AC ELECTRIC FIELD ACTIVATED SHAPE MEMORY POLYMER COMPOSITE

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Introduction

Shape memory materials have drawn interest for applications like intelligent medical devices, deployable space structures and morphing structures.[1] Compared to other shape memory materials like shape memory alloys (SMAs) or shape memory ceramics (SMCs), shape memory polymers (SMPs) have high elastic deformation that is amenable to tailored of mechanical properties, have lower density, and are easily processed.[2] However, SMPs have low recovery stress and long response times. A new shape memory thermosetting polymer nanocomposite (LaRC-SMPC) was synthesized with conductive fillers to enhance its thermo-mechanical characteristics.

Experimental

Materials. Bisphenol A diglycidyl ether was purchased from Aldrich Chemical Co. and was used without further purification. Functionalized graphene sheets (FGS) were obtained from Vorbeck Materials Co.

Instrumentation. Differential scanning calorimetry (DSC, Setaram DSC 131, France) was used to determine the glass transition temperature at a heating rate of 10°C/min. Mechanical properties at room temperature and temperatures above the glass transition temperature were measured using a microtester (Instron 5848) with a standard temperature chamber (C9100606). The AC conductivity and the dielectric constant of LaRC-SMPC film were measured as a function of frequency with a Novocontrol-Solartron 1260 impedance/gain-phase analyzer. The electric-field-activated shape memory effect was controlled using a Hewlett Packard 33120A function generator and a Trek 50/750 HV power supply.

Synthesis of shape memory polymer nanocomposite. Epoxy based thermosetting shape memory polymer resin was synthesized with bisphenol A diglycidyl ether as a resin and aromatic amines as the curing agent. The precursors were mechanically premixed and a predetermined amount of FGS was added to the resin. The premixed resin was placed in a mixing apparatus consisting of a temperature controlled sonication bath and a mechanical stirrer. The resin was mixed under high shear at 60°C and 25kHz sonication for 2 hours. After mixing, the composite resin was poured into a Teflon mold and cured in a convection oven at 125°C for 4 hours, 150°C for 4 hours and 175°C for 2 hours.

Results and Discussion

Figure 1 shows well dispersed functionalized graphene sheets (FGSs) in the shape memory polymer resin.
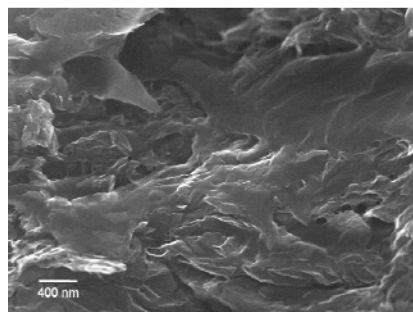

Figure 1. Cryogenically fractured cross-section SEM image of LaRC shape memory polymer composite (LaRC-SMPC).

Figure 2 shows thermo-mechanical properties of pristine LaRC-SMP. Tensile tests were performed at room temperature and at elevated temperature (T_g + 30ºC), respectively. Pristine LaRC-SMP exhibits a hard and rigid stress-strain characteristic with an elastic modulus of 2.8 GPa and an ultimate tensile strain of about 6.3% at room temperature. It exhibits a soft and elastic stress-strain characteristic with an elastic modulus of only 7.5 MPa and an ultimate tensile strain at break of higher than 38% (**Figure 2**) above the glass transition temperature (T_g (96 ºC) + 30ºC). The drastic change in elastic modulus of LaRC-SMP with temperature is a critical parameter for making a variable stiffness material. The LaRC-SMPC behaves like pristine LaRC-SMP except for a slight decrease in modulus (**Figure 3**), at room temperature and above the glass transition temperature (T_g + 30ºC).
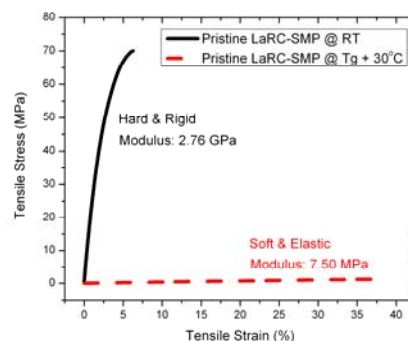

Figure 2. Mechanical properties measured at room temperature and at elevated temperature (30°C above the glass transition temperature (96ºC)) of pristine LaRC shape memory polymer (LaRC-SMP)
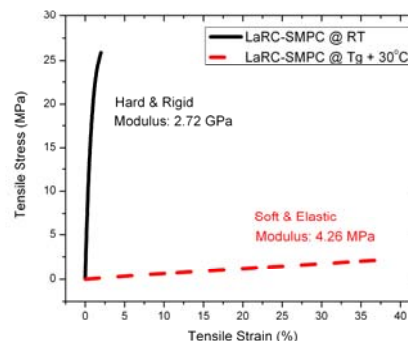

Figure 3. Mechanical properties measured at room temperature and at elevated temperature (30°C above the glass transition temperature (95 ºC)) of LaRC shape memory polymer composite (LaRC-SMPC).

