



# Light induced reversible and irreversible mechanical responses in nanotube-polymer composites



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## ABSTRACT

It is shown that visible light irradiation of carbon nanotube (CNT) dispersed polymers may yield both reversible and irreversible changes in measured mechanical stress. While light irradiation of PDMS (polydimethylsiloxane)–CNT composite yields a change of stress level at a fixed strain, which reverts to the original value after the incident light is removed, the RET (reactive ethylene terpolymer)–CNT composite exhibits such reversible characteristics as well as an additional irreversible stress-relaxation response when the composite is first irradiated by light. A microscopic model is proposed to explain the mechanism of the dual photomechanical responses of the RET–CNT composites and quantitative predictions are compared with experimental measurements.

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## 1. Introduction

Photo-responsive (PR) materials have been extensively explored for various engineering applications, from the nano- to the macro-scale [1,2], e.g., in photo-switchable ion transport [3], photon-propelled robotics [4] and photo-responsive actuators [1,4]. In comparison to other actuation mechanisms, PR material constituted devices have the advantages of not requiring physical contact to a powering source, which enable structure compactness and related weight reduction [2]. Moreover, various light irradiation parameters (intensity, frequency, polarization, etc.) [4,5], can be used for external control.

PR material has been synthesized mostly through assembling specific molecules into host materials [6–8], where upon light irradiation, configurational change related to such molecules can cause macroscopic mechanical responses [9]. For example, azobenzene molecules incorporated in photo-responsive composite yield a mechanical response due to a reversible *cis*–*trans* transformation driven by light irradiation [10–12]. To maximize the

photo-response in PR materials, polymer composed of photoactive units [13,14], such as covalently linked photoisomerizable azobenzene moieties [15] incorporated into copolymers [16,17]. Recent studies have found that composites formed through dispersing carbon nanotubes (CNTs) [4,18–20], boron nitride [19] or molybdenum disulfide [21] into polymer matrices may also behave as PR materials. Various light-induced deformation modes including bending [22], contraction [23] and expansion [5] have been shown in CNT-infiltrated polymeric composites. Such responses have been considered inherent to the composite, and were not observed in either the native polymer or CNTs [24,25]. For instance, it has been reported that multiwalled carbon nanotube (MWCNT) dispersed polydimethylsiloxane (PDMS) composites [26] can expand at small pre-strain while contracting at large pre-strain when exposed to light irradiation with wavelength  $\sim 675$  nm [18,27]. Such effects have been attributed to stretch-induced alignment of embedded CNTs and the kink-instability upon photon absorption [18].

In this paper, we study the photo-response of the MWCNT dispersed reactive ethylene terpolymer (RET) composites. RET is a commercially available thermoplastic elastomer, which has good adhesive properties to metals and is widely adopted as coatings that protect metal structure from corrosive environments and mitigate cathodic disbondments [28,29]. The elastomeric characteristics and corrosion resistance are also critical to the utility of

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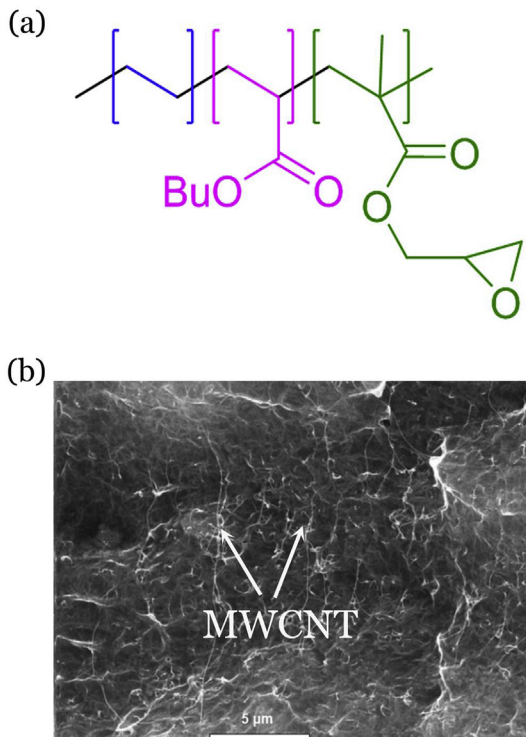
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RET as a hot-melt adhesive and coating [30] and the constituent epoxide group enables effective anchoring with functional groups (e.g., -OH, -COOH, -NH<sub>2</sub> etc.) on the CNTs [31,32].

In the stretched RET-MWCNT composite, in addition to photo-mechanical stress modulation similar to that previously observed in PDMS-MWCNT composites [18,27,33], an irreversible and significant stress relaxation was observed under initial light irradiation. We propose a microscopic model to explain such phenomena, by invoking the transformation of crystalline to amorphous domains due to light irradiation. Our results and analyses would be useful for a better understanding of PR composites and pave the way for their application.

