Mechanical Characterization of Polycarbonate-Graphene Oxide (PCG) Nanocomposite

Jaskaran Singh, Suneev Anil Bansal , and Amrinder Pal Singh

Abstract Polymer matrix nanocomposite are of great interest due to its high specific strength, low cost, and ease of processing & synthesis. Various attempts have been made to improve properties of polymer matrix by introducing nano reinforcement. In present report, effect of low cost nanosheet reinforcement, Graphene Oxide (GO), in Polycarbonate (PC) matrix was studied for mechanical properties of PC-GO (PCG) nanocomposite. PC is used in various mechanical parts and structural applications. Low cost GO was synthesized by chemical oxidation route using low cost graphite flakes. To get better dispersion of GO in PC, solution mixing method was used. First, the thin film of PCG nanocomposite was prepared by mixing sonicated GO in Tetrahydrofuran (THF) and beads of PC. After that these sheets were extruded using an injection molding machine to synthesize dog-bone sample of PCG nanocomposites. Morphological studies of samples were performed using FE-SEM machine. Dog-bone samples were characterized using micro mechanical testing machine. PCG composite was prepared for 0.05, 0.1, and 0.2 wt.% of GO reinforcement in PC matrix. As the percentage of GO reinforcement increased, both tensile strength and elastic modulus of PCG nanocomposite increased. At 0.2 wt.% of GO tensile strength and elastic modulus was increased by 57 and 13%, respectively. GO reinforcement in PC showed better mechanical performance over pure PC.

J. Singh · S. A. Bansal · A. P. Singh (✉)
Department of Mechanical Engineering, UIET Panjab University, Chandigarh 160014, India
e-mail: apsinghpu@gmail.com

J. Singh
e-mail: apsinghpu@gmail.com

S. A. Bansal
e-mail: suneev@gmail.com

S. A. Bansal
Department of Mechanical Engineering, MAIT, Maharaja Agrasen University, Baddi, HP 174103, India

1 Introduction

Two or more materials, having different types of properties, are combined in different ways to form composites that possesses superior properties than pristine matrix materials. Matrix phase has less strength whereas the reinforcement has more strength and stiffness than matrix, giving good mechanical and physical properties to composites. Thus composite materials possess better mechanical properties along with light weight, a combination difficult to find in conventional materials like metals. Weight of the finished part produced by composite has high strength to weight ratio because of the low density of composite as compared to bulk material. Uniquely, polymer composites are widely used for mechanical applications in aerospace, automobile, marine industry, etc., due to their light weight, better strength, and ease of processing [1, 2].

Polycarbonate (PC) finds variety of application like structural parts, body, automotive parts, helmets, hydrophobic surfaces, packaging, etc. [3, 4]. Various filler like carbon fibers, carbon nanotubes, glass fibers, etc., were reported to enhance properties of PC by synthesizing PC composite materials [5–10]. Still area of carbon nanoparticles reinforcement in PC is still in development. As Graphene/Graphene oxide reinforcement reported to produce excellent results with other matrix materials, GO in PC can produce exciting results [11, 12].

Graphene, sp^2 hybridized two-dimensional (2D) carbon nanostructure, has as an atomically thin sheet structure with carbon to carbon 0.142 nm. A 2D graphene sheet weighing only 0.77 mg/m^2 is 100 times stronger than the steel sheet of same size and thickness. The single-layered graphene sheet is one of the strongest materials; its breaking strength is 42 Nm^{-1} and 1.0 TPa Young's modulus. Graphene exhibits excellent thermal conductivity (5000 Wm^{-1} K^{-1}), extremely high transmittance at 97.7% over wide range of wavelengths and very large surface area of 2630 m^2/g [13]. Graphene, is extensively researched for application in various fields like battery storage, sensing, photo activity, composites, due to fascinating mechanical properties, electrical properties, and thermal properties [14–18].

Our group recently studied effect of graphene/CNT reinforcement in polystyrene and found carbon nanomaterial can enhance mechanical properties of polymer [19]. Effect of Graphene oxide (GO) reinforcement of epoxy resin was also studied and found that GO enhances modulus and hardness of epoxy-GO composites [20]. Composite synthesis requires a high quantity of GO at reasonable quality that can be suitably produced by wet chemical oxidation method. Although graphene was first synthesized from graphite precursor by simple mechanical cleavage method using an adhesive tape [21, 22].

