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# The synergistic effect of the combined thin multi-walled carbon nanotubes and reduced graphene oxides on photothermally actuated shape memory polyurethane composites



Dong Hun Yi<sup>a</sup>, Hye Jin Yoo<sup>a</sup>, Sibdas Singha Mahapatra<sup>a</sup>, Yoong Ahm Kim<sup>b,\*</sup>, Jae Whan Cho<sup>a,\*</sup>

<sup>a</sup>Department of Organic and Nano System Engineering, Konkuk University, Seoul 143-701, Republic of Korea

<sup>b</sup>School of Polymer Science and Engineering, Chonnam National University, Gwangju 500-757, Republic of Korea

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## ABSTRACT

We evaluated the synergistic effect of the hybrid-type nanocarbon, consisting of 1D thin-walled carbon nanotubes (TWNTs) and 2D reduced graphene oxide (RGO), on the shape memory performance of hyperbranched polyurethane composites. The shape recovery of the resulting composites was activated via a photothermal process using a near-infrared laser. The best laser-induced shape recovery performance was achieved for the composites with a 7/3 of TWNT/RGO ratio and a 1 wt.% of nanocarbon content. Such result can be explained by good dispersion of TWNTs and RGO in the hyperbranched polymer as well as three-dimensionally enhanced interconnection between carbon nanotubes and graphenes. The optically active TWNTs with a high optical absorption section exhibited high ability of transferring laser-induced thermal energy to polymer matrix whereas RGO provided a high mechanical property to polymer matrix. The tensile modulus and electrical conductivity of the composites also showed a similar dependence on the TWNT/RGO composition ratio as the photothermal shape recovery. Our study demonstrated an effective conversion from light, thermal to mechanical work by irradiating shape memory polymer composite containing hybrid-type fillers using a near-infrared laser.

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## 1. Introduction

Nanocarbon materials consisting of carbon nanotubes (CNTs) and graphene have been considered an ideal nanomaterial for reinforcement and functionalization of polymers due to their outstanding mechanical, electrical, thermal, and optical properties. Although many results have been reported on the mechanical and electrical properties of polymer/nanocarbon composites [1–4], the photothermal properties of nanocarbon-based polymer composites have received much attention in recent years for applications of photothermal imaging, photothermal therapy, drug delivery, photoactuators, etc. [5–8]. Nanocarbon materials have particularly good advantages in terms of efficient photothermal energy conversion and strong absorbance over a wide wavelength range as compared to metal nanoparticles [5,9,10]. Particularly, the small-sized carbon nanotubes are in resonance with the near infrared (NIR) laser line and thus exhibit a high capacity (e.g., optical absorption section) of converting the absorbed light to heat energy [11]. Recently, the photothermal properties of nanocarbon materials have been

applied to the development of polymer actuators. Miyako et al. [12] reported laser-triggered remote heating using the photothermal properties of CNT-containing composites. Panchapakesan et al. [13] developed graphene/elastomer composite-based photothermal nanopositioners that showed efficient light absorption by graphene nanoplatelets and subsequent energy transduction to the polymeric chains. The infrared-actuated recovery of reduced graphene oxide/multi-walled carbon nanotubes (MWNTs) polyurethane composites was also demonstrated by Feng [14]. Lee et al. [15] fabricated a light-controlled graphene composite hydrogel actuator in order to demonstrate rapid and tunable motion due to the photothermal effect of the composite upon irradiation with near-infrared light.

On the other hand, various nanocarbon functionalization methods including covalent and noncovalent modifications and the choice of suitable nanocarbon fillers have been investigated to improve the properties of polymer composites [16,17]. The use of highly branched polymer structures as a polymer matrix has been investigated as an alternative method for enhancing dispersion of nanocarbon particles with no surface modification. These hyperbranched polymers provide numerous functional groups on their outer surface and in interior cavities, low solution and melt viscosity,

\* Corresponding authors.

E-mail addresses: yak@jnu.ac.kr (Y.A. Kim), jwcho@konkuk.ac.kr (J.W. Cho).

and good solubility [18–21]. In addition, combinations of various types of nanocarbon fillers such as single-walled carbon nanotubes (SWNTs), thin-walled carbon nanotubes (TWNTs), MWNTs, graphene, graphene oxide (GO), graphite nanoplatelets, and reduced graphene oxide (RGO) can also significantly affect the mechanical and electrical properties of the polymer composites. Examples of such polymer composites include polypropylene/RGO/MWNTs [22], polyaniline/graphene nanosheet/CNTs [23], poly(vinyl alcohol)/MWNT/GO [24], and poly(vinyl alcohol)/nanodiamond/SWNT/graphene [25].

In this study, the photothermal shape recovery actuation behaviors of nanocarbon hybrid-based hyperbranched polyurethane (HBPU) composites resulting from irradiation by a near-infrared (NIR) laser are investigated as a function of TWNT/RGO composition ratio and nanocarbon content. The use of TWNTs among various kinds of CNTs may be effective as a bridging agent between one-dimensional nanotubes and two-dimensional graphene sheets due to the fact that they have better flexibility and a smaller diameter than MWNTs and exhibit better debundling than SWNTs [26]. With regarding to the photothermal effect, the carbon nanotubes act as energy converter from light to thermal energy, whereas graphene offer high mechanical strength. Particularly the small diameter inner tubes of TWNTs show higher ability of converting the absorbed light to heat energy, compared to MWNTs [11]. In addition, the use of the hyperbranched polymer may be also very effective for nanocarbon dispersion in the polymer matrix [27,28].