The real AC conductivity of LaRC-SMPC is shown in **Figure 4**. Pristine LaRC-SMP had lower AC conductivity like that observed for a conventional insulator, whereas conductive FGSs doped LaRC-SMPC exhibited higher AC conductivity and resembled a conventional conductor's characteristics as a function of increasing frequency.

In order to trigger shape reformation, the temperature of the material must be increased through its glass transition temperature. In this study, electric power input was employed to warm up the LaRC-SMPC to induce a faster response time than that afforded by surface conduction or radiation. The temperature of the LaRC-SMPC was monitored with an IR camera while applying DC voltage or AC voltage in the frequency range from 0.1 Hz to 1 MHz (**Figure 5**). The LaRC-SMPC exhibited a faster increase in temperature under applied AC voltage below 1 kHz than under DC voltage or AC voltage over 1 kHz. This indicates that the synergistic effect of combining loss heating (generated by dielectric losses under applied AC electric field) and Joule heating (generated by electrical currents) can boost the triggering of shape reformation.
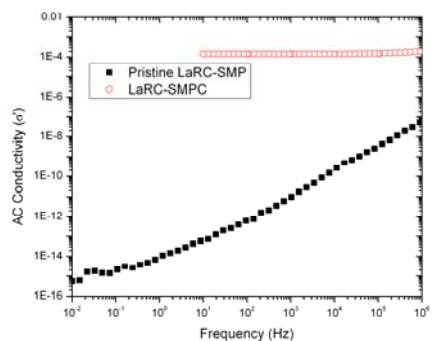
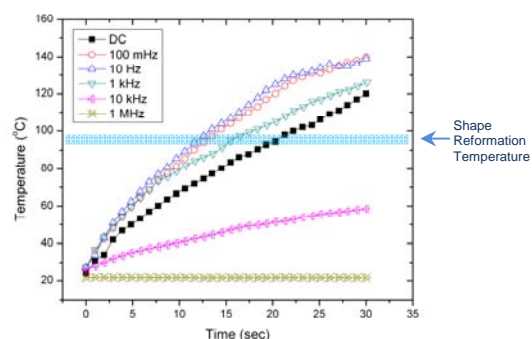**Figure 4.** AC conductivity of pristine LaRC-SMP and LaRC-SMPC.

Figure 5. Temperature change of LaRC-SMPC under applied DC and AC electric field.

Figure 6 presents a visual demonstration of the shape memory effect achieved by LaRC-SMP and LaRC-SMPC. The programmed temporary shape of pristine LaRC-SMP (**Figure 6 (a)**) was reformed to the original permanent shape by external thermal heating (**Figure 6 (b)**). The programmed temporary shape of the LaRC-SMPC (**Figure 6 (c)**) recovered its original permanent shape when an external electric field was applied (**Figure 6 (d)**).
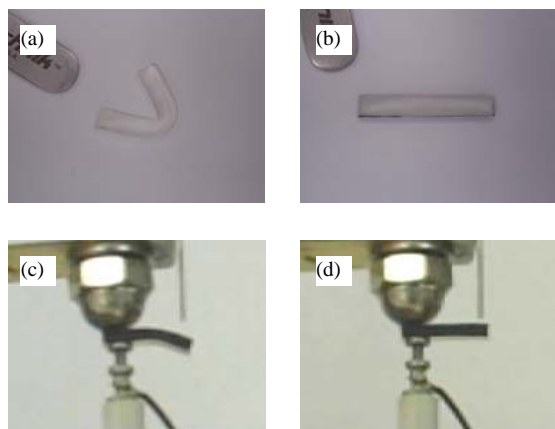

Figure 6. Shape memory effect: (a) programmed temporary shape of pristine LaRC SMP and (b) original shape of pristine LaRC SMP recovered by external thermal radiation; (c) programmed temporary shape of LaRC-SMPC and (d) original shape of LaRC-SMPC recovered by external electric field.

Conclusions

A new composition of shape memory thermosetting polymer nanocomposite (LaRC-SMPC) was synthesized with conductive functionalized graphene sheets (FGS) to enhance its thermo-mechanical characteristics. The elastic modulus of LaRC-SMPC is approximately 2.7 GPa at room temperature and 4.3 MPa above its glass transition temperature. Conductive FGSs-doped LaRC-SMPC exhibited higher conductivity compared to pristine LaRC SMP. Applying an electric field at between 0.1 Hz and 1 kHz induced faster heating to activate the LaRC-SMPC's shape memory effect relative to applying DC electric field or AC electric field at frequencies exceeding1 kHz.

References
(1) Mather, P. T. *Annu. Rev. Mater. Res.* **2009**, 39, 445.
(2) Otsuka, K.; Wayman, C.M. ed., *Shape Memory Polymers*, Cambridge University Press, Cambridge, 1998.