## 2. Experiments

The studied composite was synthesized from a RET polymeric matrix with dispersed MWCNTs. The embedded MWCNTs (obtained from Sigma-Aldrich Inc.) have an average diameter of 140 nm, with the length ranging from 5 to 9  $\mu\text{m}$  and density 2.1 g/cm<sup>3</sup>. RET (DuPont™, Elvaloy® 4170, density 0.94 g/cm<sup>3</sup>) is constituted from epoxide, butyl and ethylene groups (Fig. 1a). The epoxide group has a high reactivity [30] and is amenable [34] for effective anchoring of constituent ring bonds with functional groups on the CNTs [35,36]. The butyl groups (*soft segments*) provide high extensibility in the polymer at ambient temperature; while the ethylene groups (*hard segments*) induce formation of crystalline domains [37,38], and serve as the physical cross-linking [39] in the RET matrix. Such molecular structure characteristics make RET behave like an elastomer at room temperature and as a polymer melt at higher temperature. Therefore, RET may be regarded as thermoplastic elastomer (TPE) [9,40].



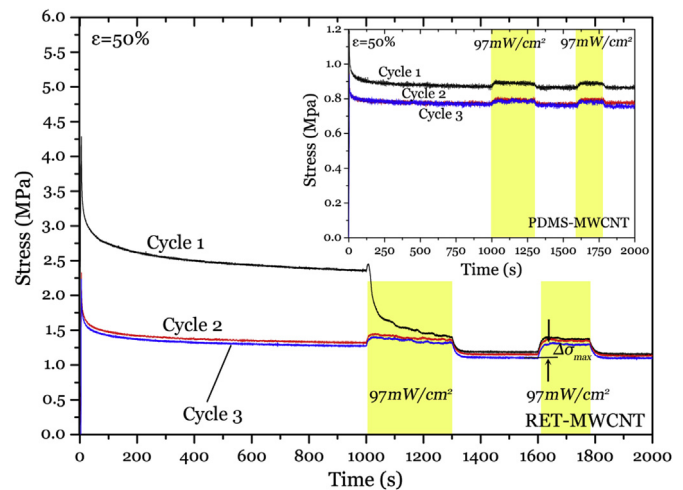
**Fig. 1.** (a) Chemical structure of the RET (Elvaloy® 4170) with three functional groups: the ethylene group (blue), the butyl group (purple) and the epoxide group (green). (b) Scanning electron microscopy (SEM) image of the RET-MWCNT composite. The sample is for a 7 wt% MWCNT composite, with initially non-aligned MWCNTs distributed among the RET matrix. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

To prepare the composites, pellets of RET were first dissolved in toluene which was heated to  $\sim 60^\circ\text{C}$  for 2 h. The CNTs were also dispersed in toluene by using a sonicator (VCX-750, Sonic Materials Inc., 750 W, 42 kHz) for 10 min. The CNT-toluene dispersion was then incorporated with the RET solution and sonicated again for 1 h. The mixture was stirred for another 2 h before being placed in a vacuum oven (under pressure at 0.866 atm) at  $60^\circ\text{C}$  for 10 h. A scanning electron microscopy (SEM) image of a RET-MWCNT composite is shown in Fig. 1b, indicating non-aligned MWCNTs distributed among the RET matrix. In this paper we report on a representative 7 wt% CNT constituted RET composite. Such a weight fraction was above the percolation threshold of CNTs (of  $\sim 2.3$  wt% for the given MWCNT length and diameter) [35,41] and consequently MWCNTs form a connected network in the polymer matrix [42].

Stress-strain diagrams of RET composite were measured (5965 Dual Column Testing systems, Instron). The RET-MWCNT composite was cut into samples of dimensions, 25 mm  $\times$  5 mm  $\times$  0.3 mm. The samples were firmly clamped by the grippers and subjected to a uniaxial tensile stress. The accuracy of the strain measured in the sample was within  $\pm 0.1\%$ . While the composites were stretched, they were exposed to irradiation (of area  $\sim 100$  mm<sup>2</sup>) from a light source (CREE XM-L2 LED, maximum intensity at  $\sim 440$  nm, 10 mW/cm<sup>2</sup> at 1 m distance) positioned at a distance of  $0.15 \pm 0.01$  m away from the surface of the sample, and the light intensity (measured by Spectra-Physics 407A) was modulated through neutral density filters (Thor Labs Inc.) in the range of 0–97 mW/cm<sup>2</sup>. The latter limit of irradiance is close to the standard of solar irradiance ( $\sim 1$  sun) of  $\sim 100$  mW/cm<sup>2</sup>.

## 3. Results and discussion

Fig. 2 indicates the photomechanical response characteristics of stretched RET-MWCNT composite under light irradiation. The composite was first stretched and held at a constant strain: 50% ( $\epsilon = 0.5$ ) for 1000 s, to enable a quasi-equilibrium state of the sample before light irradiation. Subsequently, two periods of light irradiation (marked in yellow in Fig. 2) were provided under a



**Fig. 2.** A single RET-MWCNT composite (7 wt%) under a 50% pre-strain and light irradiation of intensity 97 mW/cm<sup>2</sup>. Three cycles of test were performed, each included a mechanical equilibration step, two periods of light irradiation and a stage after removing external constraints. The RET-MWCNT composite showed a dramatic decrease in stress under the first light irradiation period in Cycle 1, with repeatable stress modulations for the subsequent light irradiations. The inset indicates the stress response in PDMS-MWCNT composite (7 wt%) under the same test procedures, showing only repeatable stress modulations for all light irradiation periods. The maximum stress responses under light irradiation were measured as  $\Delta\sigma_{\text{max}}$ .

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