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High strain rate sensitivity of epoxy/clay nanocomposites using non-contact strain measurement

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ABSTRACT

As polymers are rate sensitive to mechanical properties, dynamic tensile tests are carried out on drop mass setup, to obtain medium strain rate stress-strain response which fills the gap between quasi-static strain rate (<10 s^{-1}) and split Hopkinson pressure bar (SHPB) technique (>1000 s^{-1}). The present research work is to study the effect of medium strain rate on tensile behavior of epoxy/clay nanocomposites. Neat epoxy and nanocomposites containing 1.5, 3 and 5 wt% clay content are fabricated using mechanical stirrer. The dispersion of nanoclay in epoxy is examined using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The digital image correlation (DIC) technique is employed for evaluating full-field strain and strain rate using high speed CCD camera, which captures about 100,000 frames per second. Stress-strain measurements are reported for epoxy/clay nanocomposites for strain rates ranging from 0.008 to 450 s^{-1}. The results reveal that the tensile strength and modulus increase with increase in strain rate for epoxy and its clay nanocomposites. The fracture surfaces of tensile specimens are investigated using scanning electron microscopy (SEM).

1. Introduction

Epoxy resins are widely used in aerospace, automobile, and electronics industry because of its good mechanical, electrical, thermal, and chemical properties. Addition of nanoclay to epoxy resin results in further enhancement of properties due to its unique features such as high aspect ratio, intercalation/exfoliation characteristics, environment friendly and easy availability [1–3]. Lan and Pinnavaia [4] found significant improvement in tensile strength and modulus by addition of nanoclay in contrast to the neat epoxy. However, the improved performance of these nanocomposites guaranteed only when homogeneous dispersion of nanofillers is achieved [5].

Researchers [6–21] primarily focused on fabrication and characterization aspects of nanocomposites, but very few literature reported the medium strain effects on mechanical properties of such materials [22]. Since, the conventional servo-hydraulic machine is limited to lower strain rates (<10 s^{-1}), split Hopkinson pressure bar (SHPB) technique is widely used to achieve very high strain rate (>1000 s^{-1}) tensile properties. However, the experimental techniques to generate tensile properties in medium strain rates (1–100 s^{-1}) are not well established [23]. Chen et al. [24] modified the split Hopkinson pressure bar to investigate the dynamic properties of epoxy resin and poly (methyl methacrylate) (PMMA) under tension and compression loading. They observed that the tensile specimens failed in a brittle manner during dynamic testing with reduction in failure strain, in contrast to quasi-static testing where specimens failed in a ductile manner. Similar observations were reported by Gilat et al. [25] for two different epoxy resin systems at strain rates (5×10^{-5} to 700 s^{-1}) in tensile and shear loadings. Guo et al. [22] investigated the quasi-static and dynamic compression responses of SiO_2/epoxy nanocomposites of strain rates from 10^{-4}–10^{4} s^{-1} using SHPB apparatus. They observed that the material behavior is influenced by strain rate and nanoparticle dispersion. Evora et al. [26] used direct ultrasonification technique for producing polyester/TiO_2 nanocomposites with good particle dispersion. They observed that the presence of TiO_2 nanoparticles had a significant effect on quasi-static fracture toughness and stiffening behavior. Roland et al. [27] investigated dynamic tensile response over a range of strain rates from 0.06 to 573 s^{-1} for polyurea using a drop weight test instrument. They found an increase in stiffness and failure stress and decrease in failure strain with increasing strain

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rate. Xiao [23] carried out dynamic tensile test at strain rates of 4 and 400 s^{-1} using a servo-hydraulic testing machine on four polymer materials and validated the dynamic tensile tests by evaluating the condition of dynamic stress equilibrium using SHPB criterion. Raisch et al. [28] modified the servo-hydraulic machine to achieve a strain rate of 670 s^{-1} and introduced a curve fitting procedure to determine Young's modulus using a viscoelastic stress strain relation. They observed that both modulus and yield stress increased logarithmically per decade of strain rate and apparently yield strain was found to be constant. Gurusideswar and Velmurugan [29] studied the effect of nanoclay on epoxy and glass/epoxy composites, at low strain rates. They observed that the tensile strength increased by 15% whereas the increase in Young's modulus is about 13%, when the strain rate increased from 0.0001 to 0.1 s^{-1} for neat epoxy system.

To summarize, as polymeric materials show differences in mechanical behavior under quasi-static and dynamic tensile, an attempt is made to study the dynamic tensile properties of epoxy/clay nanocomposites on drop mass test setup using digital image correlation (DIC) technique for non-contact strain measurements.

