ELECTRICAL RESPONSE OF GRAPHENE REINFORCED COMPOSITES UNDER STATIC AND DYNAMIC LOADING

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Abstract
An experimental investigation was conducted to understand the electro-mechanical response of graphene reinforced polystyrene composites under static and dynamic loading. Graphene-polystyrene composites were fabricated using a solution mixing approach followed by hot-pressing. Absolute resistance values were measured with a high-resolution four-point probe method for both quasi-static and dynamic loading. A modified split Hopkinson (Kolsky) pressure bar apparatus, capable of simultaneous mechanical and electrical characterization, was developed and implemented to investigate the dynamic electro-mechanical response of the composites. In addition to measuring the change in electrical resistance as well as the dynamic constitutive behavior, real-time damage was captured using high-speed photography. The real-time damage was correlated to both stress-strain and percent change in resistance profiles.

1 Introduction
Extraordinary mechanical properties combined with excellent transport properties make graphene a promising addition to the future of smart composite materials. When graphene platelets are effectively dispersed within a matrix material, an electrical network can be formed and serve as an internal sensor. Understanding the electrical response of graphene-reinforced nanocomposites under dynamic loading conditions will be particularly useful in designing sensors for applications such as structural health monitoring in aircrafts and smart body-armor response systems. A comprehensive series of experiments were conducted to experimentally investigate the electro-mechanical response of graphene-PS composites subjected to static as well as dynamic split Hopkinson pressure bar (SHPB) loading. A novel SHPB apparatus, capable of simultaneous mechanical and electrical characterization, was developed to effectively investigate the electro-mechanical response of the graphene reinforced PS composites. The history between the electrical resistance change, mechanical loading, and the high-speed deformation photography are correlated to characterize the electrical-mechanical response of the fabricated composites.

2 Material and Specimen Geometry
The graphene platelets used in this study were xGnP™ Nanoplatelets (XG Sciences). These unique nanoparticles consist of short stacks of one or more graphene sheets having a lateral dimension of ~25 µm. The edges of these sheets are sites for functionalization, which may help facilitate bonding within the polymer matrix. The specific polymeric matrix chosen for this study was polystyrene (PS) (Crystal PS 1300). 5 vol.% graphene-PS composites were prepared by solution mixing followed by compression molding.

Fig. 1 illustrates specimens prepared for both quasi-static and dynamic compression loading experiments. Specimens used in quasi-static experiments were 10 mm in length and had a diameter of 6.35 mm, where the loading was exerted in the longitudinal direction of the 10 mm length. Specimens used in dynamic experiments were 8.68 mm in length and had a diameter of 15.87 mm. Two V-notch channels with a depth of 0.5 mm were machined in the middle section of both specimens. The channels were used to implement a modified four-point probe method in order to effectively
measure the change in electrical resistance of the specimen during loading.

3 Experimental Setup

3.1 Quasi-static Loading

The quasi-static loading was implemented by a screw-driven testing machine. A modified four-point probe method was utilized to measure the resistance change during the compression tests [1-2]. The experimental setup used to capture the resistance change of the composites under quasi-static loading is shown in Fig. 2. A constant current source was used to supply a DC current flow through the specimen. The graphene-PS specimen was sandwiched between two aluminum plates to guarantee uniform current flow through the specimen during the compressive loading. Silver paint was applied to the top and bottom of each specimen to minimize the contact resistance between the specimen and the plates. Each loading head was insulated from the electrical measurement system. Two electrometers were used to measure the voltage at each of the two individual inner probe rings. The difference between the two voltage readings, which corresponds to the voltage drop across the two inner probes, was measured using a digital multimeter and recorded using a LabView system.

3.2 Dynamic Loading

A modified split Hopkinson (Kolsky) pressure bar apparatus, capable of simultaneous mechanical and electrical characterization, was developed and implemented to investigate the dynamic electro-mechanical response of the graphene-PS composites. A typical SHPB consists of a striker bar, a solid incident bar and a solid transmission bar. The striker bar is propelled using an air-operated gun. A pulse shaper is commonly placed at the impact end of the incident bar with a thin layer of lubricant to improve force equilibrium conditions at the specimen-bar interfaces. The theoretical details of SHPB can be obtained from Kolsky [3]. The specimen is sandwiched between the incident bar and the transmission bar. A lubricant is applied between the specimen and the bar interfaces to minimize friction.

Several modifications were made to the existing SHPB to simultaneously capture the electrical response as well as the mechanical behavior of the specimen during the dynamic loading. A sketch of the novel SHPB device is shown in Fig. 3. The incident and transmission bars were 19.05 mm in diameter and measured 1613 mm and 1220 mm in length respectively. A similar four-point probe technique, as described in quasi-static experiments, was implemented. Lead wires were securely attached to each bar to provide a means of supplying a DC current flow through the specimen during loading. In order to obtain an accurate electrical response of the specimen, nylon bushings were fabricated and installed to isolate the incident and transmission bars from the supports. To minimize the contact resistance as well as the frictional forces present at the specimen-bar interfaces, a conductive lubricant (AI Technology Inc. ELGR8501) was applied to the specimen faces. Additionally, a pulse shaper consisting of a single layer of electrical tape and clay (~ 2 mm thick) were used to isolate the incident bar from the gas gun apparatus and to improve the force equilibrium conditions at the specimen-bar interfaces. A constant current source with high frequency response (Keithley Instruments Model 6221) was used to supply the constant DC current flow under the high rate deformation while the voltage drop between the two inner probes was measured by a differential amplifier (Tektronix ADA 400A) and recorded by a digital oscilloscope (Tektronix TDS 3014).