The present work reports mechanical characterization of polycarbonate-Graphene Oxide (PCG) nanocomposite with the percentage weight variation of GO. GO was synthesized by chemical oxidation method. To achieve better dispersion solution mixing method was used. Dog-bone samples were synthesized and tested on micromechanical testing machine. Small percentage reinforcement of GO in PC tremendously improved mechanical properties of PCG nanocomposite.

2 Experimental

2.1 Materials

Commercial grade polycarbonate (PC), in form of beads, was procured from market. Received materials were put in vacuum over at 60 °C for 8 h to remove moisture and volatile substances. Graphite flakes were procedure from Sigma Aldrich as used as received. All reagents used were of AR grade and procured from either, Thermo Fisher Scientific, India or Sigma Aldrich, India. All reagents were used as received without any purification.

3 Synthesis of GO

Graphene oxide (GO) was prepared using wet chemical oxidation method reported earlier [23, 24]. In short, 1 g of graphite flakes were stirred in H_2SO_4 and H_3PO_4 acid mixture in 9:1 ratio (by volume) on magnetic stirrer. An oxidizing agent, $KMnO_4$ (six times by weight of graphite flakes), was slowly added and mixture was left on stirring condition till color changes to dark brown. A small amount of H_2O_2 was added to stop oxidation reaction. Solution was washed several times with mild HCl solution and then with De-ionized water to neutralize pH. Solution was dried in vacuum oven to get sheets of GO.

4 Synthesis of PC-GO Nanocomposite

Fabrication of Dog-bone shaped specimen was carried out in two steps. In first step, PC was mixed with GO using solution mixing method. GO was sonicated in Tetrahydrofuran (THF) using ultra-sonicator for 15 min. After complete dispersion of GO in THF, beads of PC were added in solution under constant stirring condition. Different composition samples were obtained by varying the weight percentage of GO in PC from 0–0.2 wt.%. The nomenclature of resulting composite was designated as PCG000 where last three numeric characters represent weight percentage of GO in PC multiplied by 100, e.g., PCG010 represents 0.10 wt.% of GO reinforcement in PC (Table 1). The solution was then dried on glass perti-dish at 70 °C for couple of hours to completely evaporate solvent.

In second step, Composite films were molded using injection molding machine shown in Fig. 1 [25–27]. Temperature of injection moulding machine was set at 290^0C and pressure settings were 6–10 psi. Composite films, obtained from solution blended method, were poured into the hopper of Injection moulding machine. After the conversion of solid-state film to molten state, molten material was pushed into the die with the pressure ram. Figure 2 shows actual digital image of Dog-bone samples.

Table 1 Table shows the results of different % of Graphene in compositions

S. no	Specimen name	GO wt.%age	Tensile strength (MPa)	Elastic modulus (GPa)	Tensile strength Ratio	Elastic modulus
1	PCG000	Pure	29.141	1.280	1.00	1.00
2	PCG005	0.05	26.762	1.072	0.92	0.84
3	PCG010	0.1	31.743	1.295	1.09	1.02
4	PCG020	0.2	45.694	1.440	1.57	1.13

Fig. 1 Digital photograph of Injection Moulding Machine

Figure 2a and b shows digital images of pure and composite samples, respectively. Figure 2c shows digital image of broken dog-bone sample after testing. All the procedure was repeated to prepare pristine PC sample using same procedure without mixing GO in it.

5 Results and Discussion

Solution mixing method for polymer composite synthesis is widely used due to improved dispersion of reinforcement in matrix [28, 29]. Solvent should be compatible for matrix and reinforcement [30]. THF was used as a solvent in present work as GO can be easily dispersed and PC can be dissolved in it. Regular color of synthesized

Fig. 2 Digital camera Images of Dog-bone shaped sample **a** Pure PC sample, **b** PC/Graphene (PCG) nanocomposites and **c** broken dog-bone sample after testing

specimen also points toward better dispersion of GO in PC matrix. Better dispersion enhances interaction of reinforcement and matrix to achieve higher mechanical properties.

