



Material properties

Dielectric, mechanical and electro-stimulus response properties studies of polyurethane dielectric elastomer modified by carbon nanotube-graphene nanosheet hybrid fillers



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ARTICLE INFO

Article history:

Received 2 July 2015

Accepted 6 August 2015

Available online 10 August 2015

Keywords:

Graphene

Carbon nanotube

Polyurethane

Electro-stimulus response

Artificial muscle

ABSTRACT

The typical nano-carbon materials, 1D fiber-like carbon nanotubes (CNTs) and 2D platelet-like graphene nanosheets (GRNs), that have attracted tremendous attention in the field of polymer nanocomposites due to their unprecedented properties, are used as conducting filler to induce a considerable improvement in the mechanical, thermal and electrical properties of the resulting graphene/polymer nanocomposites at very low loading contents. This study deals with the preparation and electro-stimulus response properties of polyurethane (PU) dielectric elastomer films with such 1D and 2D nanocarbon fillers embedded in the polymer matrix. The various forms of carbon used in composite preparation include CNT, GRN and CNT-GRN hybrid fillers. Results indicate that the dielectric, mechanical and electromechanical properties depend on the carbon filler type and the carbon filler weight fraction. Here, it has been also established that embedding CNT-GRN hybrid fillers into pristine polyurethane endows somewhat better dispersion of CNTs and GRNs as well as better interfacial adhesion between the carbon fillers and matrix, which results in an improvement in electric-induced strain. Therefore, the nanocomposites seem to be very attractive for microelectromechanical systems applications.

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1. Introduction

Dielectric elastomers can generate large-strain actuation under the application of a high electric field, or vice versa, to convert mechanical energy into electricity [1]. Dielectric elastomers are one of the most promising classes of flexible actuator materials that exhibit excellent performance such as large active strain, high energy density, fast response, high electromechanical coupling efficiency, reliability, durability, as well as easiness of processing [2,3]. This field is growing rapidly, as measured by the number of research papers, performance of the technology and diversity of potential applications, including micropumps, energy harvesters, space robotics, positioners and bioinspired robotic systems [4–8]. However, a key limitation for the practical application of dielectric elastomer actuators is the high electric field (>100 MV/m) to drive them [9]. Such a high electric field is very dangerous for humans and equipment, particularly in biological and medical fields, which largely limits its wide application. Therefore, the preparation of

dielectric elastomer actuators with high actuated strain at a low electric field is the biggest challenge.

According to the well-known Maxwell equation, to obtain a dielectric elastomer with high actuated strain at a low electric field, a high electromechanical sensitivity (β) is required, which is defined as the ratio of the dielectric constant (ϵ) to the elastic modulus (Y) ($\beta = \epsilon/Y$) [1]. Therefore, an effective solution is to largely increase ϵ and largely decrease Y of the dielectric elastomer [10]. Most dielectric elastomers, such as silicones, acrylics and polyurethanes, have quite a low ϵ (<10). Composite methods, in which nanofillers are added to a polymer matrix, have been carried out to fabricate high ϵ polymeric materials. These nanofillers may be mainly divided into four categories: (1) high ϵ ceramic nanofillers (e.g., BaTiO₃, PbZrTiO₃) [11–15]; (2) organic dipoles (e.g., N-allyl-N-methyl-p-nitroaniline) [16–20]; (3) high ϵ organic semiconductor (e.g., CuPc) [21–23]; and (4) conductive nano-carbon fillers (e.g., CNT, GRN) [24–27]. Approach (1) usually requires high loading fractions (>50%) to effectively improve ϵ , and thus produces the large increase in elastic modulus. Hence, the resulting composites usually suffer from a loss of flexibility and processability. Approach (2) can achieve high uniformity of distribution of

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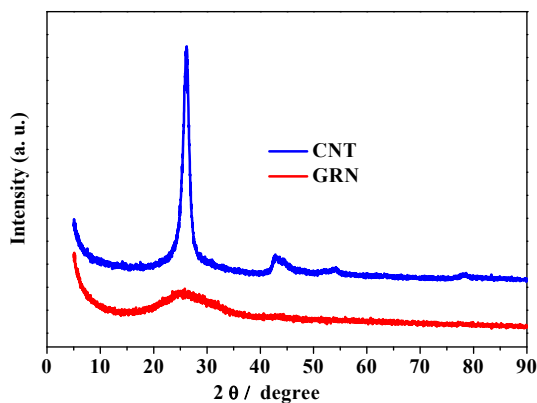


Fig. 1. XRD patterns of CNT and GRN.

the high dielectric moieties, increased dielectric constant and decreased mechanical stiffness. However, the polar group significantly reduces the polymers' dielectric strength and increases sensitivity to moisture. Approach (3) provides an ultra-high ϵ filler ($>10^5$). However, the molecular stacking of CuPc macrocycles allows intermolecular interaction, which results in a large electrical conductivity so that the oligomer suffers from a high dielectric loss. Molecular grafting of CuPc to the dielectric elastomer macromolecular network could reduce the size of CuPc, improve the dispersibility and decrease the dielectric loss. Nevertheless, the grafting procedure is complex. Approach (4) can achieve a high dielectric constant at a very low loading fraction ($<5\%$). The main drawback is the stacking and agglomeration of the carbon nanoparticles tends to form conductive pathways, and thus diminish the dielectric strength.

