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Study of the energy absorption capabilities of laminated glass using carbon nanotubes

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ABSTRACT

Laminated glass (LG) typically consists of two or more glass plies bonded together with a transparent thermoplastic elastomeric interlayer, often composed of polyvinyl butyral (PVB). This interlayer primarily serves as a means of preventing splintering and of absorbing energy upon blast/impact. This research attempted to enhance the impact resistance of LG by increasing PVB interlayer energy absorption by embedding carbon nanotubes (CNTs). Interlayers were formed by electrospinning aligned PVB fibers mat embedded with various concentrations of CNTs. Subsequently, the fiber mat was hot-pressed between two glass plies forming a composite film. The composite fibers were characterized using optical, SEM, and TEM microscopy. The mechanical and thermo-mechanical properties of fibers were determined by dynamic mechanical analysis (DMA), and the energy absorption capacities of the modified LGs were measured by applying the Charpy impact test of un-notched samples. A ~ 30% increase in composite fiber (CNT 1.5 wt.%) strength was observed, along with a ~ 70% increase in elastic modulus, measured at a strain rate of 0.1 min^{-1}, in comparison to CNT-free fibers. Increased CNT loading restricted the segmental motion of polymer macromolecules and provided the geometrical confinement effect to neighboring macromolecules in the nanoscale fiber. The energy absorption of a double-layered LG embedded with carbon nanotubes increased by nearly 341%, where experimental results demonstrated the role of the CNTs pull-out toughening mechanism. In parallel, transmission of visible light decreased by 60%.

1. Introduction

Laminated glass (LG) is widely used as an enveloping and protective element in the automotive and construction industries. It can be of critical value in providing impact damage resistance and in protecting the behind-target space from penetration of foreign objects. LG is typically comprised of two or more glass plies bonded together with a transparent, thermoplastic elastomeric interlayer, typically formed from polyvinyl butyral (PVB). The failure behavior of glass–PVB laminates subjected to dynamic impact differs from that of monolithic glass [1,2], due to both PVB elastic properties, that can vary between 0.1 MPa and 250 MPa, depending on the strain rate [3], and to its energy-absorption capacities.

Previous experimental and analytical studies have been carried out on LG subjected to small missile impact at low velocity. Behr et al. [4] reported that the thickness of both the inner glass ply and the PVB interlayer significantly influence the impact resistance of the inner glass ply. More specifically, they determined, in both simulatory and experimental setups, that PVB interlayer thickness inversely correlates with the peak of the dynamic radial strain. Finite element analysis of low velocity impact demonstrated a reduction in the maximum principal stress with increasing PVB thickness [5]. At higher values of PVB bulk modulus, increasing PVB thickness for very thin interlayers (<0.4 mm) causes an increase in the maximum principal stress, followed by a reduction. Namely, a reduction in maximum principal stress may not be sensed until sufficient PVB thickness is provided to overcome an initial increase in maximum principal stress.

Based on the above mentioned principles, this work pursues a means of enhancing the energy absorption capacities of the LG interlayers without compromising the interlayer thickness. When considering the remarkable mechanical properties and the nanoscale of carbon nanotubes (CNTs) [6], it is no surprise that polymer composites reinforced with CNTs feature improved stiffness, tensile strength and toughness [7–12]. Also, when considering the high thermal and electrical conductivity of CNTs, their incorporation in the laminate interlayer is expected to improve the electrical, thermal, and conductive properties of the LG. However, due to their elongated, graphite, tubular structures, CNTs strongly absorb visible light [13]. Therefore, CNT-embedded polymer matrices must be designed to preserve the construct's optical properties. The present work describes fabrication of electrospun [14] composite fibers comprising a CNT-embedded PVB matrix [15,16].

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Electrospinning ensured homogeneous dispersion and alignment of the CNTs along the fiber [17], which could then be deposited on a glass ply to form an anisotropic structure. Subsequently, hot press were used to transform the structure into a composite film positioned between two glass plies (see Fig. 1).

Fiber morphology and microstructure were characterized by optical, scanning electron (SEM) and transmission electron (TEM) microscopes. Mechanical and thermo-mechanical properties were examined by Dynamic Mechanical Analysis (DMA). Impact resistance of the composite LG to low-velocity demonstrated the potential of the proposed polymer interlayer in strengthening LG structures.

2. Materials and methods

2.1. Materials

Polyvinyl butyral (PVB), a random terpolymer typically formed from 76% vinyl butyral, 22% vinyl alcohol and 2% vinyl acetate, was purchased from Kuraray Europe GmbH. Multiwall CNTs of 95% purity, with an outer diameter of 10–20 nm, an inner diameter of 5–10 nm, and length of 0.5–2 μm, were purchased from NanoAmor (Nanostructured & Amorphous Materials Inc., USA) and used as provided. Analytical-grade dimethylformamide (DMF) and tetrahydrofuran (THF) were purchased from Frutarom Ltd., Israel. CNTs were suspended in 6.16 g DMF at various concentrations. The suspensions were sonicated in a water bath sonicator for 5 h and then supplemented with 1.20 g of PVB and 2.64 g THF, at room temperature, with a final DMF/THF ratio of 7:3 (w/w). The PVB-CNT suspensions were then sonicated for 5 h, yielding stable suspensions suitable for electrospinning. After solvent evaporation, CNT-PVB weight ratios were 0.06/1.20, 0.012/1.20 and 0.018/1.20. A control solution for electrospinning was prepared by dissolving 1.2 g PVB in 8.8 g DMF and THF (7:3 (w/w)) to obtain a 12% (w/w) solution.