Morphological studies of PCG nanocomposite samples were performed to understand reinforcement of GO in nanocomposite. Figure 3 PCG nanocomposite FE-SEM micrographs and presence of GO is marked by arrows. Shining spot in FE-SEM can be attributed to thin sheets of GO successfully embedded in PCG nanocomposite. Distribution of GO sheets has been fairly even too.
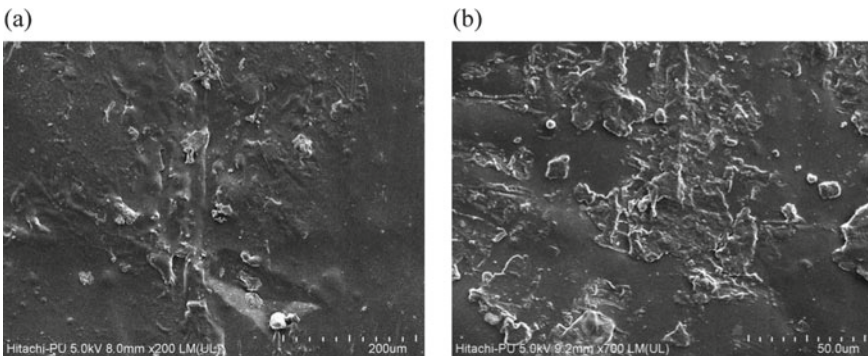

Fig. 3 FE-SEM micrograph of PC/G nanocomposite **a** Surface micrograph of PCG nanocomposite and **b** cross-sectional micrograph of PCG nanocomposite
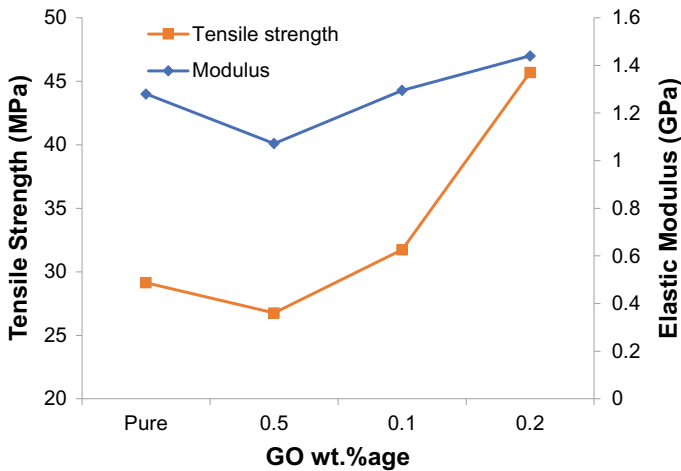
Fig. 4 Tensile strength and elastic modulus of different variations of graphene % in compositions

5.1 Tensile Strength

To characterize tensile behavior of PCG nanocomposite, a dog-bone sample was tested on micromechanical testing machine. Sample was prepared according to ASTM 638. Before testing samples were cleaned from burrs and extra materials. Surfaces of all samples were scratched with sand papers to achieve smooth finishing. Results of tensile testing are presented in Fig. 4 and Table 1.

Tensile strength of PCG upon addition of 0.05wt.% initially decreased. Decrease in tensile strength may be attributed to developments of few defects during synthesis as new materials phase was introduced. As GO concentration was further increased the tensile strength started to increase. Maximum tensile strength was achieved at 0.02 wt.% of GO in PCG020 sample. Comparing with tensile strength of pure sample PCG000 of 29.141 MPa, sample PCG020 showed tensile strength of 45.694 MPa that was 57% improvement from pure polycarbonate sample. A small reinforcement of 0.2 wt.% showed improved mechanical performance of PCG nanocomposite.

Elastic modulus characterization of PCG nanocomposites were also performed. A higher modulus represents lower strain while applying same load, i.e., better dimensional stability upon applying load. After an initial dip in modulus at 0.05 wt.% reinforcement of GO that may be attributed to small defects due to introduction of second phase modulus was increased in by further reinforcement. Modulus was increased from 1.280 GPa at pure PC to 1.440 GPa at PCG020 (GO 0.20 wt.%). It was a 13% improvement from pure PC. Once high strength GO reinforced at interface, GO gives strength to nanocomposite by increasing attachment matrix with high strength GO.

6 Conclusions

Following conclusions are drawn from the presented work:

- Solution blending was successfully used to synthesize Polycarbonate-GO (PCG) composite solution at room temperature for achieving superior mechanical properties.
- Casted thin sheets of PCG nano-composites were successful extruded Dog-bone shaped samples using Injection Molding Machine.
- Tensile strength and modulus of PCG nanocomposite at 0 wt%(pure), 0.5 wt%, 0.1 wt%, 0.2 wt% GO reinforcement were tested. Increase in tensile strength and elastic modulus of composite was observed with increase in concentration of GO in PC/Graphene composite.
- At 0.2 wt.% reinforcement of GO a rise in tensile strength and elastic modulus of 57% and 13% reported, respectively.

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