4 Experimental Results and Discussion

4.1 Quasi-Static Loading

A typical electro-mechanical response of a 5 vol.% graphene-PS composite under quasi-static compressive loading is shown in Fig. 4. During the quasi-static compression, the stress of the specimen monotonically increases to 47 MPa at 5% strain and then gradually decreases. Taking the initial resistance as a baseline, the percent change in electrical resistance increases proportionally with strain. Initially, no significant change in resistance is observed up until ~ 2% strain. Due to the brittle nature of the PS matrix, small micro-cracks begin to form as the compressive strain increases resulting in a substantial increase in electrical resistance. Since the electrical resistance of the matrix material is very high, the graphene particles exclusively conduct the
electrical current within the material. When considering the negligible change in graphene particle geometry during the compressive event, the resistance change is caused by the interruptions of the electrical networks between the graphene particles. Scanning electron microscopy (SEM) was performed on the post-mortem specimens to observe this phenomenon. As shown in Fig. 5, it is evident that in certain regions, small agglomerates of the graphene sheets served as crack nucleation sites. A series of experiments were carried out and the change in resistance showed this similar behavior.

4.2 Dynamic Loading

A typical electrical response along with the mechanical behavior of both neat PS and graphene reinforced composites is shown in Fig. 6. As the specimen undergoes dynamic compression, the electrical resistance increases proportional to the change in strain. As the stress of the specimen monotonically increases to 75 MPa at 5% strain, the bulk electrical resistance of the specimen increases ~85% due to the formation of micro-cracks within the matrix. As the internal damage grows, the electrical resistance continues to increase as the electrical efficiency of the composite is further diminished. The resistance does not abruptly jump but gradually increases as damage initiates and propagates throughout the composite. We believe that this difference may come from the non-uniform dispersion of graphene inside the matrix and complex initiation and propagation of damages. A typical electrical response along with the real-time deformation images of a 5 vol.% graphene-PS composite subjected to dynamic loading are shown in Fig. 7. The time frames used in the loading event are chosen in a manner such that they can be correlated to the time at which certain deformation mechanisms were first observed. During the first 50 µs, the specimen undergoes a slight uniform compression. Since the strain of the material is very minimal during this time, no noticeable change in electrical resistance is observed. At ~60 µs, a crack is seen to initiate and propagate through the specimen consequently causing an increase in resistance. From 60 to 100 µs, damage further propagates throughout the specimen leading to larger increases in electrical resistance.

Fig. 8 shows the effect of 5 vol. % graphene on the static and dynamic behavior of polystyrene. Despite an increase in yield stress for dynamic loading in comparison to static loading, the presence of graphene within the polystyrene matrix significantly diminishes the mechanical properties of the composite material under both static and dynamic compression.

Conclusions

The present paper describes the electro-mechanical response of graphene reinforced polystyrene composites under quasi-static and dynamic compressive loading. Graphene-PS composites with low resistance were fabricated using a solution mixing approach followed by hot-pressing. A modified four-point probe method, using line and face contacts rather than point contacts, was implemented to accurately monitor the bulk electrical resistance of the composites. Moreover, a modified split Hopkinson (Kolsky) pressure bar apparatus, capable of simultaneous mechanical and electrical characterization, was developed and implemented to investigate the dynamic electro-mechanical response of the composites. In addition to measuring the change in electrical resistance as well as the dynamic constitutive behavior, real-time damage was captured using high-speed photography. The real-time damage was correlated to both stress-strain and percent change in resistance profiles. Due to a high concentration of graphene particles, small aggregations of the graphene were inevitably formed which resulted in inadequate load transfer between the graphene particles and the PS matrix. Consequently, a significant decrease in mechanical properties under both static and dynamic loading conditions with the presence of graphene was observed. The bulk electrical resistance of the composite increased significantly due to the brittle nature of the PS matrix as well as the presence of relative agglomerations of graphene platelets which resulted in micro-crack formations.

Acknowledgements

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References


Fig. 1. Specimen geometry used for (a) quasi-static loading and (b) dynamic loading

Fig. 2. Experimental setup for measuring resistance change under quasi-static conditions
Fig. 3. Experimental setup of SHPB apparatus with dynamic electrical characterization setup

Fig. 4. Typical electro-mechanical response of 5 vol.% graphene-PS under quasi-static loading
Fig. 5. SEM image of a cross-section of a post-mortem specimen loaded to 7% eng. strain

Fig. 6. Typical electro-mechanical response of 5 vol% graphene-PS under dynamic loading
Fig. 7. Percent change in electrical resistance of a 5 vol.% graphene-PS composite subjected to SHPB loading with real-time deformation images.
Fig. 8. Comparison of pristine PS vs. graphene-PS under static and dynamic loading