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Material behaviour

Characterization of deformation induced changes to conductivity in an electrically triggered shape memory polymer

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ABSTRACT

Transforming a shape memory polymer (SMP) based part into a new configuration is typically achieved by increasing the temperature of the material to just above the glass transition temperature ($T_{\rm g}$). This paper describes the preparation and testing of an electrically conductive SMP based composite which can be heated beyond T_{g} through the passage of an electric current at low power levels and with good temperature-time response characteristics. Conductivity was achieved by impregnating the resin with conductive carbon black (CB) using two dispersion techniques. Samples were subjected to tensile deformation tests to assess the effects of strain on the carbon black based conductive networks within the matrix. A series of these tests were performed on each sample to further assess recovery behavior from repeated loading cycles and any ensuing effect on electrical properties. The SMP showed similar strain recovery in repeated loading cycles. The electrical nature of the filled materials, as measured by the resistivity, was found to change, registering an increase and/or decrease, in response to the magnitude of the strain. Three distinct regions could be identified in the resistivity versus strain plots which are explained vis-à-vis filler chain interaction and rearrangement. The data endorses the technique of using electrical actuation as a means of triggering SMPs which can then be an enabling technology in myriad applications in the aerospace, automotive and biomedical fields. Furthermore, the resistivity change data can be used in the design process to predict the response of components manufactured using reconfigurable composites.

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1. Introduction

Shape memory polymers (SMPs) have been the subject of considerable research in the past decade and the impetus has stemmed partly from their low cost and high strain recovery [1,2,3] behavior which makes them ideal candidates for applications as varied as biomedical devices [4–6], satellites [7,8] and morphing wing structures [9]. However, as simple as it is to demonstrate shape recovery behavior in the laboratory, the ability to consistently trigger strain recovery behavior in a way that would be practical for most applications is proving challenging. While direct heating is still the most common method for triggering shape recovery [10], and may well be the preferred technique in some circumstances, such as in the case presented by Ahmed M, et al. [11] where the body temperature of a patient is used to trigger shape recovery of an SMP implant, it is often unfeasible as an efficient and portable triggering system due to size, weight, cost and complexity. External heating can preclude the possibility of capitalizing on the advantages SMPs offer over other actuation methods, such as shape memory alloys [1]. This predicament has spurred research into alternative triggering mechanisms such as ultra-violet light [12], solar [13] and





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infrared irradiation [14], magnetic fields [15], changes in the chemical environment [14,16,17] and electrical current [14,18–25].

The two main advantages of electrically triggered SMPs are that, i) no external apparatus is needed to envelop the polymer, such as an oven, light source, induction coils or some sort of fluid, over its entire operational range, and ii) good response times and minimal temperature gradients are achieved through homogeneous (through-thickness) heating. Unlike many other triggering methods, an electrically activated SMP can be installed within a larger application and controlled precisely. Since polymers, including SMPs, are typically good electrical insulators, electrical triggering is contingent upon having sufficient conductivity to achieve high levels of resistive heating through the passage of an electrical current. The first task, therefore, becomes the attainment of sufficient electrical conductivity without adversely influencing the desirable set of shape recovery characteristics.

There are several different strategies that can be employed to create a conductive SMP, and they overwhelmingly employ the addition of conductive fillers prior to curing. The goal is establish a sufficiently large number of conductive pathways throughout the material. Notably, some attempts have been made to manually form these conductive chains with the application of a magnetic field during curing [18–20]. For example, Leng et al. [20] were able to show that magnetic aligning of Ni powder produced significantly lower resistivity SMP samples compared to those prepared with a random distribution of Ni particles Other attempts have been aimed at linking agglomerations of conductive particles by experimenting with combinations of conductive carbon black (CB) [21-25], carbon nanotubes (CNTs) [23] and carbon nanofibers [25] to bridge small distances and, conversely, short carbon fibers (SCFs) [24] to bridge longer distances. In general, the research shows that higher loading fractions of conductive particles yield lower resistivity in the composite [18–25]. However, there is a very practical trade-off in that the processing of the polymer often becomes more difficult as filler content is increased. This can be particularly problematic because, if the conductive material is not adequately dispersed, localized heating of the cured SMP can not only cause nonuniform recovery but in extreme cases can lead to localized melting and even present a fire hazard. Additionally, as is the case in most composite materials, the SMP's mechanical properties are typically altered by the addition of the filler [26-29]. Collectively, these issues have been the primary obstacles towards the development of conductive shape memory composites that have a good set of electrical and mechanical properties.

One of the critical considerations in the adoption of SMPs in the development of reconfigurable structures, which could be an aircraft's flight surface, wind deflector aiding heat management in an automobile, or a therapy boot used in pediatric care, is the ability of the material to be deformed predictably. This functional requirement translates into accurate knowledge of the required input energy, response time and repeatability of the deformation cycle. While filler based electrical actuation of SMPs has been widely reported in the literature, data reporting the

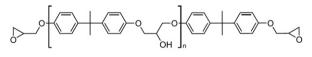
ensuing changes in the electrical characteristics in response to deformation is lacking. This research has addressed the question of how microstructural changes occurring as a result of bulk tensile deformation manifest themselves in the electrical properties of the material. The exploration of this topic has been presented with a detailed account of the process of preparing a conductive composite. This is followed by a description of the experimental protocol adopted to measure the zero strain resistivity, constant power temperature response profile, and resistivity versus strain data. The paper concludes with a discussion of the filler-chain interaction to which the deformation-resistivity characteristics can be attributed.

2. Method

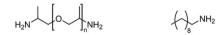
2.1. SMP preparation

The ability to achieve electrical conductivity in a SMP through the addition of a filler ideally requires access to an uncured resin. SMP availability in pellet form typically precludes the possibility of economically infusing the material with the filler in small batches because an injection molding machine must be used. While resin-hardener type systems such Veriflex-E, (Cornerstone Research Group, USA) and Diaplex (SMP Technologies, Japan) were acquired at the start of this study, they were abandoned due to lack of continued availability of the former, and short potting life (<5 mins.) of the latter. The predicament was overcome by preparing an SMP in-house using EPON resin 826 (Momentive Inc.), Jeffamine D 230 hardener (Huntsman Chemicals), and 1-Aminodecane catalyst (TCI Chemicals), with their chemical structures shown in Fig. 1. The mix ratios used were 6.296:1:1.365 by mass, see [30].

Conductive carbon black from three manufacturers was used in the initial conductivity trials; Ensaco 250G manufactured by Timcal Graphite and Carbon, Vulcan XC72R from Cabot Chemical, and conductive CB from Columbia Chemicals Co. Ensaco was selected for its favorable electrical and mixing characteristics. All filled samples used in this study were loaded to 10% by mass by directly mixing the CB into the resin before curing. The uncured mixture was placed in a vacuum chamber (~ 25 mm Hg) for 10 minutes to remove air bubbles. The slurry was then poured into casting molds with embedded copper wire electrodes as shown in Fig. 2. These molds were then cured under pressure (65 psi) in an oven. Tensile samples having



EPON 826, n = 0.085



Jeffamine D-230 (n~2.5)

Decylamine

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