2.2. Electrospinning

The PVB solution and PVB-CNT suspensions were electrospun to respectively form fiber mats of oriented polymer monoliths or composite nanofibers. The flow rate, controlled by a syringe pump, of the PVB solution was constant at 0.9 mL/h with an electrostatic field of ~1.2 kV/cm. A collector wheel, positioned at a distance of 12 cm from the spinneret (needle 23G) and with a tangent velocity of 6 m/s [18], was used to collect aligned fibers. The ambient temperature was 21 ± 1 °C, and the air humidity was 48–51%.

2.3. Structural and morphological characterizations

High resolution images of the nanofiber mesh surface were obtained using FEI E-SEM Quanta 200 at 20 kV, In Lens and E-T SE2 detectors at a sample–detector distance of 2 mm. FEI Tecnai T20 S-Twin TEM at 200 keV, a LaB6 electron source and an FEI Super Twin objective lens were used to study the microstructure of the composite fibers. This microscope is equipped with BF (bright field), DF (dark field) and STEM (scanning transmission electron microscopy) detectors. The fibers were electrospun directly onto 200 mesh copper TEM grids. Fibers orientation was analyzed using Image J software.

2.4. Thermal characterization

Thermogravimetric analysis (TGA; SETARAM TG-DTA 92.) was used to measure the amount and rate of sample mass change, as well as their compositional properties as a function of temperature. Fiber mats were transferred to platinum pans and subjected to heating at a rate of 5 °C min^{-1}, from room temperature to 860 °C, under an inert atmosphere of argon. Sample mass was limited to approximately 20 mg and fibers were slightly compacted into the pans to achieve good thermal contact with the inner surface. Two specimens of each fiber mat composition were tested.

2.5. Mechanical characterization

Tensile tests were executed by Dynamic Mechanical Analysis (TA-DMA Q800) on films and aligned fiber mats. Samples were 12 mm long, 2 mm wide and 0.09–0.12 mm thick. The strain rate varied between 0.02 and 0.2 min^{-1}, and clamp tightening torque was 5.8 in-lb. For temperature sweep experiments, samples were subjected to a sinusoidal displacement with 0.45% strain, at a fixed frequency of 1 Hz from −120 to 100 °C, at a heating rate of 2 °C min^{-1}. Four specimens were tested for each fiber mat composition.

2.6. Specimen preparation

Samples were made of laminated double layer glass reinforced with a single polymer interlayer. The specimens were glass slides ($76 \times 26 \times 1$ mm^3, soda lime glass, DIN ISO 8037/1) purchased from Marinfeld, Germany. All laminates were laid-up with fiber, aligned parallel to the longitudinal direction of the glass slides. For specimen curing, the temperature was initially raised from room temperature to 120 °C, within 1 h, held steady for 2 h and then decreased to room temperature within 0.5 h. The entire process was conducted in a vacuum oven set at 300 mbar.

2.7. Charpy impact test

All specimens were subjected to the Zwick–Roell Charpy impact test, during which the absorbed energy was recorded. The applied energy was 1.1 J. Tests were executed on un-notched laminated

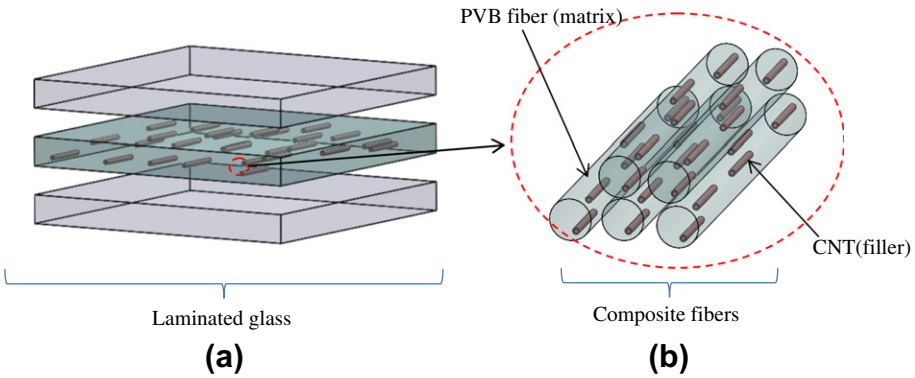

Fig. 1. A sketch of the designed laminated glass (LG) with the CNT-embedded polymer (PVB) interlayer ($76 \times 26 \times 2$ mm^3). (a) Isometric view, (b) enlarged view of the interlayer consisting of aligned electrospun composite fibers.