Digital image correlation (DIC) technique is widely used to determine in-plane strain field data and the significance of DIC for SHPB experiments was reported by Gilat et al. [30] and Koerber et al. [31] for epoxy and uni-directional carbon epoxy composites, respectively. However, due to heterogeneous nature of composites, sophisticated experimental techniques are needed to assert proper conclusions and to get reliable data.

This paper discusses the application of a drop mass system and DIC for dynamic tensile characterization of epoxy/clay nanocomposites at strain rates from quasi-static to 445 s^{-1}. Fractography of tested specimens were carried out using SEM.

2. Experimental details

2.1. Materials

Epoxy, a medium viscous diglycidyl ether bisphenol A resin (DGEBA), and the curing agent, a low viscosity aliphatic polyamine (TETA) procured from Huntsman Ltd, (India) was used for fabrication. Organic clay, Garamite 1958® with a moisture content of 4% and density of 1.7 g/cm^3 was sourced from Southern Clay Products, Inc., (USA) and used as nano filler.

2.2. Specimen preparation

The epoxy resin was preheated at 60 °C to lower the viscosity and organoclay was dried at 50 °C in oven prior to mixing to remove moisture content. Nanocomposites with various amount of clay ranging from 1.5, 3 and 5 wt% were fabricated using mechanical stirrer at an optimal speed of 2000 rpm followed by ultra sonication process. Neat epoxy resin was also fabricated for reference. An appropriate amount (100:12) of curing agent was then added to the mixture and mixed well for 5 min. The final mixture was degassed and cast in the mold with a dimension of 300 × 300 mm^2 and cured for 24 h at room temperature. The tensile specimens were cut by water jet cutting technique to get required dimension of 10 mm gauge length × 3 mm width × 2 mm thick.

2.3. Characterization techniques

X-ray diffractograms were obtained by a Bruker D8 Advance with CuKα radiation (λ = 0.154 nm) at a scan rate of 2°/min and a step size of 0.02°. High resolution scanning electron microscopy (HR-SEM) studies were performed using a Hitachi S-4800 model operating at 5 kV accelerating voltage and equipped with Energy Dispersive Spectroscopy (EDS). The surfaces were sputter-coated with a gold film prior to SEM investigation to avoid charging of epoxy samples. Transmission electron microscopy (TEM) was performed using Jeol 3010 model at an accelerating voltage of 200 kV. Ultra microtoming was carried out to get sections with a nominal section of 120 nm and placed on a carbon coated Cu grids of 200 mesh to capture bright-field TEM images.

2.4. Drop mass setup

Drop mass test setup (Fig. 1) used for generating strain rates from 10 to 1000 s^{-1} consists of two guide rods, drop mass, and stop blocks. Prior to testing, drop mass is lifted by an electric motor to a fixed height through bearing assemblies and then dropped. The present test facility is custom built to handle different specimen geometries as well as strain rates.

2.5. Digital image correlation

Digital image correlation is a technique used to determine the full-field displacement and strain contours of a material. A random speckle pattern is sprayed on the specimen surface using Aerosol spray paint. In the current study, a coarse speckle pattern (Fig. 2) is applied manually for dynamic testing, considering the image resolution of high speed camera. Commercial image correlation VIC 2D software is used for evaluating the full-field strain during quasi-static and dynamic experiments.

2.6. Quasi-static testing and dynamic testing

Quasi-static tensile tests were performed in INSTRON 3365 5 kN testing machine at a cross-head speed of 5 mm/min, which corresponds to a nominal strain rate of 0.008 s^{-1}. Tensile modulus, strength, and strain were determined for epoxy/clay nanocomposites as per the **ASTM: D638–10**. The Grasshopper3 (2/3″

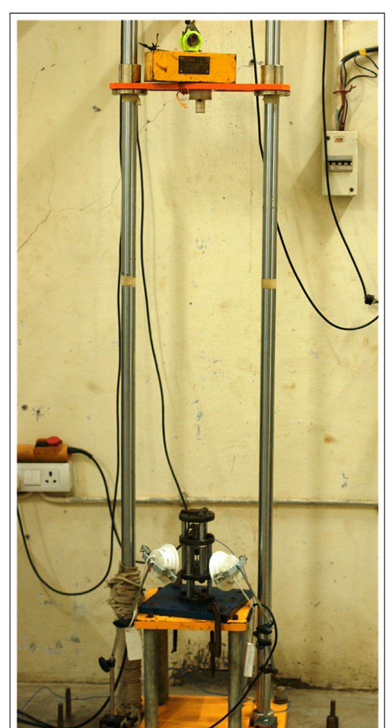

Fig. 1. Drop mass setup.