## 2. Experimental

### 2.1. Materials

TWNTs with diameters and lengths of approximately 4–6 nm and 10–20  $\mu\text{m}$ , respectively, and GO were purchased from Hanwha Nanotech, Korea, and Nanoinova Tech., Spain, respectively. Poly( $\epsilon$ -caprolactone)diol (PCL,  $M_w = 3000$  g/mole) was received from Solvay Co., UK, and 4,4'-methylenebis (MDI) and tris(hydroxymethyl)-aminomethane (THA) were received from Sigma Aldrich Co. 1,4-Butanediol (BD) was obtained from Junsei Chemical, Japan. N,N-dimethylformamide (DMF) (Duksan, Korea) was used after purification by the conventional technique and stored with 4  $\text{\AA}$ -type molecular sieves.

### 2.2. Synthesis of hyperbranched polyurethane

In a 300 mL three-neck reaction vessel equipped with a mechanical stirrer and nitrogen inlet, 6 g of PCL was dissolved in 15 mL of dry DMF, and 2 g of MDI in 10 mL of DMF solution were injected into the vessel at room temperature. The reaction temperature was increased to 70  $^{\circ}\text{C}$  and the reaction was allowed to proceed for 3 h. After completion of the prepolymer synthesis, the system was cooled to 0  $^{\circ}\text{C}$ , and 0.18 g of THA in 15 mL of DMF and 0.27 g of BD in 20 mL of DMF were added to it. The reaction temperature was then increased to 60  $^{\circ}\text{C}$  for 3 h. After completion of the reaction, the product was dried in a vacuum oven at 50  $^{\circ}\text{C}$  for 48 h to obtain the hyperbranched polyurethane (HBPU).

### 2.3. Synthesis of reduced graphene oxide

RGO was prepared from GO by using hydriodic acid with acetic acid [29]. 1.3 g of GO was dispersed in 500 mL of acetic acid and then sonicated using a bath-type sonicator for 10 min. After sonication, 26 mL of HI was added and the mixture was stirred at 40  $^{\circ}\text{C}$  for 40 h. The reaction mixture was washed with saturated sodium bicarbonate, distilled water, and acetone with filtering.

The product was then dried overnight in a vacuum oven at room temperature to obtain the RGO.

### 2.4. Preparation of TWNT/RGO/HBPU composites

The TWNT/RGO/HBPU composites films were prepared by a solvent-casting method. First, TWNTs and RGO were dispersed in

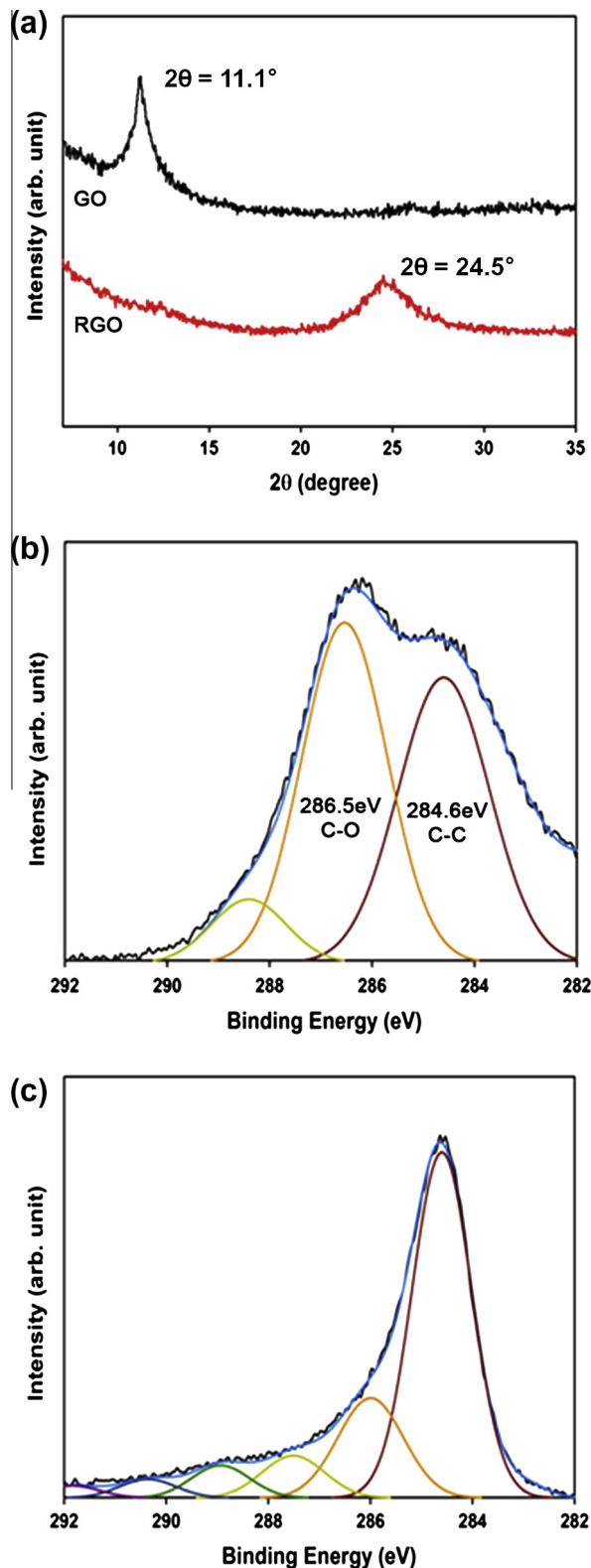


Fig. 1. (a) X-ray diffraction and XPS spectra of (b) GO and (c) RGO.

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