Multifunctional polymer composites employing carbon fillers have attracted widespread attention [28–36]. The typical nano-carbon materials, 1D CNT and 2D GRN, which have attracted tremendous attention in the field of polymer nanocomposites due to their unprecedented properties, are used as conducting filler to induce a considerable improvement in the mechanical, thermal and electrical properties of the resulting graphene/polymer nanocomposites at very low loading contents. However, in many cases, the potential applications of CNT and GRN are limited because CNTs easily entangle and agglomerate due to their size and large aspect ratio, while GRNs also tend to restack due to their large van der Waals forces and strong π - π interactions. Herein, one challenging

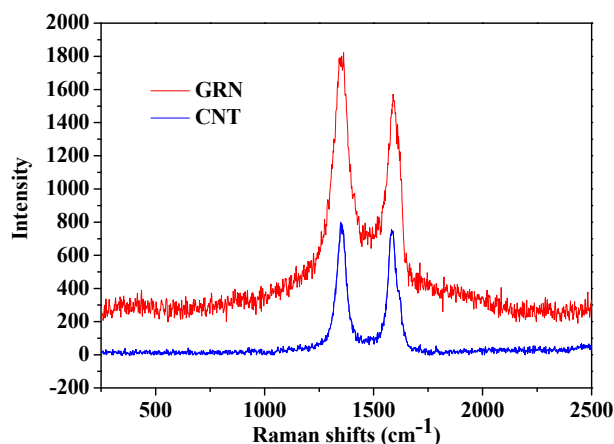


Fig. 2. Raman spectra of CNT and GRN.

task to successfully explore the structural composites lies in achieving homogeneous dispersion of CNTs and GRNs in the polymer matrix. In the present study, in order to fully harness the exceptional properties of CNT and GRN conducting nanofillers, the dielectric elastomers are filled with both CNT and GRN to form the novel CNT-GRN@PU hybrid structures. Furthermore, we present here the effect of the novel CNT-GRN@PU hybrid structure on the electromechanical performance of the polymer composite film. To the best of our knowledge, it is the first time that the electromechanical actuation performances of such novel CNT-GRN@PU intelligent composites have been investigated in detail.

2. Experimental

2.1. Materials

Polyurethane elastomer (A polyether-type thermoplastic polyurethane elastomer TPU58887, Estane) was used as the matrix material. Multi-walled carbon nanotubes (MWCNTs) with length of 5–10 μm were synthesized using CVD. Graphene oxide (GO) was prepared from natural graphite powder by the modified Hummers method. The graphene nanosheets (GRNs) were prepared by reduction of GO with mixed gas of hydrogen and argon at 450 $^{\circ}\text{C}$ for 2 h.

2.2. Preparation of CNT-GRN@PU composites

Polyurethane elastomer films were prepared using a solution casting method. PU granules were completely dissolved in N,N-Dimethylformamide (DMF) at around 75 $^{\circ}\text{C}$. Subsequently, CNT, GRN, and CNT-GRN nanofillers were added to the solution under magnetic stirring and then ultrasonically stirred for at least 2 h. The carbon weight percentage was limited to below 2.5%. The mixed solution was then poured onto a Petri dish and dried at 60 $^{\circ}\text{C}$ in air for 12 h, then in vacuum at 60 $^{\circ}\text{C}$ for 12 h to remove DMF. The films were removed from the Petri dish and then dried again at 130 $^{\circ}\text{C}$ for 3 h. Samples without any additives, denoted Pure PU, were also prepared for comparison.

2.3. Characterization

X-ray diffraction (XRD) patterns were recorded by a Bruker D8 Advance diffractometer. Raman spectra were obtained with a French JY HR800 microscopic Raman system. Scanning electron microscope (SEM) images were taken on a JSM-5610LV scanning electron microscope. Transmission electron microscope (TEM) images were taken on a FEI Tecnai G2 transmission electron microscope. Atomic force microscope (AFM) images were taken on a Shimadzu SPM-9500J3 atomic force microscope with the non-contact mode. Dielectric properties were measured by a HP4294A impedance analyzer. The tensile properties of films were measured in general accordance with ISO 527 using a universal testing machine (CM5105) at room temperature with an elongation rate of 10 mm min^{-1} . The electric field induced strain measurement was measured using a MTI Fotonic Sensor (MTI-2100), as described elsewhere [28]. The “electric induced strain” is defined as $S = d/t$, where d is the displacement measured at the center of the diaphragm and t is the sample thickness.

3. Results and discussion

3.1. Microstructure of nanofillers

3.1.1. XRD characterization

XRD patterns of CNT and GRN are shown in Fig. 1. Two distinct

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