25.4 mm diameter long-bar with a 6.35 mm diameter bull-nose tip registered against an unconstrained specimen and a 304.8 mm long, 25.4 mm diameter striker held inside the barrel of a gas-gun. Both the long-bar and the striker were of the same diameter and made of aluminum 7075-T6. This eliminated the impedance mismatch between the long-bar and the striker. Three different dynamic loading rates were achieved by using different pulse shapers between the striker and the long-bar shown in Fig. 3 [23]. A soft Aluminum 1100 disc (hereon designated as 'Al-PS') of diameter 8 mm and thickness 0.9 mm produced a *strain-rate* of 10.7/s, *measured on the long-bar* by a strain gage during impact. A combined polycarbonate washer (outer diameter 6.3 mm, inner diameter 2.2 mm and thickness 0.7 mm) and Al 1100 disc sandwich pulse shaper (hereon designated as 'PC-PS') produced a lower strain rate of 3.7/s relative to Al-PS. The highest strain rate of 42.0/s was attained when no pulse shaper (hereon designated as 'No-PS') was used. The role of the pulse shaper was to ramp up the stress wave in a controlled fashion in the long-bar during impact [24]. The striker was launched towards the long-bar using the gas-gun at a velocity of ~16 m/s. When the striker contacted the long-bar, a compressive stress wave was initiated and propagated through the bar before transmission into the specimen.

A stochastic black and white speckle pattern was sprayed on to the specimen surface for performing in-plane deformation measurement using 2D DIC method. The pattern was photographed using a Cordin-550 ultrahigh-speed digital camera (Cordin Scientific Imaging, Salt Lake City, UT, USA) equipped with 32 independent CCD image sensors (1000 × 1000 pixels) positioned

**Table 1**
Glass Filler Characteristics.

| Shape | Source | Average dimensions | Aspect ratio[*] (AR) | Density (kg/m$^3$) |
|---|---|---|---|---|
| Flake | ACF-300: Isorca Inc., USA | 30 μm wide, 5 μm thick | 30/5 = 6 | 2,500 |
| Rod | Milled Fiber: Fiberglass Supply, USA | 800 μm long, 10 μm diameter | 800/10 = 80 | 2,500 |
| Sphere | A300: Potters Industries, USA | 35 μm diameter | 35/35 = 1 | 2,500 |

[*] Aspect Ratio was determined by dividing the largest average dimension by the shortest average for each filler type, provided by the manufacturer.